Curriculum Vitae

Dr. SANTOSH K. PAL

Materials Science and Engineering, Indian Institute of Technology - IIT Kanpur, India
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SUMMARY:

I am proactive, self-motivated, innovative individual with an impeccable track record of initiating research, establishing laboratory, managing research project, and teaching. My specialties are in the field of materials development, optimization and structure-property correlation through X-ray diffraction, electron microscopy, magnetometry, calorimetry. Additionally, I have acquired an extensive experience in micro-/nano-scale materials processing, ceramics and intermetallic compounds along with commanding knowledge of theory and experimental methods in alloy design, phase transformation, microstructure, magnetism, and thermodynamics.

EDUCATION

Aug. 2010 – Nov. 2015 **Ph.D.** Materials Science
Technical University Dresden, Germany
Thesis: Anisotropic hard-magnetic nanoparticles and nanoflakes obtained by surfactant-assisted ball milling
Supervisor: Prof. Dr. Ludwig Schultz

Jul. 2007 – Jul. 2009 **M. Tech.** Metallurgical Engineering and Materials Science
Indian Institute of Technology – IIT Bombay, India
Thesis: Shaping and functionalization of magnetic nanoparticles
Supervisor: Prof. Dr. Dhirendra Bahadur

Jul. 2004 – Jul. 2006 **M. Sc.** Physics
University of Allahabad, India

Jul. 2001 – Jul. 2004 **B. Sc.** Physics, Chemistry, Mathematics
ECC, University of Allahabad, India

RESEARCH EXPERIENCE

Dec. 2018 - present **Senior Project Scientist**
Indian Institute of Technology – IIT Kanpur, India www.iitk.ac.in

- Designing open-framework-based porous high ion-conducting $Na_{1+x}Zr_2Si_xP_{3-x}O_{12}$ ceramics and studying structure-property correlations.
- Synthesizing materials by sol-gel and solid-state reaction routes, performing crystallographic, microstructural and electrical characterization through XRD, SEM, TEM, EIS, CV techniques.
- Managing the research lab and supervising Ph.D. and master's degree student.

Dec. 2015 - Dec. 2017 **Postdoctoral Fellow**
Institute for Energy Technology - IFE, Kjeller, Norway www.ife.no
Project: **Development of magnetocaloric materials for magnetic cooling systems**

- Developed the project approach, initiated the research on magnetocaloric materials & systems. Established collaboration with 3 international groups and coordinated the project meetings.
- Optimized the synthesis parameters and established magneto-structural coupling in Mn-Co-Ge by tuning the magnetic and structure degrees of freedom through crystallographic distortion

and manipulating the magnetic spin states. Investigated the magnetic, structural and thermodynamic characteristics using SQUID, PPMS, XRD, DSC, SEM, EDX techniques.

- Achieved a first-order magneto-structural phase transition and a giant magnetocaloric effect with a maximum value of entropy change (~60 J/kg.K) in this series of materials. Additionally, for the first time, observed a competitive ferromagnetic and antiferromagnetic interaction leading to a spin-glass state and cluster-glass behavior in $MnCo_{1-x}Cu_xGe$ compounds. Extensively performed ac-susceptibility and magnetic relaxation measurements to study the glassy behavior.
- Produced 3 research articles and presented the results at international conferences.

May 2014 - Sep. 2015

Research Associate

Technical University Darmstadt, Germany www.tu-darmstadt.de

Project: **Rare-earth free permanent magnets** (EU project)

- Researched on the development of rare-earth-free permanent magnets by improving the magnetization of high magnetocrystalline anisotropy materials and by enhancing the anisotropy of high-moment materials through microstructure- and crystal-engineering.
- Established high-temperature processing routes of arc- and induction-melting followed by long-term homogenization to synthesize $(Co\text{-}Fe)_3B$ intermetallic compounds. For the first time, succeeded to stabilize the metastable high-magnetic-moment τ-Fe_3B phase at room temperature *via* minute substitution of Fe by Mo.
- Studied the phase formation and investigated the microstructural and magnetic properties using XRD, SEM, EDX, VSM, and SQUID, and determined the magnetic easy-axis of $(Co\text{-}Fe)_3B$ by adopting a combinatorial approach of XRD, SEM, EBSD and VSM techniques.
- Coordinated and executed various tasks of the project and published 3 research articles. Taught Materials Technology course and trained graduate students in the lab.

Oct. 2010 - Apr. 2014

Doctoral Research Assistant

Institute for Solid State and Materials Research – IFW Dresden, Germany www.ifw-dresden.de

Project: **Hard magnetic nanostructures – fabrication and characterization** (Siemens project)

- Researched on the processing and characteristics of hard-magnetic ($SmCo_5$ and $Nd_2Fe_{14}B$) nanostructures promising for the fabrication of future high-energy-density nanocomposite permanent magnets.
- Optimized different milling parameters (e.g. surfactant and solvent type and amount, milling energy) and fabricated nanoscale single-crystalline particles in a large amount (tens-of-grams).
- Studied the microstructural and magnetic properties of single-crystalline and textured-polycrystalline flakes and nanoparticles by XRD, SEM, TEM, EDX, VSM, and SQUID. Established the formation mechanism of the nanoflakes and nanoparticles by a thorough investigation at various processing steps by a combination of X-ray and electron diffraction and magnetometry techniques. Fabricated nanocomposite magnets by hot-compaction of a homogeneous mixture of ultrafine hard magnetic particles and Fe nanoparticles, and investigated the microstructural and magnetic properties.
- Published 4 research articles and presented the results at various international conferences.

Aug. 2009 - Sep. 2010

Senior Research Fellow

Indian Institute of Technology - IIT Delhi, India www.iitd.ac.in

Project: **Electrochemical- and Electrophoretic- Deposition of Anisotropic Nanostructures**

- Demonstrated the growth of Cu/Co(Cu) multilayer nanowires using anodic-alumina templates and studied microstructural, electronic and magnetic transport properties.
- Synthesized Co nanoparticles (~10 nm) by thermal decomposition of organometallic precursor and employed electrophoretic deposition method to deposit a thin film (~100 nm thick) of the Co nanoparticles.
- Studied the electronic and magnetic transport in multilayer structure and the influence of inter-particle magnetic interaction on the blocking temperature in Co 2d-nanostructures.

TEACHING EXPERIENCE

July 2018 - Dec 2018 **Integral University Lucknow, India**
Assistant Professor in Physics

- Prepared lectures and taught Solid-State Physics, Atomic and Molecular Physics, Materials Science, and Mechanics and Wave Motion at graduate and post-graduate level.
- Carried out regular assessments by conducting, class tests, tutorials and assignments.
- Instructed and trained students in Mechanics laboratory.

Oct. 2014 - Feb. 2015 **Technical University Darmstadt, Germany**
Substitute Lecturer for **Materials Technology** course

- Prepared and delivered lectures on Materials types and various processing techniques designed for post-graduate students.

Lab Instructor for **Thermal Conductivity Lab**

- Organized the lab and trained undergraduate students with thermal conductivity measurement. Instructed the students to write lab-reports and carried out the assessment of the reports.

Jan. 2008 - Jul. 2009 **Indian Institute of Technology - IIT Bombay, India**
Teaching assistant for lecture course **Phase Transformations**

- Assisted instructor in course planning and preparing lectures, and organized tutorials for around 100 graduate students. Prepared weekly quizzes to maintain the study habits of the students, conducted the final exam and evaluated the answer sheets.

Lab Assistant for lab **Metallography and Structural Characterization**

- Prepared experiments and organized the lab with 3 lab assistants. Instructed 50 students individually and in a group for using metallography equipment and characterization tools.

TECHNICAL SKILLS

Materials Processing Methods

Nanostructures: Colloidal methods, Sol-gel method, thermal decomposition, micro-emulsion, electrochemical-/electrophoretic-deposition, dry/surfactant-assisted ball milling, mechanical alloying

Bulk: Solid-state reaction, physical metallurgy, sintering, arc melting, induction melting, melt spinning, hot-compaction, Hydrogen decrepitation

Characterization Techniques

Diffractometers: X-ray and neutron diffractometers

Microscopes: Optical and electron microscopes (SEM, TEM, EBSD, SAED, EDX), MOKE

Magnetometers: VSM, SQUID, PPMS

Thermal analysis: DSC, DTA, TGA, Mass-spectrometer

Transport: Electrochemical impedance spectroscopy (EIS), Cyclic voltammetry (CV), four-probe method

Softwares: FullProf Suit, OriginLab, MATLAB, LabVIEW, Photoshop, C++, FORTRAN, Microsoft Office

Languages known: English (fluent), German (intermediate-B1), Norwegian (basic), Hindi (native)

AWARDS and RECOGNITIONS

- Selected for postdoctoral fellowship at Institute for Energy Technology – IFE, Norway.
- Received a travel grant for IEEE Magnetics Society summer school held in New Orleans, USA.
- Won the prestigious Siemens/DAAD Postgraduate fellowship for the Ph.D. study in Germany.
- Awarded with teaching assistantship at IIT Bombay, India.
- Secured **AIR 38** with 99.9 percentile in GATE 2007 in Physics.
- Qualified in national eligibility test (**NET**) for lectureship in Physics, UGC-CSIR, India.

LEADERSHIP AND ORGANIZATION ACTIVITIES

- **Established collaboration** with internationally leading research groups, and organized meetings and seminars.
- **Managed and executed** research activity including maintaining partnership between industry and university and writing external funding proposals.
- **Led a team** of 35 graduate students on an educational tour of a glass manufacturing industry.
- **Led a group** of 5 junior teaching assistants and developed and designed electronics laboratory experiments for undergraduate students.
- **Taught** Physics and Materials Science courses to bachelor and master's degree students**, trained and supervised** students and new employees on various lab instruments.
- **Delivered invited talks and presented** the scientific results in various international conferences.
- Attended specialized courses on **Project Management,** and **Management Effectiveness Skills**.
- Certificate course on Fundamentals of **Intellectual Property and Rights (IPR)**.

PROFESSIONAL ASSOCIATIONS

- Editorial Board Member: Asia Pacific Journal of Engineering Science and Technology (APJEST)
- Member of IEEE Magnetics Society
- Invited reviewer for various scientific journal, APL, JAP, JMMM, JALCOM, Scripta Materialia, J Power Sources, Materials Research Express

LIST of REFERENCES

Prof. Dr. Shobit Omar	**Prof. Dr. Ludwig Schultz**	**Prof. Dr. Geir Helgesen**
Materials Science and Engineering, IIT Kanpur Kanpur – 208016, U.P., India +91 512 679 7427 *somar@iitk.ac.in*	Scientific Coordinator of DRESDEN-concept e.V. Editor-in-Chief JALCOM Nöthnitzerstraße 43, 01069 Dresden, Germany +49-351 46343176 *ludwig.schultz@dresden-concept.de*	Deputy Head of the department Physics, IFE Kjeller Instituttveien 18 2007 Kjeller, Norway +47 453 93 241 *geir.helgesen@ife.no*

LIST OF PUBLICATIONS

Submitted for Publication

1. **Santosh K. Pal**, Ritobrata Saha, Gundugolanu Vijay Kumar, and Shobit Omar, Improved Microstructure and Conductivity of NASICON-type Sc/Yb-doped $Na_3Zr_2Si_2PO_{12}$ Using Cubic-ZrO_2 Precursors. (under review).
2. **Santosh. K. Pal**, C. Frommen, G. Helgesen, S. Kumar, B. C. Hauback, H. Fjellvåg, On the magnetostructural transformation and giant-magnetocaloric effect in MnCoGe by Cu substitution for Mn (under review)

Published Peer-reviewed Articles

3. **Santosh K. Pal**, C. Frommen, S. Kumar, B. C. Hauback, H. Fjellvåg, T. G. Woodcock, K. Nielsch, G. Helgesen "Comparative phase transformation and magnetocaloric effect study of Co and Mn substitution by Cu in MnCoGe compounds", Journal of Alloys and Compounds 775 (2019) 22-29.
   Impact factor = 4.18, doi: 10.1016/j.jallcom.2018.10.040
4. **Santosh K. Pal**, L. V. B. Diop, K. P. Skokov, O. Gutfleisch "Magnetic properties of Mo-stabilized bulk Fe_3B magnet", Scripta materialia 130 (2017) 234-237.
   Impact factor = 4.54, doi: 10.1016/j.scriptamat.2016.12.009
5. **Santosh K. Pal**, K. P. Skokov, T. Groeb, S. Ener, O. Gutfleisch "Properties of magnetically semi-hard $(Fe_xCo_{1-x})_3B$ compounds" Journal of Alloys and Compounds, 696 (2017) 543-547.
   Impact factor = 4.18, doi: 10.1016/j.jallcom.2016.11.226
6. **Santosh K. Pal**, L. Schultz and O. Gutfleisch "Structural and magnetic properties of heat-treated ultrafine single crystalline $Nd_2Fe_{14}B$ particles obtained by ball-milling of dynamic hydrogenation disproportionation desorption and recombination powder" Scripta Materialia, 78-79 (2014) 33-36.
   Impact factor = 4.54, doi: 10.1016/j.scriptamat.2014.01.024
7. **Santosh K. Pal**, K. Güth, T.G. Woodcock, L. Schultz and O. Gutfleisch "Properties of isolated single crystalline and textured polycrystalline nano/sub-micron $Nd_2Fe_{14}B$ particles obtained from milling of HDDR powder" Journal of Physics D: Applied Physics, 46 (2013) 375004-11.
   Impact factor = 2.83, doi: 10.1088/0022-3727/46/37/375004
8. **Santosh K. Pal**, L. Schultz and O. Gutfleisch, "Effect of milling parameters on $SmCo_5$ nanoflakes prepared by surfactant-assisted high energy ball milling" Journal of Applied Physics, 113 (2013) 013913-8.
   Impact factor = 2.33, doi: 10.1063/1.4773323
9. **Santosh K. Pal**, D. Bahadur, "Shape controlled synthesis of iron-cobalt alloy magnetic nanoparticles using soft template method" Materials Letters 64 (2010) 1127-29.
   Impact factor = 3.02 doi: 10.1016/j.matlet.2010.01.086

Conference Proceeding Papers

10. **Santosh K. Pal**, L.V. Diop, K. Skokov, S. Ener, O. Gutfleisch "Synthesis and magnetic properties of $(Fe,Co)_3B$ based semi-hard magnets", Magnetics Conference, IEEE Xplore Digital Library, 2017.
    doi:10.1109/INTMAG.2017.8007861

Conference Presentations

11. **Santosh K. Pal**, C. Frommen, G. Helgesen *et al.*, "*Complex magnetic behavior and spin glass effect observed in Cu doped MnCoGe compounds*", 62nd Annual Conference on Magnetism and Magnetic Materials, 6-10 Nov. 2017, Pittsburgh, USA. **ORAL**

12. **Santosh K. Pal**, C. Frommen, G. Helgesen, *et al.*, "*Comparative phase transformation and magnetocaloric effect study of Co and Mn substitution by Cu in MnCoGe*", 62nd Annual Conference on Magnetism and Magnetic Materials, 6-10 Nov. 2017, Pittsburgh, USA.
13. **Santosh K. Pal**, C. Frommen, G. Helgesen, *et al.*, "Structural and magnetic phase transformations and magnetocaloric effect of Cu substituted MnCoGe compounds", Danish Days on Caloric Materials and Devices 2-3 Oct. 2017, Technical University of Denmark, Risø Campus, Denmark.
14. C. Frommen, M. Kristiansen, **Santosh K. Pal**, *et al.*, "Magnetostructural transitions in Fe-substituted $Mn_{1-x}Fe_xNiGe$ and $MnNi_{1-x}Fe_xGe$ (x < 0.25) compounds", Danish Days on Caloric Materials and Devices 2-3 Oct. 2017, Technical University of Denmark, Risø Campus, Denmark.
15. **Santosh K. Pal**, C. Frommen, S. Kumar, *et al.*, "*Tuning the Magnetocaloric Effect in Mn-based Intermetallic by elemental substitution*" Electronic phenomena studied in the nordic countries, Max IV, Lund, Sweden. **ORAL**
16. **Santosh K. Pal**, C. Frommen, S. Kumar, G. Helgesen, B. C. Hauback, H. Fjellvåg, "*Magnetocaloric Effect in Mn-based Intermetallic Compounds*" 6th National meeting on inorganic and materials chemistry 2017, Gardermoen, Norway. **Invited ORAL presentation**
17. **Santosh K. Pal**, L.V.B. Diop, K.P. Skokov, *et al.*, "Synthesis and magnetic properties of $(Fe,Co)_3B$ based semi-hard magnets", INTERMAG 2017, Dublin, Ireland. **ORAL**
18. **Santosh K. Pal**, C. Frommen, S. Kumar, *et al.*, "*Magnetostructural transition and magnetocaloric effect in Mn-Co-Cu-Ge compounds*", THERMAG 2016, Turin, Italy.
19. **Santosh K. Pal**, T. Groeb, S. Ener, K *et al.*, "*Structural and magnetic properties of magnetically semi-hard $(Fe_xCo_{1-x})_3B$ compounds*" ICM 2015, Barcelona, Spain.
20. **Santosh K. Pal**, K.-H. Müller, L. Schultz and O. Gutfleisch, "*Spin reorientation temperature of ultrafine $Nd_2Fe_{14}B$ particles: influence of exchange-coupling*" ICM 2015, Barcelona, Spain.
21. **Santosh K. Pal**, L. Schultz and O. Gutfleisch, "*Study on annealing and hot-compaction of ultrafine $Nd_2Fe_{14}B$ particles obtained by wet/surfactant-assisted ball milling*" INTERMAG 2014, Dresden, Germany.
22. **Santosh K. Pal**, K. Güth, T.G. Woodcock, *et al.*,"*Preparation and properties of ultrafine single crystalline and textured polycrystalline $Nd_2Fe_{14}B$ particles*" JEMS 2013, Rhodes, Greece.
23. **Santosh K. Pal**, L. Schultz and O. Gutfleisch, "*Single grain and textured submicron particles of $Nd_2Fe_{14}B$ for high energy density nanocomposite magnets*", TMS 2013, San Antonio, TX, USA. **ORAL**
24. **Santosh K. Pal**, L. Schultz and O. Gutfleisch, "*Hard magnetic nanoparticles and flakes prepared by surfactant assisted high energy ball milling*", JEMS 2012, Perma, Italy. **ORAL**
25. **Santosh K. Pal**, S. Sawatzki, L. Schultz *et al.*, "*Influence of milling parameters on textured $Smco_5$ nano-flakes prepared by surfactant assisted high energy ball milling*", REPM 2012, Nagasaki, Japan. **ORAL**
26. O. Gutfleisch, T. G. Woodcock, K. Güth, J. Thielsch, **S. K. Pal**, "*Advance processing and microstructure of high performance permanent magnets*", TMS 2012, Orlando, USA.
27. **Santosh K. Pal**, L. Schultz and O. Gutfleisch, "*Effect of milling parameters on the preparation of textured SmCo5 nanoflakes by surfactant assisted high energy ball milling*", INTERMAG 2012, Vancouver, Canada. **ORAL**
28. **Santosh K. Pal**, J. Thielsch, L. Schultz *et al.*, "*Highly coercive and textured $SmCo_5$ nanoflakes prepared by surfactant assisted high energy ball milling*", Magnetism and Magnetic Materials (MMM) 2011, Scottsdale, AZ, USA.
29. **Santosh K. Pal**, J. Thielsch and O. Gutfleisch, "*$SmCo_5$ nanoflakes prepared by surfactant assisted high energy ball milling*", IEEE Magnetics Society Summer School 2011, New Orleans, LA, USA.
30. **Santosh K. Pal**, D. Bahadur, "*Synthesis and magnetic properties of FeCo alloy nanostructures*" ICONSAT 2010, IIT Bombay, Powai, Mumbai, India.

Journal of Alloys and Compounds 775 (2019) 22–29

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Comparative phase transformation and magnetocaloric effect study of Co and Mn substitution by Cu in MnCoGe compounds

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[a] Physics Department, Institute for Energy Technology (IFE), P.O. Box 40, 2027 Kjeller, Norway
[b] Nanostructures and Functional Materials (NAFUMA), Department of Chemistry, University of Oslo, Sem Sælands vei 26, 0371 Oslo, Norway
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ARTICLE INFO

Article history:
Received 29 June 2018
Received in revised form
4 October 2018
Accepted 6 October 2018
Available online 8 October 2018

Keywords:
Magnetocaloric effect
Magnetic entropy
Magnetostructural coupling
Magnetic phase diagram
Martensitic transition
Mn-Co-Ge

ABSTRACT

Structural and magnetic phase transformations and magnetocaloric effect of Mn and Co substitutions by Cu in MnCoGe have been investigated using X-ray diffraction, differential scanning calorimetry, and magnetization measurements. Increase in Cu concentration reduces the martensitic structural and magnetic phase transition temperatures. However, nearly doubling of the amount of Co substitution is required compared to Mn for an equivalent change in the structural transition temperature. A giant magnetocaloric effect, $-\Delta S_M^{max} \approx 50\,J\,kg^{-1}K^{-1}$ for $\Delta\mu_0H = 5\,T$, resulting from coupling of concomitant structural and magnetic transformations near room temperature has been obtained for a sample with around 11 at% Mn-substitution. Fine tuning of Cu concentration (20 at%) in the case of Co substitution resulted in concurrent structural and magnetic transitions at around 260 K. However, the absence of a magnetostructural coupling resulted in peak entropy change of less than $4\,J\,kg^{-1}K^{-1}$. Samples with 15 at% or higher Co-substitution showed complex magnetic behavior and multiple magnetic transitions. The nature of magnetic phase transitions in both Co- and Mn-substituted samples have been investigated and phase diagrams for both sets of samples have been derived based on calorimetry and magnetometry results.

1. Introduction

Recently, MM'X (M, M′ − transition metals and X − p-block elements) type compounds have attracted considerable research interest because of their magneto- and thermo-responsive properties [1,2]. In particular, magnetocaloric effect (MCE), a thermal response of a magnetic material upon application or removal of a magnetic field, is of significant importance for its potential application in environmental friendly and energy efficient magnetic cooling technology. Materials going through a first-order magnetic phase transition are known to produce the highest MCE; as such systems possess different crystal structures on either side of the magnetic transition and exhibit a large magnetization difference around the transition temperature. Equiatomic MnCoGe is an important member of MM'X family with both structural and magnetic phase transformations. The martensitic structural transition is reported to take place from high temperature Ni_2In-type hexagonal (*P*6_3/*mmc*, #194) austenite to low temperature TiNiSi-type orthorhombic (*Pnma*, #62) martensite at around 500 K, and both hexagonal and orthorhombic structures order ferromagnetically at Curie temperatures (T_C) of 275 and 355 K, respectively [3–5]. Additionally, saturation magnetization of the martensitic phase ($M_s = 3.86\ \mu_B/f.u.$) is slightly higher as compared to that of the austenite phase ($M_s = 2.58\ \mu_B/f.u.$), which is also indicative of a magnetic field-induced martensitic phase transition [6]. In pristine MnCoGe, the martensitic transformation takes place in the paramagnetic (PM) austenite state, but it is possible to reduce the martensitic transition temperature (T_{str}) to be close to the T_C of the martensitic phase. In such case, the material would transform from the low moment austenite to the high moment martensite phase, leading to a large change in the magnetization accompanied by a first-order transition, which is basically the desired condition for a large change in the magnetic entropy and thus a giant MCE.

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https://doi.org/10.1016/j.jallcom.2018.10.040
0925-8388/

Scripta Materialia 130 (2017) 234–237

Contents lists available at ScienceDirect

Scripta Materialia

journal homepage: www.elsevier.com/locate/scriptamat

Regular article

Magnetic properties of Mo-stabilized bulk Fe_3B magnet

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ARTICLE INFO

Article history:
Received 29 November 2016
Accepted 8 December 2016
Available online xxxx

Keywords:
Iron alloys
Borides
Suction casting
Hard magnets
Magnetic anisotropy

ABSTRACT

Fe_3B is a metastable high temperature phase and exists in orthorhombic and tetragonal structures. Here, we report on synthesis, structural and magnetic properties of tetragonal ($P4_2/n$) Fe_3B stabilized by a very small substitution of Mo (1–3 at.%) for Fe. The $(Fe_{0.98}Mo_{0.02})_3B$ compound possesses a high Curie temperature of 780 K and high saturation magnetization of 175 $A{\cdot}m^2/kg$ (1.60 T). Magnetocrystalline anisotropy field of around 0.8 T and anisotropy energy of 340 kJ/m^3 have been determined for magnetically-oriented fine particles of $(Fe_{0.98}Mo_{0.02})_3B$.

Permanent magnets are essential components used in a range of energy efficient devices for applications in energy, conversion, data storage and magnetic cooling [1,2]. Performance of a permanent magnet is characterized by the maximum energy product and the operating temperature range. $Nd_2Fe_{14}B$ and $SmCo_5$ are considered as high performance permanent magnets, however these materials utilize significant amounts of expensive and sometimes scarce rare-earth (RE) elements. Intensive research on the search of new types of permanent magnets possessing reasonably good magnetic properties but employing reduced or no use of the RE–elements is in progress worldwide [3,4]. This type of magnet could close the huge gap in performance between low cost ferrite and high energy density NdFeB. There are two ways to achieve this objective. First, by fabricating two phase (magnetically-hard and magnetically-soft/-semi-hard phases) nanocomposite permanent magnets and second, by improving the magnetocrystalline anisotropy of high magnetic moment (magnetically-soft) materials [3–8].

Fe_3B is an interesting system because of its large saturation magnetization and reasonably strong magnetic anisotropy. It has a potential to be used as magnetic soft/semi-hard phase for the fabrication of the two phase nanocomposite permanent magnets [3,9]. However, Fe_3B is a metastable high temperature phase and has been reported to be present along with $Nd_2Fe_{14}B$ in melt spun ribbons of approximate composition $Nd_4Fe_{78}B_{18}$. The Fe_3B compound is reported to exist in orthorhombic (*Pnma*) and tetragonal ($P4_2/n$ and *I*-4) structures at high temperature and decomposes into Fe_2B and Fe below 1420 K [10, 11]. The tetragonal phase is stable in only very narrow temperature interval of 1420–1450 K, whereas the orthorhombic modification exists only between 1450 and 1480 K [12–14]. Coene *et al.* [11] has prepared the Fe_3B compound by melt spinning of $Fe_{76}B_{24}$ followed by annealing at 470 °C, however the compound was not a single phase. Around 20% of orthorhombic Fe_3B and 3% of α-Fe were detected along with tetragonal Fe_3B main phase. Coene *et al.* has also investigated the magnetic properties of these two phase ribbons by means of Lorentz transmission electron microscopy and magnetization measurements. Their studies revealed an easy-plane magnetocrystalline anisotropy, with [110] axis being the easy magnetization direction, and an anisotropy field of 0.5 T for tetragonal Fe_3B phase [11]. To the best of our knowledge there are no reports on the single phase Fe_3B system. In this work, we report on stabilizing Fe_3B compound in tetragonal symmetry ($P4_2/n$) with very small substitution of Fe by Mo (~2 at.%) atoms, and discuss the magnetic properties of the compound.

$(Fe_{1-x}Mo_x)_3B$ ($x = 0.0$–0.1) samples were prepared by melting high-purity elements (Fe-99.99%, Mo-99.99%, B-99.999%) in an arc furnace under a purified argon gas atmosphere. The resulting ingots were suction-casted from melt into thin rectangular ingots of thickness 0.5 mm. Suction-casting is a method of rapid solidification used to obtain a homogeneous elemental distribution in the solid. The ingots were then homogenized by heat treating under vacuum at 1050 °C for two weeks and then subsequently quenched in water. The ingots were cut and polished for optical microscopy and scanning electron microscopy (SEM). The phase purity and the elemental composition were checked with optical microscopy, SEM in back scattered electron (BSE) contrast and energy-dispersive X-ray spectroscopy (EDX) analysis. The content of B was assumed to be constant at 25 at.%, as it is not detectable by the EDX. The ingots (after removing the outer layer) were grounded down to fine particles of size less than 20 μm. X-ray diffraction (XRD) was carried out on a Stoe Stadti P powder diffractometer in transmission

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http://dx.doi.org/10.1016/j.scriptamat.2016.12.009
1359-6462/

Journal of Alloys and Compounds 696 (2017) 543–547

Contents lists available at ScienceDirect

Journal of Alloys and Compounds

journal homepage: http://www.elsevier.com/locate/jalcom

Properties of magnetically semi-hard $(Fe_xCo_{1-x})_3B$ compounds

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ARTICLE INFO

Article history:
Received 30 June 2016
Received in revised form
14 November 2016
Accepted 16 November 2016
Available online 17 November 2016

Keywords:
Rare-earth free permanent magnets
Semi-hard magnets
Magnetocrystalline anisotropy
Anisotropy field
FeCoB
Co_3B

ABSTRACT

The $(Fe_xCo_{1-x})_3B$ (x = 0.0, 0.1, 0.2, 0.3) compounds have been prepared by induction melting with subsequent annealing. All of the compounds form in the orthorhombic Fe_3C-type structure and exhibit ferromagnetic behaviour: the Curie temperature increases from 750 K for x = 0.0 to 923 K for x = 0.3. Magnetic domain images obtained by Kerr microscopy reveal the presence of uniaxial magnetocrystalline anisotropy. The magnetocrystalline anisotropy energy and anisotropy field of magnetically oriented fine particles have been determined. An anisotropy energy of 651 kJm^{-3} was obtained for Co_3B (x = 0.0), the anisotropy energy was found to decrease with increasing Fe content reaching a value of 507 kJm^{-3} for x = 0.3. These compounds with uniaxial magnetocrystalline anisotropy are potential candidates to be used as semi-hard magnetic phase for the preparation of rare-earth free nanocomposite permanent magnets.

1. Introduction

There has been some efforts in fabricating nanocomposite exchange-spring magnets by combining high magnetization (soft) phases, such as Fe and Fe–Co and high magnetic anisotropy (hard) phases, in order to improve the energy product $(BH)_{max}$ value [1–5]. However, the soft phases negatively affect the coercivity. It is expected that, apart from the magnetization improvement, the coercivity of nanocomposite permanent magnets would be improved by the replacement of the soft magnetic phase by a semi-hard magnetic phase [6]. According to Skomski et al. [7,8], the nucleation field in nanocomposite magnets is closely related to the overall anisotropy profile which indicates that the coercivity of composite magnets can be enhanced by the use of semi-hard phase because the magnetocrystalline anisotropy of this phase would make a positive contribution to the average anisotropy of the composite.

Mn-based compounds (e.g. Mn-Al, Mn-Bi, Mn-Ga) with reasonably good magnetocrystalline anisotropy are interesting materials for rare-earth free permanent magnets [9]. However, these materials possess a saturation magnetization far below 1 T. There is room to improve the saturation magnetization while retaining the anisotropy by combining these materials with high magnetization semi-hard magnetic materials. $(Fe_{1-x}Co_x)_2B$ compounds of tetragonal structure and an easy-axis [001] anisotropy with a magnetocrystalline anisotropy field of around 1 T, are good semi-hard magnetic materials [10,11]. However, the saturation magnetization is quite low as compared to Fe and Fe-Co. Therefore, there is a need to search for a better semi-hard magnet with higher saturation magnetization for which Fe_3B, Co_3B and $(Fe_xCo_{1-x})_3B$ are potential candidates. Fe_3B is a metastable high temperature phase which exists in both tetragonal and orthorhombic phases [12,13], and is reported to possess unfavorable easy-plane anisotropy. There are some reports on the structural and magnetic properties of Co_3B compounds [14–18], however it has relatively low saturation magnetization and information about the easy-axis for this compound has not been reported in the literature. It would be very beneficial to increase the magnetization of Co_3B compound by substitution of Fe for Co. Also, there is a lack of reports on the intrinsic magnetic properties such as magnetocrystalline anisotropy energies, anisotropy fields, easy-directions of $(Fe_xCo_{1-x})_3B$ compounds. Here, we present a systematic study of structural and magnetic properties on the $(Fe_xCo_{1-x})_3B$ compounds.

2. Experimental

$(Fe_xCo_{1-x})_3B$ (x = 0.0, 0.1, 0.2, 0.3) alloys were prepared by induction melting of Co, Fe (both 99.99% pure) and B (99.999% pure)

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http://dx.doi.org/10.1016/j.jallcom.2016.11.226
0925-8388/

J. Phys. D: Appl. Phys. **46** (2013) 375004 (8pp) doi:10.1088/0022-3727/46/37/375004

Properties of isolated single crystalline and textured polycrystalline nano/sub-micrometre $Nd_2Fe_{14}B$ particles obtained from milling of HDDR powder

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Received 25 March 2013, in final form 1 August 2013
Published 30 August 2013
Online at stacks.iop.org/JPhysD/46/375004

Abstract
Textured, polycrystalline $Nd_2Fe_{14}B$ powders, produced by dynamic hydrogenation disproportionation desorption and recombination (d-HDDR) were further processed by wet and surfactant-assisted ball milling. After 4 h of milling at 400 rpm in absolute ethanol and heptane + oleic acid, the polycrystalline d-HDDR particles had disintegrated, via intergranular fracture, into the individual grains i.e. isolated single crystalline particles of size 200 to 500 nm. An excellent degree of alignment was produced in the single crystalline particles using an applied magnetic field. This was reflected in the remanence of the field-aligned single crystalline powder (148.1 emu g^{-1}) which was far higher than that of field-aligned un-milled d-HDDR powder (119.5 emu g^{-1}). Milling the single crystalline powder further at 800 rpm in the same media produced polycrystalline flakes of size 0.2 to 1.0 μm. The polycrystalline flakes showed $(0\,0\,l)$ in-plane texture and thus oriented edge to edge in an applied field.

(Some figures may appear in colour only in the online journal)

S Online supplementary data available from stacks.iop.org/JPhysD/46/375004/mmedia

1. Introduction

There has been impressive progress in permanent magnets for most of the 20th century through discovery of new materials and improvement in their energy density value. This has led to a range of applications for high-energy density permanent magnets, e.g. energy conversion devices (motors, loudspeakers, and electric generators and alternators), household appliances and cell phones and recent environmental friendly technologies such as hybrid electric vehicles and wind turbines [1–4]. Exchange-coupled nano-composite magnets made of a fine mixture of magnetic hard and soft nanoparticles with well-controlled compositions and interfaces are potentially the future generation high-energy density magnets [5–7]. The synthesis of hard magnetic nanoparticles with precise control over the size, size distribution and phase purity is challenging. Chemical solution method, the most versatile way of controlling size and shape of nanoparticles is well studied for the preparation of FePt hard magnetic nanoparticles [8–10]. But because of complex crystal structure and high reactivity of rare-earth elements, the chemical solution method is not an efficient route for the preparation of rare-earth transition metal (RETM) nanoparticles. In recent years, surfactant-assisted ball milling (SABM) has proven to be an effective technique to prepare RETM hard magnetic nanoparticles and flakes by milling in the presence of surfactant and solvent [11–13]. Conventionally prepared arc-melted and homogenized $Nd_2Fe_{14}B$ powders with grain sizes of tens of micrometre have been used to prepare $Nd_2Fe_{14}B$ nanoparticles and flakes using SABM. Well-structured single crystalline and textured polycrystalline flakes and nanoparticles of $Nd_2Fe_{14}B$ have been obtained by this

JOURNAL OF APPLIED PHYSICS **113**, 013913 (2013)

Effect of milling parameters on $SmCo_5$ nanoflakes prepared by surfactant-assisted high energy ball milling

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(Received 10 October 2012; accepted 4 December 2012; published online 7 January 2013)

In this study, we discuss the effect of different milling parameters, such as the type and concentration of surfactants, milling energy, and milling time on the structural, morphological and magnetic properties of hard magnetic $SmCo_5$ nanoflakes prepared by surfactant assisted high energy ball milling. Two kinds of surfactants, polyvinylpyrolidone (PVP) with ethanol and oleic acid (OA) with n-heptane, were used as milling media. Increase in surfactants concentration and decrease in milling energy result in the decrease of degree of amorphization and reduction in grain size with milling time. Milling at 200 rpm results in more homogeneous and thicker flakes with fewer fractions of nanoparticles as compared to milling at 800 rpm. Increase in surfactants concentration results in the increase of the aspect ratio of flakes. Due to better capping ability of OA, the degree of flaking is higher when milling in OA than that in case of PVP. A maximum coercivity of 2.3 T was obtained after milling for 1.0 and 2.0 h for 10 and 50 wt. % of OA, respectively, at 800 rpm. A maximum $(BH)_{max}$ of 23.8 MGOe (188.9 kJ m^{-3}) and degree of texture of 93% were obtained for 10 wt. % OA after 10 h of milling at 200 rpm. The pronounced anisotropy and high coercivity of the nanoflakes should prove advantageous for the preparation of textured exchange spring magnets. © *2013 American Institute of Physics*. [http://dx.doi.org/10.1063/1.4773323]

I. INTRODUCTION

Recently, there has been a great scientific interest in the preparation of textured nanocomposite hard magnets as the development of the magnetic energy product ($(BH)_{max}$) of permanent magnets has somewhat stagnated since the discovery of rare earth transition metal (RE-TM) materials (SmCo and NdFeB). In the last couple of years, the prices of rare earth elements used in hard magnets have soared dramatically.[1] To reduce the use of rare earth elements and satisfy the growing demand for higher $(BH)_{max}$, the concept of textured exchange-spring type magnets has been proposed.[2–6] The basic principle in the nanocomposite hard magnets is to exchange couple a nanoscale textured hard magnetic phase having high coercivity with a nanosized soft phase of very large magnetization.[7,8] In general, the processing of nanocomposite magnets is mainly based on melt spinning and mechanical alloying utilizing FePt, $Nd_2Fe_{14}B$, and $SmCo_5$ as the hard phase and Fe_3Pt, Fe_3B, Fe, and FeCo as the soft phase; however, these methods produce only isotropic materials.[9–16] There is a possibility of an alternate route to make nanocomposite permanent magnets by combining separately prepared nanostructured, textured hard, and nanosized soft phases.

Various top-down and bottom-up approaches have been developed to prepare nanoparticles of different materials. Chemical or solution methods (bottom-up), which are extensively used for the synthesis of nanoparticles, are not efficient methods for the preparation of rare-earth compounds because of the high reactivity with oxygen.[17] High energy ball milling (HEBM) (top-down) is a simple, inexpensive, and efficient method for preparation of nanocrystalline powder in bulk amount. There are a few reports on the preparation of anisotropic RE-TM flakes using surfactant assisted high energy ball milling (SA-HEBM), which involves milling of RE-TM powder in the presence of surfactant and solvent.[18–24] The use of surfactants during ball milling provides a capping layer over the particles which decreases inter-particle friction, reduces the cold welding between particles, and yields particles with high shape and crystalline anisotropy. RE-TM nanoparticles of spherical and elongated rod shapes with sizes ranging from 5 to 100 nm have been prepared by SA-HEBM.[25,26] Cui *et al.*[19] studied the evolution of structure and magnetic properties of anisotropic submicron $SmCo_5$ flakes prepared with high energy milling in heptane and oleic acid. The effect of flake thickness on magnetic properties of anisotropic $SmCo_5$ flakes was studied by Knutson *et al.*[27] who obtained a maximum coercivity of 2.1 T and $(BH)_{max}$ of 22 MGOe measured in easy direction of magnetically aligned samples.

To our knowledge, there is no systematic study on the effect of different milling parameters on preparation and properties of $SmCo_5$ flakes. In this context, we set out to study the effect of different milling conditions on the structural, morphological, texture, and magnetic properties of $SmCo_5$ flakes.

II. EXPERIMENTAL METHODS

$SmCo_5$ powder (2–40 μm) was purchased from Great Western Minerals Group Ltd. The as-obtained powder was milled in a high-energy planetary ball mill (Fritsch Pulverisette 7) in a protective argon gas atmosphere with a powder to ball ratio of 1:10 for milling times varying from 0.5 to

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