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MICROSTRUCTURE REFINEMENT IN MARTENSITIC Fe-12Ni-0.25Ti: THERMAL MECHANISMS OF GRAIN AND PACKET REFINEMENT

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MICROSTRUCTURE REFINEMENT IN MARTENSITIC Fe-12Ni-0.25Ti:
THERMAL MECHANISMS OF GRAIN AND PACKET REFINEMENT

by


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ABSTRACT

The present work was undertaken to illustrate and clarify the three distinct metallurgical mechanisms that are used in thermal treatments to refine the effective grain size of lath martensitic steels: (1) the decomposition of austenite into distinct packets of martensite, (2) the recrystallization of the austenite on heating to above the $A_f$ temperature, and (3) the direct refinement of martensite packets by rapid reversion to austenite. The work was also intended to identify the effective grain size in alloys having a variety of microstructures, and to show that the effective grain size sometimes cannot be determined from optical or bright field transmission electron micrographs. To accomplish this, an Fe-12Ni-0.25Ti alloy was given four types of heat treatment: (1) an anneal-and-quench treatment, (2) an intercritical anneal, (3) a single or double reversion to austenite in a furnace, and (4) a single or double rapid reversion to austenite in an induction furnace. Two effective grain sizes were identified for each heat treatment: the packet size, or dimension over which adjacent martensite laths have a common crystallographic orientation, and the prior austenite grain size. The former controls the ductile-brittle transition temperature, the latter controls intergranular embrittlement. The heat treatments are shown to suppress $T_B$ and increase resistance to intergranular embrittlement to the extent that they decrease the relevant grain size. Rapid reversion, which causes a direct packet refinement, is the most successful of the heat treatments. It readily yields an effective grain size near 1 μm, imparting a very low ductile-brittle transition temperature and a substantial resistance to temper or hydrogen embrittlement.
INTRODUCTION

Since the ductile-brittle transition temperature (T_B) of ferritic steels increases with the ferrite grain size [1-3] the design of alloys for low temperature use requires the development of thermal treatments that refine the effective grain size. The most successful of the thermal treatments that are now used fall into three categories.

The first category includes intercritical tempering treatments, such as the 'QT' treatment of commercial 9Ni steel [4], the 'QLT' treatment of 5.5Ni steel [5,6], and the QQ'T treatments that have been applied to steels of lower Ni content [7] and to Fe-Mn steels [8]. In these treatments the steel is tempered at a relatively low temperature within the two-phase (α+γ) field to precipitate islands of austenite along the lath boundaries of the initial martensite. These austenite islands refine the effective grain size by disturbing the alignment of adjacent martensite laths [9,10], and have been used to suppress the ductile-brittle transition of Fe-Ni and Fe-Mn steels to below 77K.

The second category includes thermal cycling treatments such as multiple austenitizing treatments [11], the '2B' treatment of Fe-12Ni-0.25Ti [12,13], and its derivatives [14-16]. These treatments utilize successive thermal cycles to above the austenite reversion temperature, usually alternated with heating within the two-phase region, to force the alloy through repeated martensitic transformations. The thermal cycling treatments produce a martensitic structure with a small effective grain size. They have been used to suppress the ductile-brittle transition of Fe-Ni steels to below 4K and to achieve excellent toughness in Fe-SMn alloys at 77K [17].

The third category includes the rapid thermal cycling treatments imposed during multi-pass gas tungsten arc welding [18-21]. The multiple thermal cycles experienced during multi-pass welding also refine the effective grain size of the martensite and decrease the ductile-brittle transition temperature. The development of controlled multi-pass GTA welding has permitted the use of ferritic filler metals in industrial weldments that must remain tough at temperatures near 77K [18], and in laboratory weldments that have exhibited high toughness at 4K [19-21]. The technique has even been used to build up an all-weld ferritic plate of commercial 11Ni weld wire that had excellent fracture toughness at 4K [22].

The mechanisms of grain refinement and toughening during intercritical heat treatment have been extensively studied in recent years. While a number of important issues remain, there has been steady progress in the understanding and metallurgical control of this class of heat treatments.

On the other hand, the mechanisms of grain refinement in the thermally-cycled steels have received less attention, and are not so well understood. The research that has been done raises interesting questions. For example, while cycling through the martensitic phase transformation usually produces a microstructure that appears very fine when examined in either the optical or transmission electron microscope, the
The present work was undertaken to clarify the mechanisms of grain refinement in thermally-cycled steels by conducting detailed metallography on samples of a single alloy, Fe-12Ni-0.25Ti, that had been given a variety of simple thermal cycling treatments. The research was particularly intended to specify and interpret the microstructural differences between samples that were given rapid reversion treatments, resembling those experienced during multi-pass welding, and samples that were cycled at the slower rates encountered in more conventional heat treatments.

EXPERIMENTAL PROCEDURE

1. Material Preparation

The alloy used in this investigation had the nominal composition Fe-12Ni-0.25Ti, with less than 0.01C. Its low carbon content and Ti microalloy addition were intended to minimize precipitation during heat treatment. The alloy was melted in vacuum from pure starting materials and cast as a 9.5 kg ingot. The actual chemical composition of the cast ingot is shown in Table I.

Table I: Alloy Composition in Weight Percent

<table>
<thead>
<tr>
<th></th>
<th>Ni</th>
<th>Ti</th>
<th>P</th>
<th>S</th>
<th>O</th>
<th>N</th>
<th>C</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>12.07</td>
<td>0.20</td>
<td>0.001</td>
<td>0.002</td>
<td>0.005</td>
<td>&lt;0.001</td>
<td>0.006</td>
<td>Balance</td>
</tr>
</tbody>
</table>

The ingot was homogenized at 1200°C for 24 hours under argon atmosphere and upset cross-forged at 1100°C into a 15mm thick plate. The plate was then annealed at 1200°C for three hours, furnace-cooled to 900°C and quenched in water. This plate provided the starting material for all subsequent heat treatments. Its condition is designated 'AN'. The AN plate was machined and sliced to slightly over-sized Charpy impact specimens, which were used for the experimental work.

The austenite start and finish temperatures during heating (As and Af) and the martensite start and finish temperatures on cooling (Ms and Mf) were determined by dilatometry. The results were: As = 695°C, Af = 715°C, Ms = 448°C, Mf = 415°C.

2. Heat Treatment

The heat treatments studied in this work are diagrammed in Figure 1. They are of four types. The first is the simple anneal-and-quench that establishes the 'AN' condition. The second is an intercritical
anneal at 650°C for three hours, which leads to the 'B' condition. The third is an austenite reversion treatment at 790°C for one hour in an air muffle furnace, after which the sample is quenched in water. This condition is labelled 'A'. The 'A' treatment is repeated to give the 'AA' condition.

The fourth heat treatment is a rapid reversion treatment to the same peak temperature in an induction furnace. The specimen was heated rapidly to 790°C and then immediately dropped into an oil bath. A single such cycle constitutes the 'C' treatment; a double cycle is designated 'CC'. The temperature was monitored during the rapid reversion cycle by spot-welding a thermocouple onto the specimen surface and embedding a second into the specimen center. The two gave very similar readings and showed that the surface and bulk reached essentially the same peak temperature. An example temperature profile is plotted in Figure 2, and compared with that experienced by a sample that was heated in the furnace to the same peak temperature. Samples treated in the induction furnace reached peak temperature (790°C) in approximately 15 seconds, while those heated in the air-muffle furnace took more than 15 minutes to reach the same temperature.

3. Mechanical Tests

After heat treatment the specimens were machined into the standard Charpy V-notch configuration and tested at various temperatures to measure the ductile-brittle transition temperature. Testing temperatures between room temperature and 115K were controlled in isopentane cooled by liquid nitrogen. The specimen hardness was also measured, but did not vary significantly with heat treatment.

4. Metallography

All of the samples employed in the metallographic studies were cut from broken Charpy impact specimens. For optical microscopy samples were polished and etched with an acidified FeCl₃ solution: 200ml HCl + 200ml H₂O + 20 gr. FeCl₃. This etch reveals the transformation structure and the prior austenite grain boundaries. For transmission electron microscopy samples were spark-cut from chemically-thinned slices. They were electropolished in a twin jet polishing apparatus using a chromic-acetic acid solution (75 gr. CrO₃ + 400ml CH₃COOH + 21ml H₂O) with an operating voltage of 20 V. These specimens were examined in a Philips 301 electron microscope at 100 kV.

A dark field technique was used to distinguish high- and low-angle boundaries in the TEM images. After locating a large visible area in the specimen, bright field images and diffraction patterns were photographed. Dark field micrographs were then exposed, without specimen tilting, from diffraction spots that were uniquely associated with a single diffraction pattern. Adjacent grains, laths or subgrains that were illuminated in dark field micrographs from diffraction spots of a single diffraction pattern were assumed to be separated by low angle boundaries, while those illuminated by spots belonging to distinct diffraction patterns were assumed to be separated by high angle boundaries.
RESULTS

1. The Annealed-and-Quenched (AN) Condition

The microstructure of the alloy in the annealed-and-quenched (AN) state is illustrated by the optical micrograph in Figure 3(a). The alloy is divided into grains of the prior austenite phase, each of which is subdivided into packets of lath martensite. The boundaries of the packets are irregular, as evidenced by their jagged appearance in the plane of the micrograph and by the appearance of islands of one packet in the interior of its neighbor. The packets appear to contain only one variant of the martensite; they generally are not divided into the 'blocks' that are common in other steels [23-26]. The packet size and the prior austenite grain size were measured by the linear intercept method, and are approximately 100 \( \mu \text{m} \) and 500 \( \mu \text{m} \), respectively.

The substructure of the alloy is illustrated by the transmission electron micrograph in Figure 4, which is typical of those taken to characterize the substructure of the steel. This micrograph contains over 70 parallel laths that are long and nearly straight in the plane of the figure. Selected area diffraction patterns were taken over the whole area shown. An example is included in the inset to the figure. The diffraction patterns show that all laths have the same crystallographic orientation, documenting that only a single martensite variant is present. It follows that the laths are separated by low-angle boundaries. These observations are consistent with those of Wakasa and Wayman [27,28], who performed high-resolution studies of lath martensite in Fe-20Ni-5Mn and found that adjacent laths were misoriented by no more than about 2°.

If the packets contain a single variant of martensite then they share (100) cleavage planes and (112) twin planes, and may cleave or twin as a unit. The optical micrographs shown in Figure 5 illustrate this behavior. Figure 5(a) shows a secondary cleavage crack that emanates from the main crack in a Charpy specimen that was tested below the ductile-brittle transition temperature. The secondary crack is nearly straight within a packet, but changes direction at every packet or grain boundary and finally stops at a packet boundary. Figure 5(b) shows a set of mechanical twins that were revealed by etching an area just beneath the brittle fracture surface. The twins are also straight within a packet, but change direction at the packet or grain boundaries. These results document the simple crystallography of the packets and the high-angle character of typical packet boundaries.

It follows from these observations that the packet size is the effective grain size governing transgranular cleavage, while the larger prior austenite grains define the grain boundary network that governs intergranular fracture.

2. The Intercritically-Annealed (B) Condition

The microstructure of the alloy in the intercritically-annealed (B) condition is illustrated by the micrograph in Figure 3(b). The optical microstructure is essentially unchanged from the AN condition (Fig. 3(a)). There is no evidence of recrystallization, and no obvious re-
finement of the packet size. The holding time at the annealing temperature was extended up to 100 hours without any significant change in the optical microstructure.

During the intercritical anneal austenite islands nucleate along the martensite lath boundaries and grow by a diffusion process [29]. These are relatively lean in solute content, and re-transform to martensite on cooling back to room temperature. Previous work [9,10] suggests that the transformation is crystallographically reversible: the precipitated austenite reverts back into the common martensite variant of the original packet.

The reversibility is documented by the transmission electron micrograph shown in Figure 6, which was taken on a specimen that was annealed at 650°C for 100 hours. The regions of fresh martensite in the microstructure (labelled M) are easily distinguished from the regions of tempered martensite (labelled F). Since the equilibrium content of γ phase at 650°C is about 80% by volume, it is not surprising that fresh martensite is the dominant phase. Nonetheless, the selected area diffraction patterns show that only a single martensite variant is present.

The optical and transmission electron microanalyses suggest that the 'B' treatment does not refine the effective grain size of the alloy. If this is true, there should be no significant change in the ductile-brittle transition temperature. Figure 7 shows the variation of the Charpy impact energy of the alloy with temperature after annealing at 650°C for three hours. The corresponding curve for the alloy in the 'AN' condition is also plotted for comparison. The two curves are essentially identical, although there is a small increase in the upper shelf energy in the 'B' condition.

3. The Austenitized (A and AA) Conditions

The microstructures of alloys that were austenitized by single or double cycles to 790°C for one hour are shown in Figure 8. A single austenitizing treatment yields the partially-refined structure presented in Figure 8(a). Similar partial recrystallizations have been reported by a number of authors [30-32]. The second austenitization treatment sufficed to complete grain refinement by recrystallization. As illustrated in Figure 8(b), the second austenitization not only recrystallized unrefined areas such as that shown in the upper portion of Figure 8(a), but also recrystallized the small grains that were formed during the first cycle. The result is a mixed structure of grains that vary in size from 10 to 70 μm.

The substructure of the doubly-austenitized specimen is illustrated by the composite transmission electron micrograph in Figure 9, which includes a schematic drawing to help identify the prior austenite grain boundaries. The composite contains five grains, designated by the letters A-E, and three triple-grain junctions. The lath martensite substructure is visible. The boundary between grains B and D is highly curved, suggesting grain growth. Note the Ti(O,C) precipitate on the curved boundary, which apparently impeded the shrinkage of grain B. The width of grain B is approximately 20 μm.
A crystallographic analysis of the grains included in Figure 9 showed that they are separated by high angle grain boundaries. The analysis is illustrated in Figure 10, which includes a detailed micrograph of the triple junction between grains B, C, and D with accompanying diffraction patterns taken from the circled areas within the grains. The three diffraction patterns are distinct: they were indexed as [001], [T11] and [T13]. The accompanying dark field micrographs show that each diffraction pattern is operative only within a single grain and is bounded by grain boundaries that appear in the bright field image. While more than one dark field micrograph is needed to illuminate the whole interior of a grain, as illustrated in Figure 10(f) and (g), the complementary dark field micrographs are images of beams from a single diffraction pattern. The dark areas within the illuminated grains in the dark field micrographs hence represent regions that are slightly misaligned rather than crystallographically distinct.

Since each grain of the 'AA' structure contains a single packet of lath martensite and is separated from its neighbors by high-angle grain boundaries, it follows that the effective grain size of the 'AA' structure is essentially equal to the grain size of the recrystallized prior austenite grains, and is roughly 30 μm. This refinement of the effective grain size should cause a decrease in the ductile-brittle transition temperature. The Charpy impact energies of the 'A', 'AA' and 'AN' structures are compared in Figure 11. The decrease in T_B is evident. The fracture mode at 77K changed from transgranular cleavage in the 'AN' structure to a mixture of transgranular cleavage and ductile rupture in the 'AA' structure. The double austenitization also caused an increase in the upper shelf toughness, which was not observed in prior work on the same alloy [4] that used a slightly different austenitization heat treatment.

4. The Rapidly-Austenitized (C and CC) Conditions

The microstructures of the alloy in the 'C' and 'CC' conditions are shown in Figure 12. By comparison with the micrographs shown in Figure 8 it is clear that the microstructural change induced by the rapid thermal cycle is quite different from that associated with conventional austenitization. There is no evidence of recrystallization in the 'C' and 'CC' microstructures, but the packet substructure has been disturbed, particularly after the second rapid cycle. In the 'C' condition the prior austenite grain boundaries are roughened and partly obscured by the martensite transformation product. In the 'CC' condition the prior austenite grain boundaries can hardly be found.

The substructure of the alloy in the 'CC' condition is illustrated by the transmission electron micrograph shown in Figure 13. In contrast to the 'AN' (Fig. 4) condition, the lath morphology within the 'CC' alloy is irregular and the lath boundaries are difficult to trace. The crystallographic alignment between adjacent laths has been largely destroyed. The area shown in the bright field micrograph contains at least three different orientations, [011], [001], and [331], which yield the distinct martensite diffraction patterns shown in the figure.

Due to the absence of boundary-like features it is not possible to trace the boundaries between areas of different orientation in the
bright field micrographs. The distinct variants can be separated with dark field techniques, as shown in Figure 14. The region shown in Figure 14(a) also contains at least three martensite variants, which yield three distinct diffraction patterns that are indexed as [113], [001], and [115] (Figures 14(b,c,d)). The dark field micrographs shown in Figures 14(e,f,g) were taken from the diffraction spots that are marked with arrows in these three diffraction patterns. The results show that different martensite variants are intermixed on a very fine scale.

The results of the transmission electron microscopy suggest that the 'CC' alloy has an extremely fine effective grain size, on the order of 1 μm. This ultrafine grain size should result in a very low ductile-brittle transition temperature. Figure 15 is a plot of the Charpy energy against temperature for the 'AN', 'C', and 'CC' conditions, and documents the marked suppression of T_B in the 'CC' condition, where packet refinement is nearly complete. The upper shelf toughness is also raised. Note that the 'C' alloy, which is only partly refined in its packet structure, has only a slightly lower T_B than the 'AN' alloy and has essentially the same upper shelf toughness.

While the 'CC' alloy has an exceptionally low transition temperature, its disturbed microstructure suggested that it might have a high residual stress. To test this point two of the 'CC' specimens were stress-relieved at 600°C for one hour and tested at 77K. The average of the two tests was 149 Joules (110 ft.-lb.), which is slightly below the impact energy of the alloy as-cycled. Hence the internal stress is either small or unimportant.

The very fine effective grain size and the absence of clear prior-austenite boundaries in the 'CC' alloy suggests that the alloy may have exceptional resistance to embrittlement by tempering or hydrogen. To test this hypothesis specimens in the 'AN', 'AA', and 'CC' conditions were tempered at 450°C for 300 hours, notched, and then charged with hydrogen at a current density of 10 mA/cm² for 24 hours. The specimens were bolt-loaded immediately after charging to initiate a crack from the machined notch, and broken completely after the crack arrested. As shown in Figure 16, the 'AN' and 'AA' specimens fractured in an intergranular mode along the prior austenite grain boundaries. The 'CC' alloy was resistant to fracture, and eventually broke in a completely ductile mode.

DISCUSSION

1. The Effective Grain Size

Both the research described here and prior work [9,10] show that the brittle fracture of this and similar alloys is controlled by an effective grain size, which is related to the crystallographic coherence length within the microstructure. The proper definition of the effective grain size depends on the phenomenon of interest, since the several brittle failure modes that may occur in the alloy involve the fracture of distinct microstructural elements.
The ductile-brittle transition occurs through the intrusion of transgranular cleavage predominantly along \( \{100\} \) cleavage planes, and should hence be controlled by the mean free path of a \( \{100\} \) cleavage plane. In the present alloy this length is equal to the diameter of a coherent packet of martensite laths. As shown in the body of the paper, and in prior work \([9,10,12]\), heat treatments that refine the packet size decrease \( T_B \).

Fracture that is due to temper or hydrogen embrittlement, on the other hand, is predominantly along the prior austenite grain boundaries (or, in clean alloys, along the martensite lath boundaries \([33]\)). The 'CC' treatment essentially destroys the prior austenite grain structure, and replaces it with an extremely fine microlath structure that has a correspondingly fine effective grain size. Such an alloy is expected to resist embrittlement, and does.

However, the effective grain size of the alloy is often impossible to determine from optical or even bright field transmission electron micrographs. Low-angle lath boundaries are not easily distinguished from high-angle packet or grain boundaries, and even boundaries that separate distinct regions of the microstructure, as in the 'B' microstructure (Figure 6), may not introduce crystallographic discontinuities. Selected area diffraction and dark field microscopic techniques seem to be essential to the accurate determination of the effective grain size.

2. Grain Refinement by Thermal Treatment

In the present work the martensite transformation was used in four different ways in an attempt to refine the effective grain size of the alloy. These were: (1) transforming annealed austenite to give the 'AN' condition, (2) transforming austenite that was precipitated along lath boundaries to give the 'B' condition, (3) transforming recrystallized austenite to give the 'A' and 'AA' conditions, and (4) rapidly transforming reverted austenite to give the 'C' and 'CC' conditions. Each of these treatments influences the effective grain size in a distinct way.

a. The 'AN' treatment.

If the alloy is annealed so that it has a large austenite grain size and quenched, martensite forms in coherent packets. There are generally several distinct packets within a grain. These define the effective grain size for cleavage fracture, and hence control \( T_B \), while the larger prior austenite grain size controls intergranular embrittlement.

b. The 'B' treatment.

If the alloy is then intercritically annealed at a temperature near the top of the two-phase \((\alpha+\gamma)\) region, islands of austenite form along the martensite lath boundaries. These re-transform to martensite on cooling, but revert to that variant of martensite that is shared by the surrounding packet. Both the packet size and the prior austenite grain size remain the same.
c. The 'A' and 'AA' treatments.

If the alloy is reverted to austenite and allowed to crystallize before quenching then the prior austenite grain size is refined. If the prior austenite grain size is held in the 10-70 \( \mu \)m range, as it is in the present work, then the austenite grains usually contain a single packet of martensite. The prior austenite grain size then defines the effective grain size for both cleavage and intergranular embrittlement.

The efficiency of an austenitization treatment in refining the grain size depends on the rate and generality of recrystallization in the microstructure. While the mechanism of recrystallization is not completely known, the process is well explained by the model proposed by Maki, et al. [31] and by Nakazawa, et al. [32]. In this model the driving force for recrystallization is provided by a high dislocation density in the austenite after reversion by a shear process. Various workers [34-38] have found dislocation densities in reverted austenite that reach as high as \( 10^{11} \) cm\(^{-2} \) [35], which is approximately the dislocation density in martensite. The recrystallization temperature depends on the defect density, and may lie above the \( A_f \) temperature; the grain refinement of maraging steels often requires heating to well above the \( A_f \) temperature. The extent of recrystallization should also depend on the heating rate, since a minimum heating rate is needed to ensure a shear reversion of the austenite. However, previous work [32] has shown an uncertain dependence between heating rate and grain refinement in reverted maraging steels.

In the present work recrystallization was assured by choosing a peak temperature of 790°C, above the \( A_f \) temperature of approximately 715°C. But it still was not possible to obtain a uniform recrystallization in a single heat treatment. The incomplete recrystallization may be due to a low defect density in the reverted austenite, to a failure to obtain complete shear reversion at the heating rates used, or to a partial recovery of the transformation defects during heating between the \( A_f \) and the recrystallization temperature. In the present work complete recrystallization was accomplished by repeating the thermal cycle to give the 'AA' treatment. The more complete recrystallization in the 'AA' condition suggests that the second austenitization treatment produces a higher defect density, as has, in fact, been observed in previous work on reverted austenite [37].

While the grain refinement achieved in the 'AA' condition in this work was not sufficient to suppress T\(_B\) to below liquid nitrogen temperature, similar treatments that were used in prior work on the same alloy (but with a more refined starting microstructure) were sufficient to establish high toughness at 77K [39]. The ultimate grain refinement that can be obtained through this sort of cycle is not now clear, but this and prior work suggest that it is unlikely that an effective grain size below about 10 \( \mu \)m can be obtained by repeated austenitizing treatments [39,40].

d. The 'C' and 'CC' treatments

The rapid reversion treatment that is used in the 'C' and 'CC' treatments creates an extremely fine effective grain size by destroying
the alignment of adjacent martensite laths. We hence use the term 'packet refinement' to distinguish this mechanism from the 'grain refinement' associated with the recrystallization of the austenite. Packet refinement is clearly the most efficient means for decreasing the effective grain size. Effective grain sizes near 1 \( \mu \text{m} \) were obtained with the 'CC' treatment. This fine grain size governs both cleavage and intergranular embrittlement, since the prior austenite grain boundaries are roughened or obliterated by the transformation. The resulting alloy has a ductile-brittle transition temperature well below 77K, and fractures in a ductile mode after tempering and hydrogen charging.

While the mechanism of packet refinement in a rapid austenitization cycle needs further investigation, the results are plausible in the light of current understanding of austenite reversion. Rapid reversion prevents recrystallization, so that the austenite that retransforms into martensite on quenching has a high density of defects. The martensite is crystallographically related to its parent by the Kurdjumov-Sachs relations: \((111)_\gamma \parallel (011)_\alpha\), \([110]_\gamma \parallel [111]_\alpha\). These relations permit 24 variants of martensite to appear in a single prior austenite grain, but for reasons that are not well understood the martensite forms cooperatively, in blocks or packets of a single variant. Given the high defect density in reverted austenite, and the propensity for the heterogeneous nucleation of martensite, it is certainly possible that the cooperative growth of adjacent laths is suppressed so that martensite laths appear in small elements whose variants are more randomly chosen from among those permitted by the K-S relations.

The possibility of achieving ultrafine grain size through a rapid austenitization treatment has been used, empirically, in the development of the multipass welding techniques that permit the welding of ferritic cryogenic structural steels with ferritic filler metals [18-21], and hence has commercial significance. No comparable scheme has been developed for ferritic base plates. The thermal treatments that have successfully suppressed the \( T_B \) of the base plate to very low temperatures are relatively complex thermal cycling treatments that can be used with slower heating rates. The clear identification of the mechanisms of packet refinement does, however, hold the intriguing possibility that similar microstructures may be achieved with heat treatments that are applicable to base alloys in useful thicknesses.

CONCLUSION

The present work was primarily intended to illustrate and clarify the three distinct metallurgical mechanisms that refine the effective grain size of lath martensitic steels: (1) the decomposition of austenite into distinct packets of martensite, (2) the recrystallization of the austenite during reversion, and (3) the direct refinement of the martensite packet by rapid reversion. These treatments are successful insofar as they decrease the effective grain size of the steel, which is the martensite packet size for the ductile-brittle transition and the prior austenite grain size for intergranular embrittlement. Direct packet refinement is the most effective of the three. It readily yields an effective grain size near 1 \( \mu \text{m} \), imparting a very low ductile-brittle transition temperature and a substantial resistance to temper or hydro-
gen embrittlement. The detailed mechanism of packet refinement is, however, unclear.

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FIGURE CAPTIONS

1. Schematic drawing of the heat treatments used in this investigation.

2. Time-temperature profiles of (a) the rapid austenitizing cycle in the induction furnace and (b) the austenitization treatment in an air-muffle furnace.

3. Optical micrographs of (a) annealed-and-quenched, and (b) intercritically-annealed specimens.


5. Cross sections of Charpy impact specimens of the AN alloy broken at 77 K. Microstructure (a) shows secondary cleavage cracks; (b) shows mechanical twins.

6. Transmission electron microstructure of a specimen that was intercritically annealed for 100 hours.


8. Optical microstructures of (a) single- and (b) double-austenitized specimens.

9. Bright field transmission electron micrograph of a double-austenitized specimen. The insert is a schematic drawing of the grain boundaries.

10. Bright field transmission electron micrograph (a) of a part of the field shown in Fig. 9. The diffraction patterns shown in (b), (c) and (d) were taken from the circled areas marked by B, C and D respectively. The dark field micrographs, (e), (f), (g) and (h), were taken from the diffracted spot(s) marked with arrows in the diffraction patterns.

11. Ductile-brittle transition curves of single- and double-austenitized specimens.

12. Optical micrographs of the rapidly-austenitized specimens: (a) single, and (b) double cycled.

13. Transmission electron micrograph of the double-cycled specimen. The diffraction patterns labelled b, c, d and e, were taken from the circled areas marked B, C, D and E respectively.

14. A low magnification, bright field transmission electron micrograph of the double-cycled specimen is given as (a). The dark field micrographs, e, f, and g, were taken from the diffracted spots that are marked with arrows in the diffraction patterns b, c and d respectively.
15. Ductile-brittle transition curves of the single- and double-cycled specimens.

16. Scanning electron fractographs of specimens that were tempered at 450°C and then charged with hydrogen. (a) The 'AN' condition; (b) the 'AA' condition; (c) the 'CC' condition.
Figure 1

- Annealing Treatment
  - Fe → Ni → AN
  - $1200^\circ C, 3$ hrs

- Intercritical Annealing Treatment
  - $650^\circ C, 3$ hrs

- Austenitizing Treatment
  - $790^\circ C, 1$ hr

- Rapid Austenitizing Treatment
  - $790^\circ C, 15$ sec

Designation
- AN
- B
- A
- AA
- C
- CC

XBL841-6522
Figure 2
Figure 3  XBB 834-3068
Figure 4
Figure 5
Figure 6
Figure 7

- AN: 1200°C/3 hrs
- B: 650°C/3 hrs
Figure 11
Figure 12
Figure 15
Figure 16

XBB 830-10356
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