Isothermal Transformations in an Fe-9 Pct Ni Alloy

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Isothermal transformation from austenite in an Fe-9.14 pct Ni alloy has been studied by optical metallography and examination by scanning electron microscopy (SEM) and transmission electron microscopy (TEM). In the temperature range 565 °C and 545 °C, massive ferrite (α_q) forms first at prior austenite grain boundaries, followed by Widmanstätten ferrite (α_W) growing from this grain boundary ferrite. Between 495 °C and 535 °C, Widmanstätten ferrite is thought to grow directly from the austenite grain boundaries. Both these transformations do not go to completion and reasons for this are discussed. These composition invariant transformations occur below T_0 in the two-phase field ($\alpha + \gamma$). Previous work on the same alloy showed that transformation occurred to α_q and α_W on furnace cooling, while analytical TEM showed an increase of Ni at the massive ferrite grain boundaries, indicating local partitioning of Ni at the transformation interface. An Fe-3.47 pct Ni alloy transformed to equiaxed ferrite at 707 °C ± 5 °C inside the single-phase field on air cooling. This is in agreement with data from other sources, although equiaxed ferrite in Fe-C alloys forms in the two-phase region. The application of theories of growth of two types of massive transformation by Hillert and his colleagues are discussed.

I. INTRODUCTION

GILBERT and Owen[1] first reported massive transformations in iron substitutional alloys. Since that time, it has become apparent that there are two massive transformations in iron alloys: equiaxed ferrite[2] (α_p) and massive ferrite (α_q).[2,3] The nomenclature in brackets, here and later is attributed to Araki.[4] Wilson also contends[5–8] that the massive transformation in Cu-9.3 pct Al may occur partly if not completely by shear to resemble massive martensite in the polished and etched microstructure. In References 9 and 10, transformations occurring in an Fe-9 pct Ni alloy at various cooling rates were reported. On cooling at 0.13 K/s (equivalent to furnace cooling), composition invariant massive ferrite formed between 575 °C and 556 °C. The transformation range means that the massive ferrite forms in the ($\alpha + \gamma$) phase region, below the A_3 of 715 °C[11,12] and T_0[13,14] of 623 °C ± 8 °C.[9,10,15] This result confirms directly Massalski's observation[16] that the massive transformation occurs in an Fe-9.12 pct Ni alloy in the two-phase region beyond the retrograde $\alpha/(\alpha + \gamma)$ phase boundary on cooling at 7 K/s. A typical optical micrograph of massive ferrite and transmission electron microscopy (TEM) micrograph are shown in figures 1[3] and 2,[17] respectively. Note the irregular grain boundaries and high dislocation density. In contrast, a typical optical micrograph of equiaxed ferrite and TEM micrograph are shown in Figure 3.[18] The TEM micrograph illustrates the low dislocation density and the equiaxed microstructure similar to well-annealed metals. To return to the Fe-9.14 pct Ni alloy on cooling at 0.13 K/s, below 556 °C, Widmanstätten ferrite grew from the grain boundary ferrite exhibiting tent relief,[9,19–22] on prepolished surfaces. Subsequent microanalysis by TEM showed an enhancement of Ni at the grain boundaries of the massive ferrite in the furnace-cooled Fe-9.14 pct Ni alloy.[9,10] This indicates that Ni must partition in the interface during transformation. The observation of martensite-austenite (MA) constituent in furnace-cooled Fe-9.14 pct Ni[9,10] also indicates that C partitions in the interface, although this point is debated later under further discussion.

Current thinking is that there are six transformations in pure iron; equiaxed ferrite (α_p) at 870 °C ± 10 °C, massive ferrite (α_q) 800 °C ± 10 °C, Widmanstätten ferrite (α_W) 760 °C ± 10 °C, bainitic ferrite 725 °C ± 20 °C (α_B^0), lath (massive) martensite (α'_m) 545 °C ± 5 °C, and twinned martensite (α'_{TM}) 420 °C ± 10 °C.

The present article deals with attempts to study isothermal transformation in the same Fe-9 pct Ni alloy as studied in Reference 9.

II. EXPERIMENTAL PROCEDURE

The alloy, designated VS2241A, used in this investigation was prepared as a 25 kg ingot by vacuum melting by Corus UK Ltd., Swinden Technology Centre (Rotherham). After removal of the pipe, the alloy was upset forged at Corus UK Ltd., Stocksbridge Engineering Steels, and then homogenized for 4 days at 1200 °C under argon. Analysis showed the alloy contained 9.14 wt pct Ni, 0.002 wt pct C, and 0.008 wt pct N and less than 1480 ppm residuals by mass.

Specimens, 5 × 5 × 35 mm, were cut from the homogenized bar. A hole about 5-mm deep was drilled at one end of each specimen and temperature resistant wire attached for handling purposes. For some of the experiments, a thermocouple was attached instead. The specimens were austenitized for 20 minutes at 1150 °C under argon blown into the furnace. After austenitizing, the specimens were rapidly transferred to the isothermal salt baths, controlled to ±5 °C, and quenched in iced brine, after isothermal holding times of 20 minutes, 1 hour, 5 hours, and 24 hours. The temperature of the specimens with thermocouples attached was continuously

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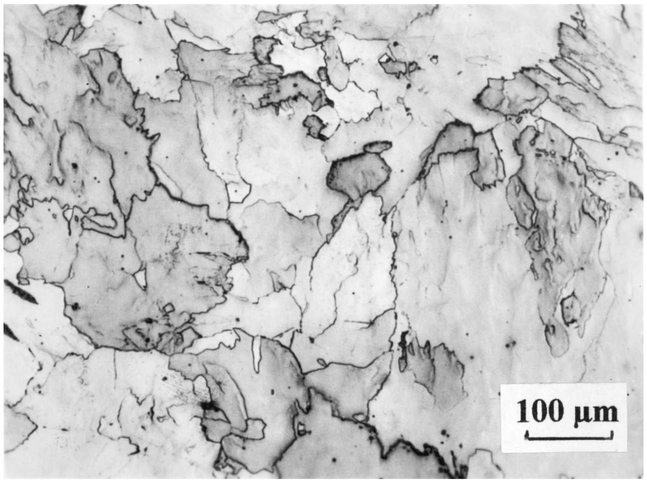

Fig. 1—Optical micrograph of massive ferrite in Fe-3.9 pct Cu-0.005 pct C taken from the work of Wilson.[3] Specimens 2.54-mm thick were austenitized for 3 days at 1050 °C under dry hydrogen and quenched into iced brine.

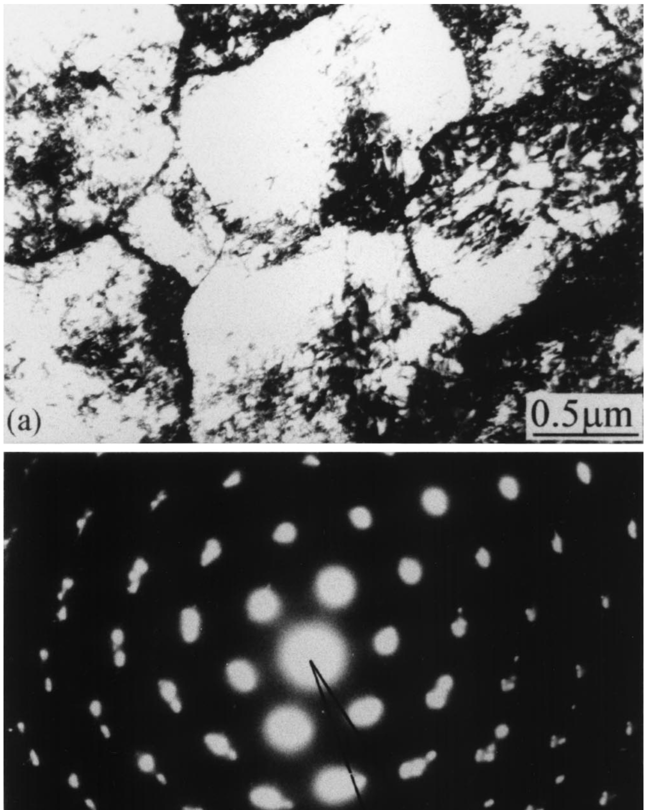

Fig. 2—Transmission electron micrograph of massive ferrite in Fe-15 pct Ni. Dilatometer specimen, 5-mm diameter × 10-mm length, cooled at 50 K/min (0.83 K/s) after 1 h at 1000 °C under vacuum. Massive ferrite formed in the temperature interval 386 °C to 372 °C. (*a*) Bright-field image and (*b*) electron diffraction pattern of (a) showing slight misorientation between grains.[17]

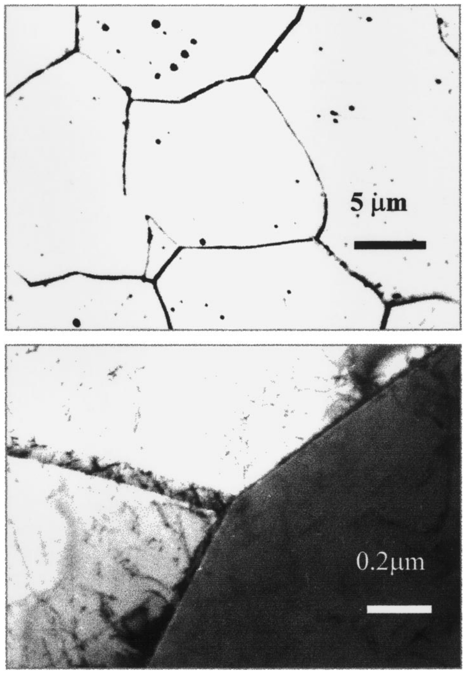

Fig. 3—Light optical and electron micrographs of equiaxed ferrite in Fe-2.5 pct Mn-0.01 pct C austenitized for 1800 s (30 minutes) at 1200 °C. Isothermally treated for 600 s at 610 °C.[18]

recorded. This enabled the effectiveness of the quenching to be observed as well as allowing the stability of the isothermal temperature to be monitored. Cross sections of the specimens were examined by metallography and by scanning electron microscopy (SEM). The extent of transformation was determined by point counting. A total of 18,000 points were used for each specimen giving an accuracy of ±2.3 pct.[23]

Discs for transformation electron microscopy (TEM) were cut from machined 3-mm rods and ground on emery paper to a thickness of 80 to 100 μm. Electropolishing was carried out using a twin jet polisher and 5 pct perchloric acid in methanol. Polishing conditions were 30 to 35 V and 100 to 200 mA at ∼−40 °C. Thin foils were examined in a PHILIPS CM20 electron microscope operating at 200 kV.

III. RESULTS AND DISCUSSION

Initially, specimens were austenitized at 1000 °C, but it was found that only material austenitized for 20 minutes at 1150 °C and isothermally transformed in salt baths could be resolved for examination under the optical microscope. Figure 4 shows the continuous cooling curve observed on quenching an Fe-9.14 pct Ni specimen into a salt bath at 455 °C. It will be seen that some athermal transformation occurs at 557 °C, prior to isothermal transformation at 455 °C. In retrospect, it would have been better to have used a thin sheet for isothermal transformation experiments to achieve a faster cooling rate. In addition, the percentages of transformation shown in Figure 5 were only determined for holding times of 20 minutes or longer. For shorter times, it was difficult to identify the transformed phases under the optical microscope. The shorter the transformation time, the more ambiguous was the phase boundary revealed by nital etching. Despite these problems, the data given in Figure 5 are similar to those obtained by Moiseyev *et al.*[24] by dilatometry, albeit with shorter time scales (seconds). This

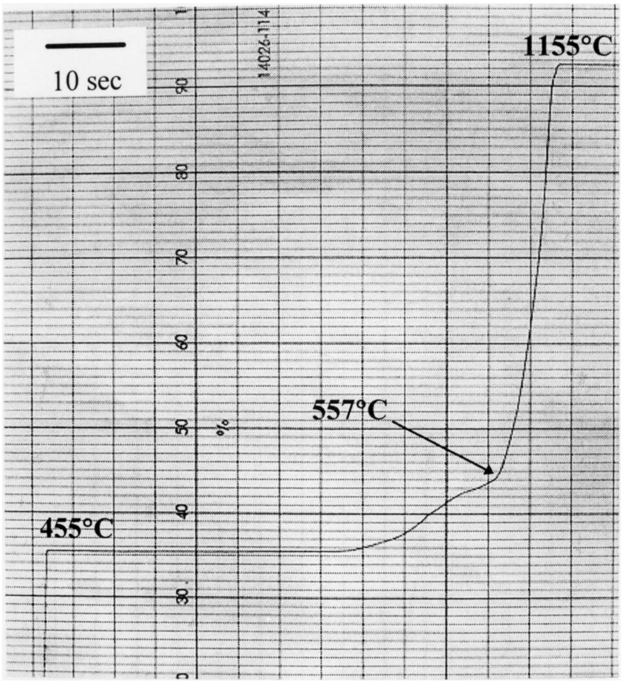

Fig. 4—Cooling curve of a specimen of Fe-9.14 pct Ni subjected to austenitizing temperature of 1150 °C for 20 min, quenched into a salt bath at 455 °C, and soaked for 1 min followed by ice brine quenching. Note thermal arrest, indicating partial transformation at 557 °C.

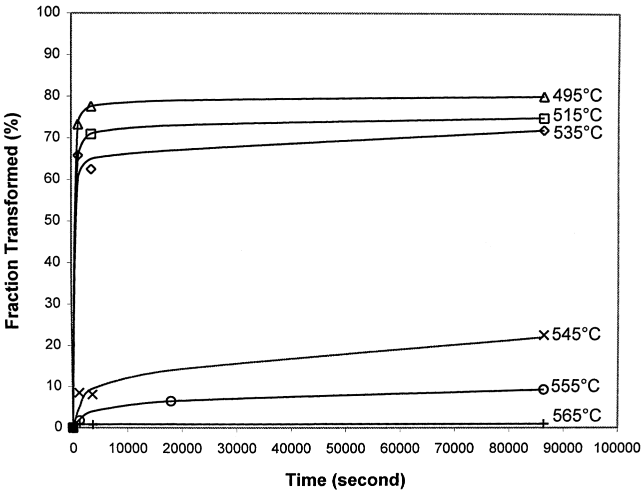

Fig. 5—Percentage transformation determined by point counting isothermally transformed specimens of Fe-9.14 pct Ni (VS2241A). Specimens were austenitized at 1150 °C for 20 min, soaked in a salt bath at various temperatures and for different periods, followed by ice brine quenching.

difference in time scale is probably due to the much lower austenitizing treatment of 15 minutes at 800 °C, used by Moiseyev *et al.* Since the ferrite observed at these temperatures is nucleated at the prior austenite grain boundaries, this will explain the faster rate of transformation in Moiseyev *et al.*'s data being due to the much smaller austenite grain size. It is apparent in Figure 5 that the data could be grouped into two sets; 495 °C to 535 °C and 545 °C to 560 °C and this is confirmed by the metallography to be described later.

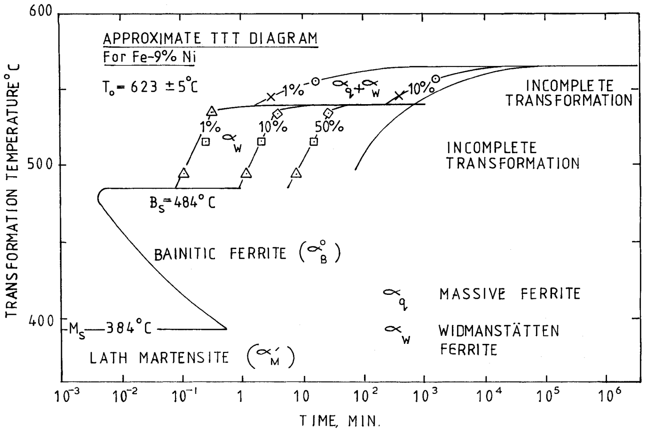

Fig. 6—Approximate TTT diagram for Fe-9.14 pct Ni alloy (VS2241A) deduced from Fig. 5, metallography and transformation points on continuous cooling.

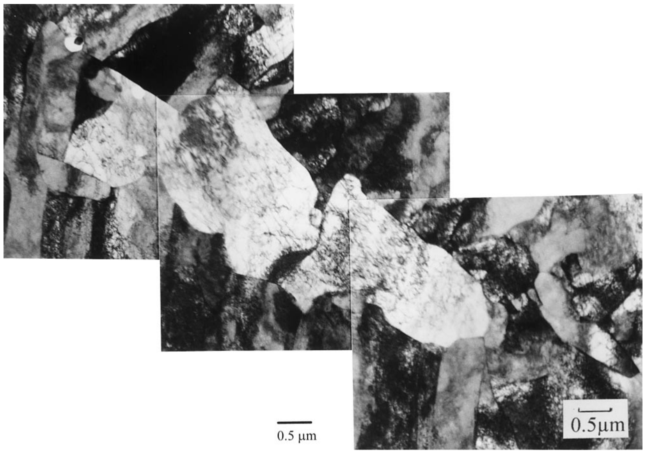

Fig. 7—TEM montage of grain boundary ferrite formed in Fe-9.14 pct Ni after austenitizing for 20 min at 1150 °C and isothermal transformation for 2 min at 555 °C. Note the absence of a trace of prior austenite grain boundary.

Two groups are also apparent in the work of Moiseyev *et al.*, 520 °C to 546 °C and 548 °C. For the temperature interval of 495 °C to 535 °C, equations of the form $y = a\{1 - \exp - (bt)\}$ were fitted to the data and $y = c + d \ln t$, for the data at 545 °C and 555 °C, where y = fraction transformed after time t. This together with the earlier continuously cooled data[9,10] yielded the approximate TTT diagram in Figure 6. No noses in the "C" curves for massive ferrite and Widmanstätten ferrite are evident in Figure 6. This is thought to be due to the paucity and low accuracy of the data. Figure 7 shows a montage of massive ferrite nucleated at a prior austenite grain boundary. Although this figure shows a considerable amount of massive ferrite, the overall effective volume fraction of massive ferrite is small because it is nucleated and only observed on prior austenite grain boundaries. The austenite grain size is large due to the high austenitizing temperature of 1150 °C. Note the absence of a trace of the austenite grain boundary within the transformed grains of Figure 7. This may be compared with Figure 8 from the same specimen also reacted for 2 minutes at 555 °C. In this case, what is thought to be the prior austenite grain boundary

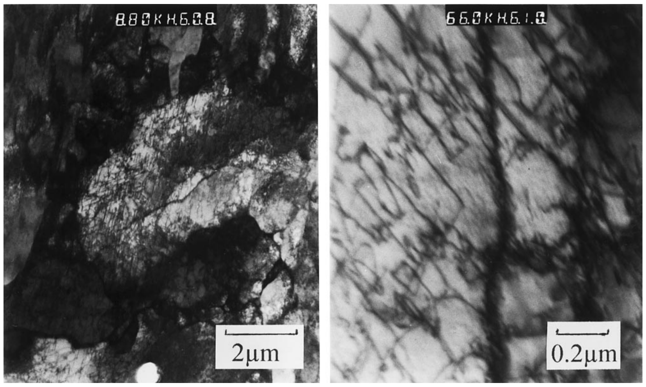

Fig. 8—Specimen given same treatment as Fig. 7. Note the presence of the prior austenite grain boundary in grain boundary ferrite and the absence of the precipitation on this boundary at high magnification.

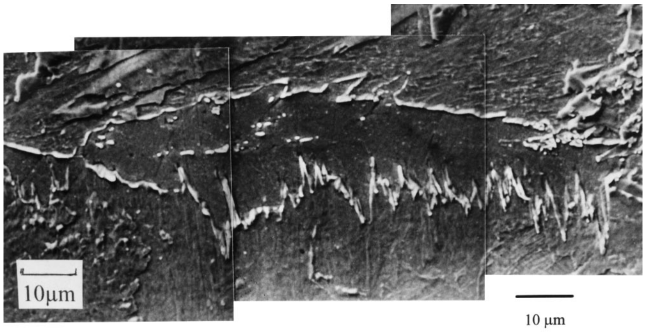

Fig. 9—SEM montage showing growth of grain ferrite with sawteeth protuberances. Specimen of Fe-9.14 pct Ni austenitized for 20 min at 1150 °C and isothermally transformed for 24 h at 555 °C.

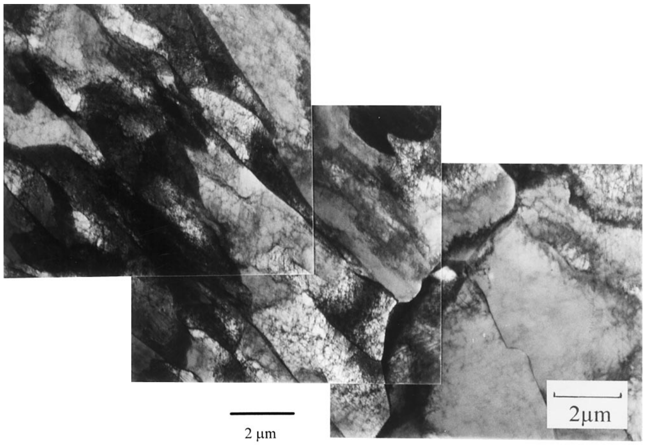

Fig. 10—TEM montage of alloy, VS2241A, Fe-9.14 pct Ni showing secondary Widmanstätten laths nucleated and growing from grain boundary ferrite. Specimen austenitized for 20 min at 1150 °C and isothermally transformed for 20 mins at 535 °C.

is visible in the TEM micrographs. The reason for the presence or absence of traces of prior austenite grain boundaries in micrographs is not known. The trace can perhaps be compared with the presence of a midrib in martensite,[25,26] as the initial site at which transformation starts. An SEM montage taken after a much longer isothermal holding time (24 hours) at 555 °C than Figure 7 is shown in Figure 9. There is some decoration of the prior austenite grain boundary in this case. Note the grain boundary ferrite develops into Widmanstätten ferrite. This process is better illustrated by the TEM montage in Figure 10, albeit at a lower temperature of 535 °C and shorter time (20 minutes). To aid identification of transformed areas, microhardness impressions with a 50 g load were made on microstructures. Typical results are illustrated in Figure 11, for a specimen of Fe-9.14 pct Ni transformed for 24 hours at 555 °C. The transformed ferrite has a hardness value of 170 to 178 HMV50, while the lath martensite obtained on quenching has a hardness of 214 to 224 HMV50.At temperatures of 515 °C and 495 °C, it was found that the fine detail in the transformed phase was practically impossible to reveal by nital etching. The isothermally transformed phase was essentially featureless. However, with prolonged isothermal holding, the transformed structure could be recognized as primary Widmanstätten laths that grew directly from the prior austenite grain boundaries, for example, Figure 12.

IV. FURTHER DISCUSSION

A. *Equiaxed Ferrite*

The microstructure of this composition invariant transformation has been described in the introduction. It is a high-temperature reaction, being the first transformation encountered in iron and substitutional alloys below T_0. It is thought to occur by nucleation as an incoherent allotriomorph at austenite grain boundaries and grain corners and to grow by rapid atom transfer across the incoherent boundary, similar to recrystallization. The theories of Hillert[27] and his colleagues[28,29] for a massive transformation with a high growth rate and weak solute drag are thought to be applicable to this transformation.

Chong[30] observed equiaxed ferrite at 707 °C ± 5 °C at a cooling rate of 4.4 K/s in an Fe-3.47 pct Ni alloy, while Rehman and Edmonds observed equiaxed ferrite in an Fe-2.5 pct Mn −0.01 pct C alloy after 600 seconds (10 minutes) at 610 °C (Figure 3). These reactions occur below T_0 of 767 °C ± 10 °C[15] and 760 °C ± 10 °C,[31,32] respectively, in the single-phase region. In the case of Fe-C alloys, the work of Räsänen shows that the equiaxed ferrite reaction occurs in the two-phase field, below T_0[32,33] to give supersaturated ferrite up to at least 0.1 mass pct C.[34] Thus, it is possible that Ni and Mn do not partition in the interface of this transformation, but interstitials do partition exerting a weak solute drag.

B. *Massive Ferrite*

This is now thought to be the main transformation observed by Gilbert and Owen[1] in Fe, Fe-Ni, Fe-Cr, and Fe-Si alloys with a chemical driving force varying between 42 J/mol in pure iron to 335 J/mol in binary alloys containing 10 at. pct solute. This microstructure has been described in Section I and was investigated by Chong in Fe-9 pct Ni on continuous cooling.[9,10] Nucleation is thought to occur at austenite grain boundaries and triple points with one or more coherent boundaries. The high dislocation density of massive ferrite is thought to arise from the volume change at lowish temperatures and coherency strains. Growth is thought to occur by movement of the incoherent boundaries with the

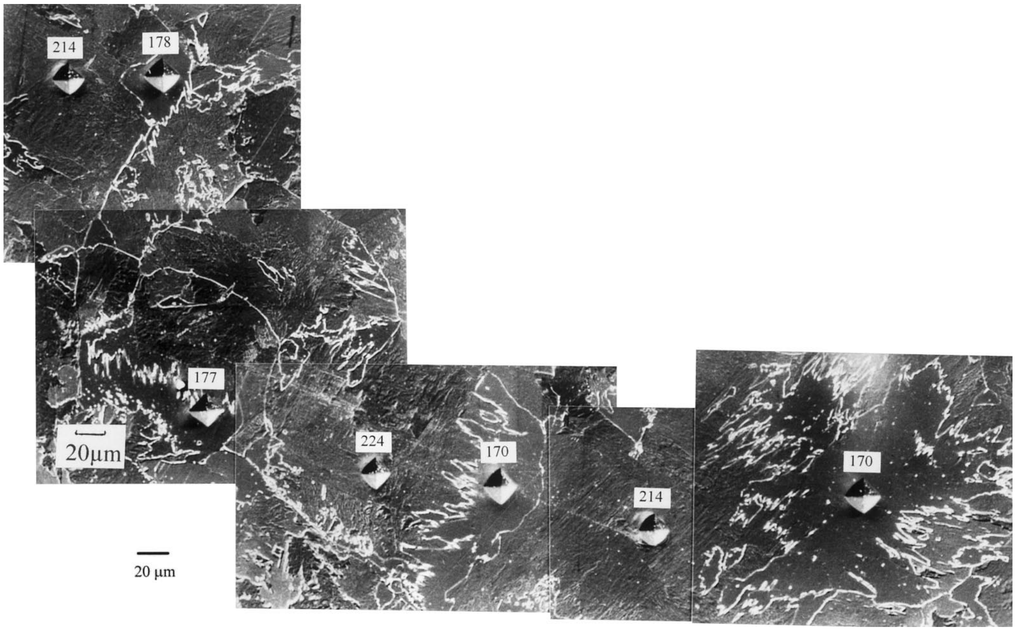

Fig. 11—SEM montage of specimen of Fe-9.14 pct Ni austenitized for 20 min at 1150 °C and isothermally transformed for 24 h at 555 °C, followed by ice brine quenching. Microhardness impressions indicate hardness of transformed grain boundary ferrite, 170 to 178 HMV50 and martensite 21 to 224 HMV50.

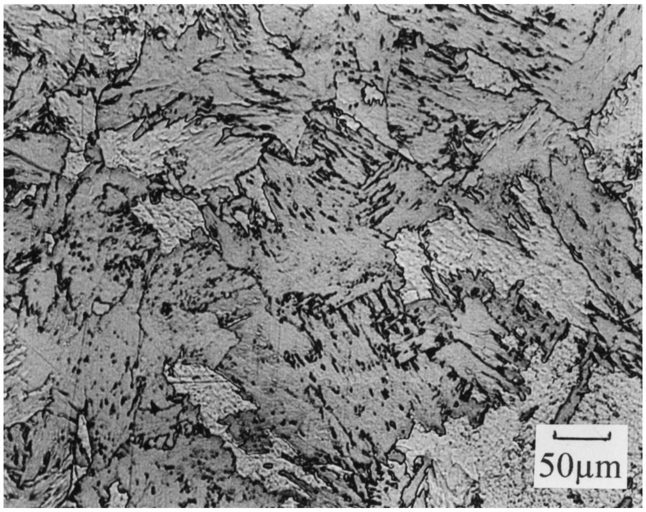

Fig. 12—Optical microstructure of specimen of 9.14 pct Ni austenitized for 20 minutes at 1150 °C, isothermally transformed for 24 h at 515 °C. Etched in pct. The structure consists of primary Widmanstätten ferrite grown directly from austenite grain boundaries.

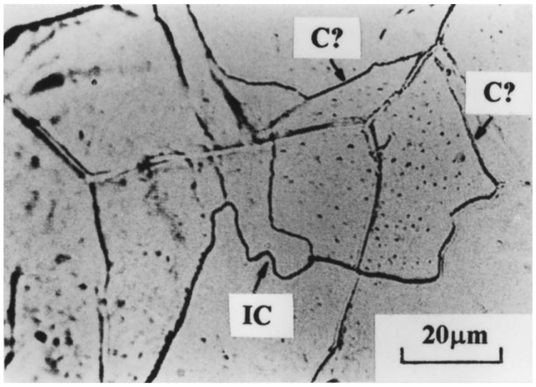

Fig. 13—Possible coherent (C?) and definite incoherent (IC) boundaries in Fe-3 93 pct Ni-0.01 pct C, austenitized 1000 °C under vacuum. Cooled under vacuum; estimated cooling rate ~100 K/s. Subsequently etched in nital. Prior austenite grain boundaries are revealed by thermal grooving. Note the ferrite boundaries freely crossing prior austenite boundaries. The structure is thought to be massive ferrite. Re-edited from Owen and Wilson.[6]

coherent boundaries keeping pace by some mechanism, possibly lateral movement of ledges. Possible evidence for this form of nucleation is given by Figure 13, although this specimen was continuously cooled. The work of Chong on Fe-9 pct Ni[9,10] shows that, at least in the case of massive ferrite obtained by continuous cooling, partition of Ni occurs at the interface. The fact that the MA constituent is observed in the same microstructure indicates that possible partitioning of carbon also occurs at the interface. However, MA could also arise from the austenite remaining from incomplete transformation, enhanced with carbon, forming martensite on subsequent cooling. The theories of Johnsson and Ågren,[28] Purdy and Brechet,[29] and Hillert[27] are thought to be applicable to this type of transformation, with slow growth and strong drag force due to the interface spike of substitutional and possibly interstitial elements.

C. *Widmanstätten Ferrite, (α_W), Bainitic Ferrite (α_B^0), and Lath Martensite (α'_M)*

On isothermal holding, Widmanstätten ferrite (α_W) forms after massive ferrite (α_q) in the range 545 °C and 565 °C and on its own between 495 °C and 535 °C. On water quenching at 210 K/s, bainitic ferrite (α_B^0) formed at 484 °C, and lath martensite at 348°C on brine quenching at 420 K/s.[9] It is not easy to distinguish between Widmanstätten ferrite (α_W), Bainitic ferrite (α_B^0), and massive (lath) martensite (α_m') unless the transformation temperature is known. Widmanstätten ferrite has slightly wider laths, 0.9 to 1.65 μm, compared to bainitic ferrite and massive martensite, ~0.65 μm. The dislocation density within the laths increases in the order Widmanstätten ferrite, bainitic ferrite, and massive (lath) martensite. In Reference 17, Widmanstätten ferrite in an Fe-15 pct Ni alloy was found to have a $\{110\}_\alpha$ habit in agreement with Reference 35, while bainitic ferrite had a $\{110\}_\alpha$ habit, accurate to 1 deg in the same alloy. In the latter case, there was retained austenite present giving a $\{111\}_\gamma$ habit parallel to $\{110\}_\alpha$. However, the lath martensite in the Fe-15 pct Ni alloy was found to have a $\{112\}_\alpha$ habit with an accuracy of 5 deg, rather than the more commonly reported habit of $\{110\}_\alpha$.

D. *Incomplete Transformation*

It will be seen from Figures 5 and 6 that neither the massive ferrite transformation nor the Widmanstätten ferrite transformation go to completion, but are asymptotic to a certain volume fraction $v_{\text{crit.}}$. In the case of α_q and α_W, this varies from less than ~ 2 pct at 565 °C to ~ 25 pct at 545 °C, and in the case of Widmanstätten ferrite (α_W), ~ 70 pct at 535 °C to ~ 80 pct at 495 °C. In both cases, v_{crit} increases as the temperature decreases. This is probably because nickel is an austenite stabilizer, decreasing T_0 and forcing the reactions to be terminated earlier. A similar phenomenon is observed in the bainite reaction, which has been explained in terms of rejection of C from the bainite into the austenite, the reaction ceasing when the local composition of the austenite reaches T_0'[36] (T_0' is the metastable equilibrium temperature in the presence of strain energy[13,37–39]). Perhaps a similar explanation applies to the case of massive ferrite and Widmanstätten ferrite observed here due to the accumulation of Ni and possibly C at the transforming interface.

On further holding of the 9.14 pct Ni alloy, some recovery of the structure was observed. Still further holding would result in the formation of equilibrium austenite from the supersaturated ferrite. This equilibrium austenite is referred to as reverted austenite in the maraging literature.[40]

V. CONCLUSIONS

1. In an Fe-9 pct Ni alloy on austenitizing at 1150 °C and transforming isothermally, massive ferrite forms first followed by Widmanstätten ferrite in the temperature range 545 °C to 565 °C. Widmanstätten ferrite is thought to form on its own in the temperature range 495 °C to 535 °C. However, the reactions do not go to completion (Figure 5). Reasons for none completion of these reactions are discussed.
2. These reactions occur below T_0 but in the two-phase field.
3. Massive ferrite consists of ragged grains and heavy dislocation density, while Widmanstätten ferrite consists of broad laths of moderate dislocation density.
4. These two morphologies inherit the composition of the parent austenite and are supersaturated with C and Ni.
5. Evidence from other alloys and authors show that equiaxed ferrite forms with smooth grains and low dislocation density[2,18,34] in the two-phase field in the case of Fe-C[34] but in the single-phase field in the case of Fe-3.47 pct Ni[30] and Fe-2.5 pct Mn-0.01 pct C,[18] both below T_0.
6. It is thought that the theories of Hillert[28] and his colleagues[28,29] apply to these reactions: high growth rate and weak solute drag to equiaxed ferrite and slow growth with strong solute drag to massive ferrite.

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