

Mössbauer study of Fe–Co alloys with Cr additions synthesized by mechanical alloying

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Abstract Cr has been added to FeCo substituting 10 at.% of Co or Fe in the alloy. The alloys Fe₅₀Co₄₀Cr₁₀, Fe₄₀Co₅₀Cr₁₀ and Fe₅₀Co₅₀ were prepared by mechanical alloying for 2, 5, 10, 20, 40 and 60 h. The formation of the alloy and the incorporation of the elements have been followed by X-Ray Diffraction (XRD) and Mössbauer Spectroscopy. The kinetics of mixing occurs by incorporation of Co and Cr into the Fe structure. After prolonged milling it seems that Cr incorporates itself into both α -Fe and α -FeCo structures and a mixture of FeCoCr rich in Cr and FeCoCr rich in Co solid solutions is obtained.

Keywords Nanostructured alloys · Mechanical alloying · Mössbauer spectroscopy

1 Introduction

Mechanical alloying is widely used to produce nanophase alloys with interesting structural and magnetic properties, due to size effects and to the disorder created by the high density of defects. Among Fe-alloys, the Fe–Co system shows a high permeability and the highest magnetization saturation. In particular the Fe₅₀Co₅₀ alloy has a considerably large permeability, low magnetocrystalline anisotropy and

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Fig. 1 XRD for different milling times of $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ alloys

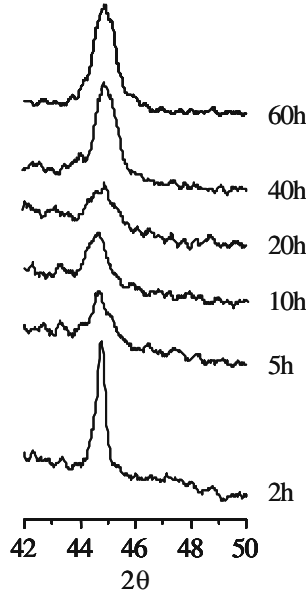
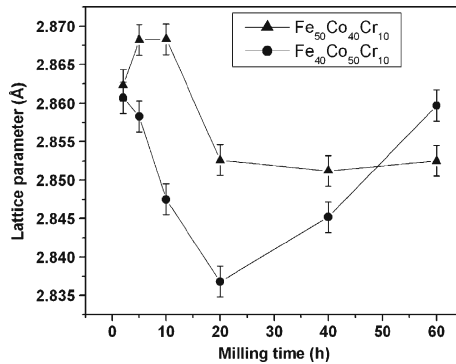


Fig. 2 Lattice parameter vs. milling time for $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ and $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ alloys



low electrical resistivity. Near equiatomic Fe–Co alloys with additions of a third element can change significantly their structural, electrical and magnetic properties [1]. The addition of Cr increases the resistivity and improves their dynamic properties, slightly decreasing the saturation magnetization by dilution of the magnetic atoms [2]. Recent work on Fe–Co–Cr has been done by Bentayeb et al. [3]; however, to our knowledge, no reports on the near equiatomic $\text{Fe}_{50}\text{Co}_{50}$ with additions of Cr, obtained by mechanical alloying have been found in the literature. In this work we present a Mössbauer Spectroscopy study of $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ and $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ alloys prepared by mechanical alloying to observe the effect of the incorporation of Cr into the Fe–Co structure. For comparison $\text{Fe}_{50}\text{Co}_{50}$ alloys were also prepared.

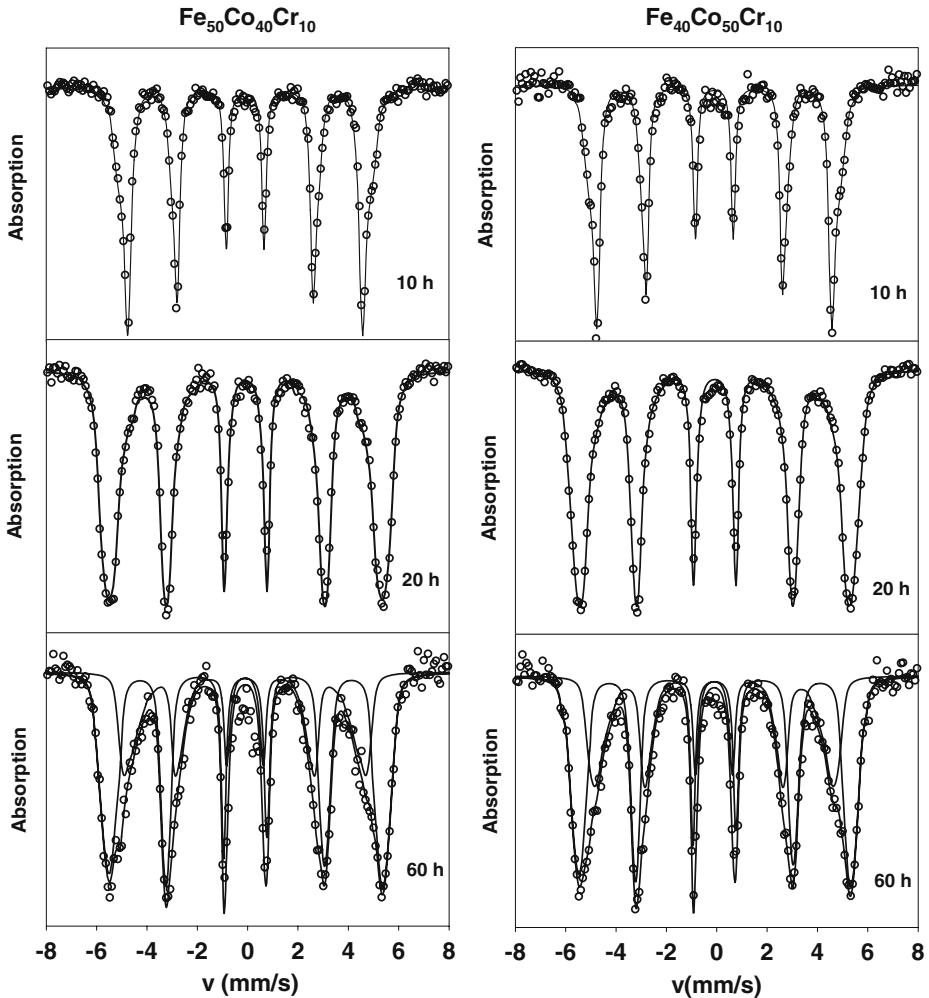


Fig. 3 Mössbauer spectra at room temperature for different periods of milling

2 Experimental

Powders of Fe, Co and Cr (ABCR Germany) with a purity of 99.8% and particle size below 50 μm were used. For each sample 10 g of powder were mixed in a Fritsch P5 planetary ball mill, equipped with hardened steel vials and balls of 10 mm diameter, with a rotational speed of 250 rpm and a ball-to-powder weight ratio of 10:1. The milling experiments and all powder handling were performed under protective argon atmosphere and the periods of milling were 2, 5, 10, 20, 40 and 60 h. Small amounts of powder were withdrawn after selected milling times for structural examinations. XRD was performed on a Siemens 5005 X-ray diffractometer, using Cu-K α (Ni filter) operating at 40 keV and 20 mA. ^{57}Fe Mössbauer spectra were obtained with a spectrometer running in the triangular symmetric mode for the velocity in

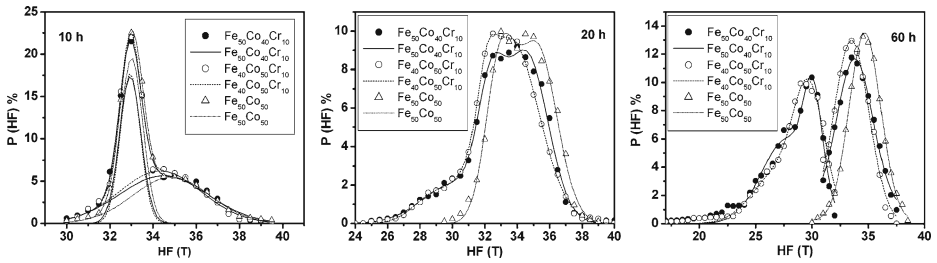


Fig. 4 Hyperfine field distribution for 10, 20 and 60 h of milling times

a transmission geometry using a ^{57}Co source in a Rh matrix. The experimental data were fitted using a distribution of hyperfine fields (DHF) and this distribution fitted with Gaussian functions in order to obtain the mean values of the hyperfine fields HF for each alloy. The isomer shifts IS are given relative to metallic iron.

3 Results and discussion

In Fig. 1 are shown the XRD for the main diffraction peak of the $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ alloys milled for different periods. Similar diffraction patterns are obtained for other alloys. The variation of lattice parameter vs. milling time is shown in Fig. 2. The lattice parameters for these two alloys reached the lowest value after 20 h of milling, when maximum strain was observed (results to be published). For $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ the lattice parameter decreases from a value near to 2.863 to 2.852 Å, after 20 h of milling and remains constant up to 60 h of milling. For the $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ alloy after 2 h of milling the lattice parameter value is 2.862 Å and progressively decreases with milling time to 2.837 Å after 20 h, and then increases to 2.860 Å after 60 h of milling, probably due to an increase in the degree of order with prolonged milling.

The decrease in the lattice parameter may be due to the allotropic transformation, of hcp Co to fcc Co, induced by deformation. For all the alloys the peak broadens with milling, suggesting a decrease in the grain size. After 60 h of milling all the alloys reached a grain size between 2 and 5 nm.

The Mössbauer spectra at room temperature of $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ and $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ alloys for milling periods of 10, 20 and 60 h are shown in Fig. 3. The HFD of both alloys were plotted together with that of the FeCo alloy for comparison and can be observed in Fig. 3 for the same milling times. For both alloys the HFD for 10 h of milling have been fitted with two Gaussian functions, Fig. 4, giving for both alloys a $\text{HF}_1 = 33.0$ T, corresponding to Fe-bcc that has not reacted yet and a $\text{HF}_2 = 34.5$ T for $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$, and a $\text{HF}_2 = 34.3$ T for $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ which correspond to an FeCo alloy [4], suggesting that diffusion of Cr is slower than that of Co.

The HFD after 20 h of milling could indicate that Cr starts to diffuse into the crystalline structure in both the Fe-bcc and the Fe-Co bcc. In this case three Gaussian functions fit best, Fig. 3, giving for $\text{Fe}_{50}\text{Co}_{40}\text{Cr}_{10}$ $\text{HF}_1 = 30.2$ T, $\text{HF}_2 = 32.5$ T, $\text{HF}_3 = 34.6$ T and IS = 0.03 mm/s, and for $\text{Fe}_{40}\text{Co}_{50}\text{Cr}_{10}$ $\text{HF}_1 = 28.9$ T, $\text{HF}_2 = 32.1$ T, $\text{HF}_3 = 33.6$ T and IS = 0.03 mm/s.

After 60 h of milling, the spectra for both alloys can be fitted with two HFD since it seems that there are two separated compounds with different values of isomer shift.

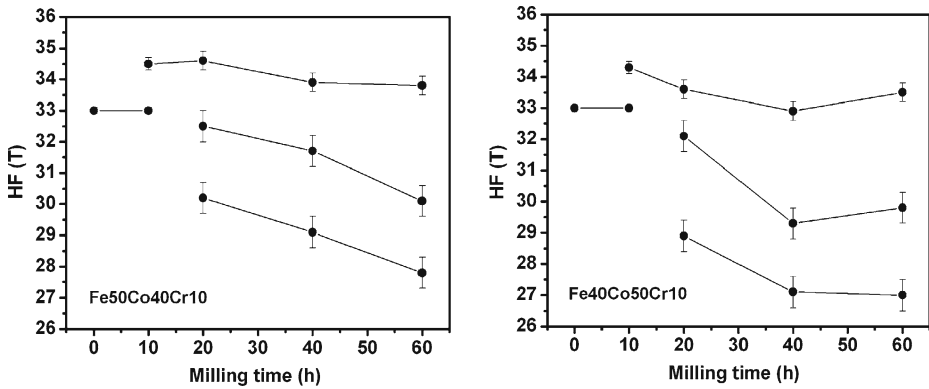


Fig. 5 Hyperfine field vs. milling time for Fe₅₀Co₄₀Cr₁₀ and Fe₄₀Co₅₀Cr₁₀ alloys

For Fe₅₀Co₄₀Cr₁₀ we obtained HF₁ = 27.8 T and HF₂ = 30.1 T with an IS = 0.00 mm/s and HF₃ = 33.8 T with an IS = 0.026 mm/s. For Fe₄₀Co₅₀Cr₁₀ we got HF₁ = 27.0 T and HF₂ = 29.8 T with an IS = 0.00 mm/s and HF₃ = 33.5 T with an IS = 0.026 mm/s. For comparison the Fe₅₀Co₅₀ alloy gave us a HF = 34.7 T and IS = 0.042 mm/s. The first two values of HF correspond to a FeCr alloy [5], with Co additions, and the third value corresponds to a FeCoCr alloy, since the additions of Cr in FeCo decreases the HF.

In Fig. 5 are displayed the HF vs. Milling time obtained from Mössbauer spectra. It is interesting to note that the alloys with more amount of iron have a HF higher than the alloys with lesser amount of iron, and that the incorporation of Cr into the structure seems to be slower and decreases the HF. The diffusion coefficient of Co in α -Fe at high temperatures is around $6.91 \times 10^{-4} \text{ m}^2/\text{g}$ and the diffusion coefficient of Cr in α -Fe at high temperatures is $2.53 \times 10^{-4} \text{ m}^2/\text{g}$ what agrees with these results.

4 Conclusion

The mixing of Fe, Co and Cr powders by mechanical alloying suggests that Cr and Co diffuse into the α -Fe, but Cr diffuses more slowly than Co. For both alloys after 10 h of milling α -Fe remains unreacted and a FeCo alloy is obtained. After 60 h Cr incorporates into both α -Fe and α -FeCo structures and a mixture of FeCoCr rich in Cr and FeCoCr rich in Co alloys seems to be obtained.

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