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**THE ROLE OF THE CONSTITUENT PHASES IN  
DETERMINING THE LOW TEMPERATURE TOUGHNESS OF  
5.5NI CRYOGENIC STEEL**

J. I. Kim\*, H. Jae Kim\*\* and J. W. Morris, Jr.

Department of Materials Science and Mineral Engineering  
University of California  
and  
Materials and Molecular Research Division  
Lawrence Berkeley Laboratory  
Berkeley, California 94720

**ABSTRACT**

Ferritic Fe-Ni steels that are intended for service at low temperature are usually given an intercritical temper as the final step in their heat treatment. The temper dramatically decreases the ductile-brittle transition temperature,  $T_B$ . Its metallurgical effect is to temper the lath martensite matrix and precipitate a distribution of fine austenite particles along the lath boundaries. Prior research suggests that the low value of  $T_B$  is a consequence of the small effective grain size of the ferrite-austenite composite. The present research was done to test this conclusion against the counter-hypothesis that the low  $T_B$  is due to the inherent toughness of the constituent phases. The approximate compositions of the tempered martensite and precipitated austenite phases in the composite microstructure of tempered 5.5Ni steel are known from STEM analysis. Bulk alloys were cast with these two compositions. Their mechanical properties were measured after heat treatment and compared to those of the parent alloy in the toughened 'QLT' condition. Both of the constituent phases are brittle at low temperature. It follows that the outstanding low-temperature toughness of the tempered alloy cannot be attributed to the inherent properties of the constituent phases, but must reflect their cooperative behavior in the composite microstructure. The austenitic bulk alloy was also used to investigate the stability of the precipitated austenite phase. The thermomechanical stability of the bulk alloy approximates that of the precipitated austenite within tempered 5.5Ni steel. This result is consistent with previous data, and supports the conclusion that the stability of the precipitated austenite is determined mainly by its chemical composition.

\*Present address: IBM Thomas J. Watson Research Center, Yorktown Heights, New York 10598

\*\*Present address: Metallurgy Department, School of Engineering, Georgia Institute of Technology, Atlanta, Georgia 43215

## INTRODUCTION

The heat treatment given to ferritic Fe-Ni steels that are intended for structural use at low temperature, such as the commercial 9Ni [1-3] and 5-6Ni [4-7] grades, is finished with an intercritical temper in the two-phase ( $\alpha+\gamma$ ) region [8]. The intercritical temper results in a composite microstructure of tempered lath martensite with small islands of precipitated austenite phase densely distributed along the lath boundaries and the prior austenite grain boundaries [5,6,9-11]. The intercritical temper improves the fracture toughness of the alloy in two senses [1,6,12-15]: it lowers the ductile-brittle transition temperature ( $T_B$ ) and it raises the upper shelf toughness above  $T_B$ .

It is generally agreed that the increase in the upper shelf toughness is due to the getting of matrix carbon and carbides by the austenite [1,15]. This conclusion is supported by at least two independent sets of experiments. First, the upper shelf toughness is inherently high in similar Fe-Ni steels that are chemically gettered by Ti [16], and is essentially unchanged by an intercritical temper. Second, matrix carbides dissolve as the austenite phase precipitates during the intercritical temper [15]. Their dissolution leads to a substantial increase in the upper shelf toughness.

However, the precise manner in which the austenite precipitation decreases  $T_B$  remains somewhat controversial. Since the ductile-brittle transition in this class of steels is caused by the intrusion of transgranular cleavage fracture [11,17,18], the intercritically tempered microstructure must effectively resist cleavage. Two distinct types of theory have been offered to explain how this may happen. The mechanisms may be generally described as 'crack-blunting' and 'grain refinement'. The 'crack-blunting' models focus on the inherent properties of the constituent phases in the composite tempered microstructure, and suggest that the cleavage resistance is due to the inherent ductility of either the relatively soft tempered ferrite matrix or the precipitated austenite phase [19-23]. The 'grain refinement' model [11,17] concentrates on the microstructural role of the precipitated austenite (or, more accurately, the fresh martensite to which it transforms under load) in disrupting the cleavage planes that traverse packets of lath martensite in the untempered alloy.

The evidence supporting the 'grain refinement' model is presented elsewhere [11,17]. The model has been developed and tested in detail in the particular case of Nippon Fe-5.5Ni cryogenic steel in the QLT condition [11].

The present work was intended to examine the plausibility of the 'crack-blunting' model by determining the mechanical properties of the constituent phases in the final microstructure of the same alloy. This approach was made possible by prior work [24,25] that used scanning transmission electron microscopy (STEM) to measure the compositions of the constituent phases in this alloy. In the present work bulk alloys were cast to duplicate the compositions of the constituent phases. Their strength and toughness were found as a function of temperature, and compared to the strength and toughness of the tempered base alloy.

A second purpose of the present work was to find the reason for the thermomechanical stability of the precipitated austenite in tempered Fe-Ni martensitic steels. The austenite introduced by intercritical tempering in 9Ni and 5-6Ni steels is thermally stable on cooling to room temperature, and is largely retained when the alloy is cooled to 77K, but transforms readily when the alloy is deformed. It is not clear whether the stability of this phase is determined mainly by its chemical composition, by its size, or by the mechanical constraint imposed by the surrounding matrix. The stability of the representative bulk alloy was, therefore, studied and compared to that of the precipitated austenite in QLT-treated 5.5Ni steel.

## **EXPERIMENTAL PROCEDURE**

### **1. Composition of the Research Alloys**

The compositions of the alloys used in this research are given in Table 1. The Fe-5.5Ni steel is a commercial grade that was supplied by Nippon Steel. It is normally used in the QLT heat treatment: Q(800°C for 1 hr., quench), L(670°C for 1 hr., quench), T(600°C for 1 hr., quench). This treatment yields a composite final microstructure that contains three elements [11]: tempered martensite, the primary constituent, precipitated austenite, which comprises about 8.5% of the volume, and a small quantity of fresh martensite that arises from the reversion of part of the austenite that is precipitated at 600°C.

The approximate compositions of the tempered martensite and austenite were previously measured [24,25] by energy dispersive x-ray analysis (EDAX) in a scanning transmission electron microscope (STEM). The results of the STEM analysis were used to set the substitutional alloy content of the simulated martensite (M) and austenite (A) alloys listed in Table 1. However, the STEM analysis does not reveal the carbon content. It may be easily shown [24] that 1 hour at 600°C is sufficient time for essentially complete equilibration of carbon by diffusion. The carbon content of the two alloys was therefore estimated by setting the carbon content of the matrix at the solubility limit in ferrite at 600°C, about 0.01 weight percent, and assigning the remaining carbon to the austenite, giving about 0.5 weight percent for the ~10 volume percent of austenite precipitated during the temper. The actual carbon contents of the cast alloys were 0.02 wt. % in alloy M and 0.42 wt. % in alloy A.

### **2. Alloy Processing and Heat Treatment.**

The base 5.5Ni alloy was annealed at 1200°C for 2 hours to remove the effects of prior deformation and heat treatment, solution annealed at 900°C for 2 hours, and then given the standard QLT heat treatment.

The two representative alloys were cast as 10 kg ingots after induction melting in an argon gas atmosphere. The ingots were homogenized under argon gas at 1200°C for 28 hours. They were then forged at 1100°C into plates 23 mm thick and 76 mm wide, and solution annealed at 900°C for 2 hrs. prior to heat treatment.

The austenite constituent of QLT-treated 5.5Ni steel is formed at 600°C during the final alloy temper (T) and quenched directly to room temperature. The representative alloy A was hence tested in the as-quenched condition.

The tempered martensite constituent of 5.5Ni steel is a product of the full cyclic heat treatment. The representative alloy M was hence given the normal QLT heat treatment. It was also tested in the as-quenched condition (Q) and the quench-and-temper condition (QT) to gain information on the development of its properties during heat treatment.

The alloy M was also tested in two other conditions to compensate for possible differences between its transformation behavior and that of 5.5Ni steel. First, the austenite reversion temperatures,  $A_s$  and  $A_f$ , of the alloy M are expected to be higher than those of 5.5Ni steel because of the lower alloy content. Dilatometric studies showed that the difference is approximately 40°C. The difference has the consequence that the L treatment temperature, which is only slightly below  $A_s$  in 5.5Ni steel, falls well below the austenite reversion temperature in alloy M. The L treatment may, therefore, have very different microstructural consequences in the two alloys. To study the effects of this difference the alloy M was also given a QL'T treatment in which the L' temperature, 710°C, was chosen to be near the  $A_s$  temperature for the alloy.

Secondly, metallographic studies of the QLT-treated alloy M suggest that it has a larger grain size than the base 5.5Ni alloy. Samples of alloy M were hence cold-worked to a 25% and 50% reduction in cross section prior to heat treatment to refine the grain size.

### 3. Mechanical Testing and Analysis.

The alloys were cut and ground under flood cooling into cylindrical tension specimens (13mm gage length by 3mm diameter) and Charpy impact specimens (10 mm x 10 mm x 55 mm). The tension tests were done at room temperature and at liquid nitrogen temperature (77K) using a strain rate of 0.04/min. The yield strength was calculated by the 0.2% offset method. Low temperature Charpy impact tests were done in accordance with ASTM Standard 23-72, using mixtures of liquid nitrogen, isopentane and methyl alcohol to achieve controllable subzero temperatures.

The microstructural and fractographic analyses employed optical microscopy and transmission and scanning electron microscopy. The phase contents of the alloys were measured by x-ray diffraction.

## RESULTS

### 1. Microstructure and Phase Stability.

The microstructure and phase content of QLT-treated 5.5Ni steel are well characterized, and are discussed in detail elsewhere [5,6,11]. The metallurgical behavior of the representative alloys M and A was investigated in the present work.

a. Alloy A

Both x-ray analysis and optical microscopy showed that alloy A is fully austenitic at room temperature. On cooling to 77K the alloy undergoes a partial transformation to a mixture of lath and twinned martensite (Fig. 1). The total martensite content is approximately 25% by volume. As a further test of the austenite stability the alloy was immersed in liquid helium (4.2K). The fraction martensite increased to ~55%.

In contrast to its thermal stability, alloy A transforms readily under mechanical load in both the tensile and Charpy impact tests. Its transformation under tension is reflected in its stress-strain behavior, described below. Its transformation under impact is illustrated in Figure 2, which is an optical micrograph of an etched cross section of a Charpy impact specimen that was broken at room temperature. The transformed region extends at least a full millimeter below the fracture surface.

b. Alloy M.

The alloy M was first given the standard thermal cycling heat treatment for Nippon 5.5Ni steel: Q(800°C, 1 hr., quench), L(670°C, 1 hr., quench), T(600°C, 1 hr., quench). This heat treatment resulted in an alloy having an apparent grain size near 10  $\mu\text{m}$  with no detectable retained austenite. Its microstructure is shown in the optical micrograph presented in Figure 3a.

This alloy was quite evidently more coarse in its grain structure than the QLT-treated base alloy. Two alternate treatments were used in an effort to achieve a more comparable grain size. The first was based on the observation that the composition difference between the 5.5Ni-0.06C base alloy and the 5Ni-0.02C representative alloy raises the austenite reversion temperature. Dilatometric measurements show an increase in  $A_s$  from approximately 700°C for rapidly-heated 5.5Ni to approximately 735°C for alloy M. To investigate whether this change has important effects on the final microstructure the alloy M was given the alternate treatment QL'(710°C, 1 hr., quench)T. However, the QL'T treatment yielded a microstructure that was essentially identical to that established by the QLT treatment.

In the second alternate treatment samples of the alloy were cold worked 25% and 50% prior to heat treatment through the QLT cycle. Optical micrographs of the resulting alloys are presented in Figure 3. The apparent grain size decreased with the amount of prior cold work from ~10  $\mu\text{m}$  to ~5  $\mu\text{m}$  after 25% cold work and ~2  $\mu\text{m}$  after 50% cold work. No retained austenite phase was detected in the final state of any of these samples.

2. Mechanical Properties.

a. QLT-Treated 5.5Ni Steel.

The low-temperature mechanical properties of 5.5Ni steel are described in detail elsewhere [5,6,11]. Typical properties are given in

Table 2. The alloy has both high strength and impact toughness at 77K. Its impact toughness decreases only slightly with temperature, as shown in Figure 4. The fracture mode at 77K is almost fully ductile.

b. Alloy A

The measured mechanical properties of alloy A are given in Table 3. The yield strength of alloy A is low at both room temperature and at 77K. The low yield strength is largely due to a stress-induced transformation to the martensite phase. Continuous transformation during the test resulted in very high work hardening. The specimen fractured before necking. The fracture stress (applied load divided by the actual area at fracture) is listed in Table 3.

The Charpy impact energy of alloy A was very small compared to that of the base alloy at both room temperature and at 77K. As is typical of austenitic steels that become brittle at low temperature, alloy A did not show a clear ductile-brittle transition, but rather exhibited a monotonic decrease in toughness as the test temperature was lowered.

c. Alloy M

The mechanical properties of alloy M are given in Table 4, for various heat treatments starting from the annealed condition. The yield strength of the alloy is weakly dependent on the heat treatment, and is comparable to that of QLT 5.5Ni steel. The ultimate tensile strength is significantly below that of QLT 5.5Ni. The impact toughness is comparable to that of QLT 5.5Ni at room temperature, but is very low at 77K in all heat treatment conditions. The change in the intermediate heat treatment temperature (QLT to QL'T) has very little effect on the mechanical properties. Scanning electron fractographs of 77K Charpy specimens of the alloy in various heat treatment conditions are given in Figure 5. All fracture surfaces are brittle and reveal a transgranular quasicleavage fracture mode that appears to be insensitive to heat treatment.

The room temperature tensile and impact properties of the samples that were cold worked prior to the QLT heat treatment are given in Table 5. There is a small increase in yield strength with prior cold work. This increase is presumably due to the grain refinement, and reflects the small Hall-Petch slope of this class of steels. The other mechanical properties, tensile strength, elongation, reduction in area, and Charpy impact energy, are unaffected by the prior cold work.

The impact toughness of the cold-worked samples is plotted as a function of temperature in Figure 6. The ductile-brittle transition temperature is well-defined and lies above 77K in all cases. The transition temperature decreases from approximately  $-130^{\circ}\text{C}$  ( $\sim 140\text{K}$ ) for the annealed specimen with a  $10\ \mu\text{m}$  grain size to  $-175^{\circ}\text{C}$  ( $\sim 100\text{K}$ ) for the 50% cold-worked specimen with an apparent grain size near  $2\ \mu\text{m}$ . As expected [9,13] the grain refinement does not significantly affect the upper shelf toughness.

## DISCUSSION AND CONCLUSIONS

In interpreting the results of this work it must be recognized that the precise compositions of the constituent phases in QLT 5.5Ni steel could not be determined, and the measured compositions are not exactly duplicated in the representative alloys. The uncertainty in the carbon content is particularly unsettling, since carbon has a profound effect on the stability and mechanical properties of both the austenite and martensite phases. Using the formulae of Steven and Haynes [26], for example, the total content of substitutional species in alloy A (~16% by weight) is predicted to lower its  $M_s$  temperature by only 335°C, while 0.42 wt.% carbon lowers the  $M_s$  by 200°C.

While the simulation attempted here is imprecise, its results seem to be both instructive and qualitatively reasonable. The compositions of the representative alloys are close to the compositions determined by chemical analysis of QLT-treated 5.5Ni steel [24,25] under the assumption that carbon is effectively gettered into the austenite. There is no reason to believe that the mechanical properties of the alloys will change dramatically with small differences in composition. The plausibility of the representation is made more credible by similarities between the behavior of the representative alloys and that of the phases in the base alloy, including the high shelf toughness of alloy M and the thermomechanical instability of the austenite in alloy A.

Assuming the validity of the representation, four conclusions can be drawn from the results of this work:

(1) The low  $T_B$  of QLT-treated 5.5Ni steel is primarily due to its composite microstructure.

QLT-treated 5.5Ni steel is tough at 77K, while both of the alloys representing its constituent phases are brittle. The representative austenitic alloy A is mechanically unstable at 77K and transforms to a brittle martensite. It is, therefore, unlikely that the austenite serves as a 'crack-blunting' constituent. Moreover, since its toughness is low in the bulk, it is unlikely that the austenite contributes any significant 'transformation toughening'. The representative alloy M is also brittle at 77K, even in a low-carbon, grain-refined and tempered condition. It is, therefore, unlikely that the tempered martensite matrix in QLT-treated 5.5Ni steel acts as a 'crack-blunting' phase.

A possible microstructural toughening mechanism has been described previously [11,17] and supported with experimental data on the composite microstructure and properties of 9Ni and 5.5Ni steel in various heat treatment conditions [11]. In this interpretation, the austenite admixture acts to refine the effective grain size of the tempered martensite matrix. In the quenched condition martensite laths are organized into packets that share {100} cleavage planes. During the QT treatment of 9Ni steel or the QLT treatment of 5.5Ni steel austenite phase precipitates along the lath boundaries. The austenite transforms to martensite when the alloy is deformed at low temperature, but the crystallographic variant of this martensite is generally different from that of the surrounding packet. The consequence is a decrease in the effective grain size of the alloy.

The present results are consistent with this microstructural toughening mechanism. Decreasing the prior austenite grain size of the alloy M to  $\sim 2 \mu\text{m}$  by cold working the alloy prior to heat treatment lowered  $T_B$  to  $\sim 100\text{K}$ . While the precise value of the effective grain size of QLT-treated 5.5Ni steel is not entirely clear, the precipitated austenite decorates lath boundaries that are  $< 0.5 \mu\text{m}$  apart [11]. It is reasonable to infer that the effective grain size is less than that in a tempered alloy with a  $2 \mu\text{m}$  prior austenite grain size. The monotonic decrease in the  $T_B$  of alloy M with the prior austenite grain size suggests that  $T_B$  would lie below  $77\text{K}$  if the grain size were sufficiently fine.

(2) The upper-shelf toughness of QLT-treated 5.5Ni steel is determined primarily by the tempered martensite matrix.

The high upper shelf toughness of the representative matrix alloy M suggests that the tempered martensite matrix of QLT-treated 5.5Ni steel is also very tough above  $T_B$ . Grain refinement does not typically cause a large change in the upper shelf toughness of Fe-Ni alloys [9,13]. Hence the austenite precipitation is expected to influence the upper shelf toughness only to the extent that it modifies the deformation behavior of the alloy or introduces ductile void nucleation sites. The slightly lower upper shelf toughness of the QLT material reflects its higher yield strength and the low toughness of the austenite constituent.

(3) Austenite precipitation raises the upper shelf toughness of QLT-treated 5.5Ni steel by gettering carbon from the matrix phase.

The representative alloy M has a high upper shelf energy (266 J) even when the alloy is tested in the as-quenched condition. This result contrasts strikingly with the behavior of as-quenched 5.5Ni steel, which has a relatively low upper shelf toughness ( $\sim 150 \text{J}$ ). Comparing the compositions of the two alloys, it seems clear that the higher shelf toughness of alloy M is attributable to its low carbon content. While STEM analyses of the compositions of the constituent phases in QLT-treated 5.5Ni steel do not provide the carbon content, carbon must segregate strongly to the austenite phase during tempering [24]. The accumulation of carbon in the austenite has, in fact, been followed in indirect experiments [15] that monitored the dissolution of matrix carbides as the austenite phase precipitated during tempering.

(4) The stability of the austenite constituent in QLT-treated 5.5Ni steel is determined primarily by its chemical composition.

The representative austenitic alloy, A, is thermally stable on cooling to room temperature, but undergoes an approximately 25% transformation on further cooling to  $77\text{K}$ . It is mechanically unstable at both room temperature and  $77\text{K}$ . Its stability is roughly comparable to that of the precipitated austenite in QLT-treated 5.5Ni steel. The volume fraction of precipitated austenite in QLT-treated 5.5Ni steel decreases slightly on cooling from the  $600^\circ\text{C}$  tempering temperature to room temperature [11,24], and drops by an additional 5% on cooling to  $77\text{K}$ . The remaining fraction of the precipitated austenite transforms on severe deformation at  $77\text{K}$ . The similarity of the two alloys in their

thermomechanical stability suggests that the stability of the precipitated austenite in 5.5Ni steel is controlled primarily by its composition, although its particle size and state of constraint in the alloy matrix may also have an influence. This conclusion is consistent with prior work [24,25] that compared the thermal stability of austenite that was precipitated in 5.5Ni steel by various heat treatments, and showed that its stability varied systematically with the composition determined by STEM analysis.

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Table 1. Chemical Composition of the Research Alloys (wt.%).

	Fe	C	Mn	Si	P	S	Ni	Cr	Mo
Fe-6Ni	Bal.	0.063	1.21	0.20	0.08	0.01	5.86	0.69	0.20
Retained Austenite*	Bal.	0.50**	3.90	0.30	-	-	9.00	2.10	2.0***
Tempered Martensite*	Bal.	0.01**	0.60	0.19	-	-	5.00	0.08	0.34***
Alloy A	Bal.	0.42	3.29	0.19	0.002	0.005	9.61	1.16	2.01
Alloy M	Bal.	0.021	0.57	0.18	0.001	0.006	4.98	0.09	0.31

\* STEM-EDAX analysis [24,25]

\*\* Calculated

\*\*\* Chemical extraction analysis [24,25]

Table 2. Mechanical Properties of the Base Alloy at 298K and at 77K.

	600°C, 4 hrs		670°C, 1 hr		670°C and 600°C, 1 hr	
Test Temperature	298K	77K	298K	77K	298K	77K
Y.S. (MPa)	640	910	680	920	660	880
U.T.S. (MPa)	830	1310	1000	1370	830	1220
R.A. (%)	73.3	73.8	70.2	73.1	75.0	77.5
Total elong. (%)	28.4	29.2	22.7	28.6	32.2	35.3
C <sub>v</sub> (J)	229.7	55.0	229.7	27.9	230.8	224.4
T <sub>B</sub>	83K		138K		below 77K	

Table 3. Mechanical Properties of Alloy A.

Test Temperature	298K	77K
Y.S. (MPa)	421	578
Fract. S (MPa)	1236	2162
Total elong. (%)	36.4	28.4
R.A. (%)	31.8	22.6
C <sub>v</sub> (J)	53.1	12.6

Table 4. Mechanical Properties of Alloy M.

Test Temperature	Q		QT		QLT		QL'T	
	R.T.	-196°C	R.T.	-196°C	R.T.	-196°C	R.T.	-196°C
Y.S. (MPa)	523.0	843.0	502.0	871.0	468.0	798.0	465.0	839.0
U.T.S. (MPa)	610.0	1034.0	585.0	949.0	567.0	904.0	572.0	910.0
Total elong. (%)	30.4	30.8	31.4	33.4	35.2	39.7	35.6	42.2
R.A. (%)	82.7	73.8	84.9	74.3	85.7	75.5	85.1	75.3
C <sub>v</sub> (J)	266.3	11.0	194.3	13.8	293.2	19.0	294.3	9.6

Q = 800°C (1 hr)

L = 670°C (1 hr)

L' = 710°C (1 hr)

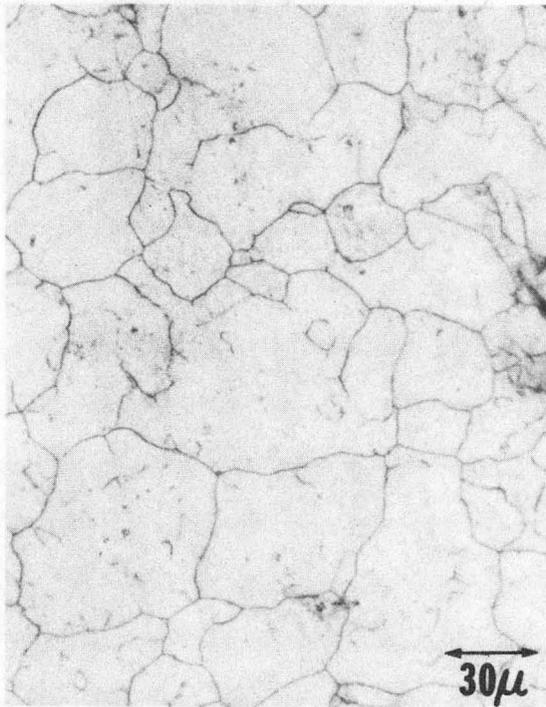
T = 600°C (1 hr)

Table 5. Room Temperature Mechanical Properties of Samples of Alloy M that were Cold Worked Prior to QLT Treatment.

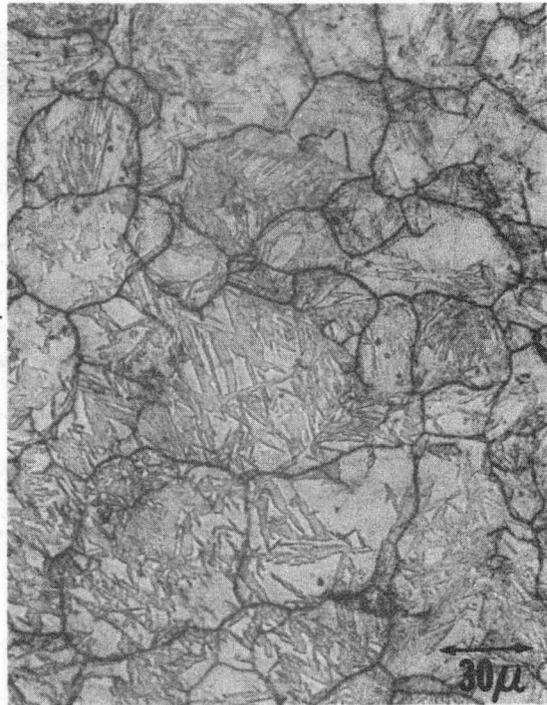
Cold Working	0%	25%	50%
Y.S. (MPa)	468.0	479.0	497.0
U.T.S. (MPa)	567.0	565.0	573.0
Total elong. (%)	35.2	35.3	34.6
R.A. (%)	85.7	86.9	83.8
C <sup>v</sup> (J)	293.2	294.3	293.9

### FIGURE CAPTIONS

1. Optical micrographs of alloy A after cooling to room temperature (RT) and after further cooling to 77K, liquid nitrogen temperature (LNT). The martensitic transformation product is evident in the sample cooled to 77K.
2. Optical micrograph of an etched cross section of a Charpy impact specimen of Alloy A that was broken at room temperature. The depth of martensitic transformation below the surface is apparent.
3. Optical micrographs of samples of alloy M that were cold worked by the amounts labelled under the respective figures before being given the QLT treatment. The micrographs illustrate the grain refinement caused by the initial cold work.
4. The variation of Charpy impact energy with temperature for QLT-treated 5.5Ni steel.
5. Scanning electron fractographs of Charpy impact specimens of alloy M that were broken at 77K. The heat treatments of the specimens were Q (a); QT (b); QL'T (c); QLT (d).
6. Charpy impact energy as a function of temperature for samples of alloy M that were cold worked by the labelled amounts before being given the QLT treatment.



**R.T.**



**L.N.T.**

Figure 1

XBB 804-5235

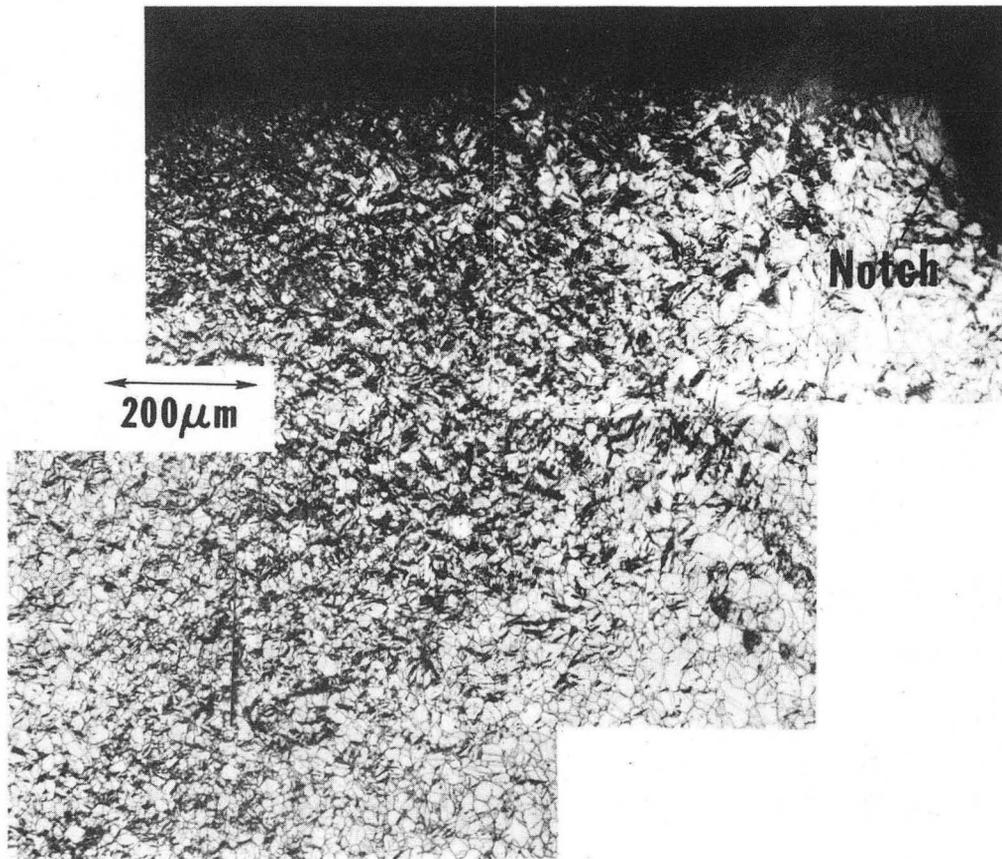


Figure 2

XBB 807-8542

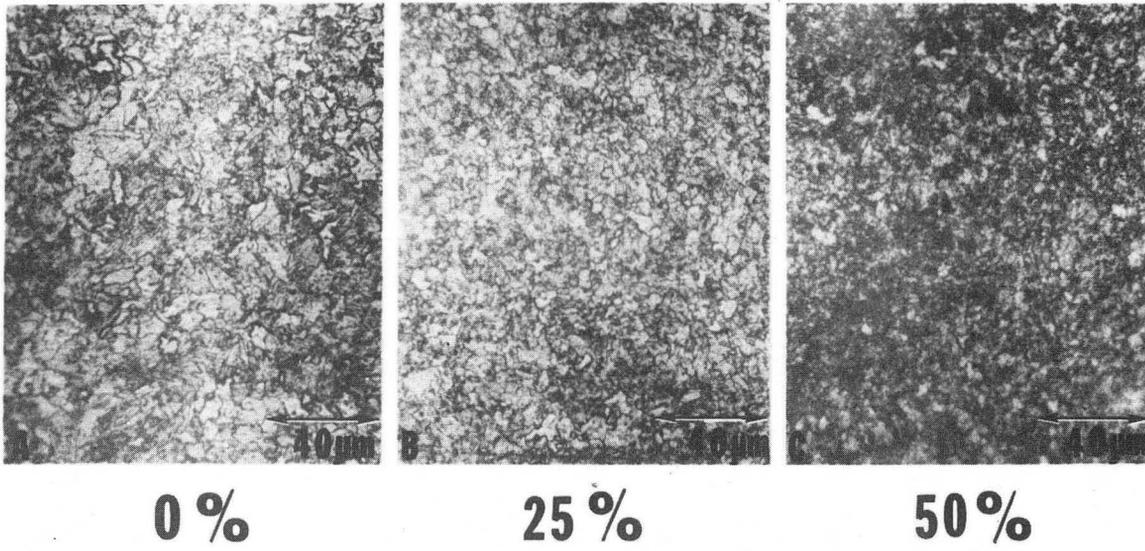
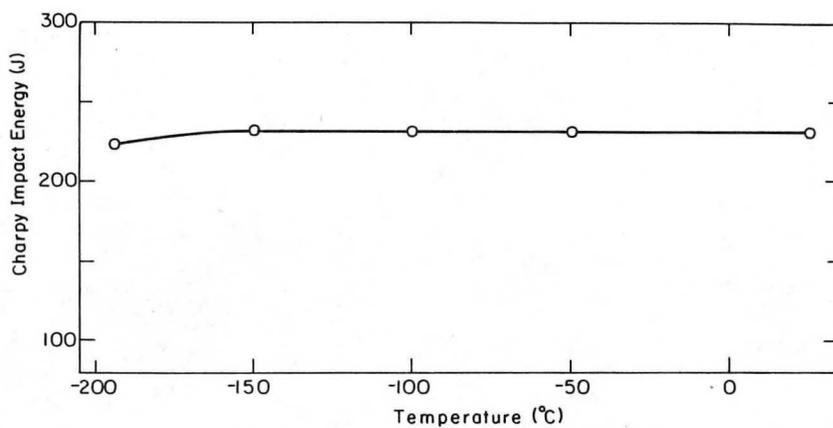


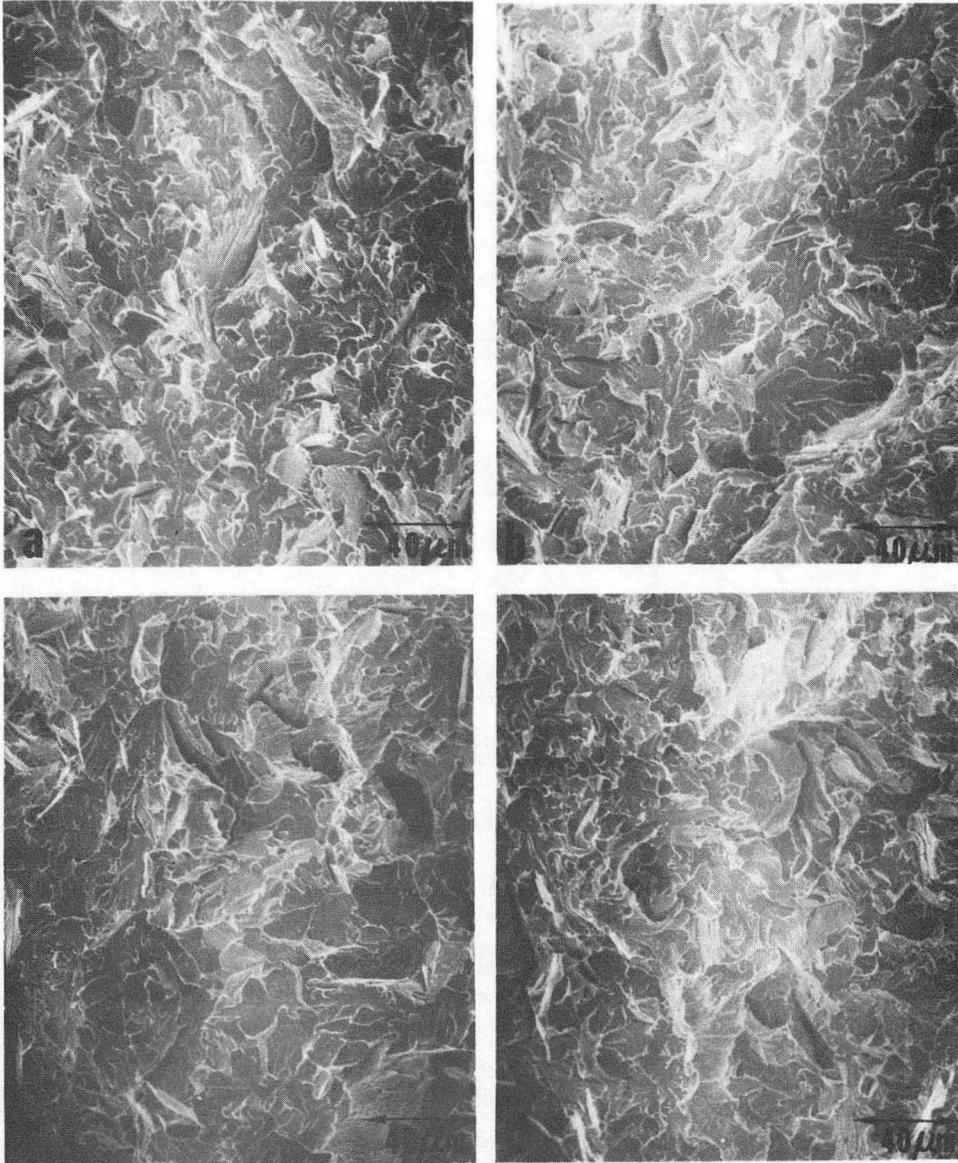
Figure 3

XBB 809-11265



XBL 835-5726

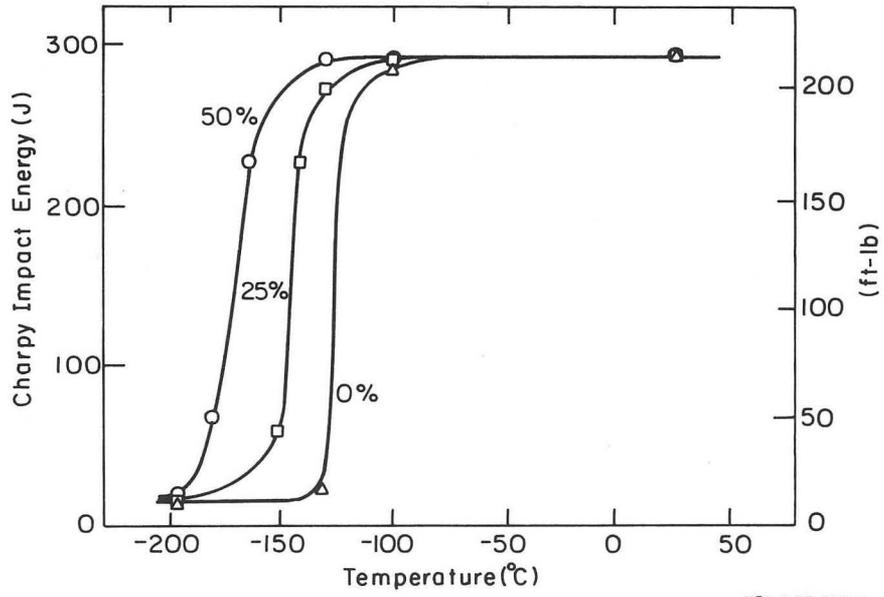
Figure 4



**L.N.T.**

Figure 5

XBB 806-7548



XBL 835-5727

Figure 6

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TECHNICAL INFORMATION DEPARTMENT  
LAWRENCE BERKELEY LABORATORY  
UNIVERSITY OF CALIFORNIA  
BERKELEY, CALIFORNIA 94720