IMPACT TESTING AND FRACTURE BEHAVIOR OF VANADIUM ALLOYS –

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OBJECTIVE

The purpose of this research is to evaluate the impact properties and fracture behavior of the large heat of V-4Cr-4Ti (heat 832665) and small heats of compositional variants with Cr and Ti contents ranging from 3 to 6 wt.%.

SUMMARY

Charpy impact testing was completed on vanadium alloys with Cr and Ti contents ranging from 3 to 6 wt.%. A large heat (~500-kg melt) of V-4Cr-4Ti (heat 832665) and small heats (~15-kg melt each) of compositional variants, V-3Cr-3Ti, V-4Cr-4Ti-Si, V-5Cr-5Ti, V-6Cr-3Ti, and V-6Cr-6Ti, were examined in this work. One-third-size Charpy impact specimens, machined from 3.81-mm-thick plates of these vanadium alloys, were used for impact testing. In a fully recrystallized condition with a grain size of ~16 μm, the large heat of V-4Cr-4Ti exhibited a high level of resistance to cleavage failure with a DBTT at ~-190°C. The small (15 kg) heat of V-4Cr-4Ti heat treated to produce the same microstructural condition exhibited similar Charpy impact properties. The small heats containing higher concentrations of Cr and Ti, in a fully recrystallized condition exhibited a DBTT at around -100°C, whereas the V-3Cr-3Ti alloy failed by pure ductile shear at liquid nitrogen temperatures.

INTRODUCTION

In the early stages of the program on the development of alloys for fusion reactor applications, vanadium alloys with 3 to 6 wt.% Cr and Ti were investigated.[1-4] This composition range was subsequently narrowed down to vanadium alloys with 4 wt.% each of Cr and Ti based on the thermal creep properties, low DBTT under Charpy impact testing, resistance to swelling, and also resistance to helium- and irradiation-induced embrittlement exhibited by a laboratory-scale heat of this alloy.[5] A production-scale heat (~500-kg, heat 832665) of V-4Cr-4Ti alloy has been fabricated by Teledyne Wah Chang, Albany, Oregon (TWCA) recently. Initial impact data have been reported from the testing conducted on the samples machined from a warm-worked plate.[6] The present work reports the Charpy impact testing on the samples machined from 3.81 mm thick plate supplied by TWCA in an annealed condition. Impact behavior of the small heats of compositional variants has also been evaluated and presented here. These alloys will give the window for permissible ranges of Cr and Ti concentrations for consistent properties.

EXPERIMENTAL PROCEDURE

The chemical compositions of the large heat of V-4Cr-4Ti and the small heats of compositional variants have been presented in the companion article in this document.[7] One-third-size blunt-notch Charpy specimens (3.33 x 3.33 x 25.4 mm), with a notch that was 0.51 mm deep, with a root radius of 0.08 mm and an included angle of 30°, were obtained by electrodischarge machining from 3.81-mm-thick plates. All the specimens were oriented such that the crack could propagate parallel to the rolling direction. The 3.81-mm-thick plate of V-4Cr-4Ti (heat 832665) was annealed by the manufacturer at 1050°C for 2 hours. Following machining, the Charpy specimens were annealed for 2 hours at either 950°C, 1000°C, or 1050·C in a vacuum better than 1×10^{-6} torr. These annealing treatments removed any residual deformation induced by cutting and machining operations. In addition, during the recovery/recrystallization studies reported in a companion article,[7] it was observed that as the final annealing temperature is increased over this range, there is an increasing tendency for a fine precipitate dispersion to develop at grain boundaries.

The compositional variants were supplied in the form of 3.81-mm-thick plate in a 40% warm-rolled (400°C) condition. The machined Charpy samples were annealed at temperatures from 950° to 1050°C for 2 h in a vacuum better than 1×10^{-6} torr ($<10^{-4}$ Pa). Impact testing was carried out in a semiautomated pendulum-type impact testing system modified for subsize specimens.[8] Test temperatures employed were from –196° to 100°C with low temperatures being achieved by using liquid nitrogen. The results from the testing were fitted with a hyperbolic tangent function for determining the DBTT and upper-shelf energy. The lower-shelf energy was fixed at 0.2 J. The midpoint between the upper- and lower-shelf energy levels was considered as the DBTT.

Fractography was done using a scanning electron microscope on the fracture surfaces obtained after the impact testing. Metallography and TEM analysis for microstructural characterization were carried out on the samples cut from the undeformed sections of the impact specimens. The grain size was measured in a plane parallel to the rolling direction using the method of lineal intercepts.

RESULTS AND DISCUSSIONS

LARGE HEAT OF V-4Cr-4Ti (Heat 832665)

Figure 1 shows the typical optical microstructures of as received (annealed at 1050°C for 2 h at TWCA), and after 2-h anneals at 950°, 1000°, and 1050°C. Since the Charpy specimens were obtained from the pre-annealed (at TWCA) sheet, they were already recrystallized in the as-machined state. The fully recrystallized structure from the pre-anneal was quite stable for temperatures from 950° to 1050°C with no appreciable grain growth. The measured grain size for the three annealing conditions was identical at ~16 ± 4 μm.

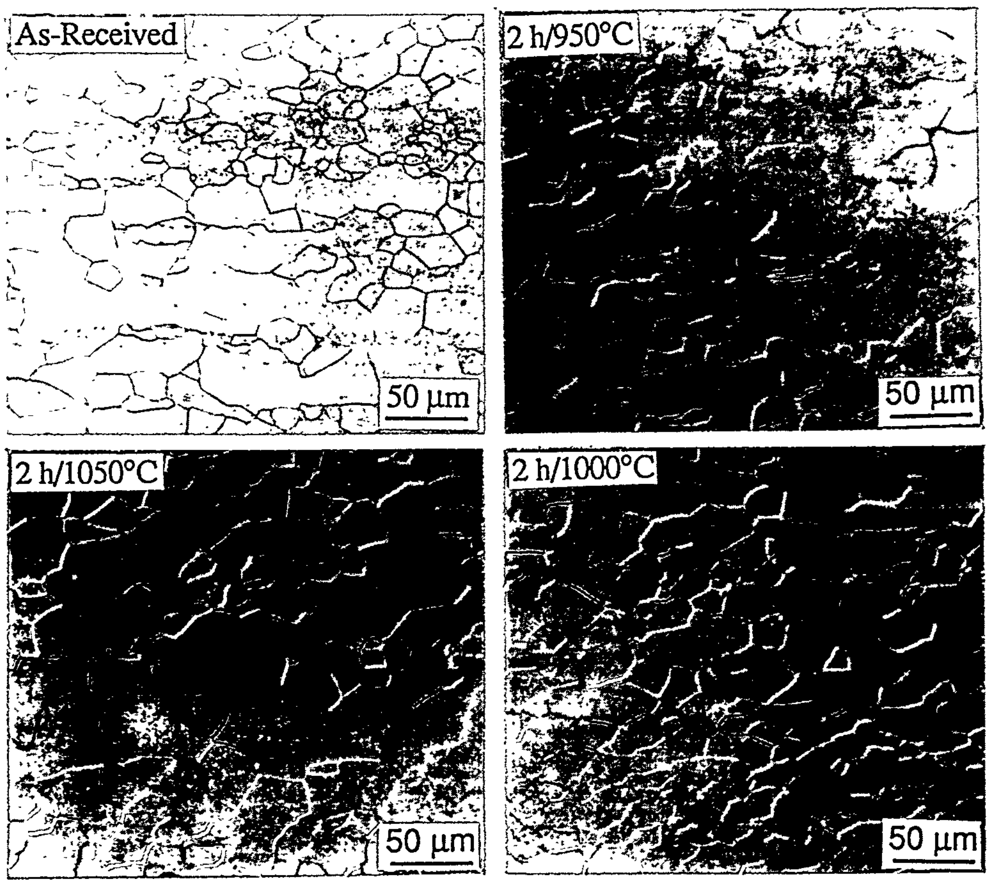

Figure 1. Typical microstructures of the large heat V-4Cr-4Ti as received, and after 2-h anneal from 950° to 1050°C.

The data obtained from Charpy impact testing are shown in Fig. 2 with absorbed impact energy plotted as a function of test temperature. There was no significant difference between the impact behavior of the three heat treated conditions indicating that the presence of varying amounts of the finely dispersed grain boundary phase has no effect on impact behavior. In Fig. 2, the data from the three anneals are fitted to a single curve. Thus, in a fully recrystallized condition a DBTT occurs at around –190°C.

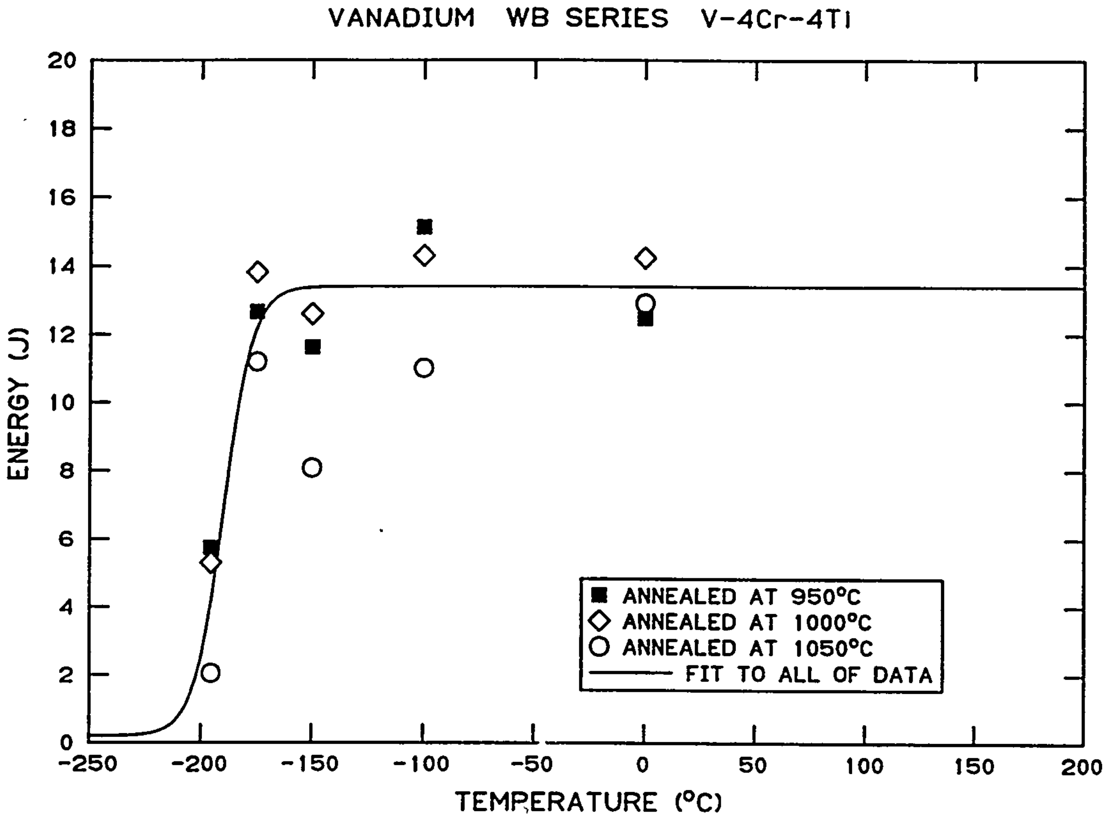

Figure 2. Third-size Charpy impact data for the large heat V-4Cr-4Ti (Heat 832665).

Figure 3 shows the typical fracture surfaces of the large heat of V-4Cr-4Ti at various test temperatures. As there was not much difference in the absorbed energy or grain size among the samples after 2-h anneals at 950°, 1000°, and 1050°C, only one set of fracture surfaces of samples annealed at 1050°C has been presented as representative of the three anneals. At a test temperature of 0°C, the fracture mode is purely ductile shear and the sample did not open up much but was bent under impact which resulted in a higher absorbed energy. With a decrease in temperature to –100°C, the fracture mode still remains as pure ductile shear. With further decrease in temperature (to –150°C), the fracture mode changes to mixed transgranular (TG) cleavage and microvoid coalescence with a drop in absorbed energy (from around 11 J to 8 J). At liquid nitrogen temperature (–196°C), the sample fails in a predominantly brittle TG cleavage mode with some secondary intergranular (IG) cracks which results in a dramatic drop in the absorbed energy to ~2 J. It is important to note here that there is no noticeable amount of IG failure in the transition or in the lower-shelf energy regions. This is in contrast to the results from impact testing of V-5Cr-5Ti (heat BL 63) which exhibited a predominant IG fracture in the transition region with TG fracture dominating the lower shelf energy region.[8]

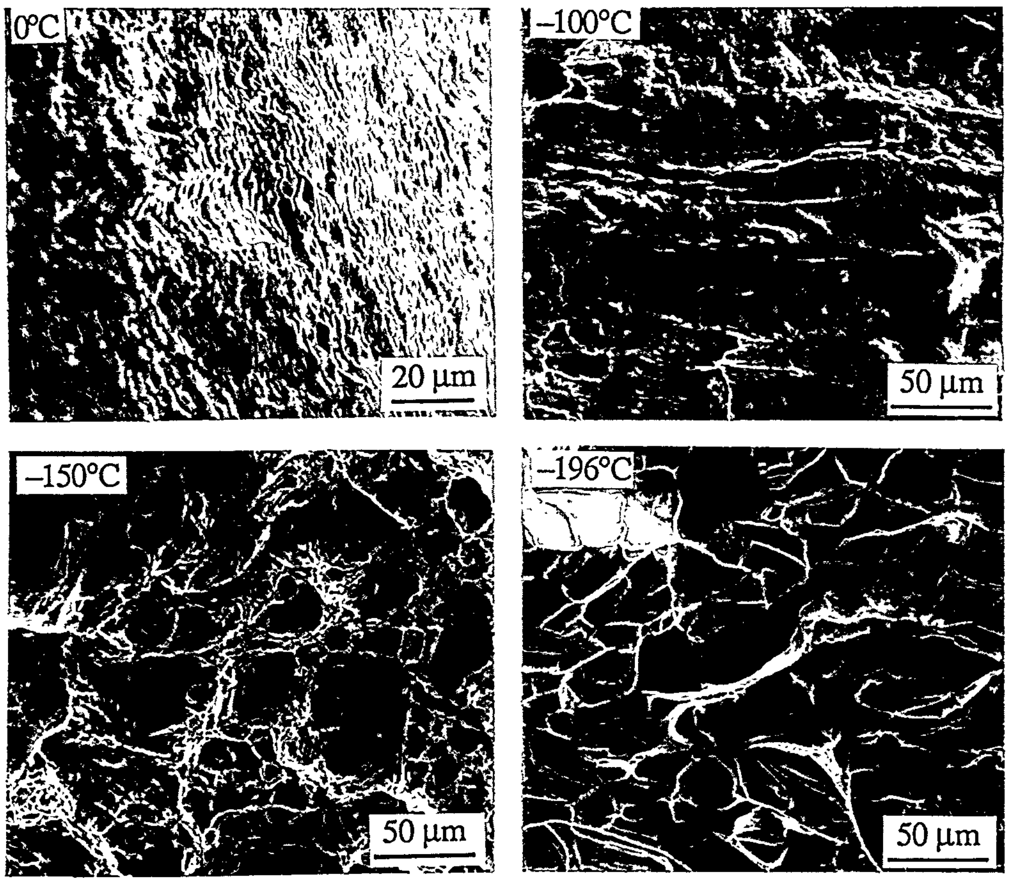

Figure 3. Typical fracture surfaces of the large heat V-4Cr-4Ti at different test temperatures. All samples are annealed at 1050°C for 2 h.

SMALL HEATS OF COMPOSITIONAL VARIANTS

Of the five compositional variants with various Cr and Ti contents, only Charpy specimens of V-6Cr-3Ti (heat T92) were annealed from 950° to 1050°C for 2 h to obtain different microstructures. The other four alloys, V-3Cr-3Ti (heat T91), V-4Cr-4Ti-Si (heat T89), V-5Cr-5Ti (heat T87), and V-6Cr-6Ti (heat T90), were annealed at 1000°C for 2 h to obtain a recrystallized structure in order to study their impact behavior.

Figure 4 shows the optical and TEM microstructures of V-6Cr-3Ti after annealing from 950° to 1050°C for 2 h. At 950°C, the microstructure consists of well-developed subgrains together with a small volume fraction (<10%) of new recrystallized grains. At 1000°C, recrystallization is 70-80% complete with isolated regions containing subgrains. Annealing at 1050°C produced complete recrystallization.

The results from the impact testing of Charpy specimens of V-6Cr-3Ti which were annealed from 950° to 1050°C for 2 h is depicted in Fig. 5. All the three microstructures produced by these anneals showed a typical ductile-to-brittle transition behavior with decrease in impact test temperature. The DBTT ranged from around –175°C for the 950°C microstructure to around –75°C for the fully recrystallized structure produced by the 2 h anneal at 1050°C. The DBTT of the microstructure with 70-80% recrystallization (after 2-h anneal at 1000°C) falls in-between at ~–125°C. This indicates that the final microstructure has a significant influence on the impact behavior which was also shown in an earlier study by Grossbeck et al[8] on the V-5Cr-5Ti alloy (heat BL 63).

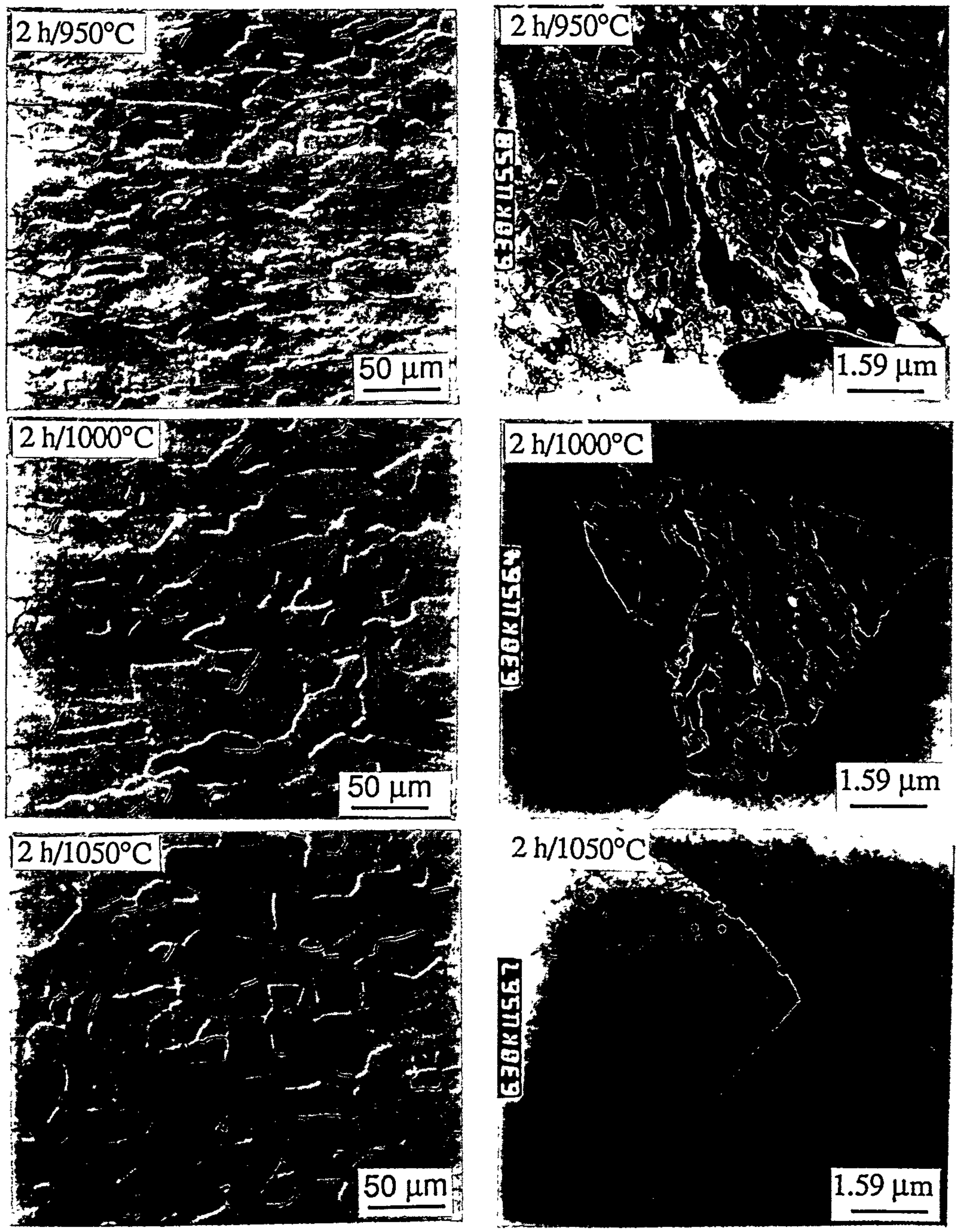

Figure 4. Typical optical and TEM microstructures of V-6Cr-3Ti after anneals from 950° to 1050°C for 2 h.

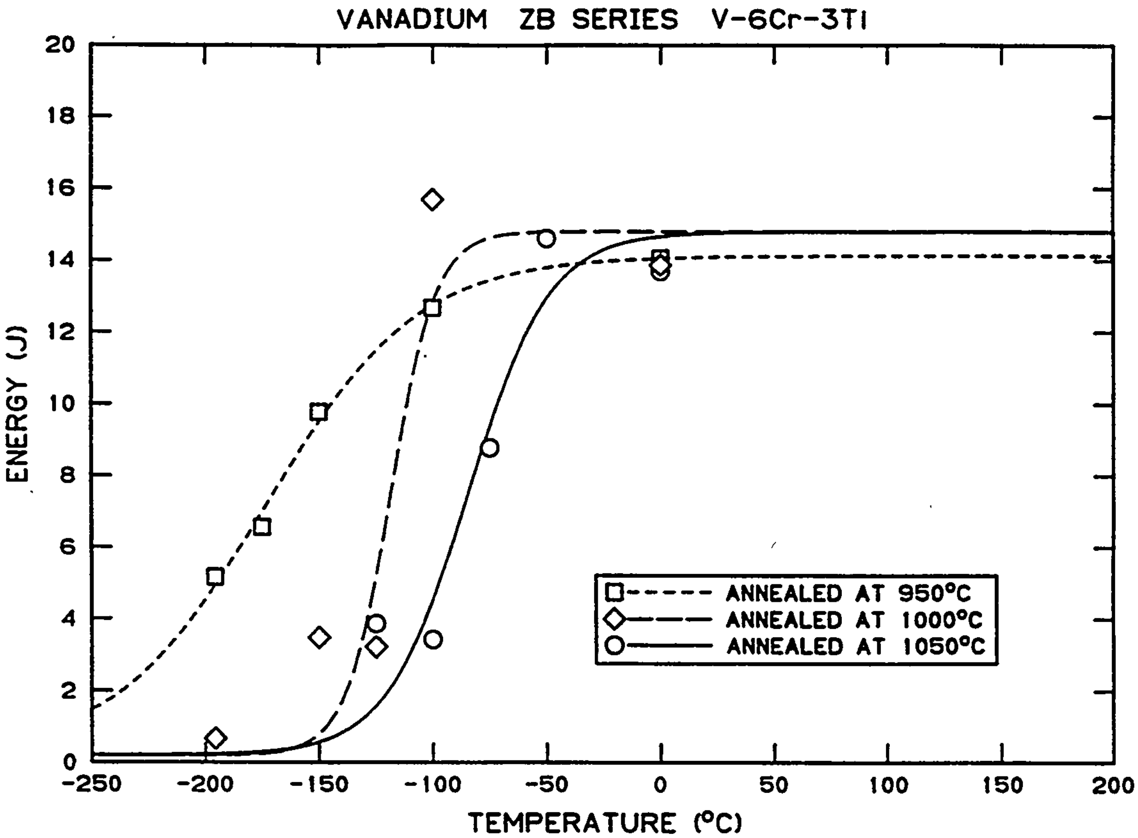

Figure 5. Third-size Charpy impact data for the small heat of V-6Cr-3Ti.

The typical fracture surfaces of the specimens after impact testing are shown in Fig. 6. At higher test temperatures where the absorbed impact energy is high, the fracture mode is a pure ductile shear irrespective of the annealing treatment. The samples which were annealed at 950°C (with high dislocation density and minor recrystallization) showed a mixture of ductile rupture and microvoid coalescence with some TG cleavage at lower temperatures (≤–175°C), and in the transition range the fracture mode was a mixture of TG cleavage and microvoid coalescence. The samples which were annealed at 1000°C (resulting in a mostly recrystallized microstructure) and 1050°C (resulting in a fully recrystallized microstructure) exhibited almost identical modes of failure at high and low test temperatures. At high temperatures, these samples failed in a pure ductile shear whereas at low temperatures, they fractured in a predominantly TG cleavage. In the transition region, the samples annealed at 1050°C showed some shear and microvoid coalescence with mostly TG cleavage.

Figure 7 shows the optical microstructures of the four compositional variants, V-3Cr-3Ti, V-4Cr-4Ti-Si, V-5Cr-5Ti, and V-6Cr-6Ti, after 2-h anneal at 1000°C. All the four alloys are almost fully recrystallized at this temperature. The third-size Charpy samples from these alloys, after annealing at 1000°C for 2 h, were tested under impact conditions. The results from the Charpy impact testing are plotted in Fig. 8 with absorbed energy as a function of test temperature. Also included in Fig. 8 are the data for V-6Cr-3Ti, the samples of which were annealed similarly (2 h at 1000°C) for comparison. The impact results shown in Fig. 8 can be placed into two groups: one group containing alloys with lower (Cr + Ti) contents, i.e. V-3Cr-3Ti and V-4Cr-4Ti-Si, and the other group having alloys with higher (Cr + Ti) contents, i.e. V-5Cr-5Ti, V-6Cr-3Ti, and V-6Cr-6Ti. The lower (Cr + Ti)-containing alloys exhibit excellent impact behavior with V-4Cr-4Ti-Si showing DBTT of around –175°C. The V-3Cr-3Ti alloy did not show any ductile-to-brittle transition and remained quite ductile even at –196°C with shelf energy around 12 J. The higher

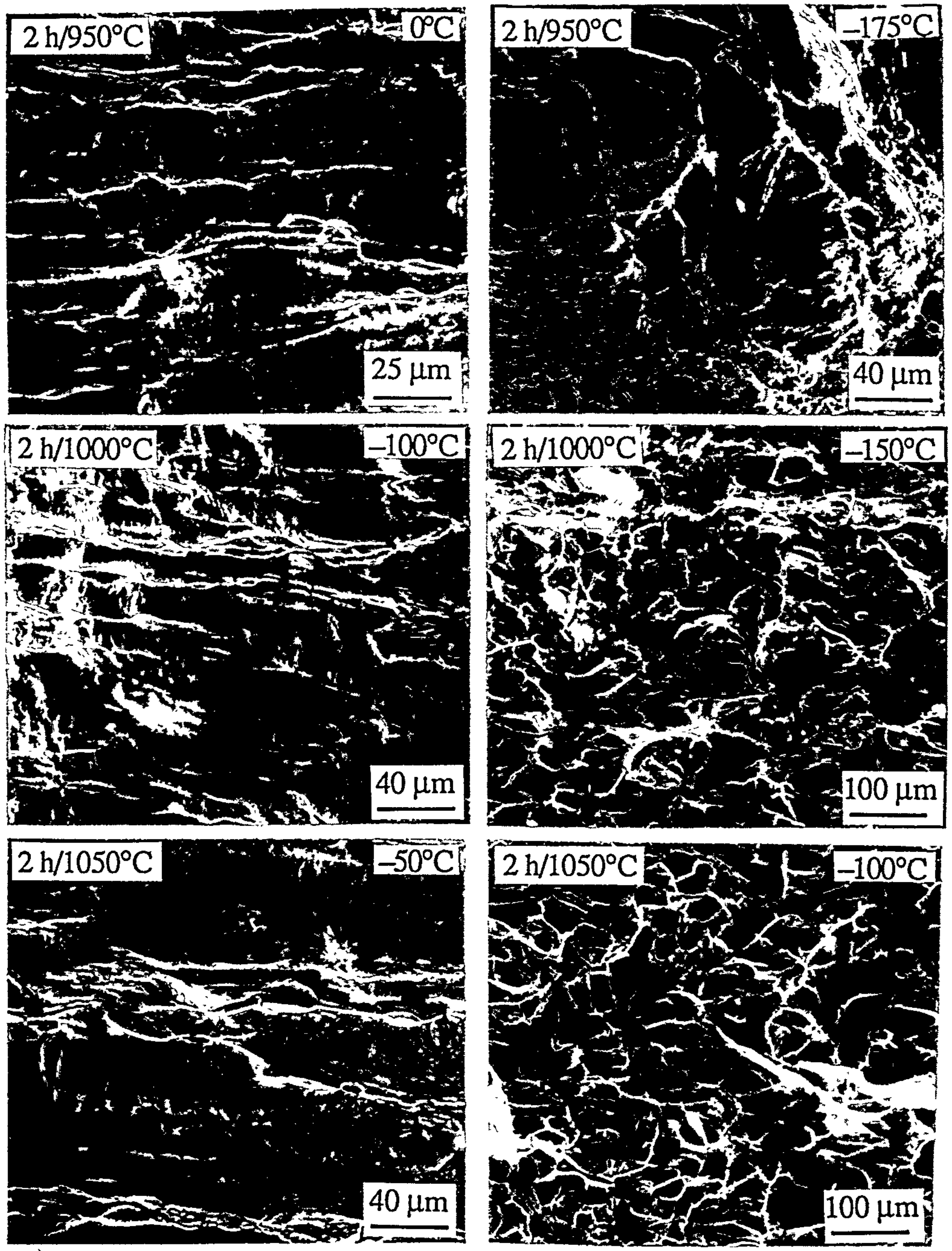

Figure 6. Typical fracture surfaces of the small heat of V-6Cr-3Ti at different test temperatures and anneals.

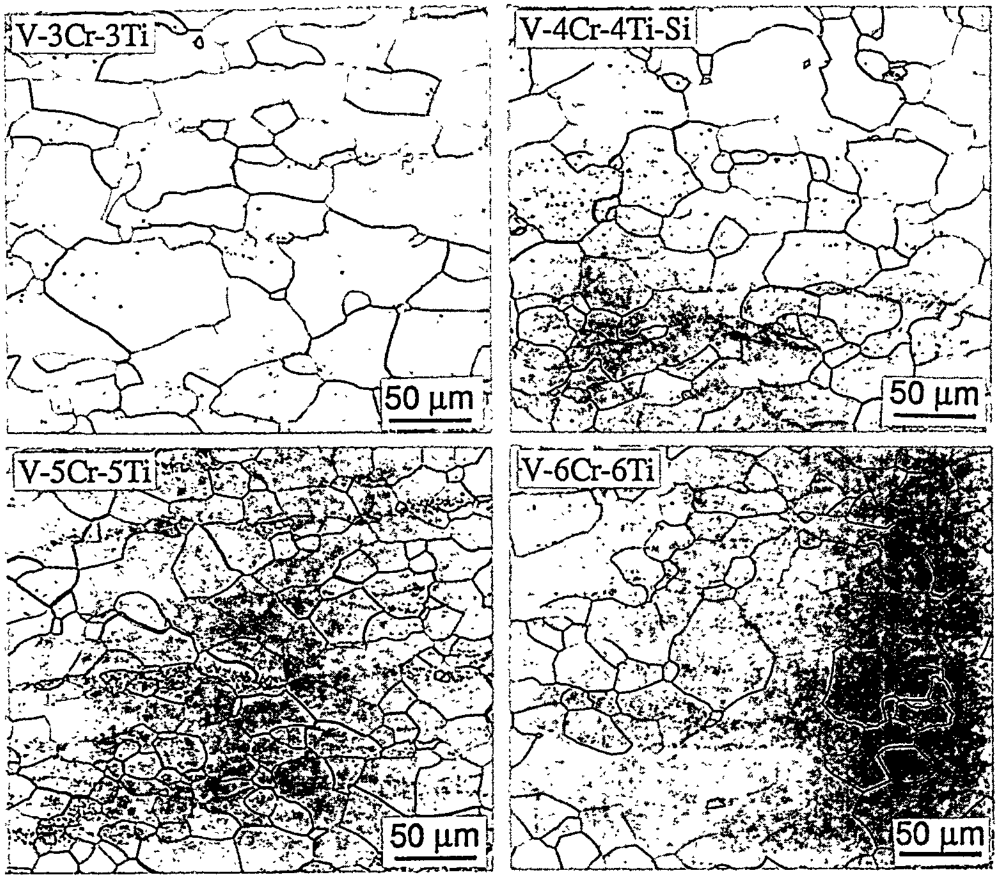

Figure 7. Typical optical microstructures of small heats of Compositional Variants after annealing at 1000°C for 2 h.

(Cr + Ti)-containing alloys, V-5Cr-5Ti, V-6Cr-3Ti, and V-6Cr-6Ti all had a DBTT of ~ –120°C. The only difference among these alloys is in the upper shelf energy with that for V-6Cr-3Ti being around 15 J and V-5Cr-5Ti and V-6Cr-6Ti having around 11 J. In a companion article in this document,[7] it was reported that the microhardness of the same alloys studied in the present work was grouped in a similar fashion. In the fully recrystallized conditions, the lower (Cr + Ti)-containing alloys had lower microhardness values (130-140 DPH) compared to the higher (Cr + Ti)-containing alloys which possessed higher hardness values (155-160 DPH). Thus, the results of the present study, in conjunction with those of the companion article,[7] indicate that there is an incremental change in the physical and mechanical properties when the combined (Cr + Ti) content exceeds ~8 wt %.

It is interesting to compare the impact behavior of large heat of V-4Cr-4Ti (heat 832665) and the small heat of V-4Cr-4Ti-Si (heat T89) both of which have similar chemical compositions. For a similar fully recrystallized microstructure, there is no significant difference in the DBTT or the upper-shelf energy (compare Figs. 2 and 8) of the large and small heats of V-4Cr-4Ti, with the DBTT of large heat being around –190°C and that for the small heat being around –175°C. It was reported earlier by Grossbeck et al[8] that the large heat of V-5Cr-5Ti (heat BL 63) exhibited a DBTT of around –120°C in the fully recrystallized condition. In the present study, the small heat of V-5Cr-5Ti (heat T87) had an identical DBTT of ~ –120°C for a fully recrystallized microstructure. This indicates that the scaling (large or small heat) of an alloy has little effect on the impact behavior provided the chemical compositions and microstructures are similar.

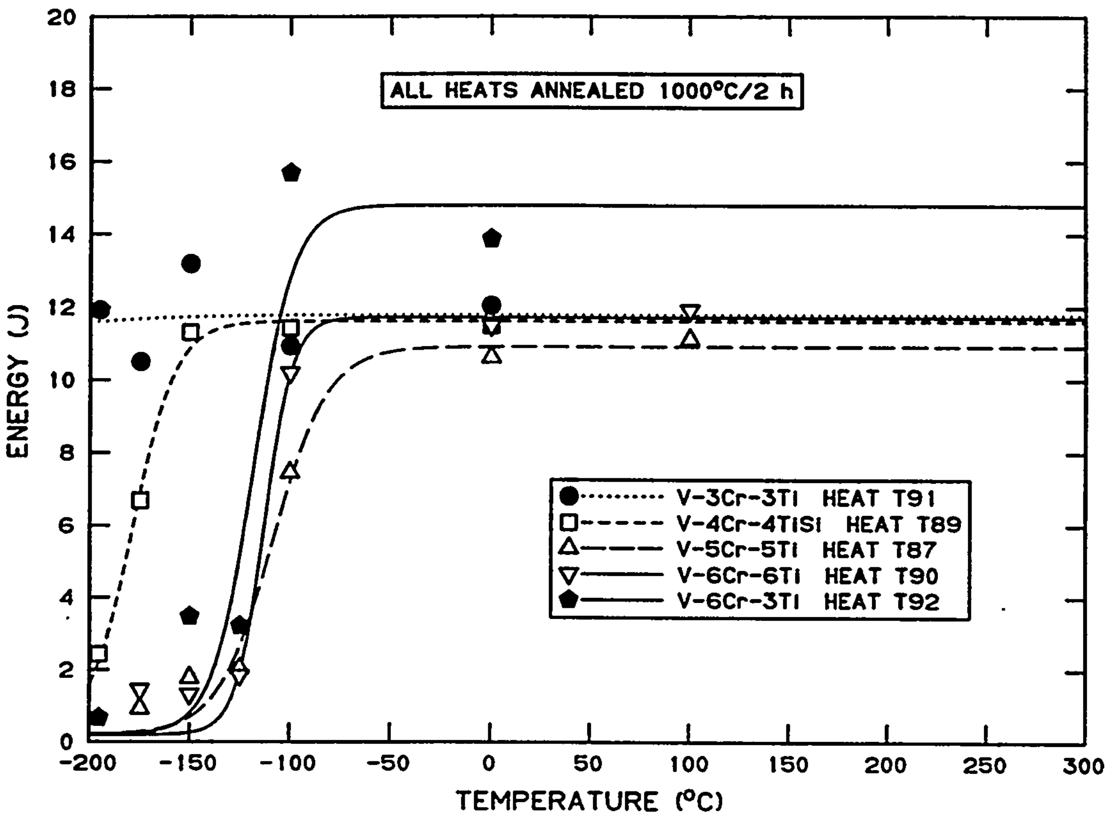

Figure 8. Third-size Charpy impact data for the small heats of Compositional Variants.

The typical fracture surfaces of the four compositional variants, V-3Cr-3Ti, V-4Cr-4Ti-Si, V-5Cr-5Ti, and V-6Cr-6Ti are shown in Fig. 9. The fractographs presented here were taken from samples tested at different impact temperatures. The three alloys, V-4Cr-4Ti-Si, V-5Cr-5Ti, and V-6Cr-6Ti, showed pure ductile shear for temperatures from +100° to –100°C with the exception of V-5Cr-5Ti which had some microvoid coalescence. At lower temperatures of –175° to –196°C, the fracture mode for these three alloys was predominantly TG cleavage with some secondary IG cracks. In the intermediate temperature range, the fracture mode was a mixture of TG cleavage and microvoid coalescence. In the whole test temperature range from 0° to –196°C, V-3Cr-3Ti, which exhibited no ductile-to-brittle transition with high shelf energy, did not fracture completely and the fracture mode was always a pure ductile shear.

CONCLUSIONS

(1) In the fully recrystallized condition with a grain size of ~16 μm, the large heat of V-4Cr-4Ti has very high fracture resistance at temperatures in the fusion operating regime with a DBTT at ~ -190°C. The fracture mode in blunt notch Charpy tests is pure ductile shear at the higher test temperatures and transgranular cleavage in the lower shelf regime.

(2) The 15 kg heat of V-6Cr-6Ti showed a significant variation in impact properties depending on the extent of recrystallization induced by the final anneal. The resistance to transgranular cleavage at low temperatures is greater when the microstructure contains a high proportion of recovered subgrains.

(3) In a fully recrystallized microstructural condition, the Charpy impact properties of the compositional variant heat falls into two groups. Alloys containing a combined (Cr + Ti) content in excess of

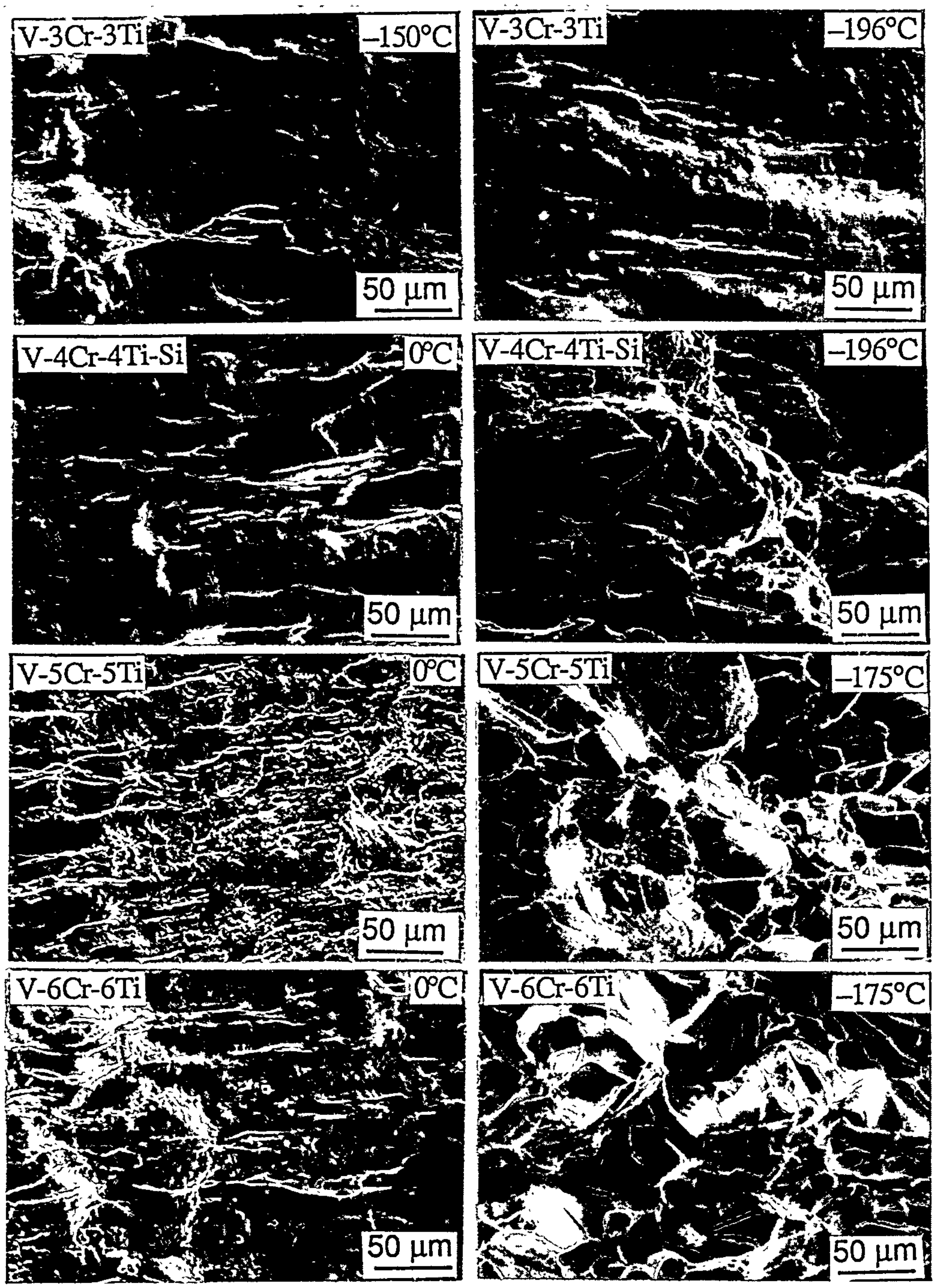

Figure 9. Typical fracture surfaces of the small heats of Compositional Variants at different test temperatures. All are annealed at 1000°C for 2 h.

~8 wt. % exhibit a DBTT at ~ -120°C. Alloys containing 3Cr3Ti and 4Cr4Ti showed better resistance to cleavage fracture at low temperatures.

(4) The results from the present study in conjunction with those of earlier works demonstrate that scaling (large or small heat) of an alloy has little effect on the impact behavior provided the chemical compositions and microstructures are similar.

WORK IN PROGRESS

The effects of varying the degree of recrystallization on the irradiated properties of the large heat will be investigated. The tensile properties of the compositional variants in the range 400 to 650°C will be determined.

ACKNOWLEDGMENT

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