MAGNETIC PROPERTIES OF FINE Fe-B PARTICLES

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Abstract

The magnetic properties of fine Fe-B particles with different boron concentration and particle sizes have been studied. The powders were prepared by reducing Fe-salts with $NaBH_4$ as a reducing agent in aqueous solution. The particle size ranged from 300-600Å. X-ray diffraction patterns show that the particles are crystalline for boron concentrations less than 20 at% and amorphous for greater concentrations. The maximum coercivity and saturation magnetization obtained for these particles are approximately 1200 Oe and 160 emu/g at room temperature. The coercivities are about two orders of magnitude higher than the values reported for Fe-B metallic glasses and α-Fe.

Introduction

The magnetic properties of ultrafine particles are expected to be different than those of bulk samples. For Fe and Co particles the saturation magnetization (M_s) was reported to be 20 to 90% of that of the bulk metals depending on the size of the particles.[1] At 10 K, the coercivity (H_c) of fine Fe particles was reported to be 1540 Oe compared to the very low H_c (50 Oe) of the bulk.[2] Studies on Fe-B particles showed H_c of only 200 Oe[3] and M_s of 130 emu/g.[4] For Fe-Co-B particles the H_c reported was as high as 800 Oe.[5] All the boron containing particles were made by chemical methods.[3,4,5]

We have started a systematic study of the magnetic properties of these Fe-B particles. In particular, we are interested on the effects of size and boron concentration on the magnetic properties and on the origin of the high H_c. In this paper we will report some preliminary magnetic measurements on these particles.

Experimental

Crystalline Fe-B particles were made using a "Y" junction through which aqueous solutions of $FeCl_3$ and $NaBH_4$ were mixed in an external magnetic field of 1 kOe. The black precipitates were separated and repeatedly washed with water and acetone and then dried in nitrogen atmosphere. The maximum amount of boron that was found in the Fe-B particles was only 15% when the "Y" junction technique was used. Particles with higher boron content were made by adding $NaBH_4$ dropwise to a well stirred $FeCl_3$ solution.[6]

A SQUID magnetometer was used to measure the magnetic properties. A JOEL 100 scanning transmission electron microscope was used to examine the particle crystal structure and particle morphology. X-ray diffraction patterns were used to determine the phases present.

Experimental Results and Discussion

Figure 1 shows the x-ray diffraction patterns for different boron concentrations. It illustrates that when the boron concentration is higher than 20 at%, the particles are amorphous; otherwise they are crystalline with a bcc structure. A similar behavior was observed in Fe-B metallic glasses [7]. Figure 2 shows the TEM pictures and electron diffraction patterns for particles of boron concentration 6 at% and 27 at%. The electron diffraction patterns are consistent with the x-ray results. The particle size as determined from the TEM micrograph is found to be in the range 300-600Å.

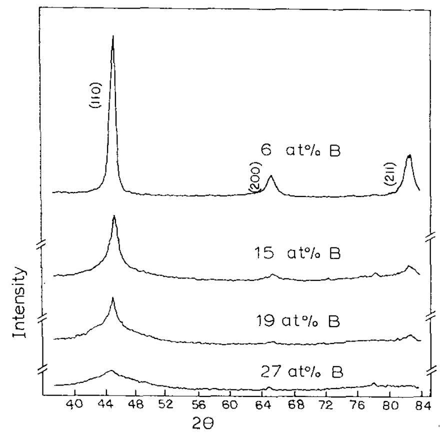

Fig. 1 X-ray diffraction patterns of Fe-B samples with different boron concentration.

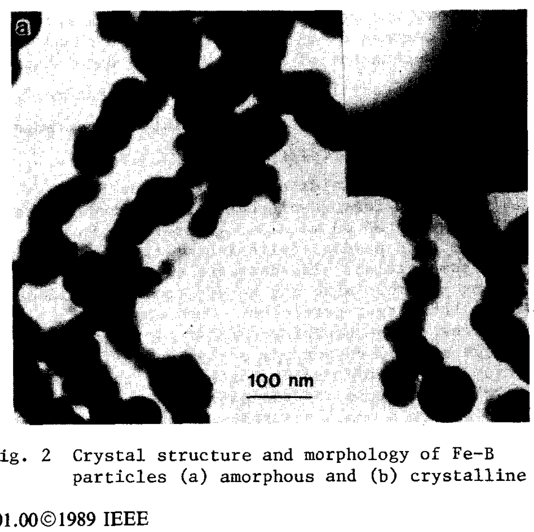

Fig. 2 Crystal structure and morphology of Fe-B particles (a) amorphous and (b) crystalline bcc.

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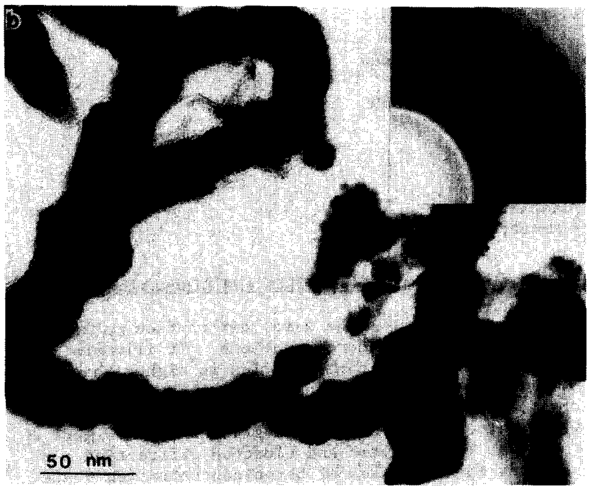

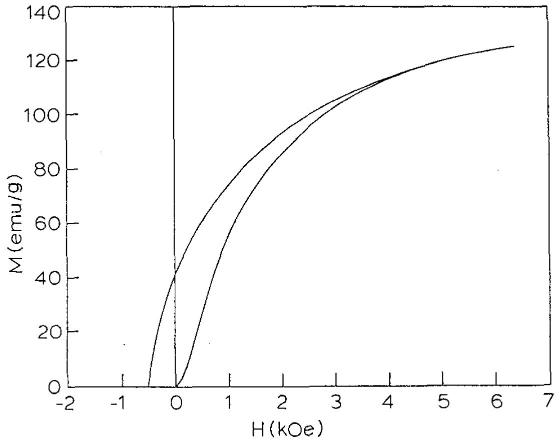

Fig. 3 Saturation magnetization (solid line) and coercivity (dashed line) as a function of temperature; circles 76.8 at% B, triangles 9.5 at% B and squares 14 at% B.

The initial magnetization and demagnetization curves of a typical sample are shown on Fig. 3. The magnetization changes steeply at low fields. Figure 4 shows M_s as a function of B concentration. M_s is found to decrease initially with B content, going through a minimum at ~15 at% and then increase again for higher B concentrations. The largest change in magnetization was observed in the smallest particles. M_s is expected to decrease with increasing B content and it is usually lower in amorphous samples than in crystalline samples [8]. The large difference between the values of the M_s for the smaller and the larger particles may be explained by oxidation effects. Smaller particles have a higher surface to volume ratio than larger particles and as a result smaller particles get more oxidized. When the B concentration is higher than 20 at%, the particles become amorphous and the particles do not oxidize as much as in the crystalline state and therefore M_s is higher. Figure 5 shows H_c as a function of B concentration with the smaller particles having larger H_c. The coercivity was found to increase with decreasing B content and go through a maximum at a B concentration around 15 at%. The large scattering of data is due to uncertainly in the size determination and various degrees of oxidation. Figure 6 shows the temperature dependence of H_c and M_s. The data show that M_s changes very little (5%) with temperature from 10 K to 300 K whereas for the same temperature range H_c changes much more (18%).

The coercive fields obtained in fine Fe-B particles are at least two orders of magnitude higher than those reported on metallic glasses and in crystalline Fe-B powders (with a bcc structure) having the same composition. The decrease of coercivity with increasing particle size may be due to the multidomain nature of the larger particles. The large coercivities can be explained either by the presence of a large surface anisotropy or by the surface oxidation layer which may be coating the particles. The lower coercivities of the amorphous powders would indicate either a smaller surface anisotropy or less oxidation. We are presently investigating the origin of these coercivities by using Mossbauer spectroscopy and other magnetic techniques.

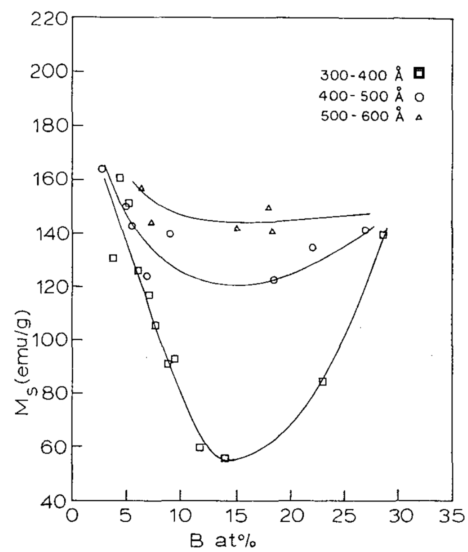

Fig. 4 Saturation magnetization as a function of boron concentration.

In summary, reasonably high H_c (1000 Oe) and M_s (160 emu/g) were obtained in fine Fe-B particles with a B-concentration of about 5 at% and a particle size in the range of 300-400 Å. The higher H_c may be due to surface anisotropy or to oxidized surface layers of the Fe-B particles.

Acknowledgments

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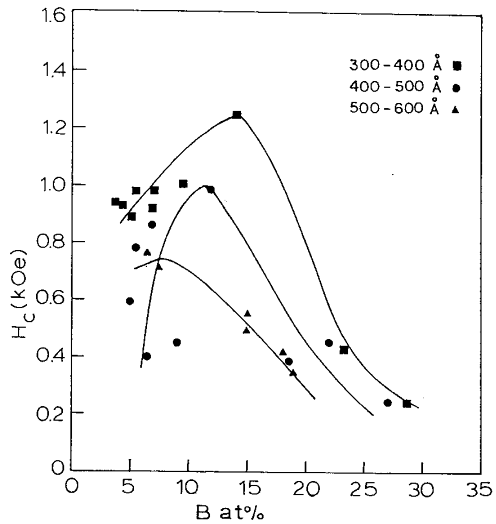

Fig. 5 Coercivity as a function of boron concentration.

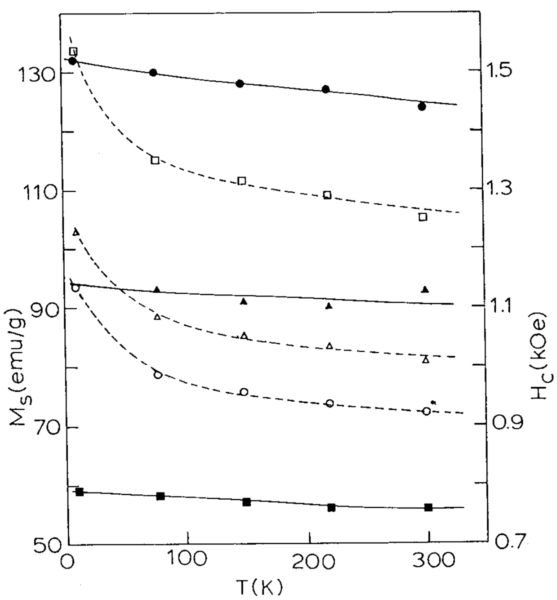

Fig. 6 Initial magnetization and hysteresis loop for a sample with 22 at% boron.

References

[1] A. Tasaki, M. Takao and H. Tokunaga, Japanese J. Appl. Phys. B271 (1974).

[2] S. Nafis, Z.X. Tang, B. Dale, K.J. Klabunde, C.M. Sorensen and G.C. Hadjipanayis, J. Appl. Phys. 64, 5835 (1988).

[3] S. Linderoth, S. Morup, C.J.W. Koch, S. Wells, S.W. Charles, J. Van Wonterghem and A. Meagher, preprint.

[4] S.G. Kim and J.R. Brock, J. Colloid and Interface Science, 116, 431 (1987).

[5] Dragieva, G. Gavrolov, D. Buchkov and M. Slavcheva, J. Less-Common Metls, 67, 375 (1979).

[6] S. Morup, private communication.

[7] C.D. Graham and T. Egami, Ann. Rev. Mater. Sci. 8, 423 (1987).

[8] F.E. Luborsky and J.L. Walter, IEEE Trans. Mag. MAC-16, 572 (1980).