The Development of Martensitic Microstructure and Microcracking in an Fe-1.86C Alloy

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The development of the martensitic microstructure in a 1.86 wt pct C steel has been followed by quantitative metallographic measurements over the transformation range of 0.12 to 0.50 fraction transformed (f). The transformation kinetics are described by the equation

 $f = 1 - \exp\left[-0.008 \left(M_s - T_q\right)\right]$

where M_s and T_q are the martensite start and the quenching temperatures respectively. Fullman's analysis shows that the average volume per martensite plate decreases by almost an order of magnitude over the transformation range studied, but this decrease is less than that predicted by the Fisher analysis for partitioning of austenite by successive generations of martensite. Microcracking increases with increasing f up to 0.3, but does not increase for f above 0.3 where transformation proceeds by the nucleation of large numbers of small martensite plates. These observations indicate that a critical size of martensite plate is necessary to cause microcracking.

THE development of a microstructure composed of plate martensite follows a unique pattern. The plates form on irrational habit planes of high multiplicity and adjacent plates generally form on nonparallel variants. The latter characteristic of plate martensite formation causes successive generations of plates to impact or impinge on those plates formed earlier in the transformation process, and the stress fields generated on impact of two plates require that accommodation occurs. If the martensite is ductile, the accommodation occurs by a slip mechanism or deformation twinning, but in brittle martensites, microcracking is often the means of accommodation.¹ The impingements between martensite plates and parent austenite boundaries also develop preferred nucleation sites for new martensite plates and hence account for the autocatalytic nucleation and zigzag plate arrangements characteristic of transformed high carbon steels and Fe-Ni alloys.

The morphological and geometric aspects of a plate martensite microstructure and their effect on microcracking in martensite can be evaluated within the established framework of experimental and theoretical observations of the sequence of plate formation and kinetics of martensitic transformations. In Fe-C alloys the kinetics of athermal plate martensite transformation have been shown to follow the relationship

$$1 - f = \exp\left[\alpha (M_s - T_q)\right]$$
^[1]

where f is the volume fraction transformed, α is a constant, M_s the martensite start temperature and T_q the quenching temperature. The relationship was first developed empirically by Koistinen and Marburger² and has recently been derived by Magee.³ The derivation shows that

$$\alpha = \overline{V}\Psi \quad \frac{d\Delta G_v^{\gamma \to \alpha}}{dT}$$
[2]

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where all terms are assumed independent of f and \overline{V} is the average volume of the martensite plates, Ψ is a constant of proportionality between the number of new plates per unit volume of austenite and an increase in driving force, and $(d\Delta G_v^{\gamma \to \alpha})/dT$ is the change in driving force for martensite (α') formation from austenite (γ) with temperature.

Several sets of data for various Fe-C alloys^{2,4} satisfy Eq. [1] and are taken³ to support the assumption that \overline{V} is independent of f. Further, in Fe-Ni-C alloys \overline{V} has been measured by quantitative metallographic techniques and no systematic dependence of \overline{V} upon f could be found.⁵ The latter experimental result differs significantly from the theoretical analysis by Fisher et al. that predicts a strong dependence of V on f because of the expected partitioning of the parent austenite into smaller and smaller compartments as transformation proceeds. In contrast, the constant \overline{V} measured in Fe-Ni-C alloys was explained by a physical model involving autocatalytic nucleation of many plates in a range of sizes within a single austenite grain followed by the spreading of such bursts of transformation to other grains. Since f increased in the Fe-Ni-C alloys by the grain by grain formation of groups of plates with almost identical size distributions, \overline{V} did not change even after as much as 55 pct of the sample volume had transformed. A similar mode of athermal transformation behavior has been noted in fine grained austenite, again in Fe-Ni-C alloys,⁷ and a spreading of isothermal transformation in Fe-Ni-Mn alloys from one grain to another up to 15 pct transformation has been discussed.⁸ The interrelationship between the number of embryos initially present in the parent austenite, the nuclei autocatalytically formed and geometrical partitioning of the austenite have been theoretically anal-yzed and confirmed^{9,10} by quantitative metallographic techniques applied to isothermal martensite transformation in Fe-Ni-Mn alloys. Autocatalytic nucleation increases with increasing grain size, but the number of initial nucleation sites has been demonstrated to be independent of grain size.¹⁰

The purpose of this investigation is twofold: 1) to in-

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vestigate the athermal transformation behavior of plate martensite in an Fe-C alloy by quantitative metallographic techniques, and 2) to relate the transformation behavior to the development of microcracking during the formation of a plate martensite microstructure. Although V has been found independent of f in Fe-Ni-C alloys and the physical reason for this aspect of transformation behavior has been demonstrated, the first objective would permit, in an Fe-C alloy, to test experimentally whether the transformation proceeds solely by autocatalytic effects, as in Fe-Ni-C alloys," or by both autocatalysis and partioning of austenite, as in Fe-Ni-Mn alloys.9 The second objective would establish a possible correlation between microcracking frequency and the size and number of plates at various stages of transformation.

EXPERIMENTAL TECHNIQUES

An Fe-1.86 wt pct C alloy that has a M_s temperature just above room temperature was chosen for this investigation. The low volume fraction of martensite at and just below room temperature in this alloy was considered essential for applying quantitative metallographic techniques that measure the dimensions of the individual plates. The composition of the alloy was as follows: 1.86 C, 0.02 Mn, 0.012 P, 0.02 S, 0.30 Si, 0.7 Ni, 0.66 Cr, 0.010 Mo, 0.001 Cu, 0.034 V, 0.002 Sn, 0.032 Ti.

Small, 10 mm by 5 mm by 5 mm samples of this alloy were homogenized in an inert argon atmosphere at 1120°C in the single phase austenite field. The temperature range of the single phase austenite field is quite narrow for this high carbon alloy and at some time during its history the ingot was apparently overheated and some porosity was developed. The presence of the porosity is not considered to affect the development of the martensitic microstructure because similar martensitic plate patterns were observed both adjacent to porosity and at boundaries well removed from any porosity. All quantitative measurements were made in regions removed from the porosity.

After the homogenization treatment, the samples were quenched to room temperature and below in alcohol cooled by liquid nitrogen to produce increasing amounts of martensite. Following the procedures of Marder *et al.*¹¹ careful polishing and etching techniques were employed to reveal the microstructure and the cracks in martensite plates. The martensite volume fractions were measured to within 3 pct by point counting.

Fullman's theoretical analysis¹² was applied to quantitative metallographic data to calculate the average volume per plate, \overline{V} , the number of plates per unit volume, N_v , and average ratio of plate thickness to radius (\overline{t}/r) according to the following formulae:

$$\overline{V} = \frac{\pi^2 f}{8\overline{E}N_a}$$
[3]

$$N_v = \frac{f}{\overline{V}}$$
 [4]

$$\left(\frac{\bar{t}}{r}\right) = \frac{32\bar{E}}{3\pi^2\bar{E}}$$
[5]

where f is the volume fraction of martensite, N_{α} is the number of martensite plates per unit area of random section and \overline{E} and \overline{F} were calculated by making 200



Fig. 1-Log of volume fraction of retained austenite, (1 - f), vs $(M_s - T_q)$. M_s -Martensite start temperature, T_q -quenching temperature.

paired measurements of plate lengths and widths on random metallographic sections.

Prior austenite grain size and microcracking sensitivity as crack area per unit volume of martensite, S_v ,¹¹ were measured by lineal analysis in units of mm and mm⁻¹ respectively. A cumulative line length of 25 mm or more was utilized to count the number of cracks.

RESULTS AND DISCUSSION

A) Transformation Kinetics

Fig. 1 shows the progress of martensite transformation in the Fe-1.86 C alloy as a function of quenching temperature. The data is plotted as $\ln (1 - f)$ vs $(M_s - T_q)$ where f is the volume fraction of martensite, M_s is the martensite start temperature, and T_q is the quenching temperature. The value of 50°C for the M_s was selected by extending a plot of collected M_s vs carbon content data.¹³

The data of Fig. 1 fit a straight line with a slope of $-0.008/^{\circ}C$ as calculated by least squares analysis, and therefore can be represented by the following empirical equation:

$$1 - f = \exp\left[-0.008(M_s - T_q)\right]$$
 [6]

This equation is of the same form as that proposed by Koistinen and Marburger² for Fe-C alloys up to 1.1 wt pct C although the slope of their line was equal to -0.011. The difference in slopes might be the result of very high carbon content or the significant amounts of chromium and nickel in the 1.86 C alloy as compared to the Fe-C alloys investigated previously. It can be seen that an error in the selection of the M_s would not affect this slope. Brook *et al.*¹⁴ have experimentally shown that for various steels a different slope parameter, df/dt, varies from -0.008 to -0.013 depending upon the particular steel. This parameter, df/dt, at small values of f is equivalent to the slope of Fig. 1 as given by Eq. [6].

The present experimental results, representing the athermal transformation kinetics of martensite in Fe-1.86 C alloy, appear to support a recent mathematical formalism³ in its final form. However further discus-



Fig. 2—Microstructure of an Fe-1.86 C alloy showing plate martensite for a specimen quenched to 25° C from single phase austenite field. Volume fraction of martensite, f = 0.123.



Fig. 3—Microstructure of an Fe-1.86 C alloy showing a higher volume fraction f = 0.362 of plate martensite for a specimen quenched to -23° C from single phase austenite field.

Table I. Plate Parameters as a Function of	Volume Fraction of Martensite in Fe-1.86C Allov

Specimen Number	Quenching Temp, T_q °C	Volume Fraction, f	Number of Plates/Area, N _a × 10 ⁻⁵ cm ⁻²	Mean Reciprocal of Plate Lengths, $\overline{E} \times 10^{-2} \text{ cm}^{-1}$	Mean Reciprocal of Plate Widths, $\overline{F} \times 10^{-3} \text{ cm}^{-1}$	Average Volume per Plate, $\overline{V} \times 10^9 \text{ cm}^3$	Number of Plates/Volume $N_{\nu} \times 10^{-8} \text{ cm}^{-3}$	Mean Plate Thickness to Radius Ratio, <i>t̄/r</i>
1	25	0.123	0.545	3.4	3.31	8.56	0.15	0.11
2	0	0.271	1.7	4.8	4.92	3.9	0.69	0.106
3	-23	0.362	3.7	7.1	7.06	1.66	2.14	0.109
4	~51	0.507	7.8	8.0	7.83	0.98	5.09	0.11

Spherical grain diameter, $\frac{3}{2}\overline{l}^{(12)} = 0.017$ cm.

sion on this point is deferred to a later stage when it will be shown that the other results of the present investigation contradict a major assumption employed in that theoretical derivation.

B) Microstructure and Quantitative Metallography

The lowest volume fraction of martensite produced in these experiments was 0.123 for the specimen quenched to 25°C. Light microscopic examination of this specimen revealed that some plates of martensite were present in each austenite grain. If spreading of the transformation from grain to grain has occurred in this alloy, then apparently the spreading is complete at this early stage of transformation. Fig. 2 shows a representative micrograph for the 0.123 transformed microstructure and shows a distribution of large martensite plates partitioning the parent austenite grains into large compartments. The characteristic features of martensite with a $\{259\}$ habit, namely, the zigzag plate patterns, the prominent midribs and the acute angle between the plates⁷ can be readily seen in the picture.

Fig. 3 is a light micrograph of a specimen quenched to -23 °C to produce a higher volume fraction, 0.362, of martensite. Many more smaller plates are present as compared to the specimen transformed to 0.123. The austenite compartment size is also reduced correspondingly. The same trend to smaller and smaller plates and austenite compartments was observed in the specimens with increasing volume fraction of martensite. For volume fractions of martensite greater than 12 pct, the visual observations on specimens of Fe-1.86 C alloy thus support Fisher's idea⁶ that the transformation proceeds by the formation of martensite plates that progressively partition austenite volumes available for transformation. The successive nucleation of smaller and smaller plates as described above would be expected to produce a smaller average volume per plate with increasing transformation.

In order to confirm these qualitative observations quantitative metallography was performed. Results of Fullman's analysis as applied to the measured values of plate lengths and widths are given in Table I. The plate parameters are given for volume fractions up to 50 pct only; at higher volume fractions the individual plate dimensions could not be measured unambiguously due to the poor delineation of individual fine plates in a highly transformed structure. It can be seen from Table I that as f increases, the decreasing size of martensite plates is reflected in the increased average reciprocals of length and width, \overline{E} and \overline{F} respectively, and the decreased average volume per plate, \overline{V} , as calculated from Eq. [3]. Fig. 4 plots \overline{V} as a function of f. There is a continuous decrease in the values of V of almost an order of magnitude as the amount of martensite increases from 12 to 50 pct. It should be emphasized that the \overline{E} and F are the arithmetic means of distributions of plate sizes from which V is calculated. These distributions become increasingly asymmetric



Fig. 4-Dependence of average volume per plate, \overline{V} , upon martensite volume fraction, f.



Fig. 5—Comparison of Fisher's theoretical prediction with experimental results. m is a constant fraction of austenite volume available for transformation and depending upon martensite plate thickness to length ratio may have a range of values. q is the volume of a spherical prior austenite grain.

with increasing degree of transformation because all plates, from the first few large plates and the more numerous smaller plates formed during the later stages of transformation, are counted for each application of Fullman's analysis.

Only a limited number of quantitative metallographic investigations of martensitic transformations utilizing Fullman's analysis have been made.^{5,10,14} Comparison of the Fe-1.86 C data with that obtained for Fe-Ni-C⁵ alloys shows that the values of \overline{E} and \overline{F} parameters are of the same order of magnitude for equivalent austenite grain size for the two kinds of alloys. The most important difference is the continuous increase in values of \overline{E} and \overline{F} with increasing transformation in the Fe-C alloy while no systematic change in these parameters was observed in Fe-Ni-C alloys. The martensitic plates of Fe-Ni-Mn¹⁰ alloys have smaller thickness to radius ratios and smaller average volumes per plate than either the Fe-Ni-C alloy or the Fe-C alloy of the present investigation. These differences have already been discussed⁵ and are attributed to the athermal, burst martensitic transformation in Fe-Ni-C alloys in contrast to the totally isothermal transformation in Fe-Mn-Ni alloys.

The results of quantitative metallography together with microstructural observations of the transformation made over many austenite grains as represented by Figs. 2 and 3 indicate that in the Fe-1.86 C alloy, above 12 pct volume fraction, the overall martensitic transformation of a specimen proceeds by the formation of smaller and smaller plates that successively partition the untransformed austenite into smaller compartments. At volume fractions much lower than 12 pct, the transformation may initiate and proceed predominantly by autocatalytic effects. The transformation may spread either grain by grain as proposed by Magee³ or outwards from a central grain as proposed by Raghavan,⁸ a process by which the transformation spreads to all grains in Fe-Ni-Mn alloys at 15 pct volume fraction of martensite.

Since partitioning of the austenite is a major element in the progress of martensitic transformation of the Fe-1.86 C alloy in the transformation range investigated, the present data was compared to that predicted by Fisher's analysis. Fig. 5 shows that 1) for f < 0.12Fisher's approach overestimates \overline{V} and 2) the predicted rate of drop of \overline{V} with f is higher than the present experimental values. McMurtrie and Magee⁵ have attributed the first discrepancy to the fact that Fisher neglected the presence of small plates produced autocatalytically even at low volume fractions, while the second discrepancy could be rationalized by assigning probabilities of plate formation to the volume of austenite compartments and relating average plate sizes at a given f to summations of the probabilities of plate formation over a range of compartment sizes. Despite the apparent oversimplification of the effects of partitioning on the dependence of V on f, according to Fisher's model the results of the present investigation confirm that partitioning of the austenite by successive generations of martensite does occur in high carbon Fe-C alloys and that the average size of the martensite plates decreased by approximately an order of magnitude as f increased from 0.12 to 0.5.

The transformation kinetics for the Fe-1.86 C alloy fit Eq. [1], and apparently support Magee's theoretical derivation³ of this equation. In this case, the calculated value of -0.008 for the slope of $\ln (1 - f)$ vs $(M_s - T_q)$ plot is equivalent to the constant α which in turn is equal to the products of the assumed constant terms, including \overline{V} , of Eq. [2]. The decrease in V measured during the course of the transformation of the Fe-1.86 C alloy indicates that one or both of the other terms of Eq. [2] must increase about an order of magnitude to maintain α constant. Brook *et al.*¹⁵ have shown that the temperature dependence of $dG_v^{\gamma \to \alpha}/dT$ is slight for low and intermediate stages of transformation. Attention is therefore focused on Ψ , the proportionality constant between the number of new plates of martensite per unit volume of austenite and an increase in driving force, $d\Delta G_v^{\gamma \to \alpha'}$. It is quite possible that Ψ increases with *f* because of the autocatalytic nucleation that occurs during martensite formation. This possibility is supported by Entwisle,¹⁶ who



Fig. 6—Microcracks in the plate martensite of an Fe-1.86 C alloy.

in reviewing the kinetics of martensite formation in steels, states that all experimental evidence to date points to an increasing autocatalytic effect with falling temperatures, *i.e.*, increasing driving force, and that the assumption of a constant average plate volume is unlikely to be satisfactory once the autocatalytic process is well under way.

C) Microcracking

Microcracks, confined to martensite plates, were observed for all fractions of martensite produced in this study. Fig. 6 is a light micrograph showing the presence of several microcracks, some along the plate boundaries and some traversing the plate thickness. The arrows in Fig. 6 show examples of microcracks that seem to be the result of direct impingement of martensite plates. In general, the microcracks were confined to the largest plates of a partially transformed microstructure.

The microcracking sensitivity as crack area per unit volume martensite, S_v , was related to the measured plate parameters: \overline{V} , the average volume per plate, and N_v , the number of plates per unit volume. These two parameters might directly influence microcracking sensitivity, *i.e.*, the number of plate impingements that result in cracking would be proportional to N_n while the possible plate size effects on cracking would be reflected in the values of V. Fig. 7 is a plot of S_v , \overline{V} , and N_v as functions of f and shows that with increasing f, S_v increases sharply, reaches a maximum and decreases slightly or more conservatively reaches a plateau if the experimental errors are considered. A similar plateau of S_v with increasing f has been observed¹⁹ in an Fe-5 Cr-1.12 C alloy over an extensive range of f. Fig. 7 shows that concomitant with this microcracking behavior, V falls continuously while N_{ij} increases with increasing f. For volume fractions up to about 27 pct, the large increase in microcracking may be explained by the increasing number of impingements of the large plates that form in this transformation range. However, for volume fractions greater than 27 pct, despite the greater number of impingements as



Fig. 7—Microcracking sensitivity, S_{ν} , average volume per plate, \overline{V} , and number of plates per unit volume, N_{ν} , as functions of volume fraction of martensite f.

reflected by the sharply increasing N_v , microcracking appears to level off or decrease slightly. Apparently the size of martensite plates is an important factor affecting the microcracking. The results of Fig. 7 indicate the existence of a critical average volume of martensite plate $\overline{V}_{\rm crit}$, below which the microcracking increases with increasing volume fraction of martensite and above which the microcracking is relatively insensitive to increasing volume fraction.

The effect of plate size upon microcracking sensitivity has already been discussed in the literature,¹¹ and austenite grain refinement has been suggested^{17,18} as a means to reduce microcracking. Recently,¹⁹ a direct correlation between plate lengths and microcracking sensitivity has been found. The present data provides quantitative evidence that the formation of microcracks is largely limited to the early stages of transformation when large plates of martensite impinge upon even larger first-formed plates. Since the largest plates are present at all stages of transformation, it appears that the size and, more directly the magnitude of the associated leading stress fields of the impinging plates, determine the microcracking behavior of a developing plate martensite microstructure as shown in Fig. 7.

CONCLUSIONS

1) The kinetics of athermal transformation for this alloy can be described by the equation:

$$1 - f = \exp \left[-0.008(M_s - T_q)\right]$$

where f is the fraction of martensite, $(M_s - T_q)$ is the difference between the martensite start temperature and the quenching temperature in °C.

2) Above f = 0.12, the martensitic transformation in this alloy proceeds by the successive partitioning of the austenite volume by subsequently nucleating plates. Quantitative metallographic data verifies the resulting continuous decrease of average volume per plate, \overline{V} , with increasing martensite volume fraction, f.

3) The effect of plate size has been shown to be a major factor in causing microcracking in plate martensite. Below a critical average plate size microcracking does not increase despite the increased number of impingements caused by formation of many small plates by which transformation proceeds in partitioned austenite.

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