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ULTRAFINE PARTICLES OF AMORPHOUS $\text{Fe}_{62}\text{B}_{38}$: A STUDY OF STRUCTURAL RELAXATION AND CRYSTALLIZATION

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Abstract. – Ultrafine particles of $\text{Fe}_{62}\text{B}_{38}$ have been produced by reduction of Fe(II) in aqueous solution using KBH_4 . Mössbauer spectroscopy and X-ray diffraction studies show that the particles are amorphous. Structural relaxation occurs at ~ 525 K and crystallization at ~ 715 K, where Fe_2B and $\alpha\text{-Fe}$ phases are formed. The particles exhibit relatively large intrinsic coercivities of 200 Oe.

1. Introduction

Amorphous alloys are normally produced as ribbons or thin films by the liquid quench or sputtering techniques [1]. Watanabe and Tanabe [2] have shown that simultaneous electroplating and chemical reaction of Ni ions with NaBH_4 in aqueous solutions results in the formation of amorphous Ni-B films. Recently it has been demonstrated that ultrafine amorphous particles with dimensions in the range 10-100 nm can be produced by reduction of transition metal salts in aqueous solution using KBH_4 or NaBH_4 [3-5]. The boron content can be varied between 11 and 42 at. % [3-7].

In this paper we present results of studies by Mössbauer spectroscopy, magnetization and X-ray diffraction measurements of as-prepared and annealed amorphous $\text{Fe}_{62}\text{B}_{38}$ particles.

2. Experimental

The particles were prepared by slowly adding 1 M FeSO_4 to 0.5 M KBH_4 in aqueous solutions with vigorous stirring. The black precipitate was washed in water and subsequently in acetone. The material was dried and passivated in a flow of Ar gas containing small amounts of oxygen.

Mössbauer spectra were obtained using a constant acceleration spectrometer with a source of ^{57}Co in rhodium. The isomer shifts are given relative to $\alpha\text{-Fe}$. Magnetic measurements were made with a vibrating-sample magnetometer. X-ray diffraction measurements were obtained using a Philips PW 1050 powder diffractometer using $\text{Co-K}\alpha$ as the X-ray radiation and a graphite monochromator mounted in the diffracted beam.

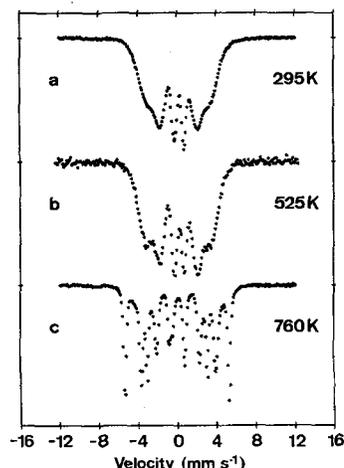


Fig. 1. – Room-temperature Mössbauer spectra of (a) as-prepared $\text{Fe}_{62}\text{B}_{38}$ particles, (b) annealed at 525 K for 4 hours, and (c) annealed at 760 K for 5 hours.

3. Results and discussion

In figure 1a is shown the room temperature Mössbauer spectrum of the as-prepared material. The spectrum exhibits very broad lines, the outer lines being broader than the inner lines reflecting a distribution of magnetic hyperfine fields. The spectrum was fitted with a sextet yielding a magnetic hyperfine field of about 20.4 T, a negligible quadrupole shift and an isomer shift of 0.16 mm s^{-1} and with a doublet with a quadrupole splitting of about 1.0 mm s^{-1} and an isomer shift of 0.39 mm s^{-1} constituting a relative area of 6 % of the Mössbauer spectrum. The doublet can

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be assigned to a Fe(III) component, presumably originating from iron oxide in the passivation layer.

A comparison of the magnetic hyperfine field with those of Fe-B amorphous alloys prepared by the liquid quench technique [8] suggests the boron content to be around 35 % (at). In accordance with this estimate a chemical analysis of the particles shows that the composition is $\text{Fe}_{62}\text{B}_{38}$.

After annealing in Ar for 4 hours at 525 K (cf. Fig. 1b) the lines become narrower and the hyperfine field increases to 20.8 T. X-ray diffraction measurements showed no significant differences between the annealed and the as-prepared samples; both give rise to a broad X-ray scattering peak. This suggests that the changes in the Mössbauer spectrum after annealing at 525 K are due to structural relaxation in the amorphous particles. Similar results have been reported from studies of amorphous ribbons [9]. The Mössbauer parameters for the doublet were not affected by the heating of the sample.

Structural relaxation in amorphous ribbons can significantly change the magnetic properties, e.g. the intrinsic coercivity and the magnetization work [10]. However, annealing the powder at 525 K for 2 hours in an Ar atmosphere only caused small changes in the magnetic properties. The saturation magnetization increased by 5 % to 117 emu/g while the intrinsic coercivity and the remanence magnetization remained at about 200 Oe and 11 emu/g, respectively. Films of similar composition exhibit coercivities 50-100 times smaller [11] than the particles prepared by the present chemical method.

After further annealing of the sample at 760 K in Ar the spectrum (Fig. 1c) can be fitted well with two magnetically split components due to α -Fe and Fe_2B [8]. This interpretation is supported by the X-ray results. Thus the particles have crystallized during the annealing at 760 K.

The magnetization, at a field of 10.55 kOe, was followed during annealing in a static Ar atmosphere with a heating rate of about 20 K/min. The results showed that crystallization takes place at 715 ± 10 K. This is in good accord with the results by Kanamura *et al.* [11] on amorphous films produced by sputtering.

4. Conclusion

Ultrafine, amorphous $\text{Fe}_{62}\text{B}_{38}$ particles have been investigated by Mössbauer spectroscopy, magnetiza-

tion, and X-ray diffraction measurements. The magnetic hyperfine fields obtained from the Mössbauer spectra are in good agreement with results on ribbons produced by the liquid quench technique. Structural relaxation occurs during annealing at 525 K. The crystallization temperature is about 715 K which is similar to that of amorphous films with a similar composition, prepared by sputtering. The results thus indicate that the amorphous structure of the particles is similar to that of films and ribbons prepared by conventional techniques.

Acknowledgements

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