

REPM 2023 BIRMINGHAM

3[®]-7[™] SEPTEMBER 2023 UNIVERSITY OF BIRMINGHAM

27TH INTERNATIONAL WORKSHOP ON RARE EARTH AND FUTURE PERMANENT MAGNETS AND THEIR APPLICATIONS







ABSTRACT BOOK

CONFERENCE CHAIRS:

A. WALTON D. BROWN R. SHERIDAN V. MANN













SUPPORTED BY

REPM2023 Contents

PREFACE

It is a great honour for the UK Permanent Magnet community to welcome all delegates to the 27th International Workshop on Rare-Earth and Future Permanent Magnets and their Applications (REPM 2023) at the University of Birmingham.

The workshop has a long history, since Karl J. Strnat initiated the series of meetings over 45 years ago in 1976. The objective of the workshop is to bring together scientists and engineers working on rare earth permanent magnets and their applications, to facilitate exchange of recent results and ideas on topics such as raw materials, resources, processing, recycling and properties of rare earth and future permanent magnets.

Over 160 abstracts were submitted and these will be presented over four days in 14 oral sessions and three poster sessions. We would like to take this opportunity to thank all who have helped organise this meeting including the international Advisory Committee, the Program Committee and the Local Organising Committee. We also express our gratitude to the organisations that have sponsored the Workshop.

The importance of permanent magnetic materials has never been greater given the impact they have on green technologies and the contribution they will make towards tackling climate change. This will be the first in-person meeting since Covid-19 and as such there has been huge interest from academia, industry and policy makers. On a personal level it will be fantastic to see many colleagues and friends.

The last REPM Workshop held in the UK was also hosted at the University of Birmingham in 1994 by the late Professor Rex Harris. 2022/2023 saw the loss of a number of key members of the magnetic materials community and we intend to commemorate their significant contributions at a session on Tuesday 5th September.

"No one is finally dead until the ripples they cause in the world die away, until the clock wound up winds down, until the wine she made has finished its ferment, until the crop they planted is harvested. The span of someone's life is only the core of their actual existence."

Terry Pratchett

The people we have lost have created massive ripples across the magnetic community.

REPM2023

CONFERENCE CHAIRS

- A. Walton
- D. Brown
- R. Sheridan
- V. Mann

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- S. King
- V. Mann

- R. Sheridan
- L. Venables
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REPM2023

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Particle size distribution of NdFeB powder after grinding and classifying in comparison to conventionally produced product.



Comparison of demagnetization curves of NdFeB magnets

NEW JET MILLING AND CLASSIFYING SOLUTION FOR PROCESSING RARE EARTH ALLOYS

- d₅₀ down to 1 μm
- lower d₉₀ / d₁₀ value
- no product residue after milling
- from lab to production solution

In the manufacturing process of rare earth magnets, the material powder is ground before being pressed and sintered. Its grinding is an essential step as the particle size is of significant importance for the quality and properties of the magnets which are subsequently manufactured from the powder. Ideally the particle size distribution should be narrow and contain an extremely low, ultra-fine fraction (< 2 μ m) and only a small amount of coarse particles (> 8 μ m).

Using jet mills and classifiers made by NETZSCH you can reliably process sensitive NdFeB compounds or other alloys under inert gas conditions and obtain a product with a narrow particle size distribution and a defined upper particle size limit.

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Better can always be improved





Possibility in every drop

High performance recycled magnet manufacturing Sustainably sourced • Low carbon footprint Ethical • Innovative • Green

HYPR®MAG

Magnet Recycling

Founded in 2018 by leading experts in magnetic materials, recycling and hydrogen technologies and with over 100 years combined experience of Rare Earth Elements and Magnets, HyProMag is establishing a short-loop recycling facility for NdFeB magnets in Birmingham, UK and Pforzheim, Germany, with plans to expand into the United States underway.

HyProMag is commercialising the highly energy efficient and patented Hydrogen Processing of Magnet Scrap (HPMS) process which has been developed over many years at the University of Birmingham and uses hydrogen to break down and demagnetise the rare earth magnet embedded in a component. The technology addresses two key issues for recycling magnets embedded in an assembly: economically viable extraction and low energy demagnetisation.

NdFeB magnet recycling is currently very limited and almost exclusively involves scrap being put back in at the chemical processing or solvent extraction stages. Short loop recycling allows us to avoid not only these stages but also electrolysis and casting. HPMS therefore enables manufacturing of rare earth magnets with a significantly reduced carbon footprint, even when compared to other recycling processes.



If you are interested in recycling NdFeB magnets and would like to speak to us about end of life and scrap solutions, or purchasing, magnet and other product sales, contact us at magnets@hypromag.com

For discussions on collaboration or consultation, contact us at technical@hypromag.com



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- Charger-fixture control via PLC interface
- Peak current measurement



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- Multiple fixture outputs
- PLC can be interfaced with automatic handling & safety systems
- Optional custom designed control panel



Custom Built Magnet Setters

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- Energies from 100 200J
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Circular Critical Materials Supply Chains Programme

The CLIMATES project will create a more resilient and sustainable UK through the opportunity of building a stronger supply chain of materials for high-performance magnets, position UK as a world leader in rare earth materials recycling and help meet the UK government's net zero targets.





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- Measurement of all hard magnetic materials acc. to
- international standards in a closed magnetic circuit J-compensated surrounding coils, pole or field coils
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- to 200°C Real time display of hysteresis during measurement





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- Determination of magnetization symmetry of permanent magnetic rotors in the range up to 500 mm diameter and height
- Easy sample mounting for rotors with and without shaft
- High resolution of up to 0.01°
- Customizable construction according to the need of the customer

Eddy Current Loss Tester ECT 200 for permanent magnets

- Determination of the AC power loss in permanent magnets under various magnetization conditions, like sinusoidal magnetization, pulse width modulation or user-defined magnetization
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- frequencies in the range from 50 Hz to 20 kHz Suitable for characterisation of various types of magnets, such as NdFeB, SmCo, AlNiCo



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- Magnetic properties testing of stators in various sizes and geometries
- Detection of influence of manufacturing processes on the magnetic properties of stator cores
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HIRST

Magnetic Instruments Ltd.

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Hirst manufacture a wide range of magnetisers, calibrators, setters, demagnetisers and fixtures. With magnetiser energies from 100J to 400kJ and voltages up to 3000V. Offering a full range of equipment from bench top to volume industrial production solutions.

Applications include

- EV motor production
- Wind turbine generator production
- Recycling equipment production

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Aerospace sensors

Instrumentation

Hirst offer a wide range of measurement solutions including gaussmeters, fluxmeters, scanners and Helmholtz coils.

- Magnet field strength and total flux measurements.
- Quality control and Production testing
- Laboratory equipment.

Magnet Characterisation

Hysteresis measurement of high-performance permanent magnets (NdFeB, ŠmCo) with the new award-winning generation 8 Pulsed Field Magnetometers (PFM) from Hirst

- Full BH curves, Coercivity (Hcj) measurements >3500 kA/m with 10.5T fields
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Over 60 years of delivering world class magnetic solutions

Some example applications include:



Baiqida Intelligent Technology (Ningbo) Co., Ltd

Baiqida Intelligent Technology (Ningbo) Co., Ltd. is located in Fenghua District, Ningbo City, Zhejiang Province, China. With over 20 years of research and production experience in the magnetic materials industry, We are a professional supplier of magnetic material production equipment. Our focus lies in forming equipment and powder processing equipment.

Our flagship equipment, the All-electric Precision Forming & Palletizing Incorporate Equipment, enables unmanned and digitized production of magnetic materials. Compared to traditional press, this approach has higher energy-efficiency and lower carbon emissions. On the other hand, our powder treatment equipment is highlighted by the Vacuum Decrepitation Furnace and Blending Machine. The Vacuum Hydrogen Decrepitation Furnace facilitates hydrogen decrepitation and degassing process of rare earth alloy materials in a vacuum environment. It also allows for the continuous automatic production of hydrogen decrepitation and degassing for recycled magnets.





Baiqida operates through three branches located in Baotou, Yantai, and Mianyang. We have established advanced production bases for magnetic material production equipment and hydrogen decrepitation process, boasting an annual output capacity of 50,000 tons. Furthermore, we possess provincial research centers and laboratories, holding more than 100 patents. Currently, Baiqida is dedicated to advancing the unmanned, intelligent, and energy-saving production within the magnetic material industry.

More information: www.nbbqd.com

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PROGRAMME OVERVIEW

SUNDAY		
16:00 – 19:00	Registration and Welcome Reception	
MONDAY		
08:00 - 08:30	Registration and Arrival Drinks	
08:30 - 09:00	Welcome Address	
09:00 - 09:30	Plenary Talk: Sagawa M	
09:30 – 11:00	[O1] Raw Materials, Resources and Mining	
11:00 – 11:30	Refreshments and Poster Session	
11:30 – 13:00	[O2] Supply Chain Development and Magnet Processing	
13:00 - 14:00	Lunch and Poster Session	
14:00 – 15:30	[O3] Alloy Design and Machine Learning	
15:30 – 16:00	Refreshments and Poster Session	
16:00 – 17:30	[O4] Recycling of Rare Earth Magnets	
17:30 – 19:30	Posters and Drinks Reception	
TUESDAY		
08:00 - 08:15	Registration and Arrival Drinks	
08:15 - 09:00	Memorial to Past Members	
09:00 – 10:30	[O5] Unconventional Processing	
10:30 - 11:00	Refreshments and Poster Session	
11:00 - 12:35	[O6] Nanocrystalline & Thin Film Magnets	
12:35 – 14:30	Lunch and Poster Session	
12:35 - 14:30	International Advisory Committee Meeting	
14:30 – 16:00	[O7] RE-Fe-B Magnet Processing and Properties	
16:00 - 16:30	Refreshments and Poster Session	
16:30 – 18:00	[O8] RE Free Magnets	
18:00 - 19:30	Posters and Drinks Reception	

REPM 2023, 3-7 SEPT, BIRMINGHAM UK

PROGRAMME OVERVIEW

WEDNESDAY	
08:30 - 09:00	Registration and Arrival Drinks
09:00 - 09:30	Plenary Talk: Coey M
09:30 – 11:00	[O9] RE-Co Magnets and Processing
11:00 – 11:30	Refreshments and Poster Session
11:30 - 12:50	[O10] Density Functional Theory (DFT) and Micromagnetic Modelling
12:50 - 14:00	Lunch and Poster Session
14:00 – 15:35	[011] Applications of Permanent Magnets
15:35 - 16:00	Refreshments and Poster Session
15:35 - 17:30	Tour of Magnet Recycling Facilities
19:00 – 23:00	REPM2023 Conference Dinner
THURSDAY	
08:30 - 09:00	Registration and Arrival Drinks
09:00 - 10:40	[O12] Applications and Sustainability of Magnets
10:40 - 11:10	Refreshments
11:10 – 12:30	[O13] Advanced Characterisation
12:30 - 13:30	Lunch
13:30 – 15:00	[O14] RE Nitride Magnets
15:00 - 15:30	Refreshments
15:30 – 17:00	Plenary: Hono K And Closing Remarks





SESSION LIST

Oral Session

- [O1] Raw Materials, Resources and Mining
- [O2] Supply Chain Development and Magnet Processing
- [O3] Alloy Design and Machine Learning
- [O4] Recycling of Rare Earth Magnets
- [O5] Unconventional Processing
- [O6] Nanocrystalline and Thin Film Magnets
- [O7] RE-Fe-B Magnet Processing and Properties
- [O8] RE Free Magnets
- [O9] RE-Co Magnets and Processing
- [O10] Density Functional Theory (DFT) and Micromagnetic Modelling
- [O11] Applications of Permanent Magnets
- [O12] Applications and Sustainability of Magnets
- [O13] Advanced Characterisation
- [O14] RE Nitride Magnets

Poster Session

- [P1] 1a. Raw Materials, Resources and Mining
 - **1b. Supply Chain Development and Magnets Processing**
 - **1c. Recycling of Rare Earth Magnets**
 - 1d. RE-Fe-B Magnet Processing
- [P2] 2a. Unconventional Processing
 - 2b. Nanocrystalline and Thin Film Magnets
 - **2c. RE Free Magnets**
- [P3] 3a. RE-Co Magnets and Processing
 - 3b. Density Functional Theory (DFT) and Micromagnetic Modelling
 - **3c. Applications of Permanent Magnets**
 - **3d. Advanced Characterisation**
 - **3e. RE Nitride Magnets**



Sunday 3rd September 2023

16:00 – 19:00 Registration and Welcome Reception, *The Bramall*

Opportunity to collect your conference resources and to network.

Monday 4th September 2023

08:00 - 08:30 Registration and Arrival Drinks

Poster set-up for Monday poster sessions (P1), The Great Hall

08:30 - 09:00 Welcome Address, Elgar Concert Hall - The Bramall

09:00 - 09:30 Plenary talk, Elgar Concert Hall - The Bramall

Plenary 1

Sagawa M - History of REPM and my Teachers in Permanent Magnets

09:30 – 11:00 [O1] Raw Materials, Resources and Mining Elgar Concert Hall - The Bramall

09:30 - 09:50 - Invited talk. O1-1.

Marte J - MP Materials: Restoring the American Rare Earth Materials and Magnet Supply Chain

09:50 - 10:10 - Invited Talk. O1-2.

Castilloux R - China's Rare Earth Resource Base

10:10 - 10:25 - Oral Talk. O1-3. De Campos M - Recent trends in the Rare-Earth industry in Brazil and in the world

10:25 - 10:40 - Oral Talk. O1-4. Dawes W - Rare Earths and Technology Solutions for the Green Transition

10:40 - 10:55 - Oral Talk. O1-5. Govender I - Development of a Dry Rare Earth Fluorination Process from Carbonates

11:00 - 11:30 Refreshments and Poster Session (P1), The Great Hall

11:30 - 13:00[O2] Supply Chain, Development and Magnets Processing
Elgar Concert Hall - The Bramall

11:30 - 11:50 - Invited Talk. O2-1.

Yang J - Development of rare earth permanent magnet material in China

PROGRAMME & CONTENT(ORAL SESSION)

11:50 - 12:10 - Invited Talk. 02-2.

Yoshida Y – Toward a sustainable supply of high-performance permanent magnets: An overview of efforts taken in Japan

12:10 - 12:25 - Oral Talk. O2-3.

Saje B - Permanent magnets - a European permanent magnets producer view

12:25 - 12:40 - Oral Talk. 02-4.

Liu J – Sm-Co Magnets and RE Supply Chain

12:40 - 12:55 - Oral Talk. O2-5.

Hatch G – Building a resilient critical-raw-materials value chain: the importance of international standards

13:00 - 14:00 Lunch and Poster Session (P1), The Great Hall

14:00 – 15:30 [O3] Alloy Design and Machine Learning

Elgar Concert Hall - The Bramall

14:00 - 14:20 - Invited Talk. O3-1.

Zhang H - Inverse Design of permanent magnets: From high-throughput calculations to machine learning

14:20 - 14:40 - Invited Talk. O3-2.

Hong Y - A high throughput machine learning-assisted study of the effect of element substitution in RE-TM films

14:40 - 14:55 - Oral Talk. O3-3.

Gopalan R - Enhancing the coercivity of Nd-Cu diffused Nd-Fe-B material by Nb assisted grain boundary pinning

14:55 - 15:10 - Oral Talk. O3-4.

Dirba I - Towards maximum utilization of heavy rare earths in sintered NdFeB magnets

15:10 - 15:25 - Oral Talk. O3-5.

Aubert A - Grain boundary engineering in Nd-based $ThMn_{12}$ magnets and their nitrides: A comprehensive study of challenges and limitations

15:30 - 16:00 Refreshments and Poster Session (P1), The Great Hall



16:00 - 17:30 [O4] Recycling of Rare Earth Magnets

Elgar Concert Hall - The Bramall

16:00 - 16:20 - Invited Talk. O4-1.

Walton A -The Works of Late Emeritus Professor Rex Harris

16:20 - 16:40 - Invited Talk. O4-2.

Burkhardt C - An overview of magnets recycling with focus on HPMS

16:40 - 16:55 – Oral Talk. O4-3.

Bacchetta G - Optimizing the short-loop recycling of Dy-rich end-of-life NdFeB permanent magnets

16:55 - 17:10 – Oral Talk. O4-4. Rebernik M - Enhancing Recycled Nd-Fe-B Magnets' Performance via (Single) Grain Boundary Engineering with Nd-Cu using Spark Plasma Sintering

17:10 - 17:25 - Oral Talk. 04-5.

Costa Macedo W - Optimising Magnetic Properties of Recycled Magnets obtained via Pressless Processing (PLP)

17:30 - 19:30 Poster (P1) and Drinks Reception, The Great Hall

Investors Forum Reception (registered attendees only)

Networking and Session: The Bramall Mezzanine / Elgar Concert Hall.

Tuesday 5th September 2023

08:00 - 08:15 - Registration and Arrival Drinks, The Great Hall

Poster set-up for Tuesday Poster Sessions (P2)

08:15 - 09:00 - Memorial to Past Members, Elgar Concert Hall - The Bramall

09:00 – 10:30 [O5] Unconventional Processing Elgar Concert Hall - The Bramall

Elgar Concert Hall - The Brama

09:00 - 09:20 - Invited Talk. - O5-1.

Brooks O - The Influence of the Disproportionated Microstructure on the Hydrogen Ductilisation Process (HyDP) for NdFeB Alloys

09:20 - 09:40 - Invited Talk. - 05-2.

Sugimoto S - Development of High Anisotropic Nd-Fe-B HDDR Powders

09:40 - 09:55 - Oral Talk. - O5-3.

Goll D - Rare earth based permanent magnets by laser powder bed fusion

09:55 - 10:10 – Oral Talk. - O5-4.

Shield J - Solidification-based Additive Manufacturing of Rare Earth Permanent Magnets

10:10 - 10:25 - Oral Talk. - O5-5.

Lewis L - Tailoring Magnetic Structures via Passive Magnetic and Strain-Field Annealing

10:30 - 11:00 Refreshments and Poster Session (P2), The Great Hall

11:00 - 12:35[O6] Nanocrystalline & Thin Film Magnets
Elgar Concert Hall - The Bramall

11:00 - 11:20 - Invited Talk. - O6-1

Sepehri Amin H - Exploring coercivity limit in SmFe₁₂-based thin films

11:20 - 11:35 - Oral Talk. - O6-2

Romero S - Magnetostatic coupling in NdFeB melt-spun magnets

11:35 - 11:50 - Oral Talk - O6-3

Tang X - (Nd,LRE)-Fe-B hot-deformed magnets for variable-magnetic-force motor applications

11:50 - 12:05 - Oral Talk. - 06-4

Kautsar Z - High Coercivity and Resistivity HRE-free Nd-Fe-B hot-deformed magnet by PrCu diffusion and CaF,-LiF eutectic mixture addition

12:05 - 12:20 – Oral Talk – O6-5

Staab F - Hard magnetic $SmCo_5$ -Cu nanocomposites produced by high-pressure torsion – microstructural evolution and magnetic properties

12:20 - 12:35 - Oral Talk. - O6-6

Kim G - Dual amorphous-precursor deformation method for high-performance Nd-saving Nd-Fe-B hot-deformed magnets

12:35 - 14:30 Lunch and Poster Session (P2), The Great Hall

12:35 – 14:30 International Advisory Committee Meeting, Careers Networking Rooms, (G30-G31)

14:30 – 16:00 [O7] RE-Fe-B Magnet Processing and Properties Elgar Concert Hall - The Bramall

14:30 - 14:50 - Invited Talk. - 07-1

Sasaki T - Achieving 2.8 T coercivity in Nd–Fe–B sintered magnets subjected to two-step grain boundary diffusion process

14:50 - 15:10 - Invited Talk. - 07-2

Miyawaki H - Heavy Rare Earth free Hot-Deformed Nd-Fe-B Magnet for Traction Motor in Electric Vehicles

PROGRAMME & CONTENT (DRAL SESSION)

15:10 - 15:25 - Oral Talk. - 07-3

Jia Z - Effect of grain boundary reconstruction and regenerated main phase shell on magnetic properties in high-abundance (NdLaCeY)-Fe-B magnets

15:25 - 15:40 - Oral Talk. - 07-4

Gassmann J - Reducing criticality of Nd-Fe-B by recycling and partial substitution using the rapid quenching technique

15:40 - 15:55 - Oral Talk - 07-5

Tomše T - A strategy for rapid sintering of Nd-Fe-B-type magnets based on intense thermal radiation

16:00 - 16:30 Refreshments and Poster Session (P2), The Great Hall

16:30 – 18:00 [O8] Rare Earth-Free Magnets Elgar Concert Hall - The Bramall

16:30 - 16:50 - Invited Talk. - O8-1

Johnson F - Iron Nitride: a Non-Rare-Earth Containing Permanent Magnet

16:50 - 17:05 – Oral Talk. O8-2 Woodcock T - Critical Materials Free MnAI-C Magnets: Recent Developments and a Perspective

17:05 - 17:20 - Oral Talk. 08-3

Zhao P - Nanoscale Chemical Segregation to Twin Interfaces in T-MnAI-C and Resulting Effects on the Magnetic Properties

17:20 - 17:35 – Oral Talk – O8-4 Capobianchi A - Direct synthesis of highly ordered L1₀-FeNi nanoparticles from Layered complexes

17:35 - 17:50 – Oral Talk – O8-5 Lewis L - Jumping the Gap: Can Tetrataenite become a "Hard" Permanent Magnet?

17:50 - 18:00 – Oral Talk – O8-6 Liu P - Hard and semi-hard Fe-based magnetic materials

18:00 - 19:30 Posters and Drinks Reception, The Great Hall

Wednesday 6th September 2023

08:30 - 09:00 Registration and Arrival Drinks, *The Great Hall* Poster set-up for Wednesday Poster Sessions (P3)

09:00 - 09:30 Plenary Talk, Elgar Concert Hall - The Bramall

Plenary 2 Coey M - Why is it difficult to make a new permanent magnet?

PROGRAMME & CONTENT(ORAL SESSION)

09:30 - 11:00 [O9] RE-Co Magnets and Processing

Elgar Concert Hall - The Bramall

09:30 - 09:50 - Invited Talk. - O9-1

Hadjipanayis G - Towards the adaptation of $Sm(Fe,Co,M)_{12}$ compounds for new rare-earth-lean permanent magnets

09:50 - 10:05 - Oral Talk. - O9-2

Castro J - Anisotropy field determination in SmCo₅ sintered magnets

10:05 - 10:20 - Oral Talk. - O9-3

Park K - Nanocrystalline Sm-Co-Cu bulk magnets prepared by low-oxygen nanopowder metallurgy process

10:20 - 10:35 - Oral Talk. - O9-4

Sheridan R - Strip Casting of Sm₂TM₁₇-type Alloys for Production of the Metastable SmTM₇ Phase

10:35 - 10:50 - Oral Talk. - O9-5

Niarchos D - Rare Earth and Transition Metal based High Entropy Alloys (HEAs) as building blocks for novel permanent magnets

11:00 - 11:30 Refreshments and Poster Session (P3), The Great Hall

11:30 - 12:50 [O10] Density Functional Theory (DFT) and Micromagnetic Modelling Elgar Concert Hall - The Bramall

11:30 - 11:50 - Invited Talk. - O10-1

Patrick C - First-principles calculations on rare earth magnets - bridging the gap between theory and reality

11:50 - 12:05 - Oral Talk. - O10-2

Staunton J - Crucial role of Fe in determining the hard magnetic properties of $Nd_2Fe_{14}B$: finite temperature, first-principles theory calculations

12:05 - 12:20 - Oral Talk - O10-3

Kulesh N - Exploring coercivity limits in ultrafine-grained Nd-Fe-B magnets with deep learning image segmentation and micromagnetic simulation

12:20 - 12:35 - Oral Talk - O10-4

Ohmer D - Micromagnetic simulation of nanostructured Sm₂(Co,Fe,Cu,Zr)₁₇ magnets



12:35 - 12:50 - Oral Talk - O10-5

Paudyal D - Discovery of rare-earth lean high-performance permanent magnets

12:50 - 14:00 Lunch and Poster Session (P3), The Great Hall

14:00 – 15:35 [O11] Applications of Permanent Magnets Elgar Concert Hall - The Bramall

14:00 - 14:20 - Invited Talk. - O11-1

Chong E - The Application of Permanent Magnets in Electrical Machines for Aerospace Full Electric and Hybrid Electric Aircrafts

14:20 - 14:35 - Oral Talk. - O11-2

Perigo E - Considerations in Magnets Selection for Industrial Motors: The Case of Permanent Magnet-Assisted Synchronous Reluctance Motors

14:35 - 14:50 - Oral Talk. - O11-3

Krengel M - Special features of the NdFeB Hot pressing process- a pump manufacturer as magnet producer

14:50 - 15:05 - Oral Talk. - O11-4

Vishwakarma A – Geometry-based reduction of rare-earth-containing raw materials for permanent magnets

15:05 - 15:20 - Oral Talk - O11-5

Klumpp A - Challenges in applications of permanent magnets in accelerators on the example of the major accelerator upgrade at DESY

15:20 - 15:35 - Oral Talk - O11-6

Dempsey N - Development and use of NdFeB micro-magnets

15:35 - 16:00 Refreshments and Poster Session, The Great Hall

15:35 - 17:30 Tour of Magnet Recycling Facilities Sign up at registration desk

19:00 - 23:00 REPM2023 Conference Dinner - The Council House Victoria Square Birmingham, B1 1BB

Pre-Dinner Talk

Plenary 3

Morcos T - Karl J. Strnat and the University of Dayton Magnetics Laboratory - Their Contributions to the Permanent Magnet Industry



PROGRAMME & CONTENT(ORAL SESSION)

Thursday 7th September 2023

08:30 - 09:00 - Registration and Arrival Drinks, The Great Hall

09:00 – 10:40 [O12] Applications & Sustainability of Magnets Elgar Concert Hall - The Bramall

09:00 - 09:20 - Invited Talk. - O12-1

Gutfleisch O - Sustainability of tomorrow's magnets and their applications

09:20 - 09:40 - Invited Talk. - 012-2

Blomgren J - Automated High-Speed Approaches for the Extraction of Permanent Magnets from Hard Disk Drives Components for Circular Economy

09:40 - 09:55 - Oral Talk.- 012-3

Delette G - Design of complex shaped REPM for increasing the performance and recyclability of synchro-reluctant machines

09:55 - 10:10 - Oral Talk. - 012-4

Grau L - Processability and separability of commercial anti-corrosion coatings in HPMS recycling of NdFe

10:10 - 10:25 - Oral Talk. - 012-5

Dias M - Improvement of magnetic properties of recycled Nd-Fe-B magnets by employing the GBD process

10:25 - 10:40 - Oral Talk. - O12-6

Ghorbanighoshchi S Recycling of Sr-ferrite Permanent Magnet Scraps

10:40 - 11:10 Refreshments, The Great Hall

11:10 – 12:30 [O13] Advanced Characterisation Elgar Concert Hall - The Bramall

11:10 - 11:25 - Oral Talk. - O13-1

Cheema N - New measurement system for evaluation of eddy current loss in permanent magnets

11:25 - 11:40 - Oral Talk. - 013-2

Mouron R - Anisotropic lattice diffusion of heavy rare earth elements (Tb, Dy) in the magnetic phase of Nd-Fe-B permanent magnets: an experimental model

11:40 - 11:55 - Oral Talk. - O13-3

Han C - Microscopic Consequences of Strain and Magnetic Field on Atomic Ordering in MnAl

11:55 - 12:10 - Oral Talk. - O13-4

Xia W - In-situ Magnetizing Holder with Strong In-plane Field for Lorentz Microscopy

12:10 - 12:25 - Oral Talk. - O13-5

Ishigami K - Understanding the Coercivity of Ga-containing Nd–Fe–B Sintered Magnets from Feature Extraction and Selection of X-ray Diffraction Patterns via Dimension Reduction and Sparse Modeling

12:30 - 13:30 Lunch, The Great Hall

13:30 – 15:00 [O14] Rare Earth Nitride Magnets Elgar Concert Hall - The Bramall

13:30 - 13:50 - Invited Talk. - O14-1

Iriyama T - Recyclable Sm-Fe-N Bonded-Magnet Using Environmentally-Friendly CNF Binder

13:50 - 14:05 – Oral Talk. – O14-2 Hirayama Y - Magnetically anisotropic nanopowder of TbCu,-type Sm-Fe-N

14:05 - 14:20 - Oral Talk. - 014-3

Luca S - Sintering of Nd(Fe,Mo)₁₂ tetragonal compounds for permanent magnets applications

14:20 - 14:35 - Oral Talk. - O14-4

Yamaguchi W - Improvement of coercivity of $Sm_2Fe_{17}N_3$ powder by coating with material designed to restore local magnetic anisotropy on the surface

14:35 - 14:50 – Oral Talk. – O14-5 Matsuura M - Microstructural Changes at The Sm_2Fe_{17} and Zn Interface

15:00 - 15:30 Refreshments, The Great Hall

15:30 – 17:00 Plenary Talk and Closing Remarks

Plenary 4 Hono K - Permanent Magnet Research at NIMS - ESICMM Succeeded by Permanent Magnet Materials Open Platform and DXMag



Monday 4th September 2023

P1-1

Tanji Karim - Recycling NdFeB waste based on a hydrometallurgical process for recovering Nd, Dy, and Pr rare earth elements: Design experiment screening and optimization

P1-2

Balasubramanian B - Nd₂Fe₁₄B-based magnets from atmoized powders

P1-3

Di J - Macroscopic demagnetisation behaviour of sintered Nd-Fe-B magnets by grain boundary diffusion of Tb

P1-4

Morcos T - Competitive Threats to NdFeB Magnets in Green Energy Applications - Electric Vehicles and Windpower

P1-5

Griffiths J - Hydrogen Decrepitation and Reprocessing of SmCo₅ Sintered Magnets

P1-6

Rathfelder S - Production of anisotropic permanent magnets from recycled Nd-Fe-B powder with metal injection and powder extrusion moulding

P1-7

Nayebossadri S - Hydrogen-assisted recycling of Nd-Fe-B magnets from the end-of-life audio products

P1-8

Bolis K - Characterization of Nd-Pr-Fe-B machining wastes for recycling purposes

P1-9

Khoshsima S - Processing of grinding slurries of Samarium Cobalt magnets for a circular economy

P1-10

Schieren L - Development of a process to remove the neodymium-rich phase from recycled NdFeB magnet powder through leaching with organic acid

P1-11

Mishra A - Chemical Recycling of Nd-Fe-B Permanent Magnets: A Sustainable Solution for the Nd2Fe14B Matrix Phase Recovery

P1-12

Schönfeldt M - Facing the challenges for a circular and sustainable rare earth permanent magnet recycling

P1-13

Leonowicz M - Tailoring the magnetic properties of multipole anisotropic magnets by using a mixture of commercial and recycled anisotropic NdFeB powders

P1-14

Petavratzi E - Dynamic stocks and flows analysis of NdFeB permanent magnets in the UK electric vehicles and wind turbines

P1-15

Podmiljšak B - Intelligent and Sustainable Processing of Innovative Rare-Earth Magnets

P1-16

Kozak V - Optimisation of grain boundary phase separation in hydrogen processed NdFeB

P1-17

Hussain S - Investigation of oxidation behaviour and passivation rates for recycled NdFeB powders

P1-18

Awais M - Extraction of NdFeB Magnets From End-of-Life Electric Vehicle Scrap, Conversion into Master Alloy and Sintered Magnets

P1-19

Rosa M - From Scrap to Magnet: On the Use of HDDR Recycled Feedstocks in the Additive Manufacturing of Composite Magnets

P1-20

Pickering L - Investigation into the Influence of Zn Coatings on Recycling of Sintered NdFeB-type Magnets

P1-21

Bacchetta G - Microstructure and phase transitions during the high temperature hydrogenation disproportionation of various NdFeB alloys

P1-22

Chang H - Coercivity enhancement of sintered NdFeB magnets by doping $R_{_{85}}AI_{_{15}}$ (R=Ce and Pr) alloys followed with grain boundary diffusion

P1-23

Yuhao L - Design of core-shell structure and magnetic property enhancement mechanism of dualmain-phase high-abundance Ce based sintered magnets

P1-24

Lostun M - Tuning the hard magnetic properties of melt-spun Misch Metal-Fe-Hf-B ribbons by heat treatment

P1-25

Ahmad T - Novel processes for the manufacturing of fine-grained Nd-Fe-B powders with steep particle size distribution

P1-26

Lai R - Attaining excellent coercivity and thermal stability in Nd-Ce-Fe-B sintered magnets

P1-27

Zhang Y - Coercivity Enhancement in (PrNd, Ce)-Fe-B Sintered Magnets: Effect of Grain Boundary Reconstruction by Low Melting Point Alloys

P1-28

Bae K - Effect of Co content of Nd-Fe-B sintered magnet on grain boundary diffusion process of TbH_{3} solution

P1-29

Opelt K - Investigation of the Diffusion Behavior for Heavy Rare Earths for Nd-Fe-B Sintered Magnets Produced by the 2-Powder Method

P1-30

Lin C - 2-Powder Method for Improving the Magnetic Properties of Ce-Containing Nd-Fe-B Magnets

P1-31

Prabhu D - Coercivity enhancement through grain boundary diffusion in sintered Nd-Fe-B magnet using DyF_3

P1-32

Costa Macedo W - On the Changes in Microstructure and Magnetic Properties of Rare Earth-based Permanent Magnets processed via Grain Boundary Diffusion

Tuesday 5th September 2023

P2-1

Luca S - Investigation of the microstructural features induced by the reaction of NdFeB powders with the degradation products during the debinding step in the Powder Injection Molding process

P2-2

Burkhardt F - An investigation into the influence of zirconium additions and processing conditions on $Nd_{12,2}Fe_{813}B_{6,5}$ strip-cast material during the Hydrogen Ductilisation Process (HyDP)

P2-3

Bulyk I - New possibilities for the use of the HDDR process in the $Nd_2Fe_{14}B$ -, $SmCo_5$ - and Sm_2Co_{17} based ferromagnetic materials

P2-4

Bulyk I - Recent about HDDR process in R-Fe-B - based materials and magnets

P2-5

Kim H - The Effect of the Solidification Microstructure on Magnetic Properties of Nd-Fe-B Magnet Manufactured by Laser Powder Bed Fusion

P2-6

Tosoni O - Rare-earth free Nd-Fe-B magnets with high coercivity by Laser Powder Bed Fusion

P2-7

Liu J - Additive Manufacturing of (Pr,Nd)-Fe-Cu-B Permanent Magnets

P2-8

Powell P - The effect of varying Nd content in NdFeB alloys on the Hydrogen Ductilisation Process (HyDP)

P2-9

Gitti Tortoretto Fim R - Laser Powder Bed Fusion of anisotropic Nd-Fe-B bonded magnets – the use of an in situ mechanical particle alignment approach

P2-10

Rosa M - The Use of Pr-Fe-Co-B HDDR Powders to Obtain Composite Magnets via Additive Manufacturing

P2-11

Chang H - Comparison on the improved magnetic properties of melt spun RCo_5 (R = Y, La, Ce, and Pr) ribbons due to Fe and C-doping

P2-12

Aubert A - Microstructure, coercivity and thermal stability of nanostructured (Nd,Ce)-(Fe,Co)-B hotcompacted permanent magnets

P2-13

Maccari F - Nanocrystalline Nd-Fe-B anisotropic magnets by flash spark plasma sintering

P2-14

Cabassi R - Steps towards the hardening of Cobalt ferrite nanoparticles

P2-15

Mori Y - Suppression of the formation of soft magnetic phase for $Sm(Fe-Co)_{12}$ -B thin films by introduction of seed layer

P2-16

Yoo J - Fabrication of high-remanence Nd-Fe-B hot-pressed magnets by manipulating coercivity of initial anisotropic HDDR powders

P2-17

Jang Y - Enhancement of crystal alignment and remanence of large-scaled Ce-substituted Nd-Fe-B magnet by hot deformation

P2-18

Payattuvalappil A - A Novel Approach for the Detection of the L10 Phase in FeNi

P2-19

Winkler J - Establishing the ultrasonic-atomization of MnAI-based alloys for the hot-extrusion of rareearth-free permanent magnets

P2-20

Arneitz S - Investigation on the influence of printing parameters on the magnetic properties of an AM rare earth free alloy

P2-21

De Campos M - Role of exchange energy on the relationship between coercivity and grain size: Application for hard ferrites

P2-22

Ohkubo T - Effect of processing methods and Cu-doping on the coercivity of $SmFe_{12}$ -based sintered magnets

P2-23

Maccari F - Effect of aging on the magnetic and physical properties of consolidated Mn-Al-C

P2-24

Zhang J - Microstructure and magnetic properties evolution in anisotropic Sm(Fe,Ti,V)₁₂-based sintered magnets by composition modification

P2-25

Niarchos D - Bulk L10-FeNi: A novel approach towards the tetragonal phase

P2-26

da Silva V - The possible formation of tetrataenite by mechanical activation and the addition of light elements.

P2-27

Dirba I - Synthesis and magnetic properties of bulk α'' -Fe₁₆N₂/SrAl₂Fe₁₀O₁₉ composite magnets

P2-28

Davis-Fowell E - Investigating Plane Strain Compression as a route to improve extrinsic magnetic properties of MnAlGa

Wednesday 6th September 2023

P3-1

Ti E - The effect of Fe, Ni substitution on Phase balance in SmCo₅ Alloys

P3-2

Brooks O - Changes in Resistivity with Temperature for SmCo type Sintered Magnets

P3-3

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Furusawa D - Influence of Oxygen Amount to Constituent Phases in Hot Isostatic Press (HIP)-
processed Sm-Y-Fe-based Alloy with ThMn, Compound
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P3-4

Hadjipanayis G - Sm-Fe(Co)-Ti phase equilibria and liquid-phase sintering of Sm(Fe,Co,Ti), magnets

P3-5

Hadjipanayis G - High coercivity in monocrystalline (Sm,Zr)(Fe,Co,Ti)₁₂ particles prepared via high-temperature calcium reduction of oxides

P3-6

Tozman P - Magnetic properties and phase diagram of Sm-Co-B: An exploratory study

P3-7

Castro J - Atomistic calculation of the domain wall energy in rare-earths with hexagonal structure

P3-8

Faria R - Electrical analogous for permanent magnets

P3-9

Bolyachkin A - Tomography-based Micromagnetic Simulations of Nd-Fe-B Magnets: the Role of Intergranular Phase Nonuniformities

P3-10

Werwiński M - Effect of transition metal doping on magnetic hardness of $CeFe_{12}$ -based compounds: DFT study

P3-11

Ferrer N - Investigating the role of interactions on the stability of magnetic anisotropy in $L1_0$ magnetic materials

P3-12

Fischbacher J - Micromagnetic study of the impact of grain boundaries on coercivity

P3-13

Zou M - Localized Demagnetization Between Unequally Sized Like Magnetic Poles and FEA Determined Occurrence Conditions for Applications

P3-14

Ahmed Y - Polymerisation of Barium Hexaferrite Ferrofluids Producing a Magnetic Nanoparticle Composite Matrix

P3-15

Baek Y - Facile Synthesis of Epsilon Ferrites via Spray Drying for Millimeter-wave Absorption

P3-16

Ye X - Voltage-driven giant modulation of magnetism in permanent magnets

P3-17

Hedlund D - Hard Magnetic Phases of CeFe₁₁W_{1-x}Ti_x

P3-18

Niarchos D - 1D-2D Assemblies of sub-millimeter NdFeB -based magnets

P3-19

Hohs D - A method for quality assessment of FeNdB sintered magnets

P3-20

Romero S - Recoil curves as a tool to identify coercivity mechanisms

P3-21

Martinek G - Vectorial Rare Earth Magnet Hysteresis Loops Measured in a Biaxial Vibrating Sample Magnetometer

P3-22

Hrushko O - Thermal stresses in the Nd₂Fe₁₄B polycrystal system with grain boundary phase

P3-23

Suwa T - Feature extraction of 3D real pictures of microstructure and magnetic domain in a Tb diffused Nd-Fe-B sintered magnet

P3-24

Günzing D - Insights into hot-deformed anisotropic $Nd_2Fe_{14}B$ magnets: Interaction domains studied by non-destructive 3D magnetic laminography and magnetometry

P3-25

Hosokawa A - Low oxygen Sm₂Fe₁₇N₃ sintered magnets produced from ball-milled powder

P3-26

Kim J - Synthesis Mechanism of Sm-Fe compounds in Low-Temperature Reduction-Diffusion Process

P3-27

Grigoras M - Preparation by gas atomization of elongated powders α"-Fe₁₆N₂ for anisotropic magnets

P3-28

Vijayaragavan G - Coercivity enhancement on Sm-Fe-N using novel low melting Zn based eutectic alloy



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION

WELCOME ADDRESS AND PLENARY TALK

PLENARY 1 - M. SAGAWA

MONDAY 4 SEPTEMBER 09:00 - 09:30 ELGAR CONCERT HALL - THE BRAMALL


Plenary - 1

History of REPM and my Teachers in Permanent Magnets

M. Sagawa

Daido Steel Co., Ltd. Nagoya, Aichi 457-8545, Japan

Welcome Address and Plenary Talk, September 4, 2023, 09:00 - 09:30

Our Workshop was started in Dayton, USA, in 1974 as the name of "The First International Workshop on Rare Earth-Cobalt Permanent Magnets and their Applications." The name has been changed in 1985 to "The 8th International Workshop on Rare Earth Permanent Magnets and their Applications," after the presentations of the Nd-Fe-B sintered and melt spun permanent magnets in 1983. Since then, our Workshop has been called in short as REPM. On this occasion, as the inventor of the Nd-Fe-B sintered magnets, I'd like to explain how I have been affected from the society of REPM. I have four teachers in this society: Prof. Karl Strnat, Prof. Alden Ray, Prof. Rex Harris and Prof. Helmut Kromuller. The REPMs are becoming more and more important. Let's contribute to human society by developing basic researches and technologies on REPMs.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (01)

SESSION 1: RAW MATERIALS, RESOURCES AND MINING

MONDAY 4 SEPTEMBER 09:30 - 11:00 ELGAR CONCERT HALL - THE BRAMALL



MP Materials: Restoring the American Rare Earth Materials and Magnet Supply Chain

J. Marte, E. Pang, A. Martinez, M. Sloustcher, A. Lund Magnetics, MP Materials, Fort Worth, Texas, USA

1. Raw Materials, Resources and Mining, September 4, 2023, 09:30 - 11:00

MP Materials is the largest rare earth materials producer in the Western Hemisphere. The Company owns and operates America's only significant rare earth element (REE) mine and processing site in Mountain Pass, California. MP's mission is to restore the full rare earth supply chain to the United States by implementing a three-stage strategy to produce REE concentrate (Stage I), separated REEs (Stage II), and NdFeB sintered magnets (Stage II).

In this talk, we will cover the progress MP Materials has made since its inception in 2017. We will provide an update on the efficiency of our Stage I operations and the exciting progress of our light and heavy REE separations plant at Mountain Pass (Stage II). We will then review the sintered NdFeB magnet factory we are building in Fort Worth, Texas (Stage III), highlighting future development and production capabilities.



ONDAY

China's Rare Earth Resource Base

R. Castilloux

Adamas intelligence, 500 King St. W. 3rd Floor, Toronto, Ontario, Canada, M5V 1L9

1. Raw Materials, Resources and Mining, September 4, 2023, 09:30 - 11:00

From 2023 through 2040, Adamas Intelligence forecasts that global demand for NdFeB magnets will increase at a CAGR of 7.5%, bolstered by double-digit growth from electric vehicle and wind power sectors, translating to comparable demand growth for the critical rare earths elements (i.e., didymium, dysprosium and terbium) these magnets contain.

By 2040, China's didymium oxide resource base will be down to less than 50 years of supply if the nation sustains its market share going forward, or less than 25 years of global supply should China supply all of tomorrow's demand alone.

This reality, coupled with ongoing efforts to develop alternative mine-to-magnet supply chains in Europe and North America, presents a generational opportunity for emerging producers of rare earth oxides outside of China in the years ahead.



MONDAY

01-3

Recent Trends in the Rare-earth Industry in Brazil and in the World

<u>**M. F. De Campos**</u>, J. A. de Castro

UFF - Federal Fluminense University, Volta Redonda, BRAZIL

1. Raw Materials, Resources and Mining, September 4, 2023, 09:30 - 11:00

The present market and production of rare-earths is reviewed, with special attention for events recently occurring in Brazil.

The rare-earth market is still driven by RE-Fe-B magnets (RE=Rare Earth). There is increasing demand for elements used in RE-Fe-B type magnets, which are Nd, Pr, Dy, Tb and also Ho and Gd. The rare-earth prices skyrocketed last year, but already decreased significantly now in April/2023.

Traditionally, three are the main applications of rare-earths: (i) magnets, (ii) luminescent phosphors (iii) catalysis.

Rare earths as Yb, Tb, Eu and Y are used in luminescent phosphors in varied applications as for example optical fibers, but the needed volume in these applications is not significant as that of magnets. There is increasing demand for Yttrium, especially for ceramics, as for example Yttria-stabilized Zirconia.

Applications in electric vehicles , where each electric car uses ~1.-2 kg of REFeB magnets are pushing the rare-earth demand. In some countries as Germany and China, plug-in electric vehicles already reached 15% or more of the sales.

RE-Fe-B magnets for motors with high temperature operation made the prices of Dy and Tb increase significantly.

Thus, mines with ionic clays became more attractive, because monazite and bastnaesite have predominance of light rare-earths as Ce, La, Nd and Pr.

Many mining projects have been recently announced in Brazil:

One of such projects aims to extract heavy rare-earths as Dy and Tb from tailings of tin mines, as for example in Ariquemes-RO. North-American company Energy Fuels acquired the site near Prado-BA, where monazite, zirconite and rutile can be found. Serra Verde is making movements to start ore extraction in Minaçu-GO, an ionic clay project.

Many acquisitions of deposits with heavy rare-earths occurred in Brazil recently, by North-American and Canadian companies. However, the rare-earth production in Brazil is still very small.



Mkango - Rare Earths and Technology Solutions for the Green Transition

W. Dawes

Mkango Resources, London, UK

1. Raw Materials, Resources and Mining, September 4, 2023, 09:30 - 11:00

Mkango's corporate strategy is to develop new sustainable primary and secondary sources of neodymium, praseodymium, dysprosium and terbium to supply accelerating demand from electric vehicles, wind turbines and other clean technologies.

Mkango is developing its flagship Songwe Hill rare earths project in Malawi with a Definitive Feasibility Study completed in July 2022 and an Environmental, Social and Health Impact Assessment approved by the Government of Malawi in January 2023.

In parallel, Mkango and Grupa Azoty PULAWY, Poland's leading chemical company and the second largest manufacturer of nitrogen and compound fertilizers in the European Union, have agreed to work together towards development of a rare earth Separation Plant at Pulawy in Poland. The Pulawy Separation Plant will process the purified mixed rare earth carbonate produced at Songwe Hill.

Through its 90% ownership of Maginito (www.maginito.com), Mkango is also developing rare earth magnet recycling in UK, Germany, US and other jurisdictions. Following a transaction announced earlier this year, Maginito holds a 100% interest in HyProMag Ltd focused on short loop rare earth magnet recycling in the UK, a 90% direct and indirect interest (assuming convertible loan conversion) in HyProMag GmbH, focused on short loop rare earth magnet recycling in Germany, and a 100% interest in Mkango Rare Earths UK Ltd, a company focused on long loop rare earth magnet recycling in the UK. A new US subsidiary, to be jointly owned by Maginito and CoTec, will be formed to develop rare earth recycling opportunities in the United States.

HyProMag is targeting first production from the UK in 2023 and Germany in 2024, with parallel technology roll-out into the US. Major competitive advantages in the rare earths magnet recycling sector include access to highly energy efficient, patented HPMS technology and the ability to manufacture rare earth magnets with significantly reduced carbon footprint



Fluorination of the Rare Earth Carbonates and Oxides

I. Govender¹, R. Pretorius¹, K. Wagener², J. le Roux², D. van Vuuren³, P. Crouse³ ¹Metal Refining Engineers, Pretoria, South Africa, ²Necsa, Pelindaba, South Africa, ³University of Pretoria, Pretoria, South Africa

1. Raw Materials, Resources and Mining, September 4, 2023, 09:30 - 11:00

Rare earth fluorides are required as a precursor to produce the rare earth metals for application in permanent magnets. The production of the NdPr alloy occurs via molten salt electrolysis of $(Nd,Pr)_2O_3$ while the production of the Tb and Dy metals occurs via metallothermic reduction of TbF3 or DyF3 with Ca metal. The production of the metals thus requires a consistent source of RE fluorides.

Traditionally, fluorination of the rare earth carbonates and oxides are performed using two routes. In the dry route, rare earth oxides are contacted with anhydrous hydrogen fluoride (AHF) at 600 °C. In the wet route, rare earth carbonates are initially chlorinated before reaction with aqueous hydrogen fluoride to precipitate the rare earth fluorides. Both of these routes have disadvantages. In the dry route, expensive materials of construction are required, and inconsistent product quality is common. In the wet route, large effluent streams are generated that require treatment.

A novel dry fluorination process was developed for the production of rare earth fluorides using an environmentally friendly process. The process produces rare earth fluorides in a batch process by reacting rare earth carbonates or oxalates with AHF at 125 °C and atmospheric pressure. The waste produced is water vapour and carbon dioxide. Any excess hydrogen fluoride may be reacted to extinction or scrubbed using wet or dry abatement systems. The technology has been demonstrated on a semi-commercial scale, in a facility that produces 80 tpa rare earth fluorides. Lower temperature operation allows the use of less exotic materials of construction and limits energy input requirements resulting in optimised CAPEX and OPEX.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (02)

SESSION 2: SUPPLY CHAIN DEVELOPMENT AND MAGNET PROCESSING

MONDAY 4 SEPTEMBER 11:30 - 13:00 ELGAR CONCERT HALL - THE BRAMALL



Development of Rare Earth Permanent Magnet Material in China

<u>J. Yang</u>

State Key Laboratory of Mesoscopic Physics and Department of Physics, Peking University, Beijing 100871, P. R. China

2. Supply Chain Development and Magnet Processing, September 4, 2023, 11:30 - 13:00

Rare earth permanent magnet materials have become indispensable functional materials in modern society, which play an essential role in energy generation and conversion applications. With the fast development of green-energy technologies such as electric transportation, wind power generation, and precision actuation, demand for permanents with high energy density, high thermal stability, and low cost has become unprecedented. As a result, the application of rare earth in manufacturing permanent magnet materials in China has increased by more than 20 times in the past 20 years. The rare earth permanent magnet material industry, represented by sintered neodymium iron boron magnets, has become the fastest-growing and most significant industry in rare earth applications. In 2021, the production of rare earth permanent magnet materials in China with an important position and significant international influence. This talk will focus on China's current development status of rare earth permanent magnet materials.



Toward a Sustainable Supply of High-performance Permanent Magnets: An Overview of Efforts Taken in Japan

Y. Yoshida¹, H. Miyawaki², T. Iriyama³

¹Daido Steel (America) Inc., VIctoria, Canada, ²Daido Steel Co., Ltd., Nakatsugawa, Japan, ³Daido Steel Co., Ltd., Nagoya, Japan

2. Supply Chain Development and Magnet Processing, September 4, 2023, 11:30 - 13:00

As the world begins the critical work of transitioning to a sustainable society, we, as members of the magnet manufacturing industry, need to accelerate this transition by tackling major challenges such as CO₂ reduction in our processes, stabilization of magnet supply, and efficient use of natural resources. For rare earth magnets in particular, the biggest problems we are facing are the environmental and geopolitical issues of the heavy rare earth raw materials supply chain and unbalanced use of rare earth resources driven by the rapid increase of Nd demand.

This presentation begins with an overview with regards to the efforts taken in Japan to stabilize the supply chain based on the published information. Then measures taken for the reduction of heavy rare earth use, the utilization of surplus rare earth elements, and efforts to develop rare earth-free magnets will be reviewed. Finally, as examples of these measures, the current status of hot-deformed NdFeB magnets and SmFeN magnets at Daido will be presented.



Permanent Magnets – an European Permanent Magnets Producer View

<u>B. Saje</u>¹, K. Kosmač², A. Drmota-Petrič¹

¹Kolektor Mobility d.o.o, R&D Magnetics, Vojkova 10, 5280 Idrija, Slovenia, ²Kolektor KFH d.o.o., CML, Vojkova 10, 5280 Idrija, Slovenia

2. Supply Chain Development and Magnet Processing, September 4, 2023, 11:30 - 13:00

Presented discussion under the above title should start with disclaimer reading »On current trends major share in permanent magnets production has China«. Then Japan. Rest of us is tiny fraction.

But, in Europe you still find some rare spots trying to process RE based, either magnets or RE containing alloys to be used in permanent magnet industry. This presentation will attempt to summarise some European activities which are part parts of:

- RE European based magnets history,
- Availability as to European capacity offered on the market (current status)
- Recycling efforts of End Of Life permanent magnets
- And some (potentially) future materials and technologies.

Since around 80% by weight are ferrites, and 70% of them end in various motors and around 65% by USD is NdFeB, and about 70% of them end in motors, focus of this talk will be more on RE-TM magnets, not so much on ferrite.

It is also going to address activities with RE's declared strategic and critical which triggered some of interesting mind sets:

- Less Rare Earths or at least Less/No Heavy Rare Earths
- No Rare Earths
- Recycle Rare Earths

Apart from research and development work it will also analyse activities on operative level whereas countermeasures the following activities are performed such as i) monitoring of supplier and stability of supplier, ii) strategic position of supplier, iii) metal clauses in supply/sales contracts, iv) security stock, v) future price trends analysis and vi) currency trends (CNY/USD/EUR).

The presentation will conclude with some future predictions starting from base position - RE-TM metals (3d-4f coupling) is magnetically unrivalled as of yet - and end with options like new materials (niche magnets), new technologies (enabling spring exchange), recycling efforts (EU based projects) and new applications (less or no magnets at all).



MONDAY

Sm-Co Magnets and RE Supply Chain

<u>J. Liu</u>

Electron Energy Corporation, 924 Links Avenue, Landisville, PA 17538, USA

2. Supply Chain Development and Magnet Processing, September 4, 2023, 11:30 - 13:00

Sm-Co magnets have been used in many applications including motors and generators, electron beam focusing systems, medical devices, oil and gas industry, space exploration, aerospace, instrumentation, and defense systems.

This presentation will cover the effect of machining on magnetic properties for very small magnets, some unique Sm-Co magnet applications, and some magnetic design considerations. We will also discuss rare earth supply chain and recent US activities in the rare earth industry.



Building a Resilient Critical-raw-materials Value Chain: the Importance of International Standards

G. Hatch^{1,2,5}, D. Hunter^{3,5}, S. Strickland^{4,5}

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2. Supply Chain Development and Magnet Processing, September 4, 2023, 11:30 - 13:00

With the increasing importance of rare earths and other critical raw materials to value chains globally, the need for coordinated, global standards has become vitally important. Globally agreed, and where possible harmonised, standards have the potential to reduce trade barriers, accelerate large-scale trade, and facilitate technology transfer. They can also improve transparency and sustainability across value-chain phases, from primary commodity production through to the circular economy. This presentation will provide an overview of the international-standards landscape for rare earths, outlining current and future plans for standardisation. It will provide further detail about some of the key international standards in development, and discuss the important role that standards play in ensuring more resilient global supply chains.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (03)

SESSION 3: ALLOY DESIGN AND MACHINE LEARNING

MONDAY 4 SEPTEMBER 14:00 - 15:30 ELGAR CONCERT HALL - THE BRAMALL



Inverse Design of Permanent Magnets: From High-throughput Calculations to Machine Learning

<u>H. Zhang</u>

Technical University of Darmstadt, Darmstadt, Germany

3. Alloy Design and Machine Learning, September 4, 2023, 14:00 - 15:30

As energy materials, permanent magnets are essential for the energy conversion between mechanical energy and electricity with applications in wind turbines and electric vehicles. To engineer sustainable permanent magnets, it is indispensable to tackle both the intrinsic (as given by the crystal structures) and the extrinsic (as dominated by the microstructure) magnetic properties. In this work, focusing on the (micro-)structure-property mapping, I am going to demonstrate the concept of inverse design and to showcase how it can be carried out in three different flavours, i.e., high-throughput combinatorial computation, multi-objective Bayesian optimization, and generative deep learning. A fully-fledged high-throughput workflow [1] has been constructed and applied to screen for transition-metal based candidate materials such as borides with nano-laminated structures [2] as permanent magnets. Additionally, I am going to show how such a workflow can be generalized for the 4f-3d magnets, where the magnetocrystalline anisotropy can be tailored via modifying the crystal fields, e.g., by hydrogen interstitials [3]. Furthermore, in order to explore the vast chemical space more efficiently, I demonstrate how forward inference machine learning models can be combined with acquisition functions to enable adaptive design via Bayesian optimization, and apply such an approach to perform multi-objective optimization of Curie temperatures [4] and magnetization of ferromagnetic materials. Last but not least, I will elaborate on our recent implementation of a diffusion model to perform generative deep learning on the microstructure and discuss how inverse design of microstructures can be carried out for permanent magnets with optimal performance.

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MONDAY

03-2

A High Throughput Machine Learning-assisted Study of the Effect of Element Substitution in RE-TM Films

Y. Hong¹, S. Grenier¹, T. Devillers¹, A. Kovacs^{2,3}, H. Oezelt^{2,3}, M. Yano⁴, N. Sakuma⁴, A. Kinoshita⁴, T. Shoji⁴, A. Kato⁴, . Schrefl^{2,3}, N. M. Dempsey¹

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3. Alloy Design and Machine Learning, September 4, 2023, 14:00 - 15:30

Combinatorial studies based on the preparation and characterisation of compositionally graded thin films are being used for the screening and optimization of a range of functional materials [1], including hard magnetic (e.g. FePt) films [2]. When combined with Machine Learning (ML), such high-throughput film-based studies hold much potential to guide data-driven design of new materials [3,4].

Here we will present results obtained on compositionally graded RE-TM films based on both the 2-14-1 and 1-5 high anisotropy phases. More specifically, compositionally graded (NdLaCe), Fe, B and Ce(CoX), films were sputtered onto 100 mm diameter Si substrates and then annealed to explore the effects of element substitution and processing parameters on both structural and magnetic properties. Characterization techniques operated in scanning mode, including Energy Dispersive X-Ray (EDX) spectroscopy, Magneto-Optic Kerr effect (MOKE) microscopy, and X-Ray Diffraction (XRD), were employed to create extensive experimental datasets, with roughly 300 positions sampled per substrate (i.e. composition spread) and per annealing condition. Machine learning models were developed to probe for correlations between composition, crystal structures, and magnetic properties, particularly coercivity [5]. Partial least squares (PLS) regression was applied to predict coercivity values, enabling the identification of key features and their respective positive or negative contribution. Our approach demonstrates the potential of combining high throughput experimentation and ML-driven data analysis for the accelerated development of rare earth permanent magnets with reduced dependence on critical elements. The resulting insights can guide the design and optimization of high-performance bulk permanent magnets which are crucial for the automotive, electronics, and renewable energy sectors.

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Enhancing the coercivity of Nd-Cu diffused Nd-Fe-B material by Nb assisted grain boundary pinning

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¹Indian Institute Of Technology Madras, Chennai, India,²International Advanced Research Centre for Powder Metallurgy and New Materials, Chennai, India

3. Alloy Design and Machine Learning, September 4, 2023, 14:00 - 15:30

Grain boundary diffusion process (GBDP) with low melting eutectics is known to enhance the coercivity of Nd-Fe-B magnet but results in growth during annealing which has adverse effect on the coercivity. Nb doping hinders the grain growth. Therefore, GBDP in Nd-Fe-Nb-B can be advantageous. Here we report the GBDP of Nd-Cu eutectic phase and the influence of Nb on the microstructure and magnetic properties of Nd-Fe-B melt-spun ribbons. The coercivity was enhanced from 1.2 T to 2.1 T with Nd-Cu GBDP. Grain size measurements estimated from the Transmission electron microscopy shows There is no significant grain size difference between the as-recd and GBDP samples. Atom probe tomography analysis indicated the presence of nano sized Nb which possibly restricts grain growth during diffusion annealing while facilitating Nd-Cu GBDP Nd-Fe-Nb-B will be discussed.



Fig. 1 (a) Magnetic Properties of as-recd and GBDP sample (b) and (c) TEM micrographs of as-recd and GBDP sample and the grain size distribution plot along with magnified images of Nd-Fe-B grains are shown in set (d) STEM-EDS mapping showing the Nd and Cu eutectic species (e) proximity histogram of the selected Nb cluster in inset along with the 3D distribution of Nb clusters delineated with 3 at.% the Nb isoconcentration surface of the GBDP sample (f) 1D Compositional profile obtained from 10 nm diameter cylindrical region of interest with 0.5 nm bin width along the Cu-enriched region (highlighted by isodensity surface) of the GBDP sample.

Towards Maximum Utilization of Heavy Rare Earths in Sintered NdFeB Magnets

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3. Alloy Design and Machine Learning, September 4, 2023, 14:00 - 15:30

Improved usage of heavy rare earths (HRE) for coercivity enhancement in NdFeB-based magnets has undergone significant evolution and is probably the main achievement in the last decades for the materials community in this field. First generation magnets were produced by direct HRE alloying [1] resulting in significant reduction in remanence and high HRE consumption. Second generation utilizes the HREs more effectively, hardening only the outer shells of the grains via the grain boundary diffusion [2] process (GBDP) or the two-alloy approach [3]. This results in preserved remanence and lower HRE consumption. Here, we investigate the third generation – strategic selective hardening of areas in the magnet that are most susceptible to demagnetization, such as corners or edges. This approach becomes especially important considering the advent of additive manufacturing which has the potential to realize such tailored approaches with local magnetic hardening based on specific application requirements.

Various HRE sources ranging from simple DyH2 to complex multi-component alloys were investigated on commercial NdFeB-based sintered magnet grade (Vacuumschmelze GmbH). Grain boundary diffusion process was applied on c-planes, edges and corners. In addition, powder mixing followed by conventional sintering (two-alloy approach) was studied to find the best utilization of the HREs. Figure 1 shows GBDP results for c-planes with different HRE amounts. In the case of 0.6 wt.% (GBDP alloy), high Tb utilization with a coercivity increase of 3866 kA/m/wt.% Tb (4.86 T/wt% Tb) is achieved with minor reduction in remanence from around 1.45 T in the initial magnet to 1.43 T after GBDP. The effectivity decreases with increasing the GBDP alloy amount. Detailed analysis of magnetic properties, microstructure and chemical composition will be presented.



Figure 1: GBDP results for c-planes with different HRE amounts

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MONDAY

03-5

Grain Boundary Engineering in Nd-based ThMn₁₂ Magnets and their Nitrides: A Comprehensive Study of Challenges and Limitations

<u>A. Aubert</u>¹, X. Liao^{1,2}, F. Maccari¹, S. Riegg¹, S. Ener¹, E. Adabifiroozjaei³, T. Jiang³, L. Molina-Luna³, K. Skokov¹, O. Gutfleisch¹

¹Functional Materials, Tu Darmstadt, Darmstadt, Germany, ²Institute of Resources Utilization and Rare Earth Development, Guangdong Academy of Sciences, Guangzhou, China, ³Advanced Electron Microscopy (AEM), TU Darmstadt, Darmstadt, Germany

3. Alloy Design and Machine Learning, September 4, 2023, 14:00 - 15:30

Grain boundary (GB) plays an important role in governing various mechanical and functional properties of polycrystalline materials. The GB engineering has been applied to different polycrystalline materials and has allowed great breakthroughs in material science. Among them, the performances of RE-based permanent magnets are known to highly depend on the grain boundary and has been intensively studied in Nd₂Fe₁₄B-type magnets, the most powerful permanent magnet which is employed for numerous applications since its discovery in 1980s [1, 2]. However, the global rare-earth (RE) crisis in 2011 has revived the interest and research in RE-lean and RE-free permanent magnets. The RE(Fe,M)₁₂ compound (M is a stabilizing transition metal) with the ThMn₁₂ (1:12) tetragonal structure requires less RE compared with Nd₂Fe₁₄B-type magnets and has comparable intrinsic magnetic properties. Thus, the investigation of Nd-based RE-lean ThMn₁₂ structure is of interest as a potential permanent magnet application [3, 4].

This work is dedicated to investigating the grain boundary phases of the Nd-based ThMn₁₂ magnets and their nitrides; which hasn't been studied previously. Its impact on microstructure, magnetic properties and sintering process is shown. For that, a series of micro- and nanocrystalline Nd_xFe_{10.5}Mo_{1.5} (at. %; x=1.05, 1.2, and 1.4) alloys with different Nd content were prepared. Adding small amounts of Cu or Ga elements was proved to tune the intergranular phase properties. After nitrogenation, the influence of the Nd excess on the phases, magnetic properties and densification is shown. We successfully obtained paramagnetic grain boundary phase, necessary to magnetically isolated the ThMn₁₂ grains. Thus, our investigation demonstrates the key role of the grain boundary also in the Nd-based ThMn₁₂ magnets and how it can be used to adjust the microstructure and magnetic properties of such compounds [5].



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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (04)

SESSION 4: RECYCLING OF RARE EARTH MAGNETS

MONDAY 4 SEPTEMBER 16:00 - 17:30 ELGAR CONCERT HALL - THE BRAMALL



The Works of Late Emeritus Professor Rex Harris

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¹School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham, B15 2SE

4. Recycling Rare Earth Magnets, September 4, 2023, 16:00 - 17:30

The late Professor Emeritus Rex Harris, former head of the Applied Alloy Chemistry Group and later the Magnetic Materials Group at the University of Birmingham, was a founding organiser of the REPM conference series.

Three short presentations will describe the main themes of his research in the theory of alloys, the interaction of hydrogen with metals and most recently his 40 years at the forefront of rare earth permanent magnet science and technology.

His lifelong interest in hydrogen and magnetism originated in his early work on Th-Ce, Ce-Intra RE and Pd-Ce alloys which led to new understanding of the anomalous alloying behaviour of cerium. In the same period, novel hydrogen purification membranes, notably based on Pd-Y alloys were developed; the first of many fruitful industrial collaborations.

Rex was a huge supporter of the hydrogen economy and worked on a range of hydrogen storage materials. He not only improved their storage capacity and functionality, but he also demonstrated their use at scale. The most prominent of these demonstrators was the Ross Barlow hydrogen powered fuel cell canal boat.

Rex had a long and illustrious career working on alloy development and processing of permanent magnetic materials. In particular, he was known for developing hydrogen-based processes for rare earth magnets including the hydrogen decrepitation process which is used globally for the manufacture of neodymium iron boron magnets. In the latter part of his career he was extremely concerned about the sustainability of rare earth magnets and started developing circular approaches to processing of rare earth magnets. This including HPMS (Hydrogen Processing of Magnetic Scrap) for separation and purification of magnets from end of life products and direct short loop "magnet to magnet" recycling of the extracted NdFeB alloy powders using a range of approaches.



An Overview of Magnets Recycling with Focus on HPMS

C. Burkhardt¹, A. Walton²

¹Pforzheim University, Pforzheim, Germany, ²School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham, B15 2SE

4. Recycling Rare Earth Magnets, September 4, 2023, 16:00 - 17:30

Rare Earths (RE) permanent magnets are essential components for electronics, communication and medical devices, renewable energy, robotics, electric vehicles, aerospace and dual use. While the EU is a world leader in the manufacturing of many of these components, it is fully importdependent along the entire value chain of RE magnet materials. In the recently published critical raw material act, the EU highlighted the dangers associated with European industry's total dependence on imported materials, including severe vulnerabilities in current global supply chain models. To mitigate the situation, the EU regulation stipulates that at least 15% of annual EU consumption of permanent magnets should be covered by recycling capacities, including all intermediate steps, in 2030. Researchers in the EU H2020 project SUSMAGPRO consortium have shown that hydrogen can be used as a very efficient recycling method to extract NdFeB magnet powder from various EOL Components in the IP protected Hydrogen-based Processing of Magnet Scrap (HPMS). On exposure to hydrogen the sintered NdFeB magnets break down into a friable, demagnetised, hydrogenated powder containing an interstitial hydride of Nd₂Fe₁₄BH_y (10 microns) and smaller particles (< 1 micron) from the grain-boundary phase NdH2.7. This process delivers a sustainable source of magnetic material for the production of sintered, polymer bonded and metal-injection moulded magnets. The talk will present numerous results along the whole value chain of magnet recycling, including automatic dismantling of magnet containing products, magnets extraction, HPMS recycling, production of recycled magnets and demonstrator testing. It will also discuss best practices and bottlenecks of the processes as an outlook for successful design-for-recycling of future applications



MONDA

04-3

Optimizing the Short-loop Recycling of Dy-Rich End-of-Life NdFeB Permanent Magnets

<u>**G. Bacchetta**</u>, S. Luca, C. Rado, J. P. Garandet Univ. Grenoble Alpes, CEA, LITEN, Grenoble, France

4. Recycling Rare Earth Magnets, September 4, 2023, 16:00 - 17:30

NdFeB permanent magnets contain approximately 30 wt.% of critical raw materials: Light or Heavy Rare-Earth Elements (LREE or HREE) [1]. Recycling End-Of-Life (EOL) NdFeB magnets is an alternative to alleviate the criticality issue of these high-technology materials. Besides, many studies have been carried out on the overall reduction of the use of HREE mainly by Grain Boundary Diffusion Process (GBDP) or dual alloy methods (HREE-rich and HREE-free), aiming at forming a core-shell type microstructure. This induces a high magnetic anisotropy close to grain boundaries thus preventing the propagation of the magnetic reversal from one magnetic grain to another. Some of the highly coercive EOL magnets used in wind turbines or electric vehicles have a high content of critical HREE typically ranging from 6.5 to 11 wt% [2]. They could be used as a future secondary source of HREE.

We propose a short-loop recycling method of NdFeB magnets, using them as the only source of HREE. A dual alloy co-sintering method is implemented using HREE-free strip cast powders to produce partly recycled anisotropic sintered magnets. Their microstructure and magnetic properties are discussed regarding the influence of several process parameters: blending ratio, sintering-annealing cycle, and initial EOL magnet composition. The coercivity is enhanced compared to reference magnets up to a 50% ratio of recycled powder, even though impurities are incorporated. Microstructural studies show an heterogeneous localization of Dy in a core-shell/anti core-shell microstructure thus involving a reduced use of HREE.

Experimental results are correlated with finite elements simulations using existing data of bulk Dy diffusion coefficients [3,4]. Cubic grains are modelled separated by a liquid phase network whose fraction is estimated using NdFeB phase diagram. Model results show interestingly good similarities with the observed microstructures. This study provides new insights into the Dy diffusion mechanisms at stake in an efficient short-loop recycling of HREE.





Figure 1:

- a) Demagnetisation curves of partly recycled magnets sintered at 1000°C-4h and anneaked at 800-520°C. Recycled powder fraction ranges from 0% (reference magnets) to 50 wt%.
- b) BSE image of a 30% recycled magnet after sintering and annealing cycle superimposed with EDS Dy elemental map. Dark contrast : Nd₂Fe₁₄B, white contrast : Nd-rich phases.
- c) Simulated Dy concentration after 4h sintering using extrapolated bulk diffusion at 1000°C (9.5 x 10⁻¹⁷ m²/s) and liquid phase diffusion coefficient of 1.0 x 10⁻¹³ m²/s.

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Enhancing Recycled Nd-Fe-B Magnets' Performance via (Single) Grain Boundary Engineering with Nd-Cu using Spark Plasma Sintering

<u>M. Rebernik</u>¹, T. Tomše¹, B. Podmiljšak¹, Z. Samardžija¹, C. Burkhardt², L. Schieren², S. Šturm^{1,3}, K. Žužek^{1,3}

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4. Recycling Rare Earth Magnets, September 4, 2023, 16:00 - 17:30

The coercivity of Nd-Fe-B magnets is governed by the interaction of local inherent magnetic characteristics and magnetization processes at the grain boundaries [1]. Recycled Nd-Fe-B-type magnets manufactured from HPMS-based powders are often burdened with oxygen and other impurities, which negatively impacts their magnetic performance. Loss of metallic Nd-rich phases due to oxidation can be partially compensated by the addition of Nd-hydride [2]. However, this approach reduces the volume fraction of the hard-magnetic RE₂Fe₁₄B phase.

To overcome the performance-limiting factors (i.e., non-magnetic impurities and ferromagnetic nature of grain-boundary phases [3]), the possibility of redesigning the microstructure of magnets based on recycled content by replacing the existing secondary phases with a fresh non-magnetic material was investigated in this work. An HPMS powder was treated with organic acid to selectively leach out the Nd-rich phases and obtain a single-phase RE₂Fe₁₄B-type powder. The single-phase powder was subsequently mixed with Nd₇₀Cu₃₀ low-eutectic alloy acting as a liquid-phase sintering aid and grain-boundary modifier. The powder mixture was consolidated into bulk magnets with a spark-plasma sintering (SPS) furnace. The effect of the Nd-Cu addition (0-20 wt.%) on the SPS densification behaviour, microstructure development, and final magnetic properties was studied. The addition of Nd-Cu increased the intrinsic coercivity of SPS samples from 16 to 492 kA/m for 0 and 10 wt.% addition of Nd-Cu, respectively, and the remanent magnetization was increased from 0.20 to 1.04 T. The grain-boundary wetting phenomenon was observed for samples blended with Nd-Cu, contrasting the microstructure of the SPS sample prepared without Nd-Cu, which was characterized by the absence of a grain-boundary phase. By increasing the amount of Nd-Cu added to the initial powder from 2.5 to 20 wt.%, the transition from partial wetting through pseudopartial wetting to complete wetting was observed.

Acknowledgement: ARRS P2-0084, HEU REESILIENCE (101058598).

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Optimising Magnetic Properties of Recycled Magnets obtained via Pressless Processing (PLP)

<u>W. C. Macedo Junior</u>¹, L. F. Antunes¹, C. . Sampietro¹, L. U. Lopes², J. C. S. Ronchi¹, M. A. Carvalho¹, L. T. Quispe¹, <u>M. . Rosa</u>¹, S. M. de Souza³, <u>P. A. P. Wendhausen</u>¹
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4. Recycling Rare Earth Magnets, September 4, 2023, 16:00 - 17:30

The pressless processing (PLP) is regarded as an advanced powder metallurgy route, and its feasibility to produce high quality magnets has been already demonstrated [1]. Besides not requiring any compaction step, it is a near net shape method, hence machining costs may be substantially reduced, opening the way for lean manufacturing of magnets. Moreover, as demonstrated by other researchers, it allows for the handling of the powders in a more controlled fashion throughout the whole processing, minimising oxygen uptake [1,2]. This is crucial, especially in the context of recycling Nd-Fe-B magnets, where in every recycling step the amount of Nd-rich phase may be depleted due to oxidation [3,4]. Guaranteeing the minimal uptake of oxygen is critical to maintain a proper amount of liquid phase during sintering, leading to recycled magnets with high coercivity and remanence. Therefore, PLP could be evaluated as a viable recycling strategy for Nd-Fe-B magnets. An important aspect of PLP is guaranteeing the magnetic alignment of the particles, since the maximum possible degree of alignment should be developed to ensure high remanence and energy product. In this work we investigated the effect of the filling factor, mould size and the magnetization strategy (number and direction of pulses) in the final texture of recycled magnets. Scrap magnets were hydrogen decrepitated and milled to a particle size ranging from 1.5 to 2.5 µm. Moulds were filled with different amounts of powder to achieve different filling factors. The filled moulds were subjected to magnetic pulses varying from 1600 to 3200 kA/m. Magnets with different sizes (10 to 100 g) were evaluated regarding the degree of texture using the adapted Fernengel function [5]. The results show that the degree of alignment is not very dependent on each of the studied variables.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (05)

SESSION 5: UNCONVENTIONAL PROCESSING

TUESDAY 5 SEPTEMBER 09:00 - 10:30 ELGAR CONCERT HALL - THE BRAMALL



The Influence of the Disproportionated Microstructure on the Hydrogen Ductilisation Process (HyDP) for NdFeB Alloys

O. Brooks¹, F. Burkhardt¹, S. Hussain¹, V. Kozak¹, Y. Kajinami², Y. Une², T. Morita², T. Iriyama², I. R. Harris¹, A. Walton¹ ¹School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham, B15 2SE,

²Daido Steel Co., Ltd., Nagoya, Japan

5. Unconventional Processing, September 5, 2023, 09:00 - 10:30

Recently, researchers at the University of Birmingham have been investigating a technique for inducing ductility in solid NdFeB alloys at room temperature, termed the Hydrogen Ductilisation Process [1,2]. The process utilises the high temperature (>650°C) solid hydrogen disproportionation [3] reaction, to transform the Nd₂Fe₁₄B matrix phase into a mixture of NdH₂, Fe₂B and Fe. It has been shown that once in this condition, the disproportionated mixture can be significantly deformed at room temperature. Once deformed, this material can be recombined, with evidence that the deformation in the intermediate step produces a degree of magnetic alignment after recombination.

HyDP was initially limited by the presence of a minority $NdFe_4B_4$ phase, which failed to fully disproportionate during hydrogen processing. Previous work has been performed to alter the initial microstructures to limit the quantity of this phase [4,5] with some success. However, this had an influence on the disproportionated microstructures that formed during hydrogen processing, and mechanical behaviours at room temperature.

The work presented here uses a multi-stage disproportionation process to significantly alter the morphology of the disproportionated microstructures, creating a series of samples ranging from fine lamellar structures to coarse spherical. It is observed that, with increased coarseness of the disproportionated microstructure the yield stress required to deform the material can be significantly improved, decreasing by ~30%, greatly improving the ductile behaviour. Furthermore, deformation caused the coarsened spherical NdH₂ to elongate in the disproportionated microstructure, influencing the kinetics for recombination and the final magnetic anisotropy.

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Development of High Anisotropic Nd-Fe-B HDDR Powders

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5. Unconventional Processing, September 5, 2023, 09:00 - 10:30

Although (BH)max of bonded magnets is lower than that of sintered magnets, the bonded magnets have the major advantage of obtaining nearly net-shaped products and having high resistivity resulting in suppressing eddy current and decreasing heavy rare earth element. The HDDR process is one of the useful methods for obtaining anisotropic Nd-Fe-B magnet powders. To increase energy products of the bonded magnets, improving anisotropy of HDDR treated Nd-Fe-B powder and the clarification of anisotropy mechanism during HDDR process are required.

This talk reviews recent progress in our work on the development of HDDR powders consisting of highly aligned Nd2Fe14B recombined grains [1]. From the microstructure observation, it is important to maintain the crystallographic orientation relationship between α -Fe and NdH2 formed in both fine and coarse lamellar structures during disproportionation reaction while suppressing the formation of spherical NdH2 structures. These lamellar structures transform into highly aligned Nd₂Fe₁₄B regions during recombination reaction, however, the degree of alignment in the regions transformed from spherical structures is low. The spherical NdH₂ structure, in which the crystallographic orientation relationship with α -Fe disappears, is transformed from both lamellar structures when hydrogen pressure, heat treatment temperature and time for disproportionation reaction increase. In addition, the spherical NdH₂ phase forms at the surface of powders and is present along grain boundaries and cracks in powders. Therefore, making single crystal starting powder, suppression of crack formation by performing hydrogen decrepitation at high temperature, and control of hydrogen pressure and temperature to maintain aligned structures are useful methods for increasing the volume fraction of lamellar structure to produce high-performance HDDR powders and bonded magnets.

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Rare Earth Based Permanent Magnets by Laser Powder Bed Fusion

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5. Unconventional Processing, September 5, 2023, 09:00 - 10:30

Additive manufacturing based on laser powder bed fusion (L-PBF) technology is challenging for rare earth (RE) based permanent magnets. It requires special processing chambers to handle the oxidation-sensitive powders. Furthermore, only quite specific microstructures enable good permanent magnet properties. Material systems such as FeNdB [1] develop nanocrystalline microstructures in the bulk by rapid solidification during L-PBF. This is enabled by the formation of a shallow melt pool during laser melting. Material systems such as CoSm [2] or FePrCuB [3] can offer advantages for L-PBF processing. Due to their solidification process and phase equilibria, they form magnetically advantageous microstructures. Permanent magnet properties can be obtained by annealing without the need of subsequent powder metallurgical processing or rapid quenching.

By proper choice of processing parameters and post-annealing conditions permanent magnet properties and partial magnetic anisotropy can be realized for printed parts of both materials: For FeNdB (Dy-free), depending on the alloy's RE-content, a maximum coercivity of 1.27 T, remanence of 0.70 T and maximum energy product of 68.1 kJ/m³ have been achieved. For (CoCuFeZr)₁₇Sm₂, depending on the composition, a coercivity of 3.05 T, remanence of 0.78 T and maximum energy product of 109.4 kJ/m³ have been obtained. For FePrCuB, depending on the used processing parameters (laser power, scan velocity, volume energy density), a maximum coercivity of 0.85 T, remanence of 0.82 T and maximum energy product of 85.4 kJ/m³ have been realized. The paper highlights the influence of processing parameters on solidification behaviour, the resulting microstructure and related magnetic properties including their temperature dependence.

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DOI: 10.3390/mi12091056.



Solidification-based Additive Manufacturing of Rare Earth Permanent Magnets

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5. Unconventional Processing, September 5, 2023, 09:00 - 10:30

In recent years, additive manufacturing has gained significant attention. Its ability to produce near-net-shape and net-shape parts, significantly alter design paradigms, and consume less raw materials offers opportunities to transform manufacturing. For permanent magnet applications, the design flexibility will impact motor design, and the reduction of waste in the processing stream will more efficiently utilize critical materials. For melt-processing additive manufacturing, such as laser powder bed fusion (LPBF), the challenge is for the solidification process to produce the appropriate microstructure (phase, grain size, etc.) that results in maximum magnetic performance. In this paper, the focus is on using LPBF additive manufacturing to produce RE-Fe-B-, Sm-Co-, and Ce-Co-based materials. For alloy development, we utilize melt spinning as a proxy, as solidification conditions (cooling rate, cooling direction) mimic those found in additive manufacturing. For Nd-Fe-B, ternary Nd-Fe-B compositions as well as the ternary modified with alloying elements (e.g., Ti and C) to alter the solidification behaviour were studied. High-B Nd₁₅Fe₇₇B₈ displayed the best as-spun behaviour at moderate wheel speeds that most closely mimic additive manufacturing. We then print the developed alloys using LPBF under a variety of processing conditions (laser power, scan speed, hatch spacing and direction) to understand the processing-structure-property relationships. For example, control and variation of hatch characteristics can produce either stronaly directional grain orientations across layers or regions of dissimilar orientation along the build direction [1,2]. We will focus on developing processing parameters that result in high density (i.e., low porosity) and optimum microstructural evolution. Melt pool imaging experiments were also done at the Advanced Photon Source. Here, the solidification dynamics were studied to help us understand the microstructural development in situ during laser melting.

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Tailoring Magnetic Structures via Passive Magnetic and Strain-field Annealing

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5. Unconventional Processing, September 5, 2023, 09:00 - 10:30

Crystallographic and microstructural control is integral to the performance of all technologically relevant magnetic materials, including advanced permanent magnets. While such control is typically exerted through alloying additions and specialized thermal treatments, application of additional processing parameters can alter the thermodynamics and kinetics of structural development and provide more efficient pathways to tune performance. To this end, the effects of passively applied magnetic field and uniaxial stress exerted during thermal treatment on atomic ordering and microstructural alignment have been investigated in two permanent magnet proxies: Mn₅₅Al₄₅ and FeNi. Calorimetry, imaging and magnetic assessment confirm a significant (~ 40%) increase of the atomically ordered T-MnAl phase fraction in magnetic field-annealed specimens relative to those treated under zero-field conditions, both subjected to the same thermal treatment. Analogously, simultaneous conversion X-ray and backscattered y-ray 57Fe Mössbauer spectroscopy (CXMS) has revealed enhanced crystallization (local order) of product phases realized during thermal treatment under applied magnetic- and strain-field conditions, relative to what is achieved via conventional (field-free) thermal processing. Overall the outcomes of this work confirm the utility of new modes of magnetic materials processing to facilitate enhanced control of crystallographic and microstructural features underlying performance.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (06)

SESSION 6: NANOCRYSTALLINE & THIN FILM MAGNETS

TUESDAY 5 SEPTEMBER 11:00 - 12:35 ELGAR CONCERT HALL - THE BRAMALL



Exploring Coercivity Limit in SmFe₁₂-Based Thin Films

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

Rare-earth lean SmFe12-based compounds have been considered as promising candidates for next generation permanent magnet materials owning to their excellent intrinsic hard magnetic properties [1]. However, one of the major challenges hindering their practical application is how to convert the excellent intrinsic magnetic properties into the extrinsic ones, large remanence and coercivity. To realize coercivity in SmFe12-based magnets, it is necessary to introduce a grain boundary phase that can magnetically isolate SmFe12-based grains [2,3]. However, it is an open question that how large coercivity can be realized in the SmFe12-based system by engineering of the intergranular phase (IGP).

In this work, we have investigated the influence of IGP with different composition on the coercivity of $Sm(Fe_{0.8}Co_{0.2})_{12}$ -based system in a modeled thin film. We found that by doping of a small amount of boron, a nanogranular structure can be formed consisting of $Sm(Fe_{0.8}Co_{0.2})_{12}$ grains surrounded by B-rich IGP. This results in an increase in coercivity from 0.1 T to 1.2 T. However, the obtained coercivity was only 10 % of the anisotropy field of the 1:12 phase [4]. Micromagnetic simulations based on transmission electron microscopy (TEM) images showed that the easy nucleation of reverse magnetic domains on the ferromagnetic IGP is responsible for the low coercivity [5]. Therefore, we have applied the AI and Si infiltration process to dilute the magnetization of the IGP (Fig. 1). We have demonstrated partial infiltration of Si into the IGP can result in an enhancement of the coercivity to 1.32 T. At the end of the talk, we will discuss how a large coercivity of 1.8 T is realized in these films by infiltration of AI into the IGP of B-doped $Sm(Fe_{0.8}Co_{0.2})_{12}$ thin films and elimination of the secondary soft ferromagnetic α -Fe phase (Fig. 1).



Figure 1: (a) Magnetisation curves obtained from Sm $(Fe_{0.8}Co_{0.2})_{12}$, Sm $(Fe_{0.8}Co_{0.2})_{12}B_{0.5}$, Si and Al infiltrated Sm $(Fe_{0.8}Co_{0.2})_{12}B_{0.5}$, thin films. Cross-sectional STEM-EDS and high resolution HAADF-STEM imagnes obtained from (b) Si-infiltrated and (c) Al-infiltrated Sm $(Fe_{0.8}Co_{0.2})_{12}B_{0.5}$ 100 nm thin films.

Acknowledgement: This work was supported by JSPS KAKENHI Grant Number JP23H01674.

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Magnetostatic Coupling in NdFeB Melt-Spun Magnets

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

The SW-CLC (Stoner-Wohlfarth-Callen-Liu-Cullen) model is reinterpreted as a mean field model useful for studying magnetostatic coupling. The SW model predicts a region of reversible rotation which can give origin to the spring effect if the particles are single domain size.

Situations for occurrence of magnetostatic coupling were discussed. A magnetostatic criterion for coupling is proposed, where both the hard and soft phases need to be single domain size or less. The Hysteresis curves of high coercivity nanocrystalline SmCoCuFeZr magnets can be fitted with the Callen-Liu-Cullen model using positive (1/d) parameter, implying in magnetostatic coupling.

A necessary condition for the Wohlfarth relationship, which is the basis for Henkel plots, is that both coupled phases have only one easy axis of magnetization. However, alpha-iron has 3 easy magnetisation axis.

The system NdFeB-iron also can be modelled with the CLC model. However, as afore mentioned, the alpha-iron has 3 easy axis. Thus, remanence higher than half in isotropic NdFeB-iron magnets may not be due to exchange coupling, and this is more likely due to magnetostic coupling. It is discussed how to estimate the volume fraction of the soft phase in magnetostatically coupled Nd2Fe14B-iron.

Suitable nanostructures for taking advantage of magnetostatic coupling were also discussed. This is the double-shell model, where a hard phase is enveloped first by a soft phase, and then by a paramagnetic phase. The double shell is inspired by real nanostructures of 2:17 type magnets.

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O6-3 (Nd,LRE)-Fe-B Hot-Deformed Magnets for Variable-Magnetic-Force Motor Applications

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

The recent development of variable-magnetic-force (VMF) motors has raised a new demand for permanent magnets with moderate coercivity (0.15-0.65 T), flat first-order reversal curves (FORCs), and high magnetization [1-2]. However, conventional Nd-Fe-B sintered magnets cannot be used for this application due to the large coercivity and unflat FORCs. In this work, we explored the potential of (Nd,LRE)-Fe-B (LRE= La and Ce) hot-deformed magnets for VMF motor applications by evaluating their extrinsic magnetic properties and FORCs.

The hot-deformed magnets with a composition of $(Nd_{0.6}La_{0.3}Ce_{0.1})_{13.4}Fe_{76.3}Co_{4.5}Ga_{0.5}B_{5.3}$ (at.%) were prepared by melt-spinning and die-upsetting processes. The magnetic properties were measured by BH-tracer and superconducting quantum interference device vibrating sample magnetometer (SQUID-VSM). The magneto-optical Kerr effect (MOKE) microscope was employed to investigate underlying mechanism for different shapes of FORCs.

Fig. 1(a) shows the selected FORCs for a commercial sintered magnet and a (Nd,LRE)-Fe-B hotdeformed magnet. The coercivity of the hot-deformed magnet can be tuned to 0.4 T, which is within the desirable range for VMF application, by substituting 40 at.% of Nd with La and Ce for (Nd_{1-x}LRE_x)-Fe-B, while keeping a relatively high remanent magnetization of 1.26 T. Unlike the commercial Nd-Fe-B sintered magnet, FORCs of the (Nd,LRE)-Fe-B hot-deformed magnet are flat as shown in Fig. 1(a). The FORCs obtained from the MOKE data in Fig. 1(a) show the same trend with those from the SQUID-VSM data, i.e., the invariable magnetization against the external field for (Nd,LRE)-Fe-B hot-deformed magnets. The selected MOKE micrographs in Fig. 1(b) revealed that flat FORCs in (Nd,LRE)-Fe-B hot-deformed magnet is due to the pining of magnetic domain walls at the intergranular phase of the (Nd_{0.6}La_{0.3}Ce_{0.1})-Fe-B hot-deformed magnets. The desirable coercivity and flat FORCs demonstrate LRE-substituted hot-deformed magnets can be good candidates for VMF motors application [3].



TUESDAY



Figure 1: (a) The selected FORCs measured from SQUID-VSM and Kerr data; (b) selected micrographs of MOKE for commercial Nd-Fe-B sintered and (Nd, LRE)-Fe-B hot-deformed magnets.

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High Coercivity and Resistivity HRE-free Nd-Fe-B Hot-Deformed Magnet by PrCu Diffusion and CaF₂-LiF Eutectic Mixture Addition

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

In electric traction motor applications, the permanent magnet temperature rises to 160-200 °C due to the eddy current loss. To maintain sufficient coercivity at these elevated temperatures, a significant amount of scarce heavy rare earth (HRE) elements are introduced into the Nd-Fe-B permanent magnets. High resistivity Nd-Fe-B permanent magnets, made from the mixture of Nd-Fe-B and high resistivity material powder, can be an alternative because they have low eddy current loss, resulting in a lower operating temperature [1,2]. The coercivity requirement is then relaxed, and the HRE content can be reduced.

One way to develop high coercivity HRE-free Nd-Fe-B magnets is by introducing low-melting point eutectic RE alloy diffusion process into the hot-deformed Nd-Fe-B magnets [3]. However, due to the formation of metallic intergranular phase, the resistivity of the magnet is further reduced, making it more susceptible to eddy current loss. We could successfully overcome this problem by developing a continuous HRE-free resistive layer surrounding the high coercivity eutectic diffusion processed Nd-Fe-B flakes in the hot-deformed magnets.

HRE-free Nd-Fe-B hot-deformed magnet with 1.87 T coercivity and 1.15 T remanence is developed from PrCu diffused Nd-Fe-B melt-spun ribbon powder mixed with CaF_2 -LiF eutectic mixture powder, which is comparable to those of Dy-containing high resistivity magnets. In addition, the resistivity of this magnet is doubled from 175 $\mu\Omega$ cm to 344 $\mu\Omega$ cm. The formation of a continuous resistive fluoride layer surrounding the ribbon was observed, explaining the origin of the high resistivity. RE enrichment in the intergranular phase inside the ribbon was also observed, explaining the origin of high coercivity. A temperature reduction of 23 °C was observed when the magnet was subjected to a magnetic field compared to an additive-free magnet. This research demonstrates a feasible way to achieve a high coercivity and resistivity Nd-Fe-B hot-deformed magnet without reliance on HRE elements.







Figure 1: (a) Magnetic properties of hot-deformed magnets produced in this work compared to high resistivity hotdeformed magnets and bonded magnets reported in the literature (b) Magnet temperature versus time profile of additive-free and 4 wt % PrCu diffused 5 wt % CaF2-LiF added hot-deformed magnets under 110.1 kHz 9 mT peakto-peak sinusoidal AC magnetic field and the experiment setup.

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06-5

Hard Magnetic SmCo₅-Cu Nanocomposites Produced by Highpressure Torsion - Microstructural Evolution and Magnetic Properties

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

In recent years a lot of research and development has been done to overcome the discrepancies between intrinsic and extrinsic magnetic properties. Beside the search on novel materials and optimizing already existing ones, there is a challenge to develop new unconventional processing techniques, that allows the adjustment of a specific micro- or nanostructure. Severe plastic deformation processes like high-pressure torsion (HPT) are well known for its potential to refine microstructures of a variety of metals and alloys. Yet, the method is not limited to processing of monolithic samples but also capable of consolidating powders and powder blends, thus, paving the way towards nanocomposite materials and microstructures that are inaccessible via conventional melting-based routes [1]. In this work, HPT is applied to powder blends consisting of hard magnetic SmCo_s and diamagnetic Cu. The ductile copper powder serves as a binder phase, carrying the plastic deformation and furthermore it decouples the hard magnetic SmCo₅ grains due to its diamagnetic behaviour. Microstructural analyses show a refinement of the brittle SmCo₅ phase with increasing applied strain up to the single domain region. At the same time the individual particles are well surround by Cu leading to magnetic decoupling of the SmCo₅ grains. The structural refinement of the SmCo₅ while simultaneously encircling by Cu, correlates with an increase in coercivity up to more than 1.3T [2]. Furthermore, a crystallographic texture with the c-axis being preferably aligned parallel to the rotation axis of the HPT disc evolves, thus, leading to anisotropic magnetic properties. The results emphasize the wide freedom of microstructural design for magnetic materials using HPT of powder blends, which enables the generation of textured nanocomposites with excellent magnetic properties.

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Dual Amorphous-Precursor Deformation Method for High-Performance Nd-Saving Nd-Fe-B Hot-deformed Magnets

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023, 11:00 - 12:35

Current challenge in development of Nd-Fe-B permanent magnets is to achieve high-performance in the magnets while reducing their Nd content by substituting Ce [1]. However, the magnetic properties of Nd-Fe-B magnets could be deteriorated after replacing Nd with Ce due to the intrinsic magnetic properties of Ce₂Fe₁₄B compared to Nd₂Fe₁₄B [1]. The dual-main-phase structure in Nd-Fe-B magnets can suppress the deterioration of magnetic properties due to Ce substitution [2]. Recent studies have successfully surpassed the limit of magnetic properties of Ce-substituted magnets by hot-deformation of amorphous precursors that contain no (Nd, Ce) Fe₂ secondary phases [3]. In this study, we developed the dual-main-phase Nd-Ce-Fe-B hotdeformed magnets using two different amorphous-precursors with different compositions. Initial powders with the compositions of Nd_{13'6}Fe_{73'6}B_{5'6}Ga_{0'6}Co_{6'6'}, (Nd_{0'6}Ce_{0'4})_{13'6}Fe_{73'6}B_{5'6}Ga_{0'6}Co_{6'6}, and (Nd_{0.4}Ce_{0.6})_{13.6}Fe_{73.6}B_{5.6}Ga_{0.6}Co_{6.6} (Ce-free, CE40 and CE60, respectively) were prepared by a melt-spinning technique and a pulverization. Then, the mixed Ce-free and CE40 powders were hotdeformed to fabricate the magnets with 30 wt.% of Ce substitution content. To establish the optimum mixing ratio of powders, fabricated the magnets using the Ce-free and CE60 powder mixture were also prepared. The magnets showed much higher coercivity (18.09kOe) and remanence (13.5kG) than conventional single-main-phase magnets (15kOe, 13kG). Interdiffusion of Nd and Ce between Ce-free and Ce-containing flakes occurred during annealing, with different diffusion behaviour of Nd and Ce. After annealing, the Ce was preferentially segregated at the RE-rich grain boundary phases in the Ce-free flakes but the Nd was distributed at the outer region of the main phase forming the Nd-rich shell in the Ce-containing flakes. The different diffusion behaviour of Nd and Ce could be a crucial factor for substantial improvement in the coercivity and remanence of Cesubstituted magnets by applying the dual-amorphous-precursors deformation method. Finally, we succeeded in developing 30% Ce-substituted Nd-Fe-B hot-deformed magnets that can replace the commercial 45H-graded Ce-free magnets.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (07)

SESSION 7: RE-FE-B MAGNET PROCESSING AND PROPERTIES

TUESDAY 5 SEPTEMBER 14:30 - 16:00 ELGAR CONCERT HALL - THE BRAMALL



Achieving 2.8 T Coercivity in Nd-Fe-B Sintered Magnets Subjected to Two-step Grain Boundary Diffusion Process

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7. RE-Fe-B Magnet Processing and Properties, September 5, 2023, 14:30 - 16:00

Nd-Fe-B magnets for traction motors for electric vehicles and wind turbines need to withstand the demagnetization field at high operating temperatures of up to ~180 °C during service. The HRE grain boundary diffusion (GBD) process is widely used to achieve high coercivity with the minimum use of HREs, and Tb is frequently used as a GBD source because of the large coercivity increment per weight. If the Dy-GBD process can achieve a comparable coercivity with that of the Tb-GBD processed magnets by exceeding the highest coercivity achievable by the Dy-GBD process, ~2.4T [1], the cost-effective Dy-GBD process could substitute the Tb-GBD process. In this study, we report the highest coercivity of 2.8 T among the Dy-free sintered magnets treated with the Dy-GBD process of $Dy_{70}Cu_{30}$ and $Pr_{68}Cu_{32}$ eutectic alloys.

The as-sintered sample exhibited coercivity, μ_0 Hc, remanence, μ_0 Mr, of 1.1 T, and 1.38 T, respectively. The μ_0 Hc substantially increased to 2.5 T by the first GBD process, and the following two-step GBD process has increased the μ_0 Hc to 2.8 T at a slight expense of the μ_0 Mr to 1.32T, Fig. 1. The microstructural analysis by SEM and TEM showed that the thin Dy-rich shell formed discontinuously on the Nd₂Fe₁₄B grain surface in the bulk center after the first GBD step, while a thick Dy-rich shell forms near the bulk surface. The second GBD step led to the continuous Dy-rich shell formation along with the slight increase in the Dy and Pr contents on the Nd₂Fe₁₄B grain surface even in the bulk center. Such a HRE-rich shell with high Dy content at the bulk center has suppressed the low-field nucleation, resulting in high coercivity.



Figure 1: Demagnetisation curves of the untreated base magnet and the magnets subjected to 1st and 2nd step GBD processes.

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Heavy Rare Earth free Hot-deformed Nd-Fe-B Magnet for Traction Motor in Electric Vehicles.

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7. RE-Fe-B Magnet Processing and Properties, September 5, 2023, 14:30 - 16:00

Generally, Nd-Fe-B magnets require the addition of heavy rare earth elements (HREEs) to secure high enough coercivities to prevent the deterioration of their remanences due to demagnetization fields when they are put into various uses. However, since HREEs are scarce and localized in certain areas of the world, the sourcing risk of HREEs is a particular concern. As electrification progresses, the demand for Nd-Fe-B magnets will increase, and the threat is expected to increase further. In 2017, Daido Steel Co., Ltd. (DS) and Daido Electronics Co., Ltd. (DEC) began mass production of HREE-free hot-deformed magnets for HEV drive motors, which consist of ultra-fine crystal grains that show high coercivities without the HREE addition. Honda Motor Co., Ltd. (Honda) recently proposed an improved performance motor design for xEV utilizing the shape- and orientation-controlled magnets through the ideal magnetic circuit. In order to realize this new design, DS and DEC have developed a technology for controlling the shape and orientation of the HREE-free hot-deformed magnets through the net-shape molding process. The newly developed magnet has been in preparation for mass production. We also continue to work on improving the performance of HREE-free hot deformed magnets by aiming to achieve finer and more uniform crystal grains than our current products.



Effect of Grain Boundary Reconstruction and Regenerated Main Phase Shell on Magnetic Properties in High-Abundance (NdLaCeY)-Fe-B Magnets

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7. RE-Fe-B Magnet Processing and Properties, September 5, 2023, 14:30 - 16:00

Nd-Fe-B permanent magnets have a wide range of applications owing to their superior magnetic characteristics¹. In recent years, the soaring prices and massive consumption of Pr, Nd, Dy, and Tb have posed a challenge for the sustainability of Nd-Fe-B magnets. La, Ce, and Y remain underutilized and high-abundance rare earth permanent magnet have attracted considerable research attention. Replacing Nd with high-abundance rare earth elements results in a decline of magnetic performance due to the lower intrinsic magnetism of RE₂Fe₁₄B (RE=La, Ce, Y) compared to Nd₂Fe₁₄B. Previous studies have demonstrated that grain boundary reconstruction and "coreshell" structure can enhance the magnetism of high-abundance magnets², but achieving high coercivity without compromising remanence is still a difficult task. This study systematically examined the (NdLaCeY)-Fe-B magnets with 45-50 wt.% high-abundance rare earth substitution by co-mixing high boron content matrix phase and Pr-Fe alloy powders during the fabrication process. The coercivity of magnets increased successively with the addition of Pr₇₀Fe₃₀ (wt.%) and reached 9.32 kOe at the addition amount of 9 wt.% from the initial 7.33 kOe, while the remanence only decreased by 0.13 kGs. The analyses showed that the B-rich phase in the high-boron powders reacted with Pr₇₀Fe₃₀ to form the main phase shell, forming a core-shell structure with a Pr-rich shell and a Y-rich core. Meanwhile, the grain boundary was significantly widened with the introduction of Pr₇₀Fe₃₀. Then, the improved anisotropy of the main phase grain surface, accompanied with the enhanced magnetic de-coupling effect between the main phase grains, resulted in a great coercivity increment. Notably, on the remanence, the positive effect of increased main-phase proportion by regenerated grain shell had a trade-off with the negative effect of the magnetic dilution by the nonmagnetic material introduction, thus under the combined effects, the remanence initially increased and then decreased with the addition of Pr₇₀Fe₃₀.





Fig. 1 BSE and EPMA images of magnets with 9 wt.% Pr₇₀Fe₃₀ added



Fig. 2 HRTEM images of the Pr-rich shell of the magnets with 9 wt.% Pr₇₀Fe₃₀ added



Fig. 3 Microstructure of grain boundary of original magnets and 9 wt.% magnets

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Reducing Criticality of Nd-Fe-B by Recycling and Partial Substitution using the Rapid Quenching Technique

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7. RE-Fe-B Magnet Processing and Properties, September 5, 2023, 14:30 - 16:00

Rare earth (RE) permanent magnets like Nd-Fe-B are vital in societies everyday life, as they are widely used in common applications. Since RE elements are designated as highly critical in terms of supply and mining, there is an ongoing search for substitute materials. Different risk mitigation strategies are applicable, however in this contribution we will focus on the general decrease of the demand for primary mining by making use of the natural RE basket and End-of-Life (EoL) materials. The natural basket of RE consist of approximately 75 % lanthanum and cerium, which are not used in total and can be seen as "free" rare earth. Moreover, materials from EoL applications will contribute to some extend to a stable supply by recycling in near future.

In case of both routes investigated, melt-spinning was used as technique for the synthesis of nanocrystalline flakes and powder from elements or scrap magnets. In case of the recycling, polymer bonded as well as hot-deformed magnets were investigated. For the La and Ce substitution only hot-deformation was used as processing technology for magnets. Magnetic properties of La and Ce containing magnets were optimized by post processing treatments to reduce losses in magnetic properties. Deformation was conducted in a conventional hot-pressing and spark plasma sintering (SPS) device.

Magnetic properties from both mitigation strategies compete well with Nd-Fe-B magnets produced from primary mining, however they excel by far those magnets in terms of environmental footprint and criticality of supply.



A Strategy for Rapid Sintering of Nd-Fe-B-Type Magnets Based on Intense Thermal Radiation

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7. RE-Fe-B Magnet Processing and Properties, September 5, 2023, 14:30 - 16:00

The intrinsic coercivity of Nd-Fe-B permanent magnets, governed by the interplay between local inherent magnetic characteristics and nanoscale magnetization processes¹, is limited to less than 30% of the anisotropy field of the hard-magnetic Nd₂Fe₁₄B phase. Examples of coercivity-limiting factors are the presence of nonmagnetic impurities, large grain size, and the ferromagnetic nature of the grain-boundary phases². The established sintering paradigms based on low heating rates (a few degrees per minute) and hours-long dwell times at temperatures above 1000 °C limit the freedom to control the microstructure formation. In this work, we show that it is possible to rapidly sinter conventional jet-milled Nd-Fe-B powders to full density utilizing intense thermal radiation (Radiation Assisted Sintering - RAS). The novel sintering strategy allows fast heating rates (100s °C/min) and drastically reduced soaking times (<5 minutes) at standard sintering temperatures (≈ 1100 °C). The total sintering time, i.e., heating and soaking, can be reduced to 10 minutes or less. Even when heating rates as high as 500 °C/min are employed, the temperature field in the powder compact is homogeneous during heating, leading to uniform microstructure formation in diskshaped samples having a diameter of 15 mm. For a powder with a nominal composition Nd₃₀₁Pr $_{0.6}$ Dy₁Fe_{63.85}Co₃Ga_{0.22}Cu_{0.15}Al_{0.15}B_{0.93} (wt.%), the as-sintered magnetic properties were Br = 1.35 T, (BH)max = 350 kJ/m³, and Hci \approx 600 kA/m. The heating rate in the 25 - 200 °C/min range had a minor effect on the properties of dense samples. Compared to conventionally-sintered magnets prepared from the same Nd-Fe-B powder, the coercivity of RAS samples was approx. 200 kA/m lower, which was attributed to incomplete grain-boundary wetting. Nevertheless, low-temperature post-sinter annealing at 520 °C for 60 minutes significantly increased the coercivity by more than a factor of 2, to >1200 kA/m, through redistribution of the grain-boundary phase.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (08)

SESSION 8: RARE EARTH-FREE MAGNETS

TUESDAY 5 SEPTEMBER 16:30 - 18:00 ELGAR CONCERT HALL - THE BRAMALL



Iron Nitride: A Non-rare-earth Containing Permanent Magnet

F. Johnson

Niron Magnetics, Inc., 650 Taft Street NE Suite 400, Minneapolis, MN 55413

8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

Niron Magnetics, Inc. is commercializing Iron Nitride, a high performance, completely rare earth free permanent magnet technology. Iron Nitride will act as an economical substitute for several grades of both sintered and bonded NdFeB magnets. Niron's Iron Nitride technology is based on progress achieved by the University of Minnesota under work supported by the Department of Energy's Rare Earth Alternatives in Critical Technologies ARPA-E REACT program. These magnets are based on the α "-Fe₁₆N₂ compound which has high saturation magnetization and a moderate magnetocrystalline anisotropy due to a tetragonal crystal structure. Iron Nitride is manufactured from low-cost, non-critical elemental components. The unique characteristics of Iron Nitride include a magnetic strength higher than most grades of NdFeB permanent magnets. Test data also indicates that iron nitride exhibits superior temperature stability when compared to NdFeB. Niron's magnets are positioned to substitute for NdFeB in applications such as motors with high torque output.



Critical Materials Free MnAI-C Magnets: Recent Developments and a Perspective

T.G.Woodcock

Leibniz IFW Dresden, Dresden, Germany

8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

MnAI-C magnets, based on the T-phase, contain no critical elements and have the potential to compete with both hard ferrites and isotropic Nd-Fe-B bonded magnets in applications, offering enhanced performance over the former, whilst contributing to sustainability when substituted for the latter. Detailed knowledge of the intrinsic magnetic properties of the T-phase are useful both for estimating the upper limits of the extrinsic properties and as input for first principles calculations and micromagnetic models. Recent measurements of the intrinsic properties will be summarised and typical values will be given. The best extrinsic magnetic properties in MnAI-C to date were achieved more than 40 years ago in commercially produced, extruded materials. Similar performance has been achieved on a lab scale since the rare earth crisis; however, the maximum energy products of these and indeed all reported MnAI-C materials still fall short of the upper limits, estimated from the intrinsic properties of the T-phase. Further improvements are both possible and necessary if MnAI-C magnets are to be attractive for applications. Various strategies to exceed the state of the art will be reviewed, including the usage of alloying additions, the development of novel precursor materials and results from advanced characterisation yielding valuable insight into the microstructural mechanisms governing the magnetic properties.



JESUA

08-3

Nanoscale Chemical Segregation to Twin Interfaces in T-MnAl-C and Resulting Effects on the Magnetic Properties

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8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

Among various candidates for the rare-earth free permanent magnets, the ferromagnetic T-MnAI-C (L10, P4/mmm) shows great potential to replace ferrites and bonded Nd-Fe-B magnets for its attractive magnetic properties and the non-critical nature of the raw elements [1, 2]. Further magnetic properties enhancement will depend on understanding of the effect of various crystallographic defects in the magnet, e.g. twin boundaries, and on developing novel processing routes for microstructure optimization.

Twin boundaries are frequently observed in both the as-transformed and hot deformed T-MnAI-C magnets [3]. Three different types of twin boundaries have been discovered in the T-MnAI-C magnet and they are described as true twins, order twins and pseudo twins [4]. Considering the tetragonal structure of the chemically ordered T-MnAI-C magnet, it is reasonable to assume that a different atomistic structure exists at these three types of twins. The magnetic properties would also differ correspondingly to these nanoscale features.

In this study, aberration-corrected scanning transmission electron microscopy coupled with electron energy-loss spectroscopy (STEM-EELS) was used to investigate the atomistic structure and chemical composition at various twin boundaries in T-MnAI-C [5]. The results show differing levels of structural disorder at the various types of twin boundaries and EELS data reveal the presence of a Mn-enriched layer at the twin boundary surrounded by AI-enriched layers. The thickness of these layers and the magnitude of the chemical segregation vary with the level of structural disorder. Micromagnetic simulations based closely on the experimental results showed that the coercivity tends to increase with increasing structural and chemical disorder at the twin interface. These results suggest that targeted doping of interfaces in T-MnAI may be a promising strategy to increase the coercivity of the material for applications.

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Direct Synthesis of Highly Ordered L10-FeNi Nanoparticles from Layered Complexes

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8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

The L10-FeNi binary alloy is a candidate for next generation rare earth-free permanent magnets (PMs)[1], which can revolutionize the high-performance PM market currently dominated by the Nd-Fe-B. However, the fabrication of the L10 phase is extremely challenging owing to the low atomic mobility below the chemical order/disorder transition temperature that kinetically limits the formation of the L10 phase [2]. Despite many efforts, the experimental results are still far from the theoretical predictions and the proposed approaches mainly involve complex and expensive protocols, which cannot be easily scaled-up for bulk production and/or result in a low amount of the L10 phase [2].

To overcome current limitations, we exploited an effective and easily scaled-up chemical synthesis method, already successfully used for other L10 alloys [3,4], to obtain highly ordered L10 FeNi nanoparticles by low-temperature reduction in H2 atmosphere of two crystalline Ni-Fe complexes (Ni-nitroprussides Ni-NPr)or Fe-tetracyanonickelate (Fe-TCN)) consisting of an ordered arrangement of Fe and Ni atoms with a 1:1 ratio on alternating planes [5]. The atomic order of the precursors, resembling the atomic arrangement of the L10 structure, allows reducing the energy and time required to order the Fe and Ni atoms thus driving the formation of the L10 phase. Carbon coated (2-4nm) FeNi alloy nanoparticles (20 – 120 nm) with a L10 phase percentage as large as 55% and noteworthy magnetic properties (Hc up to 0.055 mT, Ms = 135 KAm²/Kg Ni-NPr, and 0.059 mT, Ms 135 KAm²/Kg Fe-TCN, without further treatment) were obtained in the standard experimental conditions and very short time (3-24h). Despite the coercivity is still far from optimal for a high-performance permanent magnet, the results clearly prove the effectiveness and high potential of the developed strategy, which can be exploited, after further optimization, for mass production of highly ordered L10-FeNi nanoparticles for next generation permanent magnets.





TUESDAY

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Jumping the Gap: Can Tetrataenite become a "Hard" Permanent Magnet?

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8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

Tetrataenite, a ferromagnetic, extraterrestrial mineral comprised of iron and nickel arranged in a unique atomically ordered fashion, is under scrutiny as a sustainable advanced permanent magnet for escalating renewable energy aspirations in the transition to a global low-carbon economy. Tetrataenite's intermediate magnetocrystalline anisotropy (MCA) currently designates it as a "semihard" magnetic material. Its empirical hardness parameter is less than unity, categorizing it as a potential "gap magnet" with maximum stored energy between that of the weaker oxide magnetic materials and the rare-earth supermagnets, NdFeB, SmCo, etc. However, all experimental reports of tetrataenite's MCA, measured from unoptimized natural materials or from highly out-of-equilibrium synthesized forms, are significantly greater than ab initio computational determinations which assume perfect atomic order within the lattice [1]. This result is striking since such computational outcomes show great fidelity when applied to other ferromagnetic transition-metal-based compounds with tetrataenite's ordered crystal structure (FePt, FePd, CoPt) [2]. The computations also show a significantly diminished MCA for imperfectly ordered crystal structures [3]. In this work, new ab initio computational modelling reveals that undiscovered phenomena impacting Fe-Ni interatomic interactions must be in play. These results suggest that atomically optimized, highly ordered tetrataenite may indeed be included in the category of a truly "hard" ferromagnetic material with a hardness parameter greater than unity, accompanied by decisive technological relevance.

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Hard and semi-hard Fe-based magnetic materials

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8. Rare Earth-Free Magnets, September 5, 2023, 16:30 - 18:00

Fe-based magnetic materials are widely applied in technologies and industries, with most of the applications for soft magnetic materials, because of the low magnetocrystalline anisotropy (MCA) of bcc Fe. However, it is possible to realize magnetic hardening in Fe-based materials as we have learned from the early carbon steel permanent magnets, although the coercivity of the magnets was modest. Recent efforts to search for rare-earth-free hard magnetic materials have shown more promising evidence for achieving high MCA in Fe-based materials. In this paper, we present recent developments of Fe-based hard and semi-hard magnetic materials with a focus on mechanisms of high MCA in Fe-based phases and the related crystal and electronic structures. We have identified the structures and the magnetic properties of the Fe-based binary or ternary systems containing p-block and d-block elements with considerable MCA. Furthermore, it is also important to know and to understand that the MCA in Fe-based magnetic materials can be tailored/enhanced through chemical and/or structural modifications that will lead to "artificially engineered" hard and semi-hard magnetic materials in the future. Experimental results on the Fe-rich carbide nanoparticles and nanorods with different aspect ratio will be discussed as an example of the materials investigated.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



PLENARY TALK

PLENARY 2 - M. COEY

WEDNESDAY 6 SEPTEMBER 09:00 - 09:30 ELGAR CONCERT HALL - THE BRAMALL



Plenary - 2

Why is it so Difficult to Make a New Permanent Magnet?

M. Coey

School of Physics, Trinity College, Dublin, Ireland

Plenary Talk, September 6, 2023, 09:00 - 09:30

Rare earth permanent magnets are a mature technology, which received a jolt with the 2012 rare earth crisis, when many ideas from the 1980s and 1990s about potential new hard magnets containing little or no rare earth (or heavy rare earth) were revived. Meanwhile Nd-Fe-B magnets have been skilfully optimized for a wide range of applications with different performance-cost requirements. Sm-Co is the material of choice when high-temperature stability is required, and Sm-Fe-N is finding its way into some niche applications. The scope for improvement of these basic materials by substitution has been thoroughly explored, and the effects of processing techniques on their microstructure and hysteresis are largely understood. Of the big ideas from a generation ago, the one that had real potential to raise the record energy product significantly, was the oriented exchange spring hard/soft nanocomposite magnet, which has been very challenging. Superconducting permanent magnets proved impractical. 3D printing of permanent magnet demand. The materials genome has not yet led to the identification of a successful new permanent magnet.

The exceptionally-favourable magnetocrystalline anisotropy in SmCo₅ and serendipitous phase relations in Nd-Fe-B and in Sm-Co have underpinned the development and dissemination of high-performance permanent magnets. A useful new material would have to meet a high bar. First base for a new potential magnet are adequate intrinsic magnetic properties — magnetization M_s (magnetic moment/m³), Curie temperature, magnetocrystalline anisotropy K_{η} , which give a hardness parameter $k = \sqrt{(K_1/\mu_0 M_s^2)} > 1$ and an interesting upper limit on potential energy product $\frac{1}{4}\mu_0 M_s^2$. This is the starting point, whether for a new champion magnet, a gap magnet or a bonded magnet. The subsequent bases in the figure are the identification of a potential route to develop coercivity in the new material, the optimization of hysteresis to yield a square loop and finally creation of an industrial manufacturing process appropriate for specific mass or niche applications. On past form the whole process takes about 20 years. The problems raised by every material system are complex and different. There is hope that machine learning and artificial intelligence may shorten this time dramatically.



A different approach to meet the burgeoning demand for high-performance magnets would be to establish independent supply chains for all five steps from mining to manufacture that could match supply and demand for the rare-earth magnets. There is potential to investigate novel electric machine design with recycling in mind. There is much scope to develop new permanent applications. A sound scientific and economic case needs to be made to justify investment in continued research aimed at discovery and development of new permanent magnet materials.



The Rare Earth diamond. The one home run was SmCo₅



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (09)

SESSION 9: RE-CO MAGNETS AND PROCESSING

WEDNESDAY 6 SEPTEMBER 09:30 - 11:00 ELGAR CONCERT HALL - THE BRAMALL



Towards the Adaptation of Sm(Fe,Co,M)₁₂ Compounds for New Rare-earth-lean Permanent Magnets

G. Hadjipanayis^{1,2}, A. Gabay¹, C. Han¹, C. Ni¹ ¹University of Delaware, Newark, USA, ²Northeastern University, Boston, USA

9. RE-Co Magnets and Processing, September 6, 2023, 09:30 - 11:00

More than half a decade has passed since the Sm(Fe,Co,M)₁₂ compounds were projected to surpass the Nd₂Fe₁₄B as a base material of the high-energy permanent magnets [1,2]. This goal still seems distant, yet certain progress has been made, and the remaining obstacles are now understood much better. In this presentation, we review state-of-the-art results of the ongoing efforts to manufacture competitive 1:12 magnets, the proposed alternative approaches and the relevant materials science findings. Following the highly successful development of the Nd-Fe-B magnets, the high-performance 1:12 magnets are being pursued through liquid-phase sintering which is expected to produce fine oriented 1:12 crystallites magnetically isolated by a thin layer of Sm-rich phase. This approach has already yielded reasonably high coercivities [3,4], but the stored energy density is held back because of the large amounts of non-magnetic M elements. The method is likely to receive a boost from the recent studies of the high-temperature phase equilibria [5]. The main alternative route, hot plastic deformation [6], was also inspired by the Nd-Fe-B magnets. Other, somewhat far-fetched ideas, call for obtaining the favorable microstructures via spinodal decomposition [7] or eutectic solidification. Although Ti is still considered the best M element stabilizing the 1:12 structure, sound arguments have been presented recently for V [8] and Mo [9]. Conditions for even higher coercivities attainable in the 1:12 alloys continue to be intensively investigated, with different groups making their contributions through microscopy, studies of model systems like monocrystalline particles prepared via high-temperature calcium reduction [10] and micromagnetic simulations.

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Anisotropy Field Determination in SmCo₅ Sintered Magnets

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9. RE-Co Magnets and Processing, September 6, 2023, 09:30 - 11:00

It is usually believed that single crystals provide the best estimate for the anisotropy field. However, the anisotropy field can be extracted from any sintered sample, once the texture of the sintered magnet is considered. In this contribution, it is discussed how the anisotropic Stoner-Wohlfarth model can be used for anisotropy field determination.

The processing of sintered magnets typically involves the following steps (i) alloy production (ii) milling until single crystal size (iii) alignment under magnetic field (iv) compaction (v) sintering (vi) heat treatment.

During the alignment under a magnetic field, a significant crystallographic texture is introduced, which may be described by a Gaussian function.

Here, the anisotropy field is determined directly in sintered samples, by means of measurements parallel and perpendicular to the alignment direction of the magnet. This avoids the difficult task of obtaining single crystals, and makes possible an estimation of the anisotropy field in any sintered sample.

Not only the anisotropy field, but also the crystallographic texture and the saturation magnetization can be found with great accuracy.

Since the magnetic measurements depend upon the entire volume of the samples, better statistics are obtained than with EBSD (electron back scattered diffraction) or XRD (x-ray diffraction) Schulz pole figures, which are methods that evaluate only a portion of the surface of the samples. Furthermore, the texture can be determined directly from magnetic measurements, avoiding these techniques that require laborious sample preparation.

It is found that, at the room temperature, the anisotropy field of $SmCo_5$ is ~52 Tesla (with null K2 - second order anisotropy constant). The crystallographic texture and the saturation magnetization of several SmCo5 sintered magnets were also estimated.

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Nanocrystalline Sm-Co-Cu Bulk Magnets Prepared by Low-oxygen Nanopowder Metallurgy Process

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National Institute of Advanced Industrial Science and Technology (AIST), Nagoya, Japan

9. RE-Co Magnets and Processing, September 6, 2023, 09:30 - 11:00

Sm-Co permanent magnets are indispensable for high temperature applications as hard magnetic materials [1]. In the previous study, we successfully consolidated the isotropic nanocrystalline Sm-Co bulk magnet with a huge coercivity of 5.2 T and remarkable thermal stability using the Sm-Co alloy nanopowder synthesized by the induction thermal plasma (ITP) process [2]. However, according to the XRD profile, the volume fraction of the SmCo₅ phase in the prepared nanopowder by the ITP process is not high enough. Here, this study focuses on Cu doping to increase the SmCo₅ phase present in Sm-Co nanopowders to synthesize magnetically stronger nanopowders. The Sm-Co-Cu alloy nanopowder was prepared by the ITP process using the mixed raw powders in the ratio of Sm:Co:Cu=1:4.5:0.5 at%. According to the X-ray diffraction (XRD) measurement, almost only the SmCo₅ phase was detected as the alloy phase. Rietveld refinement showed that the SmCo₅ phase content was ~60 wt%, which is about 1.6 times higher than that of the undoped Sm-Co nanopowder. The homogeneous distribution of the third element Cu in the nanoparticles was clearly observed by scanning transmission electron microscopy (STEM). The coercivity was 1.9 T at 300 K for the Sm-Co-Cu nanopowder. The Sm-Co-Cu nanopowder which was sufficiently aligned by an external magnetic field of 2 T showed anisotropic magnetic behavior, which was better than that of the Sm-Co nanopowder without Cu doping. Using this unique Sm-Co-Cu nanopowder, the anisotropic nanocrystalline Sm-Co-Cu bulk magnet was consolidated. Thus, this study contributes to the demonstration of the potential of rare earth alloy nanopowders for the fabrication of highperformance anisotropic bulk magnets.

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Strip Casting of Sm₂TM₁₇-type Alloys for Production of the Metastable SmTM₇ Phase

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9. RE-Co Magnets and Processing, September 6, 2023, 09:30 - 11:00

Conventional book casting of Sm_2TM_{17} -type alloys (where TM=Co,Fe,Cu,Zr) leads to a coarse, highly segregated microstructure, predominantly due to slow, variable cooling rate from the mould surface towards the centre of the ingot. These cast alloys require a long homogenisation treatment to remove this segregation and develop a super-saturated, metastable $SmTM_7$ -type hexagonal phase. This $SmTM_7$ phase is phase is a vital precursor phase for magnet production in order to precipitate the Sm_2TM_{17} -type rhombohedral and $SmTM_5$ -type hexagonal phases required to develop the cellular structure responsible for high magnetic properties.

In this work, strip casting was employed to facilitate rapid solidification to develop thin flakes (<0.5 mm thick) with a columnar grain structure. Rapid cooling has the potential to produce a homogenous microstructure consisting predominantly of the metastable SmTM₇ phase. This could remove or significantly reduce the need for the energy-intensive homogenisation treatment.

This paper investigates the effect of wheel speed (and hence cooling rate) on flake thickness, microstructure and phase balance of the cast alloys. It was shown that for wheel speeds between 1.0-3.1 m/s the microstructure showed large variation, however, in all cases evidence of the columnar $SmTM_7$ phase was presented. The adhesion between the melt and the wheel was critical for nucleation of $SmTM_7$ grains and the wheel speed controlled the thickness of the flake. It was determined that in order to achieve a homogenous columnar $SmTM_7$ structure, the maximum flake thickness should be limited to 280 µm to avoid formation of equiaxed Sm_2TM_{17} grains through insufficient cooling.



Rare Earth and Transition Metal Based High Entropy Alloys (HEAs) as Building Blocks for Novel Permanent Magnets

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9. RE-Co Magnets and Processing, September 6, 2023, 09:30 - 11:00

The landscape for novel magnetic phases suitable for permanent magnets beyond the current NdFeB and SmCo is very limited by single rare-earth elements and for the last 40 years no new phase has been discovered [1]. The current limitations due to the available expensive rare earths or cobalt, present in the most common families of magnetic alloys suitable for permanent magnets, can be overcome by creating artificial multi-elements, equimolar solid solutions of rare earth (RE-HEAs) or transition elements with high magnetization (TM-HEAs), as building blocks for the new generation of magnetic alloys [2,3]. Due to strong demand and origin of supply of critical materials new approaches must be developed a) to modify the existing (PMs) with new elements that are abundant and less expensive [4] and b) develop high entropy stabilized novel magnetic alloys, that are suitable as permanent magnets. We propose, based on the idea of HEAs, to create new artificial elements based on the concept of HEAs, and replace the rare-earths or transition metals aiming to increase the degrees of freedom for designing new magnetic alloys with the nearly the same properties as the existing ones or even new ones, but at a much lower prices. The proposed novel approach entails a great scientific challenge that new multi-elements with tunable atomic radii, valence electron concentration and electronegativity can be developed, widening the base of elements acting as a replacement for expensive single elements in alloys and meets the demand driven challenge for lower price high performance magnets.

We will provide an overview of the novel approach with examples for the best known permanent magnets of the type $(RE-HEAS)_2Fe_{14}B$, $(RE-HEAS)Fe_{12}$ and the $Sm(Fe_3CoNi)$ [5] supported by experimental data that confirm the validity of our approach with excellent results.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATION (010)

SESSION 10: DENSITY FUNCTIONAL THEORY (DFT) AND MICROMAGNETIC MODELLING

WEDNESDAY 6 SEPTEMBER 11:30 - 12:50 ELGAR CONCERT HALL - THE BRAMALL



O10-1

First-principles Calculations on Rare Earth magnets - Bridging the Gap Between Theory and Reality

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10. DFT and Micromagnetic Modelling, September 6, 2023, 11:30 - 12:50

For several decades now, density-functional theory (DFT) has been employed as first-principles technique to understand the fundamental properties of rare-earth magnets, with theoretical refinements providing an increasingly accurate picture of these fascinating materials. However, the computational cost associated with DFT places limits on the complexity of the structures which can be modelled, with calculations most conveniently performed on pristine materials. This is problematic if we wish to better understand the coercivity of a magnet, since magnetization reversal and domain wall dynamics depend critically on defects, surfaces and interfaces [1]. Here, we present our efforts to obtain a more realistic picture of rare earth magnets by incorporating defects into our calculations. Specifically, we will demonstrate how defects in the transition metal sublattices can dramatically modify the single-ion magnetocrystalline anisotropy of the rare earth atoms, through perturbing the crystal field. We will show how results obtained from first principles [2] can be parameterized using a simple model which is intuitive and can be readily incorporated into larger-lengthscale atomistic spin-dynamics calculations and statistical analyses. We use as our prototype example the "one-twelve" rare earth magnets [3], where understanding transition metal substitution is critical due to the intrinsic instability of the pure RFe₁₂ compound. We will also discuss the applicability of the method to different RE magnet classes and architectures, making the case that our novel and efficient method paves the way for further understanding and development of materials with enhanced magnetocrystalline anisotropy.

Acknowledgements: We gratefully acknowledge the support of the UK EPSRC, Grants No. EP/ M028941/1 and EP/W021331/1. This work is also supported in part by the U.S. Department of Energy, Office of Basic Energy Sciences under Award Number DE SC0022168 and by the U.S. National Science Foundation under Award ID 2118164.

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Crucial Role of Fe in Determining the Hard Magnetic Properties of Nd₂Fe₁₄B: Finite Temperature, First-principles Theory Calculations.

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10. DFT and Micromagnetic Modelling, September 6, 2023, 11:30 - 12:50

 $Nd_{2}Fe_{14}B's$ unsurpassed, hard magnetic properties for a wide range of temperatures result from a combination of a large volume magnetisation from Fe and a strong single-ion anisotropy from Nd. Here, using finite temperature first-principles calculations [1,2], we focus on the other crucial roles played by the Fe atoms in maintaining the magnetic order on the Nd sub-lattices, and hence the large magnetic anisotropy, and directly generating significant uniaxial anisotropy at high temperatures. We identify effective spins for atomistic modelling from the material's interacting electrons and quantify pairwise and higher order, non-pairwise magnetic interactions among them. We find the Nd spins couple most strongly to spins on sites belonging to two specific Fe sublattices, 8j1, 8j2. Moreover the Fe 8j1 sublattice also provides the electronic origin of the unusual, non-monotonic temperature dependence of the anisotropy of $Y_2Fe_{14}B$. Our work provides atomic-level resolution of the properties of this fascinating magnetic material.



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Exploring Coercivity Limits in Ultrafine-grained Nd-Fe-B Magnets with Deep Learning Image Segmentation and Micromagnetic Simulation

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10. DFT and Micromagnetic Modelling, September 6, 2023, 11:30 - 12:50

Nd-Fe-B magnets produced by hot-deformation process offer unique flexibility in microstructure and grain boundary engineering. Ultrafine grain size and grain boundary diffusion process (GBDP) have been exploited to achieve excellent energy product and thermal stability required for applications without using heavy rare earths [1]. In this work, we took advantage of the experimentally obtained ultrafine grain size [2] to construct a micromagnetic model with both realistic scale and microstructure. To reproduce the microstructure of a real magnet, we combined FIB-SEM tomography with deep-learning (DL) segmentation and geometry reconstruction. The model was used to investigate the limits of coercivity enhancement in hot-deformed magnets with different intergranular phase (IGP) magnetization.

FIB-SEM tomography data were obtained for a $Nd_{13\cdot4}Fe_{76\cdot3}Co_{4\cdot5}Ga_{0\cdot5}B_{5\cdot3}$ hot-deformed magnet without GBDP [3]. A DL segmentation model was trained on a small part of the FIB-SEM data and used to process the remaining volume of $3\times3\times3 \ \mu\text{m}^3$. For comparison, we have also considered a less expensive and time-consuming workflow to build a synthetic model based on the experimental grain size distribution extracted from 2D SEM images with a DL segmentation model. After building the geometry, IGP and triple junctions were reconstructed (Fig.1(a,b)). Small and narrow geometric elements were eliminated using a custom algorithm to ensure high mesh quality.

Realistic texture, IGP, and grain size distribution allowed us to closely reproduce the experimental coercivity without introducing artificial nucleation sites. We have shown quantitatively that grain refinement has a limited potential for coercivity enhancement even when the magnetization of the IGP is controlled (Fig.1(c)). To explain simulation results, contributions of exchange field, demagnetization field, and thermal fluctuations to the coercivity reduction have been analyzed individually. The developed workflow and micromagnetic model provide the flexibility necessary for advanced analysis of coercivity mechanisms and can provide guidance for the design of high-performance magnets.





Figure 1: FIB-SEM tomography-based geometry with coarse (a) and fine (b) grains used for micromagnetsic simulation. IGP is shown in yellow colour, nonmagnetic triple junctions are set to invisible. (c) Dependencies of coercivity vs magnetization of IGP simulated for a geometry with coarse and fine grains.

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Micromagnetic Simulation of Nanostructured Sm₂ (Co,Fe,Cu,Zr)₁₇ Magnets

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10. DFT and Micromagnetic Modelling, September 6, 2023, 11:30 - 12:50

SmCo-based permanent magnets are of particular interest for high temperature applications, where their magnetic properties are superior to NdFeB-based permanent magnets. The comparatively high temperature stability can be attributed to the high Curie temperature TC in combination with high magnetocrystalline anisotropy [1]. The coercivity in Sm₂(Co,Fe,Cu,Zr)₁₇ magnets mechanistically depends on the pinning of magnetic domains and particularly depends on the microstructure. In our project Sm₂(Co,Fe,Cu,Zr)₁₇ magnets with record breaking energy product of up to 282 kJ/ m³ were produced with approximately 20 at% Fe. Atom probe tomography (APT) revealed thin layers of the Cu-rich Sm(Co,Cu)₅ phase around the Zr-rich platelets [2]. Using the model of Katter [3] micromagnetic simulations were performed to systematically investigate the influence of the microstructure and the distribution of the Sm₂(Co,Fe)₁₇, Sm(Co,Cu)₅, and Zr-rich phase. For the simulations, MUMAX³ [4] was used to simulate a 512 nm x 512 nm x 2 nm structure with 1 nm x 1nm x 1nm cells and periodic boundary conditions. The simulations showed, that even a single layer of the Sm(Co,Cu)₅ phase around the Zr-rich platelets leads to a significant enhancement of the domain wall pinning at the interface between the Sm(Co,Cu)₅ network structure and the coated Zrplatelets. The increased pinning strength should be resulted from the increased difference of the domain wall energies. Systematic studies with varied geometrical and compositional parameters, e.g., thickness of the Zr-platelets and Cu concentration of the Sm(Co,Cu)₅ phase surrounding the platelets, revealed that the strength of the pinning increases with the thickness of the Zr-platelets and with increasing Cu concentration. Overall, the micromagnetic simulations based on the APT data can explain the outstanding magnetic properties of the Sm₂(Co,Fe,Cu,Zr)₁₇ magnets found.





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Discovery of Rare-earth Lean High-performance Permanent Magnets

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10. DFT and Micromagnetic Modelling, 11:30 - 12:50

The development and deployment of high-performance permanent magnets depends on the utilization of suitable magnetic and bonding elements in the anisotropic crystal structure. The first prerequisite is to pinpoint which atoms in the crystal provide magnetization, ferromagnetic transition temperature, and magnetic anisotropy. If these intrinsic permanent magnet properties along with the evolution of microstructure are translated to coercivity and remanence, one can then be equipped with the high-performance permanent magnet. We present here our newly discovered crystallographic site substituted high-performance hexaferrites to rare-earth cobalt to neo magnet materials from density functional theory (DFT) [1, 2] and micromagnetic Modelling. These materials can be applicable ranging from household electronic to magnetic cooling to vehicle to wind turbine technologies.

Acknowledgements: The research is supported by the Critical Materials Institute, an Energy Innovation Hub funded by the U.S. Department of Energy, Office of Energy Efficiency and Renewable Energy, Advanced Manufacturing Office.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATIONS (011)

SESSION 11: APPLICATIONS OF PERMANENT MAGNETS

WEDNESDAY 6 SEPTEMBER 14:00 - 15:35 ELGAR CONCERT HALL - THE BRAMALL



The Application of Permanent Magnets in Electrical Machines for Aerospace Full Electric and Hybrid Electric Aircrafts

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Rolls-Royce Electrical, Derby, UK

11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

Electrification impacts all Civil and Defence Aerospace market segments. Various companies around the world including incumbent Aerospace company such as Rolls-Royce are progressing electrification in aviation. The Aerospace industry had committed to ambitious environmental targets to meet the Flightpath 2050 and net zero targets, in line with UN Race to Zero commitment. A combination of incremental and disruptive solutions in parallel will be required to meet these targets and to bring opportunities that are unlocked by electrification to the market and product.

Great progress has been made in the electrification journey of Aerospace so far and the electrical machine plays a pivotal role in accelerating the development of electric-powered propulsion. In turn, lightweight, high power density machines enable by permanent magnet technology is a key driver to facilitate electrification opportunities in this energy transition. In this presentation, the use of permanent magnets and their associated challenges and solutions in these electrical machines will be discussed through electrical machine products in the urban air mobility and regional commuter aircraft applications.



WEDNESDAY

011-2

Considerations in Magnets Selection for Industrial Motors: The Case of Permanent Magnet-Assisted Synchronous Reluctance Motors

<u>E. Perigo</u>, D. Tremelling ABB US Corporate Research Center, Raleigh, USA

11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

Industrial motors constitute a strategic area of application of permanent magnets (PMs) given their utilization in virtually all manufacturing sites and emerging areas such as e-mobility. Industrial machines that make use of PMs enable higher efficiency compared to traditional – and market-dominant – squirrel cage induction motors.

One specific machine topology that continuously gains attention is the synchronous reluctance motor (SynRM). Its construction shares similarities when compared to that of induction motors (e.g., stator preparation) delivering, concomitantly, higher efficiency even in the absence of PM implementation. Its performance can still be enhanced with the addition of PMs enabling the named permanent magnet-assisted synchronous reluctance motor (PMaSynRM). Therefore, from the technical standpoint, the PMaSynRM occupies a strategic position to substitute – depending on the application space and demands – induction motors and be the next industrial standard motor.

Improvements in the remanence – and maximum energy product – can be classified as secondary for mass implementation of PMaSynRM. Since the rotor lamination can be designed with increased saliency ratios that mitigate requirements for high(er)-Jr magnets – details presented in the full article –, all engineering magnetic materials commercially available could be utilized for these machines in either sintered or bonded forms. No other machine topology presents such a low dependence on Jr as it, which not only enables changes in the approach how to develop hard materials for this end but also highlights a potentially wider range of compounds selection that are not even yet commercialized. Focus is then required to develop compounds with a knee field as high (in magnitude) as possible combined with temperature coefficients of magnetic properties able to support the materials' performance requirements for temperature windows of the order of 110°C

Novel hard magnetic materials opportunities for application in PMaSynRM are discussed, providing details on cost performance.



O11-3

Special Features of the NdFeB Hot Pressing Process- a Pump Manufacturer as Magnet Producer

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11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

The most efficient electric motors nowadays are equipped with magnets made of NdFeB, which is well-known. However, less known is the fact that there are two manufacturing processes for this group of magnetic materials. This paper takes a closer look at the manufacturing process using hot pressing and hot deformation and highlights the peculiarities that arise from the fact that in this process, the anisotropy or alignment is not achieved in an external magnetic field in a press as usual. It is shown how this process can easily produce radially preferentially oriented magnetic geometries, but without the material waste that occurs in the usual backward extrusion pressing for the production of rings.

In addition, due to the very short thermal process time, the manufacturing process offers the possibility to produce zones of different magnetic properties in one magnet. This is not possible in a long sintering process at high temperatures because differences in concentration and chemistry would be balanced by diffusion. The need for such different zones has been demonstrated in FEM calculations and has proven to be advantageous.



Geometry-based Reduction of Rare-earth-containing Raw Materials for Permanent Magnets

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11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

We demonstrate how to reduce the consumption of critical raw materials by a proper design of a permanent magnet. Our case study is a magnetic harvester for smartphone self-charging, which is a device exploiting the principle of electromagnetic induction to generate electrical energy from magnetic fields. It is supposed to be use in emergency situations when the phone's battery is dead, and there is no access to a power outlet. The essential parts are a coil and a magnet, as the source of an inhomogeneous magnetic field. The vibration of either one of them produces an oscillating magnetic flux, resulting in the induced voltage. We focus on reducing the cost and simplifying the magnet production by introducing notches in the design. They contribute to the required inhomogeneity of the field and to a considerably lower consumption of the raw materials as compared with conventional solutions, based, for example, on Halbach arrays. The efficiency of the proposed set up is proven on the basis of the finite-element modelling.



O11-5

Challenges in Applications of Permanent Magnets in Accelerators on the Example of the Major Accelerator Upgrade at DESY

D. Völker, A. Klumpp

Deutsches Elektronen-Synchrotron DESY, Hamburg, Germany

11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

Magnets play an important role in accelerator science. Magnets guide and focus charged particle beam, and undulators and wigglers are used to emit light for photo science. Recently, the importance of energy savings has increased and the lattice structures have become smaller and more complex, so that now electromagnets are partly replaced by permanent magnets (i.e. at the facilities ESRF, MAX IV).

At DESY a major upgrade of the light source accelerator is planned and for bending of the electron beam the use of permanent magnets is foreseen. Additionally, first designs for focusing with permanent magnets are done.

But every light has its shadow. On the one hand the possible energy savings in operation are huge. But on the other hand permanent magnets are made from problematic material - rare earth elements (REE). DESY as a large research facility wants to take responsibility seriously and therefore organized a workshop on rare earths elements in the frame work of the EU-funded I.FAST project (Innovation Fostering in Accelerator Science and Technology). The topics were the entire life cycle with a focus on the problematic situation in mining and processing and also the difficulties in recycling. As result DESY supports the research on recycling of permanent magnets with old undulator magnetic material (HU Pforzheim) and is starting to develop a certification scheme for the purchase of such materials.

We would like to take the opportunity to further raise awareness for this challenge and present the results of the workshop. Furthermore we would like to introduce the upgrade plans for Petra IV (the accelerator light source) focusing on the permanent magnets. Questions of critical materials and their recyclability shall already be part of the accelerator design phase. Also, first ideas for certification schemes for future procurement will be outlined.



Development and use of NdFeB Micro-magnets

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11. Applications of Permanent Magnets, September 6, 2023, 14:00 - 15:35

NdFeB-based magnets are key components of a range of macroscopic devices (motors, generators, actuators...) and hold enormous potential for the development of micro-scaled devices (MEMS) with applications in fields as diverse as telecommunications, energy management, Internet of Things, and bio-technology. The emergence of magnetic micro-systems or MEMS using high performance hard magnetic materials such as NdFeB requires micro-magnet fabrication techniques which preserve the excellent extrinsic magnetic properties achieved in bulk magnets, while being compatible with the cleanroom microfabrication techniques used to make MEMS. The techniques also have to be up-scalable, to allow massively parallel fabrication of devices. We have already demonstrated that high rate triode sputtering can be used to fabricate coercive NdFeB micro-magnets on Si substrates (either isotropic or out-of-plane textured), and these micro-magnets have been used in a range of bio-related studies. In this talk we will present advances in film patterning and will give examples of recent applications of our high performance NdFeB micro-magnets.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



PRE-DINNER TALK

PLENARY 3 - T. MORCOS

WEDNESDAY 6 SEPTEMBER THE COUNCIL HOUSE - BIRMINGHAM



Plenary - 3

Karl J. Strnat and the University of Dayton Magnetics Laboratory -Their Contributions to the Permanent Magnet Industry

T. Morcos

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Pre-Dinner Talk, September 6, 2023

Karl Strnat is considered the "Father of Rare Earth Magnets". He invented SmCo5 in 1966 – 1967 while at the United States Air Force Materials Laboratory at Wright Patterson Air Force Base in Dayton, Ohio. In 1968, he moved to the University of Dayton (UD) and, with his colleagues Alden Ray and Herbert Mildrum, established the University of Dayton Magnetics Laboratory. Over the next 2 decades, it became the world's premier permanent magnet laboratory and the referee for commercial permanent magnet measurements and specifications. In over 20 years at the UD Magnetics Lab, Dr. Strnat trained scores of undergraduate and graduate students in the "informed alchemy", art, and science of permanent magnets. In 1974, Karl organized the first "International Workshop on Rare Earth Magnets and their Applications", and since that time there have been 25 more REPM Workshops, with this 2023 REPM Workshop being the 27th in the series. This lecture will review the contributions of Dr. Strnat and his UD Magnetics Laboratory colleagues to the scientific foundation and astronomical economic growth of the rare earth permanent magnet industry.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATIONS (012)

SESSION 12: APPLICATIONS AND SUSTAINABILITY OF MAGNETS

THURSDAY 7 SEPTEMBER 09:00 - 10:40 ELGAR CONCERT HALL - THE BRAMALL



Sustainability of Tomorrow's Magnets and their Applications

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

Magnets are key enablers for the green energy transition. High performance hard and soft magnets are crucial components of energy-related technologies, such as direct drive wind turbines and e-mobility. They are also important in robotics and automatization, sensors, actuators, and information technology. The magnetocaloric effect (MCE) is the key for new and disruptive solid state-based refrigeration. The rare earth elements (REEs), an important class of the critical raw materials (CRMs), are essential constituents of the highest performing magnets and are highlighted in the raw and advanced materials flow essential for (EU) Industrial Ecosystems and a net zero emission scenario.

Important questions arise around the different mitigation scenarios addressing the criticality of REEs; they are classified as strategic elements and there are many bottlenecks along the supply and value chain. Supply deficits will endanger the development of technologies which abate the climate change and will also impact on other strategic sectors. This supply chain has to be secure, affordable and sustainable and in order to achieve this, we need the diversification of primary CRMs, material science and process solutions for new efficient alloy and microstructure design, substitutional materials and effective short and long loop recycling routes. Assessing these different strategies in terms of their energy needs, CO₂ balance, other emissions, all impacting on a price we are paying in the short and long term is a complex task. Considering that this assessment will differ greatly, when the primary magnet making is compared with an application such as an electric vehicle with a certain lifetime in which the magnet is a key component, will make an analysis even more complicated.



THURSDAY

012-2

Automated High-Speed Approaches for the Extraction of Permanent Magnets from Hard Disk Drives Components for Circular Economy.

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

In the presented work, automated pilot plant approaches for the extraction of Rare Earth (RE) permanent magnets from computer hard disk drives (HDD) are described, demonstrating a commercially viable way to utilize abundant available sources of end-of-life (EOL) magnets. It describes different ways of data acquisition, development of tailored scanning and handling equipment and the integration into an automated HDD processing line. By full system integration, a mobile approach for on-site destruction of HDDs in server farms in compliance with the European Data Protection Regulation (GDPR) is provided, which allows the magnets to be separated and the data carrier to be shredded in an automated manner. Input and output scenarios including the future development of this waste stream is considered.

The work will also help to transfer the experience gained in the mobile pilot plant to other future EOL material sources such as wind turbines and mixed electronic scrap



HURSDA

012-3

Design of Complex Shaped REPM for Increasing the Performance and Recyclability of Synchro-reluctant Machines

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

Permanent Magnet (PM) motors are predominant in transportation domain for their performance and compacity characteristics, though industry pushes forward for lower cost, higher power densities and solutions for end-of-life (EoL) material efficient recovery. To maximize the torque of PM assisted Synchronous Reluctance (PMaSynRel) motors with the lowest quantity of PM, and to avoid traditional gluing of magnets being a lock for their EoL collection, sintered magnets should ideally exhibit functional curved shapes. Standard process fails to produce such geometries without large critical material loss during machining. The Powder Injection Molding (PIM) technique has been assessed in this work as an efficient process for producing complex shaped magnets while minimizing the environmental impacts and improving the recyclability of EoL products. The issue of the carbon residual content has been addressed showing that magnetic performances comparable to the standard sintering process can be obtained. Considering that PIM enables more degrees of freedom in the magnet and flux barrier shape, an optimized motors, representative of automotive sector needs, has been designed and evaluated. The resulting design is a motor with more than 270 N.m and 190 kW for less than 1.6 kg of magnet, which allows to obtain power and torgue densities of about 6.7 kW and 9.5 N.m per kg of active parts. This corresponds to a torgue of 125 N.m per kg of PM which is more than 30% PM economy than the state of the art. The LCA shows that, despite additional process steps required, PIM designed magnets would have lower environmental impacts compared to the standard route considering the material savings. Furthermore, owing to the possibility to design magnets with geometrical details that helps assembly and recovery of magnets from rotors, the recycling of magnets by short loops from EoL motors would become more attractive.



Processability and Separability of Commercial Anti-corrosion Coatings in HPMS Recycling of NdFeB

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

NdFeB magnet recycling is required to secure the supply of this critical raw material in a reliable, ethical way. Successful recycling in suitable quantities is challenged by product designs that do not allow the extraction and recycling of these high-performance permanent magnets without excessive effort and cost. This is especially not viable for smaller-scale motors, where NdFeB is commonly applied. Therefore, methods to recycle with low levels or even no demounting are researched. Further, the contamination of the recycled material with anti-corrosion coating residues, glues and more leads to downcycling. Different coatings commonly found in end-of-life NdFeB magnets are analyzed for their relevance to design for recycling in different categories to gauge how recycling-friendly they are. The areas of importance are as follows: protectivity, separability before and after hydrogen processing using different methods, and contamination potential by residual coatings as a consequence of failed separation.

The compatibility of different methods and coatings regarding demagnetization, de-coating, and coating separation is tested with magnets in different arrangements of limited integration. Single magnets and magnets integrated into simple arrangements with different common coatings (Zn, Ni, Cu and Epoxy based coatings as well as passivations) are tested, and the recyclability is reviewed and discussed as per what coating parameters influence recyclability and how that can be improved. Small-scale rotors and shelled magnets are trialed by hydrogen processing without manual or machined demounting. Coating removal, mechanically and chemically, is tried both on bulk magnets as well as on pulverized, hydrogen processed magnet powder, expanding previous investigations [1].

It is shown that epoxy coatings are very challenging in in-situ applications while Ni, Cu, and Zn based coatings can be handled well by employing post-HPMS separation techniques.

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Improvement of Magnetic Properties of Recycled Nd-Fe-B Magnets by Employing the GBD Process

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

The exceptional magnetic properties of Nd-Fe-B permanent magnets make them suitable for use in various industrial applications. Consequently, the growing annual requirement for rare-earth-based magnets has resulted in a scarcity of these materials, which could compromise the supply chain of a great number of devices that depend on such kinds of magnets. Therefore, recycling end-of-life magnets from decommissioned devices could be one possible solution to ease this problem [1]. This work evaluates the effect of Grain Boundary Diffusion (GBD) of a Dy-rich phase (Dy-Cu alloy) on the magnetic and corrosion properties of recycled magnets at several treatment conditions. The recycling process begins with the Hydrogen Decrepitation (HD) of end-of-life commercial magnets that have been disassembled from obsolete wind turbines. The HD process was followed by milling, magnetic alignment, isostatic pressing, and sintering at a maximum temperature of 1100 °C. The commercial and recycled magnets have a coercive field of around 1250 kA/m and 800 kA/m, respectively. Regarding the GBD process, the optimal condition was achieved at 900 °C for 10 hours followed by an annealing step at 525 °C for 1 hour, which resulted in a coercivity of around 1300 kA/m, without any significant reduction in remanence (cf. Figure 1 in Supporting Documents). The microstructural analyses revealed that a Dy-rich shell had formed around the grains of the magnet surface, without any significant coarsening of the grains [2]. Moreover, the GBD process introduces Cu in the intergranular phase, improving the corrosion resistance by minimizing the disparity of electrochemical potentials between the Nd-Fe-B phase and the Nd-rich phase [3]. In summary, the GBD technique is an efficient process to improve the magnetic properties and corrosion resistance of recycled Nd-Fe-B magnets; thus it has the potential to reduce the necessity for virgin raw materials and establish a more reliable supply chain.



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Recycling of Sr-ferrite Permanent Magnet Scraps

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12. Applications of Sustainability Magnets, September 7, 2023, 09:00 - 10:40

M-type ferrite especially strontium hexaferrite (SrFe₁₂O₁₀) has opened a particular position in the permanent magnet material market since they were discovered in 1950. The unique properties of SrFe₁₂O₁₉ are sustainability, good performance-to-cost ratio, anticorrosion, chemical stability, and relatively high curie temperature still make them the industry's leading choice. Although they're not as strong as the NdFeB magnets, 1.24 Mtons of SrFe₁₂O₁₉ permanent magnets are produced yearly throughout the global market (about 90% of all permanent magnets on a weight basis) [1]. Due to the enormous market of SrFe₁₂O₁₉ in the world, recycling could benefit the environment, reduce preparation costs, and create a circular economy [2]. M-type ferrite permanent magnets have a wide application in electric motors, ergo electric motor wastes are readily found in vast amounts in scrap yards. In this work, disassembled magnets from used electric motors were crushed and pulverized with a jaw crusher down to 2 mm and then dry ball milled to achieve <10-micron size powder. EDS analysis showed that the Sr-Ferrite magnets were composed of 2.6 at.% Sr, 39.8 at.% Fe, 57.3 at.% O, and 0.3 at.% Co, which is very close to SrFe₁₂O₁₉ composition. Figure 1a shows XRD peaks of 4-hour ball-milled Sr-Ferrite powder matching to the hexagonal M-type phase of SrFe₁₀O₁₀. As produced powders were pressed under 5Tons in a mold and green compact in desired shape and dimensions were manufactured. The sintering process takes place inside the furnace at 1200 °C for 5 minutes followed by 1100 °C for 30 minutes. In the final step, the recycled permanent magnet was revealed after final magnetization [3]. Figures 1b and 1c show the room temperature hysteresis loop of the powder before and after consolidation. The results revealed 100% recycled permanent magnet with 98% density, Mr/Ms value of 0.78, and (BH)max of 11 kJ/m³.







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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATIONS (013)

SESSION 13: ADVANCED CHARACTERISATION

THURSDAY 7 SEPTEMBER 11:10 - 12:30 ELGAR CONCERT HALL - THE BRAMALL



THURSDAY

013-1

New Measurement System for Evaluation of Eddy - Current Loss in Permanent Magnets

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13. Advanced Characterisation, September 7, 2023, 11:10 - 12:30

In the high-speed multi-pole synchronous electric machines the eddy current loss in permanent magnets has a considerable influence on their efficiency. The heat generated within the rareearth magnets, such neodymium iron boron (NdFeB), increases their temperature and reduces their strength resulting in impaired performance. To meet the growing need for characterization of the permanent magnet properties under dynamic magnetization Brockhaus Messtechnik has developed a new measurement system for evaluation of their eddy current loss under arbitrary magnetization conditions. This measurement system can be used for loss comparison of magnets with different sizes, geometries and lamination ratios, allowing their optimum selection for electric drives and automotive applications.



THURSDA

013-2

Anisotropic Lattice Diffusion of Heavy Rare Earth Elements (Tb, Dy) in the Magnetic Phase of Nd-Fe-B Permanent Magnets: an Experimental Model

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13. Advanced Characterisation, September 7, 2023, 11:10 - 12:30

Traction motors of electric vehicles and wind power generators are major applications for Nd-Fe-B permanent magnets. In order to avoid thermal demagnetization during operation (above 150°C), the coercivity has to be greater than 3.0 T at room temperature [1]. To reach this value, Heavy Rare Earth Elements (HREE) such as Tb or Dy are added by grain boundary diffusion process [2], as this reduces the amount of these critical materials and limits the decrease in remanence. This process leads to a core-shell microstructure characterized by the formation of an HREE-enriched shell surrounding the Nd₂Fe₁₄B magnetic phase.

Previous studies have highlighted various mechanisms that could be involved in the formation of the core-shell microstructure : lattice diffusion [3], partial melting and solidification of grains [4], chemically induced liquid film migration [5]. However, these studies were performed on either polycrystals or sintered magnets. For the first time, this study investigates these mechanisms on single crystals.

 $(Nd_{1-x}Tb_x)_2Fe_{14}B$ single crystals with x between 0 and 0,75 were prepared using the flux-growth method (Figure 1). Diffusion couples with different concentrations of Tb were assembled and annealed at different temperatures. We studied diffusion along the main crystallographic directions of the tetragonal structure of $Nd_2Fe_{14}B$: the axis of easy magnetization c and the perpendicular lattice direction a (Figure 2). The compositional profiles of the diffusion couples are characterised by scanning electron microscopy (Figure 3). We used an energy dispersive X-ray (EDX) detector with high collection efficiency (FlatQuad), even at low beam current, allowing high spatial resolution. Quantitative chemical analysis is provided by wavelength dispersive spectroscopy (WDS) because its high spectral resolution allows to solve EDS peak overlaps of HREE and Fe. By this method, we aim to provide new insights into the HREE diffusion mechanism in the $Nd_2Fe_{14}B$ lattice by taking into account its crystallographic anisotropy.



Figure 1: Image of a single crystal of Nd₂Fe₁₄B obtained by the flux growth method with Laue diffraction patterns used to identify its orientation.



Figure 2: Back scattered electron (BSE) SEM image of a diffusion couple along c-axis between a NdTbFe₁₄B single crystal and a Nd₂Fe₁₄B single crystal at 1050°C for 5 hours. EDS Nd and Tb elemental maps are superimposed at the interface. Red arrows indicate c-axis directions for each single crystal. The black arrow indicates the position of the profile shown in Figure 2.



Figure 3: a) EDS Nd and Tb elemental map and b) Nd and Tb concentration profiles associated for NdTbFe₁₄B and Nd₂Fe₁₄B c-axis diffusion at 1050°C for 5 hours. The yellow dotted line indicates the position of the initial contact interface.

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Microscopic Consequences of Strain and Magnetic Field on Atomic Ordering in MnAl

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13. Advanced Characterisation, September 7, 2023, 11:10 - 12:30

Identification of efficient processing routes to realize and maximize technical magnetic properties remains an important topic of interest. In particular, the ferromagnetic compound MnAl gains appreciable magnetocrystalline anisotropy when ordered into the tetragonal τ -phase (L1₀-type) crystal structure [1-3]. Recent data confirm that the transformation of the metastable disordered (ɛ-phase) precursor to the L1_o form in MnAl is increased by ~35 wt. % upon application of an applied static magnetic field during thermal treatment, relative to field-free thermal treatment. Nanoscale information on the mechanism(s) underlying this accelerated transformation was obtained using selected-area electron diffraction (SAED) and convergent beam electron diffraction (CBED) to reveal relationships between the ε- and τ-phases in the field-annealed specimen (Figures 1a & b). It is observed that the application of a magnetic field during heat treatment produces a large percentage of L1₀-structured grains located close to the initial solidification surface of meltspun MnAl ribbons (Figure 1c). It is hypothesized that the presence of residual strain localized at grain boundaries, which may have originated from the rapid solidification synthesis process, amplifies the phase transformation responsible for creating the ordered T-phase within the initial ε-phase matrix. The external application of magnetic field is proposed to change the direction of magnetization and favor ordering during the phase transformation. Ongoing research involves insitu TEM experiments to identify nucleation and growth characteristics of L1_o in MnAl subjected to magnetic-field annealing treatment.



Figure 1: a) Cross-sectional TEM image of MnAl ribbon that underwent annealing with a magnetic field. B) Convergent bean electron diffraction from the location indicated by red dot in a, revealing the super-lattice reflections from L1₀ phase. C) TEM image that displays the distribution and morphology of phases present in the melt-spun ribbon.



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13. Advanced Characterisation, September 7, 2023, 11:10 - 12:30

Lorentz electron transmission microscopy (LTEM) is an useful method to characterize the magnetic domain structure of magnetic materials. Observing the evolution of domain structure by LTEM under magnetic field is especially important to understand the micro-mechanisms of the macroproperties of rare-earth permanent magnets (REPM). In order to do it many magnetizing holders have been designed to apply a pure in-plane magnetic field to TEM specimen, however, the maximum field that can be applied in general 200 kV- or 300 kV-TEM does not exceed 0.1 T [1-4]. This low field is difficult to make changes in domains of REPM with high coercivity. The large action scale t of the applied field in current in-situ holder, usually in the millimeter scale (e.g. t ~ 2 mm), which seriously reflects the electron beam even in a low field, is the main reason. Akira Sugawara et al. [5] reduced the value of t to 300 µm, so that the maximum field strength was increased to 0.5 T in a 1MV-LTEM, but whether the holder at such a large field can be used in a general 200kV- or 300kV-TEM remains to be verified. In this work, we largely decrease t to 20 µm and hence elevate the in-plane field strength to 1.2 T (Fig. 1), at the same time, the small deflection angle resulted in can keep TEM image with high quality even with no electron beam deflection compensated. Using the newly-developed holder in a 200kV-TEM, we have researched hot-depressed NdFeB magnet with a coercivity of 2.1 T. The shifting, merging and disappearing of domain walls in the process form demagnetizaed state to saturation state were clearly observed (Fig. 1). The holder with field strength further elevated and variable temperature condition added can be widely unsed in the research of various REPM.





Figure 1: (a) Schematic diagram of the in-situ magnetizing holder, (b) Field strength in air gap as a function of exciting current, (c) In-situ observation of hot-pressed NdFeB with bulk coercivity of 2.1 T.

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THURSDA

013-5

Understanding the Coercivity of Ga-containing Nd-Fe-B Sintered Magnets from Feature Extraction and Selection of X-ray Diffraction Patterns via Dimension Reduction and Sparse Modeling

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13. Advanced Characterisation, September 7, 2023, 11:10 - 12:30

It is known that the coercivity of Ga-containing Nd–Fe–B magnets increases with microstructural changes caused by post-sinter annealing. The correlation between changes in the spatial distribution and fraction of phases and coercivity changes has been revealed qualitatively and heuristically (Fig. 1(a)) [1,2,3]. Furthermore, the causality between them has been discussed from the micromagnetic simulations, which is taken the phases and their characteristics (composition, crystal structure, magnetism, spatial distribution, and orientation) as potential factors of coercivity in a continuum model [4,5]. On the other hand, there are few studies for quantitative and experimental evaluation on the correlation between variations in microstructure and coercivity.

This study investigated the relationship between the coercivity and XRD patterns (spatial averages of microstructural features in a reciprocal space) of Ga 0.5 at% doped Nd–Fe–B magnets during post-sinter annealing using sparse modeling to objectively identify microstructural changes correlated with the coercivity enhancement and to qualitatively evaluate the importance of the microstructural features on the coercivity. For example, Fig. 1(b) shows the results of LASSO sparse modeling of the coercivity using Rietveld parameters of XRD patterns. The results objectively show that the phase state changes correlated with the coercivity enhancement are, in the order of importance, an increase in the phase fraction of Nd₆Fe₁₃Ga phase, a degradation in the crystallinity of Nd₂O₃ phase, a decrease in the phase fraction of dhcp-Nd phase, an increase in the phase fraction of Nd₂O₃ phase. These quantitative and objective results are valid in comparison to the qualitative and heuristic approaches.

This study demonstrates the usefulness of sparse modeling in revealing the critical microstructural features of the magnetic properties of magnetic materials. The objective discovery of the correlations through sparse modeling will accelerate materials research and development by automating decision-making.





Figure 1: Application of machine learning to the analysis of the correlation between coercivity and microstructure. (a) The conventional approach to correlation and causality analysis between mictrostructural feature and coercivity. (b) The resi=ults of LASSO sparse modelling of coercivity using Rietveld parameters of XRD patterns.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



ORAL PRESENTATIONS (014)

SESSION 14: RARE EARTH NITRIDE MAGNETS

THURSDAY 7 SEPTEMBER 13:30 - 15:00 ELGAR CONCERT HALL - THE BRAMALL



THURSDAY

014-1

Recyclable Sm-Fe-N Bonded-Magnet Using Environmentally-Friendly CNF Binder

<u>**T. Iriyama**</u>, S. Takagi, K. Morii Daido Steel Co., Ltd., Nagoya, Japan

14. Rare Earth Nitride Magnets, September 7, 2023, 13:30 - 15:00

High-performance rare-earth bonded magnets, along with sintered counterpart, have been playing an important role in the technological innovations for ICT (Information and Communication Technologies), FA (Factory Automation), automotive, and green technology areas. From the view point of the environmental impacts, it is important to develop recycling technologies for these magnets. Two typical production processes of the bonded rare-earth magnets are injectionmolding and compression-molding. The injection-molded magnet can be recycled, in principal, by pulverizing the scrap magnets followed by injection-molding again, since the binders for injenction-molding, typically polyamide (PA) or polyphenylene sulphide (PPS) are thermoplastic. On the other hand, in the case of compression-molded magnets using thermosetting binders (eg. epoxy), it is difficult to be recycled. In this study, we tried to use cellulose nanofiber (CNF) as a binder for compression-molding. The CNF is an environmentally friendly material, and is a promising candidate for the binder of recyclable bonded magnets, because it can be decomposed by immersing in water. Melt-spun SmFe9N isotropic powder was mixed with 12 wt% of watercontained CNF (CNF:water = 20:80). The mixture was compression-molded with a pressing machine, and then heat-treated at 150 °C for 10 minutes in vacuum. The density of the obtained bonded magnet was 6.1 g/cm³. The Br, Hcj, and (BH)max of the bonded magnet were 0.807 T, 751 kA/m, and 104 kJ/m³, respectively. These values are comparable with those of conventional compression-molded isotropic Sm-Fe-N magnets. It was confirmed that the CNF-bonded Sm-Fe-N magnet was stable in air, but it crumbled after being immersed in water. Then such crumbled powder was compression-molded to produce bonded magnets. Remarkably, no deterioration with the hard magnetic properties after the recycling was observed. We will report this work in detail at the conference.



Magnetically Anisotropic Nanopowder of TbCu₇-type Sm-Fe-N

<u>Y. Hirayama</u>, Z. Liu, W. Yamaguchi, K. Takagi, K. Ozaki National Institute Of Advanced Industrial Science And Technology, JAPAN

14. Rare Earth Nitride Magnets, September 7, 2023, 13:30 - 15:00

In 1996, Sakurada et al. reported a SmZrFeCoN compound as one of the compounds with large saturation magnetization among reported ferromagnetic materials [1]. The saturation magnetization at room temperature exceeds 1.7 T, and the anisotropic magnetic field has a sufficiently large value of 8 MA/m. However, since this compound is a metastable compound of TbCu₇-type, meanwhile the synthesis method is limited. Until now, the liquid quenching method [1] and the mechanical alloying method [2] which can prepare only polycrystalline isotropic samples were reported. Therefore, the potential as a permanent magnet material cannot be maximized. In this study, we synthesized TbCu₇-type Sm-Fe-N nanoparticles using a thermal plasma method that can produce single-crystal nanoparticles [3,4] and demonstrated their magnetic anisotropic behavior.

The average particle size of obtained Sm-Fe nanopowder was around 70 nm. According to the HAADF-STEM image, each particle is found to be single crystalline. The XRD results showed that the TbCu₇-type alloy phases were formed. Then, the Sm-Fe-N nanopowder was prepared at 400 deg.C for 15min under nitrogen flow. After the alignment of the nitrided nanopowder by the external magnetic field of 9 T, the alignment degree estimated by the pole figure measurement for TbCu₇-type Sm-Fe-N nanopowder was found better than 90%. Thus, this work demonstrated that anisotropic nanopowders with metastable phases could be prepared by an induced thermal plasma process.

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THURSDAY

014-3

Sintering of Nd(Fe,Mo)₁₂ Tetragonal Compounds for Permanent Magnets Applications

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14. Rare Earth Nitride Magnets, September 7, 2023, 13:30 - 15:00

Over the last 12 years, a lot of scientific effort has been made for the development of new materials for high performance permanent magnets with reduced or none rare earth (RE) content. $Nd(Fe,Mo)_{12}N$ compounds have been considered as good candidates, due to their excellent intrinsic magnetic properties at room temperature: TC = 320°C, saturation magnetization Ms = 1.3T and large anisotropy field μ 0Ha = 11T [1]. Despite these relatively high intrinsic magnetic properties, achievable coercivity is still low for permanent magnet applications (< 0.6T) [2,3,4]. Single-phase material and proper nitrogenation is a prerequisite to increase the intrinsic magnetic properties. In addition, proper microstructure, formed by single crystalline 1-12 grains magnetic properties, namely remanence and coercivity. Furthermore, maximum performances are expected from fully dense magnets. Nevertheless, nitrided compounds are not stable at conventional densification temperatures, limiting their use as bonded magnets.

This study focuses on the development of dense $Nd(Fe,Mo)_{12}N$ compounds by low temperature sintering process. Alloys with $Nd_{1,25}(Fe,Mo)_{12}Cu_{0.1}$ (wt.%) composition were prepared by strip casting, allowing to obtain more than 96% of 1-12 phase. Liquid phase sintering at low temperature is promoted by the addition of Cu in the $Nd(Fe,Mo)_{12}$ alloy composition, allowing also the formation of a eutectic Nd-Cu grain boundary phase that should magnetically decouple the 1-12 grains.

The SPS (Spark Plasma Sintering) was implemented in order to permit low temperature sintering assisted by the grain boundary liquid phase. The SPS makes possible to apply a homogeneous pressure during the thermal cycle and thus to reduce the densification temperature. Following this procedure, we obtained high densification rates (>98%). Microstructural analysis will allow us to establish the limitations of the process, regarding the desired magnetic performances.

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Improvement of Coercivity of Sm₂Fe₁₇N₃ Powder by Coating with Material Designed to Restore Local Magnetic Anisotropy on the Surface

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14. Rare Earth Nitride Magnets, September 7, 2023, 13:30 - 15:00

The magnetic anisotropy field of Sm₂Fe₁₇N₃ is more than three times that of Nd₂Fe₁₄B [1], but its coercivity at room temperature is at most comparable. The present study aims to elucidate one of the reasons for this anomalously low coercivity of Sm₂Fe₁₇N₃ and to find a methodology for raising it to the originally expected level. In Sm₂Fe₁₇N₃, the rare-earth element Sm is responsible for magnetocrystalline anisotropy, but the base intermetallic compound Sm₂Fe₁₇ is an in-plane anisotropic material, and it transforms into uniaxial anisotropy by nitrogen introduction [2]. The strong uniaxial anisotropy is thus significantly sensitive to the environment. Due to the abrupt discontinuity of crystal periodicity at the surface, the crystal field at the Sm site on the surface should be greatly disrupted. In this situation, there is no longer any guarantee that strong uniaxial anisotropy is maintained on the surface as well as inside [3]. Even without assuming particular nucleation centers, the entire surface is a kind of defect and could be the source of the reverse magnetic domains. We investigated a material that coats the surface of a Sm₂Fe₁₇N₃ and makes the crystal field at the Sm site closer to that inside the crystal while terminating the ferromagnetic order of Fe. By coating with a substance of this kind, we expected that strong uniaxial anisotropy was maintained up to the outermost surface of the magnet phase, which would improve the coercivity. Surface oxide free Sm₂Fe₁₇N₃ fine powder prepared by pulverization in a low-oxygen atmosphere was sputter-coated with designed materials while continuous stirring [4]. The coated powder was annealed in argon to fix the coating layer, resulting in a coercivity increase of about 47% compared to that before coating. The details of coating material design and interfacial structure will be discussed in the presentation.

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014-5

Microstructural Changes at The Sm₂Fe₁₇ and Zn Interface

<u>M. Matsuura</u>, K. Yamamoto *Tohoku University, Japan*

14. Rare Earth Nitride Magnets, September 7, 2023, 13:30 - 15:00

 $Sm_2Fe_{17}N_3$ -based metal bonded magnets has been expected for applying motors under high temperature condition. Otani et al. reported that the use of Zn as the metal binder improved the coercivity of the metal-bonded Sm-Fe-N magnets [1]. Our group has developed magnetic properties of Zn-bonded Sm-Fe-N magnets, and reported high (BH)max of 200 kJm³ [2]. To further increase the magnetic properties of Zn-bonded magnets, it is necessary to control interfacial diffusion and reaction between Sm2Fe17N3 and Zn interface. However, there has not been reported concerning the detailed diffusion behaviour of the Zn to $Sm_2Fe_{17}N_3$ phase. Furthermore, there has been no report of these properties for Sm_2Fe_{17}/Zn interface. Thus, we investigated detailed microstructural changes between Sm_2Fe_{17}/Zn interface during annealing.

We investigated the interdiffusion behaviour, phase and microstructural changes in $Sm_2Fe_{17}Zn$ diffusion couples [3]. Its is found that Zn can diffuse into Sm_2Fe_{17} phase at 320°C, which is below 100°C than melting temperature of Zn (419°C). The Zn-rich region composed of polycrystalline α - and Γ -ZnFe binary alloy phases and a ThMn₁₂-type Sm(Zn,Fe)₁₂ ternary alloy phase appeared at first diffusion stage. At the surface of the Sm_2Fe_{17} phase, a region with a fiber-like microstructure was observed. Interestingly, the fiber-like microstructure consisted of two phases of Γ -FeZn and ThMn₁₂-Sm(ZnFe) phases exhibiting a specific crystal orientational relationship of Γ -FeZn(10-1) [111]//Sm(Zn,Fe)12(01-1)[011]. The interdiffusion coefficient was also evaluated, and it is found that it depended on annealing temperature.

Acknowledgements:_This study is partially supported by Toyota Motor Corporation and Proterial Materials Science Foundation.

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REPM 2023, 3-7 SEPT, BIRMINGHAM UK



PLENARY TALK AND CLOSING REMARKS

PLENARY 4 - K. HONO

THURSDAY 7 SEPTEMBER 15:30 - 17:00 ELGAR CONCERT HALL - THE BRAMALL



Plenary - 4

Permanent Magnet Research at NIMS - ESICMM Succeeded by Permanent Magnet Materials Open Platform and DXMag

K. Hono, T. Okubo

National Institute for Materials Science (NIMS), Tsukuba 305-0047, Japan

Plenary Talk, September 7, 2023, 15:30 - 17:00

The Element Strategy Initiative Center for Magnetic Materials (ESICMM) was established at NIMS in October 2012 for the Element Strategy Project commissioned by the Ministry of Education, Culture, Sports, Science and Technology (MEXT) to develop alternative materials that do not depend on rare earth elements. ESICMM focused on advancing the fundamental understanding needed to develop high-performance magnets that do not depend on critical elements, and organized an interdisciplinary team in electronic theory, microstructural characterization, and processing, covering a wide range from fundamental science to process engineering. Although the project ended in March 2022, the results of ESICMM have had a significant impact on recent permanent magnet research in the world. We felt that the interdisciplinary human resource network on permanent magnets fostered by the ESICMM project should be maintained to continue basic research that will contribute to R&D at permanent magnet manufacturers in Japan. Therefore, NIMS has established a new research center called Permanent Magnet Materials Open Platform (PM-MOP) with Shin-Etsu Chemical, TDK Co, Daido Steel, and PROTERIAL to continue basic research that will contribute to R&D at Japanese permanent magnet manufacturers. NIMS also established a new policy to promote data-driven research using data and AI to efficiently develop materials and launched the Digital Transformation Initiative Center for Magnetic Materials (DXMag) commissioned by MEXT. PM-MOP and DXMag will be organically linked to advance the magnetic materials research needed in Japan in the next 10 years. This presentation will report on these trends in magnetic materials research at NIMS and give a preview of REPM 2025 to be held in Tsukuba City in 2025.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



POSTER PRESENTATIONS - P1

POSTERS P1-1 - P1-32

SESSION 1: RAW MATERIALS, RESOURCES AND MINING

SESSION 2: SUPPLY CHAIN DEVELOPMENT AND MAGNET PROCESSING

SESSION 4: RECYCLING OF RARE EARTH MAGNETS

SESSION 7: RE-FE-B MAGNET PROCESSING AND PROPERTIES

MONDAY 4 SEPTEMBER THE GREAT HALL

or MDNDAY

Recycling NdFeB Waste Based on a Hydrometallurgical Process for Recovering Nd, Dy, and Pr Rare Earth Elements: Design Experiment Screening and Optimization

K. Tanji, K. Ouzaouit, M. Belghiti, I. Lamsayety, H. Faqir, I. Benzakour

Research Center Reminex; MANAGEM Group, Marrakesh, Morocco

1. Raw Materials, Resources and Mining, September 4, 2023

The present work aims to investigate the recovery of Neodymium (Nd), Praseodymium (Pr), and Dysprosium (Dy) oxides from Nd-Fe-B Magnets as secondary resources of rare earth elements (REE) using hydrometallurgical process utilizing only oxalic acid as leaching agent. Oxalic acid has allowed the instantaneous and high-yield recovery of Nd, Pr, and Dy in one stage. In order to evaluate the impact of different parameters on the precipitation yield of Nd, Pr, and Dy, a screening design based on the Hadamard matrix was conducted to assess the effect of stirring speed, time, temperature, L/S ratio, and $[H_2C_2O_4]$. The results of the oxalic acid leaching experiments indicated that under the optimal conditions obtained using response surface methodology (RSM), a precipitate product was characterized using X-ray diffraction (XRD), inductively coupled plasma-atomic emission spectroscopy (ICP-AES), and scanning electron microscopy-energy dispersive X-ray (SEM/EDX) which confirmed the precipitate product as (Nd, Dy, Pr) (C₂O₄)_{3.10}H₂₀ form. Finally, the (Nd, Dy, Pr) (C2O4)3.10H2O was roasted at 750 °C for 2 hours and transformed into Pr₃O₅ and Nd₂O₃, which can be directly applied to synthesize REE alloys. On the other hand, Fe was recovered in the oxalate form of Fe(C₂O₄)_{0.2}H₂₀ using a crystallization process.



Nd₂Fe₁₄B-based Magnets from Atomized Powders

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2. Supply Chain Development and Magnet Processing, September 4, 2023

Nd₂Fe₁₄B-based magnets are used in a wide range of applications including high-performance motors, wind and water turbines, and information technology. While strip-cast alloys are conventionally used to produce NdFeB magnets, we have explored atomized Nd-Fe-B powders as starting alloys for the fabrication of magnets. Nd-Dy-Fe-B powders with different Dy content (0 - 4 wt.%) were produced using an electrode induction melting inert gas atomization (EIGA) method. The alloy composition and atomization conditions including the cooling rate were tuned to obtain NdDyFeB particles with a controlled phase purity and distribution, morphology, and size distribution. A comprehensive analysis using backscattered electron composition (BEC) image measured by scanning electron microscope (SEM) reveals very little or no iron phase in the atomized powders, for example as shown in the case of $Nd_{31}Dy_4Fe_{bal}B_{1.1}$ (wt.%) alloy (Fig. 1a). The SEM results are supported by the Rietveld refinement fitting of the experimental x-ray diffraction (XRD) data, which shows 96.3 wt.% of the main Nd Fe₁₄B phase along with 3.1 wt.% dhcp Nd and 0.6 wt.% Fe (Fig. 1b). By optimizing the subsequent process conditions during hydrogen decrepitation, milling, compaction and alignment, and sintering, magnets with a density of 7.63 g/cm³, and an intrinsic coercivity of about 1592 kA/m (or 20 kOe) and a remanent induction of about 1.13 T (or 11.3 kG) at room temperature were produced from the atomized $Nd_{31}Dy_4Fe_{bal}B_{11}$ powders. The effect of phase distribution and microstructure of the atomized powders on various magnet-making processes will be presented and a comparison between the properties of sintered magnets produced from the strip-cast alloys and atomized powders also will be discussed.



Figure 1: (a) A BEC SEM image and (b) Rietveld refinement fitting of the experimental XRD pattern for the atomized $Nd_{31}Dy_4Fe_{bal}B_{11}$ (wt.%) powders.

Macroscopic Demagnetization Behavior of Sintered Nd-Fe-B Magnets by Grain Boundary Diffusion of Tb

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2. Supply Chain Development and Magnet Processing, September 4, 2023

The demagnetization of the Tb grain boundary diffusion (GBD) processed sintered Nd-Fe-B magnets in comparison with a non-GBD one is investigated via demagnetization curves, coercivity and Dy content profiles in relation to the distributions of magnetic fields of the at various fields (Hext), and numerical simulations. Demagnetization reversal initiates at both the inner part and the surface of the GBD one before reaching their coercivity of the bulk, distinguished from that of the non-GBD one. The field distribution of the demagnetized GBD magnet tend to form a sandwich-layered structure before Hext reaches its HcJ. This can be attributed to the sheilding effect according to the numerical simulation.

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Competitive Threats to NdFeB Magnets in Green Energy Applications - Electric Vehicles and Windpower

Mr. Tony Morcos

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2. Supply Chain Development and Magnet Processing, September 4, 20235

With a few notable exceptions, most electric vehicle (EV) traction motors have employed Interior Permanent Magnet (IPM) Synchronous Motors with a significant mass of NdFeB permanent magnets in their rotors. This holds true, perhaps to a lesser extent, with windpower generators. The EV and windpower green energy markets are rapidly expanding, and there is significant amount of USA and EU investment in the manufacturing of NdFeB magnets to meet the demands of these growing markets. At the present time, China's complete control of the entire supply chain for NdFeB magnets creates significant risks to the manufacturers of these green-energy permanent magnet electrical machines, and on March 1, 2023, Tesla announced it would create an electric vehicle motor with zero rare earth elements in it. Similar "rare-earth free" efforts are being pursued by windpower generator manufacturers. This paper will examine a wide variety of electromagnetic machine topologies to compare and contrast their performance and economic suitability for use in EV traction motors and windpower generators in the 50 kW – 500 kW power range.



Hydrogen Decrepitation and Reprocessing of SmCo₅ Sintered Magnets

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4. Recycling Rare Earth Magnets, September 4, 2023

 $SmCo_5$ sintered magnets are vital for a wide variety of high-temperature applications, such as permanent magnet motors and actuators within the aerospace industry. They benefit these applications through their high coercivity values, usually > 2000 kA/m, their high Curie temperatures of ~800 °C and their low thermal coefficients of remanence and coercivity.

However, these magnets use vast amounts of Sm and Co, both of which have been deemed 'critical materials' of which there is a short supply in the UK and EU. Volatile pricing and shipping lead times have directed research towards methods with which to recycle these materials. One process that could be used as a starting point for recycling Sm_2Co_{17} magnets is hydrogen decrepitation (HD).

This process involves exposing SmCo₅ magnets to hydrogen at room temperature which then allows the hydrogen to diffuse into the material at the Sm-rich grain boundary phase and into the hexagonal SmCo₅ matrix phase. The differential volume expansion caused by the formation of the orthorhombic hydride phase within the matrix generates intergranular and transgranular cracking of the brittle material. The powder generated can then be degassed, milled, aligned, pressed and finally sintered/heat treated to form a recycled compact.

Multiple factors during the post HD processing of SmCo₅ will influence both the final density and magnetic properties of the sintered material. This study primarily looks at the effects of different milling and pressing parameters on the recycled sintered magnets using only the HD material. Smaller particle sizes and narrower particle distributions tend to generate magnets with improved magnetic properties; in some cases exceeding the as-received material. Greater pressing forces led to marginal improvements in recycled magnet density.

Further work in this area will explore the impact of other milling techniques and Sm-rich milled additions on recycled magnet density and magnetic properties.



Production of Anisotropic Permanent Magnets from Recycled Nd-Fe-B Powder with Metal Injection and Powder Extrusion Moulding

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4. Recycling Rare Earth Magnets, September 4, 2023

Over the last fifteen years, several groups have been carrying out research into metal injection moulding (MIM) of Nd-Fe-B powder to produce isotropic or anisotropic rare earth magnets with higher geometrical complexity than possible in the conventional press and sintering approach. However, difficult process ability, due to the high affinity of the powder to oxygen and carbon pickup, remains problematic in terms of obtaining sufficient remanence and coercivity.

In this paper, a novel approach to produce anisotropic Nd-Fe-B magnets from recycling material via MIM in a batch-type process and powder extrusion moulding (PEM) in a continuous process is presented. The processing route is based on the use of powder obtained from recycling of used rare earth magnets by the HPMS (Hydrogen Processing of Magnetic Scrap) process. The paper will present results on tailored powder processing, production of mouldable feedstock based on a specialized binder system and the shaping with magnetically aligned tooling in order to produce anisotropic MIM and PEM green and sintered parts. Magnetic properties and microstructures of debinded and sintered samples are presented and discussed with focus on the influence of the filling ratio and challenging processing conditions on interstitial contents and consequently density and magnetic properties.



Hydrogen-assisted Recycling of Nd-Fe-B Magnets From the end-of-Life Audio Products

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4. Recycling Rare Earth Magnets, September 4, 2023

Rare earth elements are top of the critical materials lists in the EU and US, which play a key role in clean energy technologies including electric vehicles and wind turbine generators. They are also a key component in electronic devices including mobile phones, hard disk drives, and loudspeakers. Here, we build on previous knowledge of the recycling of NdFeB magnets from hard disk drives, using a hydrogen process called HPMS (Hydrogen Processing of Magnet Scrap). This study investigates a new scrap stream from loudspeakers, specifically from vehicles and flat-screen TVs. The extracted magnets from loudspeakers typically had grades on the order of N30/N33 with relatively low coercivity values. The interactions of the extracted magnets with hydrogen were monitored by gravimetric and thermal analyses. The thermodynamics and kinetics of the hydrogenation reaction appear to differ based on their chemical composition, chemical homogeneity, coating state, and surface condition of the magnets. Overall, the HPMS process was demonstrated to successfully produce demagnetised hydrogen decrepitated powder at room temperature under 2 bar of hydrogen, in a commercially viable timeframe (approx. 4h). Zinc was found to be the dominant coating material which peeled away during the HPMS process. The zinc content of the NdFeB alloy powder could be reduced after the hydrogen decrepitation by sieving. The remaining level of zinc had no noticeable impact on the magnetic properties of the recycled magnets. The particle size of the recycled alloy powder could be reduced to below 7 µm by ball milling and knife milling but with an increase in the oxygen level, limiting its sintering ability. Sintered magnet from the recycled alloy powder demonstrated similar magnetic properties (Br: 1.06 T, HcJ: 1240 kA/m, and (BH)max: 220 kJ/m³) after blending with 5 wt% of NdH₂, making vehicles and flat-screen TV speakers ideal sources for a recycled magnet.



Characterization of Nd-Pr-Fe-B Machining Wastes for Recycling Purposes

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4. Recycling Rare Earth Magnets, September 4, 2023

Nd-Pr-Fe-B sintered magnets play a role in emerging technologies, including electric and hybrid vehicles and wind turbines. However, the production of these magnets yields significant quantities of residues during the machining process, which contain critical rare earth elements. Exploring alternative sources of rare earth materials, such as processing wastes, has become a critical aspect of the circular economy as an alternative source for rare earth elements and its products. In this study, Nd-Pr-Fe-B machining wastes from diamond cutting and grinding processes were characterized using X-ray diffraction, Mössbauer spectroscopy, vibrating sample magnetometry, scanning electron microscopy, X-ray fluorescence, elemental analysis, and X-ray photoelectron spectroscopy. The results showed that both wastes exhibited strong phase degradation, with the magnet phases decomposing into oxides, hydroxides, and hydrated oxides, such as Nd(OH)₃, ferrihydrite, and metallic iron. The presence of impurities and a wide particle size distribution resulted in reduced magnetic properties, affecting the magnetization behavior of the machining waste. However, it was found that some of the Nd₂Fe₁₄B phase remains unaltered and that oxides formed during the machining process are located on the surface of the particles. Also, the similarity in chemical composition of the two types of wastes suggests that they can be mixed before recycling. The results obtained provide essential insights into the Nd-Pr-Fe-B machining waste properties, highlighting the potential for efficient recycling and reuse strategies of these materials as secondary sources.

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P1-9

Processing of Grinding Slurries of Samarium Cobalt Magnets for a Circular Economy

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4. Recycling Rare Earth Magnets, September 4, 2023

Recycling Sm-Co magnet sludge is crucial to achieving a circular economy and sustainable production. This byproduct originates from grinding, polishing, and shaping bulk magnets to meet industry needs [1,2]. The carbon content introduced by coolants and emulsifiers is a significant issue when recycling Sm-Co sludge. The carbon deteriorates the magnetic properties of the alloy, and oxidation further complicates the remelting process [3–5]. Despite being energetically and chemically extensive, the current state-of-the-art methods for recycling this waste are pyrometallurgy or hydrometallurgy.

To overcome these challenges, a new approach to direct recycling is adopted. The sludge is decanted to remove excess oil and liquids and dried under a vacuum. The metallic particles are then separated using a permanent magnet after being extracted, hand-grounded, and sieved. The powder is subjected to pyrolysis at different temperatures. The best results were obtained at 600°C for 1 hour under a high vacuum based on the SEM, ICP-MS, and Carbon content analysis results which showed a reduction of carbon content to 0.2 wt. %. This process reduces the carbon content and prevents the oxidation of the metallic particles. Finally, the pyrolyzed powder is pressed and melted using an arc melter, producing a clean and purified metallic alloy. The final result indicated that there was no carbon in the structure of the molten button and a thin layer of Sm_2O_3 slag on the outer layer (~5 vol. %) that can be easily ground, demonstrating the successful cleaning and purification of the metallic alloy.

Overall, the pyrolysis and arc-melting techniques used in this study show promise for reducing the carbon content of Sm-Co sludge and purifying the metallic alloy. Recycling this waste can recover valuable elements and raw materials and move towards a circular economy and sustainable production.



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P1-10

Development of a Process to Remove the Neodymium-rich Phase from Recycled NdFeB Magnet Powder through Leaching with Organic Acid

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4. Recycling Rare Earth Magnets, September 4, 2023

In recent years, rare earths have become a critical supply risk in the European Union (EU. Recycling of neodymium-iron-boron magnets can be a partial solution for the supply of rare earths in the EU.

Pyro-, hydrometallurgical and a the highly efficient hydrogen processing of magnetic scrap (HPMS) process can be used for this purpose. In this study, a combined process of HPMS and a leaching process is evaluated to recycle spent magnets. The goal was to develop a process to obtain a single crystal powder of Nd₂Fe₄B by separating the partially oxidised neodymium-rich phase from the hard-magnetic phase through leaching, as this phase is detrimental for the production of recycled magnets with high remanences. End-of-life wind power magnets were used and organic acid was used as leaching agent. The magnets were first thermally demagnetized, processed with hydrogen (HPMS), ball milled in an inert atmosphere, treated with acid and then rinsed and dried under vacuum. Good results were obtained at an acid concentration of 0.5 mol/l, a solid to liquid ratio of 1/10 and a leaching time of 30 min. The surface structure and chemistry of the Nd₂Fe₁₄B grain were examined using atomic resolution transmission electron microscopy. The results revealed that a predominantly oxygen-rich amorphous reaction layer, 25 nanometers thick, had formed. At the sub-atomic level, the interface between the original grain matrix and the reacted layer was sharp, indicating a step-wise reaction mechanism during the leaching process. The leaching process initially attacks the neodymium-rich phase, but also the hard-magnetic phase, which leads to changes in the magnetic properties of the powder. Different rinsing strategies have been evaluated to prevent the magnetic phase from oxidizing. The separation of the neodymium-rich phase can help to increase the efficiency of recycling and to open up new approaches for magnet production from recycling material.



P1-11

Chemical Recycling of Nd-Fe-B Permanent Magnets: A Sustainable Solution for the Nd2Fe14B Matrix Phase Recovery

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4. Recycling Rare Earth Magnets, September 4, 2023

The year-wise rise in demand of rare earth elements (REEs) for permanent magnets and their shortage have resulted in price rise. This has led to the importance of recycling magnets to achieve sustainability, which doesn't come at any price without encompassing economic and environmental aspects. The focus of this study is to develop a short loop recycling, by leaching REE rich secondary phase and extract Nd2Fe14B grains undamaged, for novel magnet making. For this, sintered Nd-Fe-B magnets were hydrogen decrepitated (HD) to achieve pulverization and high surface to volume ratio of exposed Nd-Fe-B-based material towards acid leaching. The HD powders were treated with 1M citric acid solution by two different procedures. First is leaching by aging for 5, 15, 30, 45, 60 and 120 minutes and second by ultrasonication assisted leaching for 5, 15 and 30 minutes. The leaching efficiency and selectivity of citric acid for REEs rich secondary phase was investigated by microstructural analysis (SEM-BSE), magnetic measurements (VSM), gravimetric study and compositional analysis by ICP-MS. The results incline to preferential leaching of REEs rich secondary phase (due to it's high reactivity in Nd-Fe-B system) by citric acid at leaching times up to 30 min without ultrasonication and 15 min with ultrasonication, after which most of the REEs phase is leached away and continuous leaching results in leaching of Nd2Fe14B grains as evident from ICP-MS, gravimetric and magnetic properties study. Ultrasonication enhances the leaching efficiency and REEs rich secondary phase is leached in 15 minutes. The selective leaching process by the use of mild organic acid as reported in this study can be efficiently applied for any Nd-Fe-B magnets EOL or fresh. Citric acid as a lechant makes leaching eco-friendlier as it can be easily neutralized after completion of reaction and the process can be upscaled for industrial magnet recycling.



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P1-12

Facing the Challenges for a Circular and Sustainable Rare Earth Permanent Magnet Recycling

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4. Recycling Rare Earth Magnets, September 4, 2023

In times of scarce raw materials and climate change, a closed-loop circular economy is more important than ever. Due to their outstanding properties, rare earth permanent magnets (RE PM) play a key role in the transition of the energy and mobility sector towards a green and CO2-neutral society. Several recycling procedures for RE PM were developed and improved during the last years [1]. A recycling on an industrial scale is in initial stages but needs several efforts concerning upscaling. Assistance will come from the European Union which plans to introduce a new ecological product regulation system and the critical raw materials act [2,3]. Products will be classified regarding their environmental footprint, recyclability, CO2 emission and should contain specific amounts of recycled material.

Parallel to the enhancement of recycling procedures a further development of primary production techniques to reduce the content of critical RE elements occurs. These technologies like grain boundary diffusion are nowadays very common. However, these improved magnets which differ from scrap magnets 10 - 15 years ago in terms of microstructure and composition, can be a challenge for direct recycling techniques like hydrogen-based methods in terms of reproducible properties.

In this work we recycled different Nd-Fe-B PMs with the so-called magnet-to-magnet approach under the usage of hydrogen decrepitation and analyzed in detail the influence of several recycling cycles on different material properties like degree of alignment, impurity content, microstructure or magnetic properties [4]. A deeper understanding of the material behavior through the recycling process should help to establish an industrial scale recycling and improve the circularity of the material. Through three recycling cycles an increase of impurity content and coarsening of average particle size of sintering powder were observed. These changes lead to an adjustment of optimum sintering conditions and degree of alignment of fully dense recycled magnets.





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DNDAY

Tailoring the Magnetic Properties of Multipole Anisotropic Magnets by using a Mixture of Commercial and Recycled Anisotropic NdFeB Powders

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4. Recycling Rare Earth Magnets, September 4, 2023

Anisotropic Nd-Fe-B powders were produced from scrap computer hard drives using the HD/ degassing process. The magnets were processed without additional preparation steps (the nickel coating was not removed or affected in any way). A flake-like anisotropic powder with parameters Hci= 434 kA/m; Br= 1.15T, (BH)max= 109 kJ/m³ was obtained. The <250 µm fraction powder was mixed with commercial anisotropic Aichi MagFine MF15P powder by gradually increasing the proportion of recyclate. In this study, multipolar anisotropic PA12 bonded magnets with 50% by volume of the hard magnetic material were produced. Through the injection moulding, into specially designed moulds, aligning anisotropic NdFeB magnetic particles, 2-, 4-, and 6-pole rotors were produced. In this way, ring magnets with 8 and 16 poles could also be obtained. Rotors and ring magnets made from commercial materials and from commercial materials mixed with recycled powder were compared. The homogeneity of the filler particle distribution, the degree of particle orientation and the magnetic induction at the circumference of the sample were analysed. Progressively increasing the proportion of recyclate up to 40% results in a decrease in coercivity and (BH)max, which also means a reduction in the price of the magnets. Above 50% recyclate content, the magnetic properties no longer change significantly and remain at a similar level.



Dynamic Stocks and Flows Analysis of NdFeB Permanent Magnets in the UK Electric Vehicles and Wind Turbines

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4. Recycling Rare Earth Magnets, September 4, 2023

The UK has set ambitious Net Zero targets for the electrification of transport and renewable energy by 2050, which require significant quantities of neodymium-iron-boron permanent magnets (NdFeB PM) for the manufacture of electric vehicles (EVs) and wind turbines. The supply risk to the electrification transition is considerable, as the UK is close to 100% import reliant on NdFeB PM and rare earth elements (REE), primarily from China. A strategic response to risk minimisation is possible by developing a circular economy (CE) ecosystem of NdFeB PM. The quantification of the UK NdFeB PM stocks and flows is pivotal in decision making to enable potential reverse supply loops to develop.

The Met4Tech Centre and the CE-Hub collaborate to address two critical questions:

1) How large are the in-use stocks of NdFeB PM, embedded in EVs and wind turbines in the UK over time?

2) What is the revalorisation potential of secondary NdFeB PM and REE as supply from the UK End-of-Life EVs and wind turbines up to 2050?

A dynamic stock-driven model has been developed to estimate annual inflows and outflows of NdFeB PM and containing REE, based on public datasets, peer-reviewed papers and combined with stakeholder engagement.

Our results reveal that by 2050, EVs will have the highest in-use stocks of NdFeB PM and REE. The inflows of NdFeB PM and REE will continue to increase the stock until 2030. End-of-life NdFeB PM and REE flows will increase sharply from 2030 onwards, while potential retirement of aged wind turbines and EVs, are likely to contribute to secondary supply. Our analysis suggests that stakeholder collaboration and investment are required to develop a circular economy at scale, specifically the establishment of collection systems, reverse logistics, extended lifespan operations, and innovative dismantling and recycling technologies for NdFeB PM in the UK.



Intelligent and Sustainable Processing of Innovative Rare-Earth Magnets

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4. Recycling Rare Earth Magnets, September 4, 2023

The EU aims to become climate neutral by 2050. A successful transition fundamentally depends on Europe's ability to develop and deploy clean energy and mobility solutions in an economically and environmentally sustainable way. The raw materials need to facilitate this energy transition is massive and in addition, the industrial and home appliances will need to run under the highest energy efficiency standards. The most energy-efficient electric motors and generators contain rare earth permanent magnets. Whilst EU companies are world leaders in the manufacturing of electric motors, they are fully import-dependent along the entire value chain of rare earth magnet materials (1).

The permanent magnets (PMs) depend upon Rare Earth Elements (REEs). Magnets are critical to Europe's future, from wind turbines and hydroelectric generators to electromotors in next-generation hybrid and electric vehicles. The essential Critical Raw Materials (CRM) used in NdFeB PMs are REEs and non-REEs niobium and gallium. Although CRMs from China have been the primary source for Europe, supplies are uncertain, and the Chinese production chain is generally unsustainable. At the same time, the demand for REEs to make new PMs is projected to double in 15 years. Given these data, our work focuses on collecting end-of-life (EOL) magnets and sustainable recycling and reproducing PM from sources focusing on the most common and readily available source of economically recyclable electric motors: home appliances. We are developing new dismantling and recovery procedures for PMs on highly available scrap and reproduction lines. We use an already well-established method of hydrogen in HPMS (Hydrogen Processing of Magnetic Scrap), followed by milling, degassing and coating sensitive powders. The first pilot experiments in producing sintered and bonded magnets out of recycled magnets confirm the no-waste circle economy processing and future independence from the unstable sources of REE.



P1-16

Optimisation of Grain Boundary Phase Separation in Hydrogen Processed NdFeB

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4. Recycling Rare Earth Magnets, September 4, 2023

The University of Birmingham have developed a process to recycle NdFeB magnets from end-oflife products called "Hydrogen Processing of Magnet Scrap" (HPMS) [1]. This method enables bulk magnets to be broken down into a demagnetised alloy powder through the hydrogen decrepitation (HD) process. The hydrogenated powder can then be sieved to remove any impurities, e.g. coating material, and milled to single domain sized particles of \leq 5 µm by jet milling.

NdFeB magnets consist of a matrix $Nd_2Fe_{14}B$ phase and a Nd-rich phase, the latter of which is found at the grain boundaries and helps with the sintering process by smoothing the surface of the $Nd_2Fe_{14}B$ matrix phase [2]. This Nd-rich phase is often oxidised into Nd-oxide during primary processing but particularly during the milling stage. This is where a large proportion of the oxygen is in the final magnet. During the HPMS process this oxide breaks away from the $Nd_2Fe_{14}B$ phase during processing. If this oxide is left in the powder and then re-sintered, it can result in porosity and a reduction in the coercivity of the resultant magnets.

In the current work a cyclone and classifier has been used during jet milling to remove Nd-oxide from hydrogenated powder. HPMS powder with 500 μ m particle size was pulverized below 10 μ m using a Netzsch Jet mill under flowing N₂. It was then separated by a cyclone and a classifier. The first fraction contains particles of ~5 μ m size (coarse powder) while the second fraction is comprised of submicron particles (fine powder). The composition of the powder was analysed using O/N Analyser and ICP-OES and the effect of classifier wheel speed on particle size and Ndoxide removal was investigated. This work follows on from previous studies performed in a water cyclone by Herraiz et al [3, 4].

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Investigation of Oxidation Behaviour and Passivation Rates for Recycled NdFeB Powders

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4. Recycling Rare Earth Magnets, September 4, 2023

NdFeB is the most widely employed rare-earth magnet, utilised in technological applications, such as hard disk drives (HDDs) and permanent magnet motors, due to its exemplary magnetic properties and versatile application across industries.

Due to instability within the rare earth element supply chain, and demand expected to continue increasing in the coming years, there is a growing focus on establishing an alternative supply route of Neodymium (1). Recycling of NdFeB utilises the large quantities of spent NdFeB magnets available, reducing the dependency on rare earth element mining and its limited supply. A recycling method, the Hydrogen Processing of Magnetic Scrap (HPMS), devised at the University of Birmingham, uses hydrogen decrepitation of end-of-life (EOL) magnets to produce a demagnetised hydrogenated powder (2). The extracted powder can then be reprocessed to form new magnetic material. The oxidation behaviour of the NdFeB powder during this process and its oxidation stability have implications on the viability of the recycling process in subsequent steps. The pyrophoric powder is very sensitive to oxidation and requires passivation to safely and effectively conduct the HPMS process.

This work investigates the oxidation behaviour and passivation rates for NdFeB powders produced from EOL NdFeB magnets. By determining oxidation rates, an optimal passivation rate can be established. The powder material, extracted using hydrogen decrepitation, is studied under varying oxidation conditions, such as partial pressure and temperature, using electron microscopy and pressure-controlled gravimetry.

It has been found that varying oxygen partial pressures have an impact on the level of oxidation and the rate of passivation of the powder material.

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Extraction of NdFeB Magnets From End-of-Life Electric Vehicle Scrap, Conversion into Master Alloy and Sintered Magnets

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4. Recycling Rare Earth Magnets, September 4, 2023

Neodymium-iron-boron (NdFeB) based magnets are used in domestic electrical appliances, electric and hybrid automobiles, wind turbines, consumer electronics, and many other small electronic devices. The increasing popularity of hybrid and electric cars, wind turbines and e-bikes is expected to increase the demand for rare earth magnets exponentially. Recycling rare earth elements (REE) from end-of-life products or components can not only provide a sustainable supply for the future but can also minimise the environmental impacts associated with REE mining and processing [1]. Previous work has shown that hydrogen can be used to liberate NdFeB magnets from hard disk drive scrap [2]. This hydrogenated NdFeB can be directly reprocessed into new sintered magnets with magnetic properties approaching the performance of the original magnets [3].

In this work, however, an indirect recycling method has been proposed by melting the hydrogenated NdFeB powder. This route not only gives more control over the final composition but also the oxygen and possibly carbon can be separated into the slag phase [4]. In this work, the NdFeB magnets from automotive motor scrap were demagnetised by heating above their Curie Temperature, decoated to remove the epoxy coating and hydrogen decrepitated (HD). The resulting powder was then pressed into green compacts, partially degassed and melted in a vacuum induction furnace to form book mould cast alloy and then strip cast. The alloy composition was determined using ICP-OES and the microstructure was analysed using a scanning electron microscope (SEM). The resulting strip-cast alloy was later subjected to the HD process and subsequently jet-milled. The milled NdFeB was then sealed in isostatic bags, aligned, iso-statically pressed into green compacts, and vacuum sintered at 1080 °C for 1 hour. These recycled sintered magnets were then compared for magnetic properties, density and microstructure to the starting magnet.

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From Scrap to Magnet: On the Use of HDDR Recycled Feedstocks in the Additive Manufacturing of Composite Magnets

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4. Recycling Rare Earth Magnets, September 4, 2023

The HDDR (hydrogenation-disproportionation-desorption-recombination) process has been previously applied for recycling rare earth permanent magnets from various sources. The recycled magnets are usually prepared by mixing the HDDR powder with a polymeric binder (THERMOSET OR THERMOPLASTIC) and then moulding the mixture via compaction OR INJECTION, respectively. Another strategy to produce composite magnets is processing the HDDR powder via Additive Manufacturing (AM), although it has not been extensively explored in the context of recycling magnets. When compared to traditional processes, AM offers several advantages, such as the ability to produce complex geometries, the lesser waste of raw materials since net shape parts are obtained, and the possibility of developing functionally graded materials in situ. In this study, the HDDR process was applied for two types of Nd-Fe-B scrap magnets disassembled from wind turbines of different suppliers to produce HDDR isotropic powders with coercivities ranging from 600 to 800 kA/m and remanence of 0.7 T (bulk value), analogous to commercial powders produced via the same route using pristine alloys. Feedstocks were prepared by mixing the HDDR powder with different volume fractions of polyamide 12, and recycled composite magnets were then obtained using the powder bed fusion technique (PBF). Magnets with various geometries and less than 10% porosity were obtained, and the coercivity and remanence of the HDDR powder were preserved. In addition to a HDDR powder-based feedstock, another feedstock using a HDDR powder which was subjected to a grain boundary diffusion process (GBDP) was prepared and processed via PBF. In this case, the HDDR powder was mixed with a Dy-Cu alloy powder and the mixture was then heat treated resulting in a powder with coercivities up to 1100 kA/m, showing the feasibility of further improving the magnetic properties upon adopting the GBDP strategy.

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Investigation into the Influence of Zn Coatings on Recycling of Sintered NdFeB-type Magnets

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4. Recycling Rare Earth Magnets, September 4, 2023

Rare-earth (RE) magnets based on Neodymium-Iron-Boron (NdFeB) are used in a range of hightech industries, such as automotive, e-mobility and consumer goods. As demand for these materials has increased, their supply has come under considerable pressure and RE elements are now top of the EU critical materials list for supply risk [1]. Recycling end-of-life (EoL) products containing NdFeB is one solution to ensure a strategic long-term supply [2].

Previous work at the University of Birmingham has shown that HPMS (Hydrogen Processing of Magnetic Scrap) can be used to extract hydrogenated NdFeB powder from magnets contained within hard disk drives [3] and automotive scrap [4]. During HPMS, NdFeB magnets are broken down into a friable, demagnetised, hydrogenated powder using the hydrogen decrepitation (HD) process. The extracted hydrogenated material can be purified to remove contamination e.g., coating material using a combination of milling and sieving. The purified hydrogenated NdFeB powder can then be reprocessed in different ways, namely re-sintered to produce fully dense sintered magnets.

Another source of EoL NdFeB is the loudspeaker market which accounts for approximately 10% of the NdFeB market, making them an attractive feedstock for recycling. Loudspeakers containing NdFeB are widely found in flat screen TV's and high-end speaker systems and the magnets are typically coated in Zn.

Previous work has shown that small amounts of Zn additions in NdFeB can improve remanence and coercivity, however, above a threshold it has a detrimental effect. This work looks at whether the Zn coating needs to be removed prior to hydrogen processing and if Zn will be detrimental to the microstructure and subsequent magnetic properties of the recycled magnets.

The microstructure, composition and magnetic properties of the starting material and recycled sintered magnets were assessed using techniques including scanning electron microscopy, inductively coupled plasma optical emission spectrometry and permeameter measurements.



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Microstructure and Phase Transitions During the High Temperature Hydrogenation Disproportionation of Various NdFeB Alloys

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

Conventional Hydrogenation Disproportionation (c-HD) of NdFeB alloys has been widely studied especially for the development of highly coercive powders obtained with the Hydrogenation Disproportionation Desorption Recombination (HDDR) process. Most studies focus on attaining a nanometric microstructure while keeping the initial anisotropic material texture, suitable for the recombination process step. Disproportionation is hence performed at moderate hydrogen pressure and temperature [1]. However, an extensive phase diagram for high temperature disproportionation of various alloys is still unknown.

This work investigates the evolution of the microstructure and phases equilibria for hightemperature c-HD treatments (above 800°C) using NdFeB alloys with various Rare Earth Elements (REE) contents. Several process conditions (disproportionation temperature, time and pressure) are correlated to the obtained microstructures. The chemical compositions and crystalline structures of the disproportionated phases are analyzed by WDS, SEM, XRD and TEM. This study covers the phase transition temperatures, phase sizes and kinetics aspects for various compositions of strip cast alloys typically used for NdFeB magnets fabrication. The main finding of this work is the formation of an intermediate thermodynamically stable boride RE-Fe-B as the disproportionation temperature increases. This happens before the expected recombination into $Nd_2Fe_{14}B$, similarly to what is documented for Co-rich alloys [2], [3]. The stability domain of this phase and its chemical composition is discussed regarding the initial composition of the alloy.

Finally, a phase diagram of the reactions occurring at 0.8 bar H_2 is plotted depending on disproportionation temperature for several alloy compositions. This study provides new insights of solid gas reactions between NdFeB alloys and hydrogen.





- Figure 1: Evolution of the microstructure of a strip cast alloy subjected to c-HD under 800 mbar H₂ for three disproportionation temperature with a holding plateau of 3h (BSE images):
 - (a) 800° C : Fully disproportionated lamellar-type microstructure (initial Nd₂Fe₁₄B dendrite)coexisting with hydrogenated initial Nd-rich phase
- (b) $900^{\circ}C$: Fully disproportionated spherical-type microstructure. White contrast : Rare Earths (RE) hydrides, dark contrast : α -Fe + Fe₂B
- (c) $950^{\circ}C$: Partly disproportionated spherical-type microstructure showing the apparition of an intermediate thermodynamically stable boride. Whire contrast : RE hydrides, dark contrast : α -Fe + Fe₂B, grey contrast α -Fe + Fe₂B

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P1-22

Coercivity Enhancement of Sintered NdFeB Magnets by Doping R₈₅Al₁₅ (R=Ce and Pr) Alloys Followed with Grain Boundary Diffusion

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

How to enhance coercivity of NdFeB sintered magnets with the reduced Tb or Dy usage has long been an important issue. The dual-alloy (DA) sintering or grain boundary diffusion (GBD) is individually effective to meet this challenge. For traditional manufacturing of the NdFeB magnet, one magnetic level is made by an alloy with a specific composition. In this work, both DA and GBD are adopted to reach various magnetic levels by using one mother alloy in addition to acquire high-performance NdFeB magnets, which iHc+(BH)max>70 as well as coercivity of larger than 25 kOe. The low-melting Pr₈₅Al₁₅ (PrAI) and low-cost Ce₈₅Al₁₅ (CeAI) eutectic alloys are adopted and doped into commercial NdFeB powders to prepare DA-sintered magnets. Subsequently, the above 6-mm-thick magnets are GBD-treated with low-melting Tb75-xPrxCu25 (TbPrCu) powders. The magnetic properties and microstructure analysis for the DA as-sintered magnets as well as GBD-treated magnets are studied. For the magnet DA sintering with 2-4 % PrAI and 2-6% CeAI, the coercivity is increased from 14.9 to 16.4-18.0 kOe and 16.3-18.2 kOe, respectively. Ce or Pr and AI at grain boundary strengthens magnetic decoupling effect and thus contributes to the coercivity enhancement for DA sintered magnets. For the above magnets after GBD with TbPrCu alloy, the coercivity is further enhanced to 22.7-27.1 kOe and thermal stability is also improved. The increased Curie temperature of 2:14:1 phase indicates the entrance of Tb into 2:14:1 phase and helps to improve thermal stability. Most importantly, various magnetic levels of high-performance NdFeB magnets, including 48-52M, 42-45H, 35-50SH and 40-45UH, can be easily attained by modifying the amount of PrAI and CeAI for DA sintered magnets and/or GBD with TbPrCu alloys. This study provides a simple and cost-effective method to make various high magnetic levels NdFeB magnets using only one NdFeB mother alloy powders.



10NDA

P1-23

Design of Core-shell Structure and Magnetic Property Enhancement Mechanism of Dual-main-phase High-abundance Ce Based Sintered Magnets

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023,

Nd-Fe-B permanent magnets are widely used in various fields such as new energy vehicles, wind power generation, and consumer electronics. For the development of high-performance and lowcost Nd-Fe-B magnets, Ce is commonly used instead of Pr/Nd. However, the intrinsic properties of Ce-Fe-B are low, and the presence of a large number of impurities and poor microstructure in magnets with high Ce-content, which leads to a rapid decline in the magnetic properties of (Nd, Ce)-Fe-B magnets as the Ce content increases [1]. In order to obtain high-performance (Nd, Ce)-Fe-B magnets is crucial to regulate the distribution of Ce elements in the magnet, to suppress the formation of CeFe, impurities phases, to optimize the grain boundary distribution and to enhance the magnetic segregation effect of the main phase grains [2]. This work optimizes the dual-mainphase process, sintering a mixture of Pr (Pr₄₅Fe_{bal}B) and Ce (PrNd_{0.5}, Ce_{0.5})_{29.5}Fe_{bal}B powders. By using unconventional Pr powders the aim is that Pr forms the liquid phase during the heat treatment, which wraps around Ce₂Fe₁₄B, enriching Ce₂Fe₁₄B inside the matrix phase grains, constructing coreshell structure, and forming Pr-shell structure with the high anisotropic field. Pr powders optimizes the phase composition and elemental distribution of the magnet grain boundaries, which not only suppresses the production of impurity, but also optimizes the grain boundary structure facilitating the subsequent diffusion of heavy-rare-earths. The diffusion creates the highly anisotropic field of heavy-rare-earth shells, achieving a significant improvement in the performance of magnets containing large amounts of Ce. This work reveals the elemental distribution and microstructure of grain boundary phases in high Ce-content magnets with a core-shell structure of matrix phase grains, clarifying the mechanism of core-shell structure formation and optimization of grain boundary phases.



Figure 1: Demagnetization curves for substrate and diffusion samples at room temperature.



Figure 2: BSE and corresponding elemental distribution of Ce50 and Ce25 under EPMA.

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Tuning the Hard Magnetic Properties of Melt-spun Misch Metal-Fe-Hf-B Ribbons by Heat Treatment

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

In recent years, there has been a significant shift towards environmental awareness in the fields of energy and transport. This has made rare earth permanent magnets (REPM) one of the most important materials used in modern technology, causing a direct influence on the growing global REPM market [1,2]. Currently, strong research activity is thus focused on finding viable alternatives to permanent magnets based on rare earth (RE) elements [3], which may allow their replacement at least in those applications where high performance is not strictly required. The substitution for RE the abundant Misch Metal (MM) is a practical way to balance the utilization of RE resources and develop Misch Metal (MM)-Fe-B permanent magnets with a high performance/cost ratio.

Here, we have studied the influence of heat treatment on the magnetic properties and structure of the MM-Fe-Hf-B melt-spun ribbons. It was determined that the optimal treatment is 650 °C, a time of 20 minutes. The heat treatment led to the removal of some unwanted effects such as the existence of uneven distributions of atomic species generated by the particularities of solidification on the cooling disc and the inhomogeneous distribution of crystalline grains and to the formation of a homogeneous and refined crystalline structure, in the nanometric range, a structure associated with the manifestation of superior magnetic properties. More precisely, in the case of ribbons with 2% Hf addition, the heat treatment led to an increase in the coercivity to 9.2 kOe from 5.2 kOe, the remanence to 87 emu/g from 30 emu/g and the energy product to 9.8 MGOe from 4.1 MGOe. This paper may shed light on the further development of MM-based magnets and offer a feasible way for using RE resources efficiently.

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Novel Processes for the Manufacturing of Fine-grained Nd-Fe-B Powders with Steep Particle Size Distribution

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

The manufacturing process of rare earth (RE) magnets involves a series of steps including grinding, pressing, and sintering of the material powder. Among these, grinding is a crucial process that determines the particle size distribution and morphology of the RE alloy powder, which in turn influences the quality and performance properties of the magnets produced from the powder. It is desirable to achieve a narrow particle size distribution with minimal fines content, which is most prone to oxidation and a well-defined upper particle size limit.

To address these requirements, a new grinding process for RE alloy powders, specifically Nd-Fe-B powders, has been developed that offers several advantages over conventional fluidized bed jet mills. The new process involves a high-performance classifier that is capable of removing undesirable fine-grained particles (< 1 μ m) and achieving a d90/d10 value that is only about half of that produced using conventional methods.

The use of these powders with a narrow particle size distribution in the production of sintered magnets leads to a significant improvement of the magnetic properties such as higher coercivity and knee-field strength. The new grinding process offers a promising approach for the production of high-quality RE magnets with improved properties.



Attaining Excellent Coercivity and Thermal Stability in Nd-Ce-Fe-B Sintered Magnets

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

The development of high-abundance Ce-based Nd-Fe-B magnets has attracted much attention for the past decade because of their low cost and reduced dependence on critical rare earth (RE) elements of Pr/Nd/Dy/Tb. However, the anisotropic field HA of Ce₂Fe₁₄B is much lower than that of Nd₂Fe₁₄B. In addition, the enrichment of RE in the REFe₂ phase and RE-O phase leads to strong exchange coupling between adjacent grains, which makes the coercivity of Nd-Ce-Fe-B sintered magnets generally lower than that of commercial magnets (1.2 T), limiting their large-scale applications. To overcome these disadvantages, in this work, we employ Dy-Cu alloy to achieve grain boundary modification and magnetic hardening, expecting to obtain a balance between cost and performance. Through composition optimization, coercivity up to 1.35 T was obtained in an initial sintered Nd-Ce-Fe-B magnet with Ce replacing 30% of Nd, which is attribute to the formation of a thin and continuous grain boundary phase. After adding 2.4 wt.% Dy-Cu, the coercivity was further increased to 1.95 T, and the coercivity temperature coefficient β (20-150°C) increased from -0.575 %/°C to -0.529 %/°C. Microstructural analysis shows that Dy mainly replaces the RE in the surface layer of the grains to form a core-shell structure, with Dy-rich in the shell. The enhancement of HA effectively inhibits the nucleation of reversed domains, thereby attenuating the magnetic dilution effect. In addition, the grain boundary thickness is further increased, which weakens the exchange coupling of adjacent grains. The relationship between microstructure and coercivity was verified by magnetic domain structure observation and micromagnetic simulation. This work provides a guidance for the coercivity enhancement of Nd-Ce-Fe-B magnets, which is beneficial for their large-scale applications.



Figure 1: (a) Room temperature demagnetization curve of the original and Dy-Cu addition magnet.
MONDAY



Figure 2: BSE SEM and corresponding EPMA images of the (a) original and (b) DyCu addition magnet.

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P1-27

MONDAY

Coercivity Enhancement in (PrNd, Ce)-Fe-B Sintered Magnets: Effect of Grain Boundary Reconstruction by Low Melting Point Alloys

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

Promoting the formation of a continuous grain boundary layer and enhancing the magneto-crystalline anisotropy field on the surface of the main phase grains is an effective approach to optimize the magnetic properties of Ce-based magnets. In this work,near stoichiometric ratio (PrNd,Ce)-Fe-Bmagnets were designed. Using the grain boundary reorganization (GBR) method, alloy additives including Nd₉₅Cu₅, Nd_{47'5}Dy_{47'5}Cu₅, and Dy₉₅Cu₅ (at. %) were incorporated into the system. The addition of 3% Nd_{47'5}Dy_{47'5}Cu₅ resulted in a significant increase in coercivity from 6.05 kOe to 14.93 kOe. The remanence and coercivity of the Nd_{47'5}Dy_{47'5}Cu₅ magnets were higher compared to those of the Dy₉₅Cu₅ alloy. It is found the incorporation of the low melting point NdDyCu additive facilitated the liquid phase sintering process, leading to improved densification and the formation of continuous grain boundary layers that effectively isolated the main phase grains. The Nd-Dy-rich hard magnetic shell layer was constructed, resulting in enhanced local magnetic crystal anisotropy and weakened ferromagnetic coupling. That effectively protects the nucleation and expansion of reverse domains and thus achieves high coercivity. The findings demonstrate that NdDyCu alloy is a promising additive for enhancing the coercivity of Ce-based permanent magnets.



Figure 1: Room temperature demagnetization curves of the original magnet, NdCu, NdDyCu, and DyCu magnets.



Figure 2: The recoil loop of original magnet and NdDyCu magnet, reversible (c) and (d) irreversible portions during the magnetization reversal process of original magnet, NdDyCu magnet.

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MONDAY

P1-28

Effect of Co Content of Nd-Fe-B Sintered Magnet on Grain Boundary Diffusion Process of TbH, Solution

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

The improvement of thermal stability and the formation of a continuous and uniform non-magnetic Nd-rich grain boundary phases (GBPs) in Nd-Fe-B sintered magnets are the most important microstructural design factor to improve the coercivity without reducing the remanence [1-4]. Co addition in the magnet is effective for improving the thermal stability of the magnets, due to its high Curie temperature. The formation of a continuous and uniform Nd-rich GBP and non-magnetic Nd₃(FeCo) phase are important Co addition effects [1-3]. However, the coercivity decreases when the magnets contains over the optimum Co content (~1.0 wt.%) [3]. Tb-rich core-shell microstructure development via grain boundary diffusion process (GBDP) could enhance the anisotropy field of the near grain boundary region. Hence, the coercivity is efficiently improved and Tb usage is minimized [4]. However, the grain boundary diffusion behavior of Tb in magnets with different Co content has not yet been clearly identified. We investigated the effect of Co content on the microstructural and magnetic properties of Nd-Fe-B sintered magnet by GBDP of TbH₃ solution. The (Nd,Pr)₃₁₀-Febal.-B_{0'96}-Co_{1'0}-M_{0'8} (wt.%) and (Nd,Pr)31.0-Febal.-B_{0'96}-Co_{5'0}-M_{0'8} powders were mixed according to the ratio and sintered at 1050°C. The sintered body was coated by TbH₃-ethanol solution. The coercivity of the magnets without GBDP decreased (17.10-> 15.96 kOe) with increasing the Co content (1.0->5.0 wt.%). However, the coercivity increments in the 5.0 wt.% Co-containing magnet with GBDP (74.12 %, 27.32 kOe) was larger than that of 1.0 wt.% Co-containing magnet with GBDP (60.82 %, 27.5 kOe) compared with each corresponding magnet without GBDP. As the content of Co in magnets with GBDP was increased (1.0->5.0 wt.%), there was a coincident gradual increase in temperature coefficients (α , β) of magnets. The mechanism of the magnetic and microstructural evolution due to the change of Co content and will be discussed.

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MONDAY

P1-29

Investigation of the Diffusion Behavior for Heavy Rare Earths for Nd-Fe-B Sintered Magnets Produced by the 2-Powder Method

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

In the context of climate change and green technologies rare earth (RE)-based permanent magnets like Nd-Fe-B magnets are needed. If they are used in electrical motors of electrical vehicles, expensive and critical heavy rare earths (HREs) are required, because they increase the temperature stability.

By using the conventional production process for Nd-Fe-B-based sintered magnets in an industrially relevant scale (25 kg per strip cast batch), two Nd-Fe-B-based alloys were produced for investigation and improvement of the 2-powder method (2PM) [1,2]. Therefore, a coarse HRE-free main phase powder (D50 = 5.3 μ m) and a finer HRE containing anisotropy powder (D50 = 2.6 μ m) were blended having a final Dy content of 0, 1, 2, and 3 wt.%. Magnetic properties at room temperature and elevated temperatures up to 180 °C were measured and temperature coefficients were calculated. While no significant decrease in remanence was observed, a constant coercivity gain (120 kA/m per added wt.% Dy) was obtained. Microstructural investigations show the development of a core-shell structure, meaning that HREs were localized only at the outer regions of the grains. Compared to the industrial-used grain boundary diffusion process (GBDP), the HRE-enriched shells are still too thick. Therefore, the diffusion mechanism of the 2PM is investigated. For this, samples with a final Dy content of 3 wt.% were analyzed at different sintering stages (5, 10, 30, 60, and 120 minutes at 1090 °C). The microstructure was investigated, observing that the coreshell structure was developed, after 5 minutes already and the diffusion coefficient for the heavy rare-earth element dysprosium of D = (4.46 ±0.49) x 10-12 [cm2/s] was calculated.

Finally, the 2PM was ranked with the conventional production process and the GBDP demonstrating the protentional of the 2PM for the production of Nd-Fe-B-based permanent magnets with less critical HREs.





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P1-30

2-Powder Method for Improving the Magnetic Properties of Ce-Containing Nd-Fe-B Magnets

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

The demand for neodymium-iron-boron (Nd-Fe-B) magnets surges in recent years, driven by the expansion of wind turbines, hybrid electric vehicles, and consumer electronics. However, China's monopoly on the market of rare earth (RE) elements creates supply chain fragility and price volatility. Accordingly, the research on cerium-containing Nd-Fe-B based ((Ce,Nd)-Fe-B) magnets have received increasing interest to reduce the usage of critical Nd and maintain a balance between market demand and natural abundance [1]. Nevertheless, substituting Nd by Ce in the Nd₂Fe₁₄B phase deteriorates magnetic performance due to the inferior intrinsic properties of Ce₂Fe₁₄B.

To mitigate the deterioration, the 2-powder method (2PM), originally designed for heavy rare earth (HRE) addition in Nd-Fe-B magnets by Löwe et al. [2,3,4], is proposed for (Ce,Nd)-Fe-B magnets. In 2PM, a core-shell structure with a HRE-enriched shell is formed in Nd₂Fe₁₄B grains after the standard sintering procedure by mixing a fine HRE-containing anisotropy powder (AP) with a coarse Nd-Fe-B main powder (MP). The coercivity is effectively improved by increasing the anisotropy field merely in the surface region of Nd₂Fe₁₄B grains. Adapting the 2PM in (Ce,Nd)-Fe-B magnets utilizes a Nd-Fe-B AP with the (Ce,Nd)-Fe-B MP to form a Nd-enriched shell in (Ce,Nd)₂Fe₁₄B grains, improving the coercivity while maintaining less content of Nd.

In the current stage, the 2PM is employed on (Ce,Nd)-Fe-B magnets with Dy-containing AP for a clearer insight into the core-shell structure to examine the effectiveness of the process. An addition of 3 wt.% of Dy increases the coercivity by 610 kA/m without deteriorating remanence and energy product. The improvement can be interpreted by the microstructure, where Dy tends to substitute Ce instead of Nd in the (Ce,Nd)₂Fe₁₄B phase. In the future, Dy-containing AP will be replaced by Nd one to further decrease the amount of critical RE used in (Ce,Nd)-Fe-B magnets.





Figure 1: The demagnetization curve of (Ce,Nd)-Fe-B magnets containing no Dy and 3 wt. % of Dy manufactured by the 2PM. Both magnets exhibit the remanence of 1.25T. The coercivity increased from 750 kA/m to 1360 kA/m after 3 wt. % of Dy is included.



Figure 2: The microstructure and EDX analysis of (Ce,Nd)-Fe-B magnets containing no Dy and 3 wt. % of Dy manufactured by the 2PM.

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Coercivity Enhancement through Grain Boundary Diffusion in Sintered Nd-Fe-B Magnet using DyF₃

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

Since the invention of Nd-Fe-B magnets by Sagawa in 1983, sintered Nd-Fe-B magnet has found various application owing to the high energy product [1]. However, retaining high coercivity at elevated temperatures has always remained a challenge and partial substitution of Dy/Tb at Nd site in Nd₂Fe₁₄B is one of the approaches to enhance the coercivity of sintered magnet. However, high cost of Dy/Tb elements and the scarcity of natural resources has always been a limitation. Grain boundary diffusion process via dip coating is one of the approaches to enhance the coercivity by forming core-shell like structure of (Nd, Dy)₂Fe₁₄B with minimal use of Dy/Tb [2-3]. In the present study, the sintered Nd-Fe-B magnet (6 mm × 4 mm × 2.5 mm) was dip-coated with DyF3-ethanol slurry at a weight proposition of 60:40. After drying, the magnet was vacuum sealed in a quartz tube and subjected to thermal treatment upto 900°C for 2 h. An uncoated sample was also heat treated under similar conditions as reference sample. The Dy-diffused sample showed enhancement in the coercivity (fig. 1(a)) (Hc) of 1.8 T which is 16 % higher than the as-received sample and 37% higher than annealed uncoated sample. Back scattered scanning electron micrograph [fig. 1 (b&c)] and 3DAP results [fig1. (d & e)] clearly show the thickening of grain boundary and formation of shell (fig. 1) In this paper we report a detailed investigation on the evolution of continuous (Nd, Dy)₂Fe₁₄Bshell enveloping the Nd-Fe-B grains and the thickening of Nd-rich intergranular phase. The precise quantification of the chemistry and segregation of the elements in the various phases (shell, grain boundary, secondary, matrix and triple junction phases) will be investigated using 3-dimensional atom probe tomography to explain the observed enhancement in coercivity.



Figure 1: (a) Demagnetization curves of as-received, Dy-diffused and un-coated annealed sintered magnet showing enhanced coercivity. SEM-BSE (b) & (c) and 3DAP (d) & (e) data shows the thickening of the grain boundary and formation of the shell

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On the Changes in Microstructure and Magnetic Properties of Rare Earth-based Permanent Magnets processed via Grain Boundary Diffusion

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7. RE-Fe-B Magnet Processing and Properties, September 4, 2023

Rare earth-based permanent magnets, known for their exceptional magnetic properties, play a crucial role in various industrial sectors, including renewable energy, electronics, and transportation. However, the growing demand for these magnets, particularly Nd-Fe-B magnets, has resulted in elevated prices and scarcity of rare earth (RE) elements. Hence, recycling end-of-life magnets has emerged as a promising solution to the scarcity problem [1]. The recycling process involves the Hydrogen Decrepitation (HD) of scrap magnets, with the HD product subsequently reintroduced into the sintering route of Nd-Fe-B magnets, a magnet-to-magnet recycling approach. Nevertheless, recycling may lead to an undesirable increase in the oxygen content, affecting the magnetic properties, particularly reducing the intrinsic coercivity (Hcj) [2]. The Grain Boundary Diffusion Process (GBDP) of Heavy Rare Earth Elements (HREE) has been proposed as a potential method to address the issue, since it allows for the enhancement of Hcj. The GBDP treatment consists of two steps: the initial one at higher temperature where the diffusion of the HREE is stimulated, while the second step involves post-diffusion annealing [3]. In this study, we focus on investigating the changes in magnetic properties and microstructure of RE-recycled magnets resulting from different temperatures of post-diffusion annealing within the range of 450 to 550 °C. The GBDP method using a Dy-Cu alloy was employed for grain boundary engineering and enhancing the coercivity. Preliminary results show significant improvements in magnetic properties after GBDP, with a maximum increase in coercivity of approximately 50% compared to the original magnet. The diffusion behaviour and microstructural evolution are characterized by employing advanced analytical techniques, such as scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). Additionally, magnetic properties, including intrinsic coercivity (Hcj), remanence (Jr), and squareness factor (SF), were evaluated using a hysteresisgraph.

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POSTER PRESENTATIONS - P2

POSTERS P2-1 - P2-28

SESSION 5: UNCONVENTIONAL PROCESSING

SESSION 6: NANOCRYSTALLINE & THIN FILM MAGNETS

SESSION 8. RARE EARTH-FREE MAGNETS

TUESDAY 5 SEPTEMBER THE GREAT HALL

Investigation of the Microstructural Features Induced by the Reaction of NdFeB Powders with the Degradation Products during the Debinding Step in the Powder Injection Molding Process

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5. Unconventional Processing, September 5, 2023

Rare earth (RE) permanent magnets are strategic and essential components for many applications such as electric cars, wind turbines, robotics, aeronautics or electronics and play a crucial role in the electrification of our society, necessary to achieve carbon neutrality. The growing demand in NdFeB magnets raises the question of the availability of critical raw materials, namely the RE elements [1]

The advanced manufacturing processes reduce the amount of material required to produce a magnet and allows for complex shapes with an optimized magnetic architecture. They could offer substantial benefits by combining better system performances and material savings. Powder Injection Molding (PIM), combining the advantages of plastic injection molding and powder metallurgy, brings a large freedom of building design, without wasting material during machining. For the particular case of the NdFeB magnets, this process could be advantageous as it offers the possibility to align the magnetic particles during the injection molding step and to obtain high density anisotropic magnets. However, the resulting parts must present features of the particular microstructure of NdFeB sintered magnets and low oxygen and carbon contaminations to avoid the weakening of the magnetic performances. Several specific steps of the PIM process are crucial to avoid the organic contamination.

This work investigates the evolution of the microstructure and the magnetic properties of PIM produced parts with various carbon contamination levels. High magnetic performances, equivalent to the ones of conventionally sintered counterparts, are obtained, showing low level of carbon contamination [2]. Carbon contamination effect on the microstructure, given by structural and microstructural analysis, is correlated to the measured magnetic properties.

This study provides new insights of the reactions occurring during the debinding step of the PIM process and highlights the importance of the different process conditions of the organic contamination levels of the PIM produced magnets.







Figure 1: Evolution of the microstructure of a) the conventional processed magnet compared to the PIM produced magnets with b) 1140 ppm of C, c) 5300 ppm of C and d) 8500 ppm of C, observed by SEM-BSE

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An Investigation into the Influence of Zirconium Additions and Processing Conditions on Nd_{12.2}Fe_{81.3}B_{6.5} Strip-cast Material during the Hydrogen Ductilisation Process (HyDP)

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5. Unconventional Processing, September 5, 2023

The work presented in this paper has successfully shown that additions of zirconium coupled with improved processing conditions can eliminate currently existing issues of the Hydrogen Ductilisation Process (HyDP) [1-3]. Strip-casting has successfully reduced the amount of Nd1+ ϵ Fe4B4 phase to quantities that can be disproportionated without extended processing times, eliminating cracking during the deformation of the material. Zirconium additions and higher processing temperatures have been shown to favour the formation of coarsely disproportionated microstructures, contributing to enhanced ductility in the material, leading to deformations of up to ~26% in the initially brittle strip-cast material and improved sample shapes after the pressing.

The applied processing conditions have successfully recombined the materials, and it was shown that zirconium exhibited a cavitation-reducing effect in samples initially disproportionated at higher temperatures of 915°C. The reduced cavitation is caused by the zirconium additions inhibiting the redistribution of the Nd-rich phase during the recombination and shows promise to increase the final magnetic properties significantly.

The final remanence achieved in the strip-cast material varied depending on the processing condition and the zirconium addition and reached values of up to ~0.36T, while the maximum achieved coercivity was ~234 kA/M.

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New Possibilities for the use of the HDDR Process in the Nd₂Fe₁₄B-, SmCo₅- and Sm₂Co₁₇-based Ferromagnetic Materials

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5. Unconventional Processing, September 5, 2023

New data on the HDDR treatment of Rare Earth-Transition Metals (REM-TM) ferromagnetic materials, including $Nd_2Fe_{14}B$, $SmCo_5$, and Sm_2Co_{17} , are presented.

The nanostructure of REM-TM materials can be formed using the HDDR route with modified parameters, and this possibility has been experimentally demonstrated. It is suggested that the nanostructure forms when the grain sizes of the disproportionation products are in the nanoscale range.

After HDDR treatment, the grain sizes of the ferromagnetic phases lie in the range of 40-140 nm for the $SmCo_5$ -based alloy and 80-140 nm for the $Sm_2(Co,Fe,Zr,Cu)_7$ -based alloy [1]. The high coercivity of the sintered SmCo5-type magnet was achieved by HDDR post-sintering treatment [2].

The REM-TM materials can be sintered at low temperatures using the HDDR route. It was shown that sintering of the powders occurred during the HD stage to form a highly porous bulk material. The porosity of the sintered materials decreased to less than 1% as the compaction pressure increased and the powder particle size decreased. The decrease in sintering temperature was attributed to an increase in the diffusion rate of the alloy components caused by hydrogen-initiated phase transformations and the presence of hydrogen solid solution in the alloy. The main advantage of the new method is that it allows for the production of nanostructured materials. Ways to improve the properties of sintered materials at low temperatures are proposed, including optimizing sintering parameters and homogenizing powders by particle size.

The texture memory mechanism, which is based on the assumption that the material will be textured if it contains remnants of the main ferromagnetic phase, was experimentally proved using X-ray diffraction for texture evaluation and is proposed for customary usage.

Overall, these results indicate that the HDDR approach can be spread for producing nanostructured materials with improved properties sintered at decreased temperatures.



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Recent About HDDR Process in R-Fe-B – Based Materials and Magnets

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5. Unconventional Processing, September 5, 2023

The HDDR method was used for obtaining the sintered nanostructured Nd-Fe(Zr)-B magnets.

The initial investigation focused on understanding the correlation between grinding parameters, hydrogen treatment (HDDR) and microstructure of the sintered materials. For obtaining the anisotropic Nd-Fe-B-Zr HDDR treated alloys the remains of the Nd2Fe14B ferromagnetic phase must be present after disproportionation reaction [1]. The sintered nanostructured magnets were obtained with using treatment in hydrogen under pressure approximately of 0.05 MPa at a temperature of 700 °C and in vacuum at a temperature of 850 °C.

The relationship between the temperature of the start, peak and finish of conventional disproportionation and recombination, in hydrogen, reactions and the starting hydrogen pressure of 10-80 kPa and maximum temperature of 950–1015 °C was studied for (Nd,Pr)-Fe-B alloy powder. The non-equilibrium pressure-composition-temperature schema with four zones was built for the (Nd,Pr)-Fe-B powder alloy-hydrogen system [2].

The relationship between the parameters of the solid HDDR-route post-sintering treatment and the phase content, degree of texture, and magnetic properties of the (Nd,Pr,Gd)-Fe-B sintered magnets are presented. The low hydrogen pressure of 10-50 kPa, low temperature of 700-785 °C and dwell time of reaction up to 11 h were applied during the disproportionation of magnets. Recombination temperatures of 750, 810, and 850 °C were used. The results show that disproportionation reaction occurred in the entire pressure range. The phase content of the disproportionation products changes from the sample surface inwards as pressure increased. The starting ferromagnetic phase completely recovered across all temperature ranges. The recombined magnets showed anisotropy in the entire hydrogen pressure range studied. The study suggests that optimizing the HDDR parameters could improve the magnetic properties of sintered textured magnets.

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The Effect of the Solidification Microstructure on Magnetic Properties of Nd-Fe-B Magnet Manufactured by Laser Powder Bed Fusion

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5. Unconventional Processing, September 5, 2023

Currently, $Nd_2Fe_{14}B$ is the strongest permanent magnet but since it requires a high amount of rare earth elements (RE), the application of 3D printing is necessary to minimize material waste compared to the existing sintering process. In this work, we fabricated Nd-Fe-B magnets through LPBF (laser powder bed fusion) using an Nd-lean commercial spherical powder (MQP-S-11-9). To investigate the effect of LPBF processing parameters on the magnetic properties at similar densities, samples were prepared at the following conditions: layer thickness fixed at 30 µm, hatch distance set to 40 or 70 µm, scan speed and laser power in a range of 100-1200 mm/s and 80-160 W, respectively. The highest magnetic properties (Br= 5.3 kG, Hc= 10.2 kOe) were found at P= 90 W, V= 500 mm/s, H= 70 µm. SEM-BSE observation revealed that the grain size of Nd2Fe14B varied along the locations: fusion zone and the melt pool boundary. Based on the results, the microstructure according to process parameters was analyzed in detail, and the effect of the solidification microstructure on the magnetic properties of Nd-Fe-B magnets was reported. This study will provide insights into the design of process variables and microstructure of LPBF permanent magnet, with the ultimate goal of achieving superior magnetic properties.



Rare-earth Free Nd-Fe-B Magnets with High Coercivity by Laser Powder Bed Fusion

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5. Unconventional Processing, September 5, 2023

NdFeB permanent magnets are needed for the energy transition and the electrification of transportation, but their high rare-earth content might become an issue in the oncoming decades. Designing systems with complex-shaped magnets has proved useful to help reducing the magnet volume and the rare-earth content of systems [1] [2]. Yet, the designing effort has to be accompanied by the development of net-shape processes that are capable to efficiently realize the needed geometries at industrial scale. In this respect, Additive Manufacturing (AM) has a lot of potential. From all available AM technologies, powder bed fusion using laser beam (PBF-LB) appears best suited for producing fully dense metallic parts with high geometrical freedom and good dimensional accuracy. The first demonstration was made on a commercial Nd-lean spherical powder [3] and since then, several attempts to increase the remanence (inducing the texture) and coercivity (improving the microstructure) have been reported, particularly through the development of tailored compositions and powders [4].

A previous work reported a coercivity of PBF-LB Nd-Fe-B magnets as high as 1790 kA/m obtained with a powder composition containing 4% Dy and 1% Cu [5]. There is much literature on the role of various substitution elements in the microstructure of sintered Nd-Fe-B magnets, but conversely the effect of the chemical composition in magnets manufactured by PBF-LB has not been much investigated. Unlike the sintering process, PBF-LB implies very short characteristic times (often in the sub-ms range), which can prevent the homogenization of the composition in the melt pool when using powder blends. In this work, the possibility to study the influence of some substitution elements in the PBF-LB manufacturing of Nd-Fe-B magnets by powder-to-powder blending is investigated. Remanence of 0.63 T and coercivity of 1487 kA/m are achieved with a heavy rare earth free composition.



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UESDA

P2-7

Additive Manufacturing of (Pr,Nd)-Fe-Cu-B Permanent Magnets

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5. Unconventional Processing, September 5, 2023

Additive Manufacturing (AM) of permanent magnets is a new and challenging field in material science and engineering. Obtaining the necessary microstructure for high coercivity is by no means straightforward, especially after melting and fast cooling in Laser Powder Bed Fusion (L-PBF). The well-known Nd Fe-B sintered magnet consists of magnetically decoupled 3-10 μ m grains of the hard magnetic RE2Fe14B (RE = Rare earth element) phase, which gives rise to the highest performance. In order to achieve the desired microstructure and hard magnetic properties after printing, we propose here Pr-Fe-Cu-B as a new useful reference alloy system [1] and compare this with its Nd-based counterpart.

Our studies describe the L-PBF and the subsequent annealing optimization for L-PBF in order to understand the newly established coercivity mechanism. Specifically, we explore the 6-13-1-type grain boundary phase and grow single crystals to understand its magnetism, supported by DFT calculations [2]. Furthermore, grain boundary engineering with nanoparticle-additivation shows great potential for grain refinement and uniaxial grain growth during re-solidification during L-PBF.

Recently, fully-dense and true-to-shape cubes are printed with variable parameters. A coercivity of 0.5 T could be achieved after post heat treatment, showing the potential of AM to produce magnets.

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The Effect of Varying Nd Content in NdFeB Alloys on the Hydrogen Ductilisation Process (HyDP)

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5. Unconventional Processing, September 5, 2023

Demand for NdFeB permanent magnets is expected to triple by 2035 [1], primarily due to their use within green technologies, leading to recent research into more efficient unconventional processing techniques. The Hydrogen Ductilisation Process (HyDP) is a new processing technique for NdFeB permanent magnets. Hydrogen is introduced to solid NdFeB material at temperatures over 650°C, causing the Nd₂Fe₁₄B matrix phase to disproportionate into NdH₂, Fe₂B, Fe, while avoiding decrepitation of the material. This mixture exhibits ductile mechanical behavior [2], so HyDP has the potential to significantly reduce waste produced during magnet manufacturing, compared with conventional sintering processes.

Arc melting of NdFeB alloys produces a highly homogenous microstructure, in addition to enabling the production of specific bespoke compositions. Changes in the atomic percentage of neodymium changes the microstructure of the material. The absorption and desorption behaviour for hydrogen within the material is impacted significantly by this, as the neodymium rich grain boundary phase has been shown act as the pathway for hydrogen diffusion [3]. In addition, when the material is in a ductile state following disproportionation, the neodymium hydride is expected to exhibit different mechanical behaviour to the Fe phases which impacts the overall ductility of the material.

Research presented here shows how increasing atomic percentage of neodymium, in arc melted NdFeB alloys, impacts the processing parameters and mechanical behaviour for HyDP. Hydrogen reaction kinetics change with varying neodymium content, altering the disproportionated microstructure. Alloys with different neodymium content exhibited changes in the mechanical behaviour, as well as showing different levels of porosity due to cavitation during hydrogen desorption [4].

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TUESDA

P2-9

Laser Powder Bed Fusion of Anisotropic Nd-Fe-B bonded magnets the use of an In Situ Mechanical Particle Alignment Approach

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5. Unconventional Processing, September 5, 2023

Nd-Fe-B bonded magnets are an important class of permanent magnets, employed in many technological sectors [1]. Additive Manufacturing (AM) techniques have gained consideration as a potential route to fabricate functional magnetic components with tailored properties due to the possibility to combine both magnetic and geometrical features. The production of magnetically anisotropic components is the current challenge on AM of bonded magnets. Common approaches, presented in the literature up to now, require a post-printing step or a complex integration of a magnetic field source into the AM process. Here, we present a technique to obtain anisotropic Nd-Fe-B bonded magnets via Laser Powder Bed Fusion (LPBF), exploring a mechanical torque for the physical alignment of anisotropic particles during the printing process. Using this approach, the need for an in situ magnetic field source or a post-processing process is eliminated. For this work, a mixture of anisotropic Nd-Fe-B powder (MQA-38-14) and polyamide-12 (PA12) was used as feedstock for anisotropic bonded magnets manufacturing. This magnetic powder consists of ellipsoidal particles, where the easy magnetization axis is distributed perpendicular to their longest dimension, which can be exploited to generate magnetic texture. During the recoating step of each individual powdered layer, the magnetic particles are oriented parallel to the recoating direction, due to the mechanical torgue exerted over the particles by the recoater [2]. Magnetic texture is then developed in the perpendicular direction. The alignment degree ($\langle \cos \theta \rangle$) can be tailored according to the particle size, varying from $\langle \cos\theta \rangle = 0.63$ to $\langle \cos\theta \rangle = 0.78$. The fabricated anisotropic bonded magnets exhibited a maximum remanence of Jr = 377 mT and an energy product of (BH)max = 28.6 kJ/m³, respectively.





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The Use of Pr-Fe-Co-B HDDR Powders to Obtain Composite Magnets via Additive Manufacturing

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5. Unconventional Processing, September 5, 2023

The hydrogenation, disproportionation, desorption and recombination (HDDR) process is used to produce magnetically hard powders from homogenised alloys. HDDR powders have been produced from Pr-Fe-Co-B alloys and have been shown to be highly anisotropic due to the texture memory effect, which is desirable to achieve high remanence and high energy product composite magnets. Nevertheless, the use of such powders in Additive Manufacturing (AM) processes remains an unexplored topic in the state-of-the-art of composite magnets obtained via AM. The fabrication of composite magnets using AM allows the obtention of intricate magnet geometries, lesser waste of raw materials and the development of functionally graded materials in situ. In this work, the use of Pr-Fe-Co-B powders obtained via HDDR processing in the powder bed fusion (PBF) processing is explored. The primary objective is to produce a HDDR anisotropic powder with high coercivity and adequate particle size for the preparation of a feedstock containing the HDDR powder and various volume fractions of polyamide. Bonded magnets were fabricated processing the optimised feedstock via PBF, and the magnetic properties of the AM composites were assessed. Furthermore, the composites were subjected to a post processing by heating the composites enough to soften the polymeric matrix then applying a magnetic field to align the HDDR particles and achieve anisotropic bonded magnets. The final magnets presented magnetic properties analogous to those of commercially available bonded magnets, showing the potential of the implementation of Pr-Fe-Co-B HDDR powders in AM technologies to produce composite magnets.

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Comparison on the Improved Magnetic Properties of Melt Spun RCo₅ (R = Y, La, Ce, and Pr) Ribbons due to Fe and C-doping

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023

Developing novel permanent magnetic materials is crucial in modern technology due to everincreasing demand for the wide applications. Rare earth (R) - cobalt alloys with excellent magnetically intrinsic properties have attracted much attention. Except for SmCo5, few reports related to other RCo_{e} , such as R = Y, La, Ce, and Pr, are available, where low coercivity is found. In this work, magnetic properties of RCo5 alloys, prepared by melt spinning, are significantly enhanced by doping Fe and C, and their optimized magnetic properties are also compared. Low coercivity (iHc) of 0.2-3.3kOe is found for binary RCo5 ribbons due to extremely large size of grains. By codoping proper Fe and C, iHc and (BH)max are significantly enhance to 15.0kOe and 9.1MGOe for $YCo_{4.6}Fe_{0.3}C_{0.3}$, 11.2kOe and 11.7MGOe for $PrCo_{4.6}Fe_{0.3}C_{0.1}$, 15.0kOe and 5.1MGOe for $CeCo_{4.6}Fe_{0.3}C_{0.3}$, respectively. As to LaCo₅ ribbons, it needs Y substitution to stabilize magnetically hard 1:5 phase, and the optimized magnetic properties of iHc=8.0kOe and (BH)max=5.1MGOe are also achieved for $La_{0.5}Y_{0.5}Co_{4.6}Fe_{0.3}C_{0.3}$. The coercivity achieved in this work is superior to those reported ever before, and (BH)max is also larger than the theoretical values for each isotropic RCo5 alloys. Fe and C enter into 1:5 phase to modify lattice constant and increase TC, and the increased c/a ratio of 1:5 phase may improve Ha and contribute to enhance the coercivity. The grain size is effectively refined by doping C. Fine microstructure for C-containing ribbons contributes to attain high coercivity. Besides, Fe-doping may increase the magnetization of 1:5 phase, and therefore improve (BH)max. Magnetic properties of $RCo_{4.6}Fe_{0.3}C_{0.3}$ ribbons with fine microstructure are dominated by magnetically intrinsic properties of RCo, phase. The results of this work suggest that magnetic properties of RCo₅ ribbons could be improved by modification of 1:5 crystal and microstructure refinement due to proper Fe and C-codoping.



TUESDA

P2-12

Microstructure, Coercivity and Thermal Stability of Nanostructured (Nd,Ce)-(Fe,Co)-B Hot-compacted Permanent Magnets

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6. Nanocrystalline and Thin Film Magnets, September 5, 2023

Thermally-stable permanent magnets (PM) play a crucial role in high-temperature applications, for which Nd-Fe-B PM have intrinsic limitations. Doping heavy-rare-earth elements such as Tb and Dy is the commonly used strategy for enhancing the high-temperature magnetic properties, however, Tb and Dy are critical elements with very high price. In the efforts to reduce Tb and Dy content in Nd-Fe-B-based magnets, Ce substituted magnets attract attention, although Ce reduces the anisotropy field, spontaneous magnetization and Curie temperature in 2:14:1 phase [1,2]. This can be partially resolved by additional substitution of some amount of Fe by Co [3]. Cobalt is also a critical element, but price and criticality are lower than for Tb and Dy. Thus, if the thermal stability of the (Nd,Ce)-(Fe,Co)-B magnets can be comparable with the magnets containing HRE, the Co substitution could be an option for a cost-effective solution.

We report on microstructure, hard magnetic properties, and thermal stability of nanocrystalline $(Nd_{0.85}Ce_{0.15})_{15}(Fe_{1-x}Co_x)_{78}B_7 (x=0-1)$ hot-compacted PM. At lower Co concentrations (x<0.3), the substitution of Fe by Co changes the composition of grain boundary phase from non-magnetic (Nd,Ce)2Fe to magnetic (Nd,Ce)(Fe,Co)2, leading to degradation of the coercivity. At x≥0.6, the grain boundary phase is no longer observed between the nanocrystalline grains, resulting in strong magnetic coupling of constituent phases. With the further increase of Co concentration (x>0.6), additional RE(Fe,Co)₂, RE(Fe,Co)₄B and RECo₅ phases are gradually formed in the magnets. A new type of microstructure with nanograins of RE₂(Fe,Co)₁₄B phase, RE(Fe,Co)₂ phase and RE(Fe,Co)₄B phase, with partial or without grain boundary phase shows good thermal stability (x=0.4 and 0.6). In $(Nd_{0.85}Ce_{0.15})_{15}(Fe_{0.4}Co_{0.6})_{78}B_7$ magnets, the absolute values of temperature coefficient of coercivity $|\beta|$ can reach 0.3%/K within the temperature range of 300K-500K, and it exceeds 0.23%/K for the temperature range of 300K-650K, indicating a promising method for designing highly thermal stable PM [4].





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6. Nanocrystalline and Thin Film Magnets, September 5, 2023

Flash spark plasma sintering (Flash SPS) is an attractive method to obtain Nd-Fe-B magnets with pronounced anisotropic magnetic properties when starting from rapidly guench melt-spun powders [1]. In the Flash SPS process, a load is applied on a pre-compacted sample followed by an application of a well-defined power pulse promoting deformation and densification of the sample within seconds in a single step. Compared to the benchmark processing route via hot pressing with subsequent die-upsetting, Flash SPS promises electroplasticity as additional deformation mechanism and reduced tool wear, while maximizing magnetic properties by tailoring the microstructure - fully dense and high texture. A detailed parameter study was conducted to understand the influence of Flash SPS parameters on the densification and magnetic properties of commercial MQU-F powder. Our results reveal that pre-sintering conditions and pre-heating temperature before applying the power pulse play a major role for tailoring grain size and texture in the case of hot deformation via Flash SPS. Detailed microstructure evaluation disclose the texture enhancement with increasing temperature at expense of coercivity, see Figure 1. Magnetic domain analysis corroborates the increase in local and global texture by the appearance of wellpronounced interaction domains [2]. Magnets with 1.37 T remanence, 1195 kAm⁻¹ and (BH)max of 350 kJm⁻³ were obtained using a combination of pre-sintering at 500°C for 120 s and preheating temperature of 600°C. These findings show the potential of Flash SPS to obtain fully dense anisotropic nanocrystalline magnets with high magnetic performance.





Figure 1: Demagnetisation curves of the Flash SPS magnets produced with different pre-heating temperatures and the corresponding microstructure for the extreme cases.

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UESDA

P2-14

Steps Towards the Hardening of Cobalt Ferrite Nanoparticles

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6. Nano crystalline and Thin Film Magnets, September 5, 2023

Cobalt ferrite (CFO) is between the spinel ferrites the one that exhibits larger magnetic anisotropy. However, the coercive field of bulk oxide is small due to the cubic symmetry of the magnetocrystalline anisotropy. In fact, CFO is not considered for producing permanent magnets. Recent studies [1-5] have demonstrated that CFO powders of nanometric scale can present properties competitive with those of commercial M-type hexaferrites being due to the combination of the single domain behavior and size effects. In addition, hybrid structures containing CFO nanoparticles can present exchange bias or spring magnet behavior, interesting phenomena to develop novel nanobased magnets [6-10]. The increase of the magnetic anisotropy and of the coercive field are a key issue to obtain CFO based magnets.

In this presentation, the effects of solvent-mediated thermal annealing at low temperatures in the structural and magnetic properties of Co0.6Fe2.4O4 nanoparticles synthesized by thermal decomposition [11] are investigated. The choice of the non-stoichiometric Co composition with selected particle size allow to obtain room temperature blocked nanoparticles with high coercive field [5]. At difference of most state-of-art studies, the solvent annealing induces a non-monotonic increase of Hc that reaches a maximum of 50% larger for the treatment at 210°C. A wide structural, morphological and magnetic characterization was performed to investigate the mechanism that determines the improvement of Hc. X-Ray diffraction and High-Resolution Transmission Electron Microscopy show that the annealing process gives rise to the decreasing of the lattice parameter and a local reduction of the stress while Mossbauer spectroscopy and magnetic measurements indicate that the magnetic properties are very similar. We conclude that the thermal induced stress release reduces the random magneto-elastic anisotropies in the single domain particles giving rise to the increase of Hc. Our study shows new lights for the development of novel rare-earth free permanent magnets.



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JESDA

P2-15

Suppression of the Formation of Soft Magnetic Phase for Sm(Fe-Co)₁₂-B Thin Films by Introduction of Seed Layer

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6. Nano crystalline and Thin Film Magnets, September 5, 2023

RFe₁₂ compounds with a tetragonal ThMn₁₂-type crystal structure are expected to surpass the magnetic properties of Nd-Fe-B magnets because of excellent magnetic properties of Sm(Fe-Co)₁₂ thin films[1]. Then, we realized that a large coercivity of 1.2 T was obtained in the B-doped Sm(Fe-Co)₁₂-B thin film due to the formation of columnar structure in which the SmFe₁₂ grains were enveloped by a B-rich amorphous grain boundary phase[2]. Additionally, it was predicted that if the α -(Fe,Co) phase existing at the vicinity of the initial interface between V under layer and Sm(Fe-Co)-B main layer was suppressed, a giant coercivity of 6 T will be obtained[3]. In this study, in order to suppress the formation of the soft-magnetic phase, several seed layer materials have been introduced to the Sm(Fe-Co)-B thin films, and structure and magnetic properties have been investigated in detail.

The samples were prepared by an ultra-high vacuum magnetron sputtering system. A buffer layer and a seed layer with different materials such as V, Sm and Nb were deposited onto MgO (100) single crystal substrate at 400 °C. Then, the Sm(Fe-Co)-B layer was deposited. The composition of B and thickness of the main layer were changed. Finally, a V cover layer of 10 nm was deposited.

The peaks of (002) and (004) from ThMn₁₂-type compound were clearly observed and the intensity of the peak from α -(Fe,Co) phase was decreased for the film with a V buffer layer. On the other hand, intensity of the peaks from 1:12 phase was increased by introducing Sm seed layer. It was confirmed that from the magnetization curve measured in the in-plane direction, a rapid increase at the low applied magnetic field contributing to the generation of the soft magnetic phase was suppressed by introducing Sm seed layer.

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TUESDAY

P2-16

Fabrication of High-remanence Nd-Fe-B Hot-pressed Magnets by Manipulating Coercivity of Initial Anisotropic HDDR Powders

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6. Nano crystalline and Thin Film Magnets, September 5, 2023

The hot-deformation is one of the promising industrial processes to fabricate fine-grained anisotropic Nd-Fe-B bulk magnets and is commonly used to give the crystallographic texture to melt-spun and HDDR powders [1]. Compared to hot-deformed magnets produced from melt-spun powders, the problem with the one produced from HDDR powders is a poorer [001] texture and remanence due to their higher deformation resistance [2]. Therefore, magnetically aligning the anisotropic HDDR powders prior to densification can be a solution for achieving higher remanence in the final bulks. Additionally, if they are magnetically well aligned before densification, fine-grained anisotropic bulk magnet could be easily obtained by the hot-press alone without undergoing subsequent die-upset process. According to the previous investigation on the powder alignment under the magnetic field [3], when the remanence of particles is high, the repulsive force between the particles increases, thereby deteriorating the alignment of the particles. Thus, reducing their remanence is a key factor for improving the alignment degree of the powders. Since the remanence of the powders can be controlled by their coercivity, in this work, we manipulate coercivity of initial anisotropic HDDR powders in fabricating anisotropic hot-pressed magnets with improved remanence and systemically investigated the influences of magnetic and microstructural characteristics of initial HDDR powders on the magnetic alignment and hot-press behaviours. Notably, by manipulating coercivity of initial HDDR powder to be low, higher remanence is obtained in the hot-pressed magnets. The remanence of hot-pressed magnets fabricated from low-coercivity HDDR powders were 11.2kG, while that of the one fabricated from high-coercivity HDDR powders were only 8.2kG. A detailed method to control the magnetic and microstructural properties of the initial anisotropic HDDR powders will be explained, and the magnetic and microstructural differences between the hot-pressed magnets produced from the lower and higher coercivity HDDR powders will be discussed.

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P2-17

Enhancement of Crystal Alignment and Remanence of Large-scaled Ce-substituted Nd-Fe-B magnet by Hot Deformation

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6. Nano crystalline and Thin Film Magnets, September 5, 2023

Recently, as demand for Nd-Fe-B magnets has increased, Nd-Fe-B magnets replaced by Ce have attracted much attention due to the abundant resources and low prices of Ce. However, increasing Ce contents in (Nd, Ce)-Fe-B magnets degrades the magnetic properties because Ce₂Fe₁₄B has lower saturation magnetization and anisotropy field than Nd₂Fe₁₄B. Since Ce-based magnets have low intrinsic magnetic properties, it is necessary to fabricate anisotropic magnets by improving texture to obtain high magnetic performance. This can improve remanence. The texture improvement can be achieved by hot-pressing and hot-deforming process. The microstructure of the hot-pressed precursors prepared under suitable conditions has a uniform RE-rich distribution, and the grain alignment is improved during the hot deformation process, resulting in high magnetic properties. In this work, we report the magnetic properties of hot-deformed Ce-substituted (Nd, Ce)-Fe-B magnets prepared with various Ce contents ranging from 20 to 40 wt%. In order to suppress the formation of secondary phase of CeFe,, the melt-spun powder of the starting material was prepared in an amorphous phase by changing the wheel speed. After optimization of the hotpressing and hot-deforming conditions (pressure, temperature, and time), the final dimension of the hot-deformed (Nd1-xCex)-Fe-B magnet was Φ 24 mm \times 2 mm. The magnetic performance was measured using a B-H loop tracer. Among the various as-prepared magnets, the 40 wt% Ce-substituted magnets exhibited the highest maximum energy product, (BH)max, of 31 MGOe (Hcj = 8.9 kOe and Br = 12.2 kG). This is the first report demonstrating high performance in a large-sized hot-deformed (Nd1-xCex)-Fe-B magnet substituted with Ce 40 wt%. The mechanism of high magnetic properties has been discussed by analyzing crystal alignments and detailed microstructures under various hot-pressing conditions.


A Novel Approach for the Detection of the L10 Phase in FeNi

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8. Rare Earth-Free Magnets, September 5, 2023

The equiatomic composition of FeNi with the L10 structure is a potential candidate for rare earthfree permanent magnets. The L10 phase is only found naturally in meteorites that have cooled over billions of years. It is highly challenging to produce this phase in the laboratory due to the extremely low atomic mobilities of Fe and Ni below the critical temperature, 320°C. The extremely low intensity of the superlattice diffraction peaks and negligible change in the c/a ratio during the ordering process makes detecting the L10 phase arduous. The superlattice peaks cannot be observed using soft X-ray diffraction, but weak spots are observable in diffraction patterns in the Transmission Electron Microscope (TEM). If the superlattice peaks could also be observed by electron diffraction in the Scanning Electron Microscope, the limitations of the small volumes available for investigation in the TEM and the difficulty of sample preparation for TEM would both be overcome. The present work investigates a novel approach for detecting the L10 phase by analysing Electron Backscatter Diffraction (EBSD) band profiles. EBSD patterns are simulated for crystals with varying degrees of chemical order from which average intensity profiles of various bands can be extracted. The information from these profiles can be analysed to determine whether detection of chemical ordering may be possible in this system under a variety of experimental conditions.



Establishing the Ultrasonic-atomization of MnAI-based alloys for the Hot-extrusion of Rare-earth-free Permanent Magnets

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8. Rare Earth-Free Magnets, September 5, 2023

The high demand for Nd-Fe-B-magnets in combination with the critical nature of the rare earth elements generates interest in alternative permanent magnet materials. A particularly strong candidate with performance in the range of polymer-bonded, rare earth magnets is MnAI-C, which has promising intrinsic magnetic properties and extremely low raw materials costs. In the current work, in an effort to surpass the record-holding, commercially produced MnAI-C magnets from over two decades ago, a novel powder fabrication technique, ultrasonic-atomization (UA), has been applied to the well-established processing route of hot-extrusion. UA is shown to produce a powder with a spherical particle morphology, similar to that obtained from inert-gas-atomization; however, in UA powder almost all the particles are fully dense whereas in inert-gas-atomized powder, the majority of the particles are hollow. In addition, UA is particularly suited for the production of the small to intermediate powder volumes needed on the laboratory scale. Particular challenges in the UA of MnAl-based alloys are identified and addressed. Prior to extrusion, the UA powder was heat treated in order to optimize the content of the T-phase. The high sphericity and flowability of the UA-powder are found to be advantageous for their extrudability, thus resulting in dense MnAI-C permanent magnets. The extruded magnets show anisotropic magnetic properties due to their microstructural texture. Especially the unusually high coercivity reached in the magnets produced shows the high potential of the UA powder to further the development of MnAI-C permanent magnets.



Investigation on the Influence of Printing Parameters on the Magnetic Properties of an AM Rare Earth Free Alloy

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8. Rare Earth-Free Magnets, September 5, 2023

In recent years, additive manufacturing has become more and more relevant for producing magnetic materials due to higher demands for miniaturisation and complex-shaped magnet parts. Using laser powder bed fusion (LPBF), magnet parts of the rare earth free Fe-Cr-Co system can be produced with notable shape accuracy. For this alloy, the chemical composition can be modified directly in the printing chamber when using the in-situ alloying technique, which allows for the chemical composition to be accustomed to the specific application case required. Fe-Cr-Co obtains its hard- magnetic properties during a spinodal decomposition of a solid- solution bcc alpha phase into a ferromagnetic Fe-Co phase and a paramagnetic Cr-Fe matrix when applying a heat treatment at a given temperature in the presence of a high magnetic field. As has been demonstrated before, the LPBF process can have a considerable influence on the microstructure (e.g. homogeneity and texture), which is going to be investigated in further detail in this study. [1] Hereby, EBSD investigations have been performed as a core part of the study with the aim to correlate the changes in the magnetic properties to the changes in the microstructure observed when using different printing parameter. An additional focus has been set on the texture evolution during the thermomagnetic treatment.



Figure 1: EBSD investigations have been a key part of this study.

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UESDA

P2-21

Role of Exchange Energy on the Relationship Between Coercivity and Grain Size: Application for Hard Ferrites

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8. Rare Earth-Free Magnets, September 5, 2023

Hard ferrites, as barium and strontium ferrites are the "de facto" rare-earth-free magnet alternative to neodymium-iron-boron magnets.

However, the properties of hard ferrites are limited by their magnetization of saturation and anisotropy field (HA).

The anisotropy field of $Ba_{12}Fe_{12}O_{19}$ is 16 kOe, whereas for $SrFe_{12}O_{19}$ is 19.5 kOe.

How to improve the coercivity of hard ferrites? One possibility are the La-Co additions, but this is an expensive alternative, and this is not rare-earth-free because it involves lanthanum. Besides, cobalt is very expensive nowadays, due to strong demand in batteries. Another possibility is by considering the relationship between grain size and coercivity.

In the case of sintered hard magnetic materials as hard ferrites, NdFeB and $SmCo_5$, the coercivity depend strongly on the grain size.

There are two main situations: grain size below the single domain particle size, and grain size above the single domain particle size.

For the case of grain size above the single domain particle size, the models developed to explain the dependence of coercivity on grain size include the domain wall energy.

Methods to assess the domain wall energy, experimentally and theoretically are discussed.

A nucleation model developed in an analogy to nucleation theory of Volmer and Weber is also presented and discussed.

The developed models are compared with experimental measurements obtained for barium ferrites - BaFe12O19.

It is found that the variation of coercivity as function of the grain size in ferrites are well described by an equation where the coercivity depends on the inverse of the square root of the grain size. Barium ferrite can exhibit coercive field up to 5 kOe for very small grain size.



TUESDAY

P2-22

Effect of Processing Methods and Cu-doping on the Coercivity of SmFe₁₂-based Sintered Magnets

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8. Rare Earth-Free Magnets, September 5, 2023

Recently, the development of the next-generation $SmFe_{12}$ -based permanent magnet (PM) candidates has become a topic of interest for the PM community [1]. The $SmFe_{12}$ -based sintered magnets reported by Otsuka et al. and Zhang et al. had a coercivity of 0.8-1.0 T [2, 3]. For further improvement of the coercivity, the microstructure needs to be tuned. In this work, by varying the processing methods and composition, the microstructural features like grain size, and intergranular phase (IGP) composition were designed such that a high coercivity of 1.4 T was obtained in the $SmFe_{12}$ -based sintered magnets.

Sintered magnets made using optimally annealed as-cast ingots and strip-cask flakes as the starting materials were compared, keeping the same composition $\sim Sm_8Fe_{73}\cdot_5Ti_8V_8Al_2Cu_{0\cdot5}$. It was found that the matrix grains have better coverage by IGPs in the magnet made from strip-cast flakes than that made from ingots (Fig. 1), and consequently, the former (1.4 T) has a higher coercivity than the latter (0.85 T). The reason for the improved wettability in the former is attributed to a better homogeneity and distribution of the Sm-rich phases in the strip-cast flakes than in the ingots. Detailed investigations by TEM revealed that the IGPs are mainly composed of Fe-lean (<20 at.%) compositions (Fig. 1). It was found that not only the process but also the presence of Cu plays an important role in achieving these Fe-lean IGPs. The Fe-lean IGPs minimize the intergranular exchange-coupling in the SmFe₁₂ matrix phase, contributing to the high coercivity in contrast to the Fe-rich IGPs, which was revealed by Kerr microscopy observation. Further investigations revealed that the combination of Mg-type Sm and CsCl-type SmCu-based phases in the intergranular regions led to the Fe-lean compositions. Overall, this study provides detailed coercivity-microstructure correlations and insights for the future development of high-performance SmFe₁₂-based sintered magnets.





Figure 1: SEM images of Cu-doped SmFe₁₂-based sintered magnets made with)a) ingots and (b) strip-cast flakes as starting materials, and a high-magnification SEM along with elemental concentration profile across IGP shown for the latter.

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P2-23

Effect of Aging on the Magnetic and Physical Properties of Consolidated Mn-Al-C

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8. Rare Earth-Free Magnets, September 5, 2023

The Mn-Al material systems are one of the promising material systems which fit into the concept of the gap magnets [1]. The tetragonal τ -MnAl systems phase have the potential of theoretical energy products approximately of 100 kJ/m³. Recently, many efforts have focused on the production of the bulk Mn-Al-C material systems for specific motor application and studies on the consolidation of Mn-Al-C materials in laboratory scale has received a lot of attention [2].

In this work, we have investigated the effect of different aging conditions on the structural, microstructural, chemical and magnetic properties of the hot compacted $Mn_{56.2}Al_{42.3}C_{1.5}$ samples. High phase purity Mn-Al-C powders were prepared by Less Common Metals (LCM) using conventional casting following a KEK milling and classification process. Powders of < 300 µm were used for the hot compaction. A series of consolidation experiments were carried out at different conditions and the details of the consolidation studies will be discussed in this presentation. The structural, microstructural and magnetic studies were carried out on the consolidated samples.

Together with the magnetic and structural properties, we carried out investigations on the corrosion behaviour of the Mn_{56.2}Al_{42.3}C_{1.5} samples. The figure shows voltammetry measurement results under two different chemical environments with different pH levels. These results indicate that the T-MnAl phase oxidizes relatively easier in alkaline conditions. Considering the pH level of tap water, the evaluation of the corrosion sensibility of this material seems relevant prior to utilisation in an aqueous environment. As for the consolidated samples, structural, microstructural and magnetic characterizations of the aged samples have been carried out and the results will be discussed in detail.



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Microstructure and Magnetic Properties Evolution in Anisotropic Sm(Fe,Ti,V), -based Sintered Magnets by Composition Modification

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8. Rare Earth-Free Magnets, September 5, 2023

(Nd,Dy)-Fe-B based magnets are currently the material of choice for the applications in hybrid/ electric vehicles and wind turbines. However, the increasing demand for Pr, Nd, Tb, Dy elements has raised the necessity to reduce their usage and to diversify the utilization of rare earth elements in general [1, 2]. Therefore, research interest in rare-earth learn SmFe12-based (1:12) compounds has been revived [3]. The excellent intrinsic hard magnetic properties of SmFe12-based phases make them potential new permanent magnet materials [4]. However, the main challenge is to transfer these excellent intrinsic magnetic properties to the extrinsic ones, large coercivity and remanence, in the thermodynamically stable anisotropic SmFe12-based bulk alloy with optimal alloy composition [5]. Recently, there have been some successes in realizing coercivity in anisotropic bulk SmFe₁₂-based magnets [6, 7]. However, the obtained coercivity (μ_0 Hc = 1.0 T), which is only about 10 % of the anisotropy field of the 1:12 phase, and the small remanence (μ_0 Mr = 0.6 T) hinder their practical applications. Therefore, further fundamental studies are needed to increase their μ 0Mr and understand the coercivity mechanism.

In this work, we have demonstrated that reducing the stabilizer element Ti is an efficient approach to enhance the remanent magnetization in anisotropic Sm(Fe,Ti,V)₁₂-based sintered magnets. A record-high remanence μ_0 Mr of 0.79 T and (BH)max of 113 kJ/m³ with a moderate coercivity μ_0 Hc = 0.6 T were achieved in the Ti-reduced Sm₈Fe_{76.5}Ti₅V₈Ga_{0.5}Al₂ sintered magnet (Fig. 1). The substitution of Co for Fe in the Sm₈(Fe_{0.95}Co_{0.05})_{73.5}Ti₈V₈Ga_{0.5}Al₂ magnet resulted in a lower μ_0 Mr and μ_0 Hc. In the corresponding Co-substituted magnet, the formation of twinned grains and the ferromagnetic SmFe₂-based phase were observed. Based on the detailed microstructure analysis, we will discuss the coercivity origin and propose the optimum microstructure to increase μ_0 Hc and μ_0 Mr closer to their theoretical limits.



Figure 1: (a) Demagnetization curves for the sintered magnets, BSE-SEM images and inverse pole figure (IPF) maps of the 1:12 phase observed along the EA texture for sintered (b), (c) $Sm_8Fe_{76.5}Ti_5V_8Ga_{0.5}AI_2$, (d), (e) $Sm_8(Fe_{0.95}Co_{0.05})_{73.5}Ti_8Ga_{0.5}AI_2$ magnets

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Bulk L10-FeNi: A Novel Approach Towards the Tetragonal Phase

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8. Rare Earth-Free Magnets, September 5, 2023

Among the best candidates for rare earth-free permanent magnets the L10 FeNi alloy is very attractive because it has a large uniaxial magnetic anisotropy energy (MAE) with Ku ~ 1.3 x106J/ m3, nearly the same saturation magnetization, μ 0Ms = 1.59 T, a high Curie temperature (>450 0C) and a theoretically possible energy product (BH)max = 448 kJ/m3 [1]. The L10-type structure was first produced by Néel and Paule'v [2] with an order-disorder transformation temperature around 320 °C very low compared to other L10 alloys. Vey recently [5] it was published that there was a successful alloy casting of L10 -FeNi by adding phosphorous and carbon, which accelerate the diffusion and the formation of the tetragonal structure. In this work, we have employed a different approach. We melted together Fe, Ni at stoichiometries of Fe50Ni50 with extra addition of In at 5 and 10 % wt by RF in a cold crucible in an Ar atmosphere. From the XRD data we cannot clearly see if the L10- FeNi type structure is formed since no superlattice lines were detected. A slight indication can be seen form the broad peaks of (002) and (020). An anisotropy field, consisted with a tetragonal structure, of approximately 2-3 T was derived from measurements in epoxy oriented powders, very close to the theoretical value expected, considering an anisotropy constant Ku ~ 1.3 x106 J/m³ and a saturation magnetization μ_0 Ms = 1.38 T. The most supportive data for the formation of the L10-FeNi is derived from Mossbauer data, which clearly show the coexistence of the cubic and tetragonal structure at a ratio ~2:1 and corresponding quadrupole splitting of 0.0 and 0.45 mm/s correspondingly. Work is in progress to increase the % of the tetragonal phase and through milling to modify the microstructure in order to obtain a coercivity.

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The possible formation of tetrataenite by mechanical activation and the addition of light elements.

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8. Rare Earth-Free Magnets, September 5, 2023

Tetrataenite is an ordered tetragonal Fe-Ni phase. It is interesting for permanent magnet applications due to its intrinsic properties such as magnetocrystalline anisotropy and saturation magnetization which can result in a theoretical maximum energy product of 334 kJ/m3 [1]. The production of tetrataenite-based permanent magnets is however challenging. Indeed, the ordered tetragonal phase forms from the disordered cubic phase below an order-disorder transition temperature at 320 °C. At such temperatures the short-range atomic diffusion needed for ordering is extremely slow and estimated at one atomic jump per 2600 years, requiring millions of years for the ordering process to complete.

Several approaches have been investigated recently to facilitate the formation of tetrataenite in laboratory timescales. Severe plastic deformation techniques, such as high-pressure torsion [2], and high-energy ball milling [3] promote the formation of defects which may increase short-range atomic diffusion rates. The addition of alloying elements to stabilize the ordered tetragonal phase has also been considered [4].

This work explores the possibility to produce tetrataenite starting from a FeNi-disordered phase containing a high concentration of structural defects and carbon. To increase the density of the defects, mechanical activation techniques such as ball milling, cryomilling and cold rolling were used, while different concentrations of carbon up to 5 at.% were explored. The resulting samples were characterized by powder X-ray diffraction, scanning electron microscopy, differential scanning calorimetry and vibrating sample magnetometry. The structural and microstructural results are discussed with respect to the effect of the different mechanical activation techniques and the different concentrations of interstitial carbon. The Research Council of Norway is gratefully acknowledged for funding via the project "Rare-Earth-Free Iron-Nickel-based ordered phases for permanent magnet Applications" (303563).

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Synthesis and Magnetic Properties of Bulk α"-Fe₁₆N₂/SrAl₂Fe₁₀O₁₉ Composite Magnets

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8. Rare Earth-Free Magnets, September 5, 2023

Several recent bold public announcements from industry proposing future EV motors with no rare earth (RE) elements, further strengthens the case for development of RE-free magnets. In this light, here we present an interesting case study combining two cost-efficient and environmentally friendly RE-free magnetic materials - iron nitride α "-Fe₁₆N₂ and Sr hexaferrite.

Research on RE-based bulk exchange-coupled nanocomposites is ongoing [1,2] and similar principles can be applied also to RE-free systems. In this work, hard-soft composite magnets were produced from Al-doped Sr-hexaferrite sub-micron- and iron nitride α "-Fe16N2 nano-particles as shown in Fig. 1. Phase-pure α "-Fe₁₆N₂ nanoparticles with the average particle size of 47 nm ± 5 nm have been synthesized via a two-step route developed by Dirba et al. [3]. In short, commercial γ -Fe₂O₃ nanoparticles with an average particle size of 20 nm to 40 nm, were reduced to α -Fe in high pressure hydrogen [4] and ubsequent nitrogenation in ammonia flow was done to obtain the α "-Fe₁₆N₂ phase. The Al substituted Sr-hexaferrite powder was prepared by mixing the precursor materials Fe₂O₃, SrCO₃ and Al₂O₃ followed by calcination as described in Ref. [5]. Mixing of both powders was done by ball milling with subsequent hot pressing for consolidation.

Higher iron nitride fraction results in increased saturation magnetization, but lower coercivity. Remanence can be slightly enhanced, from 22.4 A·m²/kg in the initial ferrite powder to 26.4 A·m²/kg for 15 wt% α "-Fe₁₆N₂ [6]. Results from microstructural investigations and magnetic properties will be reported.





TUESDAY

Acknowledgements: This work was supported by the German federal state of Hessen through its excellence programme LOEWE "RESPONSE", and the BMBF within the project 03X3582.

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Investigating Plane Strain Compression as a Route to Improve Extrinsic Magnetic Properties of MnAlGa

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4. Recycling Rare Earth Magnets, September 4, 2023

A key issue with the use of MnAlGa as a permanent magnet material is that traditional methods of ensuring anisotropy and texture to build extrinsic magnetic properties into the bulk samples are ineffective as the desired τ -MnAlGa phase will thermally decompose before sintering. Experiments have shown that the precursor ϵ -MnAlGa phase undergoes plastic deformation under uniaxial compression when thermodynamically stable. As such, an alternative approach was investigated to deform the precursor ϵ -MnAlGa at thermodynamically stable temperatures to ensure a resulting texture by plane-strain compression was investigated and then the resulting texture and its impacts on the intrinsic and extrinsic magnetic properties of τ -MnAlGa following metastable transformation are discussed.



REPM 2023, 3-7 SEPT, BIRMINGHAM UK



POSTER PRESENTATIONS - P3

POSTERS P3-1 - P3-28

SESSION 9: RE-CO MAGNETS AND PROCESSING

SESSION 10: DENSITY FUNCTIONAL THEORY (DFT) AND MICROMAGNETIC MODELLING

SESSION 11: APPLICATIONS OF PERMANENT MAGNETS

SESSION 12: APPLICATIONS AND SUSTAINABILITY OF MAGNETS

SESSION 13: ADVANCED CHARACTERISATION

SESSION 14: RARE EARTH NITRIDE MAGNETS

WEDNESDAY 6 SEPTEMBER THE GREAT HALL

The Effect of Fe, Ni Substitution on Phase Balance in SmCo₅ Alloys

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9. RE-Co Magnets and Processing, September 6, 2023

SmCo₅ permanent magnets can be applied in extreme situations for their wide operating temperature (up to 400°C) and good corrosion resistance. They have found many applications from sensors to high-speed motors. One challenge for SmCo₅ magnets has been the supply chain instability of cobalt. Mining cobalt is also associated with some ethical and environmental concerns. To mitigate the supple chain risk, researchers have studied substituting a proportion of cobalt with transition metals nickel and iron, which are more abundant and have fewer supply chain constraints. To investigate the effect of nickel and iron substitution on phase balance, samples of SmCo5-xFex $(0 \le x \le 2)$ and SmCo5-xNix $(0 \le x \le 2)$ have been prepared by arc-melting. Initial microstructural analysis has shown that substituting Co with Fe causes the favored Sm(Co,Fe), phase to be replaced by undesirable Sm(Co,Fe)₇ and Sm₂(Co,Fe)₇ phases. The Sm(Co,Fe)₅ phase exists in the range of $0 \le x \le 1.2$. When $x \ge 1.4$, there is only Sm(Co,Fe)₇, Sm₂(Co,Fe)₇, and trace Sm(Co,Fe)₃. However, the nickel substitution has shown the Sm(Co,Ni)₅ can be stabilised in the range of $0 \le x \le 2$. To explore the effects of adding nickel to stabilise the Sm(Co,Fe)₅ alloys, the cobalt is simultaneously replaced by nickel and iron in the formula SmCo5-x-yFexNiy where 0≤x≤2.5 and 0≤y≤2. The microstructure, phase content and calculated phase composition shown in this paper highlights that it is possible to stabilise the Sm(Co,Fe,Ni)₅ phase in arc melted buttons with varying degree of dopant levels.



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9. RE-Co Magnets and Processing, September 6

When modelling permanent magnet motors, it is important to understand the resistivity of the magnetic material, and its eddy current flow. These motors often operate over a wide range of temperatures and thus an understanding of the dependency of resistivity with temperature is necessary for accurate modelling.

Previous works [1] have shown that for SmCo type magnets the resistivity can be anisotropic for 1:5 and 2:17 type magnets, and that operating temperature can significantly change resistivity.

The authors investigate the temperature dependence of different commercial grade SmCo 2:17 type and 1:5 type magnets, up to 200°C, in both a magnetised and non-magnetised condition. Anisotropic resistivity was observed in both types, with the 2:17 types having lower resistivity in the plane perpendicular to the c-axis, and the 1:5 type having lower resistivity in the parallel plane. At room temperature, resistivity was found to be similar in both the magnetised and non-magnetised states and a linear increase in resistivity was observed with increasing temperature for both. However, the temperature coefficient was found to be lower in the magnetised state, leading to a lower resistivity at higher temperatures than the non-magnetised state.

For 2:17 type magnets of similar composition, differences in the lamellae within the cellular structures were found to produce variations in the resistivity, in the axis perpendicular to the c-axis.

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Influence of Oxygen Amount to Constituent Phases in Hot Isostatic Press (HIP)-processed Sm-Y-Fe-based Alloy with ThMn₁₂ Compound

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9. RE-Co Magnets and Processing, September 6

Compounds having ThMn₁₂ structure [1] are attracting attention as a candidate for magnets that use less rare earth elements than Nd-Fe-B magnets. Yttrium is an interesting element from the viewpoint of stabilization of ThMh₁₂ structure and magnetic properties [2, 3]. In order to achieve high magnetic properties, it is important to suppress formation of unfavorable phases, especially soft magnetic phases such as bcc-Fe and Th₂Ni₁₇ type phases, and to form intergranular phases like Nd-Fe-B-based magnets. In this study, we applied the hot isostatic press (HIP) to Sm-Y-Fe based alloy powder with Co, Ti and Cu to fully-densify without evaporation of Sm. We especially focused on influence of oxygen to constituent phase in the sample after HIP treatment.

 $Sm_{5\cdot5}Y_{3\cdot3}Fe_{68\cdot2}Co_{13\cdot6}Ti_{4\cdot5}Cu_{4\cdot7}$ (mol%) alloy powder was applied as starting material for HIP in this study. Noted that oxygen in the sample is easily increased when particle size is small, we prepared powder with different particle sizes to discuss the influence of oxygen. Our experiments show that both oxide including Y and bcc-Fe phases increased with increasing the amount of oxygen in the HIP processed sample, which suggested that the increase of bcc-Fe phase was due to the decomposition of the ThMn₁₂ phase with the formation of the Y-containing oxide phase. In order to suppress the decomposition of the main phase, we prepared HIP-processed sample with different amount of rare earth especially Y, and we found compositional region in which bcc-Fe and Th₂Ni₁₇ type phases suppressed.

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Sm-Fe(Co)-Ti Phase Equilibria and Liquid-phase Sintering of Sm(Fe,Co,Ti)₁₂ Magnets

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9. RE-Co Magnets and Processing, September 6, 2023

A revision of the Sm–Fe–Ti high-temperature phase diagram is long overdue, as the data acquired in the 1990s [1] are insufficient for guiding the ongoing efforts to develop new rare-earth-lean permanent magnets based on the ThMn₁₂-type tetragonal compound. Recently, we have studied the Fe-rich corners of isothermal sections at 1000 °C [2], 1100°C and 1200 °C, including those of a cobalt-substituted Sm–Fe_{0.8}Co_{0.2}–Ti quasi-ternary system. The newly constructed phase diagrams show that the equilibrium between the high-anisotropy Sm(Fe,Ti)₁₂ phase and a liquid phase, which is needed for liquid-phase sintering of the magnets, becomes possible above the decomposition temperature of the non-magnetic Sm(Fe,Ti)₁₁ phase between 1000 °C and 1100 °C. After the monoclinic Sm₃(Fe,Ti)₂₉ phase is replaced at around 1200 °C by a hexagonal Sm₂(Fe,Ti)₁₇ phase [3], this 1:12 + L equilibrium becomes possible for the 1:12 phase stabilized by less than one Ti atom per formula unit (Fig. 1).



Figure 1: Backscattered-electrons SEM micrograph of a $Sm_{_{9.9}}Fe_{_{67.0}}Co_{_{16.5}}Ti_{_{6.6}}$ alloy quenched from 1200 °C: grains of the 1:12 phase are surrounded by remnants of Sm-rich liquid.

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High Coercivity in Monocrystalline (Sm,Zr)(Fe,Co,Ti)₁₂ Particles Prepared via High-temperature Calcium Reduction of Oxides

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9. RE-Co Magnets and Processing, September 6, 2023

Monocrystalline $Sm_{(1-x)}Zr_{(x)}(Fe,Co)_{(12-y)}Ti_{(y)}$ particles with the ThMn₁₂-type structure exhibit a record-high room-temperature coercivity of 16.3 kOe when prepared via calcium reduction of mechanically activated metal oxides at 1200 °C [1,2]. The high coercivity values are attainable if the 1:12 compound forms in a Sm-depleted environment starting from a primary α -Fe particles, but they are not attainable if the formation of the 1:12 compound is preceded by a Sm-enriched TbCu₇-type phase – despite a similar size and crystal structure of the resulting particles. In this work, we studied the intermediate stages of the reduction synthesis to better understand how different formation in the Sm-depleted environment is confirmed as occurring at the expense of the primary α -Fe phase (Fig. 1). On the other hand, the 1:12 phase forming alongside the Sm-enriched 1:7 phase initially emerges as very Ti-rich. Its evolution in this case involves major changes in the chemical composition that may result in an imperfect crystal structure.



Figure 1: Growth of ThMn₁₂-type monocrystalline Sm-Zr-Fe-Co-Ti particles in Sm-depleted environment: (a) evolution of phases and coercivity, (b) TEM elemental map after synthesis for 30 s.

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Magnetic Properties and Phase Diagram of Sm-Co-B: An Exploratory Study

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9. RE-Co Magnets and Processing, September 6, 2023

SmCo₅ magnets is the material choice in aerospace engineering and satellite communication areas due to their unique properties such as resistance to extreme temperature (-300°C to 550°C) including rapid temperature changes and being less reactive and corrosive. By substituting B in SmCo4B, the anisotropy field can be enhanced from 40 T to 90 T (estimated) at 300 K [1], while also reducing the price but it also lowers the magnetization (µ0Ms) and Curie temperature (Tc). In this work, to address low µ0Ms and Tc, we synthesised SmCo₄B, SmCo₃₈Fe₀₂B and Sm₀₇Nd₀₃Co₃₈Fe₀₂B by induction melting. Nd is substituted for Sm due to its reported higher µ₀Ms compared to Sm, and Fe is included to enhance the magnetization as it is observed in RCo₅ system [2,3]. Chemical compositions of nearly single-phase ingots were determined by inductively coupled plasma mass spectroscopy. Crystal structure and microstructure characterization were carried out by X-ray diffraction (XRD). The magnetic measurements were carried out using a 14 T PPMS-VSM and the vertical single-turn coil equipment up to 100 T [4]. The XRD results confirm that all the ingots have a single SmCo₄B (Fig. 1 (a)). The µ0Ms increases from 0.35 T to 0.48 T for SmCo_{3.8}Fe_{0.2}B and to 0.55 T for Sm_{0.7}Nd_{0.3}Co_{3.8}Fe_{0.2}B. Tc increases from 503 K to 563 K with Fe substitution but it decreases to 550 K by substitution of Nd. Our preliminary magnetic measurements indicates that Fe substitution (Fig. 1 (b)) doesn't have any effect on anisotropy field and remains around ≈ 60 T and it decreases to \approx 30 T by replacing Sm with Nd. We will also discuss our results on the exploration of phase diagram of Sm-Co-B which is aiming to find the co-existence of Sm2Co17 (high µ0Ms) and SmCo4B (high µ0Ha) phases.



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Atomistic Calculation of the Domain Wall Energy in Rare-earths with Hexagonal Structure

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10. DFT and Micromagnetic Modelling, September 6, 2023

In the case of hard magnetic materials, the magnetization changes abruptly inside the domain wall. Thus the Bloch continuum domain wall model is not valid for phases with high magnetocrystalline anisotropy.

Bloch approximation is based on the MacLaurin expansion of the cosine function, where the first term is 1 and the second term is $-x^2/2$ (i.e. an order two polynomial) neglecting higher order terms (order 4,6,8, etc... polynomials).

In the Bloch wall, the exchange interactions, which are described by a scalar product between two vectors, are approximated by a polynomial of order two.

The cosine function varies between -1 and 1, and thus there is no possibility of infinities. However, a polynomial of order two can go to infinite if $x \sim \infty$. Thus, the Bloch approximation may significantly overestimate the exchange energy contribution.

Formulated in 1932, the Bloch wall model assumed that the electron is localized, and neglects antiferromagnetism.

In the 1950s and 1960s, Neutron Diffraction, Mossbauer and other experimental techniques showed antiferromagnetism even in phases considered as soft as bcc iron. Also in the 1960s it becomes clear that the electron is itinerant.

The traditional Landau Lifishitz equation, presented in 1935, assumes that the electron is localized, and neglects antiferromagnetism. These assumptions are typical in the models developed in the 1930s.

The Hamiltonian of the Landau-Lifshitz model can be altered to take into account antiferromagnetism and also that the electron is itinerant.

Here, it is given an example on how to address the exchange energy in an atomistic simulation of domain walls in rare-earths with hexagonal structure and only one easy axis. This is the case named as the narrow domain wall.

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Electrical Analogous for Permanent Magnets

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10. DFT and Micromagnetic Modelling, September 6, 2023

Expressions to simulate the intrinsic demagnetizing curve of permanent magnets are derived from the classical electrical analogy of a 2RLC circuit as an equivalent to a magnet under demagnetization in a closed magnetic circuit. Comparisons between experimental and theoretical intrinsic M x H curves were carried out for hard magnetic material, and the possibility of using this analogous circuit with less than three components has also been investigated. The Stoner-Wohlfarth model theoretical curves have also been used as a comparison. At last, the electrical analogous parameters have been systematically varied in order to correlate them to the magnetic properties.



Tomography-based Micromagnetic Simulations of Nd-Fe-B Magnets: the Role of Intergranular Phase Nonuniformities

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10. DFT and Micromagnetic Modelling, September 6, 2023

High-performance $Nd_2Fe_{14}B$ -based permanent magnets are among the key functional materials for green energy technologies such as wind turbines and electric/hybrid vehicles. Although decades of research have pushed the maximum energy product of Nd-Fe-B magnets close to its theoretical limit, the coercivity is still far below its potential [1]. The required coercivity enhancement is strongly related to the fine tuning of the intergranular phase (IGP), i.e., its thickness, magnetic properties and the local variation of both. Other microstructural features such as grain size, crystallographic texture, secondary phases, etc. should also be well controlled.

Up-to-date, the contributions of listed microstructural features have been investigated in simplified micromagnetic models under certain assumptions living a room for debate and hindering a quantitative comparison with experimental data [2,3]. In this report, we present a large-scale micromagnetic model of hot-deformed Nd-Fe-B magnets constructed based on a FIB-SEM tomographic data. This model can accurately reproduce the microstructure of real magnets. In particular, the IGP is represented by a set of thin individual regions localized between contacting faces of adjacent platelet-shaped grains. All such IGPs are isolated from each other by triple junctions. Here we performed micromagnetic simulations to study how the variations of both IGP thickness and magnetization, which have been experimentally observed [4], affect the coercivity of the hot-deformed Nd-Fe-B magnets. Also, we will emphasize another important factor for coercivity engineering that is triple junctions. Some relatively thin triple junctions are supposed to be ferromagnetic and can act as nucleation centers for magnetization reversal reducing coercivity.

Developed tomography-based micromagnetic model reproduces all major microstructural features of hot-deformed Nd-Fe-B magnets. Such a model can be considered as a digital twin of Nd-Fe-B magnets which can be useful to define the most efficient strategy for further coercivity improvement.



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Effect of Transition Metal Doping on Magnetic Hardness of CeFe₁₂-Based Compounds: DFT Study

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10. DFT and Micromagnetic Modelling, September 6, 2023

ThMn₁₂ -type ternary cerium alloys with tetragonal structure (s.g. I4/mmm) are considered as promising materials for permanent magnets. In this work, compositions of CeFe_{11x} (s.g. Pmmn) and CeFe₁₀₂₂ (s.g. P4/mmm) with all 3d, 4d, and 5d transition metal substitutions are considered. Since many previous studies have focused on the CeFe₁₁Ti compound, this particular case became the starting point of our considerations and we gave it special attention. We first determined the optimal symmetry of the simplest CeFe₁₁Ti structure model. We then observed that the calculated magnetocrystalline anisotropy energy (MAE) correlates with the magnetic moment, which in turn strongly depends on the choice of the exchange-correlation potential. MAE, magnetic moments, and magnetic hardness were determined for all compositions considered. Moreover, the calculated dependence of the MAE on the spin magnetic moment allowed us to predict the upper limits of the MAE. We also showed that it does not depend on the choice of the exchange-correlation potential form. The economically justifiable compositions with the highest magnetic hardness values are CeFe₁₁W, CeFe₁₀W₂, CeFe₁₁Mn, CeFe₁₀Mn₂, CeFe₁₁Mo, CeFe₁₀Mo₂, and CeFe₁₀Nb₂. However, calculations suggest that, like CeFe12, these compounds are not chemically stable and could require additional treatments to stabilize the composition. Further alloying of the selected compositions with light elements embedded in interstitial positions confirms the positive effect of such dopants on hard magnetic properties. Subsequent calculations performed for comparison for selected isostructural La-based compounds lead to similar MAE results as for Ce-based compounds, suggesting a secondary effect of 4f electrons. Our preliminary results obtained using the intra-atomic Hubbard repulsion term showed a relatively small difference for CeFe, compared to the results without this correction. Calculations were performed using the full-potential localorbital electronic structure code FPLO18. The above results were published in the paper [1].

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WEDNESDAY

P3-11

Investigating the Role of Interactions on the Stability of Magnetic Anisotropy in L1, Magnetic Materials

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10. DFT and Micromagnetic Modelling, September 6, 2023

Recent studies have revealed the superior magnetic properties of $L1_0$ magnetic materials which lead to a vast number of applications ranging from magnetic recording to medical imaging. While there is a wealth of experimental studies and numerical simulations aimed at finding ways to tune the magnetic properties of these $L1_0$ magnetic materials, there is inadequate attention given to understanding the underlying mechanisms that govern the magnetic properties of these materials, such as their magnetic anisotropy. Hence, this study aims to elucidate how fundamental interactions such as the electron-electron interaction combined with crystal symmetry affect the magnetic anisotropy of $L1_0$ magnetic materials. To achieve this, the material is modeled by a tight-binding Hamiltonian with electron-electron interactions accounted for using a Hartree-Fock mean-field approximation. This approach allows us to calculate the magnetic anisotropy as a function of the interaction strength and work through crystal symmetry-related trends in the anisotropy. These trends can be directly compared against material-specific ab initio calculations.



Micromagnetic Study of the Impact of Grain Boundaries on Coercivity

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10. DFT and Micromagnetic Modelling, September 6, 2023

The crystal structure, ferromagnetic properties and thickness of grain boundaries (gb) separating the grains of a Nd₂Fe₁₄B-type permanent magnet significantly influence the figures of merit such as coercivity, remanence or the energy density product (BH)max. Grain boundaries act as anisotropy defects. We use micromagnetic simulations to quantify the influence of those. The saturation magnetization (Ms) of Fe_100-xNd_x strongly depends on the iron content, changing from the Ms of pure Fe to nonmagnetic for high Nd concentrations [1]. Soft magnetic grain boundary phases reduce coercivity with increasing thickness of the gb. A 2 nm thick gb with μ_0 Ms = 0.7 T reduced coercivity by 25% compared to a 2 nm nonmagnetic gb, a 6 nm thick gb caused a reduction by 60%. On the other hand, nonmagnetic gb increased coercivity slightly with increasing thickness (+4% comparing 1 nm to 4 nm thick nonmagnetic gb) but reduced remanence.

Another reduction of coercivity might be due to surface anisotropy. Rare-earth ions may exhibit planar magnetic anisotropy on the surface of grains [2]. Micromagnetic simulations revealed that such an effect reduced coercivity by 7% up to 30% even for grain sizes larger than 100 nm.

Strain in the range of 1.2% was measured in $Nd_2Fe_{14}B$ sintered magnets [3] near grain boundaries. Such strains may reduce the magnetocrystalline anisotropy by around 10 percent [4]. Micromagnetic simulations showed that a reduction of the anisotropy constant K₁ by 10% in a 1.5 nm thick surface region of a 300 nm $Nd_2Fe_{14}B$ cube reduced coercivity by 6.5%.

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Localized Demagnetization Between Unequally Sized Like Magnetic Poles and FEA Determined Occurrence Conditions for Applications

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11. Applications of Permanent Magnets, September 6, 2023

Applications of magnets commonly involve forces between magnets with different sizes. When two unequally sized magnets with their like poles approaching each other, the force between them may transform from repulsion to attraction as they reach a critical distance. The attractive force between the initially like poles does not violate the basic law of magnetism. The origin of this phenomenon is the localized demagnetization, i.e., the magnet with a high permanence coefficient in the pair can locally reverse the polarity of the one with a low permanence coefficient, either temporarily or permanently, resulting in an unlike poles region with respect to each other. Finite element analysis (FEA) has verified the experiment observation of the like-poles attraction due to the localized demagnetization. By processing our FEA results, the level of localized demagnetization can be quantified by integrating the magnetic flux density changes near the magnet with and without the unequally sized like pole counterpart. Understanding this phenomenon is crucial in designing novel magnetic devices that require precise control of magnetic forces.

In this talk, the latest experimental and theoretical studies of the origin, characteristics, and potential applications of this phenomenon will be presented. The working principles of the magnetic devices based on localized demagnetization will be discussed. It will show that a powerful tool has been provided by FEA simulations' verification, which can be used to systematically study the occurrence conditions for various applications. Embodiments of these principles will be illustrated, including magnetic vibrating assembly, micro electric generator, magnetic propulsion device, container sealing assembly, in-situ magnetic patterner, magnetic docking device, and energy harvester.



WEDNESDAY

Polymerisation of Barium Hexaferrite Ferrofluids Producing a Magnetic Nanoparticle Composite Matrix

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11. Applications of Permanent Magnets, September 6, 2023

Recently discovered barium hexaferrite (BaFe₁₂O₁₉) nano-platelets form a ferromagnetic-ferrofluid which exhibits interesting magnetic properties [1]. Manufacturing the barium hexaferrite nanoplatelets via hydrothermal synthesis remains of fundamental interest and allows for optimisation of structural, magnetic, and morphological properties, which all impact on ferrofluid properties. We have developed a hexadecyltrimethylammonium bromide surfactant assisted manufacturing method for ethylene glycol based ferrofluids with barium hexaferrite. The ethylene glycol based ferrofluid and succinic acid. The proposed in-situ method creates a homogeneous nano-platelets polymetrice matrix was chemically characterised by Gas Chromatography (GPC) and magnetically characterised by Vibrating Sample Magnetometry (VSM). The proposed anisotropic magnetic polymer is highly stable with potential applications in thermal, electrical, medical, and analytical devices.

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11. Applications of Permanent Magnets, September 6, 2023

A simple and scalable method of synthesizing high-purity epsilon iron oxide (ϵ -Fe2O3) NPs with a large coercive field (~22 kOe) was successfully developed for millimeter-wave absorption applications. The epsilon ferrite has garnered considerable attention because of its high coercive field of more than 20 kOe at room temperature, which is 3–4 times higher than those of ferrites and similar to those of rare-earth permanent magnets. In addition, ϵ -Fe2O3 exhibits electromagnetic (EM) wave absorption at a high frequency (~182 GHz) because the zero-field ferromagnetic resonance is proportional to its strong magnetic anisotropy field. However, the previous methods to synthesize ϵ -Fe2O3 involve multiple time-consuming steps, produce low yields, and require expensive sacrificial templates, which limit large-scale production and practical applications.

This synthesis approach based on rapid spray drying of the precursor solution enables the spatial confinement of Fe salts in the silica microspheres, which restricts excessive crystal growth during the annealing step. The particle size required to form the ε -phase can be easily tuned by controlling the precursor molar ratio and annealing temperature. Thus, high-purity ε -Fe₂O₃ particles can be obtained via a simple, fast, and scalable process without utilizing surfactants or porous templates. Additionally, the use of a simple and continuous process to produce precursor powders can considerably reduce the duration of the synthetic process. Furthermore, the ability to absorb millimeter waves was demonstrated using the ε -Fe₂O₃ composite film, which the resonant frequencies (fr) were shown from 105 GHz to 71 GHz. This synthetic strategy is expected to pave the way for industrial applications of ε -Fe₂O₃ such as permanent magnets, magnetic recording media, informational storage, and millimeter-wave absorbers for use in high-speed wireless communication.



Voltage-driven Giant Modulation of Magnetism in Permanent Magnets

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11. Applications of Permanent Magnets, September 6, 2023

Magnetism and magnetic properties of rare-earth permanent magnets are typically tailored and optimized by engineering their compositions, crystal structures and microstructural fea-tures, which involve irreversible processes. However, achieving reversible control of mag-netism using small voltages is desirable for both fundamental interests and potential techno-logical advancements. A well-known example of this concept is the electric-field control of electron transport in semiconductors, which has led to the creation of field-effect transistors, revolutionizing modern society.

Here we demonstrate that the magnetism and magnetic properties of rare earth permanent magnets can be reversibly controlled with voltages as low as 1 V. This is accomplished through electrochemically-driven insertion/extraction of hydrogen atoms in interstitial sites of the crystal structure. By applying a voltage of only 1 V, we show that the magnetocrystalline anisotropy and coercivity of SmCo5 and Sm2Co17-based magnets with micrometer-sized particles can be reversibly modified by more than 1 T [1,2]. As a result, we have achieved voltage-assisted and -controlled magnetization reversal in permanent magnets at room temperature. We extend this concept of voltage-control of magnetism to Sm-Co thin films and bulk SmCo magnets with millimeter sizes. In the former case, we achieved the reversible switching of coercivity between 2.5 T and nearly zero tesla, as well as the voltage control of demagnetization and re-magnetization without applying any magnetic fields. In the latter case, we can switch the magnetization of the entire bulk magnet by applying small voltages. Our research work demonstrates the possibility of reversibly controlling the magnetic properties of permanent magnets, and analyzing the dynamic tuning process will improve our understanding of magnetism in these materials. Moreover, the ability to tune magnetic properties with small voltages has potential applications in data security, archival storage, and magnet recycling.

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Hard Magnetic Phases of CeFe₁₁W_{1-x}Ti_x

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11. Applications of Permanent Magnets, September 6, 2023

We report on the hard magnetic phases of $CeFe_{11}W_{1-x}Ti_x$ and compare with the well-known CeFe11Ti. The W compounds were suggested to exist by Gollet al. (JOM 2015,https://doi.org/10.1007/s11837-015-1422-8), but have not reported in the bulk before. In this study, using a combination of X-ray diffraction, magnetometry and computational methods the properties of $CeFe_{11}W_{1-x}Ti_x$ are explored. Overall, the performance of $CeFe_{11}W$ (x=0.0) as compared to the well-known $CeFe_{11}Ti$ (x=1.0) has lower potential from magnetic point perspective. However, W, as compared to Ti may have additional benefits, such as inhibition of the detrimental $CeFe_2$ Lavesphase or superior mechanical strength. Here, we report saturation polarization at 10 K for $CeFe_{11}W(x=0.0)$, $CeFe_{11}W_{0.5}Ti_{0.5}(x=0.5)$ and $CeFe_{11}Ti$ (x=1.0) to be 1.13 T, 1.29 T and 1.37 T, respectively and compare it to the theoretical results. Magnetic anisotropy, using the law of approach to saturation is estimated to be1.29 MJ/m³, 1.45 MJ/m³ and 2.23 MJ/m³ for $CeFe_{11}W$ (x=0.0), $CeFe_{11}W_{0.5}Ti_{0.5}$ (x=0.5) and $CeFe_{11}Ti$ (x=1.0), respectively. The Curie temperature is reported to be479 K, 494 K and 519 K for $CeFe_{11}W$ (x=0.), $CeFe_{11}W_{0.5}Ti_{0.5}$ (x=0.5) and $CeFe_{11}Ti$ (x=1.0), respectively.



1D-2D Assemblies of Sub-millimeter NdFeB-based Magnets

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11. Applications of Permanent Magnets, September 6, 2023

Small-scale permanent magnets, which offer high magnetic field density, are pivotal components for the development of high performance magnetic microelectromechanical systems (MEMSs) [1,2]. Promising applications utilizing these microflux sources involve electromagnetic actuators [3] linear encoders [4] energy harvesters [5], etc.

Magnetic measuring offers numerous advantages over other positioning and measuring methods for industrial applications allowing the measurement of a distance with high accuracy attributed to the magnetic field's irrelevance to the non-magnetic disturbances such as dirt, dust, or liquid droplets. A typical measuring solution consists of a measuring head with a magnetic field sensor and a magnetic linear scale (1D) composed of a highly accurate pattern of magnetic North(N) and South(S) poles. Via counting the number of NS alternations the distance is determined with high accuracy and sensitivity. Ferrite-based micromagnets are the building block materials of the current state-of-the-art magnetic scale applications presenting a favorable compromise between the desired properties and costs. However, these structures provide the lowest magnetic field strength of all permanent magnetic materials with a direct consequence on the sensitivity of magnetic scale applications.

We propose the replacement of Ferrite-based micromagnets with 1D or 2D- arrangements of NdFeB micromagnets, which deliver up to 20 times stronger magnetic field per unit volume leading to enhanced sensitivity and accuracy of these structures. Our new generation NdFeB-based magnetic scales present smaller pole pitch, improved tolerance, and position accuracy compared to their Ferrite-based counterparts. Simulated results using a COMSOL software will be compared with experimental data. Our novel 2D-arrangements will create magnetic metasurfaces, that can find applications in microsystems such miniature electron accelerators, novel 2D electromagnetic actuators, biomedical and energy harvesting applications.

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A Method for Quality Assessment of FeNdB Sintered Magnets

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13. Advanced Characterisation, September 7, 2023

For high quality electrical energy converters, a constant quality of materials such as FeNdB sintered magnets must be ensured. The quality may vary between manufacturers, batches and grades. Differences in the chemical composition and manufacturing process can lead to variations in the microstructure, for example, the occurrence of oxides or η -phase. Also, other defects such as abnormal grain growth can appear if the chemical composition, the powder processing or the heat treatment are not suitable. The number of defects can be significant, but still rare and therefore easy to be overlooked with conventional optical microscopy. To identify such defects, large-scale optical microscopy methods are needed. In combination with machine learning (ML) methods a semi-autonomous analysis is possible. With ML thousands of microstructure images of a magnet can be reduced to a small number of the worst ones. The resulting worst picture gallery can be used to compare different magnets based on their defects. This enables the quality assessment across batches and gives a benchmark tool to compare different manufacturers.

The focus of this work lies on two types of magnets, a low and high temperature magnet, from four different manufacturers. Although the magnetic properties are the same, the microstructure may differ significantly. With different analysis methods such as SEM (EDS, EBSD) and large-scale optical microscopy these magnets are fully analyzed. With different ML methods (supervised and unsupervised) various defects typical for each investigated magnet are found. The method provides a promising benchmark tool to compare different types of magnets and enables the detection of imperfections in the microstructure.


Recoil Curves as a Tool to Identify Coercivity Mechanisms

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13. Advanced Characterisation, September 7, 2023

Recoil curves can be used to identify coercivity mechanisms.

In the case of hard magnetic materials, there are two main situations: (i) grain size above the single domain particle size (i.e. multi-domain grains), which is typical of sintered NdFeB magnets and (ii) grain size below the single domain particles, also known as monodomain Stoner-Wohlfarth particles.

The spring effect observed in the 2nd quadrant of the hysteresis in NdFeB melt-spun magnets is according to the prediction of the Stoner-Wohlfarth model, and it is atributted to reversible rotation of single domain size particles.

If the coercivity mechanism were pinning, then the domain walls would be trapped at the grain boundaries, and no spring effect would be observed.

Calculations indicate that, in the case of multidomain size grains, the recoil effect is very limited, and very difficult to be observed [1,2].

Spring effect observed in several different materials, as hard ferrites (BaFe₁₂O₁₉), SmCo 2:17 type magnets [3] and melt-spun NdFeB are presented and discussed

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Vectorial Rare Earth Magnet Hysteresis Loops Measured in a Biaxial Vibrating Sample Magnetometer

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13. Advanced Characterisation, September 7, 2023

The behavior of permanent magnets in magnetic fields acting at an angle to the alignment direction is of great interest for the designers of permanent magnet electrical machines. The fields in the machines are at large angles, in particular in the regions between the poles, and motor designers find it difficult to determine the risk for demagnetization in these fields. Measurements of the angular dependence of the demagnetization curves are not just rare. The few published curves, measured in standard magnetometers, also suffer from the fact that they only show the projection of the magnetization in the direction of the applied field. For large angles, this information is of very limited value. Similar problems are encountered by the designers of magnetizing equipment. It is highly desirable for motor manufacturers to assemble the rotors with unmagnetized magnets, applying multipole magnetizing fields to the finished rotor. But modeling the result of the magnetizing process with any accuracy is currently not possible, as it would require a detailed knowledge of the initial magnetizing curves and of the resulting magnetization state as a function of both the magnitude and direction of the magnetizing fields.

Biaxial VSM's, equipped with electromagnets and two perpendicular sets of detection coils, are available and have been used for studying magnetic recording media. The large fields required for rare earth magnets, however, require using superconducting coils, where the implementation of a bidirectional detection coils system is less straightforward. The presentation reports angular dependent hysteresis loops in fields up to 9 T, measured with a biaxial detection coil system that was implemented in a commercial VSM.



3D representation (red curve) of the hysteresis loop J(H) of a sintered NdFeBmagnet, measured with the external field applied at an angle of 60°. J_{axial} and $J_{transv.}$ Refer to the polarization components in parallel and perpendicular to the applied field. The blue projected curves show the signals of the two detection oil systems "axial" being the curve as observed in a traditional VSM measurement. The sample was a cylinder $\Box 4 \times 2 \text{ mm}$, with diametric orientation of the easy axis.

Thermal Stresses in the Nd₂Fe₁₄B Polycrystal System with Grain Boundary Phase

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13. Advanced Characterisation, September 7, 2023

Strain in the range of 1.2 % has been measured in $Nd_2Fe_{14}B$ sintered magnets [1] near grain boundaries. Such strains may reduce the magnetocrystalline anisotropy by around 10% and in turn reduce the coercive field [2].

The present study focuses on the evaluation of residual stresses caused by temperature changes in production and recycling of $Nd_2Fe_{14}B$ permanent magnets. Using a mechanistic approach we derive an analytical solution, for the contact of two infinitely large crystals (Fig. 1). This is equivalent to a system of alternating plates of two materials with different thickness and constant period of alternation. For checking the analytical solutions, modelling has been performed by the FEM [3]. Strain values were measured in the stationary area. We computed the strain for different systems such as direct contact of two $Nd_2Fe_{14}B$ grains at different orientations and $Nd_2Fe_{14}B$ – Cu interfaces. The results of the analytical calculation and simulation have shown satisfactory matching.

For the model system, the computed thermal strains are in the order of 0.1% to 0.2%, which is a factor of 10 smaller than the maximum strain measured in [1]. However, strain hot spots may occur at triple junctions [4] not considered in the current analysis. Nevertheless, our work shows the impact of different scenarios on the stress level. In the system of two differently oriented identical Nd₂Fe₁₄B crystals the maximum residual thermal stresses are 330 MPa as the temperature changes by 500 K. Assuming a Cu grain boundary phase, which may be achieved in recycled Nd₂Fe₁₄B magnets, the maximum stresses in Nd₂Fe₁₄B reduces to 11 MPa. The use of grain boundary materials with low stiffness and small thickness should significantly reduce the level of residual thermal stresses in magnetic crystals.



Figure 1: Two-crystal system: schematics and modelling [3].

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Feature Extraction of 3D Real Pictures of Microstructure and Magnetic Domain in a Tb Diffused Nd-Fe-B Sintered Magnet

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13. Advanced Characterisation, September 7, 2023

To reveal the relationship between microstructures and magnetic domain structures of permanent magnets, historically, this has been studied on the surface of magnets by two-dimensional methods such as Kerr microscopy [1]. However, due to the influence of surface defects and surface demagnetization field, the magnetic domain on the surface differs from that inside magnet. Therefore, measuring the three-dimensional microstructures and magnetic domain of the same volume has been a longstanding goal for many magnet researchers.

Very recently, the method that combines a hard X-ray magnetic tomography with a three-dimensional SEM reconstruction has made it possible to observe the three-dimensional microstructures and magnetic domain [2]. However, the feature extraction and matching in these are not easy by means of classical empirical analyses based on human recognition. In this study, to overcome this issue, we attempted to extract data-driven features from the three-dimensional microstructure and magnetic domains of a Nd-Fe-B sintered magnet.

We employed the datasets of three-dimensional microstructure and magnetic domains of a Tb diffused Nd-Fe-B sintered magnet [2], which are the magnetic domain of the residual magnetization state by varying the magnetic field from +5.5 T to -5.5 T and the microstructure of reconstructed 3D-SEM images for the same volume. The measurement volume was 18 μ m × 10 μ m × 18 μ m (parallel to the c-axis).

The AKAZE algorithm [3] was employed to perform the feature extraction of microstructure. The extracted features mainly correspond to the interfaces between Nd2Fe14B phases and Nd-rich phases (Fig(a)), indicating that this process successfully extracts the Nd-rich phase. After this, the feature matchings were performed for among the different parts of microstructures. For the magnetic domains, automatically segmented three-dimensional magnetic domains along the magnetic hysteresis were plotted (Fig(b)). From these processes, we are trying to find features of the microstructure correlated to the magnetic domains.



- a. The feature extraction and feature matching of the microstructure in the Nd-Fe-B magnet.
 - b. The reversal magnetic domain mapping from -2.2 T to -2.7 T in the Nd-Fe-N magnet.

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Insights into Hot-deformed Anisotropic Nd₂Fe₁₄B Magnets: Interaction Domains Studied by Non-destructive 3D Magnetic Laminography and Magnetometry

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13. Advanced Characterisation, September 7, 2023

We demonstrate that magnetic laminography can be used to determine the 3D magnetic structure of permanent magnetic material exhibiting interaction domains to correlate the crystal and magnetic structure. For this purpose, the 3D magnetic and electronic structure of a hot-deformed anisotropic nanocrystalline Nd₂Fe₁₄B magnet is evaluated by the recently developed x-ray-based technique so-called magnetic ptychographic laminography. The technique used is non-destructive and element-specific. We evaluate the 3D interaction domain propagation through the specimen of a disc-shaped sample size ca. 5 µm thickness and 8 µm diameter in a macroscopic demagnetized state. We confirm the surface magnetic moment configuration determined by previous studies with techniques like Kerr or MFM and reveal the novel complex domain structure in deeper sections of the permanent magnet, which show no signs of isolated domains. We could quantify the interaction domain widths in these highly anisotropic samples to be around 0.9 µm, which is about 2-3 times the grain size. Furthermore, the texture and macroscopic magnetic behavior were characterized to compare and correlate them to the static 3D magnetic microstructure.



Low Oxygen Sm₂Fe₁₇N₃ Sintered Magnets Produced from Ball-milled Powder

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14. Rare Earth Nitride Magnets, September 7, 2023

Sm₂Fe₁₇N₂ compound is known to be an excellent candidate as one of the post-Nd-Fe-B magnets due to its high intrinsic magnetic properties [1, 2]. However, the thermal decomposition temperature of this compound is 620 °C, making it difficult to produce high density magnets [3]. In our research group, we combined pressure current sintering (PCS) and cemented carbide, and successfully produced high density sintered magnets [4-8]. Another problem however occurred wherein the improved coercivity of the pulverized powder severely degraded upon sintering at even below the decomposition temperature range [5, 9]. In our group, we considered this degradation is due to the formation of α -Fe due to the reduction of Fe oxides on the powder particle surfaces during the sintering processes [9]. To avoid this redox issue, we developed what is called low-oxygen powder metallurgical system wherein all the powder metallurgical processes (i.e. pulverization, magnetic alignment and PCS) are carried out inside glove-boxes filled with purified inert gas [2, 10]. By this approach, we successfully produced high coercivity sintered magnets without coercivity degradation for the first time ever [2, 10]. However, our recent investigations revealed that the magnetizations and squareness of these Sm₂Fe₁₇N₃ magnets are not excellent due to the necessarily introduced lattice defects introduced during pulverization processes using jet-milling (JM) [11-13]. In this work, we employed wet ball-milling (BM) as a mild pulverization method to minimize the density of lattice defects. It was clarified that the achievable particle size by BM is not as small as that of JM and thus the coercivity and the powder (BH)max cannot be improved as high as the powders produced by jet-milling. However, both types of sintered magnets produced from JM/BM powders exhibited similar coercivity and (BH)max. During the talk, the possible mechanism will be discussed based on the microstructural information.

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14. Rare Earth Nitride Magnets, September 7, 2023

The development of high-performance permanent magnets is essential to increase the efficiency of motors for the electric vehicles. Sm-Fe based permanent magnets such as $TbCu_7$ -type Sm-Fe-N [1], which is metastable phase, are suggested as next-generation magnets to surpass Nd-Fe-B magnet. However, in order to produce metastable TbCu7-type Sm-Fe-N powder alloy into a magnet, the following two techniques are necessary: (1) production of the raw material powder with a single crystal for crystal-oriented sintered magnet, (2) x>9 of SmFex (Fe rich).

Recently, our research group has developed a low-temperature reduction-diffusion (LTRD) process [2-4] and succeeded in synthesizing single-crystal metastable TbCu₇-type SmFex powder for the first time. On the other hand, the metastable SmFex powder synthesized by the current LTRD process has insufficient Fe as x~8.5. Therefore, the development of improved LTRD process that can synthesize metastable-phase SmFex powder with x>9 is necessary. For this development, firstly the synthesis mechanism of the current LTRD process should be clarified in terms of mass transfer and thermodynamics, and the possibility of synthesizing TbCu₇-type Fe-rich SmFex powder by the present LTRD process should be investigated.

In this study, we used a-Fe and SmCl₃ powders as precursors and LiCl-KCl molten salts as a solvent of Ca reductant. Following reactions are predicted during the LTRD process: 2xFe (s) + $2SmCl_3$ (s) + 3Ca (s) + LiCl-KCl (s) at R.T. $\rightarrow 2xFe$ (s) + $2SmCl_3$ (l) + 3Ca (dissolved in molten salts) + LiCl-KCl (l) at over $350 \text{ °C} \rightarrow 2xFe$ (s) + 2Sm (s) + $3CaCl_2$ (l) + LiCl-KCl (l) $\rightarrow 2SmFe_x$ + $3CaCl_2$ (l) + LiCl-KCl (l). In order to clarify the mechanism in terms of mass transfer and thermodynamics, the diffusion/phase change of each of elements for the sample in the process of synthesis was investigated by observation of electron microscopes in this study.

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Preparation by Gas Atomization of Elongated Powders α"-Fe₁₆N₂ for Anisotropic Magnets

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14. Rare Earth Nitride Magnets, September 7, 2023

Permanent magnets (PMs) based on rare-earth elements are essential components of modern technologies such as in electric vehicles, wind turbines or medical equipment. However, concerns about environmental degradation from rare-earth mining, cost and availability issues, have driven the development of rare-earth-free magnetic materials. The α'' -Fe₁₆N₂ compound with giant saturation magnetization contains widely available, inexpensive and completely non-polluting elements, making it a promising candidate for rare-earth-free magnets. There were several successful efforts to synthesize a α'' -Fe₁₆N₂ nanoparticles and powders in the past with high saturation magnetization [1-2]. However, the manufacture of bulk magnet with reasonable magnetic properties is still an obstacle that has not been overcome, primarily due to low coercivity. High coercivity can be achieved by either the materials intrinsic high magnetocrystalline anisotropy, fine particle/grain size, or shape anisotropy.

Here, we report the effect of particle size and shape on the morphology, structure, and magnetic properties of Fe-N powders prepared by gas-atomization of iron ingots in nitrogen atmosphere. By controlling the parameters involved in the gas atomization process, such as atomization pressure, the diameter of the ejection-nozzle, the angle between the gas nitrogen-jet and the molten iron-jet, and the diameter of the gas nozzle, we managed to control the size and shape of the particles. Thus, we found that: (i) the particle size decreases as the atomizing pressure increases and/or the diameter of the gas nozzle decreases, (ii) the particle shape changes from sphere to ellipsoid for an angle between the gas jet and the molten iron jet less than 45 degrees, (iii) the α "-Fe₁₆N₂ phase content of the powders increases with increasing breaking gas pressure. Fe-N particles prepared in an elongated shape and with a diameter of less than 60 µm have a high potential to be used for the production of high performance α "-Fe₁₆N₂ anisotropic magnets.

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14. Rare Earth Nitride Magnets, September 7, 2023

 $Sm_2Fe_{17}N_x$ is a promising hard magnetic intermetallic compound for high temperature applications in automotive sectors owing to its excellent intrinsic magnetic properties. However, consolidation of these powders into magnet remains as a major challenge through conventional sintering due to the decomposition of $Sm_2Fe_{17}N_x$. The present work reports the coercivity enhancement and the reason thereof achieved in Sm-Fe-N powders by annealing with Zn-Al eutectic powder mixture.

Sm₂Fe₁₇N_x powder (Magvalley China, isotropic Hc≈ 9kOe, particle size = 0.5-3µ) was mixed with Zn-5AI (wt. %) alloy prepared through melt spinning in different proportion (10,20 and 30 wt. %) by ball milling. Optimal annealing of the mixture in a temperature range between 400- 460 oC yielded a coercivity enhancement of 66% compared to original as received powder measured in the non-aligned condition. Detailed microstructural characterization has been carried out using SEM, TEM and 3D-APT techniques. The observed enhancement in coercivity could be attributed to effective reduction in the free-Fe by the formation of non-magnetic r-FeZn (Fe₃Zn₇ phase) and the formation of smooth coating of Sm-Fe-Zn-N phase observed from 3D-APT studies. Diffusion of Al into matrix of Sm-Fe-N grains determined from the 3dimensional distribution explains the observed Tc enhancement in the annealed sample.





Figure 1: a) DSC curve of Zn-Al alloy, b) demagnetization curve of Sm-Fe-N, plain Sm-Fe-N annealed and Zn-Al added sample, c) 3 dimensional elemental distribution map of different constituent elements, (d) Zinc rich regions in the elemental map (inset) delineated with 8 at.% isoconcentration surface and the representative proximity histogram obtained from the rectangular (red color) region with 0.1 nm bin width.

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