

Study of Fe–Ni alloys produced by mechanical alloying

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Abstract

Fe_{1-x}Ni_x samples with 0.225 ≤ x ≤ 0.400 were prepared by mechanical alloying. XRD studies showed that this group of samples presents the bcc and fcc phases in all the composition ranges. The variation of the lattice parameter, *a*, of the two phases as a function of the Ni content, remains independent of the composition. In this case all Mössbauer spectra were fitted using a hyperfine field distribution (HFD). Samples between 22.5 and 32.5 at% Ni present a paramagnetic site. For sample with 25 at% Ni, it was used an additional sextet to obtain a good fit. From the Mössbauer fitting in the field distributions, besides the field associated to the bcc and fcc microcrystalline phases (between 30 and 33 T), additional peaks at smaller fields can be noticed (between 10 and 27 T). These small fields can be associated with the contribution of those Fe magnetic sites localized in the surface and interfaces of the grains. This contribution is postulated as a consequence of the nanometer size of the bcc and fcc grains obtained by DRX.

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1. Introduction

The use of high-energy milling and of other new technological methods of preparation allows powdered samples with different structures and novel properties to be obtained. This is the current field of nanotechnology, which gives rise to nanostructured materials, as example Fe–Ni alloys [1–3]. Due to their small grain size, these materials are characterized by the rather high number of atoms located in the grain boundary. Also, they are very interesting from the magnetic point of view because the grain size approaches that of a magnetic domain, offering thus the possibility to eliminate the influence of the domain walls. It is well established that the intermetallic compounds prepared by mechanical alloying (MA) have a high structural disorder and are unstable [2]. These factors implied new physical properties, which are very different compared to those of massive materials. For this reason, several studies have been carried out investigating the structural and magnetic properties by alloys prepared by MA [3–5].

2. Experimental method

Pure Fe and Ni powders were used as the starting materials. Fe_{1-x}Ni_x samples with 0.225 ≤ x ≤ 0.400 were prepared by MA, using a planetary ball mill. The ball mass-to-powder mass (BM/PM) ratio was about 10:1. The milling time used was 10 h. The final products were characterized by X-ray diffraction (XRD) and ⁵⁷Fe Mössbauer spectroscopy (MS). For Mössbauer calibration a foil of α-Fe was used. The XRD patterns were refined by using the MAUD program [7] while the MS were fitted by using the MOSFIT program [8].

3. Results and discussion

X-ray patterns reveal the presence of both bcc and fcc crystalline phases, whatever the composition is. Fig. 1 shows the variation of the volume phase versus Ni content, as estimated from the Rietvelt refinement of the X-ray patterns. This system is thus comparable to the equilibrium thermodynamic phase diagram, where the phase coexistence extends across a large composition range [4]. The lattice parameter of each of the two phases remains nearly constant in all composition ranges (*a*_{bcc} ≈ 2.86 Å,

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$a_{fcc} \approx 3.59 \text{ \AA}$), because of the similar atomic sizes of Fe and Ni atoms: indeed, neither the substitution of Ni by Fe atoms in the fcc lattice of Ni, nor that of Fe by Ni atoms, the bcc lattice of the Fe does alter the crystalline cell size.

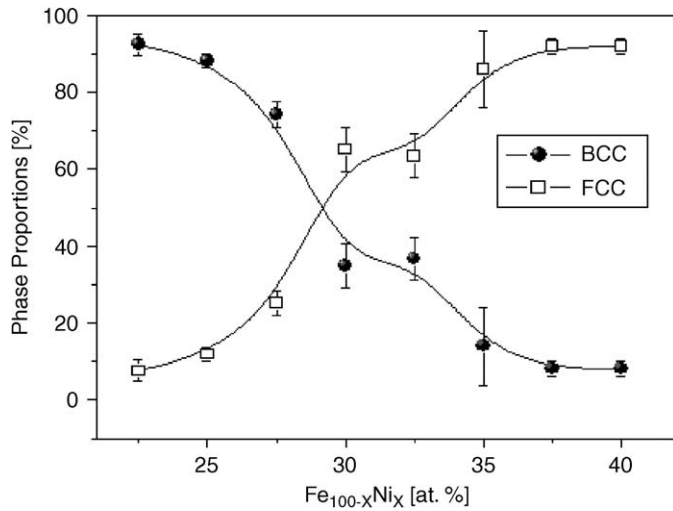


Fig. 1. bcc and fcc volumetric phase proportions versus Ni content.

The grain size of the fcc phase which varies between 14 and 22 nm is slightly larger than that of the bcc phase that varies between 6 and 15 nm. This difference is a consequence of the fragile and ductile characters of bcc and fcc phases, respectively. XRD clearly evidences the nanostructured character of the alloys obtained by AM.

Fig. 2 shows typical MS and their corresponding hyperfine field distributions (HFDs) at room temperature of some samples. In this case all the spectra were fitted using a magnetic HFD. In addition, samples between 22.5 and 32.5 at% Ni present a paramagnetic site. For sample with 25 at% Ni an additional sextet was used to obtain a good fit. The alloys with 22.5, 25 and 27.5 at% Ni show MS with narrow peaks and narrow HFDs. They have a mean hyperfine field equal or higher than 33 T, typical of bcc microcrystalline Fe–Ni melted disordered alloys [6]. Restrepo et al. [6] also found, for fcc microcrystalline alloys, distributions with high probabilities around fields between 30 and 33 T. The distributions for alloys above 27.5 at% Ni are broader, indicating a wide distribution of hyperfine field around 30 T and lower in some cases. These results indicate that a mixture of two magnetic phases exists (bcc + fcc), in agreement with XRD results.

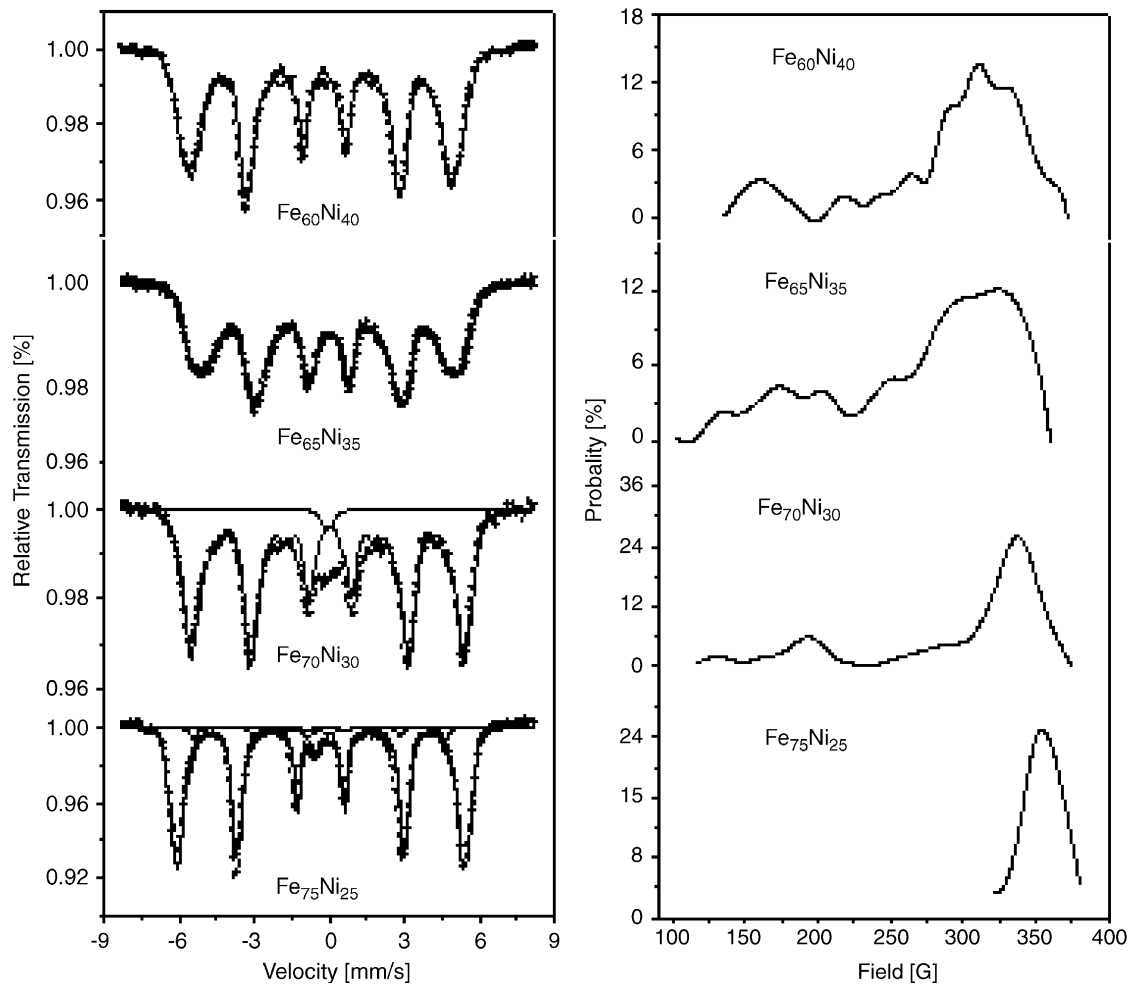


Fig. 2. 300 K Mössbauer spectra and their corresponding hyperfine field distributions.

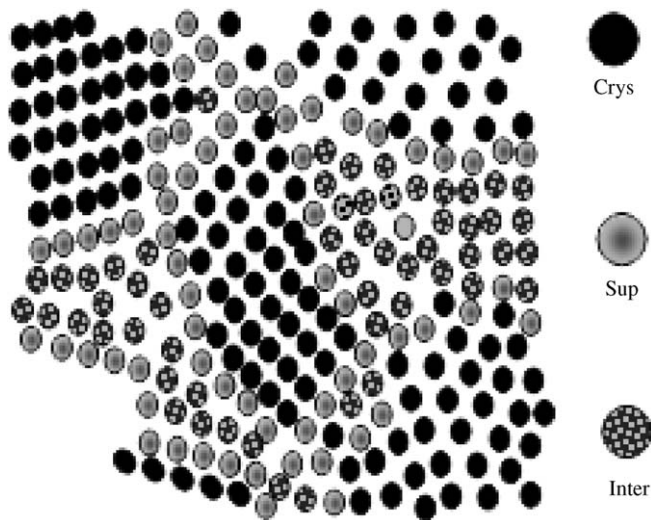


Fig. 3. Transversal schematic section of nanocrystalline material prepared by MA.

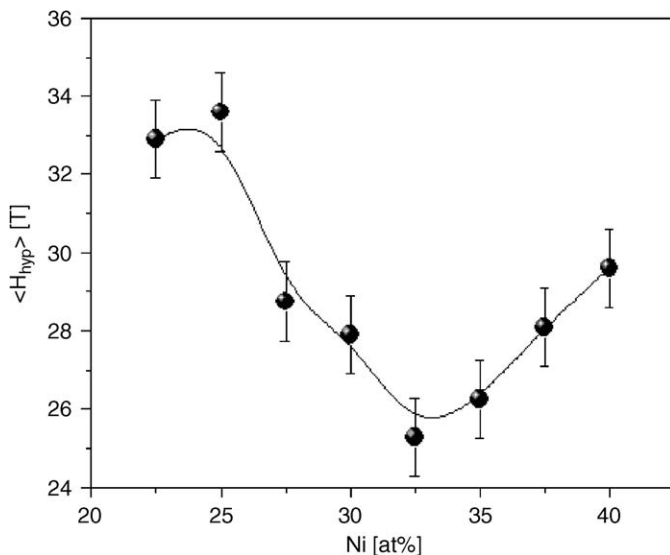


Fig. 4. Mean hyperfine field in function of Ni concentration.

Additional peaks at smaller fields (between 10 and 27 T) can be associated to the contribution of the Fe magnetic sites localized either at the periphery of grains and/or at the grain boundaries. This contribution is postulated as a consequence of the nanometer size of the bcc and fcc grains proved by DRX. Fig. 3 presents an outline of the possible nanostructure which can be present in these alloys. Fig. 4 shows an anomaly in the mean hyperfine field, which is typical of the Fe–Ni alloys around the Invar composition. A binomial theoretical model is now in progress in order to simulate the HFD and to prove the contribution of the surface and interface of the grains.

Acknowledgments

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