ELECTRICAL RESISTIVITY AND MICROHARDNESS MEASUREMENTS OF VANADIUM AND V-4Cr-4Ti ALLOY - D.T. Hoelzer, S.J. Zinkle, and A.F. Rowcliffe (Oak Ridge National Laboratory) and M.K. West (University of Tennessee)

SUMMARY

The purpose of this study was to investigate the interactions between Ti and interstitial solutes over temperature ranges corresponding to thermally activated processes such as precipitation, dislocation recovery and recrystallization, and grain growth. In this study, room temperature electrical resistivity and microhardness measurements were performed on cold-worked (CW) vanadium, CW V-4Cr-4Ti, annealed V-4Cr-4Ti, and the fusion zone of welded V-4Cr-4Ti plate over the isochronal annealing temperature range from 200°C to 1200°C. The results suggested that Ti solutes in the vanadium alloy interacted with interstitial O,C, and N solutes at temperatures of 200°C and higher. Below ~400°C, these interactions influenced processes such as solute diffusivity and dislocation atmosphere formation. Above ~400°C, recovery, recrystallization and precipitation processes had the most significant effect on the property measurements.

INTRODUCTION

Vanadium alloys with compositions near V-4Cr-4Ti are candidate structural materials for first wall/blanket applications in fusion energy reactors because of their attractive low activation characteristics and combinations of good thermal conductivity, strength, and low ductile-to-brittle transition temperature (DBTT) in the unirradiated condition. The current fabrication practices used for producing plate and sheet products from the large heats of V-4Cr-4Ti (500 kg heat #832665 and 1200 kg heat #832864) have typically resulted in cumulative interstitial O, C, and N solute concentrations ranging from 400 to 700 appm. In general, some of the interstitial solutes react with Ti atoms to form globular-shaped Ti-oxycarbonitride (Ti-OCN) precipitates and possibly other phases such as platelet-shaped Ti-oxides and the unreacted interstitial content remains in solution in the bcc vanadium matrix. However, both the precipitates and the interstitial solutes in solution can affect the thermomechanical processing and the mechanical properties of vanadium alloys. For example, the formation of an inhomogeneous distribution of Ti-OCN precipitates during thermomechanical processing was related to formation of banded grain structures in plate products[1] and increases in DBTT[2] and observation of the dynamic strain aging[3] (DSA) phenomena has been attributed to interstitial solutes in solution.

The purpose of this report is mainly to provide the measured microhardness and electrical resistivity data, since a detailed discussion of the results from this study was presented at ICFRM-9, which will be published as a conference proceeding in the Journal of Nuclear Materials.

EXPERIMENTAL PROCEDURE

The microhardness and electrical resistivity measurements were performed on type SS-3 sheet tensile specimens with nominal lengths of 25.4 mm and gage dimensions of 0.76 x 1.52 x 7.6 mm. The specimens were electro-discharge machined from 50% CW unalloyed vanadium plate (heat #820642), 40% CW V-4Cr-4Ti (heat #832665) plate, and fusion zone of a gas tungsten arc welded V-4Cr-4Ti (alloy) plate. Half of the 40% CW V-4Cr-4Ti specimens were annealed at 1000°C for 2 hours in a vacuum of <~$3x10^{-7}$ torr.

The isochronal annealing experiment was performed with six specimens of the CW vanadium, CW alloy, and annealed alloy and three specimens of the welded alloy. Measurements were performed at room temperature after each isochronal annealing was performed at temperatures ranging from 200°C and 1200°C for 1 hour. The specimens were wrapped in tantalum foil and the annealings were performed in a vacuum of ~(0.7-1.2)x10^{-6} torr using a constant heating rate of 10°C/min and furnace cooling. A four-point probe technique covered in ASTM Standard Method of Test for Resistivity of Electrical Conductor Materials, ASTM B 193-87 (reapproved 1992) and described by Zinkle et al.[4] was used to make five measurements per specimen, which were averaged and corrected to a reference temperature of 20°C. The typical standard error (SE) of the mean for the five measurements was ±0.5 nΩ-m. The SE of the mean ranged from ±0.3 to ±1.4 nΩ-m for resistivity measurements of specimens within each category. A Vickers pyramidal indenter with a 1 kg load was used to make 2 to 4 indents per specimen near the end tab region of the SS-3 tensile specimens. The CW and annealed alloy specimens consistently showed less scatter in hardness values compared to the CW vanadium and welded alloy specimens. The typical SE of the mean was ±3 DPH for the CW and annealed alloy, ±5 DPH for the CW vanadium, and ±8 DPH for the welded alloy specimens.

RESULTS AND DISCUSSION

Chemical Analysis

The chemical analysis of the interstitial O, C, and N contents on selected specimens before the isochronal annealing and after the final annealing temperature of 1200°C is shown in Table 1. The results obtained for the CW vanadium and welded V-4Cr-4Ti specimens indicated that no substantial uptake of O, C, and N impurities occurred during the isochronal annealing and property testing. The interstitial content measured in these specimens are more representative of the testing conditions since chemical analysis was conducted on SS-3 tensile specimens before and after the testing. This was not the case for the CW V-4Cr-4Ti and annealed V-4Cr-4Ti specimens. The SS-3 tensile specimens of these material showed substantial increases in O, N and C content compared to interstitial contents reported for the ingot (heat #832665). It is possible that much of the contamination occurred during thermomechanical processing of the ingot into plate and then into the SS-3 tensile geometry.

Hardness and Electrical Resistivity Measurements

Figure 1 shows the room temperature hardness and electrical resistivity measurements of the CW vanadium specimens. The significant points shown in this Figure are, the increase in hardness of ~26 DPH and decrease in electrical resistivity over the initial temperature range from 200°C to ~400°C; the decrease in hardness above temperatures of 400°C, minima at ~1000°C,

Table 1. Chemical analysis of specimens used in the property measurement study.

Material	Annealing	Specimen	O (wppm)	N (wppm)	C (wppm)
50% CW Vanadium	Before	SS-3	340	177	198
	After	SS-3	320	222	157
40% CW V-4Cr-4Ti	Before	Ingot*	310	85	80
	After	SS-3	492	109	196
Annealed V-4Cr-4Ti	Before	Ingot*	310	85	80
	After	SS-3	390	102	165
Welded V-4Cr-4Ti	Before	SS-3	457	95	235
	After	SS-3	484	108	225

* Chemical analysis has not been performed on SS-3 tensile specimens of the V-4Cr-4Ti alloy.

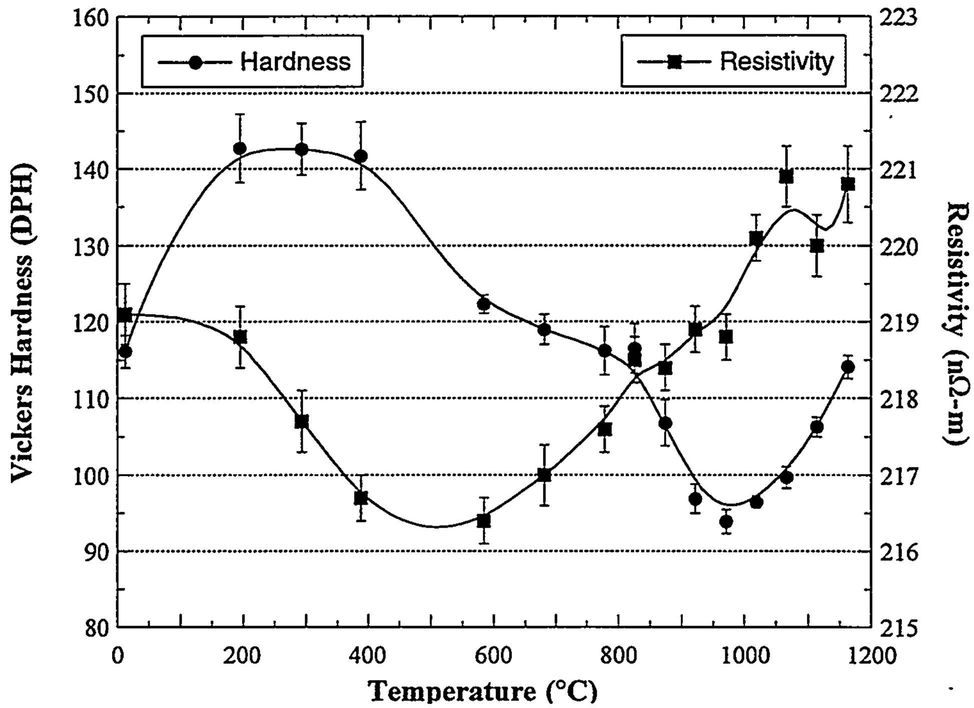

Figure 1. Hardness and electrical resistivity measurements of the 50% cold-worked vanadium specimens over the isochronal annealing temperature range from 200°C to 1200°C.

and subsequent increase in hardness above 1000°C; and the minima in electrical resistivity between ~400°C to 600°C and subsequent increase at higher temperatures.

Figure 2 and 3 show the measured hardness and electrical resistivity for CW V-4Cr-4Ti and annealed V-4Cr-4Ti specimens, respectively. The room temperature measurements indicated that cold-working caused a significant increase in hardness and only a small increase in electrical resisitivity as observed by comparing Figure 2 (CW) to Figure 3 (annealed). For the annealed specimens, the measured hardness was 139.7 DPH and electrical resistivity was 285.1 nΩ-m. Compared to the cold worked specimens, the measured hardness increased by 71 DPH to 210.7 DPH while the resistivity increased by 1.1 nΩ-m to 286.3 nΩ-m.

The most significant result observed for the annealed specimens shown in Figure 3 was the positive change in electrical resistivity compared to room temperature over the entire isochronal annealing temperature range from 200°C to 1200°C. A initial increase in electrical resistivity of ~5 nΩ-m was measured after annealing at 200°C where it then remained positive up to 1200°C. However, no significant change in hardness was observed over this temperature range except for a small peak between 600°C and 900°C. The measured properties for the CW specimens shown in Figure 2 showed considerable departure from those measured for the annealed specimens (Figure 3). Although the electrical resistivity initially increased with annealing temperature, reaching a maximum of 3.6 nΩ-m at 300°C, it then decreased at higher annealing temperatures. The electrical resistivity then reached a minima between 700° to 800°C and subsequently increased up to 1200°C. The hardness showed no appreciable change from the room temperature value until a small peak occurred near 700°C followed by a steady decrease above 700°C. A minimum in hardness was measured near 1050°C to 1100°C which was followed by a small increase at the highest temperatures.

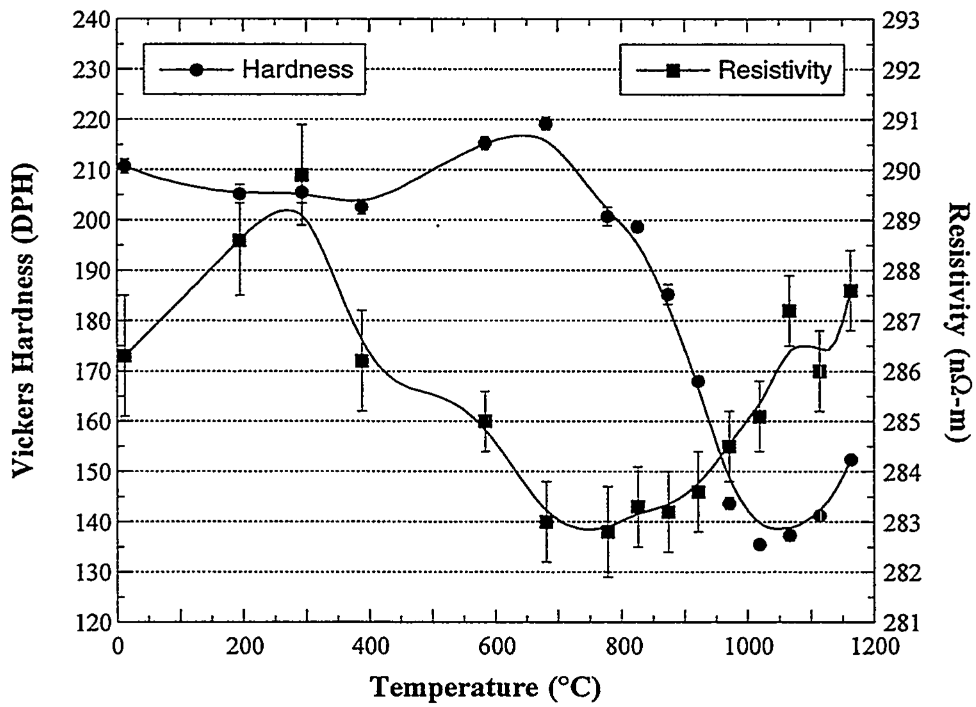

Figure 2. Hardness and electrical resistivity measurements of the 40% cold-worked V-4Cr-4Ti specimen over the isochronal annealing temperature range from 200°C to 1200°C.

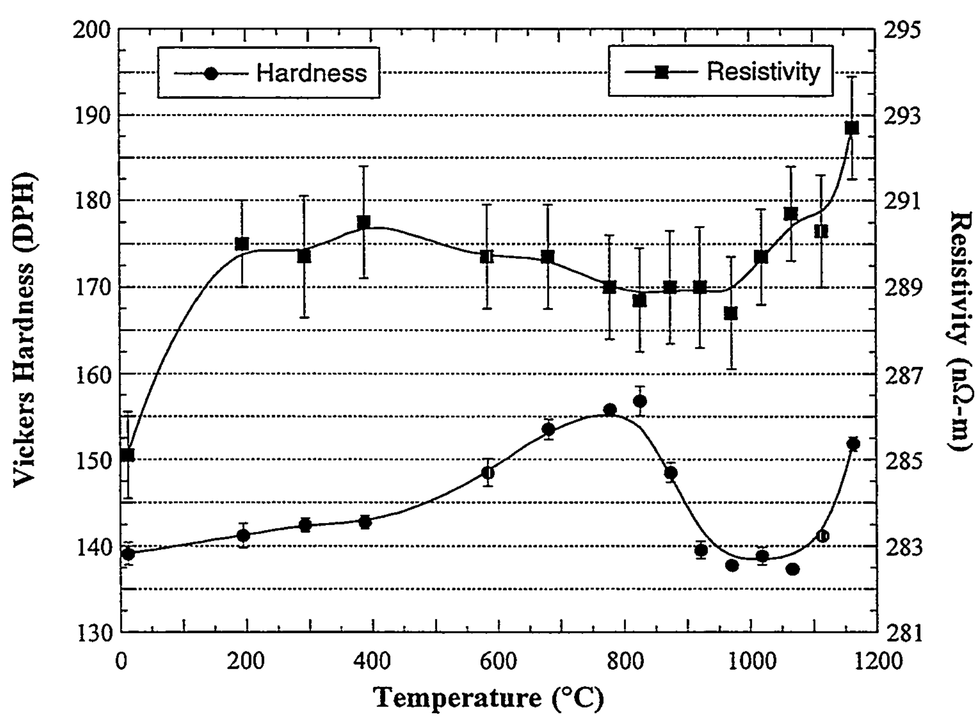

Figure 3. Hardness and electrical resistivity measurements of the annealed V-4Cr-4Ti specimen over the isochronal annealing temperature range from 200°C to 1200°C.

The temperature dependence of hardness and resistivity measured for the GTA weld specimens is shown in Figure 4. The measurements at room temperature showed that the hardness was 213.4 DPH and resistivity was 294.2 nΩ-m. The results showed that isochronal annealing caused the formation of a minor and major hardness peak near 200°C and 700°C, respectively, a minimum in hardness near 1100°C, and a continuous decease in resistivity from the room temperature value to a minimum of ~283.5 nΩ-m that remained nearly constant from 800°C to 1200°C.

The results showing the exostence of the hardness maxima from ~700°C to 800°c in the CW alloy (Figure 2) and weld alloy (Figure 4) specimens was attributed to a precipitation reaction. This precipitation reaction is believed to be associated with formation of Ti-oxide phase. In general, the electrical resistivity decreases over this temperature range and is consistent with the idea that interstitial solutes are being removed from the matrix by the precipitation reaction. However, recrystallization is occurring at temperatures above 700°C. This process also causes a decrease in the electrical resistivity since the dislocation density rapidly decreases during this process. Further work is in progress in order to clarify whether precipitation is occurring along with recrystallization in this temperature range.

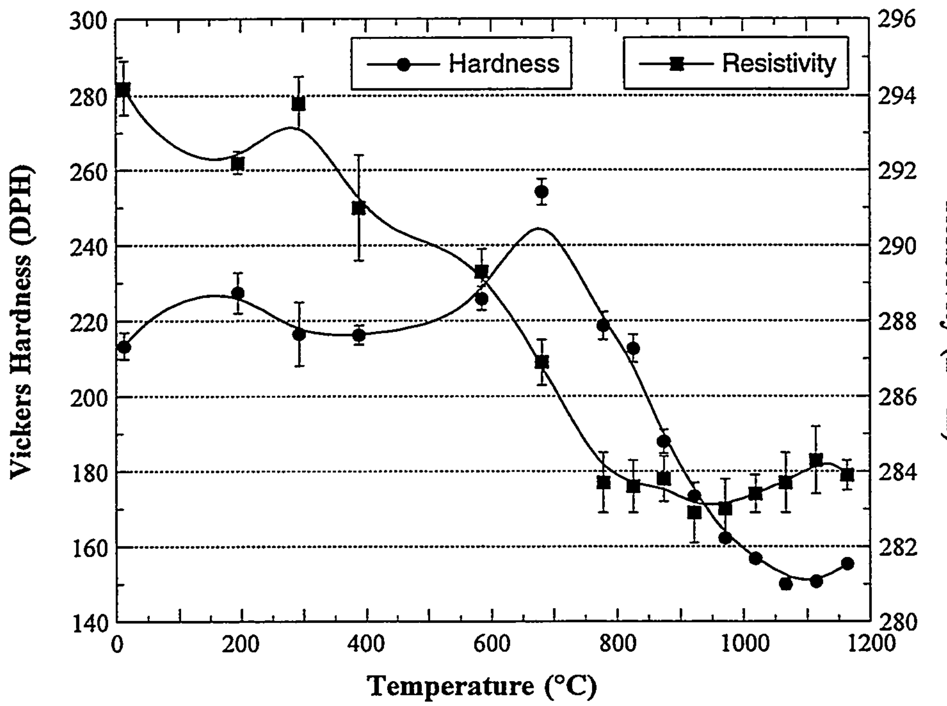

Figure 4. Hardness and electrical resistivity measurements of the welded V-4Cr-4Ti specimen over the isochronal annealing temperature range from 200°C to 1200°C.

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