



# A workshop on Microstructure-properties of Magnetic Materials

October 27, 2008  
2nd Seminar Room, 1st Floor of the Main Building, Sengen, NIMS

*organized by*  
*International Center for Nanoarchitronics & Magnetic Materials Center*  
*National Institute for Materials Science*

*cosponsored by*  
*the JSPS Amorphous Nanomaterials 147 Committee*

- 9:00 - 9:10 Opening Remarks  
K. Hono, NIMS
- 9:10 - 9:40 Engineering the microstructure and properties of ultra-High density magnetic recording media  
D. E. Laughlin, Carnegie Mellon University, USA
- 9:40 - 10:00  $L_{10}$  FePt-C nanogranular perpendicular anisotropy films with narrow size distribution  
A. Perumal, NIMS
- 10:00 - 10:30 Exchange spring and graded magnets for high density magnetic storage  
T. Schrefl, University of Sheffield, UK
- 10:30 - 10:45 Break
- 10:45 - 11:10  $H_c$  control of FePt thin films and nano particles  
Y.K. Takahashi, NIMS
- 11:10 - 11:30 Micromagnetics of spin torque nano-oscillators  
G. Hrkac, University of Sheffield, UK
- 11:30 - 12:00 Current driven domain wall dynamics in magnetic nanowires  
M. Hayashi, NIMS
- 12:00 - 13:30 Lunch Break
- 13:30 - 13:50 Search of Heusler alloy half-metals by point contact Andreev reflection  
A. Rajanikanth, NIMS
- 13:50 - 14:10 Tunnel magnetoresistance in magnetic tunnel junctions with half-metallic  $Co_2FeAl_{0.5}Si_{0.5}$  Heusler alloy electrodes  
H. Sukegawa, NIMS
- 14:10 - 14:30 Current-perpendicular-to-plane giant magnetoresistance in spin-valve structures using Heusler alloy electrodes  
T. Furubayashi, NIMS
- 14:30 - 15:00 Spin dependent tunneling in nanoparticles  
S. Mitani, NIMS
- 15:00 - 15:20 Break
- 15:20 - 15:50 Novel functional magnetic materials based on magneto-structural transitions  
O. Gutfleisch, IFW Dresden, Germany
- 15:50 - 16:10 Microstructure-coercivity relations in Nd-Fe-B magnets studied on artificially developed Nd/Nd-Fe-B interfaces  
S. Hirosawa, Hitachi Metals
- 16:10 - 16:30 Multi-scale characterization of Nd-Fe-B permanent magnets  
T. Ohkubo, NIMS
- 16:30 - 16:50 Magnetization process in hard magnetic materials  
T. Shima, Tohoku Gakuin University
- 16:50 - 17:10 Interaction domains in high performance NdFeB thick films for application in micro-electro-mechanical systems (MEMS)  
T. Woodcock, IFW Dresden, Germany
- 17:10 - 17:30 Discussion
- 18:30 - Get-together party

## Abstract

9:10 - 9:40

### **Engineering the microstructure and properties of ultra-High density magnetic recording media**

**D. E. Laughlin**, Materials Science and Engineering and Electrical and Computer Engineering Departments, Data Storage Systems Center, Carnegie Mellon University, Pittsburgh, PA 15213 USA  
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The microstructure of current magnetic recording media will be reviewed including discussion on the magnetic grain size, shape, crystallographic texture, magnetic anisotropy, grain to grain isolation, magnetic surface anisotropy, switching field distribution and soft magnetic underlayers. What is learned from the current media will then be applied to the next generation of magnetic media which will almost certainly be FePt/oxide granular heat assisted magnetic recording media (HAMR). Our discussion will include the various challenges of FePt media including the ordering transformation, the stability of microstructure during annealing and the choice of underlayers which allow for rapid heat transfer.

9:40 - 10:00

### **L1<sub>0</sub> FePt–C nanogranular perpendicular anisotropy films with narrow size distribution**

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We report the study of L1<sub>0</sub> FePt–C nanogranular perpendicular anisotropy films fabricated on oxidized Si substrates with a (100) textured MgO intermediate layer. The addition of a small amount of C (<12%) to a FePt(4 nm) film results in the formation of interconnected FePt particles, while a higher C addition leads to the formation of well-isolated L1<sub>0</sub> FePt nanoparticles with a c-axis texture. The magnetic properties of the Carbon doped FePt films show good agreement with the microstructural change. The FePt particle size can be reduced to 5.5 nm with a size distribution of 2.3 nm in variance by adjusting FePt thickness. Perpendicular coercivity is controllable between 8 and 15 kOe with high squareness. These results demonstrate that the FePt-C system can accomplish a nanogranular structure suitable for ultrahigh density perpendicular recording media.

10:00 - 10:30

### **Exchange spring and graded magnets for high density magnetic storage**

**Thomas Schrefl**, Department of Engineering Materials, University of Sheffield, S1 3JD, UK  
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Tuning the coercive field by exchange coupling of multiple magnetic layers is the key to increase the storage density. Careful choice of the intrinsic magnetic properties makes it possible to decrease the switching field of storage media while keeping a sufficiently high thermal stability. Exchange spring magnets are well known in the permanent magnet community. In this talk I will give an overview of the basic physical mechanism that governs switching field, remanent magnetization, and energy barrier in exchange composite magnets. Design guidelines for nanocomposite permanent magnets will be reviewed and applied to find magnetic storage media. Magnetic recording simulations show that storage densities of 1 Tbit/in<sup>2</sup> can be achieved in granular media when the anisotropy constant increases gradually from top to bottom of the grain. Applied to bit patterned recording exchange spring media will enable storage densities of 8 Tbit/in<sup>2</sup>.

10:45 - 11:10

### **H<sub>c</sub> control of FePt thin films and nano particles**

**Y.K. Takahashi**, Magnetic Materials Center, National Institute for Materials Science, Tsukuba 305-0047, Japan  
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H<sub>c</sub> control is important for the magnetic thin film devices. In the magnetic recording media, nano-size ferromagnetic particles with high thermal stability are required to realize high areal recording density. L1<sub>0</sub>-FePt has been received much attention due to its high magnetocrystalline anisotropy. However,

L10-FePt nano-particle shows very high  $H_c$  when its size becomes nano order. To use it as recording media, the reduction of  $H_c$  with maintaining the thermal stability is required. To control the  $H_c$  of nano-particles, we should control the microstructure. In this talk, I present  $H_c$  control of FePt thin film and nano particle by controlling their microstructure.

11:10 - 11:30

**Micromagnetics of spin torque nano-oscillators**

**Gino Hrkac**, Department of Engineering Materials, University of Sheffield, S1 3JD, UK  
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Spin transfer effects in magnetic nanometre scale devices is an active field of research and can lead to high density, low power magnetic random access memory or direct current driven nanometre microwave oscillators. It originates from the interaction of the electrons' spin angular momentum with a spin polarized current which exerts a torque on the ferromagnet of the device. Experiments of this effect have shown either reversal of the ferromagnetic magnetization or high frequency steady state precession. We combine quasi static Maxwell, Landau Lifshitz Gilbert and inhomogeneous current theory with finite element and boundary element methods to simulate realistic nano pillars, point contact and MTJ devices. It is shown that the oscillation frequency is a function of applied current and applied field strength and orientation. The simulations can predict effects like spin wave propagation, phase-locking of RF sources, thermal excitations and the generation and dynamics of vortex structures.

11:30 - 12:00

**Current driven domain wall dynamics in magnetic nanowires**

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A comprehensive understanding and control of the magnetization dynamics in magnetic nanostructures is key to the development of novel memory and logic devices. New paradigms for domain wall-based devices are made possible by the direct manipulation of domain walls using spin polarized electrical current via spin transfer torque. We have studied the field and current driven dynamics of domain walls in magnetic nanowires in pursuit of magnetic racetrack memory[1]. Controlled motion of a series of domain walls along magnetic nanowires using current pulses in permalloy nanowires are shown[2]. Fascinating effects arising from the interplay between domain walls with spin polarized current will be discussed.

[1] S. S. P. Parkin et al., Science 320, 190 (2008).

13:30 - 13:50

**Search of Heusler alloy half-metals by point contact Andreev reflection**

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$X_2YZ$  type of ternary Heusler alloys, where  $X$  is a high valent transition metal atom,  $Y$  is a low valent transition metal atom, and  $Z$  is a  $sp$  atom, are predicted to be half-metallic. Half-metallic materials are those materials whose spin up density of states are metallic and spin down density of states are semiconducting with a band gap around the Fermi level. Their properties can be tuned by quaternary element additions. This method has been shown to be promising in enhancing the properties of Heusler alloys. However some of the quaternary Heusler alloys form two phase instead of a single phase due to thermodynamics. We will discuss that; such a two phase microstructure can also be helpful in tuning the Fermi level.

13:50 - 14:10

**Tunnel magnetoresistance in magnetic tunnel junctions with half-metallic  $Co_2FeAl_{0.5}Si_{0.5}$  Heusler alloy electrodes**

**Hiroaki Sukegawa**, Magnetic Materials Center, National Institute for Materials Science, Tsukuba 305-0047, Japan  
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Half-metals, which have 100% spin-polarized electrons at  $E_F$ , are ideal materials for spintronic devices,

such as spin MOSFETs. A group of Co-based full-Heusler alloys with Co<sub>2</sub>XY type L21 structure is the most promising candidate due to their high Curie temperature (around 1,000 K), which is expected to realize room temperature half-metallic operation. In this study we fabricated magnetic tunnel junctions with the structures of sputtered full-epitaxial L21-CFAS/MgO/L21-CFAS and L21-CFAS/MgO/B2 (disordered structure)-CFAS on MgO-buffered MgO (001) substrates using a magnetron sputtering. High TMR ratio of 312% was observed in L21-CFAS/MgO/L21-CFAS at low temperature. Additionally, the evidence of half-metallicity in L21-CFAS films was found by measuring the bias voltage dependence of tunneling conductances for these MTJs, which is dramatically changed by the crystalline structure of CFAS films. The conductance of these structures is analytically explained with calculated half-metallic band structure of L21-CFAS from first principle calculations. Microstructure-coercivity relations in Nd-Fe-B magnets studied on artificially developed Nd/Nd-Fe-B interfaces

14:10 - 14:30

**Current-perpendicular-to-plane giant magnetoresistance in spin-valve structures using Heusler alloy electrodes**

**T. Furubayashi**, Magnetic Materials Center, National Institute for Materials Science, Tsukuba 305-0047, Japan  
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Current-perpendicular-to-plane giant magnetoresistance (CPP-GMR) read heads are expected in future high-density hard disc drives because of the intrinsically low resistance that is suitable for achieving high-speed read out. However, the MR ratio of CPP-GMR spin valves is still quite low and it is crucial to increase the MR ratio of the current CPP-GMR devices substantially for potential applications. Using highly spin-polarized Heusler alloys as ferromagnetic electrodes is considered to be promising for achieving a higher MR ratio. We prepared a CPP-GMR spin valve using epitaxial layers of Co<sub>2</sub>FeAl<sub>0.5</sub>Si<sub>0.5</sub> (CFAS) Heusler alloy as ferromagnetic electrodes. A multi-layer stack of Cr/Ag/CFAS/Ag/CFAS/Co<sub>75</sub>Fe<sub>25</sub>/Ir<sub>22</sub>Mn<sub>78</sub>/Ru was deposited on a MgO (001) single crystal substrate. MR characteristics and microstructures examined by TEM observations are reported.

14:30 - 15:00

**Spin dependent tunneling in nanoparticles**

**S. Mitani**, Magnetic Materials Center, National Institute for Materials Science, Tsukuba 305-0047, Japan  
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Spin dependent tunneling is a major topic in the research field of spintronics. In order to develop new functionalities, novel spin dependent tunneling phenomena in magnetic nanostructures have been intensively studied in recent years. Since the electric and electronic properties of nanoparticles depend significantly on their sizes, a variety of spin dependent tunneling phenomena are expected to appear in nanoparticles. I will show our recent experimental results on the growth process of magnetic and nonmagnetic nanoparticles on a monocrystalline oxide layer, and will discuss the sizes of nanoparticles and their stability. Spin dependent single electron tunneling and the effect of spin accumulation in single-nanometer-scale nanoparticles are also shown and discussed. Finally, a possible device application using nonmagnetic nanoparticles will be introduced.

15:20 - 15:50

**Novel functional magnetic materials based on magneto-structural transitions**

**O. Gutfleisch**, Institute for Metallic Materials, IFW Dresden, D - 01069 Dresden, Germany  
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Coupling effects between magnetic and structural degree of freedom have become one of the key features for the development of novel functional magnetic materials. New and enhanced giant magnetocaloric effects and magnetic shape memory (MSM) properties are a result of this interplay between order parameters coupling and phase transformations. The talk will be divided in two parts: Near room-temperature magnetic refrigeration based on the magnetocaloric effect (MCE) is becoming a promising technology to replace the conventional gas-compression/expansion technique. Several materials with a so-called giant MCE have been discovered recently, such as Gd<sub>5</sub>Si<sub>2</sub>Ge<sub>2</sub>, MnAs<sub>1-x</sub>Sbx, (Mn,Fe)<sub>2</sub>(P,As)- and La(Fe,Si)<sub>13</sub>-type. Common to all these materials is the occurrence

of a first-order magnetic phase transition near room temperature that gives rise to an abrupt change of the magnetization near the transition point. This, in turn, results in a large magnetic entropy change  $\Delta S_M$ . Advantages and disadvantages of the different alloy systems will be discussed. The talk then focuses on  $\text{LaFe}_{13-x}\text{Si}_x$  alloys. Among important advantages of this alloy system are the reduced thermal- and field-hysteresis in melt-spun ribbons, the abundance and non-toxicity of main constituents and the possibility of adjusting the Curie point – and thus operation temperature of the refrigerant - by varying  $x$  and/or by hydrogenation. The effect of substitutional and interstitial modification and preparation conditions on the thermally and field-induced first-order phase transitions and, consequently, on the magnetocaloric properties is reviewed. It is also pointed out that a peculiar electronic structure makes the  $\text{La}(\text{Fe},\text{Si})_{13}$ -type compounds distinct from other giant MCE materials exhibiting a first-order phase transition. In the second part, magnetic field driven phase transformations in  $\text{Ni}_2\text{MnGa}$  as the most prominent magnetic shape memory (MSM) alloy are elucidated. The idea is to move from single crystals to polycrystalline material and finally the preparation of textured MSM composites. The talk ends with some ideas on alternative MSM/MCE systems.

15:50 - 16:10

**Microstructure-coercivity relations in Nd-Fe-B magnets studied on artificially developed Nd/Nd-Fe-B interfaces**

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In order to study changes in the Nd-rich/ $\text{Nd}_2\text{Fe}_{14}\text{B}$  interface microstructure and to investigate their direct impact on coercivity in sintered Nd-Fe-B magnets, microstructures of Nd films deposited on the surface were investigated and related to coercivity of the surface  $\text{Nd}_2\text{Fe}_{14}\text{B}$  grains which could be measured independently of the coercivity of the bulk body of the magnet. It has been found that onset of a large coercivity of the surface grains is always associated with formation of an fcc Nd-oxide ( $a = 0.548 \text{ nm}$ ) in the Nd film adjacent to the interface, which was originally composed of metallic Nd with an dhcp structure. Another experiment has clearly shown that oxygen to form the fcc phase was provided from the residual gas in the vacuum annealing environment.

16:10 - 16:30

**Multi-scale characterization of Nd-Fe-B permanent magnets**

**T. Ohkubo**, Magnetic Materials Center, National Institute for Materials Science, Tsukuba 305-0047, Japan  
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Nd-Fe-B permanent magnets exhibit highest coercivity. However, the coercivity must be increased for high temperature application or downsized components with less rare earth element because of the limitation of cost and natural resource. In order to improve the performance of Nd-Fe-B permanent magnets, the additional elements and the process conditions must be optimized. Therefore, it is very important to know the relation between the properties and the microstructure. In the case of Nd-Fe-B sintered magnets, multi-scale analysis from micro to nano meter scale is required since the grain size is larger than few  $\mu\text{m}$ . So, the microstructure was analyzed by SEM/FIB, TEM and 3DAP techniques. Also, these complementary techniques were applied for HDDR processed Nd-Fe-B magnet which is anisotropic magnet and has possibility to achieve high coercivity because of the small grain size and the aligned microstructure. In this talk, we will show our recent results of the characterization and discuss about the relation with magnetic properties.

16:30 - 16:50

**Magnetization process in hard magnetic materials**

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Magnetization process in hard magnetic materials with a large magnetocrystalline anisotropy is conveniently classified as the nucleation-type and the pinning-type. For example, the former is typically observed for Nd-Fe-B magnets, and the latter, for Sm-Co magnets. However, in the both cases, a high magnetic field is necessary to saturate the magnetization completely for getting a high

coercivity ( $H_c$ ); in other words, generally it is not easy to fully wipe out reversed domains at a low magnetic field. Previously, we reported that high  $H_c$  exceeding 70 kOe was achieved in highly ordered FePt (001) films with island structure which were epitaxially grown on MgO (001) substrates. In this talk, the structure and the magnetization behavior of FePt films will be introduced in comparison with the observation of the domain structure, indicating that they are ideal nucleation-type nanomagnets. Furthermore, the latest results on Nd-Fe-B thin films will be partly introduced.

16:50 - 17:10

**Interaction domains in high performance NdFeB thick films for applications in micro-electro-mechanical systems (MEMS)**

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Thick sputtered films (5-300  $\mu\text{m}$ ) of NdFeB have excellent hard magnetic properties which make them attractive for applications in micro-electro-mechanical systems (MEMS). A two step process consisting of triode sputtering and high temperature annealing produced films with energy densities approaching those of sintered NdFeB magnets. Optimum magnetic properties were achieved for a substrate temperature of 500°C during deposition and an annealing temperature of 750°C for 10 min with a rapid heating rate. Magnetic force microscopy (MFM) using hard magnetic tips showed that the films deposited without substrate heating and at 300°C exhibited magnetic domains typical of low anisotropy materials. These films were reported to be amorphous in the as-deposited state. The film deposited at 500°C was reported to be crystalline and to display hard magnetic properties. This was reflected in the magnetic microstructure which showed interaction domains typical of highly textured and high magnetic anisotropy materials with a grain size below or equal to the critical single-domain particle limit. On annealing, the domain pattern for films processed without substrate heating changed reflecting crystallisation and corresponding magnetic hardening. With increasing substrate temperature, the domain patterns of the annealed films indicated higher degrees of texture. Similar interaction domains have also been observed in hot pressed and die upset NdFeB magnets and SmCo 2-17 type materials. The local texture of the annealed NdFeB films was investigated by electron backscatter diffraction (EBSD) in a FEG-SEM. EBSD analysis showed that although the sample deposited at 300°C had a large fraction of well oriented grains (*i.e.* those grains with  $<20^\circ$  misorientation from the ideal orientation: [001]/film normal), the sample at 500°C had a higher degree of texture. This was also shown by the misorientation distribution which was much sharper and had its maximum at a lower angle for the film deposited at 500°C. Higher texture leads to higher out-of-plane magnetic anisotropy and therefore to an improved energy product ( $BH_{\text{max}}$ ).

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