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Structural imaging of mechanically alloyed remanence-enhanced $Sm_2Fe_{17}N_3/\alpha$-Fe

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Abstract

The nanostructure of mechanically alloyed $Sm_2Fe_{17}N_3/\alpha$-Fe mixtures was studied using high resolution SEM, TEM and AFM. The remanence and coercivity are presented as a function of Sm content together with SEM micrographs obtained by a delicate etching procedure. TEM examination shows that both phases have grains of similar sizes which are in the range 20–30 nm.

Keywords: Mechanical alloying; Nanostructures; Remanence enhanced

Numerical micromagnetic calculations show that the magnetic properties of isotropic nanostructured permanent magnets are extremely sensitive to the microstructure [1]. To achieve a significant enhancement of the remanence and to preserve a high coercivity the grain size should be about twice the domain wall width of the hard phase or ≈ 10 nm. The calculated dependences of remanence and coercivity on grain size are in agreement with experimental results obtained by Davies et al. [2] for the $Nd_2Fe_{14}B/\alpha$-Fe system and by Ding et al. [3] for the $Sm_2Fe_{17}N_3/\alpha$-Fe system. The nanocomposite materials produced to date have grain sizes deduced from TEM to be in the range 20–30 nm in both Nd–Fe–B [2] and Sm–Fe–N systems [3]. According to the calculations, a reduction in grain size from 20 to 10 nm for a 60/40 nanocomposite mixture of $Nd_2Fe_{14}B$ and α-Fe should increase the coercivity from 0.6 to 1.2 T and the remanence from 1.4 to 1.5 T. Here we discuss observation of two-phase nanostructures in the Sm–Fe–N system by SEM and describe a simple procedure for preparation of samples for TEM.

Details of material preparation are given in Refs. [4]. Particle size analysis of these two-phase nanocomposites shows that over 50% of the as-milled material is in the 125–250 μm size bracket, an order of magnitude larger than in as-milled single phase Sm–Fe. Powder mixed with Cu-doped epoxy, ground, polished and examined in an optical microscope showed these 125–250 μm particles made up of agglomerates of much smaller particles. SEM analysis of powder sprinkled on silver paint revealed, with increasing magnification, fractal type agglomerates of particles down to 1 μm in size which was the resolution limit of the JEOL 5200 (SEM) used. To see the granular nanostructure a high resolution Hitachi S-4000 (FEG-SEM) and a Philips CM20 (TEM) were used. The as-polished samples showed no features when viewed in the high resolution SEM in secondary or backscattered modes. However, brief etching for 5 s in Villelas agent preferentially removed the Fe grains revealing the nanostructure of the $Sm_2Fe_{17}N_3$ grains from which the grain sizes could be estimated. Due to the agglomerate nature of these samples it was possible to use a very simple preparation technique to examine samples in the TEM. Powders were crushed under methanol and the mixture placed in an ultrasonic bath for a few minutes to break up any remaining agglomerates. Allowing the solution to settle for a few minutes

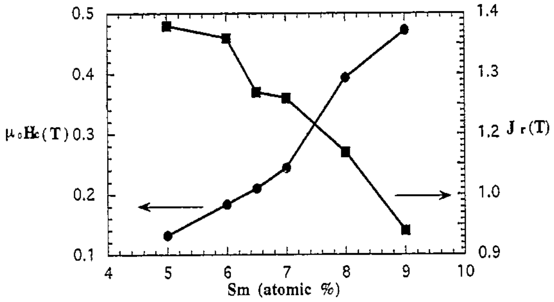

Fig. 1. Coercivity and remanence as a function of Sm content in two-phase $Sm_2Fe_{17}N_3/\alpha$-Fe.

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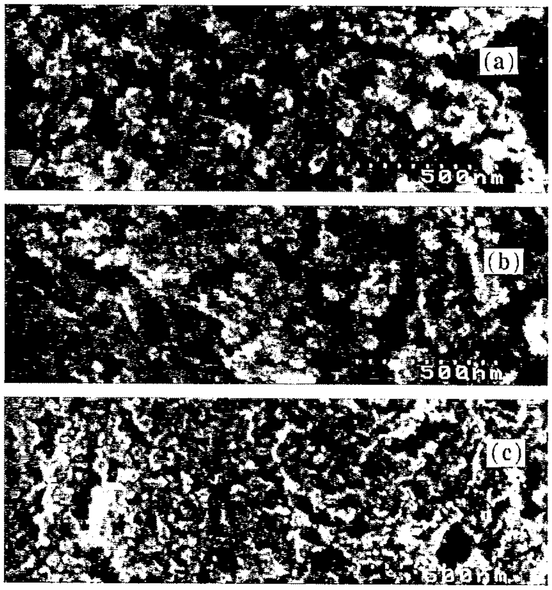

Fig. 2. High resolution scanning electron micrographs of $Sm_2Fe_{17}N_3/\alpha$-Fe nanocomposites for starting compositions (a) Sm_5Fe_{95}, (b) Sm_7Fe_{93} and (c) Sm_9Fe_{91}.

resulted in $500 \times 300 \times 80$ nm agglomerates suspended near the top of the solution which were trapped on carbon films placed on copper grids dipped into this solution. The composition was verified by EDX in TEM and phases were identified by electron diffraction.

The dependence of coercivity and remanence on the

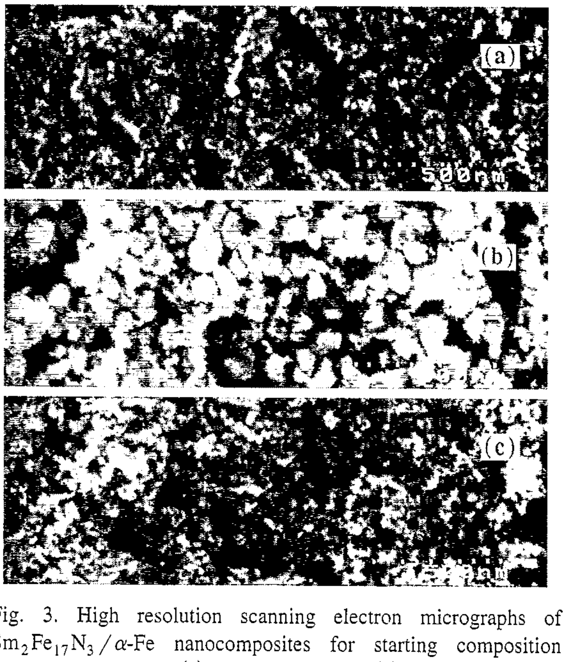

Fig. 3. High resolution scanning electron micrographs of $Sm_2Fe_{17}N_3/\alpha$-Fe nanocomposites for starting composition Sm_7Fe_{93} annealed at (a) 575°C for 30 min, (b) 625°C for 1 h and (c) with 2 at% Zr annealed also at 625°C for 1 h.

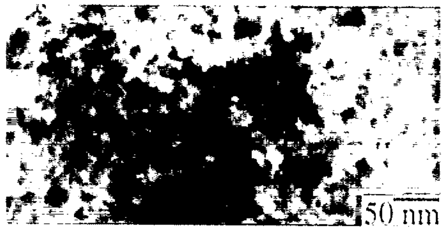

Fig. 4. Transmission electron micrograph of Sm_2Fe_{17}/α-Fe nanocomposite showing grains of diameter 20-30 nm.

amount of soft phase present (shown in Fig. 1) is similar to that obtained in other systems [2,3]. Calculations of Schrefl et al. [1] for the 20-30 nm grain sizes are in good agreement with these data. Fig. 2 shows HRSEM micrographs of Sm_xFe_{100-x} samples for $x = 5$, 7 and 9 nitrided with percentage hard phase of 43, 61 and 74, respectively. The grain size in each micrograph is in the 20-30 nm range. The annealing treatment in each case was 625°C for 10 min.

Fig. 3 shows HRSEM micrographs of samples of starting composition Sm_7Fe_{93} annealed at (a) 575°C for 30 min and (b) at 625°C for 1 h. The sample in Fig. 3c contains 2 at% Zr which inhibits grain growth and yielded optimum magnetic properties after annealing at 625°C for 1 h. The grain size in (a) is 10-20 nm but it is not fully crystallized in the Th_2Zn_{17} crystal structure, it contains some $SmFe_2$ phase. The grain size in (b) is 30-50 nm and the sample exhibits two-phase magnetic behaviour. The grain size in (c) is 10-20 nm and the material is fully crystallized but the addition of Zr reduces the remanence.

Fig. 4 shows a TEM micrograph of a sample of a 60/40 mixture of Sm_2Fe_{17} and α-Fe prepared as described above. The grain size is 20-30 nm as observed in the HRSEM. AFM of a polished 60/40 $Sm_2Fe_{17}N_3/\alpha$-Fe sample shows a 'grain' size twice as big as that observed in HRSEM and TEM, suggesting that the variation in surface topography obtained by polishing shows clusters and not individual grains.

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References

[1] T. Schrefl, J. Fidler and H. Kronmüller, Phys. Rev. B 49 (1994) 96100.

[2] H.A. Davies, A. Manaf and P.Z. Zhang, J. Mater. Eng. Perform. 2 (1993) 579.

[3] J. Ding, Y. Liu, R. Street and P.G. McCormick, J. Appl. Phys. 75 (1994) 1032.

[4] K. O'Donnell, C. Kuhrt and J.M.D. Coey, J. Appl. Phys. 76 (1994) 7068.