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Anomalous structural and magnetic behaviour of Fe–25 at.% Ni alloy due to mechanical strain

Brajesh Pandey and H C Verma¹

Department of Physics, Indian Institute of Technology, Kanpur 208 016, India

E-mail: hcoverma@iitk.ac.in

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Abstract

Mechanical strain was induced in Fe–25 at.% Ni alloy by filing the ingots into powder form. The phase transformations induced by the deformation thus caused and subsequent annealing were studied by x-ray diffraction, magnetization measurement and Mössbauer spectroscopy. It is concluded that around one-third of the Fe atoms became segregated from the Fe–Ni alloy due to filing. Annealing of the filed powder led to recombination of this segregated Fe to form an apparently body-centred cubic (bcc) phase of Fe–Ni which shows little magnetic ordering at room temperature in magnetization measurements or in Mössbauer spectroscopy. The single-line Mössbauer spectrum of this phase splits into a six-line pattern below 100 K.

1. Introduction

Fe–Ni alloys are of great interest due to their magnetic and mechanical properties. These alloys are used industrially for their high saturation magnetization, low coercivity and good anticorrosion properties. This system also presents a variety of alloy properties such as those of Invar [1] and Permalloy [2]. Many researchers have investigated the effect of grain boundaries on martensitic transformation behaviour [3–6], but the exact nature of such boundaries has not been identified yet. Fe–Ni alloys in the range 25–35% Ni show specially rich phase behaviour. While 25% Ni will be close to martensitic transformations, 35% Ni gives Invar properties.

Fe and Ni are essential constituents of a large number of meteorites, the parent bodies of which were cooled at an extremely slow rate. This has allowed researchers to discover Fe–Ni phases that could not have been obtained in the laboratory.

Mössbauer spectroscopy has played a central role in the study of Fe–Ni alloys, and especially in meteorites. In particular, Mössbauer measurements gave the first conclusive evidence [7–9] for the existence of a ferromagnetic atomically ordered Fe–Ni (50 at.% Ni) phase that was later called tetrataenite [10]. Mössbauer spectra of taenite lamellae

¹ Author to whom any correspondence should be addressed.

Table 1. Sample codes of Fe–25 at.% Ni alloys studied and their saturation magnetization M_s .

S.N.	Sample description	Sample code	M_s (A m ² kg ⁻¹)
1	As-melted ingot, cut into pieces	FENI-c	138.4
2	FENI-c homogenized at 900 °C for 100 h	FENI-h	16.2
3	Powder filed from FENI-c	FENI-f	173.2
4	Powder filed from FENI-h	FENI-hf	172.9
5	FENI-f powder annealed at 800 °C for 100 h	FENI-fa	15.8

from octahedrites [7–9, 11–13], ataxites [14–19] and the metal fractions of Fe–Ni-bearing chondrites [20, 21] also show a single-line paramagnetic contribution at room temperature (RT) that is referred to as the ‘paramagnetic phase’ or ‘paramagnetic γ -phase’. The low-Ni phase crystallizes in a bcc structure in which Fe and Ni atoms occupy lattice positions randomly. For the bcc Fe–Ni alloys, the hyperfine magnetic field (B_{hf}) increases by 1.22 T by replacement of one nearest-neighbour (nn) Fe atom by a Ni atom whereas it decreases by 0.59 T when a Ni atom replaces a next-nearest-neighbour (nnn) Fe atom.

It is known that cold working on alloys introduces strain in the crystalline structure that often leads to metastable phases not available in normal conditions [22–27]. For example, it was established that filing Fe₇₂Al₂₈ in the DO₃ phase leads to randomization of atomic ordering [22–24]. There are several studies on cold working, such as rolling, of Fe–Ni alloys [25–27]. In order to look for phase changes due to even more severe cold working, we have studied the effect of extracting powder from the ingot by filing it with a diamond coated file. In the present work XRD, magnetization measurement and Mössbauer studies are used to investigate the effect of filing on Fe–Ni 25 at.% alloy. The kinds of information derived from the above techniques are complementary, and collectively they reveal some information about the mechanism of the deformation process.

2. Experimental procedures

99.99% pure metal powders of Fe and Ni were mixed in stoichiometric proportion for Fe–25 at.% Ni alloy and melted in a non-consumable electrode arc furnace. Melting was done in an argon atmosphere and was repeated several times to achieve homogeneity as far as possible. The ingot thus formed was cut into two pieces for further studies. These pieces are referred to as ‘as-cut’ samples and are given the sample code FENI-c (c for cut). Part of the first piece was filed with a diamond coated file to extract powder from it; this sample is called FENI-f (f for filing). Some of this powder was annealed at 800 °C for 100 h and this sample is called FENI-fa. The second piece (FENI-c) was annealed at 900 °C for 100 h in order to achieve proper chemical homogeneity. The annealing was done in sealed quartz tubes under an argon atmosphere at a pressure of about 10⁻³ Torr. The sample at this stage is called FENI-h (h for homogenized). After annealing, the ingot was filed to extract powder from it, the filed powder this time is given the sample code FENI-hf. So we have five different kinds of Fe–Ni sample. The codes for all these samples are given in table 1.

All the samples were subjected to x-ray diffraction (XRD) study to find the phases formed in the alloy during different processes. The instrument used was a Seifert ISO-DEBYFLEX 2002 diffractometer with a copper target. The tube was operated at 30 kV and 10 mA. The radiation used was Cu K α having a wavelength of 0.154 nm. The melted ingot was cut with an ISOMET low speed saw with a diamond coated blade. The surface of the as-cut sample FENI-c and the annealed piece FENI-h were cleaned and polished with 50 μ m alumina powder. The

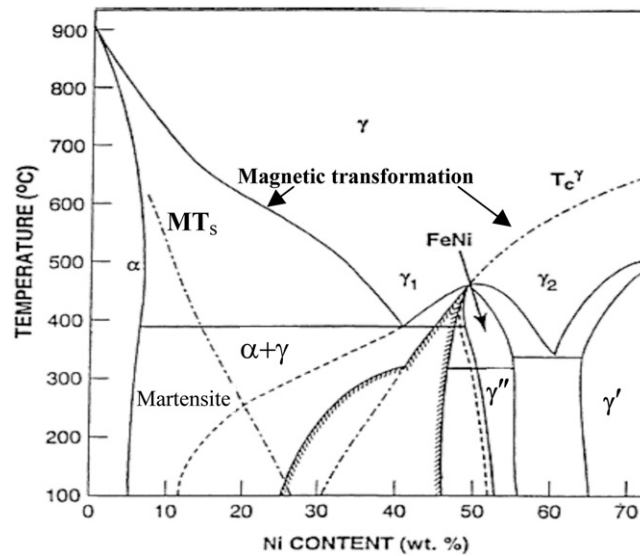


Figure 1. Fe–Ni phase diagram (taken from [28, 29]). The dotted–dashed lines show the martensite start temperature (MT_s) and the Curie temperature T for the γ_1 phase. Dashed lines show metastable extension of the miscibility gap from the tricritical point, and the hatched lines delineate the spinodal decomposition region.

polished surface in the case of FENi-c and FENi-h and the powders in the case of the other three samples were used for x-ray diffraction. The scanning rate in the x-ray powder diffraction was kept at 3° min^{-1} .

A piece of about 25 mg was cut by a low speed diamond cutter from each of the FENi-c and FENi-h samples. These pieces were subjected to magnetization measurement in an ADE Technologies model-EV7 vibrating sample magnetometer (VSM). Similarly, about 25 mg of powder each from FENi-f, FENi-hf and FENi-fa was subjected to magnetization measurement.

Mössbauer spectroscopy was performed on the powder samples only. To make the Mössbauer absorber, powder samples (about 35 mg) were sandwiched between two layers of Sellotape in a copper ring of inner diameter 13 mm. The data were recorded at room temperature in a transmission geometry using a conventional ^{57}Fe constant acceleration Mössbauer spectrometer employing a 25 mCi $^{57}\text{Co/Rh}$ source. The low temperature data were recorded in a closed cycle DMX-20 cryogenic system, manufactured by ADP Science. The spectra were analyzed using a least squares method assuming Lorentzian lineshapes.

3. Phases of Fe–Ni alloys

The crystal structure of Fe–Ni alloys is mainly body-centred cubic (bcc) or face-centred cubic (fcc). The phase diagram of the Fe–Ni system, as proposed by Reuter *et al* [28] and modified and explained by Yang *et al* [29], is given in figure 1. Below a certain temperature given by the phase boundary, the Fe-rich region (<8 at.% Ni) presents a bcc lattice and is known as the α -phase or kamacite phase. In the intermediate composition region, between 8 and 65 at.% Ni, the system presents a mixture of the bcc and the fcc lattice. This fcc phase (called the γ -phase at higher temperature) breaks into structures γ_1 and γ_2 below about 400 °C. The phase γ_1 is a paramagnetic Fe-rich phase while γ_2 is a ferromagnetic Ni-rich phase at room temperature. In the Ni-rich region (>65 at.% Ni), the diagram presents only the

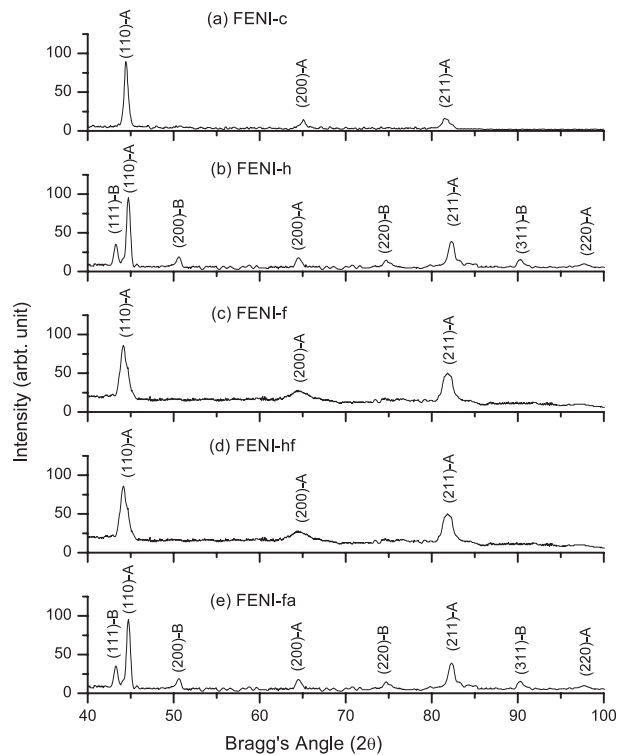


Figure 2. XRD patterns of the five samples of Fe–Ni alloys. The plane indexing of bcc is referred to A and that for fcc is referred to B.

ferromagnetic fcc or γ_2 phase. At low temperatures of around 110 K the γ_1 phase orders magnetically [26] giving a well-split six-line pattern in the Mössbauer spectrum. For the low Ni concentration, the Fe–Ni alloy is a typical system undergoing fcc \rightarrow bcc martensitic transformation (MT). The martensite transformation temperature is an important parameter to know if one wants to achieve shape memory alloys. The transition characteristics in nanograined Fe–Ni particles are significantly altered with respect to those of coarse-grained materials with similar composition [30–32].

4. Results and discussion

4.1. X-ray diffraction

Figure 2 gives the XRD patterns of the five samples. From the XRD patterns it is clear that only two types of crystallographic structure have been formed in the alloys subjected to different treatments. One of these is pure bcc while the other is a mixed phase of bcc with fcc. From the phase diagram we expect that the homogenized alloy should have crystallized in the $\alpha + \gamma$ phase. According to the calculations based on the probability distribution of a hyperfine magnetic field by Lima *et al* [34], for a Ni concentration of 25 at.% the alloys should contain 72–75% of the α -phase and 25–28% of the γ -phase at the low temperature equilibrium. Out of this γ -phase around 20% should be the γ_2 -phase and only 5–8% the γ_1 -phase.

The XRD pattern of FENI-c (figure 2(a)) shows a strong bcc crystalline structure with a sharp line width. There is no trace of fcc phase in this sample. In an arc furnace alloys cool

down quite rapidly from their molten stage and are expected to retain the high temperature phases. Therefore we should expect a high fcc content (γ -phase), but we are getting only bcc (α -phase) crystallinity. If we look at the XRD pattern of the FENI-h sample it is a mixture of bcc and fcc structure as expected from the phase diagram. The bcc peaks are quite intense as compared to the fcc peaks. The line widths of the peaks are quite sharp. This pattern (figure 2(b)) gives about the same fraction of fcc phase as estimated from the Lima *et al* [34] model.

Filing of alloys produces lot of deformation and destroys the long range order. Our XRD patterns of FENI-f and FENI-hf samples (figures 2(c) and (d)) show only peaks corresponding to the bcc phase with wider line broadening. We see that the peak corresponding to the (220) plane, which is quite pronounced in the homogenized sample, has diminished. This means that filing has destroyed its equilibrium phases and has brought it into a random α -phase. One can easily see a kink in the (110) peak in figures 2(c) and (d). The main peak of this plane is centred at a 2θ value of 44.05° , whereas the kink appears at 44.70° . It is known that the spacing between the planes for the α -phase of Fe–25 at.% Ni alloy is larger than that of the α -phase of pure Fe. And hence, the 2θ value for the α -phase of Fe–25 at.% Ni alloys should be lower than that for pure Fe. The appearance of the kink at 44.70° along with broadening of all other peaks indicates that some fraction of Fe may have segregated from the alloys during filing, resulting in a pure Fe phase or a very low Ni phase. The pattern of FENI-fa is similar to that of FENI-h, indicating that annealing removes filing-induced additional deformations/defects/phase segregation.

FENI-c alloy was cut from the as-melted ingot which was rapidly cooled from its molten state in the arc furnace and is expected to show γ -phase. But the XRD pattern of this sample corresponds to only the α -phase. Heat treatment of this sample at 900°C brings in the fcc phase. On the other hand, both the filed powder samples (extracted from the pieces after and before homogenization) also show a bcc ordered phase with somewhat wider line broadening. Annealing of the powder sample also brings in the fcc phase. Assuming that the polished surface of the as-cut sample gives information about the bulk sample too, we conclude that rapid cooling and filing produce similar types of α - γ phase equilibrium in the alloys. Both favour the formation of an almost pure α -phase which is not the thermodynamically stable phase for this composition. Indeed the segregation of an Fe-rich phase, as indicated by the kink in the (110) peak, is specific to the filing process.

4.2. Magnetization measurements

A piece of about 25 mg was cut with a low speed diamond cutter from each of the FENI-c and FENI-h samples. These together with powders (about 25 mg) of FENI-f, FENI-hf and FENI-fa samples were subjected to M – H measurement in a VSM. A typical M – H curve is given in figure 3. The saturation magnetizations (M_s), of all these samples are given in the last column of table 1. Looking at the saturation magnetization, we find three distinct ranges of values. Both the filed powders show M_s of about $173 \text{ A m}^2 \text{ kg}^{-1}$, whereas both the heat treated samples FENI-h and FENI-fa give M_s of about $16 \text{ A m}^2 \text{ kg}^{-1}$. The arc melted ingot without any homogenization heat treatment gives an intermediate value of $138 \text{ A m}^2 \text{ kg}^{-1}$. It is interesting to note that the saturation magnetic moment of Fe is $220 \text{ A m}^2 \text{ kg}^{-1}$ and that of Ni is $56 \text{ A m}^2 \text{ kg}^{-1}$ and if we mix Fe and Ni powders in a ratio of 75:25 and calculate the magnetic moment using unitary method we get

$$M_{s,\text{cal}} = 220 \times 0.75 + 56 \times 0.25 = 179 \text{ A m}^2 \text{ kg}^{-1}.$$

M_s for both the filed powder samples is very close to this value. Does this mean that filing has segregated Fe and Ni from the alloy phase? Ni in itself is fcc and XRD patterns of FENI-f and

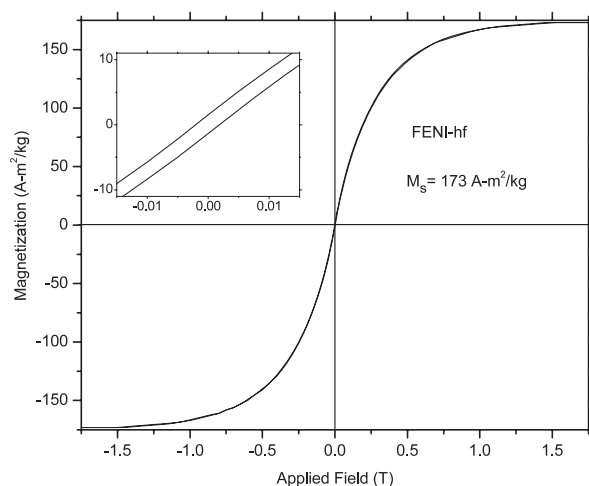


Figure 3. $M-H$ curve of the FENI-hf sample, filed powder of the homogenized Fe-25 at.% Ni alloy. The data close to zero values of H are magnified and shown in the inset to give a clear view of the shape and coercivity.

FENI-hf do not show any trace of segregated Ni. However, the possibility of a good fraction of Fe segregation cannot be denied. As we have mentioned in the previous section, a kink appears at a 2θ value of 44.70° , which corresponds to a pure Fe phase. This means some segregation of Fe atoms during filing might have occurred, enhancing the saturation magnetization. Fe and Ni atoms retain their magnetic moments, and under saturation each atom contributes its own moment and the net result is the weighted average. This means the bond effects become negligible on filing. Now let us concentrate on the saturation magnetization of the FENI-c sample. This is the sample which is quickly cooled from the molten stage in the arc furnace. If we assume that this process quenches the equilibrium high temperature γ -phase, the sample should have shown little magnetization, but here the M_s value of $138 \text{ A m}^2 \text{ kg}^{-1}$ is reasonably large but significantly smaller than what we got from the calculation for a random mixture of Fe and Ni powder.

Mathematical trial and error shows that the value of $138 \text{ A m}^2 \text{ kg}^{-1}$ can be approached from the saturation magnetic moments of Fe and Ni and their relative concentrations if we assume that the effective Fe magnetic moments are 25% weaker than the normal ones:

$$M_{s,\text{cal}} = [220 \times 0.75 \times (1 - 0.25) + 56 \times 0.25] = 138 \text{ A m}^2 \text{ kg}^{-1}.$$

This calculation suggests that the atomic and magnetic structure of this alloy is such that the Fe magnetic moments are effectively reduced while Ni magnetic moments remain intact. Indeed there may be many other ways in which the sample can attain this value of saturation magnetization.

If we compare the XRD patterns of FENI-c, FENI-f and FENI-hf we find that all of them show bcc peaks only. The line widths of the peaks in the filed samples are considerably larger than those in FENI-c. That is understandable as the filing will create a lot of crystal breaking and the XRD peaks will broaden. Though filing and rapid cooling give the same crystalline structure, apart from a finer grain size, the magnetic structures obtained are significantly changed. While filing seems to keep the magnetic moments intact, rapid cooling leads to a reduction of the effective magnetic moment.

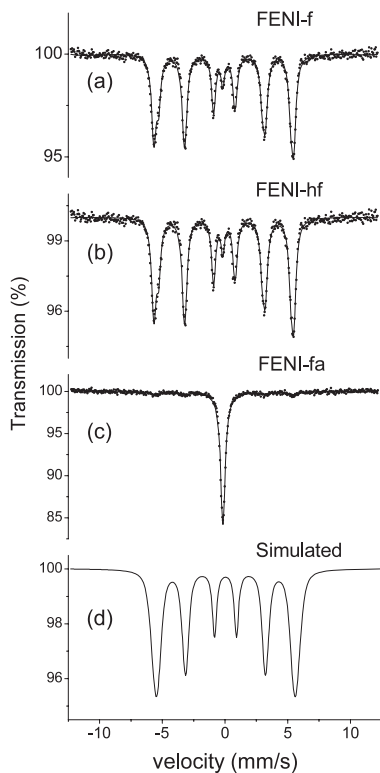


Figure 4. Mössbauer spectra (parts (a), (b) and (c)) of Fe-25 at.% Ni alloys at 300 K. (d) Simulated spectrum corresponding to a totally random atomic distribution of Fe and Ni in α -phase.

The most surprising observation comes from the saturation magnetization of homogenized and annealed samples. Its value is only $16 \text{ A m}^2 \text{ kg}^{-1}$ even though the crystallographic structure of the sample is largely bcc. From the XRD patterns we can see that only a small fraction (about 20%) of the alloy has fcc phase, also consistent with the phase diagram. The VSM observation shows that this bcc dominated phase is nonmagnetic. A similar observation was reported by Abdu *et al* [35], on the basis Mössbauer study.

4.3. Mössbauer spectroscopy

We performed transmission Mössbauer spectroscopy with the three samples in the powder form. The XRD patterns of FENI-f and FENI-hf are similar and their saturation magnetizations are also the same, so we expect that their Mössbauer spectra should have similar behaviour. We recorded spectra for FENI-f and FENI-hf samples and found that they are indeed very similar. The Mössbauer spectrum of the FENI-fa sample, (the powder annealed at 800°C) is completely different from the other two. Figure 4 gives the Mössbauer spectra of FENI-f, FENI-hf and FENI-fa samples. The magnetic splitting in the Mössbauer spectra of FENI-f and FENI-hf is expected because we obtained high saturation magnetization in magnetization measurements showing that the alloys are magnetically ordered. Samples FENI-c and FENI-h have different magnetic structure, as revealed from VSM measurement. But powders filed from them show identical magnetization and also identical Mössbauer spectra. So filing has introduced such a strong deformation that any preheating history is completely washed out.

The as-filed sample gives only bcc phase in XRD and a strong saturation magnetization of $173 \text{ A m}^2 \text{ kg}^{-1}$. These observations are very close to what one would expect from a random

Table 2. Mössbauer parameters of Fe–25 at.% Ni samples. Uncertainties in isomer shift (IS) and quadrupole splitting (QS) are 0.02 mm s^{-1} (with respect to α -Fe), in B_{hf} is 0.2 T and that in relative area is 1%. Isomer shifts are given with respect to pure Fe.

Sample code	IS (mm s^{-1})	QS (mm s^{-1})	B_{hf} (T)	Area (%)
FENI-f	−0.09			2
	0.02	−0.02	34.6	60
	0.02	0.04	33.0	38
FENI-hf	−0.09			3
	0.03	−0.025	34.6	62
	0.03	0.035	32.9	35
FENI-fa	−0.08			79
	−0.13	0.08	34.6	13
	0.22	−0.19	33.0	8

arrangement of Fe and Ni atoms in the α -phase. We have simulated a Mössbauer spectrum for such a random distribution using the fact that induction of one Ni atom in the nnn shell of Fe increases B_{hf} by about 1.22 T, and in the nnn shell it decreases B_{hf} by 0.59 T [33]. The simulated spectrum is also included in figure 4.

Comparing the Mössbauer spectra of the filed powder with the simulated spectrum (figure 4(d)) of the α -phase with completely randomized Fe–Ni distribution, we see that they are significantly different. The difference can even be seen visually. The spectrum in figure 4(d) looks like a single sextet, but the spectra in figures 4(a) and (c) clearly show the existence of two sextets. So the distribution of B_{hf} in the filed samples is bimodal whereas that for a completely random distribution of FeNi should be unimodal. Though the filed powders show only a bcc structure, the arrangements of Fe and Ni do not confirm to a single α -phase.

In order to investigate this aspect further, we made discrete Lorentzian fittings to the spectra and the parameters obtained are given in table 2. We see a small trace of a single-line component in the filed samples showing the slight presence of the γ_1 -phase. The amount is less than the XRD sensitivity and it went unnoticed in XRD analysis. There is a component of 35–38% absorption at $B_{\text{hf}} = 33.0 \text{ T}$ with almost zero isomer shift and quadrupole splitting. These parameters correspond to pure bcc Fe with no Ni substitution or very small Ni substitution. It is known that as Ni is alloyed with Fe, and for small Ni content B_{hf} increases from its value of 33.0 T for pure Fe. Its value steadily increases to 33.8 T for 5% Ni. A strong component at 33.0 T shows an almost Ni-free Fe. From XRD we had suspected that filing has resulted in Fe segregation from the alloy and magnetization measurement had supported it. Now the Mössbauer studies tell us more conclusively that indeed Fe has segregated from the alloy due to filing.

The unsegregated Fe forms an alloy with Ni. If we take the absorption area to be proportional to the number of Fe atoms present in the corresponding phase, the unsegregated Fe should form an alloy of composition near Fe–34 at.% Ni. This Ni-rich phase gives us an average B_{hf} of around 34.6 T, which may result from almost complete randomization of the atomic arrangement of Fe and Ni in the alloys.

Thus the role of filing is very special and drastic in the Fe–Ni system. It gives rise to segregation of Fe, in contrast to the case of the Fe–Al system where filing only randomizes the atomic distribution [22–24].

Annealing of the filed powder at 800°C makes most of the segregated Fe recombine with the rest of the alloy as the 33.0 T component is greatly reduced (figure 4(c) and table 2). The dominant Mössbauer component is single-line, and comes from the γ_1 -phase. This is

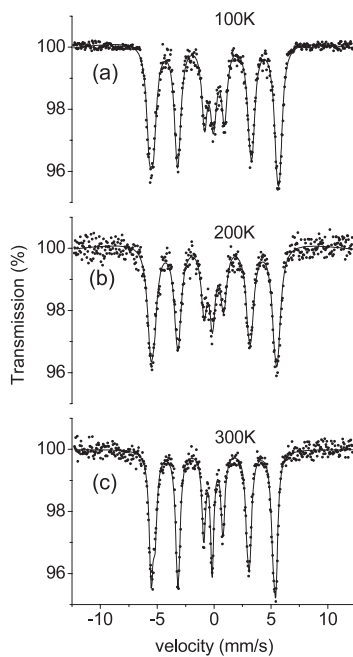


Figure 5. Mössbauer spectra of the annealed powder FENI-fa at (a) 100 K, (b) 200 K and (c) 300 K in the heating cycle.

understandable because annealing at 800 °C should take the sample to the γ -phase, which may get quenched during cooling. The problem arises when we look at the XRD pattern (figure 2(e)) which shows strong bcc peaks with only a small fcc contribution. So we have a phase apparently with bcc structure which is not magnetically ordered. It gives a single-line spectrum in Mössbauer spectroscopy and very small M_s ($15.8 \text{ A m}^2 \text{ kg}^{-1}$) in VSM measurement. This seems to be a martensitic nonmagnetic phase.

To explore the Fe–Ni nonmagnetic phase further, we recorded the Mössbauer spectra of the FENI-fa sample at low temperatures, and found that a large fraction of the paramagnetic singlet component breaks into a well-resolved six-line pattern at 100 K (figure 5(a)). The spectra at 200 and 300 K while heating back to room temperature show that part of the sample retained its paramagnetic single-line character and the rest was irreversibly changed to a magnetically ordered phase at 100 K. This behaviour is also similar to what has been observed with the γ_1 -phase of Fe–30 at.% Ni alloy [26]. The unanswered question is, if the annealed powder shows the same magnetic ordering and temperature dependence as the γ_1 -phase of Fe–Ni alloy, why does XRD show strong bcc peaks? Mössbauer spectroscopy essentially probes the immediate environment of the ^{57}Fe probe atoms and hence is much more sensitive at short range ordering than x-ray diffraction. The portion of alloy showing this anomalous crystalline versus magnetic structure could still be fcc at a very short-range not picked up by XRD.

Further experiments, based on other technique such as neutron diffraction, are needed to get more information about this phase, showing bcc peaks in XRD but a hyperfine field and magnetization analogous to the γ_1 -phase.

5. Conclusion

In contrast to our earlier work on Fe–Al based alloy [22, 24], where filing only destroys the long range order and creates vacancy-type defects and leads to randomization of atomic

arrangement, in the case of Fe–Ni alloy filing brings in a totally different order in the alloy. It leads to segregation of a good fraction of Fe atoms from the Fe–Ni alloy and the unsegregated Fe makes alloy which is much more Ni rich. Annealing of filed powder makes the whole system go to a dominantly bcc phase which has no magnetic ordering. The crystal ordering of the rapidly cooled sample is more like the filed one as both of them show a bcc-phase notwithstanding the expectations from the equilibrium phase diagram.

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