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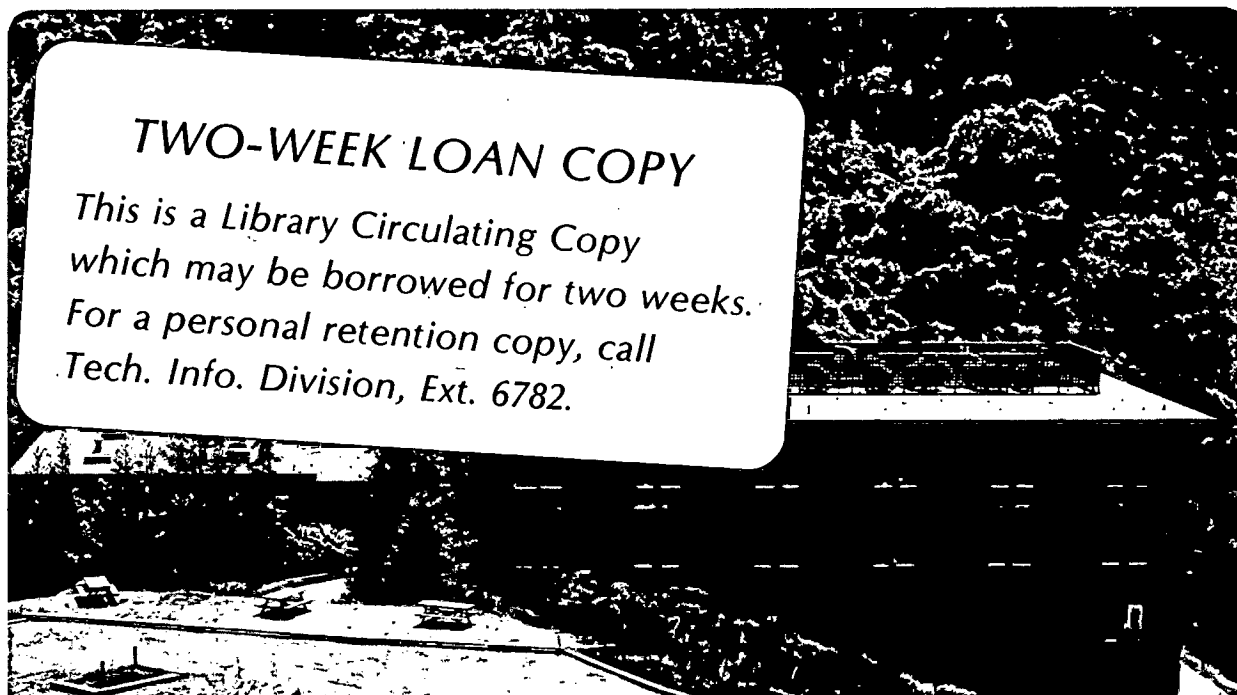
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ON THE SCAVENGING EFFECT OF PRECIPITATED AUSTENITE  
IN A LOW CARBON FE-5.5NI ALLOY

By

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ABSTRACT

The scavenging effect of precipitated austenite in a low carbon, commercial Fe-5.5Ni cryogenic alloy was investigated through observation of the dissolution of cementite precipitates during intercritical tempering and study of the associated change in Charpy impact toughness. Cementite precipitates initially located along prior austenite grain boundaries were gradually dissolved into reverted austenite as the intercritical tempering proceeded. The austenite tends to form at or around the carbide particles and may be catalyzed by their presence. The Charpy impact energy is changed through both a decrease in the ductile-brittle transition temperature and an increase in the upper shelf energy. The latter effect is specifically associated with the dissolution of the carbides which act as preferential void nucleation sites in the untempered alloy.

## I. INTRODUCTION

Good low temperature fracture toughnesses have been achieved in ferritic cryogenic steels through grain refining and the introduction of austenite phase into the ferritic matrix<sup>1,2</sup>. While the beneficial influence of grain refinement on the fracture toughness is well established<sup>3</sup>, the role of the retained austenite remains unclear. Recent hypotheses include the behavior of the retained austenite acting as a crack blunter<sup>4,5</sup>, shock absorber<sup>6</sup>, and sink for elements deleterious to fracture toughness<sup>7</sup>.

The retention of austenite in the ferritic matrix after tempering is presumably due to the enrichment of alloying elements in the austenite. Among the austenite stabilizing elements, carbon is most likely to diffuse into the austenite during the short-time tempering conventionally used due to its high diffusivity and relatively high solubility in the austenite. Furthermore, since both free carbon and precipitated carbides tend to lower the toughness of ferrite, the action of retained austenite in getting carbon should be reflected in an improvement in fracture toughness.

It is commonly observed<sup>8</sup> that cryogenic steels containing carbon show a toughness improvement after intercritical tempering which differs qualitatively from that observed in steels which are carbon-gettered<sup>1</sup> or nominally carbon-free<sup>9</sup>. In both cases, the ductile-brittle transition temperature is depressed. However, the carbon-containing steels also show a marked improvement in impact toughness above the ductile-brittle transition which is not found in steels which are carbon-free. There are indications from prior research<sup>10</sup> that the toughness improvement is associated with the dissolution of matrix carbide precipitates as austenite is precipitated during tempering. The present research was undertaken to further

clarify this phenomenon. A commercial cryogenic steel (Nippon Fe-5.5Ni) was treated so as to introduce coarse carbides. Intercritical tempering treatments were then applied and microstructural analyses conducted to study the dissolution of carbide, the concomitant formation and retention of austenite phase, and the resulting changes in impact toughness.

## II. EXPERIMENTAL PROCEDURE

A commercial low carbon Fe-5.5Ni alloy was provided by the Nippon Steel Corporation. Its composition was found to be in wt.%, Fe-5.75 Ni-0.93Mn-0.5Mo-0.19Si-0.16Cr-0.06C-0.006P-0.005S.

The as-received alloy was annealed at 1200°C for 2 hours to remove the effects of prior deformation and heat treatments. The plate was then soaked at 1200°C for 20 minutes, hot rolled 75% and slowly cooled in air. A low finish-rolling temperature of 750°C was used to promote the precipitation of carbide during subsequent cooling. The air-cooled specimens were then tempered at 670°C for 5, 15, 30, and 60 minutes respectively and water-quenched.

The microstructural changes following tempering were investigated through transmission electron microscopy. Impact toughnesses were obtained through standard Charpy V-notch impact tests and the resulting fracture surfaces were analyzed through scanning electron fractography. X-ray diffraction was used to determine the fraction of retained austenite phase.

## III. RESULTS AND DISCUSSION

### A. Tempering Temperature and Kinetics

The transformation temperatures of the Fe-5.5Ni alloy were studied through dilatometric analysis, using heating and cooling rates roughly corresponding to those pertinent to alloy heat treatment. The austenite

and martensite transformation temperatures were determined from the inflection points on the dilatometric curves to be:  $A_S = 682^\circ\text{C}$ ,  $A_f = 721^\circ\text{C}$ ,  $M_S = 420^\circ\text{C}$ ,  $M_f = 310^\circ\text{C}$ . The tempering temperature used in this work,  $670^\circ\text{C}$ , was chosen to lie slightly below  $A_S$  to insure diffusional transformation.

As is well known<sup>1,2,10</sup> the austenite precipitated during short-time intercritical tempering of steels of the class treated here is only partly retained after subsequent cooling to room temperature. The remainder, which is usually the greater part of the precipitated austenite, retransforms martensitically. To gain information about the fraction of austenite actually formed during tempering, the kinetics of austenite precipitation were estimated from the dilatometric curves, under the approximation that the sample contraction on isothermal holding is linearly proportional to the volume fraction of austenite formed. The method is illustrated and the results tabulated in Fig. 1. This method is not precise but the austenite fractions found in this way were in reasonable agreement with subsequent metallographic observations.

#### B. Microstructural Changes on Tempering

When the low-carbon 5.5Ni steel studied here is quenched after rolling, it develops a fine dislocated lath martensite substructure illustrated in Fig. 2a. When the alloy is cooled slowly in air, on the other hand, it forms a coarser but still heavily dislocated substructure which is illustrated in Fig. 2b. In this case, large cementite precipitates are formed, principally along prior austenite grain boundaries (Fig. 2b). No retained austenite is detected in either structure.

The air-cooled specimens were then heat treated at  $670^\circ\text{C}$  for 5, 15, and 30 minutes.

Figure 3 illustrates the microstructure of a specimen tempered at



670°C for 5 minutes. The structure is similar to that in the as-cooled condition. However, dilatometric tests indicate ~3% transformation to austenite during tempering and TEM studies suggest associated carbide dissolution; the cementite particles are smaller and show shape changes indicating progressive dissolution. As shown in a dark field micrograph of the cementite particle in the lower center of the figure (Fig. 3c) one side of this particle has apparently dissolved into a new phase. This phase was identified as martensite through selected area electron diffraction and presumably originates from austenite precipitated during tempering. Neither x-ray diffraction nor transmission electron microscopy revealed any retained austenite phase at room temperature.

Tempering for 15 minutes produced significant changes in the microstructure, as demonstrated in Fig. 4. Fine lath martensites are seen clearly in the ferrite matrix. Assuming that this martensite phase results from the retransformation of austenite, its volume fraction is in reasonable agreement with the dilatometric estimate (Fig. 1) of 16% austenite formed during tempering. While retained austenite was not found in the TEM specimens, x-ray analysis revealed a residual austenite content of ~1%.

TEM analysis of the remaining carbide precipitates showed that these were invariably located either within or beside the martensite laths as shown in Fig. 4. The size of the cementite precipitates was noticeably smaller than that in the original specimen suggesting their gradual dissolution into the precipitated austenite phase.

The common, and seemingly universal, association between the carbides and the austenite precipitated during tempering (lath martensite in the final structure) suggests that austenite nucleation is catalyzed by the carbide particles, which may promote the kinetics of the two-phase

decomposition during tempering, as pointed out by Speich et al<sup>11</sup>.

The microstructure of the specimen tempered for 30 minutes (Fig. 5) contained a large fraction of the lath martensite structure, reflecting the reversion of the ~85% austenite formed during tempering (Fig. 1). A marked polygonization of the dislocations present in the untransformed ferrite was also noted. Cementite precipitates were no longer observed in or near the martensite laths, although very small carbide precipitates were still found in the ferrite matrix. Retained austenite is clearly distinguishable in TEM samples. X-ray diffraction indicated a retained austenite content of about 6%.

### C. Changes in Charpy Impact Toughness

The low carbon Fe-5.5Ni alloy used in this research showed a response to intercritical tempering similar to that observed in Fe-9Ni steel. As shown in Fig. 6, tempering at 670°C for 1 hour resulted in both a decrease in the ductile-brittle transition temperature and an increase (~50 ft-lb) in the upper shelf energy. The increase in impact toughness seems clearly attributable to the scavenging effect of precipitated austenite since the formation of the retained austenite by tempering in the ( $\alpha + \gamma$ ) region was accompanied by the removal of the cementite precipitates.

Figure 7 contains scanning electron micrographs of the fracture surfaces of the tempered and untempered specimens broken at room temperature. The untempered specimens exhibit a typical low energy tear mode with large dimples. As is usual, the large dimples were found to initiate from precipitate particles. Energy dispersive analysis of x-ray emission (EDAX) established that the precipitates were not the sulfide inclusions (such as MnS and CrS) usually observed in a ductile dimple fracture surface. While

the EDAX technique cannot analyze matrix carbides, the nucleating precipitates may be safely identified as matrix carbides on the basis of their morphology, their distribution, and the failure of the EDAX to detect other species in them. The fracture surface of the tempered specimen, on the other hand, contained no cementite particles. All particles in the dimple fracture surface were identified as MnS or CrS inclusions.

#### IV. CONCLUSIONS

Investigation of the dissolution of cementite precipitates during intercritical tempering of a commercial Fe-5.5Ni alloy leads to the following conclusions:

1. The cementite precipitates on prior austenite grain boundaries are progressively dissolved as austenite forms during intercritical tempering. The evidence suggests that the carbides catalyze the formation of the austenite.

2. Intercritical tempering induces both a decrease in the ductile-brittle transition temperature and an increase in the upper shelf impact energy. The latter effect is due to the removal of carbides which act as void nucleation sites in the untempered alloy. This observation apparently explains why no corresponding enhancement of the upper shelf is observed in similar but interstitial-free cryogenic steels.

#### ACKNOWLEDGMENT

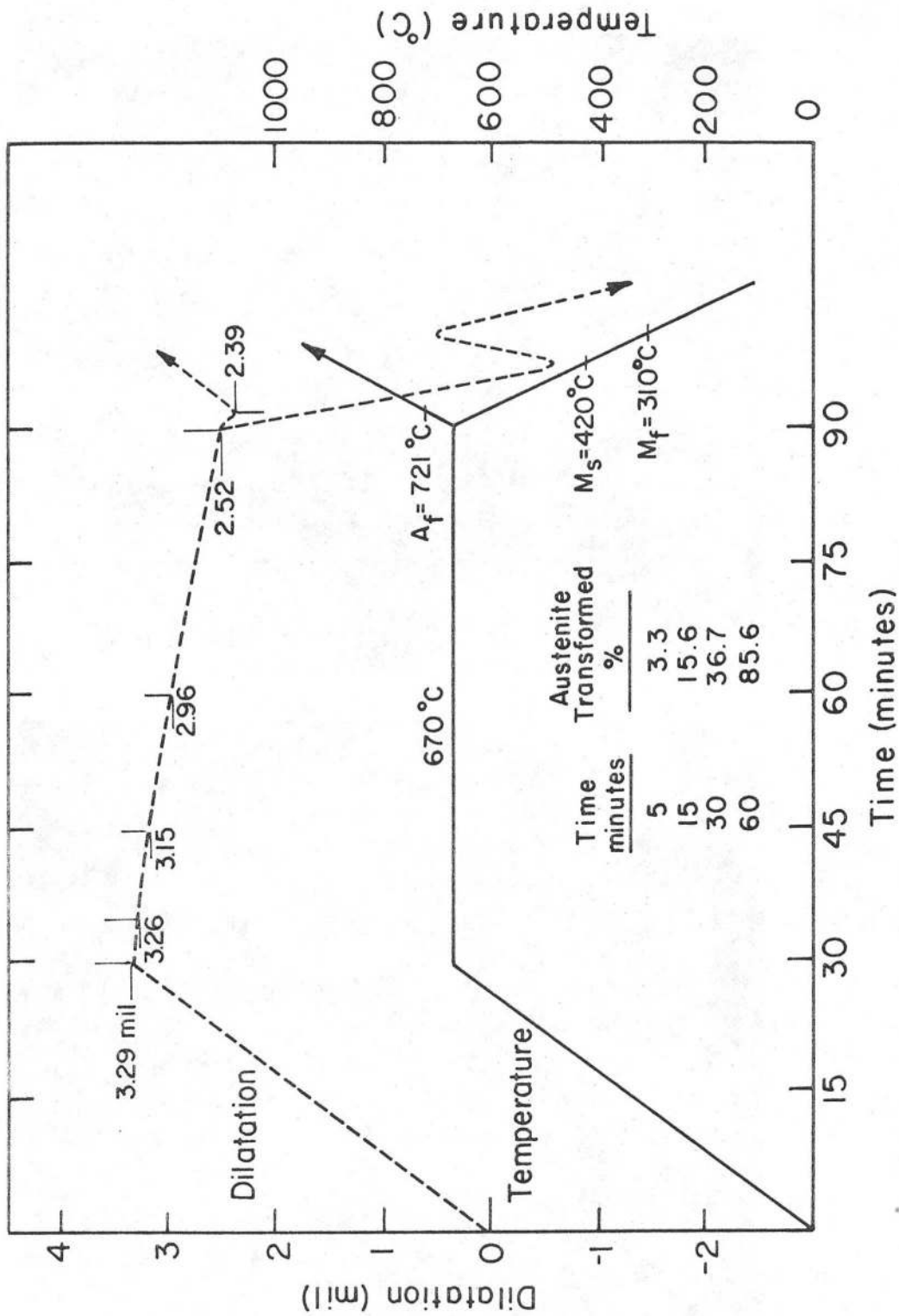
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## FIGURE CAPTIONS

- Figure 1. Dilation curve of Fe-5.5Ni alloy during tempering at 670°C.
- Figure 2. Transmission electron micrographs Fe-5.5Ni. (a) Quenched after cementite rolling, (b) air cooled after rolling, c denotes/precipitation.
- Figure 3. Microstructure of Fe-5.5Ni tempered at 670°C for 5 minutes. (b) (c) Dark field images of cementite precipitates of different orientations.
- Figure 4. (a) Bright field microstructure after tempering at 670°C for 15 minutes. (b) Corresponding dark field image of cementite precipitates.
- Figure 5. (a) Bright field. (b) Corresponding dark field image of the retained austenite present after tempering at 670°C for 30 minutes. Very small carbide precipitations are still visible inside the ferrite matrix.
- Figure 6. Charpy impact curve of Fe-5.5Ni in the initial state and after tempering at 670°C for one hour.
- Figure 7. Scanning electron micrograph of the fracture surface of Fe-5.5Ni. (a) Untempered state, as-cooled; (b) after tempering at 670°C for 1 hour, WQ. Both specimens were broken at room temperature (X1000).



XBL 791-5632

Figure 1

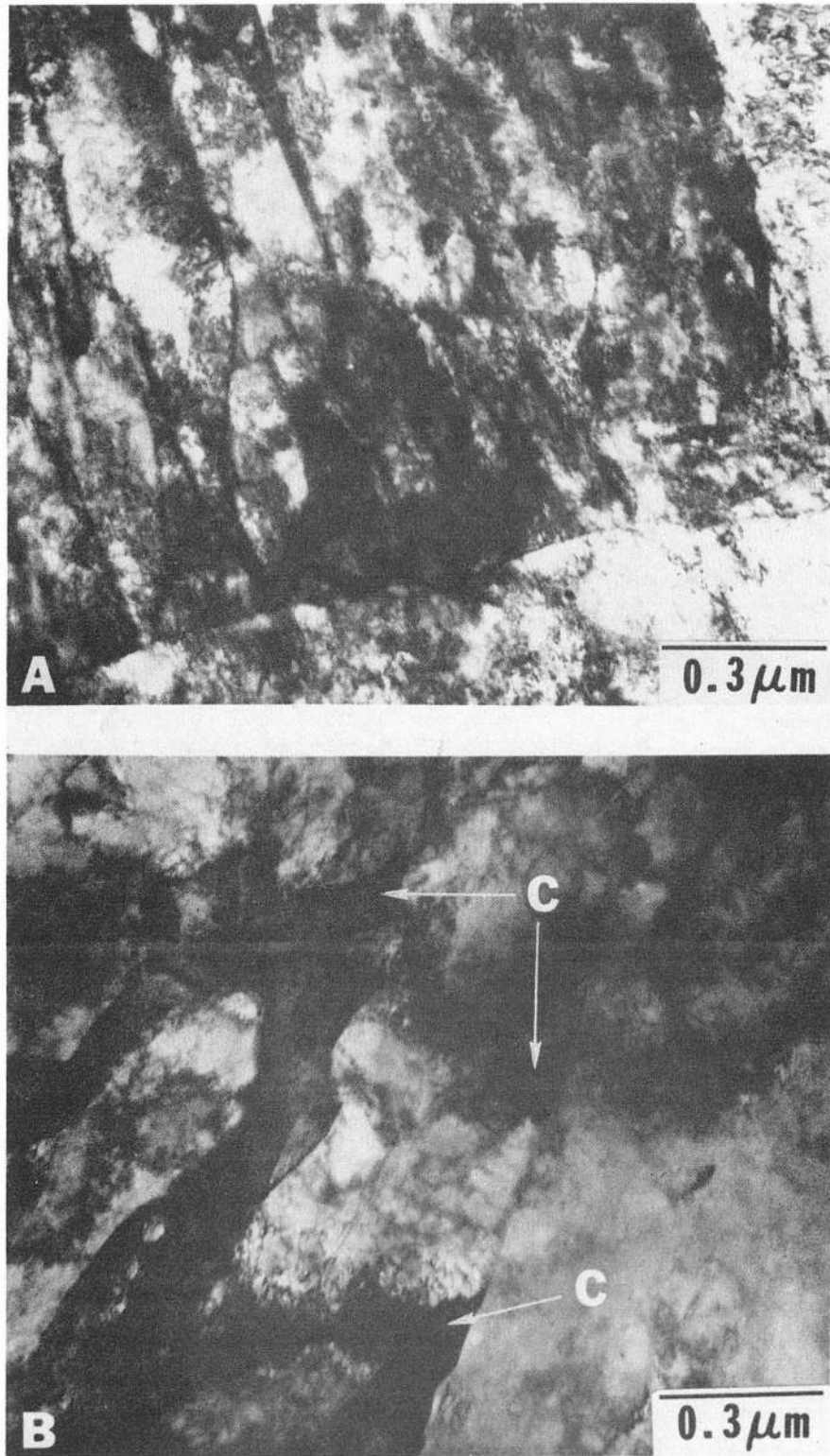


Figure 2

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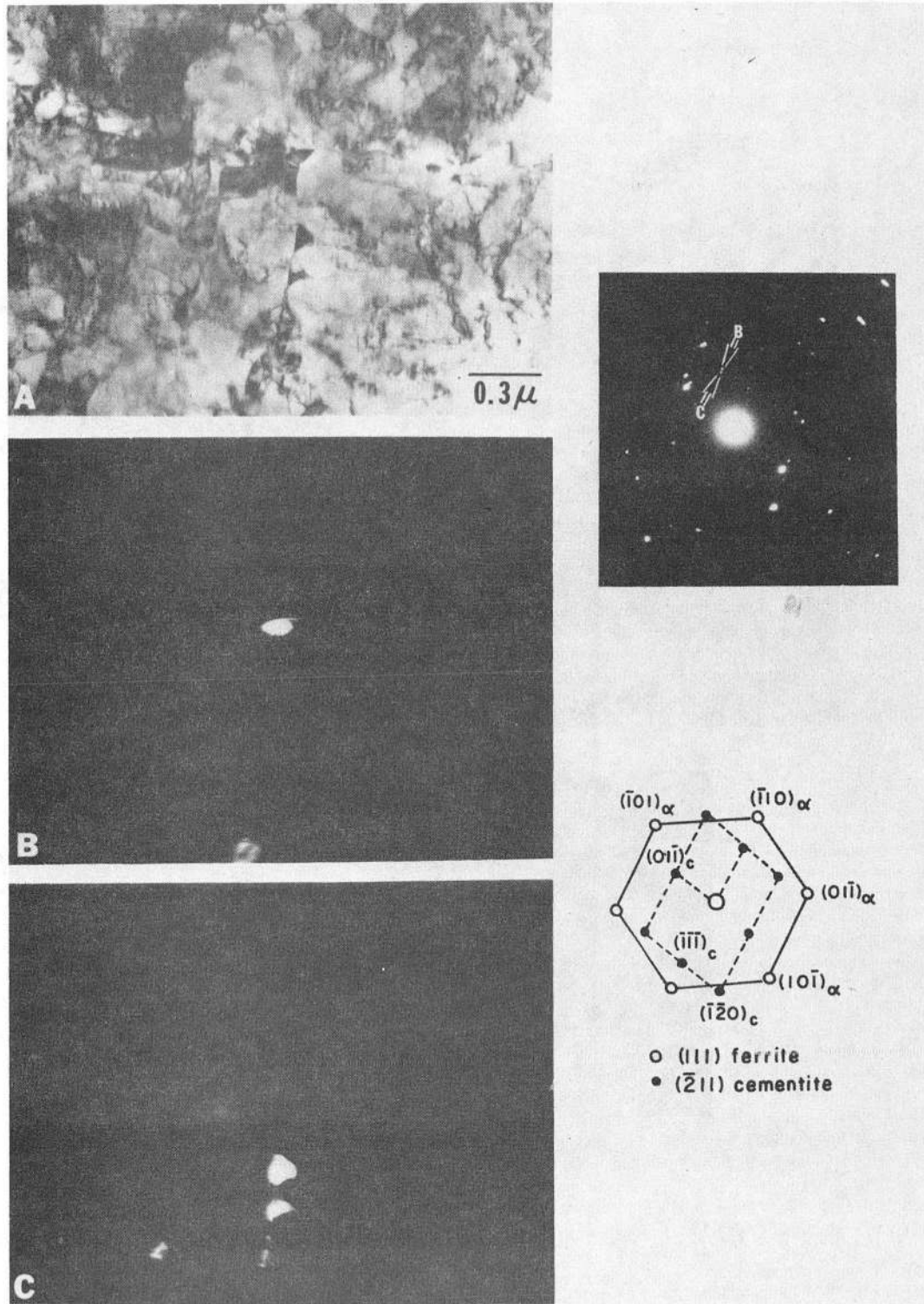


Figure 3

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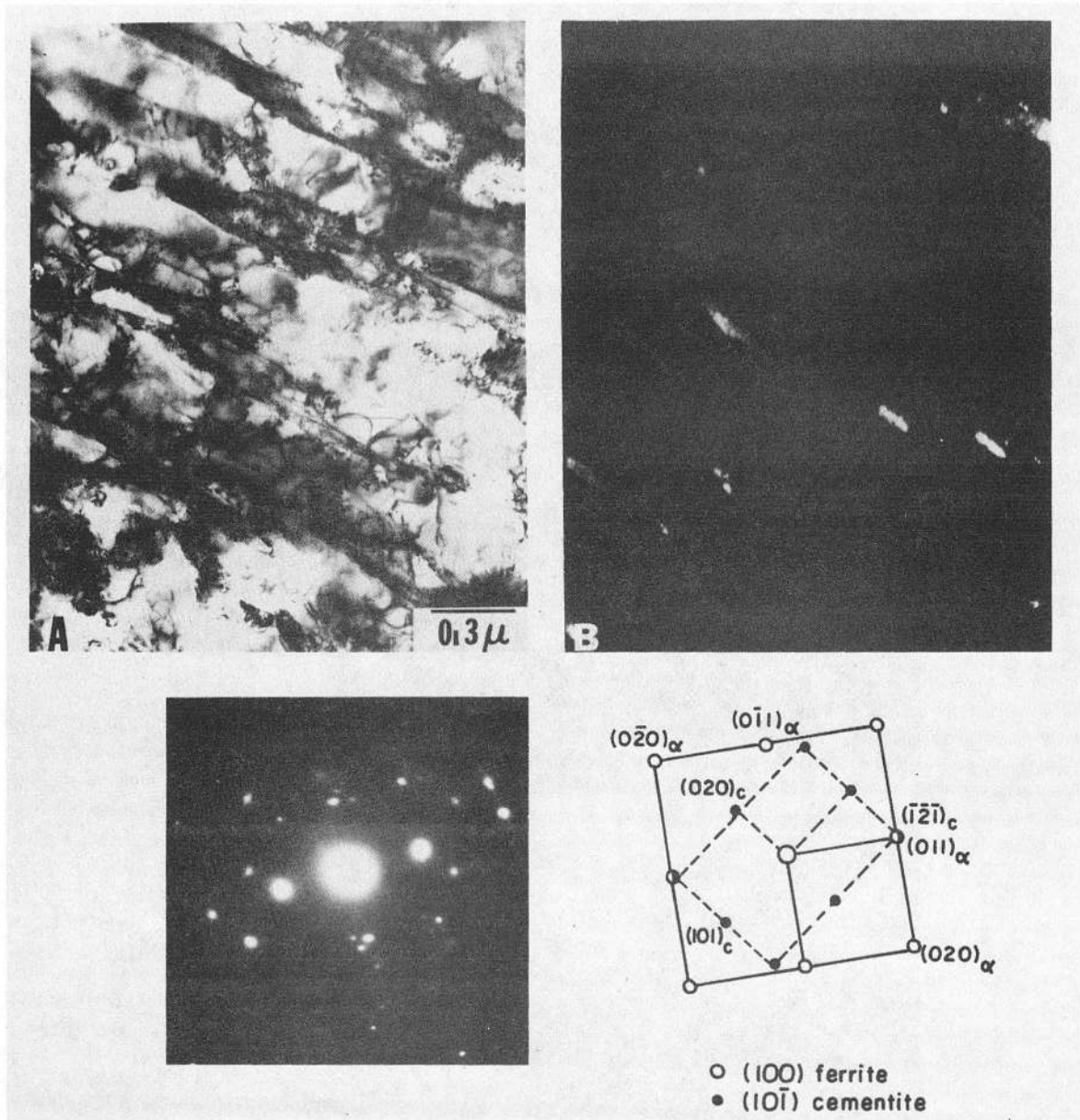
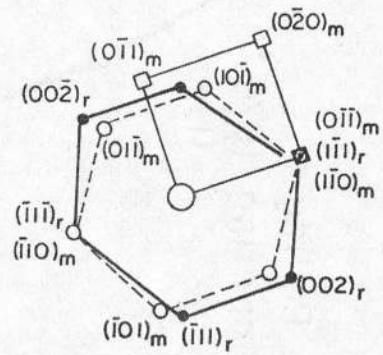
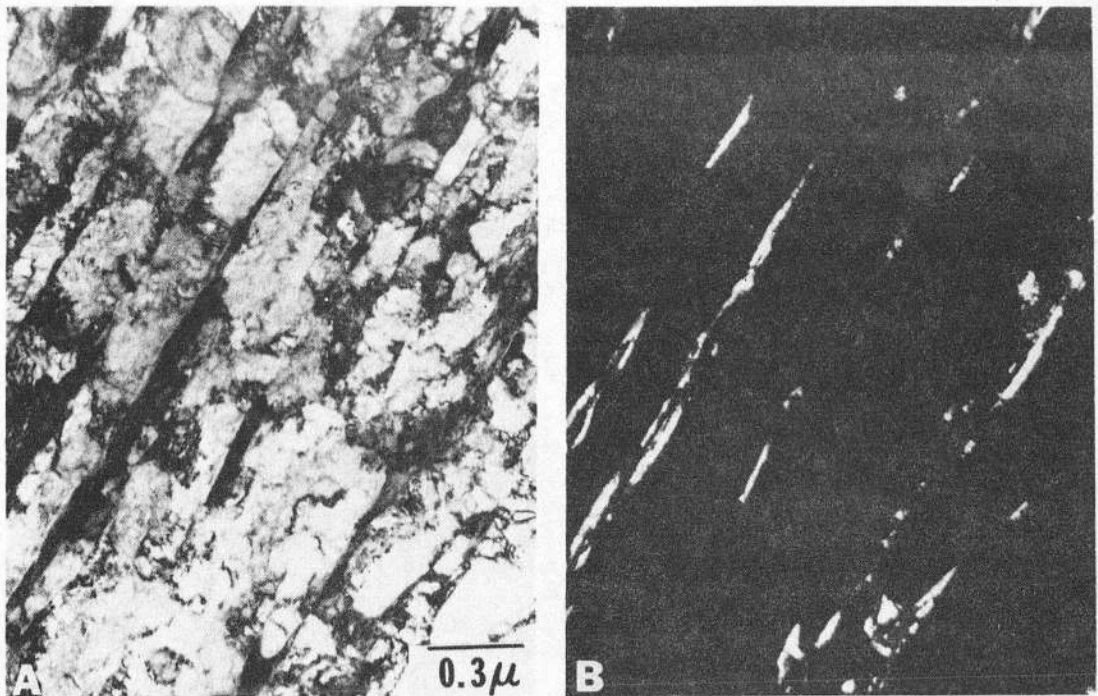


Figure 4

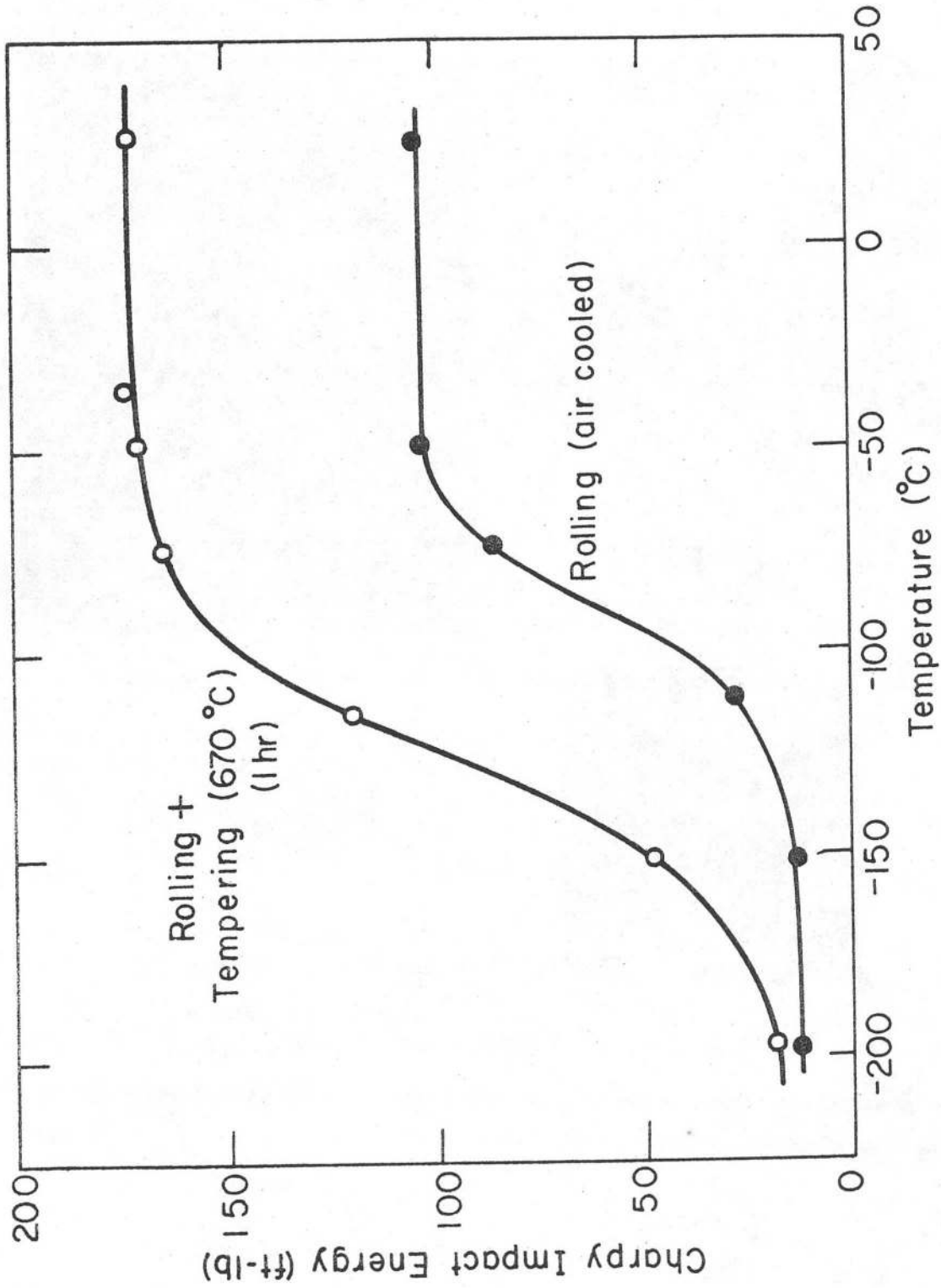
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- $[\bar{1}\bar{1}\bar{1}]$  martensite
- $[\bar{1}\bar{0}0]$  martensite
- $[\bar{1}\bar{1}0]$  austenite

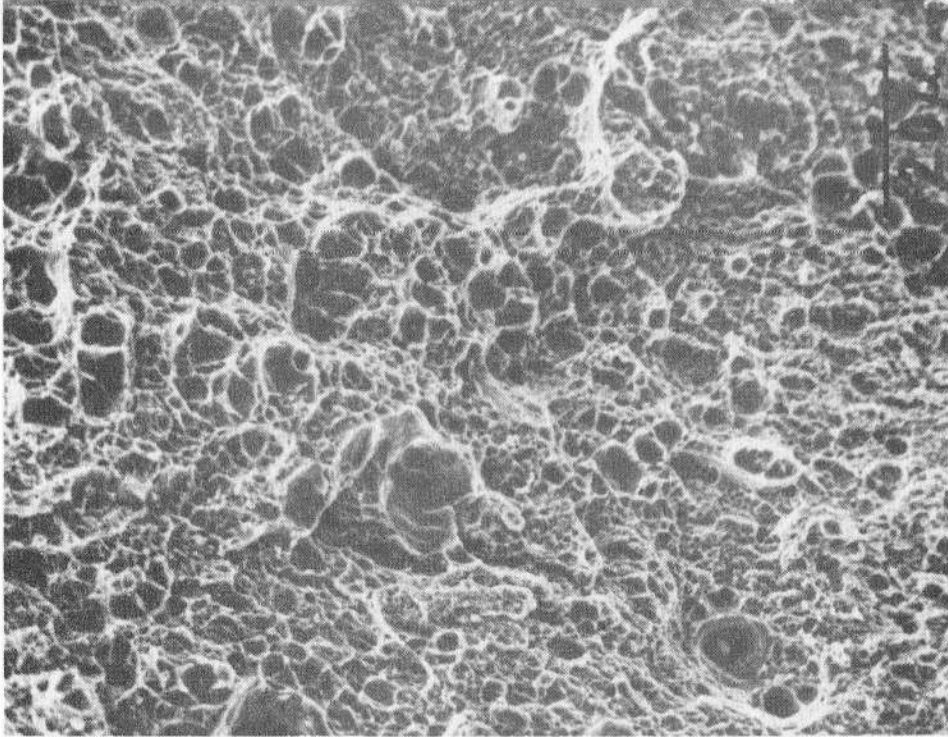
Figure 5

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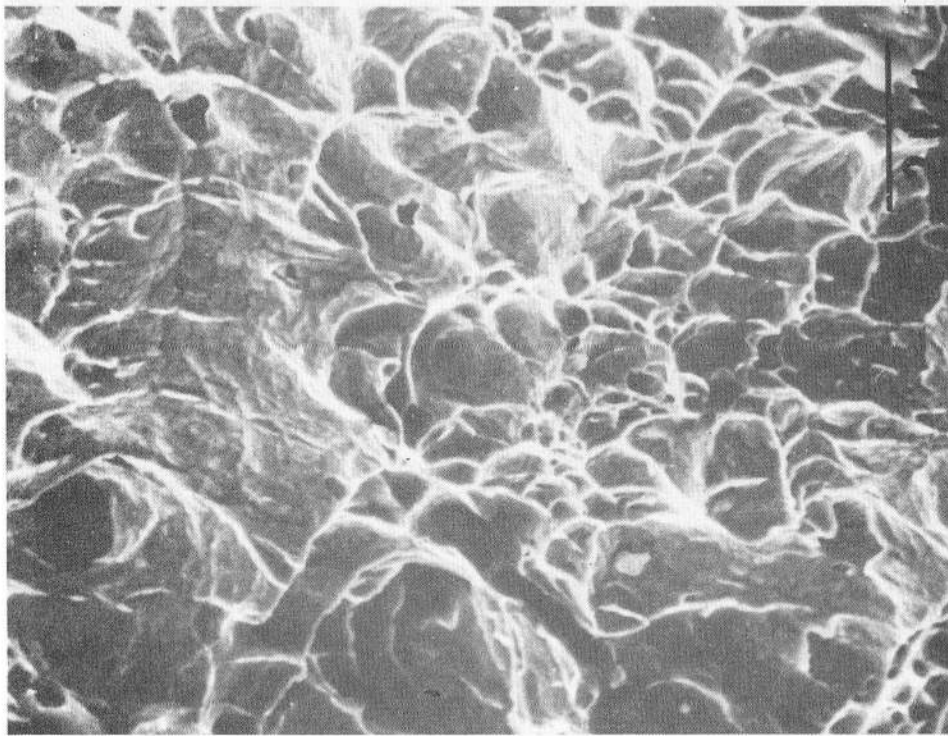


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Figure 6



**670°C, 1hr/ WQ**



**As - Cooled**

Figure 7

XBB 792-2162

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