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13. ABSTRACT (Maximum 200 words)

This research is focused on (1) design of novel nanocomposite structures for high energy-product permanent magnets, (2) design of enhanced high-temperature Co-based alloys by compound-alloy modifications, and (3) development of enhanced hard and soft magnets via novel processing methods including mechanical alloying, cluster-assembled materials, and rolling techniques. Model nanocomposite hard-soft combinations have been developed with energy products of about 50 MGOe and coercivities of about 40 kOe. A theoretical understanding has been achieved of the structural characteristics needed to produce high energy products in the exchange-coupled nanocomposites. A variety of 2:17, 1:7, 5:17 and modified compounds have been investigated both experimentally and theoretically. It has been shown that transition-metal additions such as Ti, Cu, and Zr, are capable of stabilizing the disordered hexagonal Sm(CoM)₇ phase with beneficial increases in anisotropy. Considerable development work on processing methods has been performed in this period.

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1. List of Manuscripts

1. J.P. Liu, Y. Liu, R. Skomski, and D.J. Sellmyer, HIGH ENERGY PRODUCTS IN EXCHANGE-COUPLED NANOCOMPOSITE FILMS, IEEE Trans. Mag. 35, 3241 (1999).
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2. Scientific Personnel

Professors David J. Sellmyer, Sitaram S. Jaswal, Diandra Leslie-Pelecky, J. Ping Liu, Ralph Skomski, Renat Sabiryanov, Y. Liu, Jeffrey Shield; Drs. Hong Tang, You Qiang, I. Al-Omari, Lanping Yue, Tom Bui, E. Kirkpatrick; Ms. H. De Silva, Mr. Michael Leonard, Mr. J. Zhou.

3. Report of Inventions: None.

4. Scientific Progress and Accomplishments

A. ELECTRONIC STRUCTURE AND MAGNETIC PROPERTIES OF $\text{Nd}_5\text{Fe}_{17}$

R. F. Sabiryanov and S. S. Jaswal

$\text{Nd}_5\text{Fe}_{17}$ compound has attracted some attention lately as a possible permanent-magnet material. This compound has a very complicated crystal structure (264 atoms per unit cell with 14 and 7 different Fe and Nd sites respectively!). Using linear-muffin-tin-orbital method, self-consistent spin-polarized studies of $\text{Nd}_5\text{Fe}_{17}$ have been carried out to determine the electronic structure, magnetic moments, and the Curie temperature. The results show strong effect of the local environment on the magnetic properties of individual Fe sites. The

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calculated moments are compared with the magnetization data. Total exchange parameters show well defined correlation with the local volume. The Curie temperature estimated for the iron sublattice using the mean-field approximation is compared with the experimental result.

B. VOLUME DEPENDENCE OF MAGNETISM OF Y_2Fe_{17} COMPOUNDS

H. de Silva and S. S. Jaswal

The magnetic moment and exchange parameters were calculated for expanded and compressed volumes. As the volume is expanded further and further, the magnetic moment tends to saturate. The magnetic moment of the nitrogenated compound is close to that of the expanded 2-17 compound at the same volume. The calculated value of the compressibility for this compound is not in agreement with the available experimental results. However, it is in agreement with other theoretical calculations. Monte Carlo estimate of the Curie temperature is 490 K while the experimental value is 325 K.

C. MICROMAGNETISM OF EXCHANGE-COUPLED MAGNETS

R. Skomski

To explain the hysteresis loops and energy products of the materials investigated by Prof. J. P. Liu and to predict the behavior of structurally and chemically similar materials, model calculations have been made. Emphasis is on a basic understanding of the involved magnetization processes and on a theoretical guidance towards improved materials, as opposed to full-scale numerical simulations of small magnets. The calculations are based on an expansion of the magnetization in terms of differential operators which describe the interatomic exchange. The key problem is the incorporation of the anisotropy randomness associated with the presence of hard and soft phases and with the partial alignment of the hard crystallites. Going beyond earlier attempts (Chudnovsky *et al.*, 1986), approximate hysteresis loops have been obtained. Since the quality of the approximation is best in the region of the second quadrant responsible for the energy product, reliable energy-product estimates are obtained for nearly ideal magnets, that is for not too big soft regions and moderate grain misorientation. There is, in general, a comparatively narrow window where exchange coupling actually improves the energy product. The position of this window depends on parameters such as chemical composition, volume fraction of the soft phase, and temperature.

Theoretical efforts are being made to understand magnetic anisotropy and coercivity in the limit of high temperatures. Tentative statistical calculations underline the importance of intersublattice exchange interactions as the anisotropy-limiting factor and indicate that substitutions of heavy 5d or 5f atoms in suitable intermetallic compounds may improve the high-temperature anisotropy. An alternative, and practically more feasible, approach is to utilize highly anisotropic 3d materials such as Co 1:5 materials and to substitute heavy rare earths to improve the coercivity.

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D. MODEL NANOCOMPOSITE SYSTEMS WITH HIGH ENERGY PRODUCTS

J. Ping Liu, Yi Liu, D.J. Sellmyer

We have switched our focus to the Sm-Co system this year, after the successful work in the Fe-Pt and Pr-Co systems. We consider that the Sm-Co system is the best system so far for power applications requiring high Curie temperatures, and there are several phases in this system that can be used for bulk permanent magnets. Our goal is to determine the relationship between the structure and properties of the nanocomposite through the prototype of physically deposited samples, as we did for the previous systems.

We have investigated three compositions of the nanocomposite $\text{SmCo}_x\text{:Co}$, with $x=3.5, 4,$ and 4.5 (target composition). It was found that the highest coercivity can be obtained in the $\text{SmCo}_{3.5}\text{:Co}$ system. TEM observation has found that in the composite the soft phase is the hexagonal Co phase while the hard phase is the D019 SmCo_3 phase. Different from the FePt and PrCo systems, magnetic hardening can be realized in the as-deposited samples. We have investigated the effect of the multilayer configuration on the magnetic hardening. Systematic study has been performed on heat treatment, because a proper heat treatment results in amazingly high coercivity. Multi-step treatment has been applied to the samples and huge coercivity (up to 43 kOe) has been achieved. More importantly, the squareness of the loops was also improved with increasing coercivity. As a new finding in this year, results in this investigation suggest that grain boundary conditions have effect on the intergrain exchange coupling.

E. STABILIZED NOVEL TERNARY MAGNETIC COMPOUND WITH SmFe_7 TYPE STRUCTURE

H. Tang, J.P. Liu, D.J. Sellmyer

Recently, it was reported that a novel tetragonal-structure compound SmFe_7 has large spontaneous magnetization, a huge magnetocrystalline anisotropy (which is superior to that of $\text{Nd}_2\text{Fe}_{14}\text{B}$), and a Curie temperature ($T_C \sim 608$ K). This result suggests that the SmFe_7 compound appears to be a potential host material for a practical high performance hard magnet. However, the equilibrium single phase SmFe_7 polycrystalline is difficult to obtain and has never been reported up to now. In addition, regarding the permanent magnet application the Curie temperature (T_C) is still low. In the present study, we have synthesized stabilized SmFe_7 type ternary intermetallic compound by substitution of suitable amount of Ti.

As we know from the equilibrium phase diagram of Sm-Fe system, there are $\text{Sm}_2\text{Fe}_{17}$, SmFe_3 , and SmFe_2 equilibrium phases at lower temperature. The thermomagnetic analysis (TMA) and X-ray diffraction (XRD) results also show that in the composition range from 1:6 to 1:10, one cannot obtain any novel phase other than those mentioned above. The sample with nominal composition within that range consists of SmFe_2 and SmFe_{17} and $\alpha\text{-Fe}$. With a

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small amount of Ti substitution, however, a novel intermetallic phase with Curie temperature of around 610 K appears in samples with nominal composition $\text{Sm}(\text{Fe},\text{Ti})_z$ ($z = 6.0-7.0$). At this point, the novel intermetallic phase looks like the SmFe_7 -related tetragonal phase. The TMA results shows that the content of 2:17 and 1:2 phases have changed with Ti content; the content of $\text{Sm}(\text{Fe},\text{Ti})_7$ phase could be increased up to 85%, and the 2:17 phase almost disappears in the optimum sample. Further studies are needed in order to identify the structure of this phase. The formation of the novel phase SmFe_7 is sensitive to the processing conditions. The optimum processing conditions are necessary to obtain single-phase samples.

F. DEVELOPMENT OF ANISOTROPY IN $\text{Sm}(\text{CoTi})_7$ PHASES

J. Zhou, J.P. Liu, D.J. Sellmyer

The TbCu_7 -type disordered structure (1:7 type) exists in Sm-Co based compounds with composition both 1:7 and 2:17. A metallic element M is necessary to stabilize the $\text{Sm}(\text{CoM})_7$ compound. Ti with amount 0, 3%, 5%, 7% was substituted for Co. X-ray diffraction shows single phase $\text{Sm}(\text{CoTi})_7$ with TbCu_7 disordered structure at Ti amount around 3%-5%. $\text{Sm}_2(\text{CoTi})_{17}$ always has the desired 1:7 phase. Lattice parameters were determined.

The Curie temperature is measured with a high-temperature VSM. T_c drops with increasing Ti amount. Aligned samples were measured with field normal and parallel to the easy axis. Room temperature anisotropy field (H_a) is obtained by the method of extrapolation. H_a of $\text{Sm}(\text{CoTi})_7$ reaches as high as about 180 kOe, which is much higher than the ordinary rhombohedral $\text{Sm}_2\text{Co}_{17}$ (65 kOe), and also higher than the 1:7 type $\text{Sm}_2(\text{CoTi})_{17}$ (as high as 120 kOe). Saturation magnetization decreases with the increasing of doping Ti. Further work on magnetic properties is underway.

G. MAGNETIC PROPERTIES OF MODIFIED 2:17 AND 1:7 PHASES

I. Al-Omari, J. Zhou, D.J. Sellmyer

Samples of the form $\text{Nd}_2\text{Fe}_{17-x}\text{M}_x$ ($M=\text{Cu}, \text{Cr}, \text{V}, \text{Nb}, \text{and Zr}$), where $x=0, 0.5, 1, 1.5, 2, 2.5, 3, \text{ and } 4$ have been prepared and studied. X-ray diffraction show that these compounds form the rhombohedral $\text{Th}_2\text{Zn}_{17}$ structure with small amount of Fe for some of the samples with small x values. We find that for some of these compounds the rhombohedral structure cannot be formed at large values of x . X-ray diffraction on magnetically aligned samples show that these samples have in-plane anisotropy. The lattice parameters (a and c) are dependent on the composition of M . The unit cell volume is found to increase with x for $M=\text{V}, \text{Nb}, \text{ and Zr}$ and decrease with x for $M=\text{Cu}$ and Cr . The saturation magnetization for all the compounds studied decreases with x for all M atoms and both at room temperature and at a temperature of 25 k. Curie temperature for these compounds increases with x for small values of x and it decreases for large values of x .

Samples of the form $\text{SmCo}_{7-x}\text{Cu}_x$ ($x = 0, 0.1, 0.2, 0.3, 0.4, 0.5, \text{ and } 0.7$) and $\text{SmCo}_{6.75-x}\text{Fe}_x\text{Zr}_{0.25}$ ($x=0, 0.27, 0.41, 0.54$) have been prepared and studied. X-ray diffraction shows

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that these compounds form the hexagonal TbCu₇ structure. X-ray diffraction on magnetically aligned samples show that these samples have uniaxial anisotropy. The lattice parameters (a and c) are dependent on the composition of Fe and Cu. The unit cell volume is found to increase with x. The saturation magnetization increases with x for all samples with Fe substitution while it decreases with x for all samples with Cu substitution both at room temperature and at a temperature of 25 k. Curie temperature for compounds with Cu substitution increases with x for small values of x and it decreases for large values of x.

H. PROCESSING OF 2:17 POWDERS

H. Tang, J.P. Liu, T. Bui

We have begun the cooperation with Arnold Engineering Company, in association with Dr. Tom Bui. Dr. Bui has visited our laboratory and discussed with us possible joint research projects. The first cooperative work is on the improvement of magnetic properties of the commercial Sm-Co powder. Arnold Engineering has provided 1 kg commercial Sm-Co powder of the 2:17 type, of which the magnetic hysteresis has been measured. The work that will be done in Lincoln includes following aspects: (1) The effect of mechanical milling and annealing on the permanent magnetic properties. Through this investigation, we are studying the effect of disorder induced in mechanical milling, and the induced refinement of the grain size (down to 10 nm level) on the permanent magnetic properties. The correlation between the dimension of grain size, the disorder induced and the magnetic hardening will be developed. (2) Nanocomposite magnets will be developed by means of mechanical alloying technique based on the mixture of the SmCo 2:17 hard magnetic phase and a suitable amount of soft phase Co or (Fe,Co) alloy. The effective exchange coupling between the hard and soft magnetic phases will be explored in the nano-scale level, with the purpose of increasing the energy product of the Co-based bonded magnet. The effect of mechanical alloying condition and the heat treatment condition on the grain size, further on the magnetic properties will be investigated systematically. (3) New magnets made of two hard magnetic phases. This new type of magnet will be developed by means of mechanical alloying or mechanical milling techniques. The exchange coupling between two hard magnetic phases will be investigated in nano-scale level (down to 10 nm), in order to improve the magnetic properties. The homogeneous and coherent nano-structure is the key to obtain effective exchange coupling. The first step with respect to this novel magnet will be carried out on the SmCo-based hard magnetic phases, with the purpose to explore the possibility of improving the permanent magnetic properties, especially at high temperature. This new type magnet is expected to combine the individual advantages (high saturated magnetization, high coercivity) of the mixed phases which are suitable to energy application.

I. MAGNETOVOLUME EFFECT IN Fe-RICH RARE-EARTH COMPOUNDS: **Nd₅Fe₁₇H₁₆**

R. F. Sabiryanov and S. S. Jaswal

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Curie temperatures (T_c) of Fe-rich rare-earth compounds are very sensitive to the changes in the hybridization. This is responsible for the well-known large increase in T_c of 2-17 compounds with interstitial and substitutional modifications. The Nd_5Fe_{17} compound has attracted some attention recently as a possible permanent-magnet material. Hydrogenation of this compound ($Nd_5Fe_{17}H_{16}$) produces 14.4% volume expansion and increases its T_c and magnetization by 14% and 20% respectively. Because of the large number of atom per unit cell in this crystal (264 atoms) and the lack of information on the positions of hydrogen atoms, we perform first-principle electronic structure calculations for different volumes to study the effect of interstitial impurities such as H on the magnetic properties of Nd_5Fe_{17} . This is a reasonable procedure because the primary effect of minimally hybridizing impurity such as H is the magnetovolume effect. Because of the similar close-packed environment in Fe-rich compounds and fcc iron these calculations are repeated for fcc iron and the variation of T_c with the average Wigner-Seitz radius is quite similar for the two systems giving a universal curve for Fe-rich compounds. The calculated results are also in good agreement with the experimental data for $Nd_5Fe_{17}H_{16}$.

J. THEORY OF NANOMAGNETS

R. Skomski

Theoretical and experimental efforts have been devoted to the understanding and improvement of magnetic nanostructures. One topic is exchange interactions at nanocrystalline grain boundaries, and between magnetic clusters in a nonmagnetic metallic matrix. Magnetization profiles in the vicinity of grain boundaries have been calculated, and it is shown that grain boundaries act as perturbations whose effect decays exponentially inside the grains (crystallites). On the other hand, explicit formulas for the effective intergranular exchange and for long-range RKKY interactions in complicated metallic structures have been obtained. One result of the calculations is that magnetostatic interactions between well-separated clusters dominate RKKY interactions for grains larger than about 1 nm.

A second key area is the development of cobalt-containing permanent-magnet materials and the understanding of hysteresis loops. Among other things it is shown that in two-phase magnetic nanostructures there is a novel 'bulging' nucleation mode characterized by negative demagnetizing factors and therefore favorable from the point of view of coercivity.

K. MECHANICALLY MILLED AND ALLOYED HIGH TEMPERATURE COBALT-BASED MATERIALS

D. Leslie-Pelecky, R. Skomski, E. Kirkpatrick, Lanping Yue, Michael Leonard

Recent calculations by Ralph Skomski indicate that rare earths with a positive electrostatic quadrupole moment are likely to have significant anisotropies when combined with cobalt. Arc melting, followed by mechanical milling, has been used to produce $TmCo_5$.

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The coercivities of the TmCo_5 made thus far have been < 1 kOe, and we are investigating the reason for the discrepancy with theory. We have also investigated the behavior of mechanically alloying SmCo_5 with C. The resulting materials have exceptional oxidation resistance, even at C volume fractions as low as 10%. Samples made in this way can be left out in the air for months with less than a 1% change in the coercivity or saturation magnetization.

L. $\text{RCO}_5\text{:Co}$ EXCHANGE-COUPLED ALLOYS

I.A. Al-Omari and D.J. Sellmyer

The goal of this project is developing materials with high coercivity and high energy-product at high temperatures. The systems under investigation are $\text{RCO}_5\text{:Co}$ (Sm, Y, and Dy) alloys. We have prepared some of the samples by mechanical alloying and annealing. These samples are produced by milling the powders of each individual element together for times between 10 hrs and 40 hrs. Preliminary results show that the structure depends on the milling time and annealing temperature. Systematic study will be performed on milling time and heat treatment. The magnetic properties and behavior will be investigated at high temperatures for samples with different RCO_5 to Co ratios.

M. NANOSTRUCTURED $\text{Sm}(\text{Co,Zr})$ AND $\text{Pr}(\text{Co,Fe})$ MAGNET POWDERS SYNTHESIZED BY MECHANICAL MILLING

H. Tang, J.P. Liu, Y. Liu and D.J. Sellmyer

Nanostructured (nanocomposite and nanocrystalline) $\text{Sm}(\text{Co,Zr})$ magnets have been synthesized by mechanical milling and subsequent annealing of $\text{Sm}(\text{Co,Zr})$ alloys with a small amount of Zr addition. Nanocomposite $\text{Sm}(\text{Co,Zr})_7\text{:Co}$ magnets with optimal hard magnetic properties, such as coercivity (H_{ci}) of 6.9 kOe, remanence (M_r) of 8.13 kG, remanence ratio (M_r/M_s) of 0.58 and energy product $(\text{BH})_{\text{max}}$ of 9.2 MGOe, have been obtained in the $\text{Sm}_{10.5}\text{Co}_{89.5-x}\text{Zr}_x$ ($x = 0-5$) system. Nanocrystalline $\text{Sm}(\text{Co,Zr})_7$ magnets with optimal hard magnetic properties, coercivity H_{ci} of 18.6 kOe, remanence magnetization $4\pi M_r$ of 8.0 kG and maximum energy products $(\text{BH})_{\text{max}}$ of 13.0 MGOe, have been synthesized in the $\text{Sm}_{12.5}\text{Co}_{87.5-x}\text{Zr}_x$ ($x = 0-5$) system. Coercivity H_{ci} of 32.0 kOe has been obtained in the nanocrystalline $\text{Sm}_{15.4}\text{Co}_{84.6-x}\text{Zr}_x$ ($x = 0-5$) magnet powders, which consists of a mixture of hard phases $\text{Sm}(\text{Co,Zr})_5$ and $\text{Sm}(\text{Co,Zr})_7$ with nanoscale grain size, and shows the magnetization behavior of single phase. We found that, in the above systems, the magnetic properties are strongly dependent on milling, annealing condition, and the Zr addition. A small amount of Zr addition is essential to obtain higher coercivity, and is very helpful to enhance remanence in some cases. Further optimization of hard magnetic properties are expected to carry out in the $\text{Sm}(\text{Co,Zr})$ or $\text{Sm}(\text{Co,Fe,Zr})$ nanostructured magnet systems, and hard magnetic properties at high temperatures will be explored.

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We have also synthesized nanostructured Pr(Co,Fe) magnet powder systems by mechanical milling and subsequent annealing. Coercivity H_{ci} as high as 14.7-20 kOe has been obtained in the Pr(Co,Fe) nanostructured magnetic powders. Further investigation on this nanostructured system, such as small amount of Zr addition, will be carried out soon, to optimize the hard magnetic properties.

N. MAGNETIC PROPERTIES AND STRUCTURE OF (Sm,Er)(Fe,Co)₁₁Ti COMPOUNDS

H. Tang, J. Zhou, J.P. Liu and D.J. Sellmyer

Prerequisites for permanent magnets are high saturation magnetization M_s , larger anisotropy H_A and good environment-resistant properties. $SmFe_{11}Ti$ system compounds have relatively high Curie temperature T_C , high M_s , large H_A . Previous studies show that T_C and M_s increase, H_A varies slightly with Co content in a suitable Co concentration range. In addition, Er and Sm have the same sign of Stevens coefficient so combining them may lead to better anisotropy and improve the relative temperature dependence of saturation magnetization and anisotropy field in $SmFe_{11}Ti$ compounds. Hence, better hard magnetic properties at higher temperature may be expected to develop in $(Sm,Er)(Fe,Co)_{11}Ti$ compounds with the $ThMn_{12}$ type structure. We have investigated magnetic properties and structure of $Sm_{1-x}Er_xFe_{11-y}Co_yTi$ ($x = 0, 0.2, 0.4$, and $y = 3, 4$) compounds. We found that the crystalline structure is of $ThMn_{12}$ type tetragonal one, lattice parameters a , c and cell volume V decrease with Er substitution. Curie temperature increased to 820-890 K compared to their parent compound $SmFe_{11}Ti$ (~ 600 K). The saturation magnetization varies slightly with Er content. The anisotropy field at 300 K is between 79.7 kOe and 92.6 kOe, which is higher than that of $Nd_2Fe_{14}B$. Additionally, an anomalous temperature dependence of anisotropy field H_A has been observed in the temperature range of 5 – 300 K with variation of Er content, which can be attributed to the mutual contribution to the anisotropy favoring c -axis due to the second-order crystal field of Er and Sm. The anomaly on the $H_A(T)$ curves at lower temperature is associated with the complex spin-structure of Er at low temperature. The results show that a higher anisotropy field can be developed in the compounds $Sm(Fe,Co)_{11}Ti$ with appropriate amount of Er substitution. It is suggested that $(Sm,Er)(Fe,Co)_{11}Ti$ compounds may be a potential candidate to develop better hard magnetic properties at higher temperature.

O. DEVELOPMENT OF INERT-GAS-CONDENSATION-COMPACTION SYSTEM

D. L. Leslie-Pelecky

The re-designed source has been fabricated and the rest of the system is complete. The project had to be moved to a different lab and a Roots pump was installed. We are now able to produce samples and will produce nanostructured $SmCo_5$, $TmCo_5$ and $GdCo_5$. These results will be compared to theory by Skomski and to the results of the mechanical alloying experiments. The finer control over size and size distribution of the clusters possible with the IGC system will allow us to test size effects in a way not previously possible.

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P. MAGNETIC PROPERTIES OF CLUSTER-BEAM-SYNTHESIZED COBALT: MATRIX MATERIALS

Y. Qiang, J.M. Meldrim, D.J. Sellmyer (in cooperation with H. Haberland (Freiburg) and H. Fert (Orsay))

In this project we are assembling a new cluster-beam deposition system for the design of nanostructured two-phase magnetic materials with outstanding hard or soft magnetic properties. The technique relies on growth of clusters of 3-15 nm diameter and codeposition of these with a second phase with different magnetic properties. Components of the system have been purchased and assembly is underway. In parallel with the construction we are researching Co clusters embedded in nonmagnetic matrices including noble metals such as Cu and Ag, and nonconducting matrices such as SiO₂. Zero-field and field-cooling experiments are used to probe the interactions of the clusters through the matrix, and it has been found that the presence of conduction electrons enhances the strength of the interactions. Theoretical work, along two different paths, is also being pursued on these systems.

Q. MAGNETIC AND STRUCTURAL PROPERTIES OF SmCo_{6.75-x}Fe_xZr_{0.25} COMPOUNDS

I.A. Al-Omari, J. Zhou, D.J. Sellmyer

Bulk alloys of SmCo_{6.75-x}Fe_xZr_{0.25}, where $x=0, 0.27, 0.41,$ and 0.54 were prepared by arc-melting. The alloys were characterized by x-ray diffraction, vibrating sample magnetometer, and SQUID magnetometer. X-ray diffraction shows that these compounds form the hexagonal TbCu₇-type structure for $x<0.67$ and for large values of $x(x\geq 0.67)$ the hexagonal TbCu₇-type structure cannot be formed. The lattice parameters (a and c) are dependent on the iron concentration. The unit cell volume is found to increase with x which is due to the larger atomic volume of Fe. X-ray diffraction and magnetization measurements on magnetically aligned samples show that all the samples studied have uniaxial anisotropy. The saturation magnetization increases with x at both room temperature and 5 K. The Curie temperature for these compounds is about 770°C for all values of x under investigation.

R. TEMPERATURE DEPENDENCE OF MAGNETIC HYSTERESIS OF RCo_x:Co NANOCOMPOSITES (R = Pr and Sm)

J.P. Liu, R. Skomski, Y. Liu, D.J. Sellmyer

The temperature dependence of magnetic hysteresis of RCo_x:Co (R = Pr and Sm) nanocomposite films is reported. These films are prepared by dc and rf sputtering and subsequent thermal processing. It is found that the squareness of the hysteresis loops deteriorates with decreasing temperature. This is attributed to the enhanced magnetic anisotropy of the hard phases at low temperatures. The analysis of the magnetic reversal

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process shows that the anisotropy enhancement leads to a transition form cooperated to independent behavior. This analysis agrees well with the experimental results.

S. PHASE CONSTITUTION AND MAGNETIC PROPERTIES OF Nd₁₀Fe₇₆B₄M₁₀ AND Nd₁₀Fe₇₆B₂M₁₂ (M = Fe, Ti, V, Cr, Mn, Co and Al) ALLOYS PREPARED BY MECHANICAL ALLOYING

W. Liu, Z.D. Zhang, J.P. Liu, X.K. Sun, D.J. Sellmyer, X.G. Zhao

Phase constitution and magnetic properties of Nd₁₀Fe₇₆B₄M₁₀ and Nd₁₀Fe₇₆B₂M₁₂ (M = Fe, Ti, V, Cr, Mn, Co and Al) alloys prepared by mechanical alloying and subsequent annealing have been systematically studied. It is found that the components of phases in the alloys critically depend on the additive transition metals M. In all the alloys, only the addition of Ti give significant enhancement to the permanent-magnetic properties. The increase of the Curie temperature in the Ti-doped alloys with an excessively low B content implies that part of the Ti atoms may occupy crystalline sites other than the Fe sites. In all the M additive alloys, with increasing content of the additive elements, the quantity of 1:7 phase increases and that of Nd₂Fe₁₄ B-type phase decreases.

T. NANOSTRUCTURED FePt:B₂O₃ THIN FILMS WITH PERPENDICULAR MAGNETIC ANISOTROPY

C.P. Luo, S.H. Liou, L. Gao, Y. Liu, D.J. Sellmyer

FePt/B₂O₃ multilayers were deposited by magnetron sputtering onto 7059 glass substrates. By annealing the as-deposited films at 550°C, nanostructured FePt:B₂O₃ films consisting of FePt grains with L₁₀ structure, embedded in a glassy B₂O₃ matrix, were obtained. The c axes of the FePt grains can be made to align with the film normal direction, which results in a perpendicular anisotropy constant of 3.5x10⁷ erg/cc. The films remain layered structures after annealing when the B₂O₃ layer thickness exceeds 16 Å. The nanostructure of the films was investigated by transmission electron microscopy. The coercivities and the average grain sizes of the films are dependent on the B₂O₃ concentrations, with coercivities varying from 4 to 12 kOe, while average grain sizes vary from 4 to 17 nm. Strong perpendicular anisotropy, adjustable coercivity, and fine grain size suggest this nanocomposite system might have significant potential as recording media at extremely high areal density.

U. STRUCTURE AND MAGNETIC PROPERTIES OF SmCo_{7-x}Ti_x WITH TbCu₇-TYPE STRUCTURE

J. Zhou, I.A. Al-Omari, J.P. Liu, D.J. Sellmyer

The SmCo_{7-x}Ti_x, x = 0-0.56 bulk samples are prepared by arc melting. X-ray diffraction indicates that samples with 0.2 < x < 0.4 form a single disordered TbCu₇-type structure phase and other minor phases appear for other values of x, which indicates that Ti helps stabilize

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the 1-7 phase. The lattice parameters ratio (c/a) increases with increasing Ti concentration. Room temperature saturation magnetization and Curie temperature decrease with increasing x . X-ray diffraction and magnetization measurements on aligned samples show that all samples studied have uniaxial anisotropy. The anisotropy field is found to increase with increasing x reaching a maximum of 175 kOe at $x=0.28$ and then decreases for higher values of x . This anisotropy field is 20% higher than that of the same compound with $\text{Th}_2\text{Zn}_{17}$ -type structure.

V. Sm-Co-Cu-Ti HIGH-TEMPERATURE PERMANENT MAGNETS

J. Zhou, R. Skomski, C. Chen, G.C. Hadjipanayis, D.J. Sellmyer

A class of promising permanent-magnet materials with an appreciable high-temperature coercivity of 8.6 kOe at 500°C is reported. The Sm-Co-Cu-Ti magnets are prepared by arc melting and require a suitable heat treatment. Magnetization measurements as a function of temperature and x-ray diffraction patterns indicate that the samples are two-phase mixtures of 2:17 and 1:5 structures. Depending on heat treatment and composition, some of the magnets exhibit a positive temperature coefficient of coercivity. The promising high-temperature behavior of the coercivity is ascribed to the temperature dependence of the domain-wall energy, which affects the curvature of the walls and the pinning behavior.

W. COERCIVITY OF TITANIUM-SUBSTITUTED HIGH-TEMPERATURE PERMANENT MAGNETS

J. Zhou, R. Skomski, D.J. Sellmyer

The temperature dependence of the coercivity of Sm-Co based magnets is investigated by magnetization measurements and model calculations. The Zr-free titanium-substituted Sm-Co material exhibits a positive temperature coefficient dH_c/dT of the coercivity (TCC) above room temperature, a reasonable hysteresis-loop shape, and an appreciable coercivity of 12.3 kOe at 500°C for the nominal composition $\text{Sm}(\text{Co}_{6.2}\text{Cu}_{0.8}\text{Ti}_{0.3})$. The samples were produced by heat-treating and disordered 1:5 alloy commonly referred to as the TbCu_7 (or 1:7) phase. X-ray diffraction analysis shows that, upon annealing at 1165°C, the starting material segregates into more-or-less stoichiometric 1:5 and 2:17 phases. The TCC is explained by taking into account that two-phase Sm-Co magnets are of the pinning type, that is the coercivity is realized by capturing (or repelling) domain walls at 1:5/2:17 phase boundaries. Starting from a planar-defect approach, the TCC is modeled as a function of the anisotropy constants of the involved phases. The present approach yields a fair agreement between theory and experiment, and explains the existence of a coercivity maximum in terms of the Cu concentration.

X. HIGH-TEMPERATURE MAGNETIC PROPERTIES OF MECHANICALLY ALLOYED SmCo_5 AND YCo_5 MAGNETS

I.A. Al-Omari, R. Skomski, R.A. Thomas, D. Leslie-Pelecky, D.J. Sellmyer

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The high-temperature coercivity of mechanically alloyed and subsequently annealed $R\text{Co}_5$ ($R = \text{Sm}$ and Y) is studied. The annealed materials have the hexagonal CaCu_5 structure with 2:17 (or 1:7) regions as a minor phase. High-temperature magnetic measurements show that the coercivities of materials decrease with increasing temperature from room-temperature to 873 K, but that the temperature coefficient of the coercivity of YCo_5 is much smaller than that of SmCo_5 . This behavior is explained in terms of the intrinsic temperature variation of the magnetocrystalline anisotropy.

Y. FIRST-PRINCIPLE STUDIES OF Sm-Co HARD MAGNETS

Renat Sabiryanov, Sitaram Jaswal

Substantial efforts are being made to improve the properties of permanent-magnet materials, *i.e.*, to increase the energy product $((\text{BH})_{\text{max}})$ and the Curie temperature (T_c). The search for magnets for high-temperature applications has been difficult because the magnetic properties of a ferromagnet deteriorate as the temperature increases toward T_c . However, it has been found recently that this trend can be slowed or reversed for the coercivity of $\text{Sm}(\text{Co},\text{TM})_z$ ($z = 7-8$, $\text{TM} = \text{Cu},\text{Ti},\text{Zr}$) with small amounts of Cu and TM. This breakthrough for high-temperature magnets is ascribed to the two-phase cellular microstructure with $\text{Sm}_2\text{Co}_{17}$ as the nucleus surrounded by a layer of SmCo_5 . It has been found that Cu is mainly confined to the SmCo_5 region and hence is thought to be responsible for the phase separation. There is no clear experimental evidence as to the location of TM atoms. We are studying the relative phase stability of SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ doped with TM-elements to understand the two-phase microstructure observed experimentally. We calculate the solution energies of TM elements at various substitution sites in SmCo_5 and $\text{Sm}_2\text{Co}_{17}$ using FP-LMTO method. The calculated energies are used to find preferred occupation sites and the relative stability of two phases. Impurity interaction energies are calculated to look for clustering or phase segregation. For each impurity site and possible segregated phases we calculate changes in magnetization, effective exchange parameters and anisotropy change with respect to the base phase. The changes in anisotropy due to the impurities in two phases help in locating pinning regions. Assuming typical variation of anisotropy with temperatures in each of the phases present in the system we will perform micromagnetic simulation at finite temperature to find the coercivity as a function of the temperature.

Z. HIGH SURFACE AREA MAGNETS

Jeffrey E. Shield (formerly at University of Utah; currently at University of Nebraska);
Eric Billings (formerly at University of Utah)

This project investigated the processing of multilayered Fe-Cu via deformation processing. Initial stacks containing 8 Fe and 8 Cu foils of 0.1 mm thickness were diffusion bonded in a controlled atmosphere press. These were then cold-rolled to approximately 50 percent deformation. Stress-relief anneals were utilized after each deformation cycle.

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Periodically, it was necessary to cut and re-stack the material. Consequently, the original 16 layer stack propagated to over 200,000.

The texture, microstructure, and magnetic properties were characterized for the multilayer materials, with most interest in materials below 100 nm. A dominant {211} in plane texture was observed, with a competing texture, likely {100} in-plane texture, observed at thicker layers and with higher deformation. Rocking curves and inverse pole figures revealed stronger {211} texture in thinner layers.

Scanning and transmission electron microscopy was used to examine the multilayer microstructure. Extremely uniform layers were observed, with Fe deforming to a slightly greater extent than Cu. TEM also revealed a higher dislocation density in Cu; its greater thickness was attributed to a higher strain hardening rate, making deformation more difficult. Additionally, both Fe and Cu layers were observed to be single crystalline below a critical value of layer thickness, which also corresponded to when {211} texture increased dramatically. The development of single crystals eliminated the opportunity for competing texture. Alas, a finite layer thickness was reached despite continued deformation processing. This limit, determined by TEM, was found to be approximately 50 nm. It is thought that further deformation is accommodated in near-surface regions and leads to grain fragmentation rather than a reduction in layer thickness.

The magnetic properties could be related to the increasing texture observed in the multilayer materials. For example, an increase in coercivity was observed, which was explained by an increased texture along the easier <110> rolling direction.

This work led to a Master of Science degree for Eric Billings through the Department of Materials Science and Engineering at the University of Utah.

5. Technology Transfer: None.