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A STUDY ON THE RETAINED AUSTENITE IN FINE-GRAINED Fe-12Ni-0.25Ti ALLOY

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A STUDY ON THE RETAINED AUSTENITE IN FINE-GRAINED Fe-12Ni-0.25Ti ALLOY

by

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ABSTRACT

The behavior of stabilized austenite in a fine-grained Fe-12Ni-0.25 Ti cryogenic alloy system was investigated. When the alloy was held at a temperature within the two phase ($\alpha + \gamma$) field austenite reversion occurred by a diffusion-controlled process. The redistribution of solute elements appeared to control the stability of the reverted austenite. The microstructural appearance of the retained austenite was examined using transmission electron microscopy. A preferrential distribution of the austenite phase along martensite lath boundaries was observed. A precipitate-correlated austenite was also found to occur. The beneficial effect of introducing retained austenite appeared in the improvement in the tensile elongation as well as in the Charpy impact toughness at low temperatures. The retained austenite in this system did not improve K_{IC} at 77°K. On the contrary, samples containing retained austenite showed increased susceptibility to unstable crack propagation in low temperature fracture toughness tests.

INTRODUCTION

The development of the retained austenite phase in ferritic steels has been the subject of a significant body of research in recent years. The motivation of this research comes from two sources: fundamental interest in the phase transformation behavior and the possibly beneficial effect of retained austenite on the subsequent mechanical properties.

In an Fe-20Ni-0.8C alloy some austenite was observed to remain untransformed after continuous cooling to a sub-zero temperature. Kelly and Nutting[1] attributed the stability of this partially transformed austenite to the difficulties in the cooperative movement of atoms to form martensite. In low-carbon Fe-Ni based alloys, an over-aging (maraging steels)[2-4] or a tempering (low-nickel cryogenic steels)[5-6] process often yields stabilized austenite. During these processes the solute elements seem to play a significant role in the stabilization of the two-phase decomposed austenite. The effect of ultra-fine grain size on the stability of austenite was also reported.[7]

Although there have been numerous reports on the mechanical properties of ferritic steels containing some volume fraction of retained austenite, the exact role of the austenite is not well understood. A deleterious effect on strength has been reported in maraging steels.[4,8-10] A significant lowering of the ductile-brittle transition temperature has been observed in low-nickel cryogenic steels.[5-6] An enhanced fracture toughness has been reported in a TRIP steel[24] as well as in a thermal-cycled maraging steel[17] where metastable austenite undergoes martensitic transformation during testing. The

improvement in the fracture toughness was also reported in an Fe-Cr-C alloy where interlath retained austenite films were present.[23]

Recently a grain-refining technique for the Fe-12Ni-0.25Ti cryogenic alloy system,[11] which utilizes the γ-α phase transformation through an alternate thermal-cycling process, has been developed. The alloy, after grain-refinement (to about 1μm), showed unusual ductility in low temperature (77°K, 4°K) fracture toughness tests. Retained austenite was introduced into this fine-grained, ductile matrix in the hope of achieving a further improvement in cryogenic ductility without sacrificing strength. Given the large grain boundary area, austenite reversion can take place easily. The objective of this paper is to present some results of a study of stabilized austenite found in this way. The investigation was undertaken firstly to examine the morphology and stability of the retained austenite. Then, the cryogenic mechanical properties were studied after introducing a moderate amount of retained austenite.

EXPERIMENTAL

Low-carbon alloys of nominal composition Fe-12Ni-0.25Ti were induction melted in an argon gas atmosphere, then cast into 9.1 Kg ingots in a copper chill mold. The ingots were homogenized under vacuum at 1150°C for 80 hours, then forged at 1100°C into plates. Each plate was annealed at 900°C for two hours under argon gas atmosphere, then grain-refined through the thermal cycling procedure described elsewhere.[11] A schematic diagram of the heat treatment for grain-refining is shown in Fig. 1. A chemical analysis of a typical ingot is presented in Table I.

The specimens for X-ray diffraction analysis were prepared metallographically. To remove any mechanically-induced transformation effect a chemical polishing was carried out using a hydro-fluoric acid solution (100 ml H_2O_2 + 4 ml HF). X-ray diffraction was conducted using Cu-K_α radiation with a LiF monochrometer. The calculation of the retained austenite volume percent was based on Miller's method,[12-13] comparing average integrated intensities of the (220) and (311) austenite peaks to that of the (211) martensite peak. No correction was made to take the precipitate into account. Possible preferred orientation may also cause an error in the estimation of the exact amount of retained austenite.

For transmission electron microscopy (TEM) thin foils were made by a conventional jet-polishing technique using the chromic-acetic acid electrolyte (75 gm CrO_3 + 400 ml acetic acid + 21 ml H_2O). The electron microscopes used were mainly a Hitachi Hu-125 operated at 100 kV and a Siemens Elmiscope 1 operated at 100 kV. The fracture surfaces of Charpy specimens tested at 77°K were examined in a JEOLCO JSM-U_3 scanning electron microscope (SEM) operated at 25 kV.

Modified cylindrical specimens of 12.7 mm gage length and 3 mm gage diameter were used for the tensile tests at 77°K and 4°K. Charpy V-notched impact tests were conducted at 77°K and 4°K on a MTS universal testing machine. Since the thickness of the compact tension specimens did not meet the ASTM requirement the equivalent K_{IC} values were calculated by methods based on the "Equivalent energy" and "J-Integral" concepts.[23] In calculating an equivalent K_{IC} from the "J-Integral" concept the deflection was measured on the loading axis.

EXPERIMENTAL RESULTS AND DISCUSSION

Microstructures

As shown in Fig. 1, the heat treatment employed in this experiment consisted of two parts. The first part accomplishes grain-refinement through alternate thermal cycling: 730°C/2 hours/AC (air cooling to room temperature) + 650°C/2 hours/AC, two cycles. The second part is the reheating treatment at various temperatures within the two phase range.

A TEM micrograph of the grain-refined structure, Fig. 2(a), shows a typical martensite lath structure with a high density of dislocations. In a reheated specimen, coarsened precipitates were resolved, Fig. 2(b). In light of extensive data from precipitate studies in maraging steel,[2] these precipitates are presumed to be Ni_3Ti although an elaborate identification was not made.

While the grain-refined structure was completely α-phase, the presence of retained austenite after the reheating treatment was revealed by a dark-field technique in TEM. Illuminated by a (002) austenite diffraction spot a preferrential distribution of retained austenite elongated along martensite lath boundaries was observed, Fig. 3(a). The retained austenite distribution along martensite lath boundaries was not unexpected in view of prior results on similar systems.[10,18-20] In some areas retained austenite associated with precipitates was also resolved, Fig. 3(b). This observation supports the hypothesis that austenite reversion can take place directly on previously formed precipitates.[4-5,17] Over-aged precipitates of high solute concentration may serve as favorable sites for the austenite nucleation.

Amount and Stability of Retained Austenite

As noted in the previous section retained austenite was not identified in the grain-refined structure by TEM work. The X-ray diffraction analysis gave a consistent result, i.e., the starting material (just grain-refined) was completely ferritic (α). Therefore the austenite stabilization mechanism claimed by Kelly and Nutting[1] in a carbon containing alloy does not seem applicable to the present system.

After the reheating treatment, the material was shown to contain a certain amount of retained austenite by both TEM and X-ray study. Fig. 4 shows the variation in the amount of retained austenite measured by X-ray after different reheat treatment.

The appearance of maxima at the 550°C treatment is noticeable. It could be understood in terms of solute redistribution since the reheating process is diffusion-controlled. At low temperature the reversion process is too sluggish. At high temperature the concentration of the solutes Ni and Ti, which lower Ms,[15] will tend to attain an equilibrium composition. A similar appearance of the maxima was also reported in a tempering process of a low nickel system.[5]

The rate of austenite reversion at 550°C was estimated through isothermal study. A roughly linear slope was observed in the plot of the volume percent retained austenite vs. the logarithm of the holding time, Fig. 5.

The stability of reverted austenite can be discussed in two ways. One is the thermal stability, as measured by the M_s and M_f temperatures. The other is mechanical stability, as measured by M_d. The reverted

austenite in the present system did not show a well-defined M_f. As is illustrated by the open circles in Fig. 5, there was no substantial change in the stability of the retained austenite when it was cooled to 77°K from room temperature. This remained true even after cooling to 4°K. While the retained austenite showed extremely high thermal stability the stability against mechanical deformation was low. After 50% cold reduction at room temperature the martensitic transformation was essentially completed. The M_d temperature for this system is apparently higher than room temperature.

Mechanical Properties

The effect of retained austenite on the mechanical properties of ferritic steels remains unclear. A direct loss of strength proportional to the amount of retained austenite has been reported from several studies.[4,8,10] Certain other observations remain controversial.[18,20-21] For example, a positive contribution of retained austenite to the yield stress was suggested on the grounds that austenite retained along martensite lath boundaries may cause difficulties in transmitting slip across these boundaries.

In the present investigation changes in the low temperature mechanical properties were manifested in three ways: in the tensile properties, the Charpy impact toughness and the fracture toughness. The tensile properties at 77°K after reheating 2 hours at various temperatures are plotted in Fig. 6. The yield stress slightly increases after a low temperature (475°C) reheating compared to that of initial grain-refined material. After a 550°C/2 hours/WC treatment it remains roughly

the same. As the reheating temperature was raised the yield stress began to decrease. On the whole, the total and uniform elongations increased together.

Several effects such as precipitation, recovery and retained austenite might be considered to account for the variation in yield stress. As these factors presumably are combined to give a net change it is difficult to isolate one from the others. However, it seems plausible to take the retained austenite effect for the appreciable increase in the uniform elongation. An increase in elongation with increasing amounts of retained austenite has been reported in several systems.[3,17,20,22] Nevertheless, it must be recalled that most of these improvements in the elongation were accompanied by a loss in strength. In the present study about a 70% increase of uniform elongation at 77°K after 550°C treatment was obtained without sacrificing yield strength. An improvement in the elongation was reproduced in a 4°K test whose engineering stress-strain curve is shown in Fig. 7.

The Charpy impact toughness at 77°K was markedly increased after the reheating treatment. The results are presented in Fig. 8(a), where the reheating temperature is taken as the abscissa. The C_V (absorption energy in the Charpy V-notched impact test) values for the 8-hour treatments are generally higher than those for the 2-hour treatments. The increment for a 475°C treatment was small compared to the others. A plot of C_V data in the 4°K test is shown in Fig. 8(b), where an increment ranging from 30 to 40 ft-lbs compared to that of the just grain-refined material is evident. The role of retained austenite in cryogenic low nickel steels has been ascribed to the supression of the ductile-brittle

transition temperature.[5-6] Nonetheless, the exact mechanism to explain how retained austenite improved the impact toughness remains unclear. Often it has been attributed to a matrix purifying effect or to a crack tip blunting effect caused by the characteristic properties of a f.c.c. structure. The ductile-brittle transition temperature of the present alloy is already lower than 77°K after grain-refinement. Therefore the role of retained austenite in the improvement of C_V after reheating would be difficult to interpret in terms of the transition temperature. However, an appreciable increase in C_V was obtained at the same yield stress level after 550°C treatment.

The equivalent K_{IC} values measured at 77°K in the compact tension fracture toughness test are shown in Table II. The two approaches, "Equivalent energy" and "J-Integral" calculations showed excellent agreement in the static tests (Cross head speed: 1.3 cm × 10^{-3}/sec). Several studies on metastable austenite reported a beneficial effect on the fracture toughness.[17,23-24] Referred to the results of the tensile test and Charpy impact test some improvement in the fracture toughness might have been expected. However, the reheating treatment turned out to be ineffective in improving the K_{IC} values further. In fact, a deleterious aspect set in as an unstable crack propagation, Fig. 9. One critical difference between the Charpy impact test and the usual fracture toughness test (static) is the strain rate. Therefore a 10^3 times faster cross head speed was employed to examine the strain rate effect. Although K_{IC} value calculations based on the two different approaches showed significant discrepancies in this test, the comparative value was not improved by adding the reheating treatment to the grain-

refined material. Finally a possible influence of the retained austenite phase on the fracture mode was examined. A fractograph taken from the fractured surface of a Charpy bar tested at 77°K, Fig. 10, shows that retained austenite does not give rise to an apparent change in the mode of failure, micro-void nucleation and coalescense.

CONCLUSIONS

Based on the present study on the retained austenite phase in a fine-grained Fe-12Ni-0.25Ti alloy, the following conclusions are made:

1) The diffusion-controlled reverted austenite can be stabilized chemically. The thermal stability of the reverted austenite depends on the temperature at which it was produced.
2) The retained austenite appears predominantly along martensite lath boundaries. Precipitate-correlated austenite nucleation was also observed.
3) Most of austenite stabilized by the reheating process is unstable against mechanical deformation.
4) Along with the austenite retention, significant improvements were obtained in the low temperature tensile ductility and Charpy V-notched toughness at the same strength level.
5) The reheating treatment did not improve low-temperature fracture toughness in the present ductile alloy system. Unstable crack propagation was observed in some specimens containing retained austenite.

ACKNOWLEDGMENTS

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Table I. Chemical composition of steel (wt. %)

Fe	Ni	Ti	C	N	O	P	S
Bal.	11.99	0.20	0.005	<0.001	<0.001	0.001	0.002

Table II. Fracture toughness at 77°K

C.H.S.: 1.3 cm × 10^{-3}/sec

units; ksi$\sqrt{in}$

H.T. \ Equivalent K_{IC}	From E.E.	From J-Int.
G.R.	298	300
G.R.+450°C/20hrs/WC	230	232
G.R.+475°C/2 hrs/WC	258	258
G.R.+550°C/2 hrs/WC	300	304
G.R.+550°C/60hrs/WC	280	281
G.R.+600°C/2 hrs/WC	274	274

C.H.S.: 1.3 cm/sec

H.T. \ K_{IC}	From E.E.	From J-Int.
G.R.	154	255
G.R.+550°C/2 hrs/IBQ	162	242
G.R.+550°C/2 hrs/AC	157	248

E.E.: Equivalent energy approach

J-Int.: J-Integral approach

C.H.S.: Cross Head Speed

FIGURE CAPTIONS

Fig. 1. Schematic diagram for the heat treatments.

Fig. 2. Microstructures (TEM) of (a) Grain-refined, (b) Reheated specimen (550°C/8 hours/wc) with coarsened precipitates.

Fig. 3. Distribution of the retained austenite (TEM). Reheated (525°C/1 hour/wc) after grain-refinement.

(a) Bright-field image and dark-field image illuminated by $(002)_\gamma$ spot.

(b) Same as (a), different area.

Fig. 4. Volume percent of retained austenite with different reheat treatment.

Fig. 5. Volume percent of retained austenite by isothermal holding at 550°C.

Fig. 6. Tensile properties at 77°K.

Fig. 7. Engineering stress-strain curve in a 4°K tensile test. Grain-refined and reheated at 550°C for 1 hour.

Fig. 8. Charpy V-notched impact toughness

(a) at 77°K

(b) at 4°K.

Fig. 9. Characteristic curves in fracture toughness test at 77°K.

Fig. 10. Fractograph (SEM). Reheated (550°C/2 hours/wc) after grain-refinement.

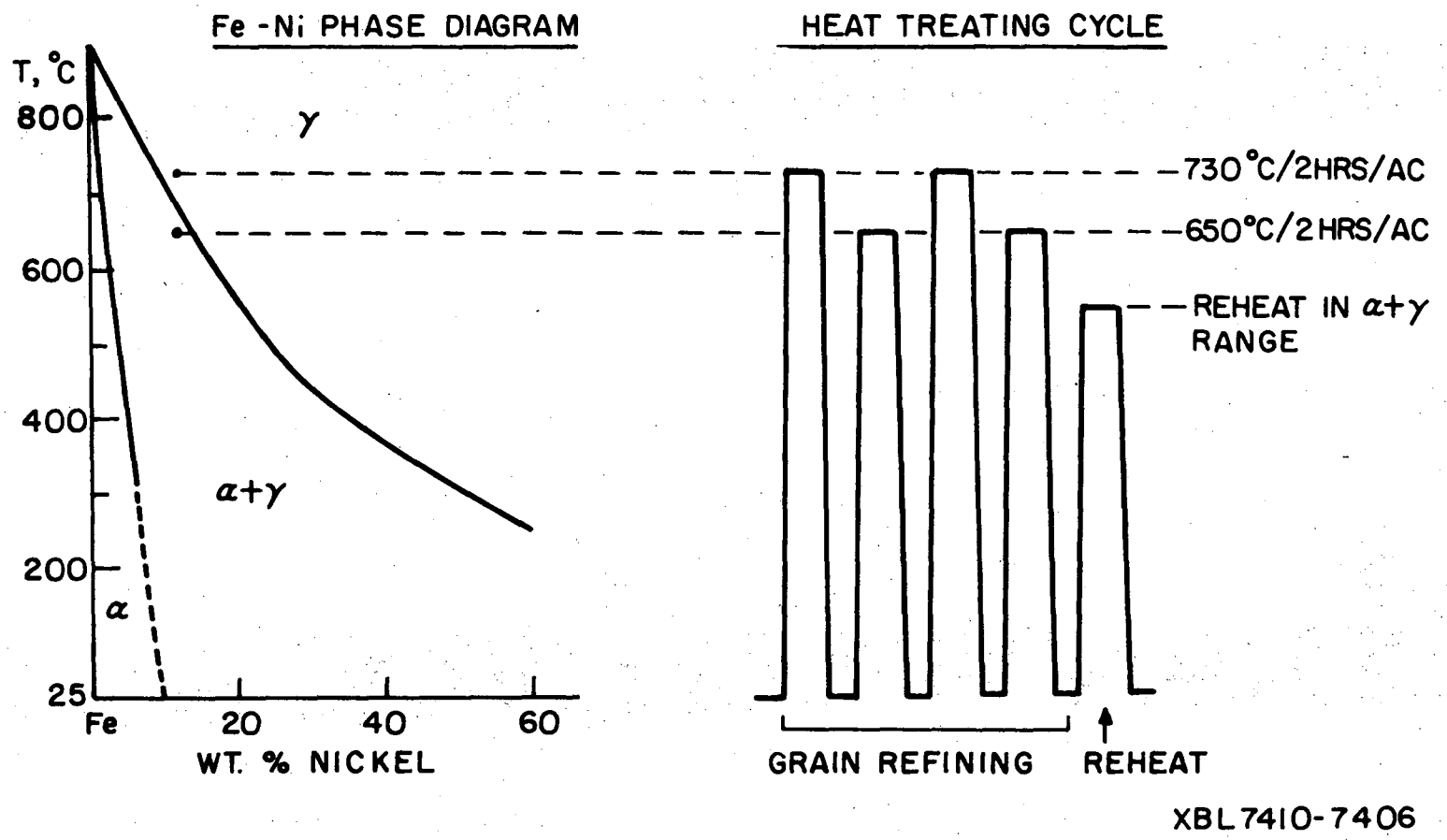
Fe -Ni PHASE DIAGRAM
T, °C
800
600
400
200
25
γ
α+γ
α
Fe
20
40
60
WT. % NICKEL
HEAT TREATING CYCLE
730°C/2HRS/AC
650°C/2HRS/AC
REHEAT IN α+γ RANGE
GRAIN REFINING
REHEAT
XBL7410-7406

Fig. 1

0 0 0 0 4 3 0 2 3 0 3

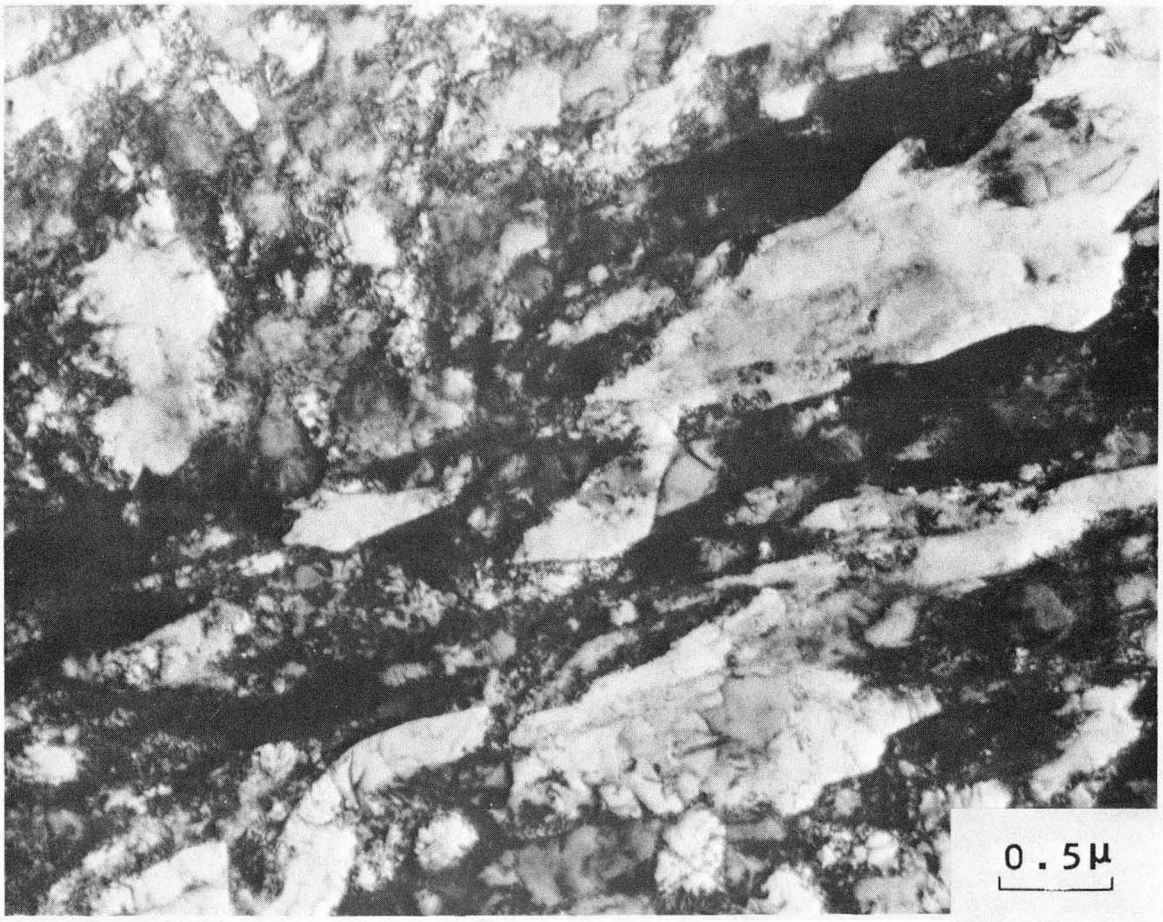

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Fig. 2a

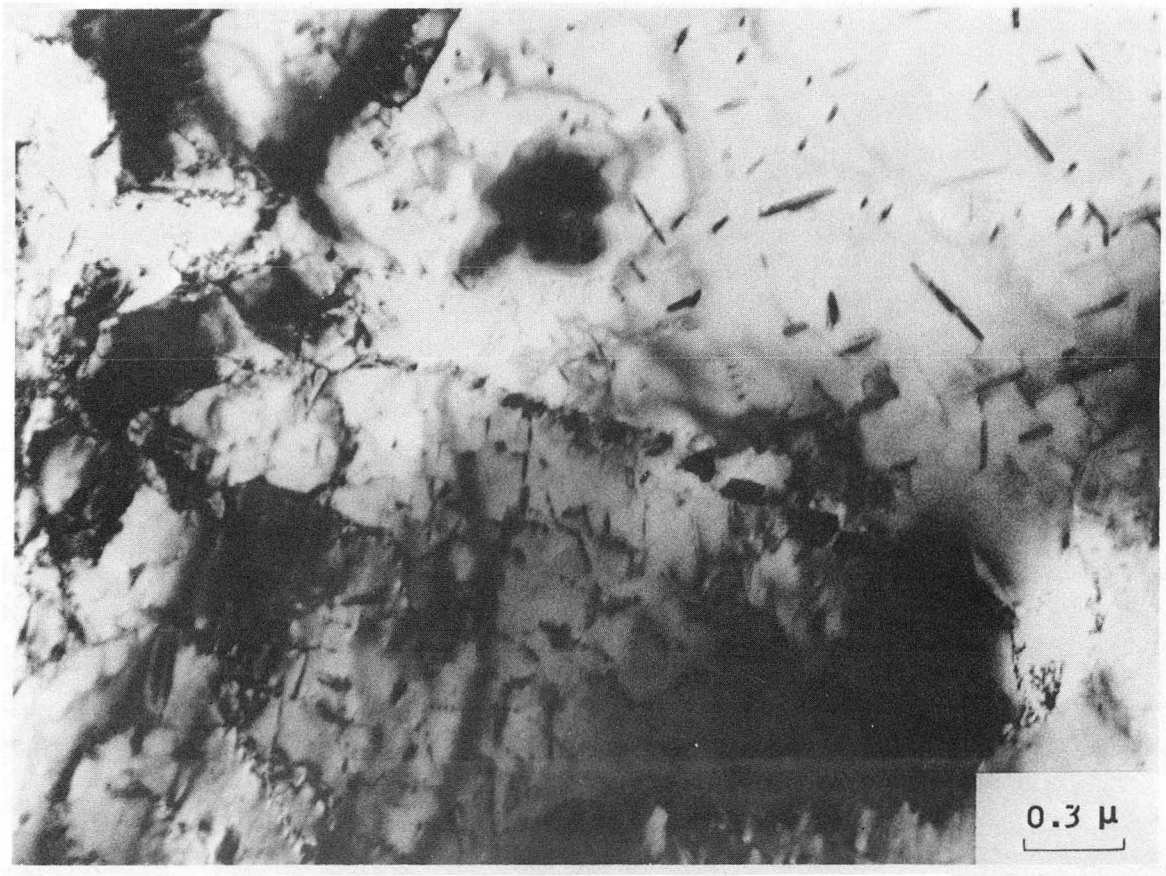

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Fig. 2b

0 0 0 0 4 3 0 2 3 0 4

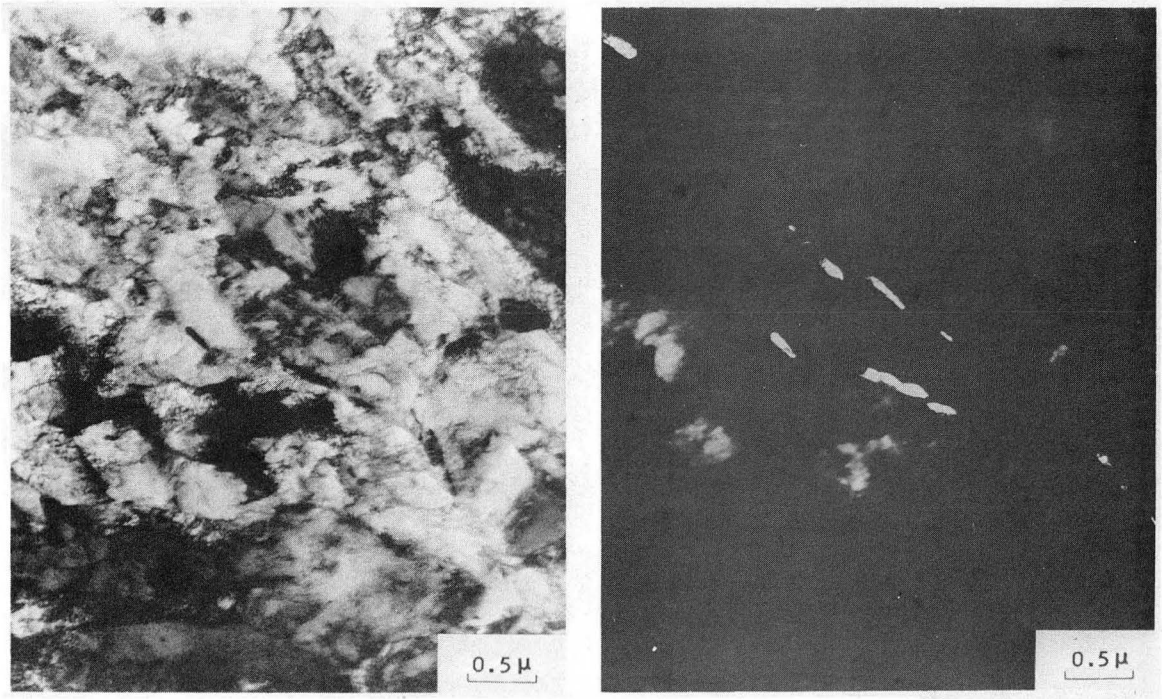

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Fig. 3a

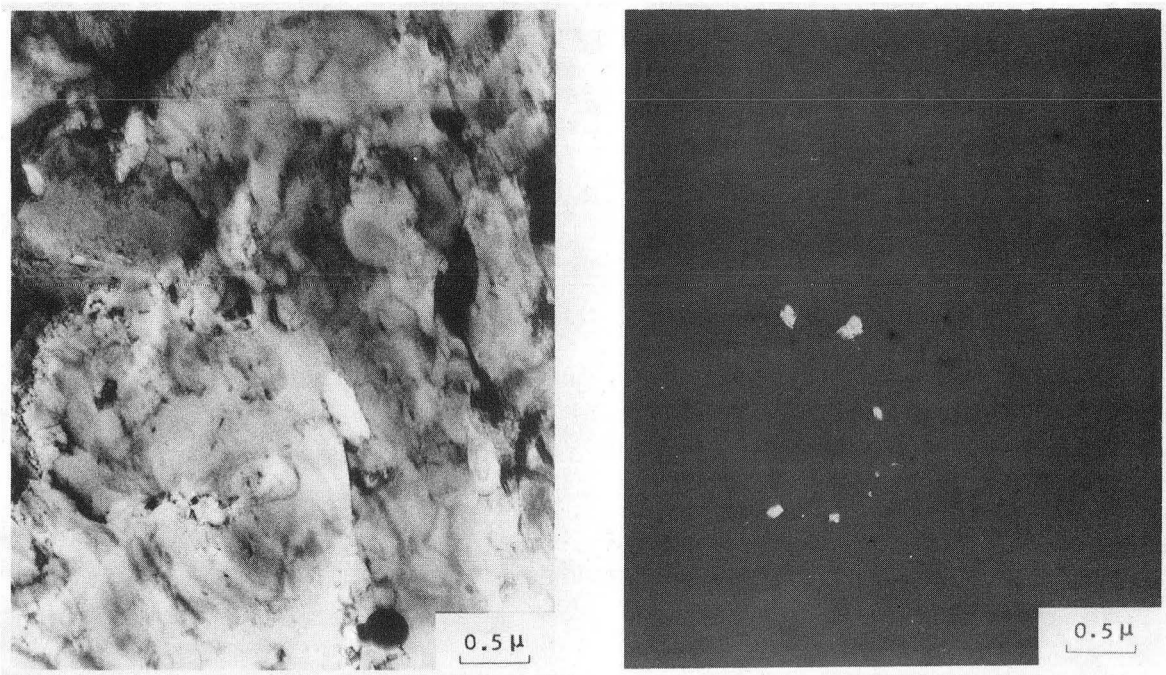

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Fig. 3b

0 0 0 0 4 3 0 2 3 0 5

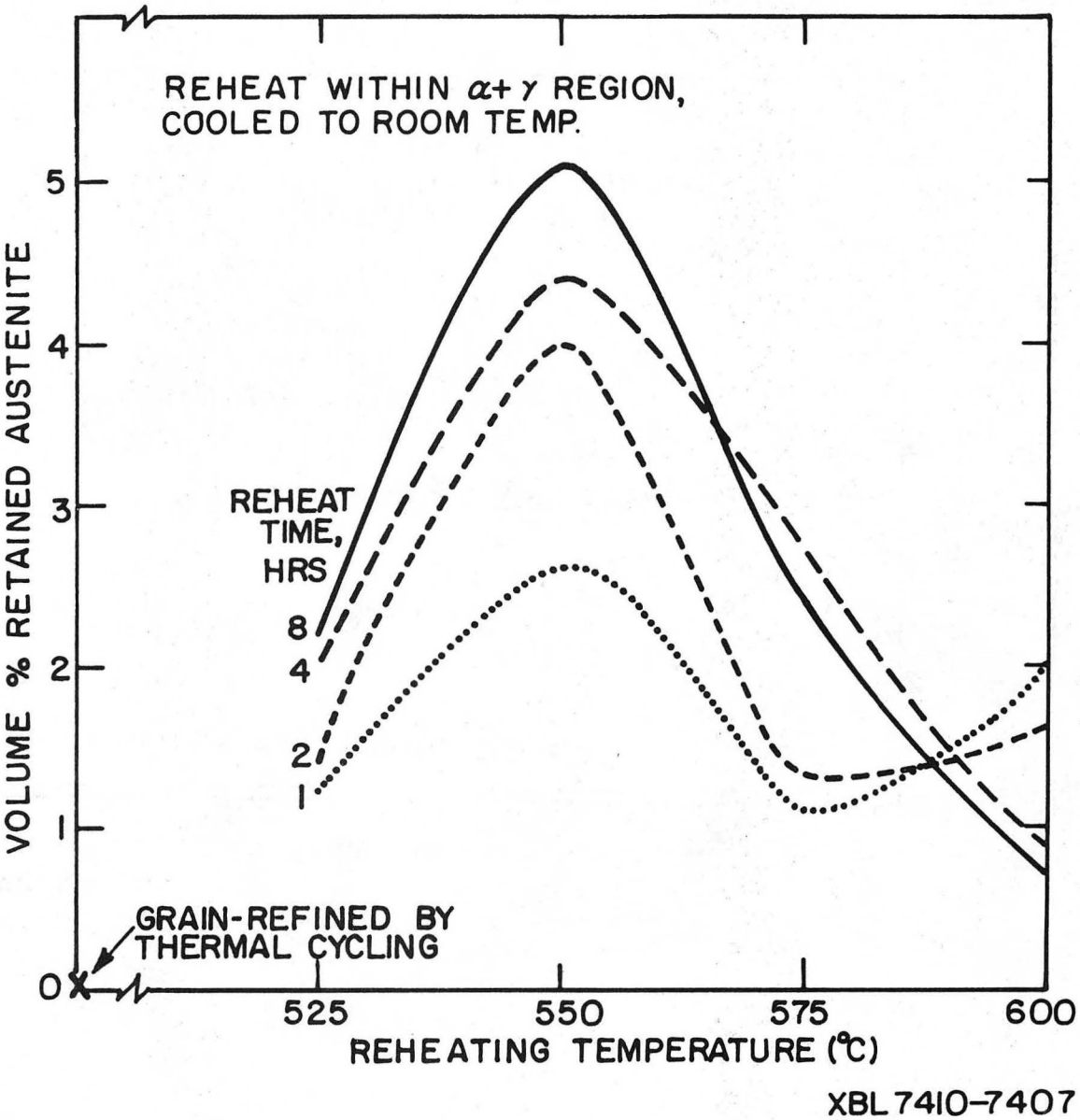

Fig. 4

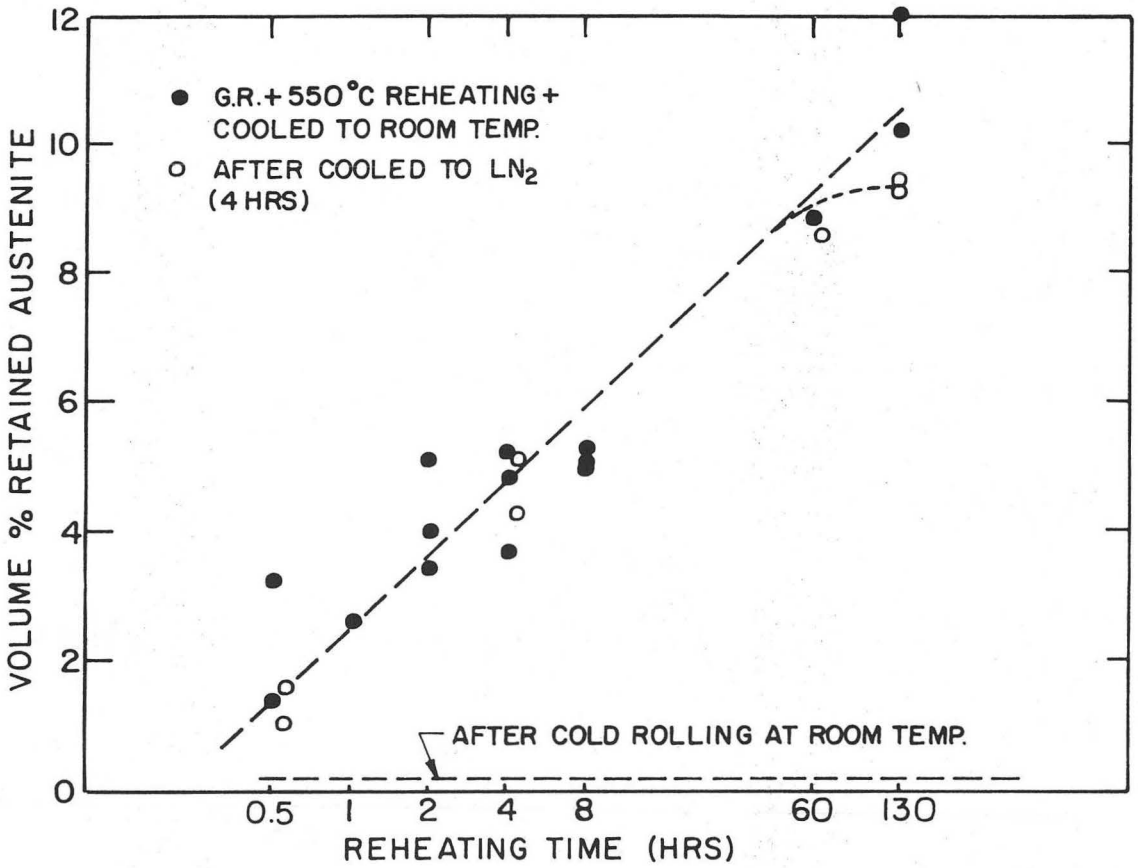

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Fig. 5

0 0 0 0 4 3 0 2 3 0 6

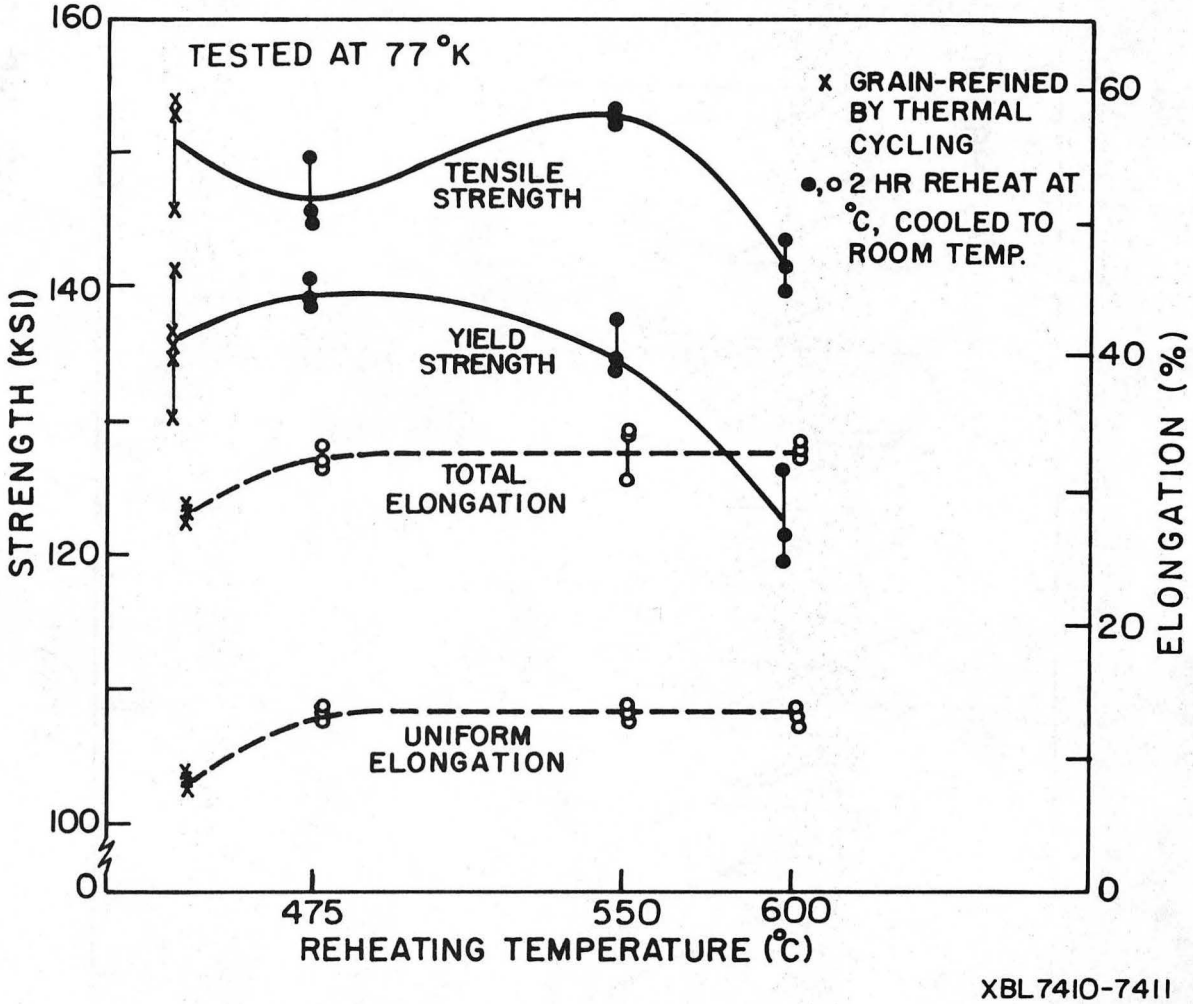

Fig. 6

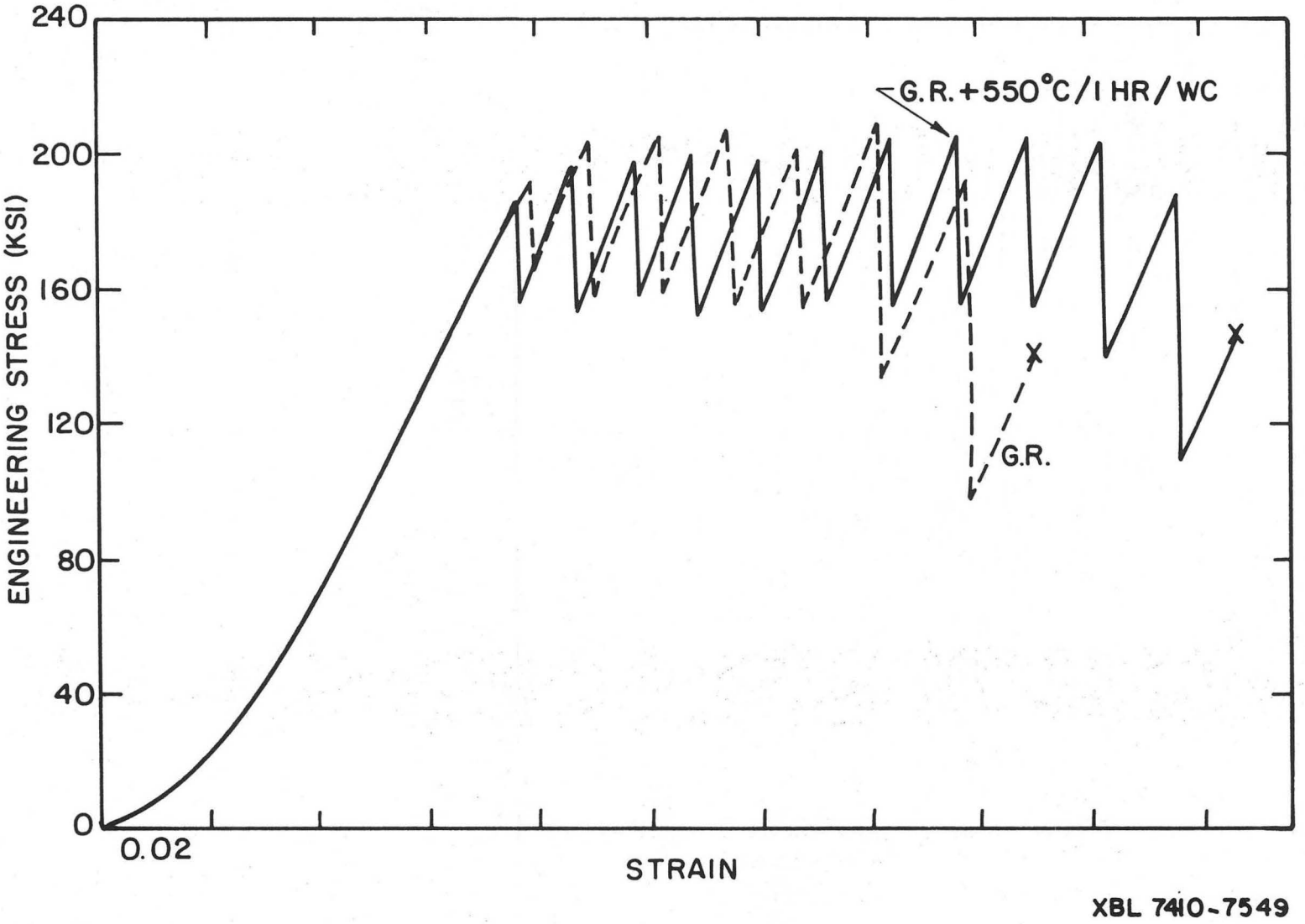
240
200
160
120
80
40
0
ENGINEERING STRESS (KSI)
0.02
STRAIN
G.R. + 550°C / 1 HR / WC
G.R.
XBL 7410-7549

Fig. 7

0 0 0 0 4 3 0 2 3 0 7

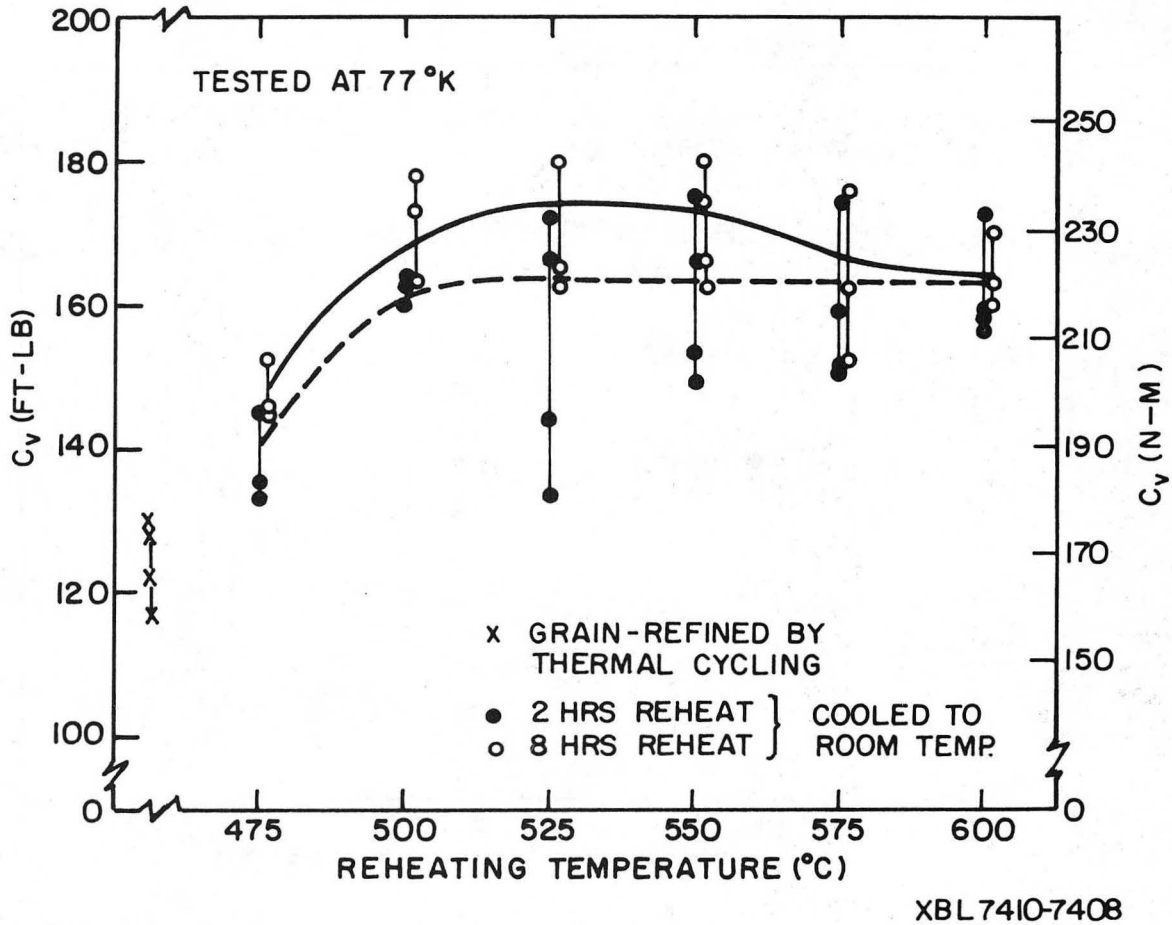

Fig. 8a

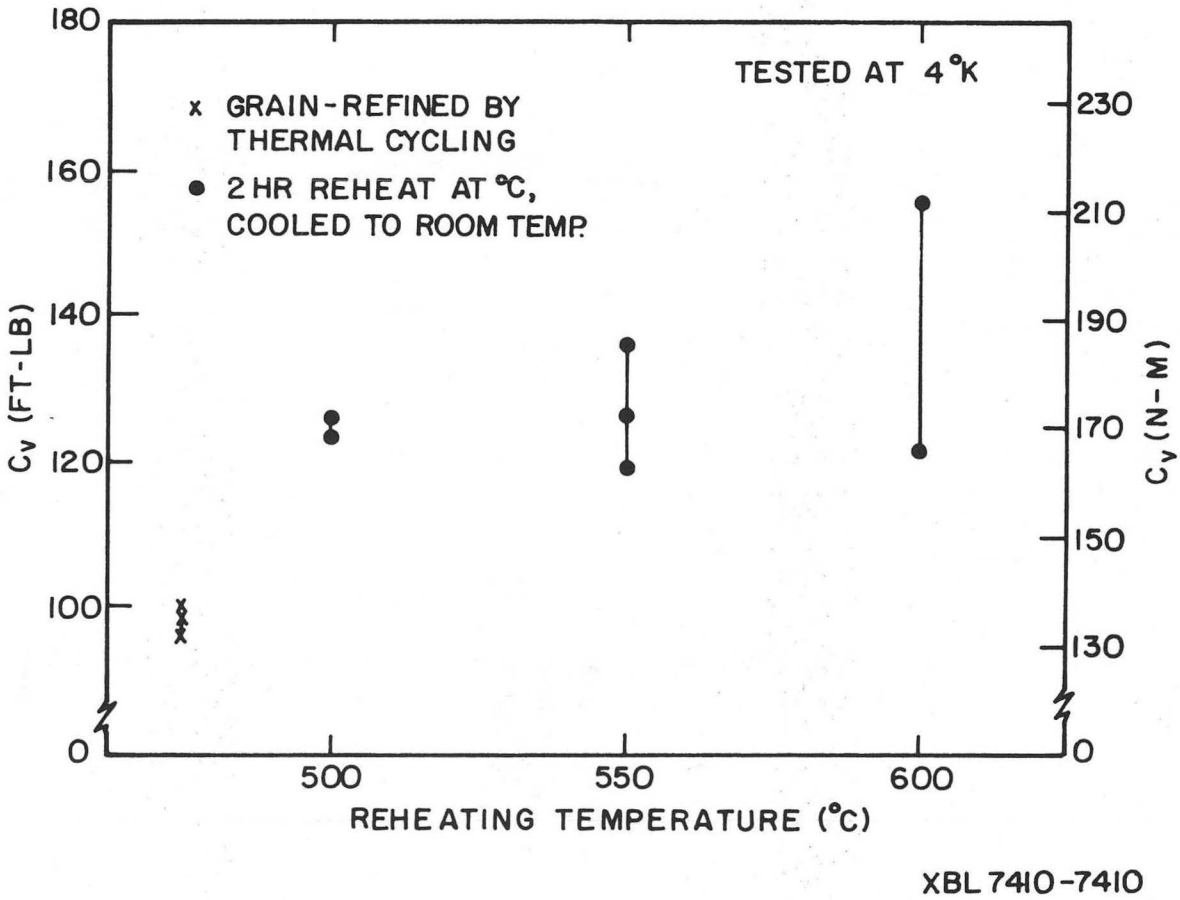
TESTED AT 4°K
x GRAIN-REFINED BY THERMAL CYCLING
● 2 HR REHEAT AT °C, COOLED TO ROOM TEMP.
180
160
140
120
100
0
C_v (FT-LB)
230
210
190
170
150
130
0
C_v (N-M)
500
550
600
REHEATING TEMPERATURE (°C)
XBL 7410-7410

Fig. 8b

0 0 0 0 4 3 0 2 3 0 8

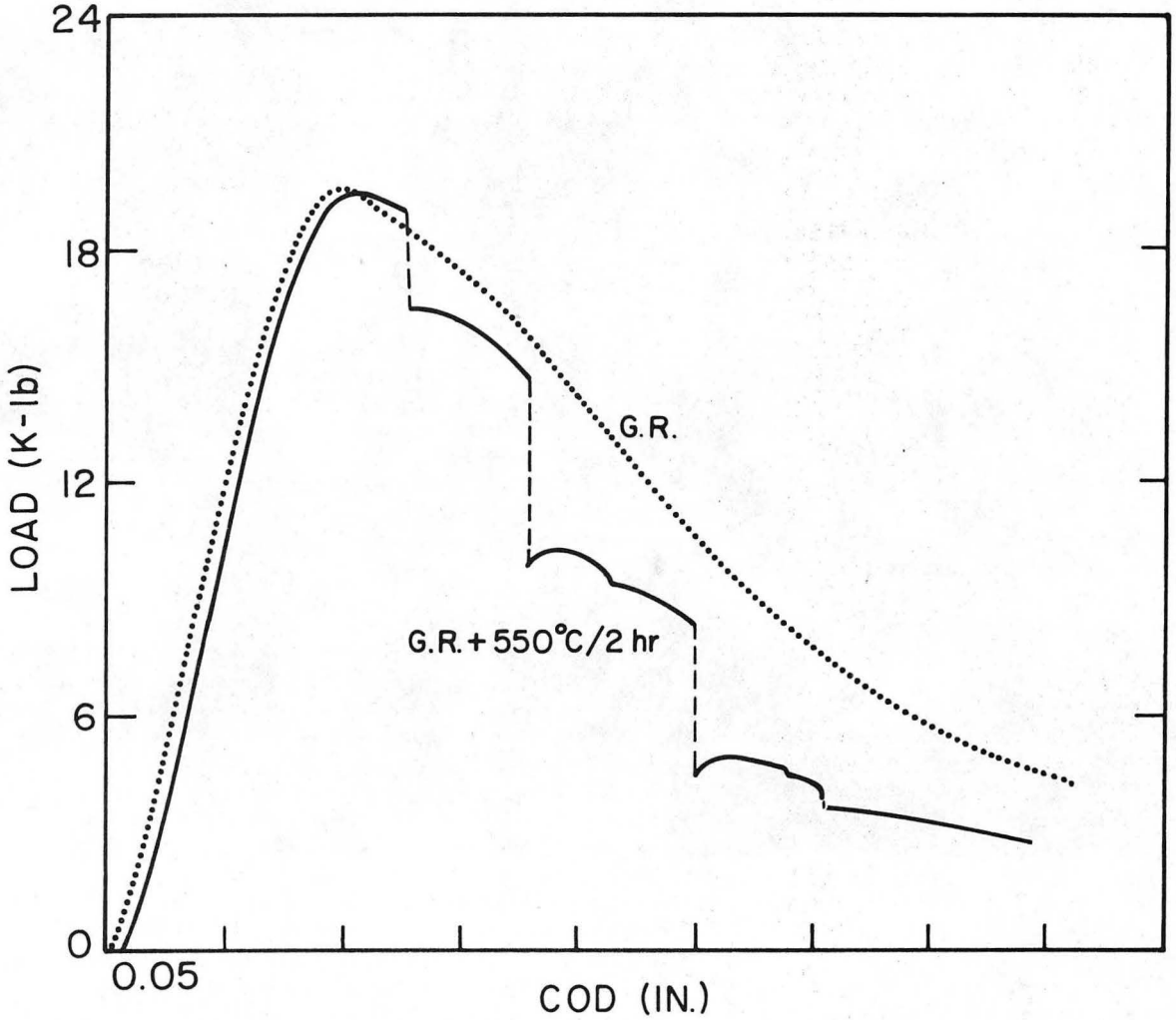

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Fig. 9

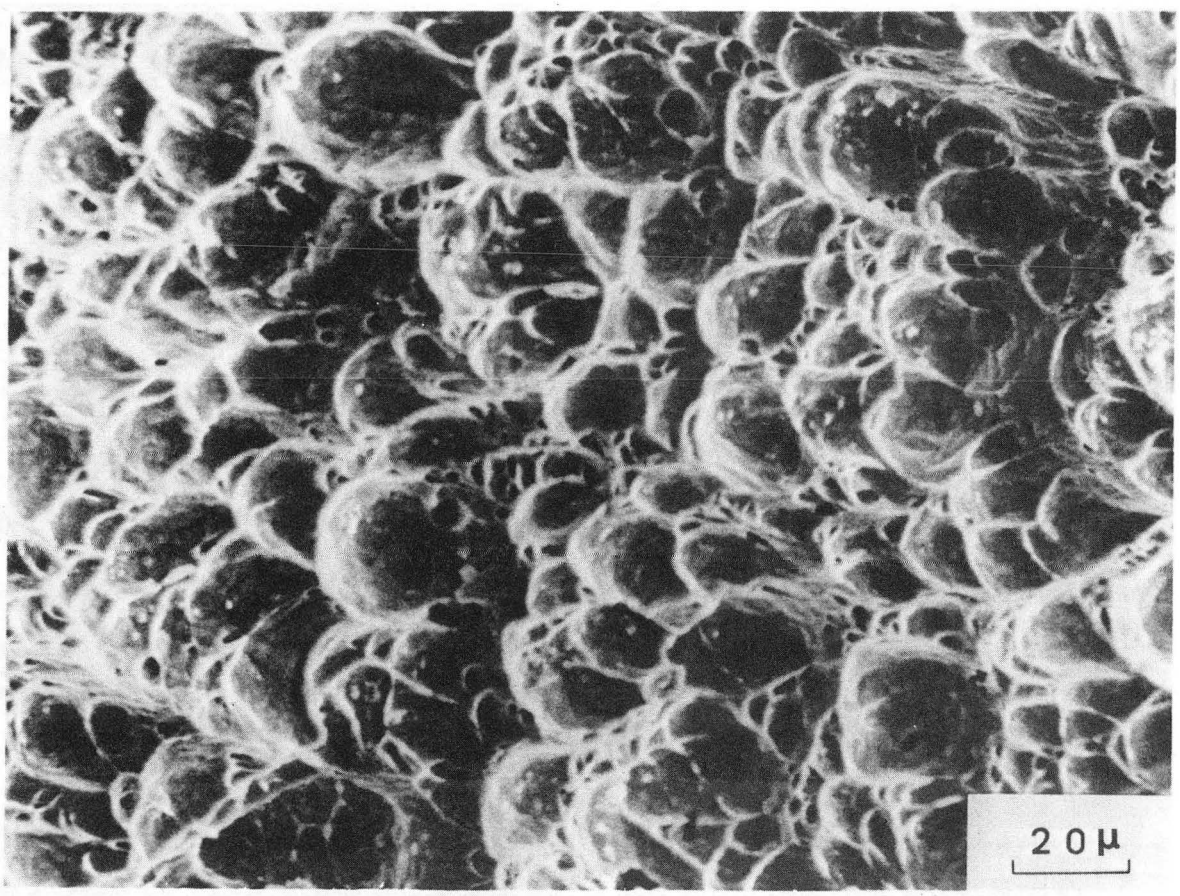

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Fig. 10

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