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A METALLOGRAPHIC INTERPRETATION OF THE QLT HEAT TREATMENT OF FERRITIC CRYOGENIC STEELS

by

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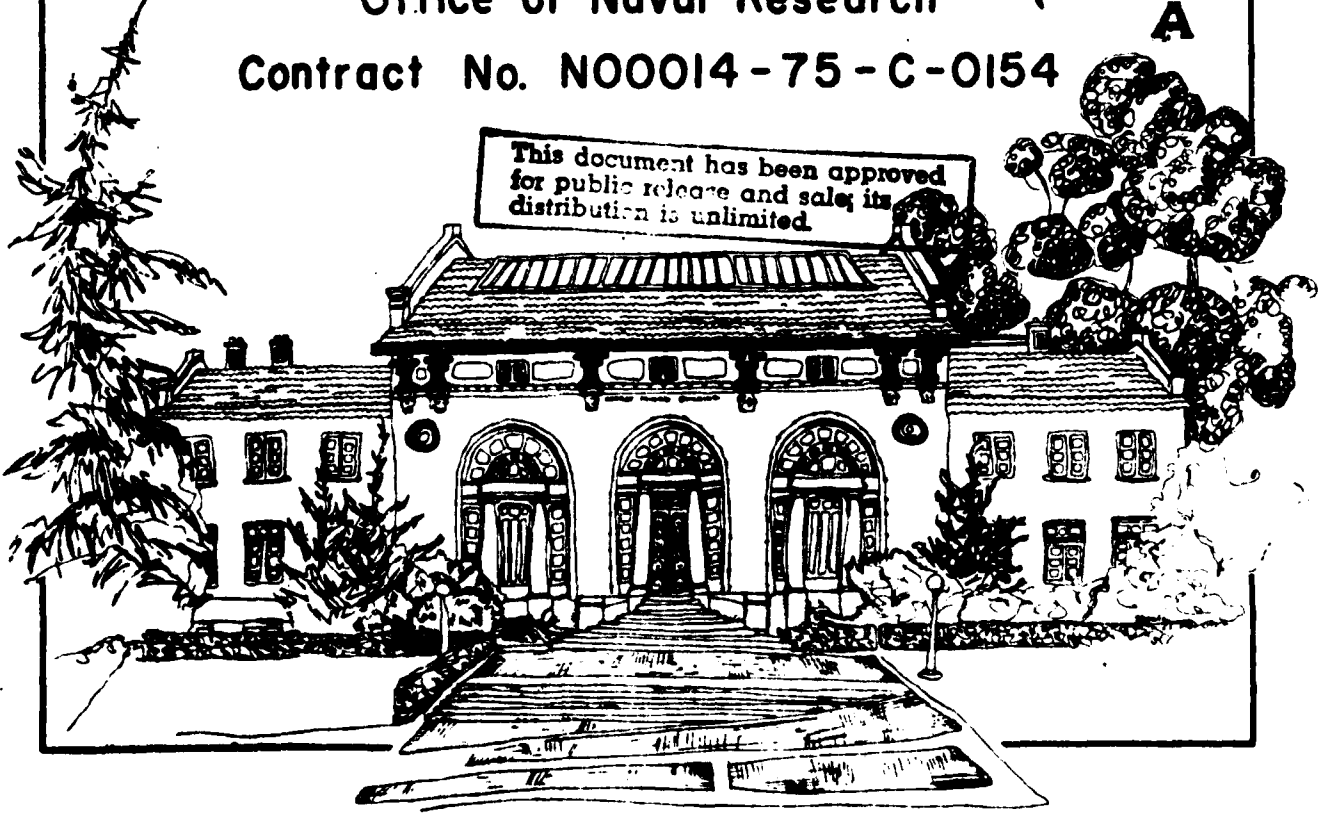and Mineral Engineering

UNIVERSITY OF CALIFORNIA, BERKELEY

MAY, 1983

REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM

1. REPORT NUMBER #13	2. GOVT ACCESSION NO. AD-A133 428	3. RECIPIENT'S CATALOG NUMBER

4. TITLE (and Subtitle)	5. TYPE OF REPORT & PERIOD COVERED
A METALLOGRAPHIC INTERPRETATION OF THE QLT HEAT TREATMENT OF FERRITIC CRYOGENIC STEELS	6. PERFORMING ORG. REPORT NUMBER

7. AUTHOR(s)	8. CONTRACT OR GRANT NUMBER(s)
J. I. Kim and J. W. Morris, Jr.	N00014-75-C-0154

9. PERFORMING ORGANIZATION NAME AND ADDRESS	10. PROGRAM ELEMENT. PROJECT. TASK AREA & WORK UNIT NUMBERS
University of California, Berkeley Department of Materials Science & Mineral Eng. Berkeley, California 94720	NR 031-762

11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE
NAVAL SYSTEMS COMMAND Washington, D.C. 20362	May, 1982
	13. NUMBER OF PAGES 5

14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)	15. SECURITY CLASS. (of this report)
OFFICE OF NAVAL RESEARCH 800 N. Quincy Street Arlington, VA 22217	Unclassified
	15a. DECLASSIFICATION/ DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

Presented at the International Cryogenic Materials Conference, Kobe, JAPAN
Published in "ICMC, Kobe, Japan, 11-14 May 1982", Butterworth, England, 1982.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Steel, Fe-Ni Alloys, Cryogenic Structural Materials, Heat Treatment,

Fracture, Ductile-Brittle Transition

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Commercial 5-6Ni steels are toughened for service by a 3-step heat treatment which is sometimes designated the "QLT" treatment. The present investigation was undertaken to determine why this treatment is necessary and successful. It was concluseded that the multi-step heat treatment is necessary because of the low nickel content. The intercritical anneal (L) serves to create regions of high solute contents along the martensite lath boundaries. The intercritical temper (T) then precipitates a dense distribution of high solute, stable austenite within these enriched regions.

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INTRODUCTION

Ferritic steels which are intended for structural use at cryogenic temperatures are thermally processed to lower the ductile-brittle transition temperature to below the intended temperature of service. In conventional 9Ni steel this treatment involves a straightforward intercritical temper at a relatively low temperature within the two phase ($\alpha + \gamma$) region. During the intercritical temper a fine, dense distribution of the austenite phase is precipitated along the boundaries of the dislocated laths of the martensite structure. This austenite is thermally stable on cooling to at least 77K. It lowers the ductile-brittle transition temperature by breaking up the crystallographic alignment of the martensite laths within packets [1]. Ferritic steels of lower nickel content can also be toughened for cryogenic service [2,3] as can ferritic iron manganese steels containing no nickel at all [4,5]. But in these cases more elaborate thermal treatments are required. The most commercially important of these alternative heat treatments are required. The most commercially important of these alternative heat treatments is the QLT treatment, which is a 3-step heat treatment employed for toughening the 5-6Ni steel developed some years ago by the Nippon Steel Corp. in Japan and by ARMCO steel in the United States. The QLT treatment is the fourth of those diagrammed in Fig. 1. It involves two sequential intercritical heat treatments. The first of these (1) is an intercritical anneal in the upper range of the two phase ($\alpha + \gamma$) field. The second (T) is an intercritical temper at lower temperature within the two phase field.

While the QLT treatment has been used with commercial success for some years, the fundamental reasons for its effectiveness remain unclear. To clarify the microstructural sources of toughening we have studied the interplay of composition and microstructure in determining the ductile-brittle transition temperature benefit achieved from the QLT treatment. The alloy used in this investigation was a commercial grade 5.5Ni steel which was provided by the Nippon Steel Corp. Its composition was determined to be: Fe-5.86Ni-1.21Mn-0.69Cr-0.20Mo-0.2Si-0.06C-0.01S-0.08P. The alloy was annealed at 1200°C for two hours to remove the effect of prior thermal mechanical treatment. It was then solution annealed at 900°C for 2 hours. Experimental samples were cut from the annealed plates and subjected to one of the four heat treatments diagrammed in Fig. 1. The microstructure of the alloys was studied as a function of heat treatment using optical, transmission and scanning transmission electron microscopy. The latter technique was used to determine the chemical composition of the austenite present in the heat-treated samples [6]. The volume fraction of precipitated austenite was determined by x-ray diffraction as suggested by Miller [7] and by backscatter Mossbauer spectroscopy using the apparatus and techniques described by Fultz [8].

RESULTS

The volume fraction of austenite in the microstructure at room temperature and after chilling to 77K is tabulated as a function of heat treatment in Table 1, together with the concentration of detected solutes in the austenite,

as determined by STEM measurements [6]. The variation of microstructure with heat treatment is described in detail in Reference 9. The results may be summarized as follows:

1. **The Q condition.** In the as-quenched condition the alloy has a dislocated lath substructure in which martensite laths are organized into aligned packets which subdivide the prior austenite grains. The martensite laths within a given packet are almost always in a single variant separated by low-angle lath boundaries.

2. **The QT_2 Microstructure.** In the QT_2 condition the primary constituent is martensite whose lath boundaries are decorated by a mixture of retained austenite and fresh martensite (from retransformation of the austenite nucleated during the tempering step). The austenite is retained in relatively low volume fraction and has the Kurdjemov-Sachs relation to the parent martensite. This austenite is relatively lean in substitutional solute content. Its low solute content and small volume fraction are apparently due to a combination of the low tempering temperature, the relatively short tempering time, and the low diffusivity of the substitutional species which preferentially segregate to the austenite phase. The low solute content is presumably responsible for the thermal instability of this austeniteμ the bulk of it reverts to martensite on cooling to 77K.

3. **The QT_{100} Microstructure.** A 100 hrs heat treatment gives the structure shown in Fig. 2. The tempered martensite retains a fine polygonized substructure. The austenite is present in much higher volume fraction, and the precipitated austenite particles have coarsened and spheriodized into more equiaxed subgrains of approximately 0.5μ diameter. This austenite is much richer in substitutional solute content (Table 1) and is thermally stable. It is almost entirely retained after the sample has been cooled in liquid nitrogen.

4. **The QL Microstructure.** Increasing the heat treatment temperature to $670^{\circ}C$ for 1 hour yields a microstructure containing three distinct microstructural constituents. These are: 1) tempered martensite having a fine, polygonized structure, 2) fresh, highly dislocated martensite which presumably arises from the transformation of the greater part of the austenite precipitated during the intercritical anneal and 3) retained austenite. The austenite is not as rich in substitutional solutes as that after QT_{100} and must be expected to be lower in carbon content because of its higher temperature of formation. It is thermally unstable with respect to martensitic transformation on cooling to 77K (Table 1). Given the combination of extensive chemical redistribution and cycling phase transformation during the L treatment one might expect a substantial decomposition of the martensite packet structure. However, this is not the case. The cyclic $\alpha \rightarrow \gamma \rightarrow \alpha$ transformation has a strong memory. When precipitated austenite reverts to martensite on cooling, it almost invariably returns to the martensite variant which gave it birth, with the consequence that the martensite packet size remains virtually unchanged.

Table 1. Composition and Volume Fraction of Precipitated Austenite.

Heat Treatment	Fe	Ni	Mn	Cr	Si	Volume Fraction (%) 298K	77K
QT_2	89.5	6.6	2.2	1.2	0.1	5.9	2.8
QT_{100}	85.0	9.1	3.8	1.5	0.2	8.2	7.9
QL	86.1	8.4	3.7	1.3	0.1	8.8	2.0
QT	82.8	8.8	4.3	3.2	0.5	9.2	8.8
Base Alloy	bal.	5.86	1.21	0.69	0.2	-	-

5. **The QLT Microstructure.** When the QL treatment is followed by a temper at 600°C for 1 hour, the result is the QLT treatment. The microstructure of the steel after this treatment (Fig. 3) consists of three constituents. The L treatment causes a chemical decomposition of the alloy into regions which are relatively rich or relatively poor in solute content. The subsequent T treatment causes a secondary decomposition of the solute-rich islands. The final microstructure consists of a low solute martensite matrix containing elongated regions of solute-rich tempered martensite which themselves contain elongated subregions of high-solute precipitate austenite with a slight admixture of fresh martensite. The austenite phase is shown in the dark field micrograph in Fig. 3. Its composition is very nearly equal to that of the austenite formed after 100 hours tempering at 600°C. Virtually all of this austenite is retained on refrigeration at 77K.

The microstructural changes which occur during heat treatment have almost no effect on the tensile properties of the alloy, but they profoundly influence its impact toughness. The Charpy impact energy is plotted as a function of temperature for each of the four heat treatments in Fig. 4. The evident changes include the pronounced increase in the upper shelf Charpy energy after the QL treatment, and the dramatic decrease in the ductile-brittle transition temperature in the QLT condition.
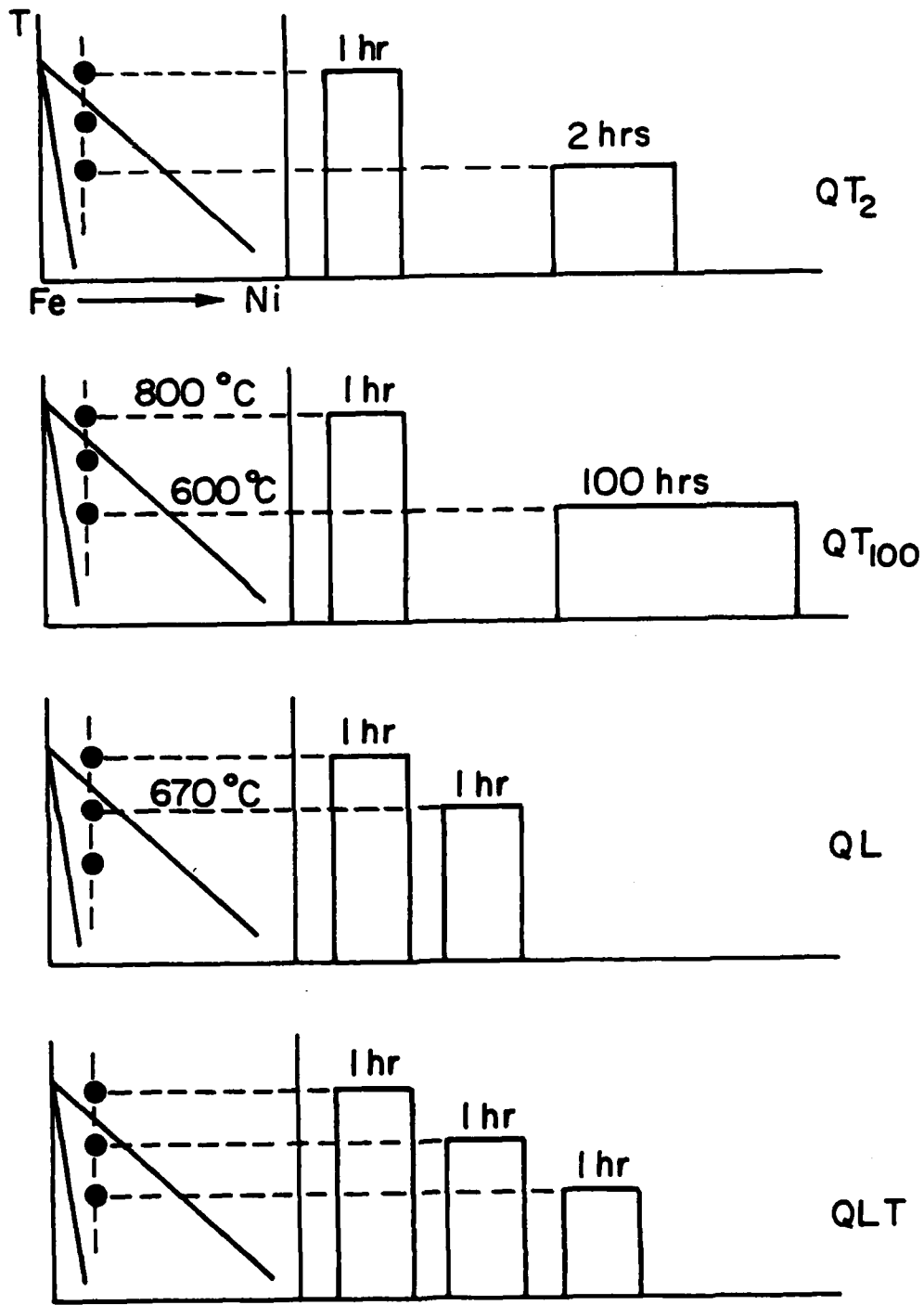
DISCUSSION AND CONCLUSION

Previous research [1,9,10] suggests that intercritical heat treatment influences the toughness of ferritic cryogenic steels primarily because of the precipitation and retention of the austenite phase. The precipitation of austenite can affect both the level of toughness above the ductile-brittle transition and the value of the ductile-brittle transition temperature. The level of toughness above the ductile-brittle transition principally reflects the distribution of carbon and carbides, which can be effectively gettered during austenite precipitation. The carbon redistribution is the probable

cause of the dramatic increase in the upper-shelf toughness after the QL treatment [10]. The decrease in the ductile-to-brittle transition temperature reflects the role of austenite in refining the effective grain size of the alloy by breaking up the crystallographic alignment of martensite laths in the packet structure.

To accomplish grain refinement the precipitated austenite must have two attributes: it must have sufficient thermal stability to be retained at the testing temperature (though it ultimately transforms during mechanical deformation [1,9] (and it must be sufficiently dense in distribution to accomplish grain refinement. The austenite introduced by the QT_2 and QL heat treatments has insuffjicient thermal stability. The QT_{100} treatment introduces a thermally stable austenite phase, but the austenite is blocky and widely dispersed through the matrix. Such an austenite is ineffective in grain refinement since it is relatively easy to find transpacket cleavage paths which avoid the austenite entirely. The QLT treatment, on the other hand, establishes an austenite phase which is both thermally stable and dense in its distribution because the precipitation of austenite in the T treatment occurs in interlath regions which have already been enriched in solute species by the prior L treatment.

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FIGURE 1. The Four Heat Treatments

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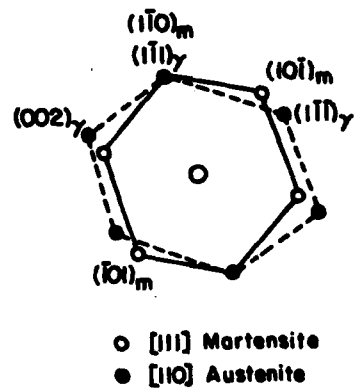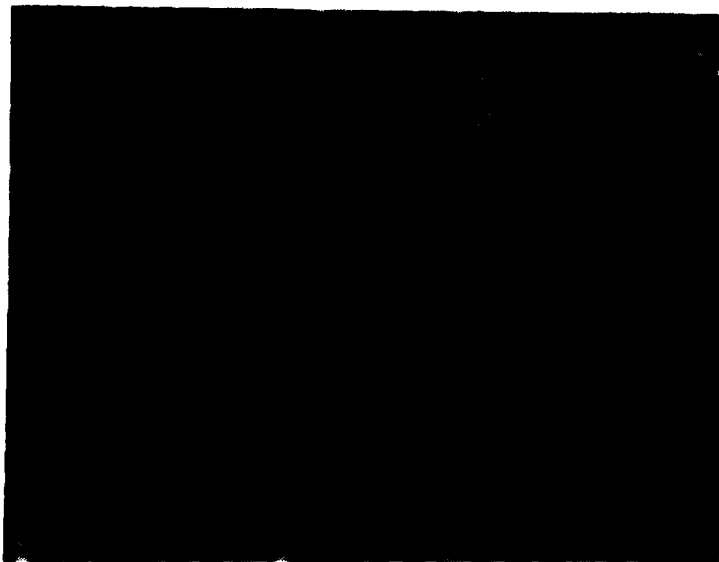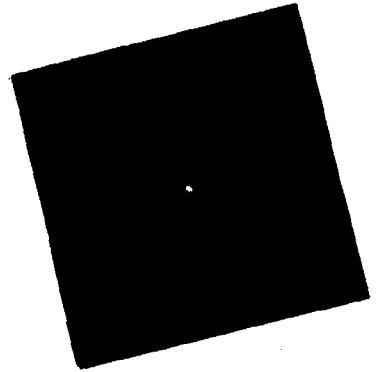
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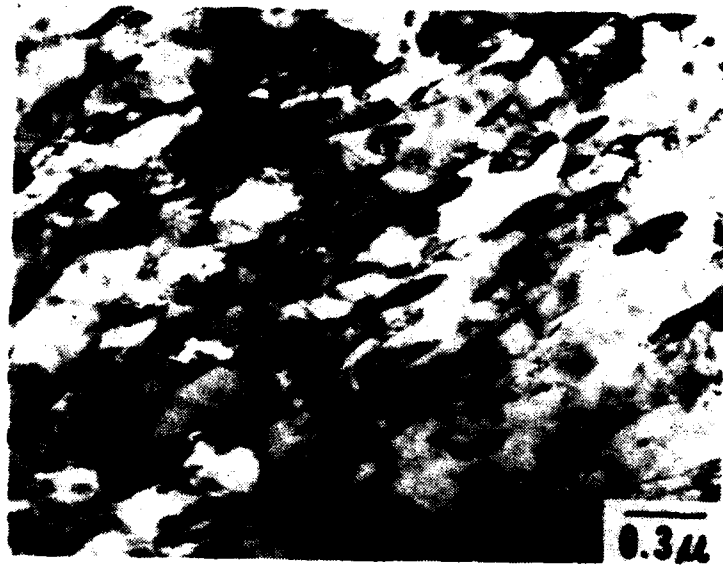O [111] Martensite
● [110] Austenite
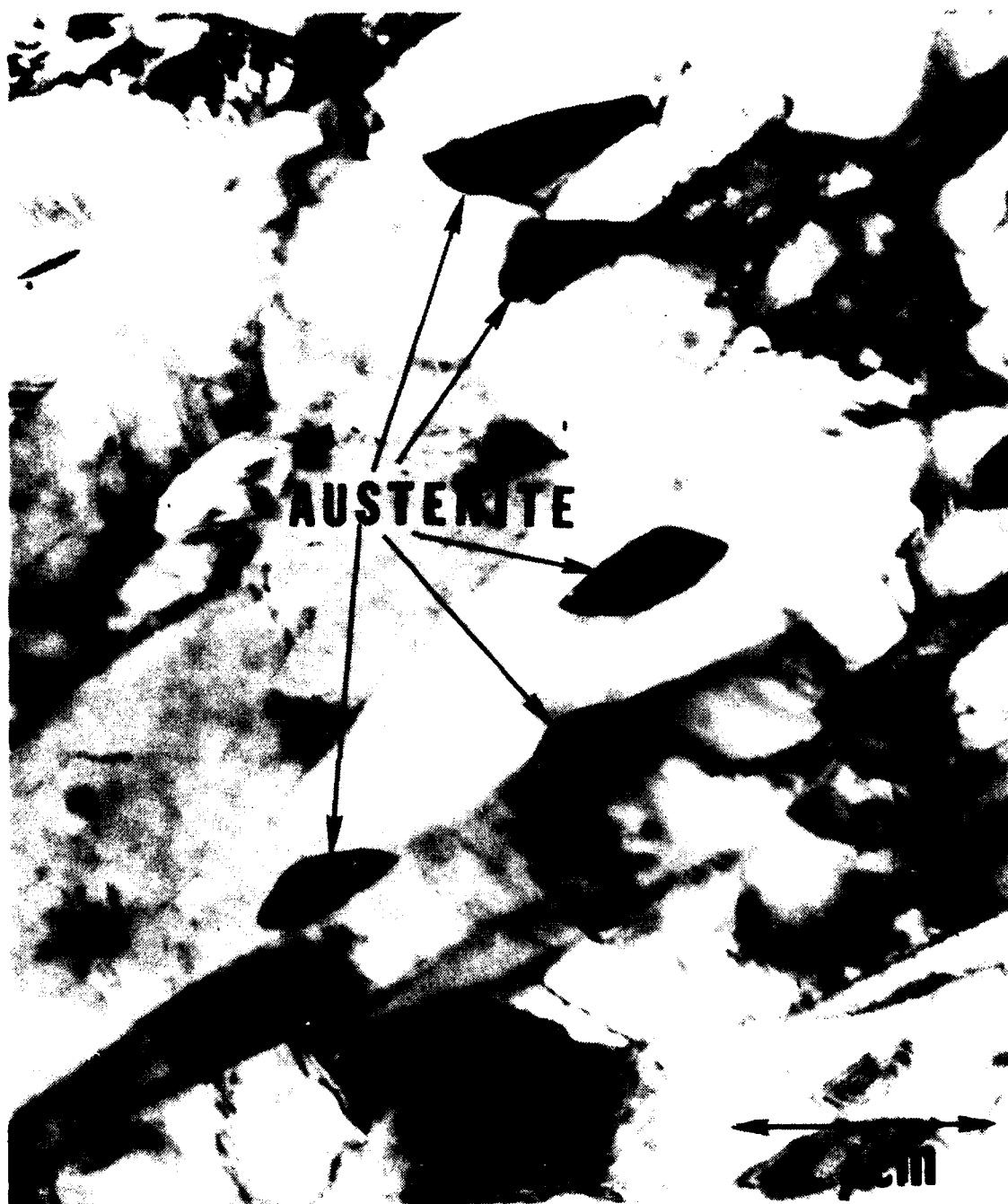
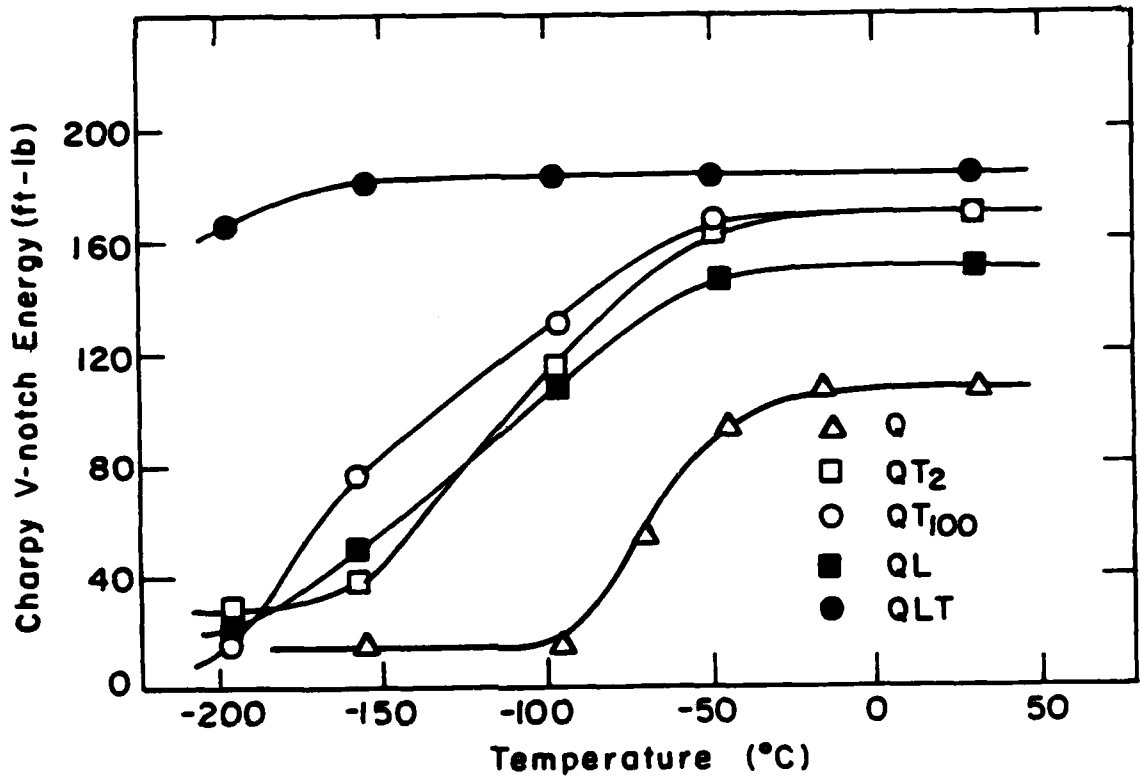FIGURE 2. The QT_{100} Microstructure

FIGURE 3. The QLT Microstructure

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FIGURE 4. Ductile Brittle Transition Behavior