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EXAFS and Mössbauer Study of the Crystallization of Fe₉₁Zr₉ Metallic Glass

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Abstract. The crystallization process of the Fe₉₁Zr₉ amorphous alloy has been followed by means of two experimental techniques, EXAFS and Mössbauer spectroscopy. X-ray absorption spectra on Fe K-edge and Mössbauer spectra of six samples with different degrees of crystallization have been measured at room temperature in order to obtain information about the structural changes that take place. From the beginning of the crystallization process it is clearly seen that a crystalline phase with a bcc-type structure appears. From the obtained data we can conclude that this phase is nearly pure bcc-Fe, with small amount of impurities in its structure.

1. INTRODUCTION

In recent years Fe_{100-x}Zr_x Fe-rich metallic glasses ($7 \leq x \leq 14$), have been attracted considerably attention because their striking magnetic properties [1, 2]. It has been observed that in this composition range the addition of 1 at.% of Zr increases the Curie temperature in 20 K [3]. On the other hand, there are not many works in the literature related with the crystallization of these alloys [4], however, the changes in behaviour that could occur on the magnetic properties due to the structural evolution during the crystallization process is actually subject of discussion. In the present work, we present a preliminary study of the structural evolution of the Fe₉₁Zr₉ sample during the first steps of the crystallization process. X-ray absorption fine structure, EXAFS, and Mössbauer spectroscopy are ideal experimental techniques to perform this study.

2. EXPERIMENTAL

A sample of nominal composition Fe₉₁Zr₉ was prepared by the melt spinning method under Ar atmosphere with cross section of about 2 mm x 20 µm. The amorphous state of the as quenched sample was checked by X-ray diffraction. In order to obtain a set of samples with different degrees of crystallization, six strips of the same sample were annealed during one hour at 623, 673, 698, 723, 748 and 773 K in a conventional furnace under Ar atmosphere. In the following we shall call the samples FeZr/aq, FeZr/623, FeZr/673, FeZr/698, FeZr/723, FeZr/748 and FeZr/773 respectively.

Room temperature X-ray absorption spectra have been recorded on Fe K-edge at 7.1 station at Daresbury Laboratory (U.K.) using a Si(111) channel monochromator and 50% of harmonic rejection. The storage ring was typically running at 2 GeV and 150 mA. ⁵⁷Fe Mössbauer transmission spectra of the samples have been recorded at room temperature in a conventional constant acceleration spectrometer using a ⁵⁷Co - Rh source. A 20 µm thick α-Fe foil has been used also to compare the results.

3. DATA ANALYSIS AND RESULTS

The absorption spectra have been analyzed following standard procedures [5]. The average absorption above the edge was fitted in the k range between 2 and 12 Å⁻¹ with a smooth spline formed by three cubic polynomials to simulate the atomic cross section. The origin of the k scale was taken at the inflection point of the absorption edge. The amplitude of the EXAFS oscillations, $\chi(k)$, decreases from the FeZr/aq sample to the FeZr/623 and increases again for the FeZr/673 sample. In the rest of the samples the amplitude increases as the crystalline fraction does. The Fourier transforms of the $\chi(k)$ functions, $\Phi(R)$, presented in fig. 1 (right), have been performed using a k^3 weight and a Hanning window function in the 2-12 Å⁻¹ k range. In the samples annealed at temperatures below 773 K, the crystalline fraction increases very slowly in the samples, hence, the change in shape of the Fourier transforms is not notorious. Furthermore, in the case of the FeZr/773 sample the percentage of crystalline fraction is clearly higher, and the shape of the Fourier transform, including the position of the mean peak, has changed towards the corresponding to the bcc-Fe, (fig. 1 right).

The fitting of the Mössbauer spectra has been performed with the NORMOS program, developed by Brand et al. [6], a program which allows a simultaneous fit of several crystalline spectra and a possible amorphous phase characterized by a distribution of hyperfine fields (B_{HF}). In fig. 1 (left) the Mössbauer spectra of the samples are represented. All the samples show a typical doublet corresponding to the Quadrupole Splitting of a paramagnetic compound. However, in all the spectra corresponding to the annealed samples the six peaks characteristic of a ferromagnetic phase appear. The hyperfine parameters of

this ferromagnetic phase correspond to the bcc Fe ones, with slight differences in the value of the B_{HF} . These values differs no more than 0.5 Tesla from the value obtained for the bcc Fe foil (33 Tesla) and evolve towards this value as the crystallization goes on. It is also clear, as in the Fourier transforms, that the amount of crystalline bcc Fe is quite low for the samples annealed below 773 K.

Figure 1: Left part, Mössbauer spectra of some of the studied samples compared with the bcc-Fe spectrum. Right part, Fourier transforms of the same samples obtained from EXAFS spectra.

4. DISCUSSION AND CONCLUSIONS

In this preliminary study of the crystallization of an Fe-rich FeZr metallic glass, it is clearly shown that the crystallization process has begun at rather low annealing temperatures (623 K), but for temperatures below 773 K the amount of crystalline phase is very low ($\approx 3\%$ in the FeZr/623 sample and $\approx 10\%$ in FeZr/748 sample). The slight differences observed in the hyperfine parameters of this partially crystallized samples, respect to the bcc Fe ones, are probably due to the small size of the crystals (about 20 nm). The large number of Fe atoms in the surface of the nanocrystals, in contact with the paramagnetic remaining amorphous phase, feel lower exchange coupling leading to a decrease in the value of the B_{HF} . The evolution of the structure in the studied samples changes drastically in the case of the FeZr/773 sample. The amount of crystalline phase reaches $\approx 80\%$ and the size of the crystals is above 0.1 μm , leading to nearly the same structure and hyperfine values than the polycrystalline bcc Fe foil corresponding ones. Magnetic measurements are needed to elucidate more accurately the changes occurring during the crystallization of these compounds.

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