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Size distribution of martensite plates in an Fe–Ni–Mn alloy

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In this paper, the size distribution of the martensite plates in an Fe–23.2 Ni–2.81 Mn (wt%) alloy, which transforms isothermally at subzero temperatures, is reported. The distribution of the martensite plates has been determined as a function of the reaction temperature, volume fraction of martensite, the austenitic grain size, a superimposed elastic stress and prior plastic strain (at room temperature) of austenite. Increasing the driving force either by decreasing the reaction temperature or by a superimposed elastic stress changes the size distribution by enhancing the extent of radial growth of the martensite plates. Pre-straining of austenite does not allow the martensite plates to grow to the full extent. The present results show that the radial growth of the martensite plates increases with increasing driving force and decreases due to work-hardening of austenite. The transformation is found to progress through a combination of the spreading-out of clusters and filling-in of pockets, both occurring simultaneously. However, the extent of filling-in, i.e. compartmentalization of austenite grains, is more in the coarse-grained (0.09 mm) and medium-grained (0.048 mm) specimens compared to that in the fine-grained (0.019 mm) specimens.

1. Introduction

Apart from the traditional interest of kinetics, crystallography etc., measurement of the dimensions of fully grown martensite plates has become of interest to research workers [1–8] in recent years. This is because the size and mean volume of the martensite plates play a crucial role in phenomena related to commercial applications such as the formation of microcracks [4], retained austenite, and martensitic grain size. Two major factors which affect the dimensions of martensite plates [8] are: (a) fundamental factors (like the driving force, properties of the austenite matrix, a superimposed elastic stress/magnetic field) and (b) geometrical factors (including austenitic grain size, details of plate arrangement and sequence of formation of the plates as the transformation progresses).

The average dimensions of the plates, such as the mean semithickness, the mean radius, etc., can be determined by Fullman's [9] disc analysis and also by characterizing the size distribution of the plates. It is to be noted that the determination of the average dimensions from the size distribution is less restrictive compared to Fullman's method where a specific shape of the plate is assumed. Also, Fullman's method involves determination of the harmonic mean of the inverse of the plate lengths, which is unfortunately too much sensitive to inaccuracies in the determination of dimensions, especially in the range of small sizes.

The size distributions of martensite plates have been reported for Fe–Ni–C [3] and Fe–C–P [5] alloys which transform athermally. The problem with these experiments is that one has to cool the specimen to different temperatures to obtain different martensitic fractions. As a result, it sometimes becomes difficult to delineate the effect of the above mentioned factors on the size distribution of martensite plates. On the other hand, isothermal transformation provides an ideal opportunity to rationalize some of the fundamental issues. For example, the effect of the fundamental factors on the plate dimensions can be studied by keeping the geometrical factors constant and vice versa, because different extents of transformation can be obtained at a constant temperature. Also, the transformation can be carried out at the same temperature with a superimposed elastic stress or with a prior plastic strain in austenite. The size distribution of the martensitic plates obtained in such cases can be compared with those obtained when no external stress was applied and in the undeformed samples. Such comparisons will be very meaningful as the reaction temperature can be kept constant in all cases. At the same time, it is necessary to have a good description of the stereological properties of the martensite plate distribution in order to predict the kinetics of isothermal transformation.

In the present work, an Fe–Ni–Mn alloy has been chosen which transforms isothermally at subzero temperatures. The size distribution of martensite plates has been determined as a function of (a) isothermal test temperature, (b) extent of transformation, (c) austenitic grain size, (d) prior plastic strain (ε_P) of austenite and (e) a superimposed uniaxial tensile elastic stress (σ_E).

2. Experimental procedure

An Fe–Ni–Mn alloy was obtained in the bar form and

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homogenized in an evacuated, sealed Vycor tube at 1150° C (1423 K) for 60 h. This heat treatment was followed by surface grinding to remove the manganese-depleted layer. The chemical analysis after homogenizing treatment was 23.2 Ni, 2.81 Mn and 0.009 C (wt %). The final wire samples, of diameter 1.25 mm, were obtained by cold-swaging the bar with intermediate anneals. Each intermediate anneal was followed by electropolishing to remove the surface layer. The final annealings were carried out at 1050° C (1323 K) for 10 min, 1000° C (1273 K) for 10 min and 900° C (1173 K) for 10 min to establish the grain sizes (mean linear intercept) of 0.09 mm for coarse grains (CG), 0.048 mm for medium grains (MG) 0.019 mm for fine grains (FG), respectively. Nickel lead wires for resistance measurements were spot-welded to wire specimens of 60 mm length and a "doping" anneal was given at 800° C (1973 K) for 10 min to prevent preferential surface nucleation [10].

Wire samples of different grain sizes were transformed at various temperatures between − 70° C (203 K) and − 196° C (77 K). Isopentane was used as a low-temperature bath cooled by liquid nitrogen. The bath temperature was monitored by means of a copper–constantan thermocouple and was maintained within ± 0.3° C. The transformation was followed by means of an Autobalance Universal Bridge B642 (Wayne Kerr, UK) which can detect 0.1% martensite with good accuracy. The change in resistance was calibrated in terms of the volume fraction of martensite by means of point counting. Some of the doped wires were deformed at room temperature (20° C or 293 K) to various strains (ε_P) from 0.005 to 0.05 in a tensometer and then isothermally transformed at − 85° C (188 K), − 100° C (173 K), − 140° C (133 K) and − 196° C (77 K). The grain size used for this experiment was MG. The elastic stress experiment was carried out with the FG samples. A constant load was maintained throughout the duration of the isothermal tests at − 85° C (188 K), − 143° C (130 K) and − 196° C (77 K).

Quantitative metallography for all the samples was performed in an optical microscope at 1000 × magnification. For each sample 400 random plates were chosen and measurements were carried out. An eyepiece (10 ×) with a square grid (10 mm × 10 mm) was used for this purpose. All the plates falling within the grid were selected and measurements were carried out. The reproducibility of such experimental data was tested by dividing any set of data into two halves and subsequently determining the size distribution of the plates in each half; this was found to give rise to almost identical distributions. This also confirms the "randomness" with respect to the selection of the plates and their measurements. The major experimental difficulty in such measurements was non-uniformity of the microstructure, especially at the early stages of transformation. However, in the present investigation, in such cases more than one polished section was examined to get 400 plates.

3. Determination of size distribution

Saltykov's analysis [11] for the determination of the size distribution of spherical particles has been extended by DeHoff [12] to that of aggregates of circles of different diameters. Assuming martensite mid-planes to be circular [3], which is quite reasonable, DeHoff's analysis gives

$$N_j = \frac{4}{\pi\Delta}\sum_{i=j}^{k} n_i\beta(j, i) \tag{1}$$

where N_j is the number of plates per unit volume with diameters in the j^{th} class, Δ is the width of the class, n_i is the number of mid-planes with lengths falling within the i^{th} class per unit area of section plane, and $\beta(j, i)$ is the Saykov coefficient. Values of this coefficient are given by DeHoff [12] and the class width, Δ, was arbitrarily taken as 10, 15 and 20 μm for FG, MG and CG specimens, respectively.

The mean plate radius, $\bar{r}$, can be written as

$$\bar{r} = (\Sigma N_j L_j)(2\Sigma N_j)^{-1} \tag{2}$$

where

$$L_j = \frac{\Delta}{2}(2j - 1) \tag{3}$$

4. Results and discussion

The histograms obtained for different reaction temperatures, at early stages of transformation for CG specimens, are shown in Fig. 1. The normalized plate lengths, $2\bar{r}/d$, where $2\bar{r}$ is the mean diameter of the

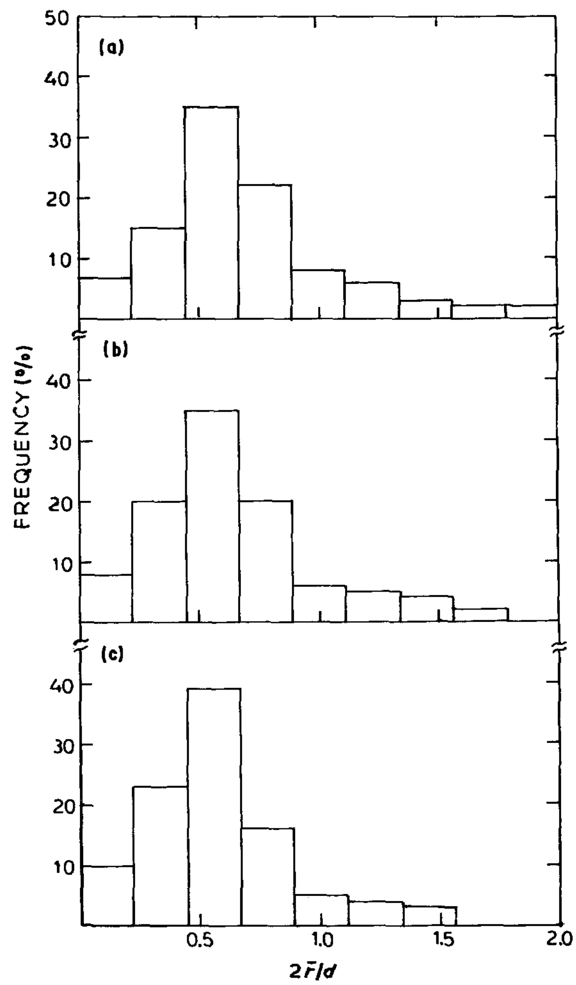

Figure 1 Distribution of martensite plates in CG specimens (grain size = 0.09 mm) at different temperatures and a martensite fraction of 0.01: (a) 77 K, (b) 133 K, (c) 173 K.

martensite plates and d is the mean linear intercept length of austenite grains, have been plotted against frequency to obtain the distribution. The frequency in all the figures is expressed as the percentage of the total population, i.e. $100(N_j/\Sigma N_j)$. If $2\bar{r}/d > 1.0$, this indicates that the mean diameter of a certain fraction of plates is greater than the mean linear intercept length of austenite. It is obvious that as the transformation temperature decreases, the relative frequency of larger plates increases. Also some higher class sizes, which are absent at higher temperatures, start appearing at lower transformation temperatures. Therefore the radial growth of the plates in a uniform temperature field is related to the driving force of transformation. This also supports Knorovsky's observation [13] that radial growth of the plates is related to the driving force when the plates are grown up a temperature gradient. A similar type of variation is found for MG and FG specimens after reacting at different test temperatures.

It is worth mentioning that the size distributions of martensite plates reported for Fe–Ni–C [3] and Fe–C–P [5] alloys were for the plates which form over a range of temperature. So in those cases it is not possible to obtain size distribution of the plates which form at the particular temperature. As far as matrix properties are concerned, the increase in yield strength due to decrease in the temperature does not have any effect on the radial growth of the plates. Otherwise one would expect the mean radius of the plates to decrease with decreasing test temperature as the yield strength of austenite increases appreciably. However, other parameters like mean semithickness and semithickness-to-radius ratio of the martensite plates are very much dependent on the yield strength of austenite at the reaction temperature [14]. Since the shear modulus of austenite is only mildly temperature-dependent, we can take it as temperature-independent.

Fig. 2 shows the size distribution of the plates for CG specimens transformed at $-120°C$ (153 K) for two different martensite fractions (f). With increasing volume fraction of martensite, the relative frequency of smaller plates increases. This implies that with the progress of transformation the austenite grains are divided into smaller compartments, i.e. the partitioning of austenite [15] does take place to some extent. MG and FG specimens are also found to behave similarly for all reaction temperatures. However, for a given amount of martensite the extent of partitioning in FG specimens was less compared to CG and MG specimens for any isothermal test temperature.

According to Raghavan [16] the progress of transformation can take place in two steps: (a) "spreading" of the clusters of plates from one grain to the other, and (b) "filling-in" of the austenite grains. In a recent study [14] it has been reported that the progress of isothermal martensitic transformation takes place by a combination of "spreading" of the clusters and "filling-in" of the pockets, both occurring simultaneously; the decrease in $\bar{r}$ with increasing martensitic fraction is found to be less compared to a case where only "filling-in" of austenite pockets takes place. On a relative basis, "filling-in" of austenite pockets in CG

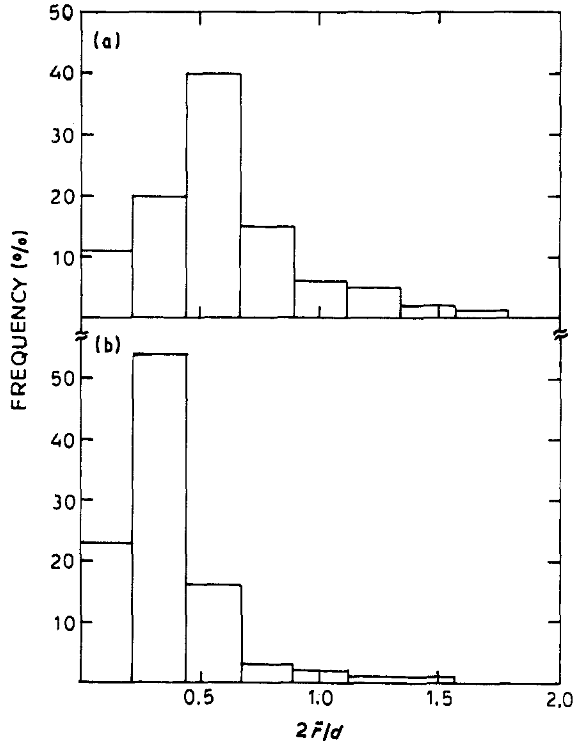

Figure 2 Distribution of martensite plates in CG specimens (grain size = 0.09 mm) at a temperature of 153 K and martensite fractions of (a) 0.015, (b) 0.30.

and MG specimens is more than that in FG specimens. Since the size distribution of the martensite plate changes with the progress of transformation, the effects of the other variables like test temperature, austenitic grain size, a superimposed elastic stress and a prior plastic strain of austenite have been studied at a constant martensite fraction.

The effect of grain size on the size distribution of the martensite plates at initial stages of transformation is shown in Fig. 3. As the grain size increases, the relative frequency of larger plates decreases and some higher class sizes may not be present at all. This is in line with the earlier report [3] for an Fe–Ni–C alloy. The distribution indicates that for FG specimens the radial growth of martensite plates is restricted by obstacles like twin boundaries, grain boundaries and interphase boundaries [17]. In addition, some other obstacles may play a crucial role in the growth dynamics of the martensite plates in larger grain-sized specimens. It is to be mentioned here that the variation of the size distribution of the martensite plates with increasing austenitic grain size may arise from the non-homogenity of the austenitic grains. This was quantitatively assessed by measuring the diameter of the largest grain on the observed section plane. The ratios of the largest grain diameter to the mean linear intercept for CG, MG and FG specimens were found to be 3.0, 3.3 and 3.5, respectively. Since these values are almost the same, the differences in frequency distribution in Fig. 3 should be attributed to transformation phenomena.

Fig. 4 shows the distribution of martensite plates for MG specimens, without and with a prior plastic strain ($\varepsilon_P = 0.05$) and transformed at $-140°C$ (133 K). With increasing ε_P, the size distribution changes systematically by increasing the relative frequency of smaller plates and decreasing that of larger

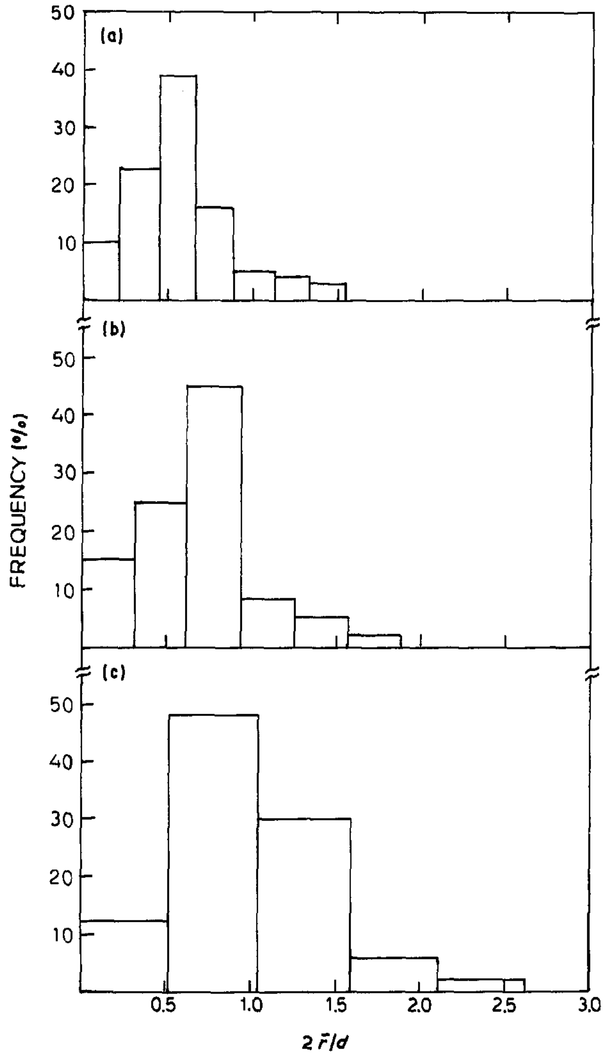

Figure 3 Distribution of martensite plates for different grain-sized specimens at a temperature of 173 K and a martensite fraction of 0.02: (a) CG (0.09 mm), (b) MG (0.048 mm), (c) FG (0.019 mm).

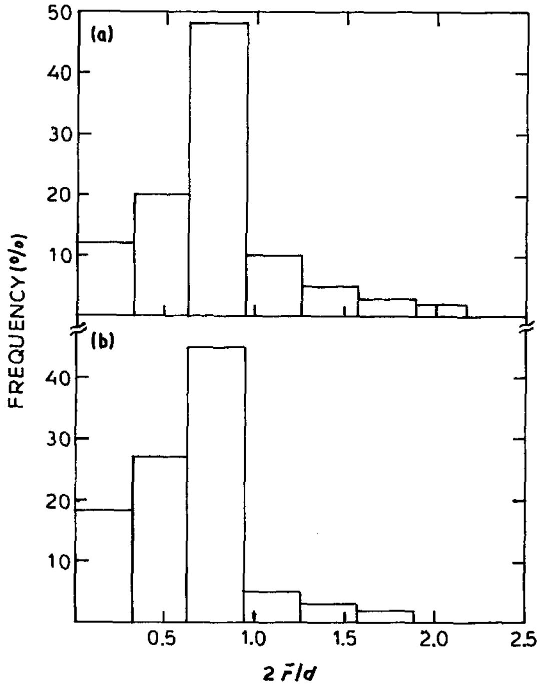

Figure 4 Distribution of martensite plates (a) without and (b) with a prior plastic strain (ε_P) of 0.05, at a temperature of 133 K and a martensite fraction of 0.01. Grain size = 0.048 mm (MG).

plates. This is expected, because pre-straining causes work-hardening of the austenitic matrix and as a result the plates are not able to grow to their full extent. For $\varepsilon_P > 0.05$, the reaction becomes very sluggish and no martensite could be obtained even after 3 h soaking at subzero temperatures. On the other hand, a superimposed elastic stress helps the plates to grow. The effect of such superimposed elastic stress, at initial stages of transformation, is shown in Fig. 5 for FG specimens isothermally transformed at -196° C (77 K). The total available driving force increases due to the free-energy contribution of elastic stress that adds to the chemical free energy [18]. At a stress level of 118 MPa, the relative frequency of larger plates increases and some higher class sizes were found which were otherwise absent when no stress was applied. Therefore, the elastic stress experiment also supports the observation that radial growth is directly related to the available driving force at the test temperature.

In most of the cases, the histograms can be fitted with a logarithmiconormal distribution function which is skewed left and has a long tail to the right.

A comparison has been made between the mean plate radius ($\bar{r}$) values obtained by Fullman's method and by distribution analysis. Fig. 6 shows that the $\bar{r}$ values obtained from the distribution analysis are smaller than those from Fullman's method by about 20%. Data from all the experiments have been plotted and they show a consistent variation from the ideal line. Guimarães *et al.* [3] found this deviation to be about 14% for Fe–Ni–C alloy. Though at the moment it is difficult to assess why the difference should be within 15 to 20%, it is clear that the average plate radius obtained by Fullman's method is more than that obtained from distribution analysis.

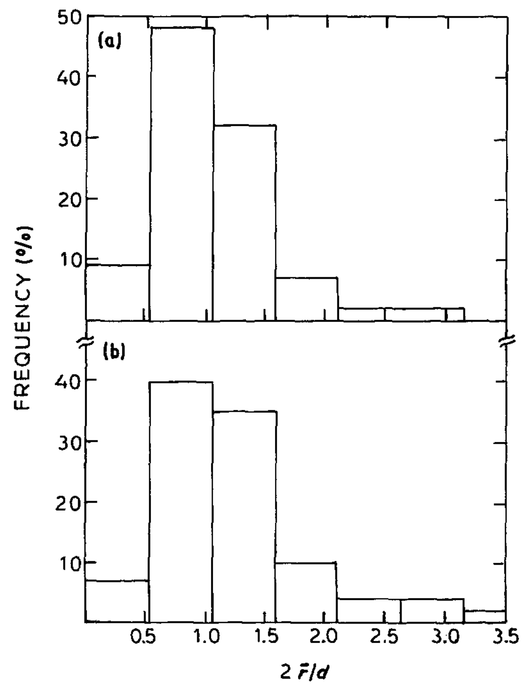

Figure 5 Distribution of martensite plates (a) without and (b) with a superimposed elastic stress (σ_E) of 118 MPa at a temperature of 77 K and a martensite fraction of 0.02. Grain size = 0.019 mm (FG).

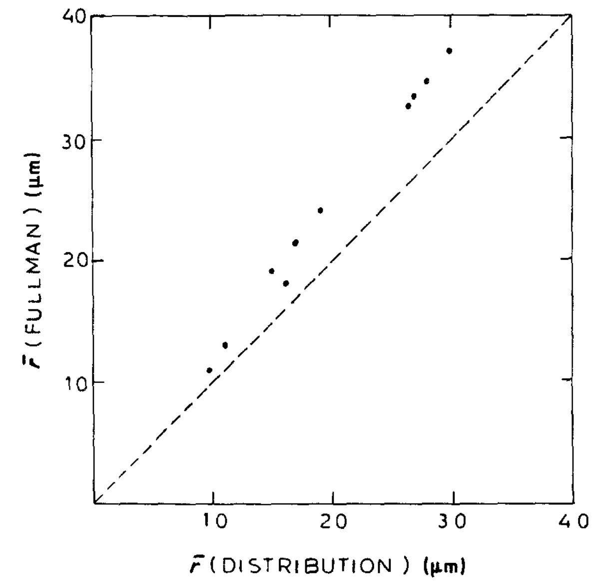

Figure 6 Comparison of mean plate radius ($\bar{r}$) obtained from Fullman's method and by distribution analysis.

5. Conclusions

With the assumption that the mid-planes of the martensite plates are circular, DeHoff's analysis has been applied to isothermal martensite to characterize the distribution of martensite plates. The following conclusions can be drawn from the present investigation:

1. The distribution of martensite plates depends on the reaction temperature. With decreasing reaction temperature, the density of larger plates increases. A superimposed elastic stress also changes the distribution by increasing the relative frequency of larger plates. Since the average plate radius increases with decreasing temperature or a superimposed elastic stress, it clearly indicates that radial growth of the plates is directly related to the available driving force.

2. With increasing grain size, barriers other than grain boundaries, twin boundaries and interphase boundaries may become effective to stop the radial growth of the plates at early stages of transformation. As a result the size distribution also changes correspondingly.

3. Pre-straining the austenite causes the martensite plates not to grow to their full extent, resulting in different size distributions with increasing prior plastic strain.

4. Isothermal martensitic transformation enables us to separate the effects of some fundamental and geometrical factors on the size distribution of the martensitic plates.

5. The $\bar{r}$ obtained from distribution analysis is about 20% less than that obtained by Fullman's method.

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