# EFFECT OF ANNEALING ON IMPACT PROPERTIES OF PRODUCTION-SCALE HEAT OF V-4Cr-4Ti*

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## OBJECTIVE

Following reports of excellent properties of a laboratory heat of V-4Cr-4Ti, the alloy identified as the primary vanadium-based candidate for application in fusion reactor structural components, a large production-scale (500-kg) heat of the alloy was successfully fabricated. The objective of this study is to identify an annealing procedure that produces optimal impact properties in this heat in the unirradiated state.

## SUMMARY

A 500-kg heat of V-4Cr-4Ti, an alloy identified previously as the primary vanadium-based candidate alloy for application in fusion reactor structural components, has been successfully produced. Impact tests were conducted at -196 to 150°C on one-third-size blunt-notch Charpy specimens of the scaleup heat in as-rolled condition and after annealing for 1 h at 950, 1000, and 1050°C in a high-quality vacuum. The annealed material remained ductile at all test temperatures; the ductile-brittle transition temperature was lower than -200°C. The upper-shelf energy of the production-scale heat was similar to that of the laboratory-scale (≈30-kg) heat of V-4Cr-4Ti investigated previously. The effect of annealing temperature between 950 and 1050°C was not significant; however, annealing at 1000°C for 1 h not only produced the best impact properties but also ensured a sufficient tolerance to the effect of temperature inhomogeneity that is expected when large components are annealed. The effect of the notch geometry of the Charpy-impact specimens was also investigated. When annealed properly (e.g., at 1000°C for 1 h), impact properties were not sensitive to notch geometry (45°-notch, root radius 0.25 mm; and 30°-notch, root radius 0.08 mm).

## INTRODUCTION

To develop and identify an optimal vanadium-base alloy for application in fusion reactor first wall/blanket structures, extensive investigations were conducted earlier on the swelling behavior, tensile properties, creep strength, impact toughness, and microstructural stability of V-Ti, V-Cr-Ti, and V-Ti-Si alloys before and after irradiation by fast neutrons at 420°C-600°C. These investigations revealed that V-Cr-Ti alloys that contained ≈4 wt.% Cr, ≈4 wt.% Ti, 500-1000 wt. ppm Si, and <1000 wt. ppm O+N+C were most desirable because they exhibit superior physical and mechanical properties.[1-5] These results were obtained, however, on laboratory-scale (<30-kg) heats, including a small heat (ANL ID BL-47) of V-4Cr-4Ti that exhibited excellent resistance to thermal creep,[4] irradiation-induced embrittlement,[1,2] swelling,[3,5] and helium embrittlement.[6-9] In the previous reporting period, a large (≈500-kg) production-scale heat of V-4Cr-4Ti (Heat ID #832665) was successfully fabricated in a joint effort between Argonne National Laboratory and Teledyne Wah Chang (Albany, Oregon). The objective of the effort was to demonstrate reliable industrial production of good-quality V-4Cr-4Ti.[10] This report describes results of an investigation of the effects of high-temperature (950-1050°C) annealing on the impact properties of the 500-kg heat. The objective was to identify an annealing procedure that produces optimal impact properties in the unirradiated V-4Cr-4Ti alloy class. The Charpy-impact test (at -196 to 150°C) was chosen because it is known to be most sensitive to conditions of thermomechanical treatment of vanadium-base alloys.

## EXPERIMENTAL PROCEDURE

The elemental composition of the 500-kg heat is given in Table 1. Also in the table is the elemental composition of the laboratory-scale heat of V-4Cr-4Ti (ANL ID BL-47) that was shown earlier to exhibit excellent properties.[1-9]

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*Table 1. Chemical composition (impurities in wppm) of industrial– (500 kg) and laboratory–scale heats of V–4Cr–4Ti*

| Heat ID | ANL ID | Heat Type | Cr | Ti | Cu | Si | O | N | C | S | P | Ca | Cl | Na | K | B |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| – | BL–47 | laboratory 30 kg | 4.1 wt.% | 4.3 wt.% | 6 | 870 | 350 | 220 | 200 | 20 | <40 | 1 | 1 | 0.1 | 0.1 | 15 |
| 832665 | BL–71 | production 500 kg | 3.8 wt.% | 3.9 wt.% | <50 | 783 | 310 | 85 | 80 | <10 | <30 | <10 | <2 | – | – | <5 |

One–third–size Charpy specimens (3.33 x 3.33 x 25.4 mm) were machined from 3.81–mm–thick plates of the material, some of which had been annealed in the production factory (Teledyne Wah Chang, Albany, Oregon) for 2 h at a nominal temperature of ≈1050°C and some of which were received in as–rolled (≈40–50% worked at 400°C) condition. The Charpy specimens were machined so that the plane of crack propagation was perpendicular to the rolling direction (i.e., the L–S direction in Fig. 1). To investigate the effect of notch geometry, two types of V–notch geometry were investigated; one with a 45° notch angle and 0.25–mm root radius and the other with a 30° angle and 0.08–mm root radius. Notch depth in both types of specimens was kept constant at 0.61 mm. The impact specimens machined from the factory–annealed plates were tested after a degassing heat treatment at 400°C for 1 h in vacuum, a customary procedure used to expel hydrogen that could be picked up during specimen machining and preparation. To identify the optimal annealing condition, specimens machined from as–rolled (cross–rolled at 400°C)[10] plates were annealed at 950, 1000, and 1050°C for 1 h in a vacuum of ≈6 x $10^{-6}$ Pa prior to testing. The cooling rate down to ≈400°C was ≈50°C per minute. Details of the drop–weight–type impact test have been described elsewhere.[2]

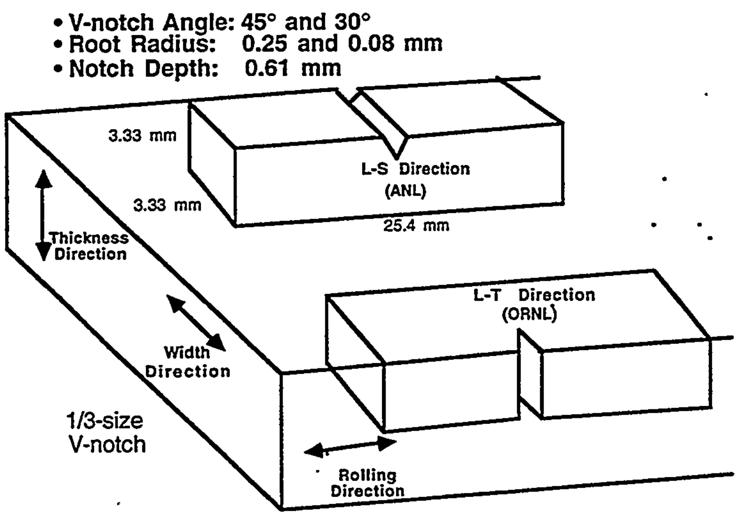


*Fig. 1. Orientation of Charpy impact specimens with respect to rolling direction of production–scale heat of V–4Cr–4Ti*

## RESULTS AND DISCUSSION

Impact energies, measured on the 45°–notched (root radius 0.25 mm) Charpy specimens from the production–scale heat of V–4Cr–4Ti in as–rolled condition and after annealing for 1 h at 950, 1000 and 1050°C, are shown in Fig. 2 as a function of impact test temperature. Also shown in the figure are the impact energies of the heat that was annealed in factory (nominally at 1050°C for 2 h in oil–diffusion–pumped vacuum). The geometry of the specimens in the figure was the same as that used in previous investigations, including the laboratory–scale (30–kg) heat of V–4Cr–4Ti (BL–47, Table 1).[2] From the results in the figure, the optimal annealing temperature appears to be ≈1000°C, the same as that found to produce minimum hardness in ≈85% cold–worked V–4Cr–4Ti (i.e., the 30–kg laboratory heat, BL–47, Table 1).[11] Results in Fig. 2 show that the impact properties of the production–scale heat are as good as those of the smaller laboratory heat, which was fabricated by essentially the same procedure. The ductile–brittle–transition temperature (DBTT) of the production–scale heat is no higher than ≈–200°C, similar to that of the laboratory–scale heat. To show a direct comparison, Charpy energies of the two heats are plotted in Fig. 3.

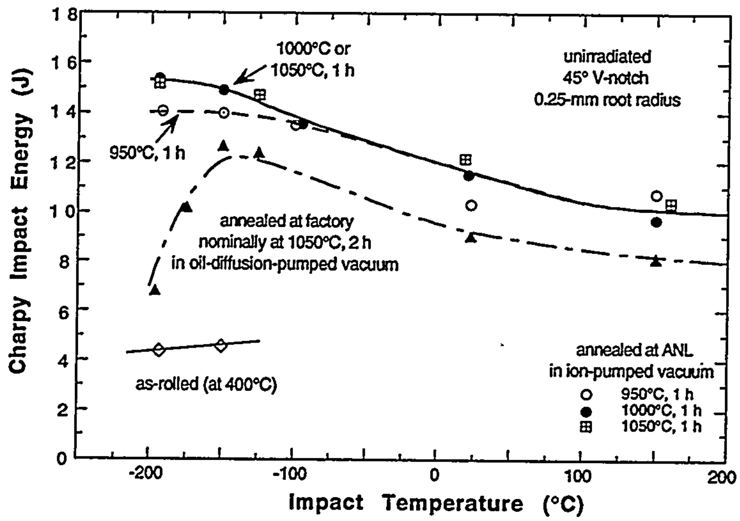


*Fig. 2.*
*Charpy energy as function of impact temperature of production–scale heat of V–4Cr–4Ti after annealing for 1 h at 950, 1000, and 1050°C. Optimal annealing temperature is ≈1000°C*

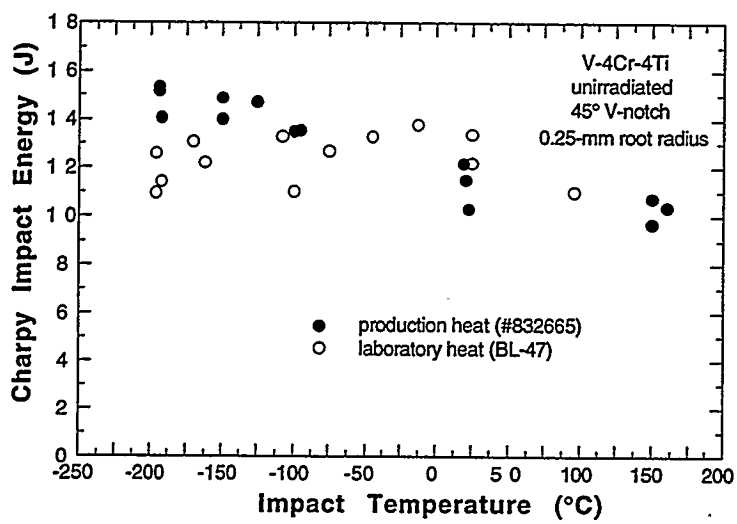


*Fig. 3.*
*Comparison of impact properties of production– and laboratory–scale heats of V–4Cr–4Ti*

Impact properties similar to those in Fig. 2 were also determined for 30°–notched specimens (root radius 0.08 mm). The results are shown in Fig. 4. Slight effects of notch geometry were observed only for specimens (average grain size ≈28 mm) annealed in the factory at a nominal temperature of 1050°C for 2 h. However, the 30°–notched specimens exhibited impact properties as excellent as those of the 45°–notched specimens (DBTT <–200°C) when annealed at 950, 1000, or 1050°C for 1 h in an ion–pumped vacuum system. When annealed at 1100°C for 1 h, there was an indication of a slight increase of DBTT in the heat.

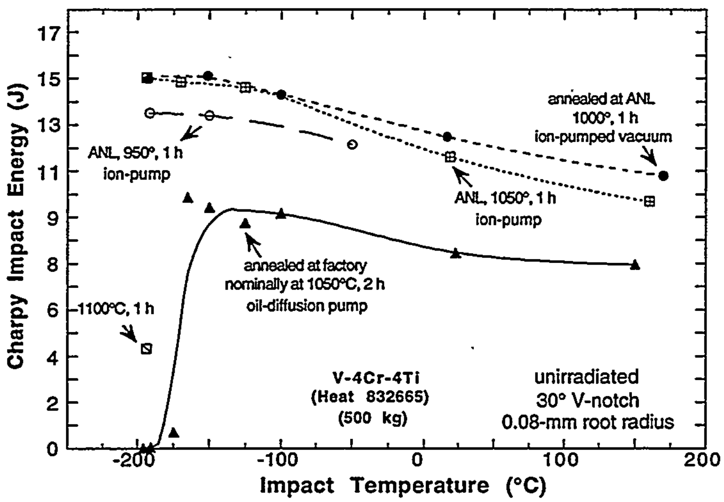


*Fig. 4.*
*Effect of annealing on Charpy impact energy of 30°–notch specimen as function of test temperature for production–scale heat of V–4Cr–4Ti*

When annealed at 1000 or 1050°C for 1 h in a high–quality vacuum, the production–scale heat exhibited negligible effect of notch geometry on impact properties, as shown in Figs. 5A and 5B, respectively. In Fig. 6, the effect of vacuum quality (i.e., ion–pumped vs. oil–diffusion–pumped) is shown. In the latter vacuum system, Charpy specimens were wrapped with a titanium shroud and annealed in a vacuum of $\approx 1.5 \times 10^{-3}$ Pa. Apparently, annealing in an ion–pumped vacuum produces higher impact toughness under otherwise identical condition.

Based on the results given in Figs. 2–6, the optimal annealing procedure seems to be to anneal at 1000°C for 1 h in a high–quality vacuum system. These annealing conditions provide a sufficient tolerance (at least ±50°C) to temperature uncertainties and nonuniformities that are expected during annealing of larger and thicker field components. The material annealed at 1000°C for 1 h in the laboratory exhibited partially recrystallized grain structure.[10]

## CONCLUSIONS

(1) Impact tests were conducted on a production–scale (≈500-kg) heat of V-4Cr-4Ti at –196 to 150°C. Following annealing at 950–1050°C for 1 h in a high–quality vacuum system, the material remained ductile at –196°C and that the ductile–brittle transition temperature (DBTT) was no higher than –200°C. Upper–shelf energies of the production–scale heat were similar to those of a laboratory–scale heat.

(2) The effect of annealing temperature (950–1050°C) on impact properties of the production–scale heat was not significant, a finding similar to that obtained for a laboratory–scale heat. This is in contrast to the very significant effect of annealing temperature on the impact properties of an incorrectly fabricated heat of V–5Cr–5Ti (ANL ID Heat BL–63), which exhibited inferior mechanical properties.

(3) Annealing at 1000°C for 1 h in a high–quality vacuum not only produces optimal impact properties in the production–scale heat but also provides sufficient tolerance to temperature inhomogeneity. Following annealing under these conditions, impact properties of the production–scale heat were not sensitive to notch geometry, and excellent impact toughness was observed at >–200°C.

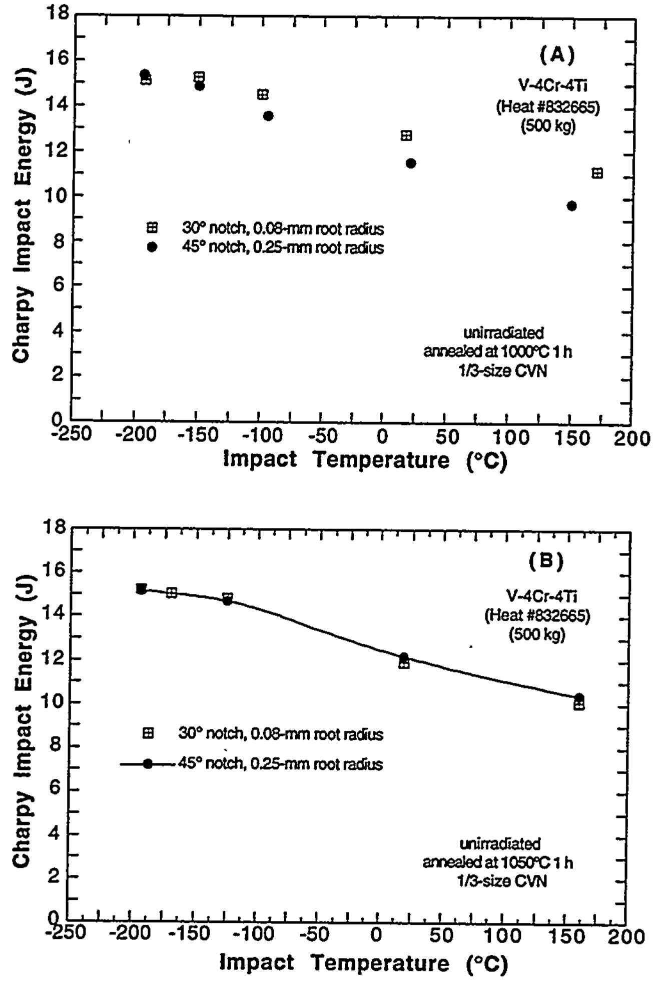


*Fig. 5. Effects of notch geometry on impact properties of 500–kg V–4Cr–4Ti annealed at (A) 1000°C and (B)1050°C for 1 h*

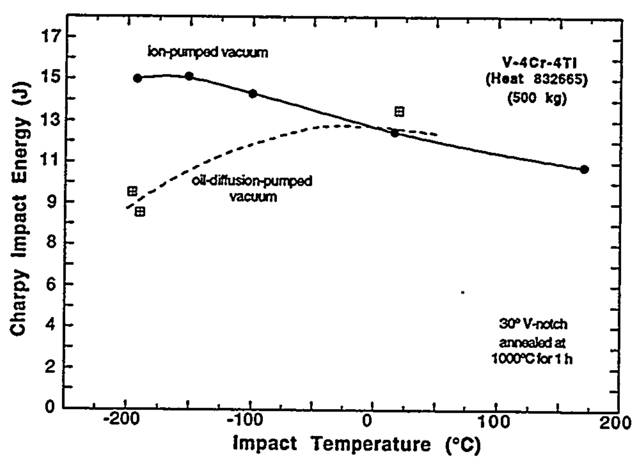


*Fig. 6. Effect of vacuum quality of annealing furnace on Charpy–impact energy of production–scale heat of V–4Cr–4Ti*

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