EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE AND FRACTURE TOUGHNESS OF A V-5Cr-5Ti ALLOY - H. Li (Associated Western Universities--Northwest Division), M. L. Hamilton and R. H. Jones (Pacific Northwest Laboratory)

OBJECTIVE

The purpose of this research is to investigate the effect of heat treatment on microstructure and fracture toughness in the range of -50 to 100°C for a V-5Cr-5Ti alloy.

SUMMARY

Fracture toughness and impact tests were performed on a V-5Cr-5Ti alloy. Specimens annealed at 1125°C for 1 h and furnace cooled in a vacuum of 1.33×10^{-5} Pa were brittle at room temperature (RT) and experienced a mixture of intergranular and cleavage fracture. Fracture toughness (J_{IQ}) at RT was 52 kJ/m^2 and the impact fracture energy (IFE) was 6 J. The IFE at -100°C was only 1 J. While specimens exhibited high fracture toughness at 100°C (J_{IQ} is 485 kJ/m^2), fracture was a mixture of dimple and intergranular failure, with intergranular fracture making up 40% of the total fracture surface. The ductile to brittle transition temperature (DBTT) was estimated to be about 20°C. When some specimens were given an additional annealing at 890°C for 24 h, they became very ductile at RT and fractured by microvoid coalescence. The J_{IQ} value increased from 52 kJ/m^2 to ~1100 kJ/m^2. The impact test failed to fracture specimens at RT due to a large amount of plastic deformation. The IFE at -115°C was 4 J, four times as much as when annealed only at 1125°C. The specimens became brittle at -50°C and fractured by cleavage, giving a J_{IQ} value of 50 kJ/m^2. The DBTT was estimated to be -40°C. Analysis of Auger electron microscopy showed significant sulfur segregation (6 at%) on grain boundaries in the specimens annealed only at 1125°C, but only 0.9 at% on grain boundaries if the additional annealing at 890°C was given. Moreover, significantly more second phase particles were found in the specimens annealed at 1125°C plus 890°C. The possible mechanism by which heat treatment affects fracture toughness is discussed.

PROGRESS AND STATUS

Introduction

We have reported[1] that a V-5Cr-5Ti alloy (ANL Heat BL-63) was brittle at RT and experienced mixed intergranular and cleavage fracture when heat treated at 1125°C for 1h and furnace cooled. While it became ductile at 100°C, it still failed by a mixture of intergranular and microvoid coalescence modes. The mixed mode I/III fracture toughness (J_{TQ}) at a crack angle of 45° (where the mode III loading component is equal to the mode I loading component) was much less than pure mode I fracture toughness, as shown in Fig. 1. The data from a ferritic/martensitic stainless steel (F82-H) is included for comparison. Specimens fractured in an Auger electron microscope (AEM) vacuum chamber at low temperature (estimated about -20°C) consisted of intergranular and cleavage fracture. AEM analysis revealed a significant enrichment of S (6 at%) on the grain boundaries, as comparing to 0.3 at% S on the cleavage facets. On the other hand, a specimen annealed at 1050° for 1h fractured in the AEM by microviod coalescence. To produce an intergranular fracture surface, hydrogen had to be introduced by cathodic charging. The result indicated that the lower annealing temperature improved the ductility of the V-5Cr-

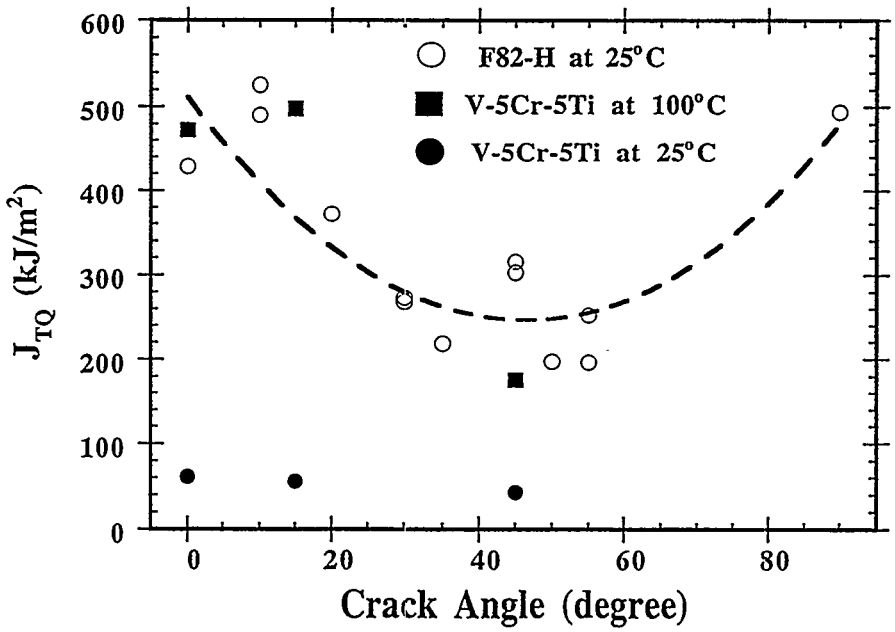


Fig. 1. The dependence of critical total J-integrals of V-5Cr-5Ti on crack inclination angles and temperatures. The results for a reduced activation ferritic/martensitic steel (F-82H) is included for comparison.

5Ti alloy. This research is an ongoing effort to understand the embrittlement observed in the V-5Cr-5Ti alloy at RT following annealing at 1125°C for 1h. The present study considers the effect of heat treatment on fracture toughness and impact fracture energy.

Material and Experimental Methods

A 6.35 mm thick V-5Cr-5Ti alloy plate (ANL Heat No. BL-63) was produced by Teledyne Wah Chang-Albany. The chemical composition of the plate (as provided by the vendor) is listed in Table 1.

Table 1. Chemical Composition of V-5Cr-5Ti (BL-63)

(in wt%)	Cr	Ti	(in wt ppm)	O	C	N	Si	V
	4.6	5.1		440	73	28	310	bal.

In order to gain more information about effect of heat treatment on grain size, precipitates, and grain boundary chemistry, the following heat treatments were investigated.

(1). 1125°C / 1 h/ furnace cooled (FC) (HT1);
(2). HT1 plus 890°C / 24 h/ FC (HT2);
(3). 1050°C / 1 h/ FC (HT3);
(4). HT3 plus 890°C / 24 h/ FC (HT4);
(5). HT3 plus 730°C / 24 h/ FC (HT5).

All of the heat treatments were conducted in a vacuum of 1.33×10^{-5} Pa. Grain boundary chemistry was analyzed by means of a scanning Auger electron spectrometer (AES) (PERKIN-ELMER-660). Specimens

were cooled down to low temperature by extracting heat with liquid nitrogen and were fractured in-situ in the AES chamber in a vacuum of 1 X 10^{-7} Pa or better. Auger spectra were taken at an accelerating voltage of 5 kV and an incident electron current of about 250 nA. At least 22 intergranular facets and 6 grain interiors were analyzed; sometimes duplicate tests were performed. Microstructure was analyzed using an optical microscope. Investigation using a scanning electron microscope (SEM) equipped with energy dispersive x-ray spectrometry (EDS) and a transmission electron microscope (TEM) is currently under way.

Based on the results from the AES and the optical metallographic analysis, only HT1 and HT2 were used to heat treat the fracture test specimens. The specimens used for fracture toughness and impact tests were cut in the T-L orientation as specified in ASTM E399-90. The geometry of compact tension specimen is shown in Fig. 2. ASTM E813-89 was used to determine critical J-integrals (J_{IQ}) and ASTM E399-90 was used to determine the critical stress intensity factor (K_{IQ}). The subscript "Q" is used because our specimen size did not totally satisfy plane strain conditions. Mechanical properties used for determination of J_{IQ} were provided by Loomis[2], which are listed in Table 2. Charpy V impact specimens of 23.6x3.33x3.33 mm (1/3 scaled) were tested in the range of -115°C to 50°C to estimate DBTT. Fracture toughness was determined at -50, 25, and 100°C. Fracture surfaces of all specimens were investigated by means of a SEM.

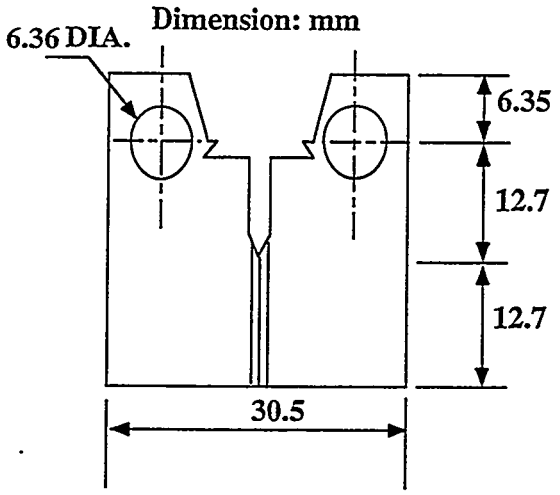


Fig. 2. The geometry of the compact tension specimen used in this study.

Table 2. Mechanical Properties of a V-5Cr-5Ti Alloy (ANL Heat No BL-63)[2]

Temp. (°C)	Yield strength (MPa)	UTS* (MPa)	Elongation (%)
25	387	454	34

* UTS: ultimate tensile strength.

Fracture toughness was determined by the J-test method for an HT1 treated specimen tested at 100°C and an HT2 treated specimen tested at RT. Fracture toughness was measured using the K-test method for an HT1 treated specimen tested at RT and for an HT2 treated specimen tested at -50°C. The K_{IQ} values were then converted to J_{IQ} values by means of Eq. 1. Temperatures were controlled within ±5°C during testing using either a heating tape or a refrigerator, respectively. The single-specimen technique was used in this study, which allows a J-R curve (J vs crack extension Δa) to be generated with one specimen. At least 40 pairs of J-Δa data were used to construct a J-R curve.

$$J_{IQ} = \frac{K_{IQ}^2 \ (1 - \nu^2)}{E} \qquad (1)$$

where E is Young's modulus and ν is Poisson ratio.

Results

Effect of Heat Treatment on Microstructure and Grain Boundary S Concentration

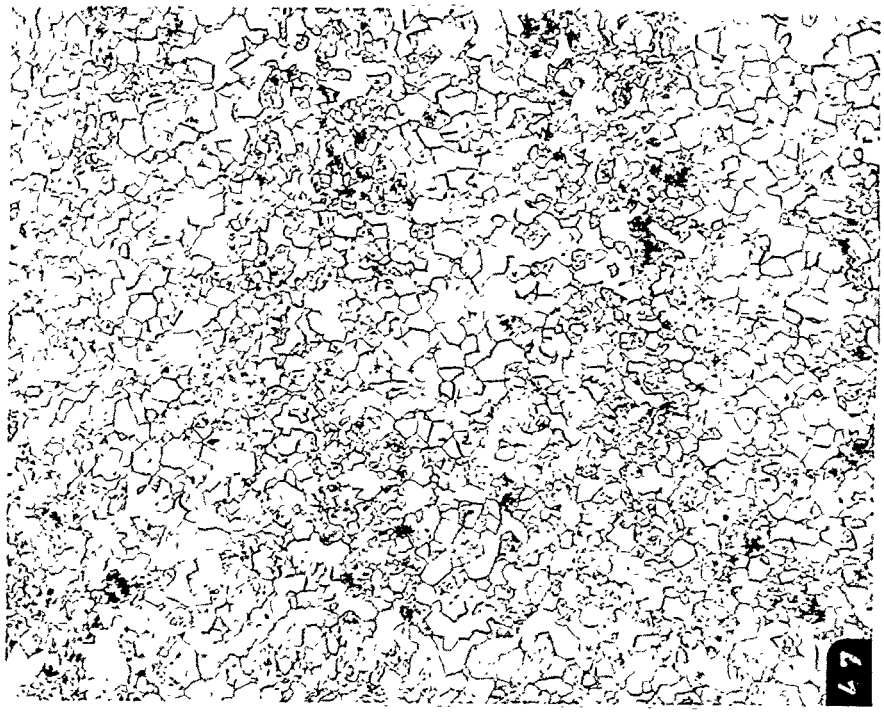
The specimens subjected to HT1 were brittle at RT and fractured with a mixture of intergranular and cleavage modes. The result indicated that the grain boundary strength was low and the interior of grains was brittle. AES analysis of more than 40 intergranular facets in two specimens showed significant enrichment of S (6 at%), as compared to 0.3 at% on the cleavage facets. The specimens treated by HT3 displayed improved ductility and fractured in the AES by microvoid coalescence. To produce intergranular facets, hydrogen had to be introduced by cathodic charging. AES analysis showed that the S concentration on grain boundary facets (more than 20 facets) was only 2 at%, three times less than that in the specimens treated by HT1. Except for the specimens treated using HT1 in order to obtain an intergranular facet, all of the specimens had to be charged with hydrogen before fracture in the AES chamber. The effect of heat treatment on grain size, grain boundary chemistry, and microstructure was summarized in Table 3. The P, O, N, and C concentrations were also investigated with AES, but their concentrations did not vary significantly with heat treatment, so only S was listed in Table 3. The microstructure produced by selected heat treatments is shown in Fig. 3. From Table 3, it is evident that HT2 gives the second lowest grain boundary S concentration, HT1 gives the highest. The HT2 treatment also produced significantly more second phase particle precipitates than HT1, as shown in Fig. 3c and 3d. However, the grain sizes were almost same in the specimens treated using either HT1 or HT2. Therefore, HT1 and HT2 were chosen to investigate the effect of grain boundary S and microstructure on fracture properties of the V-5Cr-5Ti alloy.

Table 3. Effect of Heat Treatment on Grain Boundary S Concentrations and Fracture Toughness/DBTT

Heat treatments	G.S.(μm)*	GB S	PCT	DBTT(°C)	J_{IQ} at RT	Fracture
1125°C/1h	45	6	few	20		IG, CL**
1125°C/1h	48	6	few		52 kJ/m^2	IG, CL
1125°C/890°C/24h	45	.9	many	<-40	~1100 kJ/m^2	Dimple
1050°C/1h	40	2	some			
1050°C/890°C/24h		.5				
1050°C/730°/24h		1.5				
As received	37		some			

* G.S.: grain size; GB S: grain boundary sulfur (at%); PCT: Precipitates.

** Heat treated at Oak Ridge National Lab., the temperature may have been 1150°C as indicated by the plastic bag containing the specimens; IG: intergranular; CL: cleavage.



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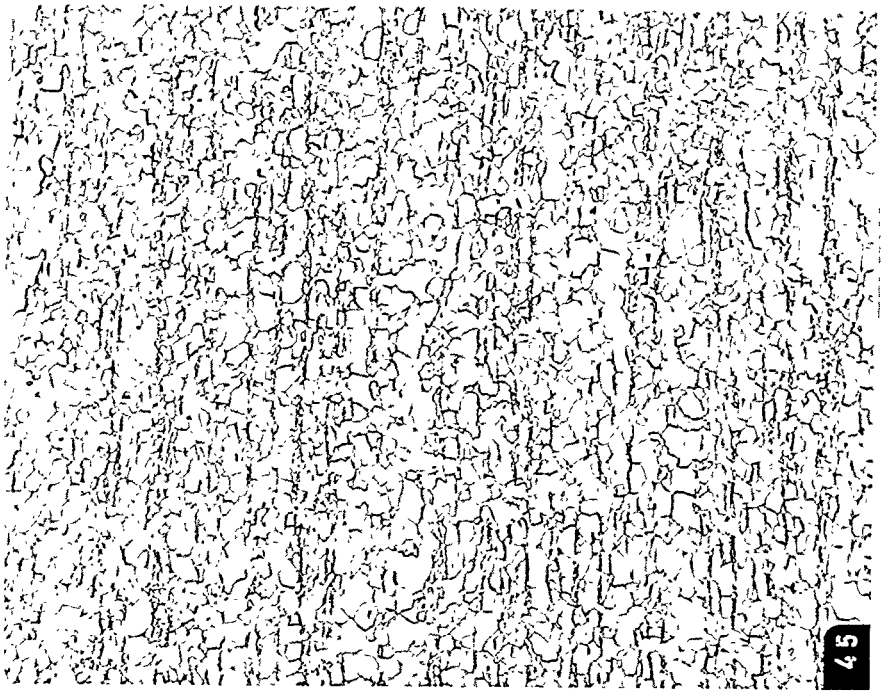
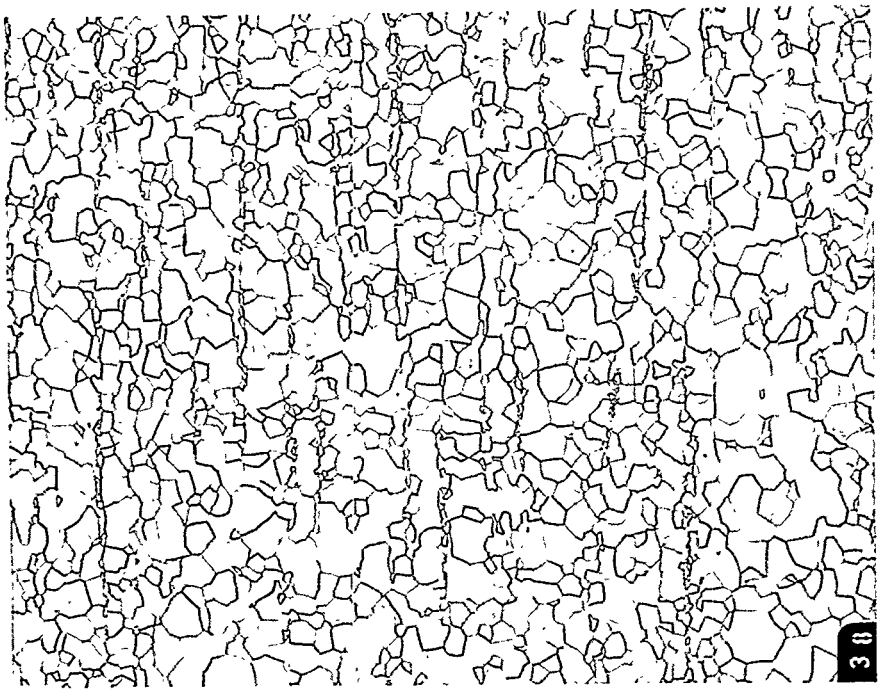
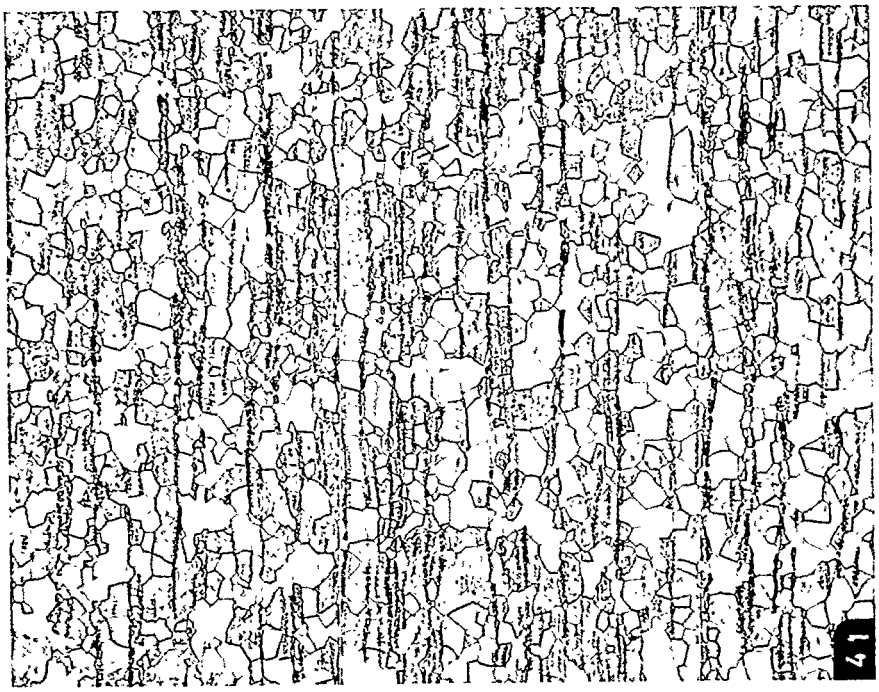


b.

Fig. 3. Overleaf.



c.



d.

Fig. 3. Optical micrographs (50 X) showing microstructures resulting from: a.) as-received; b.) 1050°C/ 1h/ furnace cooled (FC); c.) 1125°C/ 1h/ FC; and d.) 1125°C/ 1h/ FC plus 890°C/ 24h/ FC.

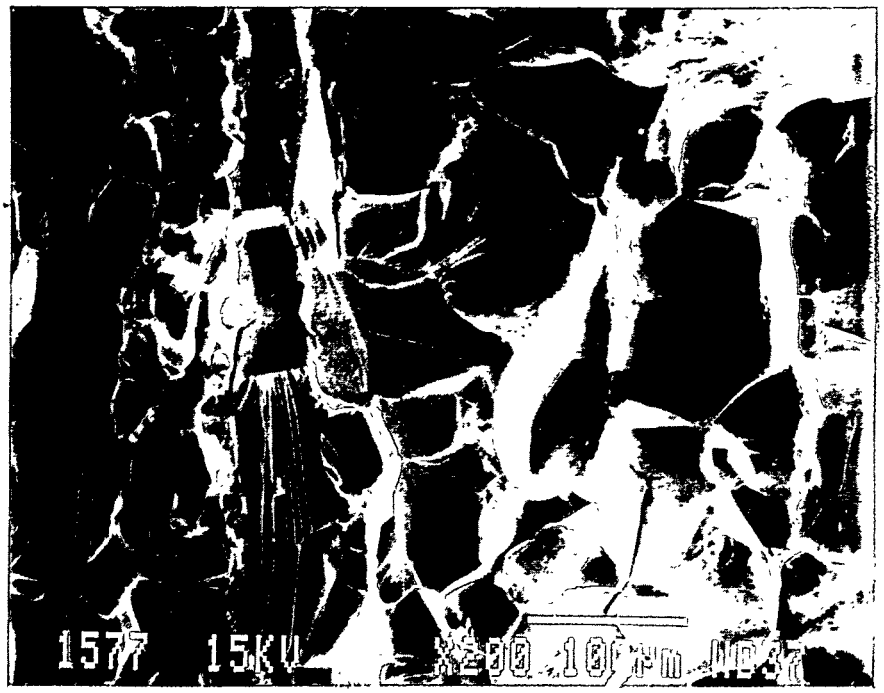
Mechanical Properties

Specimens Treated Using HT1

The specimens treated using HT1 were brittle at RT and lower. They fractured with a mixture of intergranular and cleavage failure. Fig. 4 shows the fracture surface of a specimen which failed at RT during K-testing. From Fig. 4b, it can be seen that the cleavage facets appear to initiate at the grain boundaries and pass through the whole grain. The dependence of the IFE on temperature is presented in Fig. 5. It can be seen that IFEs at RT and -100°C are 6 J and 1 J, respectively. The DBTT was estimated to be 20°C. J_{IQ} at RT was 52 kJ/m^2. When tested at 100°C, specimens were ductile and exhibited stable crack growth. J_{IQ} was about 485 kJ/m^2 and the tearing modulus was 250 mJ/m^3. Despite the high J_{IQ} value at 100°C, the fracture surface consisted of intergranular and microvoid coalescence features, as shown in Fig. 6. The intergranular portion makes up about 40% of the total fracture surface. However, it is evident from Fig. 6b that the grains experience a large amount of plastic deformation before they fracture along the grain boundaries, giving high fracture toughness.

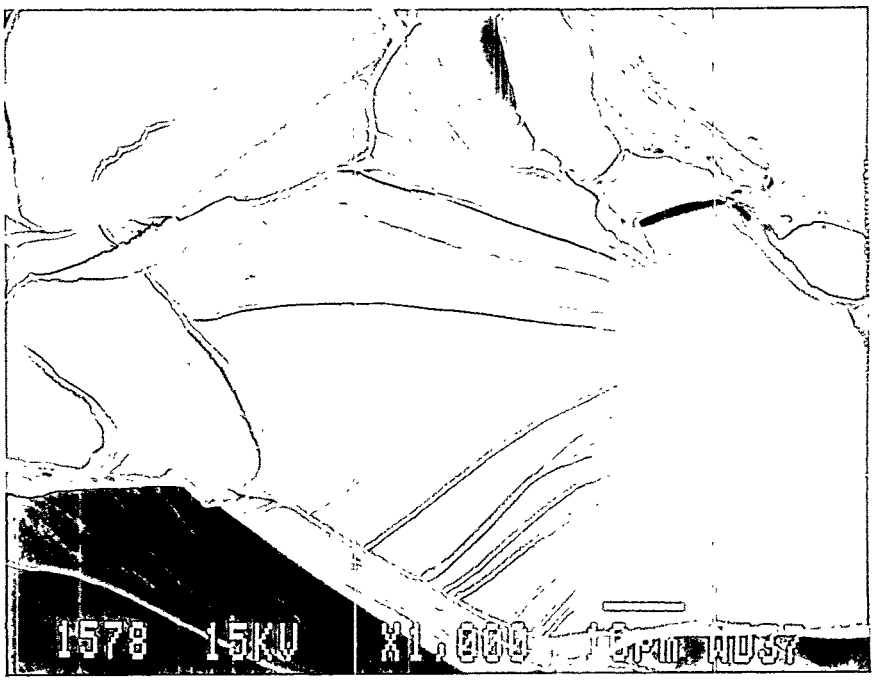
Specimens Treated Using HT2

The specimens treated using HT2 were very ductile at RT and fractured by microvoid coalescence during J-integral testing, as shown in Fig. 7. Specimens were so ductile that a complete J-R curve could not be constructed with our compact tension specimen (Fig. 2). The J_{IQ} estimated from the partial J-R curve (Fig. 8) is about 1100 kJ/m^2. Charpy testing at RT failed to cause fracture (two specimens) due to a large amount of plastic deformation. The IFE could not be determined. The data point marked by the arrow in Fig. 5 represents mainly plastic deformation energy, not including the energy for crack initiation and



(a)

Fig. 4. Overleaf.



(b)

Fig. 4. Fracture surfaces of specimens annealed at 1125°C for 1h and fractured at room temperature during K testing. a.) a mixture of intergranular, cleavage, and a little dimple failure; b.) a cleavage fracture facet showing the cleavage crack initiates at a grain boundary and passes through the entire grain.

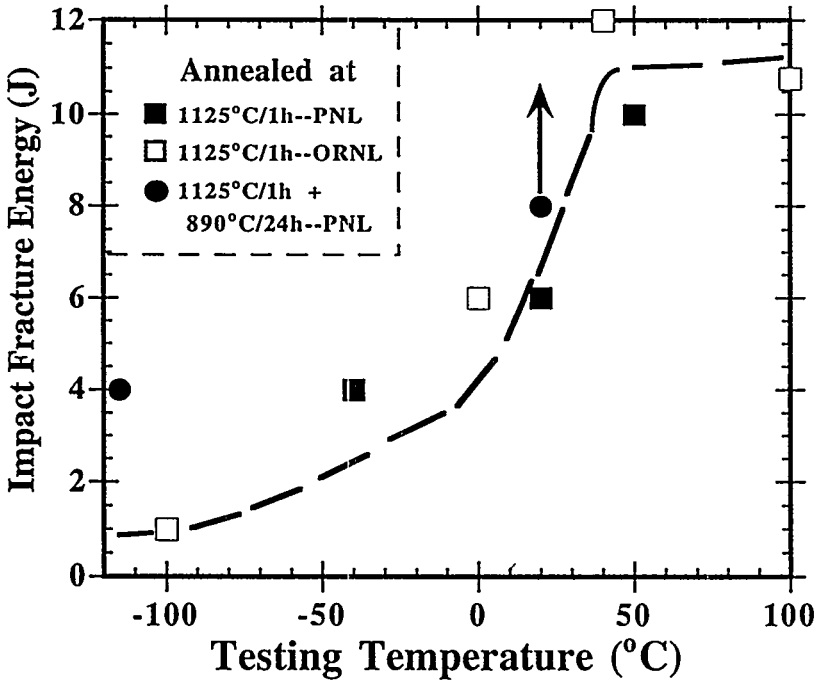
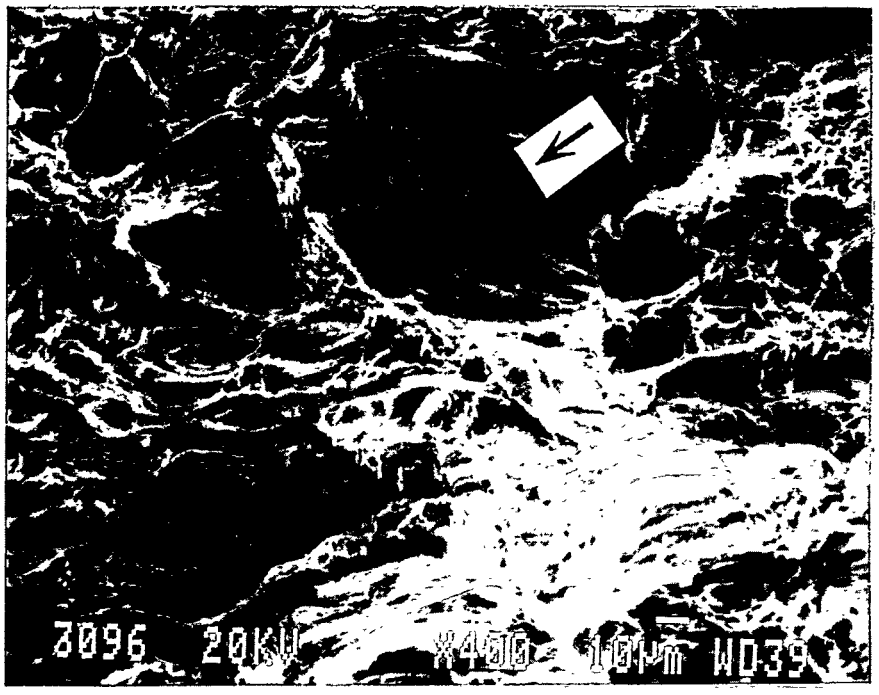


Fig. 5. Impact fracture energy vs testing temperature. PNL and ORNL indicate the tests were performed at Pacific Northwest Laboratory and Oak Ridge National Laboratory, respectively.

(a)



(b)

Fig. 6. Fracture surfaces of specimens annealed at 1125°C for 1h and fractured at 100°C during J-integral testing. They are a mixture of dimple and intergranular (about 40%) failure. The arrows indicate typical intergranular feature.

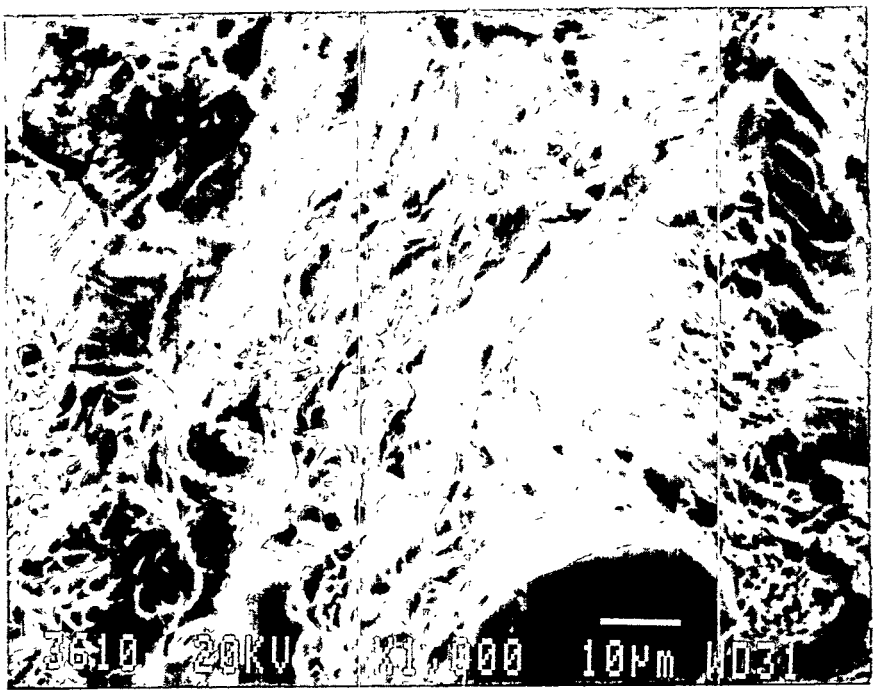


Fig. 7. The dimpled fracture surface of a specimen annealed at 1125°C for 1h plus 890°C for 24h and fractured at RT during J-integral testing.

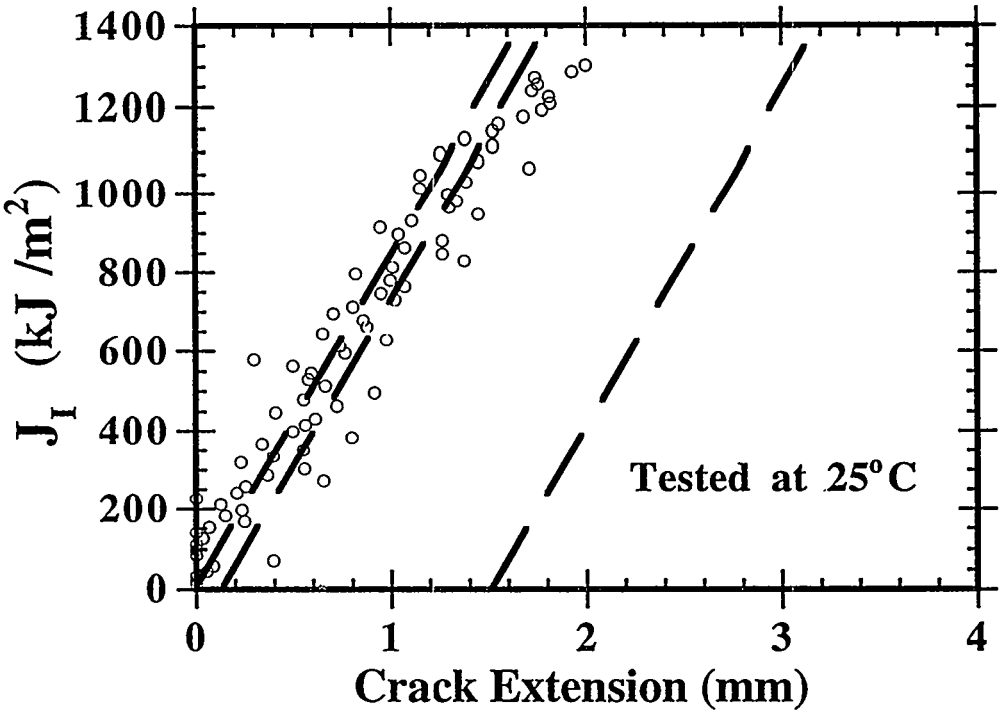


Fig. 8. The J-integral vs crack growth curve generated from a specimen annealed at 1125°C for 1h plus 890°C for 24h during J-integral testing at RT. J_{IQ} is estimated as about 1100 kJ/m^2.

propagation. Therefore, the IFE might be much higher than that in Fig. 5 which is indicated by the arrow. The IFE at -115°C was about 4 J, four times higher than the IFE for HT1 tested at -100°C. The fracture toughness was very sensitive to temperature. J_{IQ} at -50°C was 45 kJ/m^2, only about 1/20 of that at RT. The fracture mode also changed to cleavage fracture, as shown in Fig. 9. The DBTT was estimated to be less than -40°C. The effects of heat treatment and temperature on fracture properties of the V-5Cr-5Ti alloy are summarized in Table 3, Table 4, and in Fig. 10.

Fig. 9. the cleavage fracture surface of a specimen annealed at 1125°C for 1h plus 890°C for 24h and fractured at -50°C during K testing.

Discussion

The brittleness of the V-5Cr-5Ti treated using HT1 seems to stem from the low grain boundary fracture strength and brittleness of the grains themselves. It is known that impurity segregation to grain boundaries in a metallic material can reduce grain boundary strength, and interstitial impurities can enhance cleavage fracture. Because the grain size in the specimens treated using either HT1 or HT2 was almost identical, the increase in fracture strength produced with HT2 probably resulted from two factors. One of them is that HT2 increases grain boundary strength by reducing grain boundary S concentration, and the other is that it improves the ductility of grains by decreasing interstitial impurity concentration via precipitate formation. However, because our experiments are limited and not comprehensive (some data has not beenanalyzed yet), it is highly possible that as our research continues, some new results may surface requiring additional fundamental research. For example, why is this heat of V-5Cr-5Ti alloy sensitive to intergranular fracture, but previous heats of V-5Cr-5Ti and V-4Cr-4Ti alloys are not ?

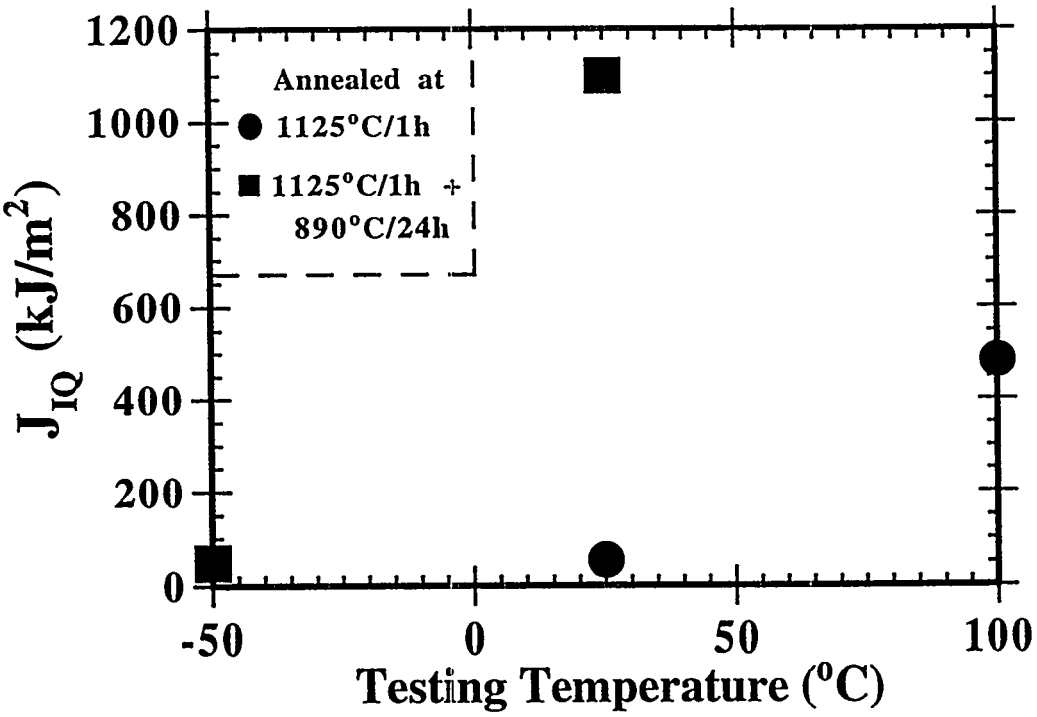


Fig. 10. Fracture toughness vs testing temperature for specimens annealed either at 1125°C for 1h or at 1125°C for 1h plus 890°C for 24h.

Table 4. Effect of Heat Treatment and Temperature on Fracture Toughness

Heat treatment	J_{IQ} (kJ/m^2)@ -50°C	25°C	100°C
HT1		52	485
HT2	45	~1100	

CONCLUSIONS

1. Specimens of V-5Cr-5Ti annealed at 1125°C (HT1) were brittle at room temperature and fractured by intergranular and cleavage modes. The J_{IQ} was about 52 kJ/m^2. The DBTT was about 20°C.

2. At 100°C HT1 treated specimens were ductile, giving a J_{IQ} value of 485 kJ/m^2, but fracture surfaces were composed of both intergranular and dimple fracture.

3. Annealing at 1125 and, then at 890°C (HT2) improved RT fracture toughness dramatically, from 52 to about 1100 kJ/m^2 and changed the mode of fracture to microvoid coalescence. The DBTT was decreased to below -40°C.

4. Specimens treated with HT2 were brittle at -50°C. The J_{IQ} value was 45 kJ/m^2, as compared with about 1100 kJ/m^2 at RT.

5. HT2 reduced grain boundary S concentration (0.9 at%) greatly as compared to HT1 (6 at%) and produced more precipitates.

FUTURE WORK

Further research will be performed with the new heats of V-5Cr-5Ti and V-4Cr-4Ti alloys.

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2. B. A. Loomis, private letter, April 9, 1994.