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## Pressure Induced Reversal of the Volume Expansion Caused by Interstitial Nitrogen in $\text{Nd}_2\text{Fe}_{17}\text{N}_3$

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tures between 0.5 bar and 8 bar by means of hydrogen differential thermal analysis (HDTA) showed, that the disproportionation reaction becomes stronger and proceeds at lower temperatures for increasing hydrogen pressures. X-ray diffraction investigations of the HDTA samples showed a decreasing content of the 2:17 phase for increasing hydrogen pressures, leading to a completely disproportionated structure for all the compounds under investigation at 8 bar. Microstructural changes within the disproportionated mixture as a result of the applied pressure have been documented in detail by high resolution scanning electron microscopy. The complete disproportionation of the samples with high Ga content is very promising for an application of the HDDR process under increased hydrogen pressures for the preparation of highly stable and coercive gas carbonated  $\text{Sm}_2\text{Fe}_{17-x}\text{Ga}_x\text{C}_y$  powders, which could be used for the production of fully dense permanent magnets by hot compaction.

<sup>1</sup>M. Kubis, L. Cao, A. Handstein, B. Gebel, K.-H. Müller, and L. Schultz J. Appl. Phys. **81**, 6485 (1997).

9:48

**ED-05. THE HARD-MAGNETIC PROPERTIES OF MELT-SPUN  $\text{Sm}_2\text{Fe}_{15}\text{Ga}_2\text{C}_{1.5}$  AND  $\text{SmFe}_{15-x}\text{M}_x\text{Ga}_2\text{C}_{1.5}$  ( $\text{M}=\text{Cu}, \text{Nb}; 0.5(x(2.0))$  ALLOYS\*.** Shao-ying Zhang, Bao-gen Shen, and Hong-wei Zhang (State Key Lab. of Magnetism & Ctr. for Condensed Matter Phys., Inst. of Phys., Chinese Acad. of Sci., Beijing 100080, P.R. China)

Our group has reported previously that the coercivity of 1.5T could be obtained in  $\text{Sm}_2\text{Fe}_{14}\text{Ga}_3\text{C}_{1.5}$  ribbons by direct quenching at the optimum wheel velocity. Recently, it was discovered that the coercivity up to 2.2T could be achieved in melt-spun  $\text{Sm}_2+(\text{Fe}_{15}\text{Ga}_2\text{C}_2)$  with overstoichiometric Sm content by annealing the amorphous ribbons. In this paper, the coercivity and microstructure of melt-spun  $\text{Sm}_2\text{Fe}_{15}\text{Ga}_2\text{C}_{1.5}$  and  $\text{SmFe}_{15-x}\text{M}_x\text{Ga}_2\text{C}_{1.5}$  ( $\text{M}=\text{Cu}, \text{Nb}; 0.5(x(2.0))$  alloys have been studied as a function of composition and annealing temperature and time. The as-quenched almost amorphous ribbons were annealed at 823-1173K. Before annealing, the ribbons showed low coercivity ( $<0.5\text{T}$ ). After annealing, the ribbons with the main phase of  $\text{Th}_2\text{Zn}_{17}$  and a small amount of  $\gamma\text{-Fe}$  were detected from X-ray diffraction patterns and SEM. The coercivities of all ribbons first increase monotonically with increasing annealing temperature. The maximum coercivities at room temperature is about 1.4T, 2.2T and 1.6T for  $\text{Sm}_2\text{Fe}_{15}\text{Ga}_2\text{C}_{1.5}$ ,  $\text{SmFe}_{14}\text{Cu}_1\text{Ga}_2\text{C}_{1.5}$  and  $\text{SmFe}_{14}\text{Nb}_1\text{Ga}_2\text{C}_{1.5}$ , respectively, after annealing at 1073K for 15min. Then coercivities decrease at the higher annealing temperature. For  $\text{SmFe}_{14}\text{Cu}_1\text{Ga}_2\text{C}_{1.5}$  ribbons annealing at 1073K for 5-60min., the maximum coercivities were obtained between 15min. and 20min. Although the substitution of Cu or Nb for Fe has a small effect on the average size of crystalline grain, but affects the coercivity obviously. From the initial magnetizing curves and the applied magnetic field dependencies of  $H_c$  and  $M_r/M_{rmax}$ , the mechanism of the coercivity has been analyzed.

\*This work was supported by the National Natural Science Foundation of China.

<sup>1</sup>J. van Lier, M. Seeger, and H. Kronm (Ier, J. Magn. Magn. Mater. **167**, 43 (1997).

10:00

**ED-06. ELECTRON MICROSCOPY STUDIES OF HIGH COERCIVE MELT-SPUN  $\text{Sm}_{2+\delta}\text{Fe}_{15}\text{Ga}_2\text{C}_2$  PERMANENT MAGNETS.** J. van Lier, A. Zern, H. Labitzke, J. Thomas, M. Seeger, and H. Kronmüller (Max-Planck-Inst. für Metallforschung, Heisenbergstrasse 1, D-70569 Stuttgart, Germany)

The influence of overstoichiometric Sm contents on the magnetic properties of melt-spun Ga stabilized  $\text{Sm}_2\text{Fe}_{17}$  carbides has been investigated. For optimum heat treated  $\text{Sm}_{2+\delta}\text{Fe}_{15}\text{Ga}_2\text{C}_2$  ribbon flakes an increase of the room temperature coercivity could be observed from  $\mu_0 H_c = 1.7\text{T}$  for stoichiometric samples to  $\mu_0 H_c = 2.2\text{T}$  for  $\delta = 0.15$ . This effect is very interesting because of an improved temperature stability. At 500 K a maxi-

mum energy density of  $(BH)_{max} = 31.4\text{kJ/m}^3$  for  $\delta = 0.13$  can be observed. It is shown, that a Sm rich nonmagnetic intergranular phase decouples the grains magnetically and is consequently the reason for the high coercivities of these samples. Electron microscopy studies of these melt-spun  $\text{Sm}_{2+\delta}\text{Fe}_{15}\text{Ga}_2\text{C}_2$  samples with different Sm contents have been performed by SEM and TEM. A broad grain size distribution (50-250 nm) is observed in the SEM, but there is no difference between samples with different Sm contents. TEM studies show no change in grain shape with variation of the  $\delta$  value. High resolution TEM studies indicate the existence of a very thin intergranular phase for samples with an excess of Sm. To prove this we performed high resolution EDX studies with a scanning-TEM. These investigations show a significant increase of the Sm content at the grain boundaries for these high coercive samples.

10:12

**ED-07. PRESSURE INDUCED REVERSAL OF THE VOLUME EXPANSION CAUSED BY INTERSTITIAL NITROGEN IN  $\text{Nd}_2\text{Fe}_{17}\text{N}_3$ .** G. K. Marasinghe, W. J. James, P. C. Ezekwenna (Univ. of Missouri-Rolla, Rolla, MO 65409), Robert Luo, W. B. Yelon (Univ. of Missouri Res. Reactor, Columbia, MO 65211), Y. Zhao, R. B. Von Dreele (Los Alamos Natl. Lab., Los Alamos, NM 87545), M. Ellouze, and Ph. l'Heritier (ENS Physique de Grenoble, BP 46, 38402 St. Martin d'Heres, France)

Crystallographic properties of  $\text{Nd}_2\text{Fe}_{17}\text{N}_3$  have been investigated using powder neutron diffraction techniques at sample pressures up to 7.23 GPa. With increasing sample pressure, the unit cell volume of  $\text{Nd}_2\text{Fe}_{17}\text{N}_3$  decreases almost linearly at a rate of  $6.1\text{Å}^3/\text{GPa}$  without undergoing a phase transformation or expelling the interstitial nitrogen atoms. At 7.3 GPa, the unit cell volume of  $\text{Nd}_2\text{Fe}_{17}\text{N}_3$  becomes approximately equal to that of  $\text{Nd}_2\text{Fe}_{17}$  at atmospheric pressure. Even though the unit cell of  $\text{Nd}_2\text{Fe}_{17}$  expands anisotropically due to nitrogeneration, contraction of the  $\text{Nd}_2\text{Fe}_{17}\text{N}_3$  unit cell under pressure appears to be isotropic with both a and c lattice parameters decreasing by approximately 1.8% as the sample pressure increases from ambient to 7.3 GPa.

10:24

**ED-08. A NEUTRON DIFFRACTION STRUCTURAL STUDY OF  $\text{R}_2\text{Fe}_{17-x}\text{Al}_x(\text{C})$  ( $\text{R}=\text{Tb}, \text{Ho}$ ) ALLOYS.** W. B. Yelon, H. Luo, M. Chen (Res. Reactor Ctr., Univ. of Missouri-Columbia, Columbia, MO 65211), W. C. Chang, and S. H. Tsai (Dept. of Phys., Natl. Chung Cheng Univ., Ming-Hsiung, Chia-Yi, Taiwan, ROC)

As part of a comprehensive study on the effect of combined substitutional and interstitial modification on the structure and magnetic properties of  $\text{R}_2\text{Fe}_{17}$  compounds, especially with higher substituent concentration,  $\text{Tb}_2\text{Fe}_{17-x}\text{Al}_x\text{C}$ ,  $\text{Ho}_2\text{Fe}_{17-x}\text{Al}_x$  and  $\text{Ho}_2\text{Fe}_{17-x}\text{Al}_x\text{C}$  ( $x = 1-4$ ) have been studied by using neutron diffraction. All these samples were made by arc melting and were confirmed to have the  $\text{Th}_2\text{Zn}_{17}$  type rhombohedral structure (space group  $R\bar{3}m$ ) except for  $\text{Ho}_2\text{Fe}_{16}\text{Al}$ ,  $\text{Ho}_2\text{Fe}_{15}\text{Al}_2$  which have the  $\text{Th}_2\text{Ni}_{17}$  type hexagonal structure (space group  $P6_3/mmc$ ), and  $\text{Ho}_2\text{Fe}_{13}\text{Al}_3$ , a mixture of the two phases with the hexagonal phase dominant. The size of the unit cell seems to be the determinant factor in forming an ordered rhombohedral phase or a disordered hexagonal phase. Both the substitution of Fe by Al and the insertion of C can lead to the formation of an ordered rhombohedral phase. The site preference of Al is somewhat different with and without C. In the Ho carbided samples, Al prefers the 18h and 18f sites in the rhombohedral structure and totally avoids the 9d site while in the Ho noncarbided samples, a small amount of Al is found at the hexagonal 6g site which is the cognate of the rhombohedral 9d. In the Tb carbided samples, for Al  $x=1, 2$  and 3, Al has a higher occupancy at the 18h site and a much lower one at 6c as compared to  $\text{Tb}_2\text{Fe}_{17-x}\text{Al}_x$ . At higher Al concentration, Al occupancy at 18h tends to saturate around 40% and the Al site occupancies are similar with and