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Mössbauer study of martensitic transformations in an Fe–29.6% Ni alloy

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The application of an external stress may form "band" shaped strain-induced martensites in the austenite structure of Fe alloys. Mössbauer spectroscopy and transmission electron microscopy techniques were used to clarify certain properties of strain-induced martensite in an Fe–29.6% Ni alloy. The reverse transformation mechanism between thermal plate martensite and the matrix austenite was also studied. Mössbauer spectroscopy made it possible to examine the same area of the austenitic thin foils during the $\gamma \rightarrow \alpha \rightarrow \gamma$ thermal cycles, and the volume fraction changes were determined. The habit plane and orientation relationship of strain-induced martensite were measured from the electron diffraction patterns and the latter parameter was found to be K-S type as with thermal plate martensites of the Fe–Ni alloys. The isomery shifts caused by the deformation and cycling procedures were also calculated for both austenite and martensite structures and the hyperfine magnetic field parameter of Fe–29.6% Ni strain-induced martensite was found to be equal to that of Fe–Ni–C alloys reported earlier.

1. Introduction

Several studies have been made on the formation characteristics of martensite in different Fe–Ni alloys. A detailed study of austenite–martensite transformation in an Fe–Ni alloy by Machlin and Cohen [1] showed the burst-type transformation behaviour of this alloy. However, a recent study of martensite formation with thin foil samples of an Fe–33% Ni alloy also revealed that the plates formed at the M_s temperature may continue to grow on further cooling [2]. Besides the thermal activation of austenite–martensite transformation, martensitic transformation can, in general, also be induced in Fe alloys by the plastic deformation of matrix austenite [3–6]. Although there is no work reported on a strain-induced martensite to austenite reversal yet, the thermally-induced martensite may transform back to austenite exhibiting certain morphological and crystallographic properties [7].

The characteristics of deformation-induced martensite and the nature of martensite to austenite reverse transformations have been observed using different techniques, but the recent experiments on Fe alloys with Mössbauer effect measurements have given valuable results [8–11].

This paper reports an application of the Mössbauer spectroscopy technique to a study of some properties of strain-induced martensite transformation and also austenite–martensite–austenite periodic transformations in an Fe–29.6% Ni alloy.

2. Experimental details

Fe–29.6% Ni alloy ($M_s \approx -40^\circ$ C) specimens were austenized at 1000° C in vacuum and cooled in a methanol–liquid nitrogen mixture for austenite–martensite transformation. The A_s temperature was measured as 440° C and martensitic samples were reversed to austenite by vacuum heating at 600° C with a heating rate of $\sim 4^\circ$ C sec^{-1} and held there for $\frac{1}{2}$ h before cooling to room temperature for examination. The austenitic bulk samples were also deformed plastically by rolling at room temperature to obtain strain-induced martensite and then observed in Hitachi HS9 and H500 transmission electron microscopes. Thin foils of Fe–29.6% Ni alloy were prepared

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by mechanical and chemical thinning procedures and used in electron microscopy and Mössbauer spectroscopy experiments.

The Mössbauer spectroscopy was carried out by using a 25 mCi ^{57}Co source diffused in Pd.

3. Results and discussion

3.1. Strain-induced martensite

It is well known that the application of an external stress near or above the upper temperature limit of stress-assisted martensite formation initiates the formation of strain-induced martensite in Fe alloys [12–14]. However, there is still some doubt as to the nature of the "band" shaped strain-induced martensites since they also exhibit some very characteristic properties of deformation twins formed in these alloys [15–18]. Although recent observations on the formation of strain-induced martensites in different Fe–Ni–C alloys [18] have helped to explain the nature of the observed "bands", it was still thought worthwhile to examine these properties of the transformation products in a non-carbon Fe alloy. Fig. 1 shows a transmission electron micrograph of Fe–29.6% Ni strain-induced martensites formed after 49% deformation of the matrix austenite at room temperature, with the corresponding electron diffraction pattern taken from the austenite–martensite interface. The Mössbauer spectra of the same sample recorded at room temperature is also given in Fig. 2a. The peak labelled γ corresponds to the spectrum of paramagnetic austenite and the rest of the pattern represents the typical six-line spectrum [8–11] of the ferromagnetic martensite in Fe alloys. The internal hyperfine magnetic field of the martensite was calculated using magnetic dipole moment values of $-0.153\,\mu_n$ and $0.090\,\mu_n$ for excited and ground states and found 340 ± 10 kOe, where μ_n is the nuclear magneton parameter; 3.152×10^{-18} MeV Oe [19], which is equal to that of Fe–Ni–C alloys [1]. The same sample was also immersed in liquid nitrogen to produce thermal plate martensites and used again for the Mössbauer measurements. Fig. 2b shows the Mössbauer spectrum of this sample which contains the peaks of both thermal plate and strain-induced martensites in addition to the matrix austenite. The martensite percentages of these samples were determined and are given in Table I with the calculated isomery shifts of austenite and martensite peaks.

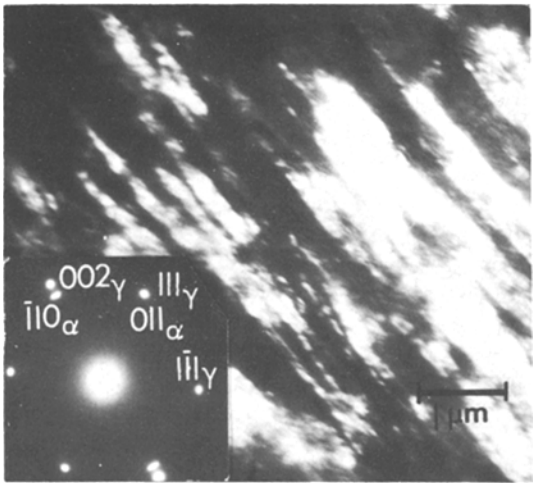

Figure 1 Transmission electron micrograph showing strain-induced martensites, and corresponding electron diffraction pattern of the matrix and product structures.

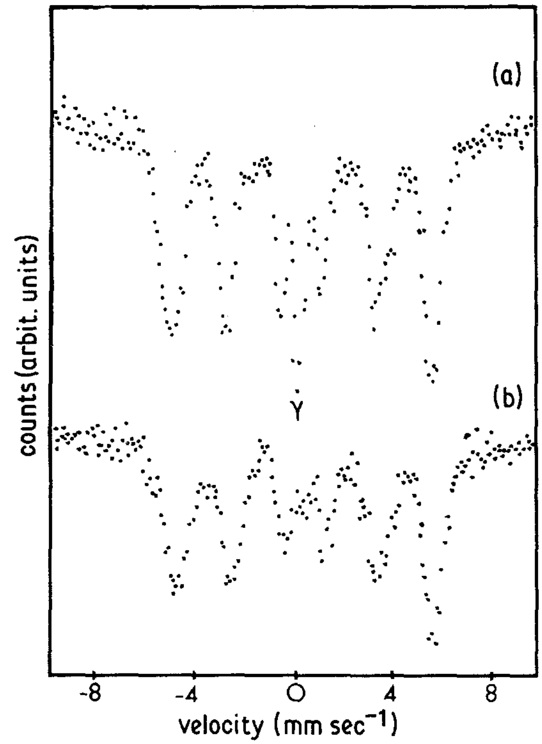

Figure 2 Mössbauer spectra of (a) deformed, and (b) deformed and cooled thin foil austenite.

This experiment shows the formation of thermal plate martensite on strain-induced type martensite as reported earlier for some Fe–Ni–C alloys [6]. The extent of plate martensite was found to be about 9% in the cooled sample. The observed changes in the isomery shifts may indi-

TABLE I Summary of the Mössbauer results obtained with a 49% deformed sample

Sample	% martensite	Isomery shifts (mm sec^{-1})	
		Austenite	Martensite
49% deformed	83 ± 1	0	0.23 ± 0.10
49% deformed then Cooled in liquid nitrogen	92 ± 1	0	0.23 ± 0.10

The figures given are mean values ± standard deviation.

cate the deformed nature of both the austenitic and martensitic crystal structures.

Some crystallographic parameters of the Fe–29.6% Ni strain-induced martensite were also determined during the study. The habit plane of the martensites was found from the electron diffraction patterns using single-surface analysis and the results are given in Fig. 3. The orientation relationship between austenite and thermal plate martensites of Fe–Ni alloys is known to be very close to a Kurdjumov–Sachs or a Nishiyama relationship [20]. This relationship was also determined by the strain-induced martensite observed in Fe–29.6% Ni alloy using the electron diffraction pattern shown in Fig. 1. The pattern consists of $[10\bar{1}]$ austenite and $[11\bar{1}]$ martensite zones and shows that the orientation between two crystal structures is very close to the Kurdjumov–Sachs type relationship. Crystallographic determinations of the habit plane and austenite–martensite orientation relationship of Fe–29.6% Ni alloy revealed that despite the formation of strain-induced martensite under different physical conditions, the main crystallographic properties of the strain-induced martensite are very close to those of thermal plate martensites in Fe–Ni alloys with Ni contents near to 29.6% [20].

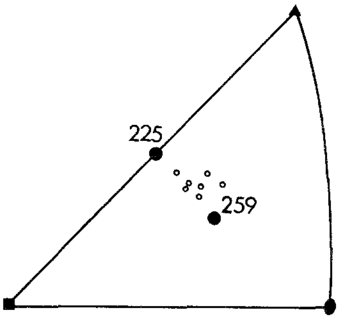

Figure 3 Unit triangle showing the strain-induced martensite habit planes.

3.1. Martensite to austenite reverse transformations

It is well known that under certain physical conditions the martensite phase of Fe alloys may be transformed back to austenite. The general nature of this behaviour was described by Wayman with the thermoelastic properties of materials and it was noted that, in general, materials exhibiting thermoelastic behaviour are crystallographically reversible [7]. The change in several properties of Fe–Ni alloys with reverse transformations was investigated by Krauss and Cohen [21] and it was shown that the heating rate of this transformation has an important effect on the nature of the transformations. They also showed the importance of γ → α → γ cycles on the austenite stabilization. Studies on the structural aspects of this type of transformation in Fe alloys also revealed that complex structural changes take place during the reversal process [5, 22–25].

The Mössbauer spectroscopy technique was applied to determine some properties of martensite to austenite reverse transformations in the Fe–29.6% Ni alloy. The austenite foil samples

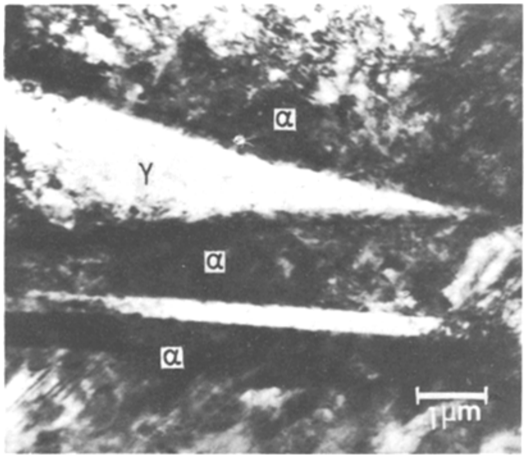

Figure 4 Transmission electron micrograph of thermal plate martensites.

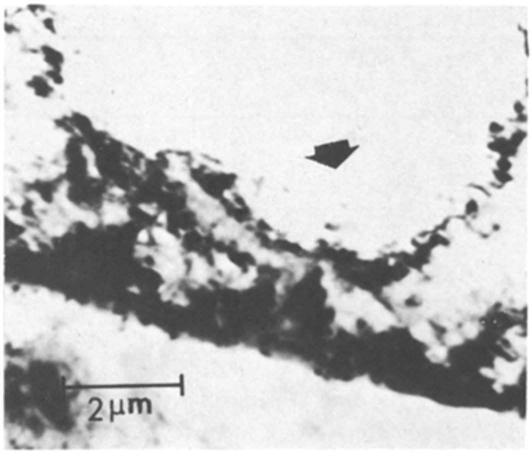

Figure 5 Transmission electron micrograph of a plate martensite after heating to 480° C, light area indicated by arrow is re-transformed austenite.

were transformed to martensite by immersing in liquid nitrogen and reversed back to austenite by heating at 600° C. The experiments were carried out with successive $\gamma \rightarrow \alpha \rightarrow \gamma$ cycles and the same area of the foil was examined by employing the Mössbauer technique. Fig. 4 is a transmission electron micrograph of the martensitic structure formed in Fe–29.6% Ni thin foil at liquid nitrogen temperature. Fig. 5 also shows an electron micrograph of a martensite plate which is partly re-austenized at 480° C. Since the martensite to austenite reversal was completed at 510° C the reverse transformation temperature of 600° C was found to be sufficiently higher than the austenite finish (A_f) temperature but ~ 100° C lower than the recrystallization temperature of the alloy used, therefore the structure was completely austenitic after the heating process, and there was no possibility of recrystallization.

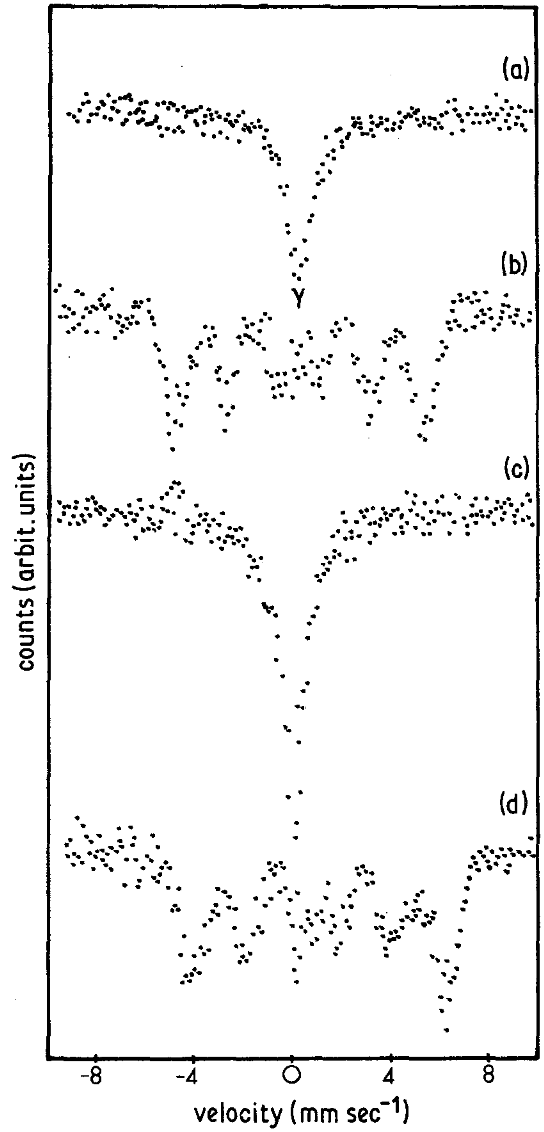

Figure 6 Mössbauer spectra of austenite (a, c) and martensite (b, d) structures after the first and third thermal cycles.

Fig. 6 shows the Mössbauer spectra of the same foil after the first and third cycles. Examination of the spectra after successive cycles revealed no change in the spectral profiles. However, comparison of the spectra after each thermal cycle showed changes in the areas occupied by martensite peaks, and relative changes of martensite volume fractions were determined. The calculated martensite percentage and the isomery

TABLE II Summary of the results of Mössbauer measurements for the sample after three successive cycles

Cycle	% martensite	Isomery shifts (mm sec^{-1})	
		Austenite	Martensite
Annealed austenite	73 ± 1	0.45 ± 0.10	0.14 ± 0.10
I	91 ± 1	0	0
II	94 ± 1	0	0.20 ± 0.10
III	95 ± 1	0	0.20 ± 0.10

The figures given are mean values ± standard deviation.

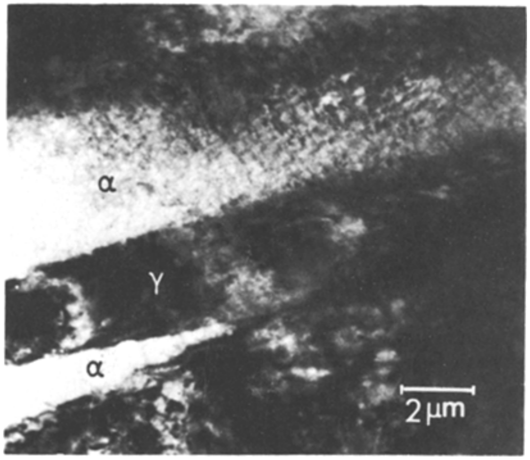

Figure 7 Transmission electron micrograph showing martensite plates formed in the reversed austenite.

shifts of both phases are listed in Table II for the first and third cycles. The same values are also given for the annealed austenite to enable comparison to be made. As it is seen in Table II, the martensite percentage changed considerably with the first cycle and no important change could be detected after this. This result agrees with those of other Fe alloys reported earlier that the biggest effect comes from the first cycle [5, 21], but does not agree with the Mössbauer spectroscopy results of an Fe–Ni–C alloy [26] which showed that the martensite percentage is decreased with successive cycles. Fig. 7 is a transmission electron micrograph of the martensite plates formed in the reversed austenite. The observed difference in the martensite volume changes can be explained in terms of nucleation and growth behaviour of Fe–29.6% Ni alloy martensite in the reversed austenite. The thermal cycling creates a high density of dislocations [5, 23] and, as has been observed in the deformation studies with Fe–Ni–C alloys [27], the low density of the dislocations may favour the martensitic transformation and big plates can grow easily. But the increased dislocation density caused by the successive cycles may obstruct the martensite nucleation and can act as important barriers to the growth in thin foils. Therefore, the dislocation density may be responsible for the observed changes, and the decreasing difference in the volume fraction of martensite plates after the first cycle is most probably the result of austenite stabilization.

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