JOURNAL OF MATERIALS SCIENCE **39** (2004) 2231–2232

Structural and magnetic studies in $Nd_{60}Fe_{20}Al_{10}Co_{10}$ amorphous powder made by mechanical alloying

HUI XU, XIAOCHUAN LU, XIAOHUA TAN, YUANDA DONG
Institute for Material Research, Shanghai University, Shanghai 200072, People's Republic of China
E-mail: xuhui01@public6.sta.net.cn

Bulk amorphous Nd-Fe-Al system magnets, first reported in 1996 [1, 2], have attracted a number of studies due to the appreciable room temperature coercivity (up to 318.5 kA/m). These studies revealed that the solidification at different cooling rates leads to different magnetic behavior. The mechanism that determines the hard magnetic behavior of the bulk amorphous samples containing more than 50 at.% Nd and soft magnetic behavior of melt spun amorphous samples with the same composition are not very well understood. Most of the development has been on the bulk amorphous alloys and ribbons [3–5]. However, mechanical alloying is an effective and important method for making amorphous alloys. In this letter, we investigated the formation of Nd-base amorphous alloy by mechanical alloying, and studied the change of structural and magnetic properties of the Nd-base alloy during the milling process.

Commercial elemental powders of Nd, Fe, Al, Co were used as starting materials. The as-received powders had a purity of 99.9 wt% or better and particle size smaller than 75 μm. The mixture of elemental powders having the desired composition of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ together with hardened steel balls was loaded in a hardened steel vial under purified argon atmosphere. A ball-to-powder weight ratio of about 20:1 was employed. The structures of powder milled for different time (2 h, 5 h, 10 h, 20 h, 50 h, and 100 h) were measured by X-ray diffraction (XRD) in a Siemen D5000X diffractometer using Cu K_α radiation. Heat treatment of the samples was carried out in a vacuum furnace at 523, 667 and 723 K for 10 min. Magnetic measurements under an applied field of 1.8 T at room temperature were carried out using a vibrating sample magnetometer (VSM).

X-ray diffraction spectra for the mixture powder $Nd_{60}Fe_{20}Al_{10}Co_{10}$ are shown in Fig. 1 as a function of milling time. The intensities of diffraction peaks from Al decrease quickly with increasing milling time. After milling for 2 h, the diffraction peaks of Al disappear, and a small amount of an amorphous phase is observed. The intensities of diffraction peaks from Nd and Co decrease with increasing milling time, then disappear at 20 h. After milling for 20 h, a broad diffuse diffraction maximum at $2\theta = 25$–$35\ ^\circ$ is the main feature in all these patterns, suggesting that the alloyed powders are predominantly amorphous. However, crystalline peak of α-Fe phase is still identifiable in the patterns of all alloys even after milling for 100 h.

Fig. 2 shows the milling time dependence of magnetic properties of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ mixture powder. As the milling time increases, the coercivity, H_{cj}, of the mixture powder increases significantly and reaching a high value at 20 h, H_{cj} remains at this value with increasing milling time. In contrast over most of the time period the saturation magnetization of the powder mixture decreases gradually and reaches a constant value at milling 100 h. The hysteresis loops of the MA sample for 100 h is shown in Fig. 3. It exhibited hard magnetic behavior with coercivity of 43 kA/m. Results of Fig. 1 shows that the mixture powder after milling for 100 h consisted of an amorphous phase and α-Fe. α-Fe is soft magnetic, so the high coercivity of the mixture powder must come from the amorphous phase.

Fig. 4 shows the XRD patterns of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ powder which had been milled for 100 h after annealing at various temperatures for 10 min. After annealing at 523 K, a small amount of Nd phase was observed. After

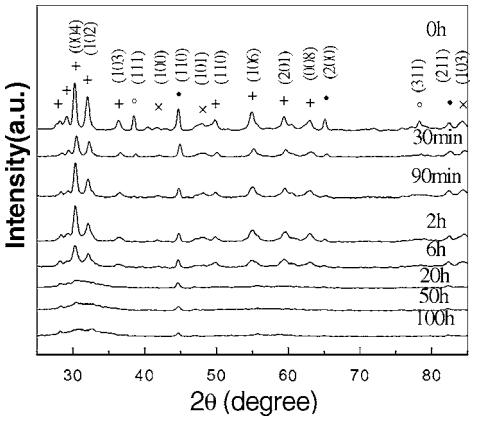

Figure 1 X-ray diffraction patterns of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ samples milled for various times. +, Nd; ●, α-Fe; ×, Co; ○, Al.

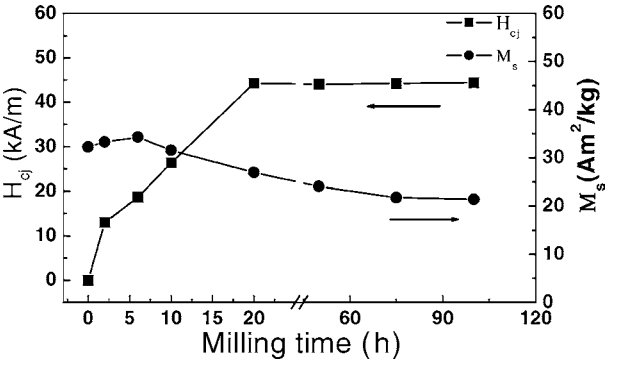

Figure 2 Curves of the milling time dependence of magnetic properties of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ amorphous powder.

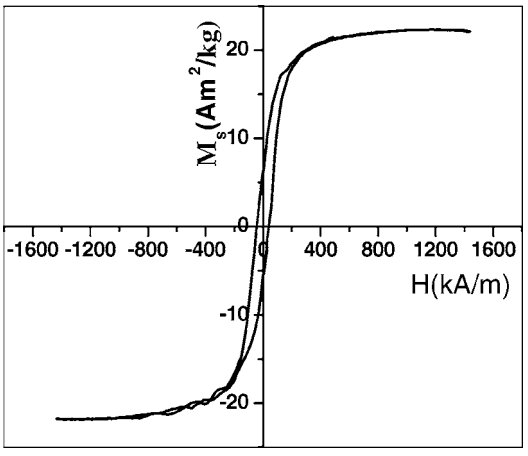

Figure 3 The hysteresis loops of the sample of milling for 100 h.

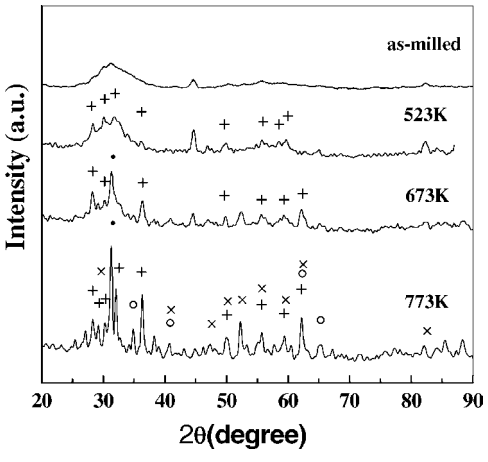

Figure 4 Results of XRD for $Nd_{60}Fe_{20}Al_{10}Co_{10}$ amorphous powder annealed at different temperatures for 10 min. +, Nd; ●, unknown phase; ×, AlCo; ○, Co_2Nd.

annealing at 673 K, an unknown phase was obtained which is assumed to be a Nd phase. After annealing at 773 K, the sample was completely crystallized; Nd, AlCo, Co_2Nd and an unknown phase are formed.

Fig. 5 shows the annealing temperatures dependence of magnetic properties of $Nd_{60}Fe_{20}Al_{10}Co_{10}$ mixture powder that had been milled for 100 h. With increasing annealed temperature, the coercivity and saturation magnetization decrease gradually. After annealing at

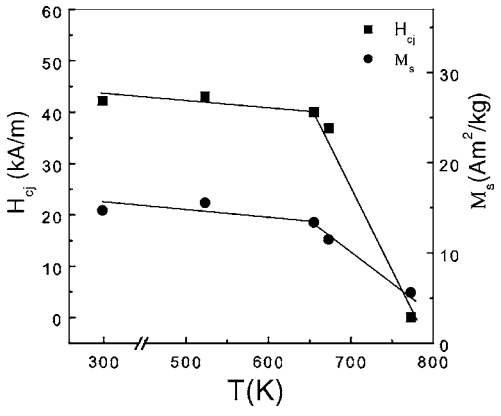

Figure 5 Curves of the annealing temperatures dependence of magnetic properties of amorphous powder which were milled for 100 h.

773 K, the magnetic properties of mixture powder are significantly degraded.

Based on the results of Figs 4 and 5, we supposed that the hard magnetic properties of the mixture powder come from the amorphous phase. When a small amount of crystalline phase was obtained, the magnetic properties of the mixture powder decrease slightly. When the sample was crystallized completely, the magnetic properties of the sample were unacceptable.

Acknowledgment

The financial support of the Nation Nature Science Foundation of China (Grant Nos. 50001007 and 50031010) is gratefully acknowledged.

References

1. A. INOUE, T. ZHANG, W. ZHANG and A. TAKEUCHI, *Mater. Trans. JIM* **37** (1996) 99.
2. A. INOUE, T. ZHANG, A. TAKEUCHI and W. ZHANG, *ibid.* **37** (1996) 63.
3. G. J. FAN, J. ECKERT, W. LOSER, S. ROTH and SCHULTZ, *Mater. Sci. Forum* **343–346** (2000) 97.
4. H. Z. HONG, Y. LI and J. DING, *J. Magn. Magn. Mater.* **217** (2000) 65.
5. B. C. WEI and W. H. WANG, *et al.*, *Mater. Sci. Eng.* A **334** (2002) 307.

Received 13 May
and accepted 8 October 2003