Title

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ABSTRACT

The effect of microstructural changes on the ductile-brittle transition temperature (DBTT) was studied in a titanium-gettered Fe-8Ni-2Mn-0.15Ti alloy. A fairly strong grain size dependence of the transition temperature, 8°C/mm$^{-1/2}$, was found. Grain size refinement from 38μm (ASTM #6.5) to 1.5μm (ASTM #15.5) through a four-step thermal treatment lowered the transition temperature by 162°C. A small amount of retained austenite was introduced into this grain-refined microstructure, and the transition temperature was reduced by an additional 120∿150°C. The reduction of the DBTT due to retained austenite was smaller when the austenite was in a large-grained structure (64°C). The distribution and stability of retained austenite were also studied.

INTRODUCTION

The effect of grain size on the ductile-brittle transition temperature (DBTT) of interstitial-free iron* base alloys has been studied by several investigators. Leslie, et al.[1] reported a grain size dependence of the DBTT of 6°C/mm$^{-1/2}$ in an Fe-Ti alloy while a slightly larger grain size dependence was observed by Bucher, et al.[2] and Gupta[3] in Fe-Ti-Al alloys. Recently Jin, et al.[4,5] were able to suppress the transition temperature of a titanium-gettered ferritic Fe-12Ni alloy to below liquid helium temperature by obtaining an ultrafine grain size through thermal cycling.

The reports on the effect of retained austenite on the mechanical properties of ferritic (or martensitic) steels are somewhat contradictory. A beneficial effect of retained austenite on tensile ductility in maraging-type steels has been reported.[6,7] Rack, et al.[8] and Pampillo, et al.,[7] however, found little benefit in impact toughness from retained austenite. In 9-Ni steel[9] and 6-Ni steel,[10] the supression of the DBTT and the improved low-temperature toughness were attributed to the presence of retained austenite formed during tempering. Recently Hwang[11] observed a considerable increase in tensile ductility and impact toughness by introducing retained austenite into a fine grained Fe-Ni-Ti cryogenic alloy, but little improvement was found in fracture toughness.

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*The term "interstitial-free iron" is commonly used in reference to titanium-gettered or aluminum- and titanium-gettered iron-base alloys. It does not represent an alloy which is completely free of interstitial species.

In many of the previous studies the accompanying effect of reduced strength during the formation of retained austenite has been neglected. This paper examines the influence of grain size and retained austenite at similar strength levels on the tensile ductility and the ductile-brittle transition of a titanium-gettered Fe-8Ni-2Mn-0.15Ti alloy.

MATERIALS AND EXPERIMENTAL PROCEDURE

Two twenty-pound ingots were prepared by induction melting under argon gas atmosphere. The compositions are shown in Table I. The ingots were homogenized under vacuum at 1200°C for 24 hours and furnace cooled, and then upset cross-forged at 1100°C to 10 cm wide by 1.3 cm thick plates. These plates were annealed at 900°C for 1 hour under argon gas to remove prior deformation strain and air cooled. This annealed material has a grain size of 38μm diameter and is designated as AN.

Table I. Chemical Composition (wt. %)

	Fe	Ni	Mn	Ti	C	N	S	P	O
Ingot I	bal	7.99	1.93	0.14	0.004	0.002	0.005	0.001	0.010
Ingot II	bal	8.03	1.97	0.17	0.001	0.001	0.006	0.001	---

Optical metallography and transmission electron microscopy were performed by standard laboratory procedures. The amount of retained austenite was measured by conventional X-ray diffraction analysis, comparing the integrated intensity of a $(211)_{\alpha}$ peak with the mean value of $(220)_\gamma$ and $(311)_\gamma$ peaks.[12] The specimen surface was ground on emery papers and then chemically polished before the final polishing on a 1μm diamond wheel.

Tensile tests were conducted at liquid nitrogen temperature in an Instron machine using subsize round specimens of 12.7 mm gauge length and 3 mm gauge diameter at a crosshead speed of 0.05 cm/min. Impact tests were carried out with standard Charpy V-notch specimens at various temperatures in accordance with ASTM specifications.[13] Hot water, a mixture of isopentane alcohol and liquid nitrogen, and liquid nitrogen were used to obtain various temperatures. Specimens were immersed in each liquid bath for at least 15 minutes and the accuracy of the temperatures was maintained within ±2°C. Tests at liquid helium temperature (-267°C) were performed using the "Lucite box technique."[14] At least three specimens were tested to obtain each datum in tensile and impact tests.

RESULTS AND DISCUSSIONS

(a) Phase Transformation and Microstructures

The phase transformation of the Fe-8Ni-2Mn-0.15Ti alloy were studied by dilatometric analysis for rough estimation of proper heat treating temperatures of grain refinement.[4] Transformation temperatures at a heating and cooling rate of ∿13°C/min. were determined as follows: $A_s \approx 697°C$, $A_f \approx 748°C$, $M_s \approx 468°C$ and $M_f \approx 396°C$. After simple austenization and cooling to room temperature, the substructure of this alloy was quite similar to that of a typical dislocated Fe-Ni martensite,[15] as shown in Fig. 1. Grain size refinement was achieved through an alternate thermal cycling[4] in the γ range and the $(\alpha+\gamma)$ two-phase range, as shown schematically in Fig. 2. The appropriate heat treating temperature and time for each step were determined by careful dilatometric and metallographic analyses. After an annealing treatment at 900°C for 1 hour (designated as AN), the alloy was reannealed in the γ range (labelled as 1A) and then transformed isothermally in the $(\alpha+\gamma)$ two-phase range. The latter two steps were repeated and a very fine-grained microstructure (labelled 2B) was obtained, as shown in Fig. 3. After the first reannealing treatment (1A), the average grain size was reduced to 38μm to 12μm in diameter. After the treatment 2B a grain size as fine as ∿1.5μm was obtained. Additional steps of thermal cycling refine the grain size further but the effect is rather small.

Retained austenite was introduced by an additional heat treating step at either 550°C (represented by a suffix r) or 600°C (suffix R) for 2 hours followed by a water quench. Introduction of retained austenite apparently does not change the grain size very much, as can be seen in Fig. 3. The transmission electron microstructure of the grain-refined alloy (2B, 2Br and 2BR) consists predominantly of equiaxed grains and subgrains with some elongated laths, as shown in Fig. 4, while a lath-like substructure is commonly observed in an annealed alloy (Fig. 1). X-ray diffraction analysis revealed no retained austenite in specimens AN, 1A and 2B, while in specimens 2Br and 2BR approximately 5 pct. of retained austenite was detected, which remained stable on cooling to liquid helium temperature (-269°C). However, on plastic deformation in a rolling mill (40% reduction in area) at liquid nitrogen temperature (-196°C), the retained austenite apparently transformed to martensite.

X-ray analysis on several different directions of the rolled specimen failed to reveal any austenite peaks. The distribution of retained austenite in the microstructure was studied by thin foil electron microscopy. Fig. 5 shows the diffraction analysis of retained austenite. A comparison of the bright field with corresponding dark field microstructure obtained from the $(200)_\gamma$ diffraction spot clealry indicates that retained austenite is located primarily at grain boundaies and martensite lath boundaries.

(b) Tensile Properties

Tensile properties of various microstructures measured at liquid nitrogen temperature (-196°C) are given in Table II. Also included in the table are the tensile properties of a large-grained specimen which contains retained austenite (labelled 1Ar: the microstructure 1A was given an additional treatment at 550°C for 2 hours). The purpose of this treatment was to separate the effect of retained austenite from that due to grain refinement. While the yield strengths varied only slightly on thermal cycling (except specimen 2BR which showed ∿6 to 13 ksi (41∿90 MPa) lower yield strength than the other microstructures), considerable changes occurred in tensile ductility. Because of grain refinement, the ductility (measured by reduction in area) improved from 45 pct. to 69 pct. The introduction of retained austenite further increased it to 73∿75 pct. A similar improvement was also observed in tensile elongation, as shown in Table II.

The microstructure 1Ar, in which approximately 3 pct. of stable retained austenite was detected in X-ray analysis, exhibited a much better tensile ductility than the microstructure 1A, as can be noticed in Table II. This indicates that retained austenite alone also improves the tensile ductility considerably without grain refinement. A yield point was observed in this titanium-gettered alloy heat treated at 550°C (specimens 2Br and 1Ar) and tested at -196°C, as shown in stress-strain curves, Fig. 6. The yield point, however, disappeared on tensile testing at room temperature (also shown in Fig. 6). These observations suggest that an insufficient thermal activation at -196°C might have required additional stress for the dislocations to tear away from a solute atom atmosphere or precipitates formed by residual titanium. This aspect requires further investigation. In Fig. 6, the progressive increase of tensile ductility and decrease of fracture stress on thermal cycling are also noticeable.

Table II. Tensile Properties at -196°C and DBTT

	Y.S.* ksi	T.S. ksi	Elong. pct.	R.A. pct.	Grain Size μm	Retained Austenite vol. pct.	DBTT** °C
AN	131	149	22.5	45	38	0	44
1A	134	151	26.6	62	12	0	-10
2B	137	157	28.5	69	1.5	0	-118
2Br	135	146	34.7	73	1.5	~5	~-236
2BR	124	148	36.0	75	1.5	~5	<-267
1Ar	130	139	33.8	72	12	~3	-74

*0.2% offset yield strength.

**Corresponding to the impact energy of 75 ft-lbs (100 NM).

†To convert to SI units, 1 ksi = 6.89 MPa.

(c) DBTT - Effect of Grain Size

The impact energy transition of the various microstructures is shown in Fig. 7. For the convenience of comparison, the ductile-brittle transition temperature was arbitrarily taken as the temperature at which the Charpy V-notch impact energy had fallen to below 75 ft-lbs (100 NM) which corresponds to near one-half of the upper shelf energy. Since the tests were conducted at about 20°C intervals, the determination of the DBTT involves an error of at least ±5°C. It is evident from Fig. 7 that the DBTT of this interstitial-free alloy was lowered considerably by reducing the grain size: Refinement of grain size from 38μm (specimen AN) to 1.5μm (specimen 2B) resulted in a suppression of the DBTT by 162°C. The DBTT of 44°C in the starting microstructure AN is substantially higher than those of other interstitial-free irons and low nickel alloys.[16-18] However, our Fe-8Ni-2Mn-0.15Ti alloy has a dislocated martensite substructure and shows a much higher strength than these alloys. The room temperature yield strength of our alloy is 70∿75 ksi (482∿517 MPa) compared with 10 ksi (69 MPa) in Fe-0.15Ti alloy and 30 ksi (207 MPa) in Fe-3Ni-0.15Ti alloy[16] at similar grain sizes and strain rates. The relatively high oxygen content in the present alloy (Table I) could also be partially responsible for the high DBTT. The grain size dependence of the DBTT in carbon-containing steels is well known.[19,20] In interstitial-free Fe alloys, however, there has been a slight difference in reported values of grain size dependence and their interpretation. The data obtained in this work are plotted together with those of other investigators[1,2,3] in Fig. 8. A fairly strong grain size dependence , 8°C/mm$^{-1/2}$, was obtained. As the dislocation theory of the ductile-brittle transition[21,22] suggests, the DBTT is also a function of surface energy (γ), the resistance to propagation of slip across grain boundaries (K_y), shear modulus (G), etc. These variables may depend on alloy composition, test temperature, and structural changes due to heat treatments depending on the alloy system. While there appears to be no reason for the grain size dependence of DBTT to be the same for the interstitial-free iron alloys with different base composition and structure, it is interesting to note that the DBTT in our interstitial-free Fe-8Ni-2Mn alloy shows roughly similar grain size dependence as the other alloys in previous research (Fig. 8). In the region of larger grain size (between the data point AN and 1A in Fig. 8), the slope of the curve approaches those of Bucher[2] and Gupta[3] in Fe-Ti-Al alloys. However, considering the experimental inaccuracy frequently involved in measuring the transition temperature or grain size, it seems to be inappropriate to draw any decisive conclusion from the limited data.

(d) DBTT - Effect of Retained Austenite

Approximately 5 pct. of retained austenite introduced to a grain-refined alloy suppressed the DBTT by 105°C, as can be seen by comparing the transition curves for microstructure 2B with that for 2Br (550°C treatment) in Fig. 7. These two microstructures exhibit similar yield strengths although there is a slight difference in the degree of work hardening (Table II). Microstructure 2BR (600°C treatment) showed a much lower DBTT than that for 2Br. In fact the ductile-brittle transition did not occur at least down to liquid helium temperature. While there could be some metallurgical factors involved, the effect of reduced yield strength of microstructure 2BR, 124 ksi (854 MPa) at -196°C, compared with that for 2Br, 135 ksi (930 MPa) at the same temperature, should not be neglected in explaining the lower DBTT in 2BR.

Leslie, et al.[23] observed an increased stability of retained austenite in a fine-grained alloy. The fine grain size of specimen 2B might have affected the amount and stability of retained austenite introduced in the subsequent reheating step. Also, the structure 2B contains an inhomogeneous distribution of alloying elements because of decomposition in the $\alpha+\gamma$ range, which could have aided the formation of retained austenite. To determine the net effect of retained austenite, a large-grained structure (1A) was heat treated at 550°C for 2 hours to produce austenite (structure 1Ar). Approximately 3 pct. of retained austenite (stable to -196°C) was detected in 1Ar compared with 5 pct. in 2Br and 2BR. It is obvious that grain-refining affects the stability or amount of retained austenite in the subsequent process. The optical microstructure of the specimen 1Ar is shown in Fig. 9. In Fig. 10, the effect of retained austenite (1Ar) on the ductile-brittle transition temperature of a large-grained alloy (1A) is shown. These two microstructures had similar yield strengths (Table II). The suppression of the DBTT, because of the introduction of retained austenite into a large-grained alloy, was smaller (by 64°C from 1A to 1Ar) than that in the grain-refined alloy (by 118°C from 2B to 2Br). This may arise from the differences in the amount and stability of retained austenite in the two microstructures and the possibly different response of retained austenite to deformation at various temperatures.

There have been several speculations on the role of retained austenite in improving ductility and toughness. It has been suggested that the retained austenite may serve as a sink for deleterious elements present in the matrix[9,24] that retained austenite may have a shock-absorbing[10,24] or crack-blunting effect[7,10,24] and that retained austenite improves the ductility of the matrix by transforming to martensite on deformation[25] (the TRIP mechanism). However, the exact mechanism is still unknown. The role of retained austenite may differ from one alloy system to another, and the amount and stability of it varies widely from system to system. Other reactions, i.e. tempering, overaging of precipitates, temper brittleness, etc., which affect the mechanical properties significantly, occur at the same time as the formation of retained austenite. Furthermore, the effect of retained austenite may be contradictory in different mechanical tests of the same material.[11] Clearly more research is needed to clarify the role of retained austenite.

CONCLUSIONS

1) A fairly strong grain size dependence of the ductile-brittle transition temperature (8°C/mm$^{-1/2}$) was observed in a titanium-gettered Fe-8Ni-2Mn-0.15Ti alloy. The refinement of grain size through a four-step thermal cycling from 38μm to 1.5μm lowered the DBTT by 162°C.

2) The introduction of a small amount of stable retained austenite into a grain-refined alloy lowered the DBTT by an additional 120∿150°C. This effect of retained austenite was more pronounced when it was introduced into a grain-refined alloy than into a large-grained alloy.

ACKNOWLEDGMENT

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FIGURE CAPTIONS

Fig. 1 - Lath-like dislocated martensite in an annealed Fe-8Ni-2Mn-0.15Ti alloy (specimen AN). Thin foil.

Fig. 2 - Thermal Cycling Procedures of grain refinement and introduction of retained austenite.

Fig. 3 - Optical micrographs. Nital etch.

Fig. 4 - Transmission electron micrograph of specimen 2Br.

Fig. 5 - Retained austenite in a specimen heat treated at 600°C after grain refinement. (a) Bright field, (b) Dark field electron micrograph taken from $(200)_{\gamma}$ diffraction spot.

Fig. 6 - Engineering stress-strain curves obtained in tensile tests.

Fig. 7 - Effect of microstructural changes on the impact energy transition in an Fe-8Ni-2Mn-0.15Ti alloy.

Fig. 8 - Grain size dependence of the ductile-brittle transition temperature in interstitial-free iron-base alloys.

Fig. 9 - Optical micrograph of the specimen 1Ar. Nital etch.

Fig. 10 - Decrease of the impact transition temperature by retained austenite in a large grained alloy.

(XBB 751-177)

Fig. 1.

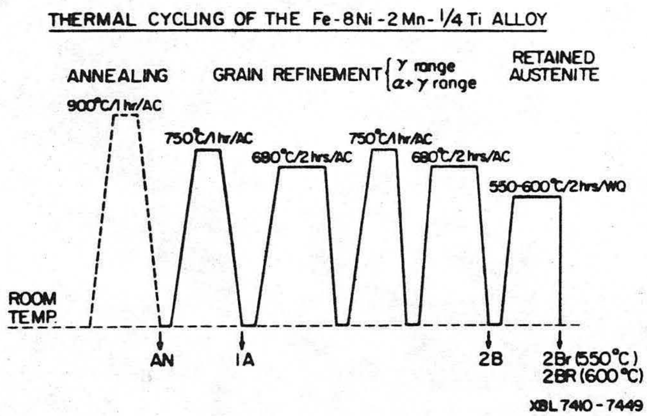
THERMAL CYCLING OF THE Fe-8Ni-2Mn-1/4Ti ALLOY
ANNEALING
GRAIN REFINEMENT {γ range / α+γ range
RETAINED AUSTENITE
900°C/1 hr/AC
750°C/1 hr/AC
680°C/2 hrs/AC
750°C/1 hr/AC
680°C/2 hrs/AC
550-600°C/2 hrs/WQ
ROOM TEMP.
AN
1A
2B
2Br (550°C)
2BR (600°C)
XBL 7410-7449

Fig. 2.

0 0 0 0 4 2 0 8 2 1 5

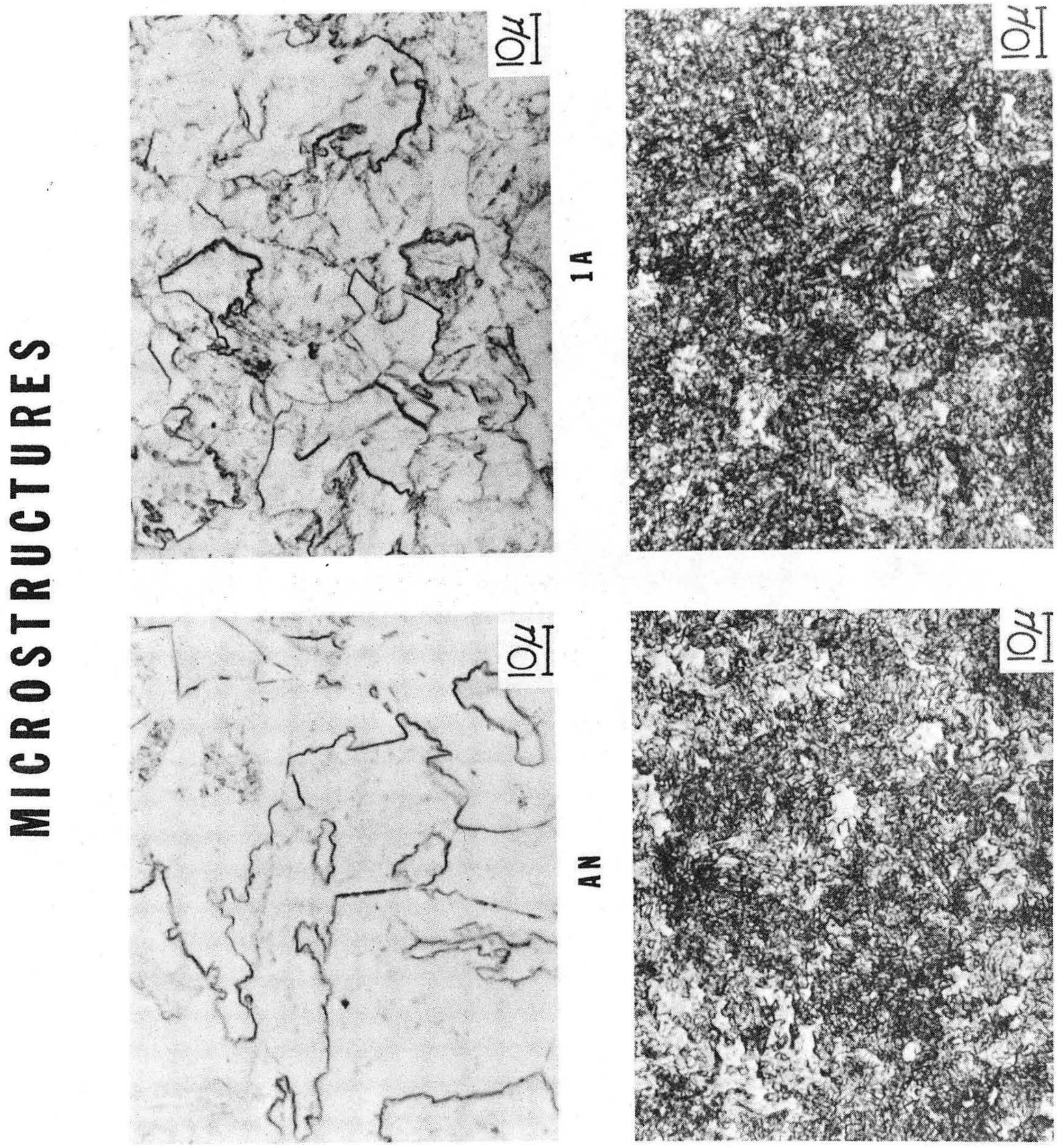

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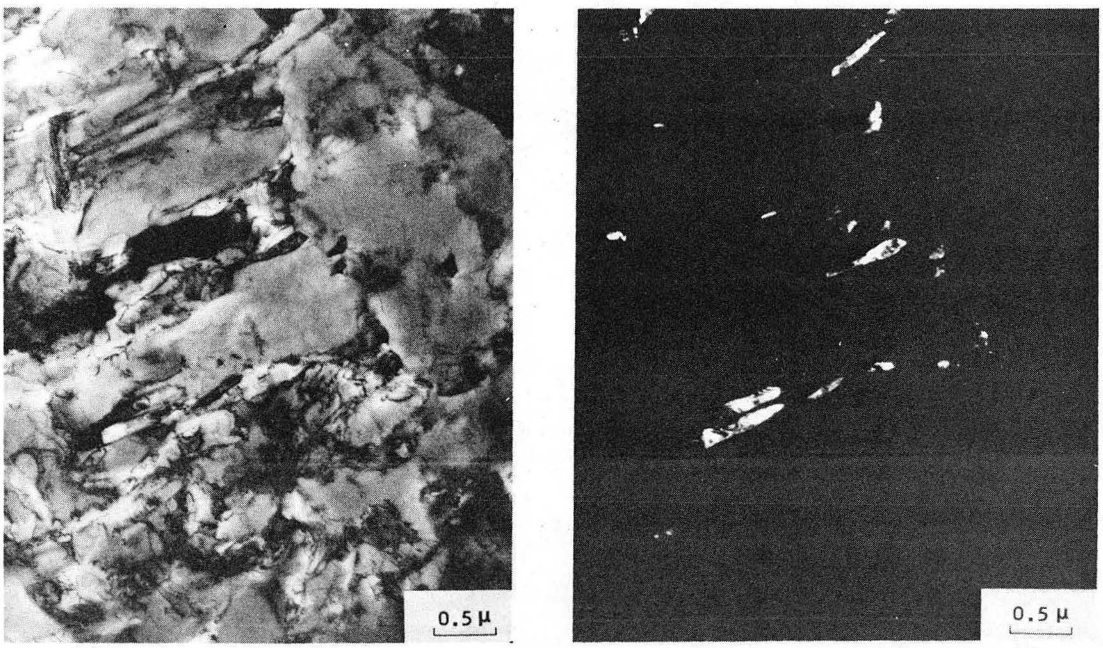
Fig. 3.

(XBB-751-176)

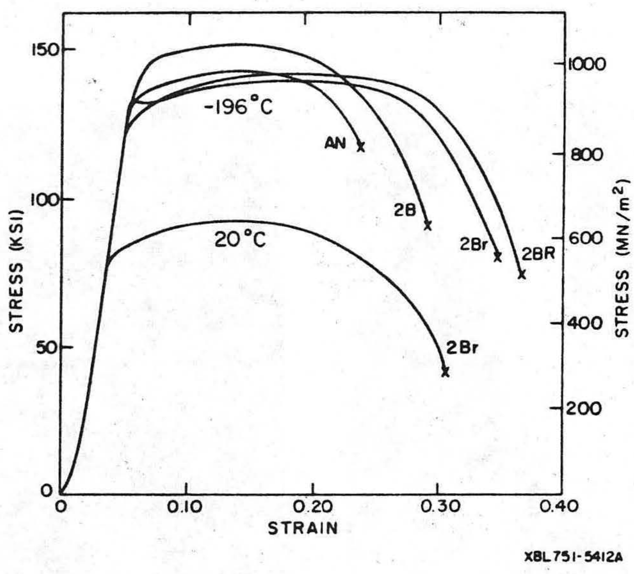
Fig. 4.



(a) (b)

(XBB 7410-7592)

Fig. 5.


150
100
50
0
STRESS (KSI)
1000
800
600
400
200
STRESS (MN/m²)
-196°C
AN
2B
2Br
2BR
20°C
2Br
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0.10
0.20
0.30
0.40
STRAIN
XBL751-5412A

Fig. 6.

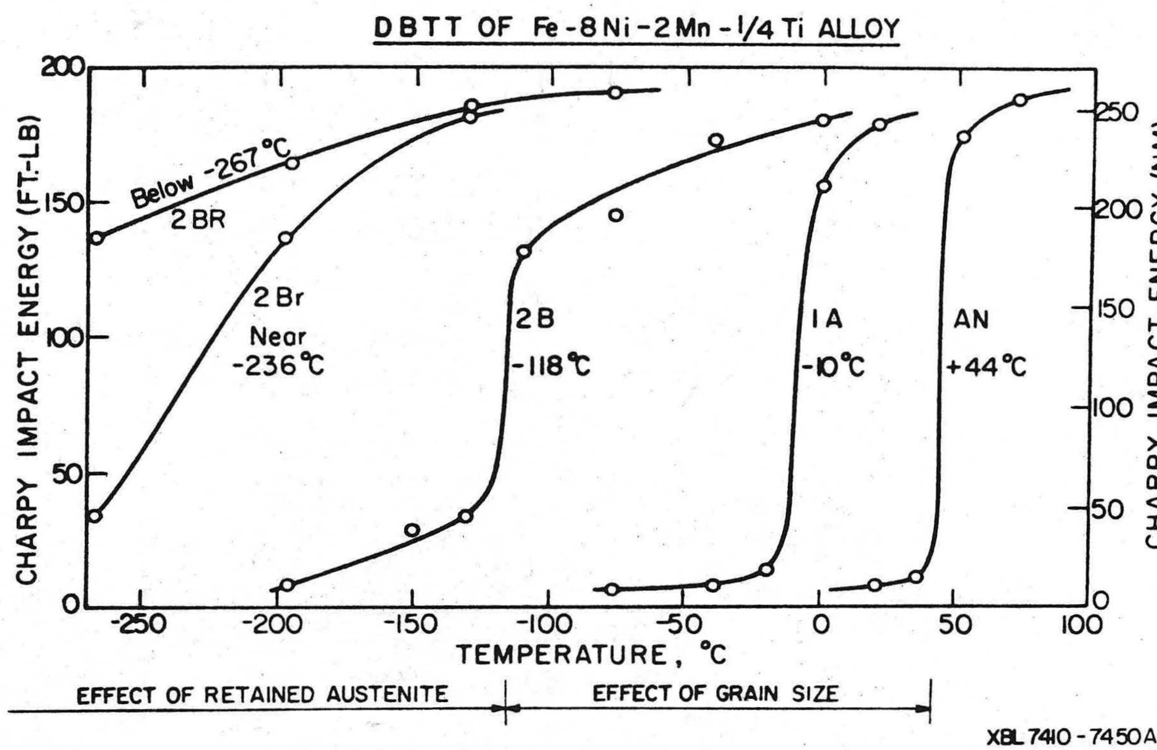

DBTT OF Fe-8Ni-2Mn-1/4 Ti ALLOY
CHARPY IMPACT ENERGY (FT.-LB)
CHARPY IMPACT ENERGY (NM)
TEMPERATURE, °C
Below -267 °C
2 BR
2 Br
Near
-236 °C
2 B
-118 °C
1 A
-10 °C
AN
+44 °C
EFFECT OF RETAINED AUSTENITE
EFFECT OF GRAIN SIZE
XBL 7410-7450A

Fig. 7.

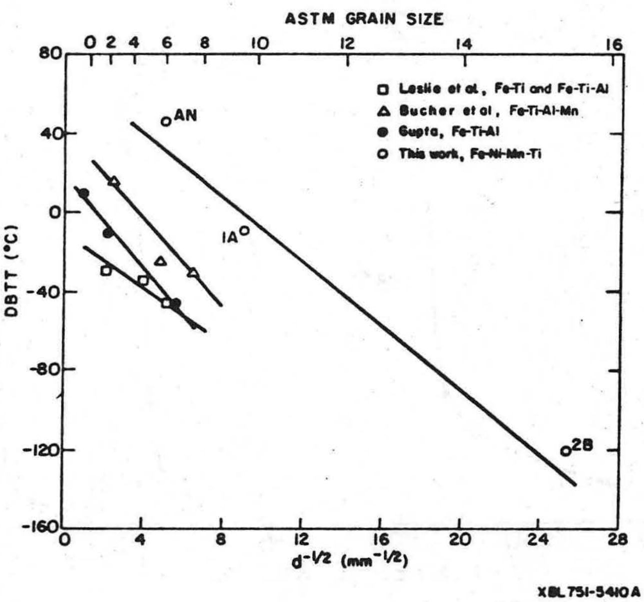
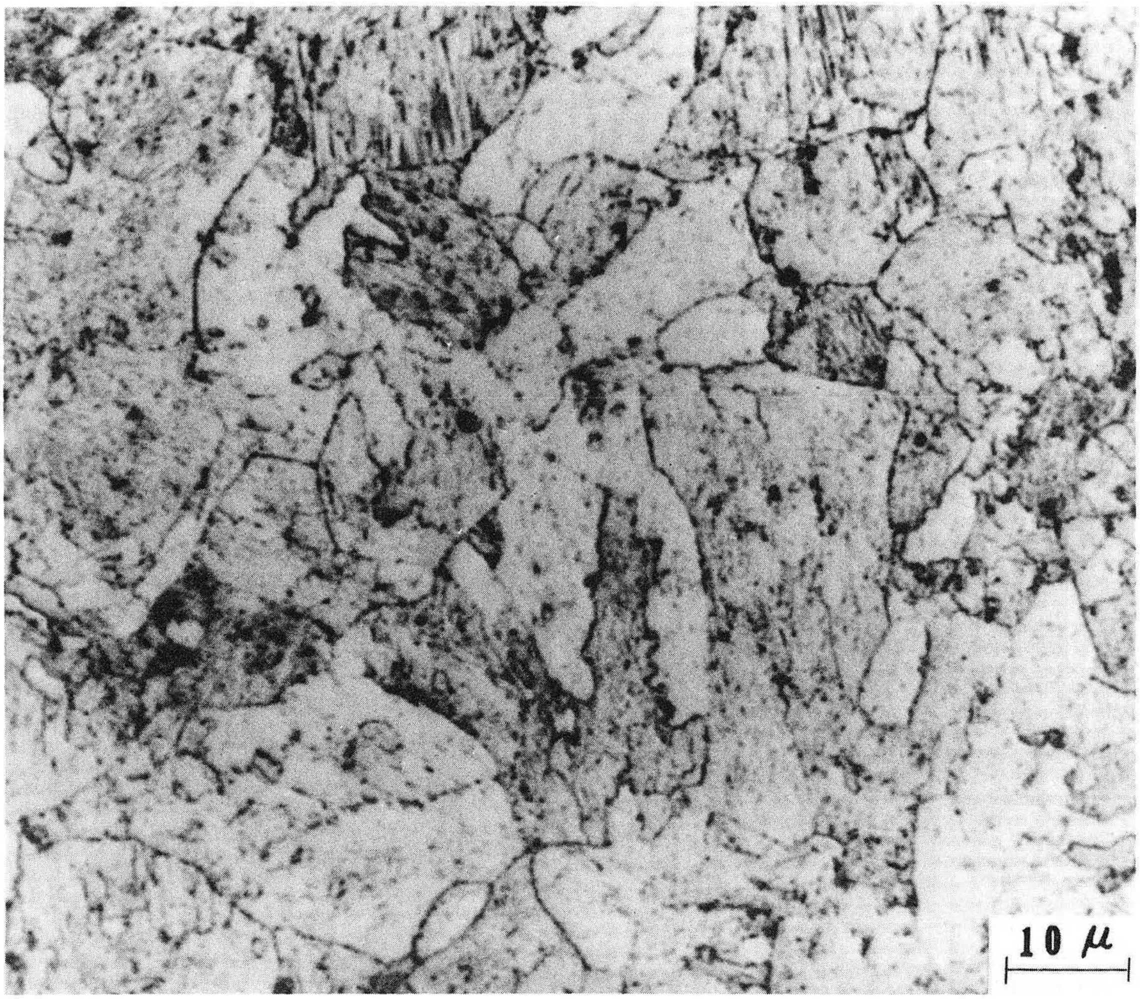

ASTM GRAIN SIZE
0 2 4 6 8 10 12 14 16
Leslie et al, Fe-Ti and Fe-Ti-Al
Bucher et al, Fe-Ti-Al-Mn
Gupta, Fe-Ti-Al
This work, Fe-Ni-Mn-Ti
AN
1A
2B
DBTT (°C)
80
40
0
-40
-80
-120
-160
0 4 8 12 16 20 24 28
$d^{-1/2}$ ($mm^{-1/2}$)
XBL751-5410A

Fig. 8.



(XBB 751-175)

Fig. 9

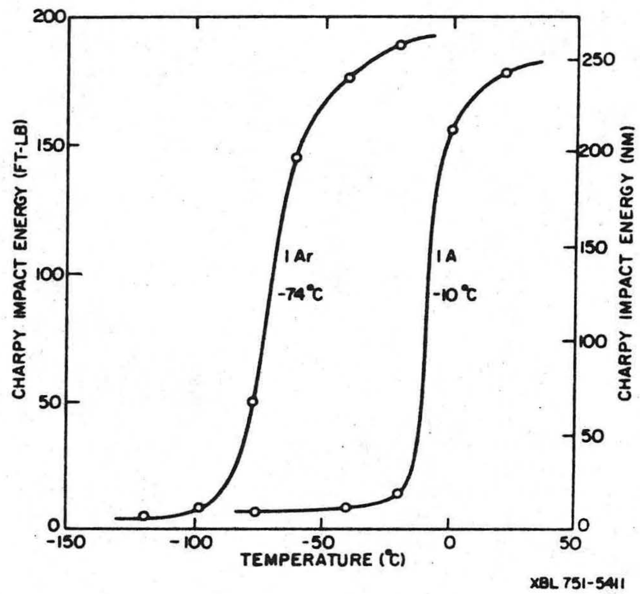

200
150
100
50
0
-150
-100
-50
0
50
TEMPERATURE (°C)
CHARPY IMPACT ENERGY (FT-LB)
CHARPY IMPACT ENERGY (NM)
250
200
150
100
50
0
1 Ar
-74°C
1 A
-10°C
XBL 751-5411

Fig. 10.

0 0 0 0 4 2 0 8 2 1 9

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