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Hard magnetic properties of the novel compound $\text{Sm}_3(\text{Fe, Cr})_{29}\text{N}_y$

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Abstract. The novel nitride $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ with monoclinic $\text{Nd}_3(\text{Fe, Ti})_{29}$ structure is synthesized by gas–solid reaction. Its hard magnetic properties have been investigated by ball milling and magnetic measurements. The intrinsic coercivity $\mu_0 H_c$ increases with decreasing average particle size d and, after reaching a maximum at $d = 0.4 \mu\text{m}$, then decreases slowly. $\mu_0 H_c$ increases also with increasing magnetizing field. A value of $\mu_0 H_c = 0.79 \text{ T}$ has been attained. The magnetizing field for obtaining coercivity saturation is about 1.5 T. The hard magnetic properties of remanence $B_r = 0.87 \text{ T}$ and energy product $(BH)_{\text{max}} = 104.8 \text{ kJ m}^{-3}$ for $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ are achieved at 293 K.

1. Introduction

Recently a new ternary rare-earth iron compound family $\text{R}_3(\text{Fe, M})_{29}$ ($\text{M} = \text{Ti, V, Cr, Mn}$ or Mo) with a monoclinic $\text{Nd}_3(\text{Fe, Ti})_{29}$ (3:29) structure were discovered by Shcherbakova *et al* [1] and Collocott *et al* [2]. The 3:29 structure is an intermediate structure between the well known rhombohedral $\text{Th}_2\text{Zn}_{17}$ (2:17 R) and tetragonal ThMn_{12} (1:12) structure and consists of the alternate stacking of the 2:17 R and 1:12 segments [3, 4]. Their interstitial compounds $\text{R}_3(\text{Fe, M})_{29}\text{X}_y$ ($\text{X} = \text{N}$ or C) are similar to those of 2:17-type and 1:12-type compounds, exhibit excellent intrinsic magnetic properties (high magnetic ordering temperature and magnetization), and have a strong uniaxial anisotropy when $\text{R} = \text{Sm}$ [5, 6] which is a potential candidate for permanent magnet materials. In our previous work we reported the hard magnetic properties of $\text{Sm}_3(\text{Fe, Ti})_{29}\text{N}_y$ [7, 8] and the intrinsic magnetic properties of $\text{R}_3(\text{Fe, Cr})_{29}\text{X}_y$ ($\text{R} = \text{Nd}$ or Sm ; $\text{X} = \text{N}$ or C) [9]. Suzuki *et al* [10] studied the hard magnetic properties of coarse powder of $\text{Sm}_3(\text{Fe, Cr})_{29}\text{N}_y$ with variation in N concentration at room temperature. In this work, we investigated the size dependence of the hard magnetic properties of $\text{Sm}_3(\text{Fe, Cr})_{29}\text{N}_y$ at room temperature and low temperatures.

2. Experimental details

Ingots with the composition $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5$ were prepared by argon arc melting with starting elements of purity at least 99.9%. The ingots were then wrapped in molybdenum foil and sealed in quartz tubes under an argon atmosphere for a subsequent anneal at 1273–1323 K

for 1–5 d, followed by quenching in water. The starting compositions contain 10 wt% excess Sm in order to compensate for the loss during the melting. The 3:29 single-phase character of alloys was examined by both x-ray diffraction and thermomagnetic analysis. Before preparing the nitride phase, the single-phase 3:29 ingots were pulverized into fine powders with a size of 20–35 μm . Nitrogenation was performed by heating the fine powders in nitrogen at 1 atm in a temperature range 773–873 K for 3–4 h. The nitride powders were ball milled using metal balls of 4–10 mm diameter with a sample-to-metal-ball weight ratio of 1 to 60. The magnetically aligned cylinder samples of different ball-milled times were made by mixing the ball-milled nitride powders with epoxy resin and solidifying in an applied field of 1.2 T at room temperature. The average particle sizes d of the ball-milled nitride powder were estimated by means of observation on the polished end surface of the cylinders using an optical microscope. After the cylinder samples were magnetized at 293 K in open circuit with a pulse field of about 4 T, the hysteresis loops of samples were measured using a vibrating-sample magnetometer at 293 K and a SQUID magnetometer at 4.2 K. The demagnetization factor of samples was considered.

3. Results and discussion

It has been shown in our previous work [9] that $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ is nearly a 3:29-type single-phase compound with a trace of $\alpha\text{-Fe}$. $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ exhibits a strong uniaxial anisotropy ($B_a = 11.0$ T) and has a high $T_c = 488$ K and a high room-temperature magnetization $\mu_0 M_s = 0.95$ T. These promising intrinsic properties might lead to excellent extrinsic hard magnetic properties for materials based on $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$.

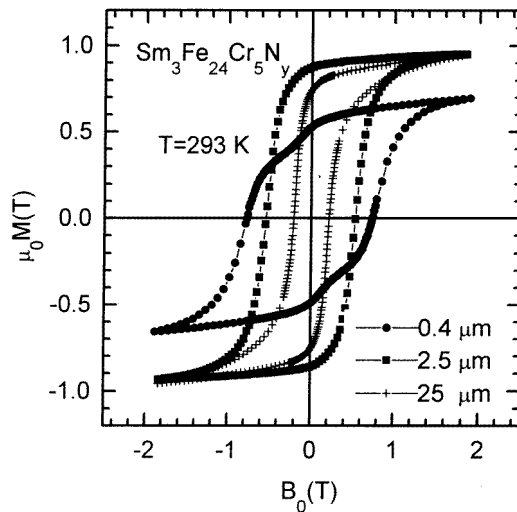


Figure 1. Typical room-temperature hysteresis loops of samples with average particle sizes $d = 25$, 2.5 and 0.6 μm for $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$.

Figure 1 shows typical room-temperature hysteresis loops of aligned samples with average particle sizes $d = 25$, 2.5 and 0.6 μm for the $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ after magnetization in a pulse field of about 4 T. It can be seen that the particle size is an important parameter which affects the hard magnetic properties of the resulting nitride. It also affects the squareness of

the hysteresis loops. The original coarse nitride powders with particle sizes of 20–35 μm have a poor squareness of loops owing to the very small coercivity. This result is different from that reported in [10] where the coarse nitride powders around 30 μm in diameter exhibit the largest coercivity. For the sample with $d = 2.5 \mu\text{m}$ the squareness of the loop is improved and the remanence obviously increases since the coercivity has been developed by the ball milling. However, when $d = 0.6 \mu\text{m}$, the squareness of the loop again becomes poor, which is probably due to oxidation of the fine powders at this stage.

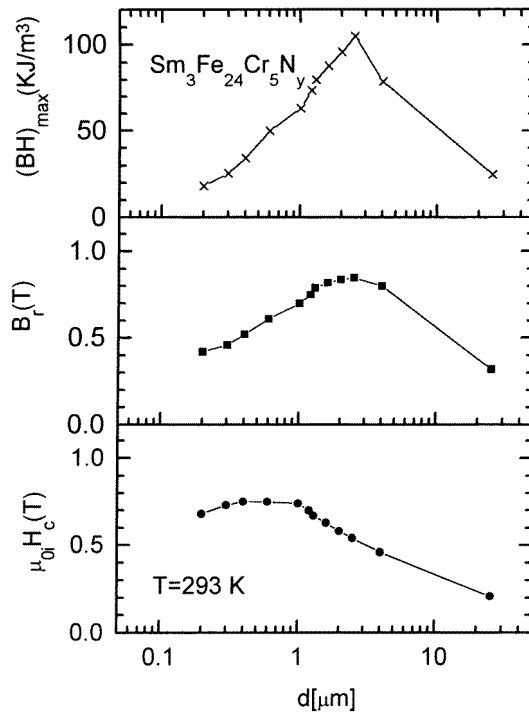


Figure 2. Variation in B_r , $\mu_0 i H_c$ and $(BH)_{\text{max}}$ for magnets at 293 K with average particle sizes d for $\text{Sm}_2\text{Fe}_{24}\text{Cr}_5\text{N}_y$.

The remanence B_r , intrinsic coercivity $\mu_0 i H_c$ and energy product $(BH)_{\text{max}}$ of these ball-milled $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ samples at 293 K are shown in figure 2 as functions of the average particle size d . With decreasing d , first B_r , $\mu_0 i H_c$ and $(BH)_{\text{max}}$ increase rapidly, then B_r and $(BH)_{\text{max}}$ achieve maxima at $d = 2.5 \mu\text{m}$ and finally they decrease rapidly, while $\mu_0 i H_c$ continuously increases through $d = 2.5 \mu\text{m}$ up to $d = 1 \mu\text{m}$, achieves a plateau of 0.75 T between $d = 1.0 \mu\text{m}$ and $0.4 \mu\text{m}$ and then decreases very slowly. Since such particle sizes (0.2–25 μm) are larger than the critical diameter D_c (about 0.1 μm) which forms a single-domain particle, these nitride particles must have multidomains. Usually, the coercivity of these multidomain particles increases with decreasing particle size [11], as seen at $d > 1 \mu\text{m}$ in figure 2. The ball milling not only reduces the size of the particles but also increases the surface defects. The former tends to increase the coercivity and the latter tends to reduce it. The plateau at $0.4 \mu\text{m} < d < 1 \mu\text{m}$ is just a consequence of both mechanisms, and the decrease in the coercivity at $d < 0.3 \mu\text{m}$ can be attributed to oxidation of the very fine powders. For a magnetizing field of about 4 T, the maximum values of

B_r , $\mu_{0i}H_c$ and $(BH)_{max}$ at room temperature reach 0.87 T, 0.75 T and 104.8 kJ m^{-3} , respectively.

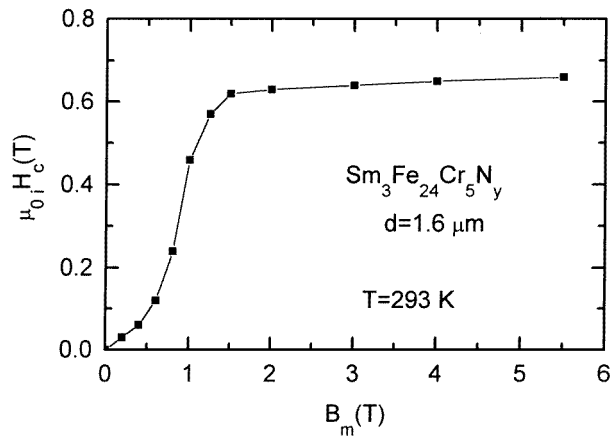


Figure 3. Magnetizing field dependence of the coercivity $\mu_{0i}H_c$ at 293 K for the sample with average particle size $d = 1.6 \mu\text{m}$.

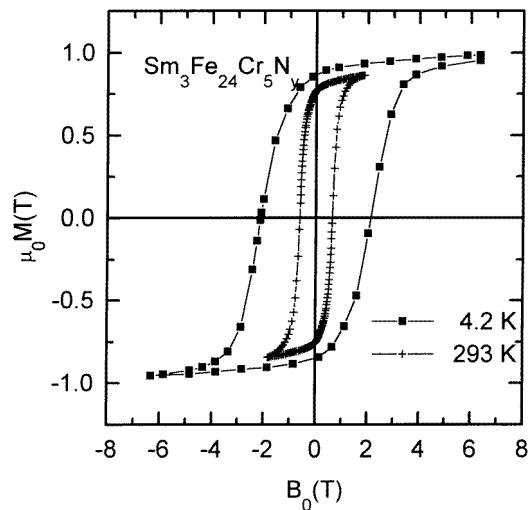


Figure 4. Hysteresis loops of the $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ sample with $d = 1.6 \mu\text{m}$ at 4.2 and 293 K.

The magnetizing field dependence of the coercivity $\mu_{0i}H_c$ at 293 K for the sample with $d = 1.6 \mu\text{m}$ is shown in figure 3. It can be seen that, on increasing the magnetizing field B_m , $\mu_{0i}H_c$ first increases slowly at $B_m < 0.5 \text{ T}$, then increases sharply at $0.6 \text{ T} < B_m < 1.5 \text{ T}$ and finally achieves saturation at $B_m > 1.5 \text{ T}$. The maximum $\mu_{0i}H_c$ is 0.69 T for the sample with $d = 1.6 \mu\text{m}$. A similar magnetizing field dependence of $\mu_{0i}H_c$ for the sample with $d = 0.6 \mu\text{m}$ was also observed. The value of $\mu_{0i}H_c = 0.79 \text{ T}$ at $B_m = 5.5 \text{ T}$ is slightly higher than the value of 0.75 T obtained at $B_m = 4.0 \text{ T}$. It can be seen from figure 3 that there is no sharp step in the magnetizing field dependence of the coercivity,

and the saturation magnetizing field required to obtain coercivity saturation is low. These results suggest that the coercivity of the $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ powders is mainly controlled by a nucleation mechanism, as observed for $\text{Sm}_3(\text{Fe}, \text{Ti})_{29}\text{N}_y$ [8] and 2:17 nitrides [12]. In addition, the small step appearing in the curve at low fields in figure 3 may result from some single-domain particles, which exist in the sample with $d = 1.6 \mu\text{m}$ from optical microscopy observation. Therefore the coercivity of the $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ powders may be partly controlled by the single-domain mechanism.

Figure 4 shows hysteresis loops of the sample with $d = 1.6 \mu\text{m}$ at 4.2 and 293 K for $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$. $(BH)_{max}$, B_r and $\mu_{0i}H_c$ at 4.2 K obviously increase in comparison with those at 293 K, which change from 84.8 kJ m^{-3} , 0.80 T and 0.63 T at 293 K to 136 kJ m^{-3} , 0.88 T and 2.14 T, respectively, at 4.2 K. The considerable increase in $(BH)_{max}$, B_r and $\mu_{0i}H_c$ at 4.2 K results in enhancements of the saturation magnetization and the magnetic anisotropy at low temperatures, which are similar to the data for $\text{Sm}_3(\text{Fe}, \text{Ti})_{29}\text{N}_y$ [7].

In conclusion, the intrinsic coercive force $\mu_{0i}H_c = 0.79 \text{ T}$ at 293 K and 2.14 T at 4.2 K have been found for the novel $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ compound. Values of $(BH)_{max} = 104.8 \text{ kJ m}^{-3}$ at 293 K and 136 kJ m^{-3} at 4.2 K are obtained for $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$. The magnetizing field required to obtain coercivity saturation is about 1.5 T for this nitride magnet. These studies show that $\text{Sm}_3\text{Fe}_{24}\text{Cr}_5\text{N}_y$ is a promising candidate for permanent magnet materials.

Acknowledgments

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References

- [1] Shcherbakova Ye V, Ivanova G V, Yermolenko A S, Belozherov Ye V and Gaviko V S 1992 *J. Alloys Compounds* **182** 119
- [2] Collocott S J, Day R K, Dunlop J B and Davis R L 1992 *Proc. 7th Int. Symp. on Magnetic Anisotropy and Coercivity in Rare-Earth Transition Metal Alloys (Canberra, July 1992)* pp 437
- [3] Li H S, Cadogan J M, Davis R L, Margarian A and Dunlop J B 1994 *Solid State Commun.* **90** 487
- [4] Hu Z and Yelon W B 1994 *Solid State Commun.* **91** 223
- [5] Yang F M, Nasunjilegal B, Wang J L, Pan H Y, Qing W D, Zhao R W, Hu B P, Wang Y Z, Liu G C, Li H S and Cadogan J M 1994 *J. Appl. Phys.* **76** 1971
- [6] Hu B P, Liu G C, Wang Y Z, Nasunjilegal B, Tang N, Yang F M, Li H S and Cadogan J M 1994 *J. Phys.: Condens. Matter* **6** L 595
- [7] Hu B P, Liu G C, Wang Y Z, Nasunjilegal B, Zhao R W, Yang F M, Li H S and Cadogan J M 1994 *J. Phys.: Condens. Matter* **6** L197
- [8] Hu J F, Yang F M, Nasunjilegal B, Zhao R W, Pan H Y, Wang Z X, Hu B P, Wang Y Z and Liu G C 1994 *J. Phys.: Condens. Matter* **6** L411
- [9] Wang Y Z, Li H S, Han X F, Liu G C, Hu B P, Yang C P, Tang N and Yang F M 1997 *J. Appl. Phys.* to be published
- [10] Suzuki S, Suzuki S and Awasaki M K 1995 *IEEE Trans. Magn.* **31** 3695
- [11] Luborsky F E 1961 *J. Appl. Phys.* **32** 171 S
- [12] Hu B P, Rao X L, Xu J M, Liu G C, Wang Y Z, Dong X L, Zhang D X and Cai M 1993 *J. Appl. Phys.* **74** 489