Characterization of V–4Cr–4Ti Heat 832665 – M. L. Grossbeck, D. J. Alexander, J.J . Henry, Jr., W. S. Eatherly, and L. T. Gibson (Oak Ridge National Laboratory)

OBJECTIVE

A liquid lithium-cooled vanadium structure is a favored concept for an advanced breeding blanket. The objective of this task is to define the processing parameters for optimal alloy performance in the V-Cr-Ti alloy system.

SUMMARY

A new 500 Kg heat of V–4Cr–4Ti, Heat 832665, is now being characterized by various national laboratories. The Oak Ridge National Laboratory has received sheet of several thicknesses from Argonne National Laboratory (ANL); characterization by chemical analysis, Charpy impact testing, and metallography is in progress. The Charpy tests have shown that the material does not experience a ductile to brittle transition temperature even at temperatures as low as –196°C.

PROGRESS AND STATUS

Introduction

A 500 Kg heat of V–4Cr–4Ti has been procured by Argonne National Laboratory from Teledyne Wah Chang Albany Corporation. This alloy is considered to be the most promising candidate vanadium alloy in the U.S. program for future fusion devices. This alloy will be used to evaluate optimization of processing, welding and fabrication, irradiation properties, and liquid metal compatibility of the low–Cr, low–Ti vanadium alloys. The present contribution is a status report of the recent work at Oak Ridge National Laboratory (ORNL) to characterize plate material 0.150 in. thick which had been annealed by the vendor at 1050°C for 2 hours.

Chemical Analysis

Chemical analysis was done by three methods to supplement the ingot analysis provided by the vendor. Table 1 shows the results reported by the vendor and subsequent analyses. The inert gas fusion analysis was performed by Leco Corp., St. Joseph, MI. The glow discharge mass analysis and the inductively coupled plasma emission (ICP) were performed at ORNL. The inert gas fusion method has been found to be reliable for interstitials. The ICP method dissolves the metal allowing an accurate solution standard to be used. This method is believed to be the most reliable for the major elements. The methods used by the vendor are not known, so no evaluation will be made of their results.

The inert gas fusion analysis provided higher concentrations of the interstitials O, N, and C. This is not inconsistent with the ingot analyses since the inert gas fusion analyses were made on a 3.8 mm warm–rolled and annealed plate. It is possible, and in fact expected, that the interstitial concentrations, especially of oxygen, would increase in processing. The glow discharge analysis is not considered reliable for the interstitial elements. Glow discharge mass analysis identified about 11 ppm Hf which was not reported by the vendor. If this later proves to be a concern for neutron economy in a future breeding fusion device, more careful analysis will have to be done. There is also a discrepancy in the Cr concentration. Since Cr is a major element, more analysis must be done to obtain more accurate values for this element. With these exceptions, the values are reasonably consistent.

Table 1. Chemical Analyses of V–4Cr–4Ti (Ht. 832665)

Element	Teledyne Analysis 3 Ingot Positions (wt. ppm)			Glow Discharge Mass Analysis (wt ppm) (±10-30%)	Inductively coupled plasma-emission (±5-10%)	Inert gas fusion analysis (wt. ppm)
Al	180	190	105	<200		
As				<3		
B	7	<5	<5	<6*		
C	64	80	94	<310		139
Ca	<10	<10	<10			
Cl	<2	<2	<2			
Cr	3.76%	3.72%	3.83%	3.0%	3.1%	
Fe	180	220	270	256		
Hf				10.8		
Mo	330	350	280	244		
N	82	80	93	<4		102
Nb	<60	60	<60	<80		
Ni				<12		
O	280	360	290	<190		383
P	<30	<30	<30	<61		
Ru				<7		
S	<10	<10	<10	40		8
Si	790	840	720	1000		
Sr				<60		
Ta				<3*		
Ti	4.16%	3.78%	3.80%	5.3%	4.1%	
V				91.6		
W				22.8		
Zr				<65		

*known contaminant

Metallography

The annealed plate was prepared using standard polishing procedures and etched with 30% HNO_3-10% HF-60% H_2O. The microstructure is shown in Fig. 1. The bonded appearance derives from the presence of stringers of particles of titanium oxycarbonitrides, which presumably became aligned during the hot extrusion process. In the vicinity of stringers, grain growth during the final anneal is impeded; in the regions between stringers grain growth is more rapid and the final grain size is much larger. The overall average grain size is around 25 µm.

Charpy Testing

Charpy impact specimens 3.33 mm square and 25.4 mm long were used for the tests. They had a 30° notch 0.51 mm deep with a root radius of 0.08 mm. The specimens were oriented such that the crack propagated perpendicular to the direction of the stringers (L–T direction). Following machining and etching, the specimens were divided into two groups: One was annealed at 950°C for 2 hours, and the other was outgassed at 400°C for one hour to remove hydrogen.

The specimens were tested in a semiautomated pendulum type Charpy impact testing system modified for testing subsize specimens.[1] The testing temperature was achieved by cold gas from liquid nitrogen for

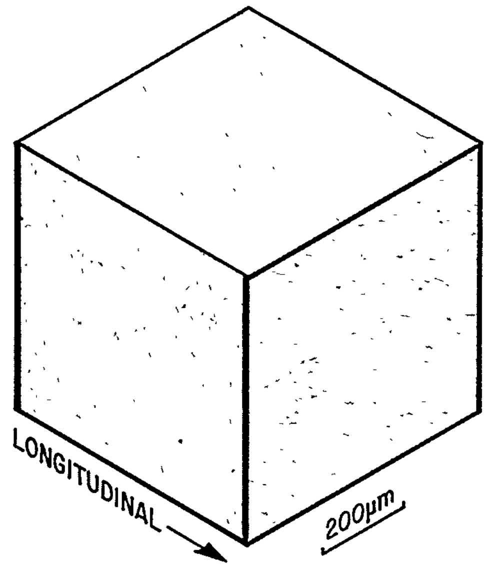

Fig. 1. Three orthogonal views of the grain structure of V–4Cr–4Ti (Ht. 832665) in the as-received annealed condition.

cooling or hot air for heating. The results of absorbed energy were fitted with a hyperbolic tangent function to allow the DBTT and the upper shelf energy to be determined at an energy level midway between the upper and lower shelf energy levels.

The results are plotted in Fig. 2 for both heat treatments. As can be seen from the figure, no ductile-to-brittle transition was observed to temperatures as low as –196°C.

DISCUSSION

The excellent Charpy impact properties shown if Fig. 2 are largely in agreement with previous results on an earlier Teledyne heat of V–4Cr–4Ti (designated BL–47 by ANL). It is also evident from the Charpy impact curves that the absorbed energy increases with decreasing temperature. This phenomenon has also been observed by Loomis et al[2] in Charpy tests of V–Cr–Ti alloys. A similar phenomenon has also been observed in tensile tests by Loomis and Carlson.[3] Such results are very similar to behavior observed by Hardie and McIntyre[4] in vanadium and niobium and by Gahr et al. in niobium.[5]

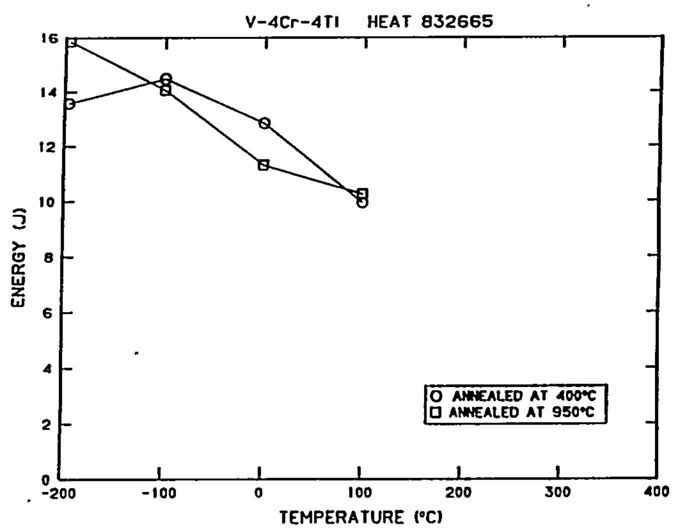

Fig. 2. Absorbed energy as a function of test temperature from Charpy impact tests of V–4Cr–4Ti (Ht. 832665) in an annealed (950°C/2 hrs.) condition and following an outgassing treatment (400°C/1 hr).

A suggested mechanism is that hydrogen is diffusing to points of stress concentration in the material initiating hydrogen embrittlement where hydrogen diffuses to the crack tip and forms brittle hydride. Another mechanism is the diffusion of hydrogen to dislocations causing a strain–aging effect. Since only hydrogen is mobile at the temperatures of minimum ductility, both of these mechanisms appear to be viable. At still lower temperatures, even hydrogen does not diffuse sufficiently fast to cause either strengthening or hydride formation, and ductility returns. In the case of the Charpy tests, the result is higher absorbed energy.

To investigate the effect of hydrogen, or to remove effects of hydrogen, one set of Charpy specimens was heat treated at 400°C for 1 hour to reduce hydrogen to very low levels. Previous experience has shown the hydrogen concentration following such heat treatment to be about one ppm or less. The results also appear in Fig. 2 where it can be seen that the process had no apparent effect. It is therefore likely that either the phenomenon is not related to hydrogen or that only fractions of ppm are sufficient to produce an effect. If either of the previously suggested mechanisms are active, lower strain rate testing should exacerbate the effect of hydrogen. This hypothesis will be tested with tensile testing in future experiments.

FUTURE WORK

Mechanical testing will continue on this particular heat of V–4Cr–4Ti with tensile and fracture toughness testing. Welding research as well as interstitial impurity studies will also be shifted to this heat.

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