DISLOCATION DEVELOPMENT IN V-5CR-5TI AND PURE VANADIUM - D. S. Gelles, (Pacific Northwest Laboratory)[a] and M. L. Grossbeck, (Oak Ridge National Laboratory)

OBJECTIVE

The objective of this work is to explain notch sensitivity noted in the candidate alloy V-5Cr-5Ti.

SUMMARY

Microstructural examinations have been performed on deformed tensile specimens of V-5Cr-5Ti and pure vanadium in order to explain notch sensitivity noted in the candidate alloy V-5Cr-5Ti. SS-3 tensile specimens have been prepared, stress relieved and deformed to 5% strain. The resulting deformation structures have been examined by transmission electron microscopy. It is found that 5% deformation in V-5Cr-5Ti produces a higher dislocation density consisting of long straight dislocations, typical of Stage II, and many small loops, whereas in pure vanadium, the dislocation arrangements are more complex, typical of Stage III, and the small loops are at a lower density.

These results are interpreted in light of the tendency for enhanced notch sensitivity found in V-5Cr-5Ti.

PROGRESS AND STATUS

Introduction

A vanadium alloy in the composition range V-5Cr-5Ti is being considered as an alternate structural material for the International Thermonuclear Experimental Reactor (ITER) design.[1] Research and Development task 5.4.1.1 calls for compatibility and irradiation tests for vanadium alloys. Vanadium may provide a major improvement in performance in comparison with other options such as austenitic and ferritic steels because of inherently better high temperature properties and inherently reduced long term radioactivity after neutron irradiation. However, a great deal remains to be learned about the behavior of this alloy class before it can be code qualified for reactor construction.

Recently, efforts have been started to quantify fracture toughness response in V-5Cr-5Ti.[2] Those efforts demonstrated that at 100°C, the fracture toughness remains very high and shows response similar to the very tough Martensitic steel F-82H, whereas at 25°C, toughness is much lower. Also, both preliminary fracture toughness testing and fractographic examination suggested that V-5Cr-5Ti is notch sensitive.[2] Similar indications can be deduced from V-5Cr-5Ti Charpy impact specimens tested with a range of notch root radii, and may indicated that V-5Cr-5Ti has a higher notch sensitivity than ferritic steels.

As a part of the effort to understand the notch sensitivity of V-5Cr-5Ti, the present work was undertaken. The original concept was to examine deformation behavior in V-5Cr-5Ti by deforming single crystals and comparing the response to pure vanadium. The intent was to determine if the number of slip planes was reduced, or if some other details could be identified in the dislocation structure to explain the behavior. However, the work scope was reduced by limiting experiments to polycrystalline material and making use of V-5Cr-5Ti miniature tensile specimens already available. Pure vanadium was tested for comparison.

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Experimental Procedure

Pure vanadium sheet 1.44 mm in thickness was provided by R. Peterson, Teledyne Wah Chang, Albany, Oregon. V-5Cr-5Ti sheet 1 mm in thickness was from heat BL63 #832394, also produced by Teledyne Wah Chang. Composition information is given in Table 1. Specimens to SS-3 specifications with a gage section 1.52 x 0.76 x 7.62 mm in length were machined from the sheet, etched in 10% hydrofluoric acid and 30% nitric acid in water, and annealed for 1 hour at 1125°C. During heat treatment, they were wrapped in tantalum foil in a vacuum below 10^{-5} Pa.

Table 1. Composition of pure vanadium and V-5Cr-5Ti (Heat BL63 #832394) as supplied by Teledyne Wah Chang.

Alloy	Composition in ppm unless noted, balance Vanadium								
	Al	B	C	Cr	Fe	H	Hf	Mo	Ni
Pure Vanadium	<300	<10	<100	<20	<200	<5	<100	<200	<20
V-5Cr-5Ti	200	<5	73	4.6%	<370	NA	NA	500	NA
	Nb	O	P	S	Si	Ta	Ti	U	Zr
Pure Vanadium	<250	<400	<100	<20	<400	<300	<50	<2	<100
V-5Cr-5Ti	<50	<5	<30	NA	310		5.1%	<1	NA

Tensile specimens were deformed to 5% strain at a strain rate of $1.1 \times 10^{-3}\ s^{-1}$ at room temperature. The gauge section was cut into 3 mm sections. Specimens were prepared for transmission electron microscopy using a Tenupol twin jet electropolishing device with a solution of 5% sulfuric acid in methanol at 40 V and moderate pump speed. Foils with acceptable thin area were possible using a small dimple size. Cleaning was in methanol.

Microstructural examinations used a JEM 1200EX transmission electron microscope equipped with a double tilting goniometer stage. Procedures involved a series of dislocation imaging conditions using $\vec{g}$ = 200 as a stereo pair, 01$\bar{1}$, 21$\bar{1}$, 2$\bar{1}$1, 1$\bar{2}$1, 12$\bar{1}$, and $\bar{1}$10, (or the equivalent) for a foil near an [011] orientation. This notation is defined in Figure 1, and the resultant imaging conditions are defined in Table 2. A review of Table 2 will show that all $\frac{a}{2}$<111> Burgers vectors are imaged using $\vec{g}$ = 200, half are imaged with $\vec{g}$ = 110, and for each of the $\vec{g}$ = 211 images, one Burgers vector is invisible, and one is in stronger contrast than the remaining two. From the series of imaging conditions used in the present work, shown in bold in Table 2, all Burgers vectors can be uniquely identified. The procedure will be described in greater detail in the results section.

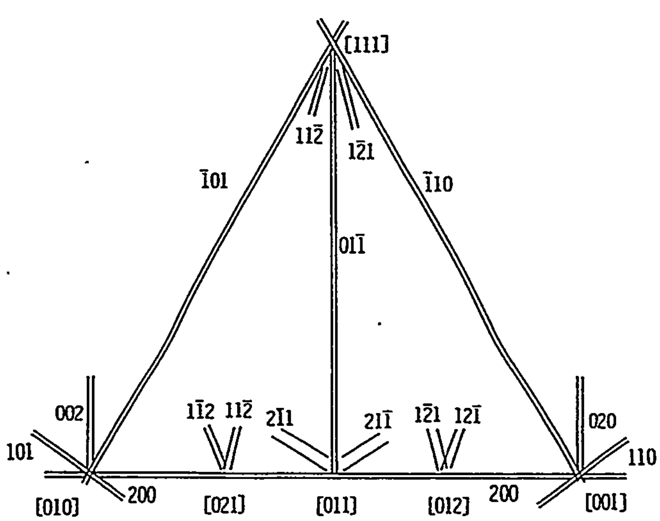

Figure 1. Definition of notation as represented on a Kikuchi map for bcc dislocation imaging conditions.

Table 2. The $\vec{g} \cdot \vec{b}$ imaging criteria for various bcc imaging conditions and Burgers vector combinations.

$\vec{g}$	Burgers vector			
	$\frac{a}{2}[111]$	$\frac{a}{2}[\bar{1}11]$	$\frac{a}{2}[1\bar{1}1]$	$\frac{a}{2}[11\bar{1}]$
200	**1**	**1**	**1**	**1**
020	1	1	1	1
002	1	1	1	1
$01\bar{1}$	**0**	**0**	**1**	**1**
$\bar{1}10$	**0**	**1**	**1**	**0**
$\bar{1}01$	0	1	0	1
101	1	0	1	0
$11\bar{2}$	0	1	1	2
$1\bar{2}1$	**0**	**1**	**2**	**1**
$2\bar{1}1$	**1**	**1**	**2**	**0**
$21\bar{1}$	**1**	**1**	**0**	**2**
$1\bar{1}2$	1	0	2	1
$12\bar{1}$	**1**	**0**	**1**	**2**

Results

Tensile Testing

The tensile test traces for specimens deformed to 5% strain are shown in Figure 2. Figure 2a shows response for pure vanadium strained to 5% and indicates load drop behavior. The upper yield strength was 409.5 MPa (59.4 ksi), and the tensile strength at 5% strain was 435.7 MPa (63.2 ksi). Figure 2b shows similar response for V-5Cr-5Ti with a slightly higher yield strength, but no yield drop and at a lower work hardening rate. The yield strength was 421.3 MPa (61.1 ksi), and the tensile strength at 5% strain was 421.3 MPa (73.6 ksi).

Microstructures

Dislocation development as a result of 5% deformation at room temperature was found to differ as a function of alloying. Pure vanadium developed a complex dislocation structure typical of stage III deformation (forest dislocation development) with large spaces between tangles, whereas V-5Cr-5Ti dislocation structures were less complicated but more uniformly distributed, often containing long straight dislocations typical of stage II. Both materials also showed dislocation loop formation with loop sizes on the order of 10 nm in diameter. Slip band orientations could not be identified based on stereoscopic examination of the dislocation structures.

Examples of these dislocation structures following deformation are shown in Figures 3 and 4. Each figure is arranged to provide a stereo pair using $\vec{g} = 200$ taken near the (011) orientation in Figures 3 a) and b), using $\vec{g} = 01\bar{1}$ near (011) in 3 c), $\vec{g} = \bar{1}10$ near (112) in 3 d), and $\vec{g} = 21\bar{1}$, $2\bar{1}1$, $1\bar{2}1$, and $12\bar{1}$, in 3 e), f), g) and h), respectively.

From Figure 3 for pure vanadium, it is apparent that the dislocation structure in deformed pure vanadium consists of dislocation tangles and isolated loops. Comparing the dislocation images in Figure 3, the following can be demonstrated. Several of the possible Burgers vectors are present, as different imaging conditions show different structures. The Burgers vectors can be determined based on the information in Table 2. For example, the dislocations in Figures 3 c) and 3 d) are different, but all can be found in Figures 3 a) and b). Therefore, the dislocations are indeed of type $\frac{a}{2}\langle 111\rangle$, and the dislocations appearing only in 3 c) have Burgers vector $\frac{a}{2}[11\bar{1}]$, and the dislocations appearing only in 3 d) have Burgers vector $\frac{a}{2}[1\bar{1}1]$. {Those appearing in both 3 c) and d) have Burgers vector $\frac{a}{2}[1\bar{1}1]$, and those appearing in 3 a) and b) but not in 3 c) and d) have Burgers vector $\frac{a}{2}[111]$.} Similarly, the loops are predominantly of Burgers vector $\frac{a}{2}[11\bar{1}]$, but examples of $\frac{a}{2}[1\bar{1}1]$ can be noted at the upper right in Figure 3 d). These conclusions can be verified from Figures 3 e) to h) by noting that $\frac{a}{2}[1\bar{1}1]$ dislocations do not appear, and $\frac{a}{2}[11\bar{1}]$ are in strong contrast in Figure 3 e); $\frac{a}{2}[11\bar{1}]$ dislocations do not appear, and $\frac{a}{2}[1\bar{1}1]$ are in strong contrast in Figure 3 f); $\frac{a}{2}[111]$ dislocations do not appear, and $\frac{a}{2}[1\bar{1}1]$ are in strong contrast in Figure 3 g); and $\frac{a}{2}[\bar{1}11]$ dislocations do not appear, and $\frac{a}{2}[11\bar{1}]$ are in strong contrast in Figure 3 h).

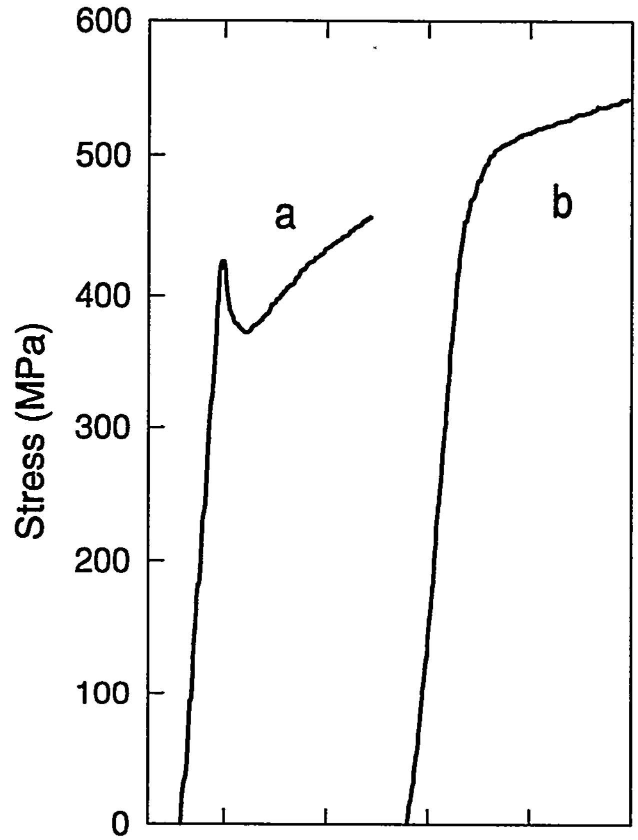

Figure 2. Stress strain curves for a) pure vanadium and b) V-5Cr-5Ti deformed at room temperature to 5% strain.

An example of the dislocation structures found in V-5Cr-5Ti are shown in Figure 4. The imaging conditions match those in Figure 3, but the dislocation structures are different. Dislocations appear as straight parallel lines, and many loops are visible. Burgers vector analysis gives similar results, however, with the highest density of Burgers vector type $\frac{a}{2}[11\bar{1}]$, and type $\frac{a}{2}[1\bar{1}1]$ at lower density.

Microstructural Analysis

The dislocation and loop structures were quantified, and the results are provided in Table 3. From Table 3, it can be shown that the dislocations structures of the two conditions examined are similar in many ways, the major difference being a somewhat higher dislocation density, and a factor of three times higher loop density for V-5Cr-5Ti. It can be noted for example, the most prevalent Burgers.vector present is the same in both cases and by a factor of two, that being $\frac{a}{2}[11\bar{1}]$.

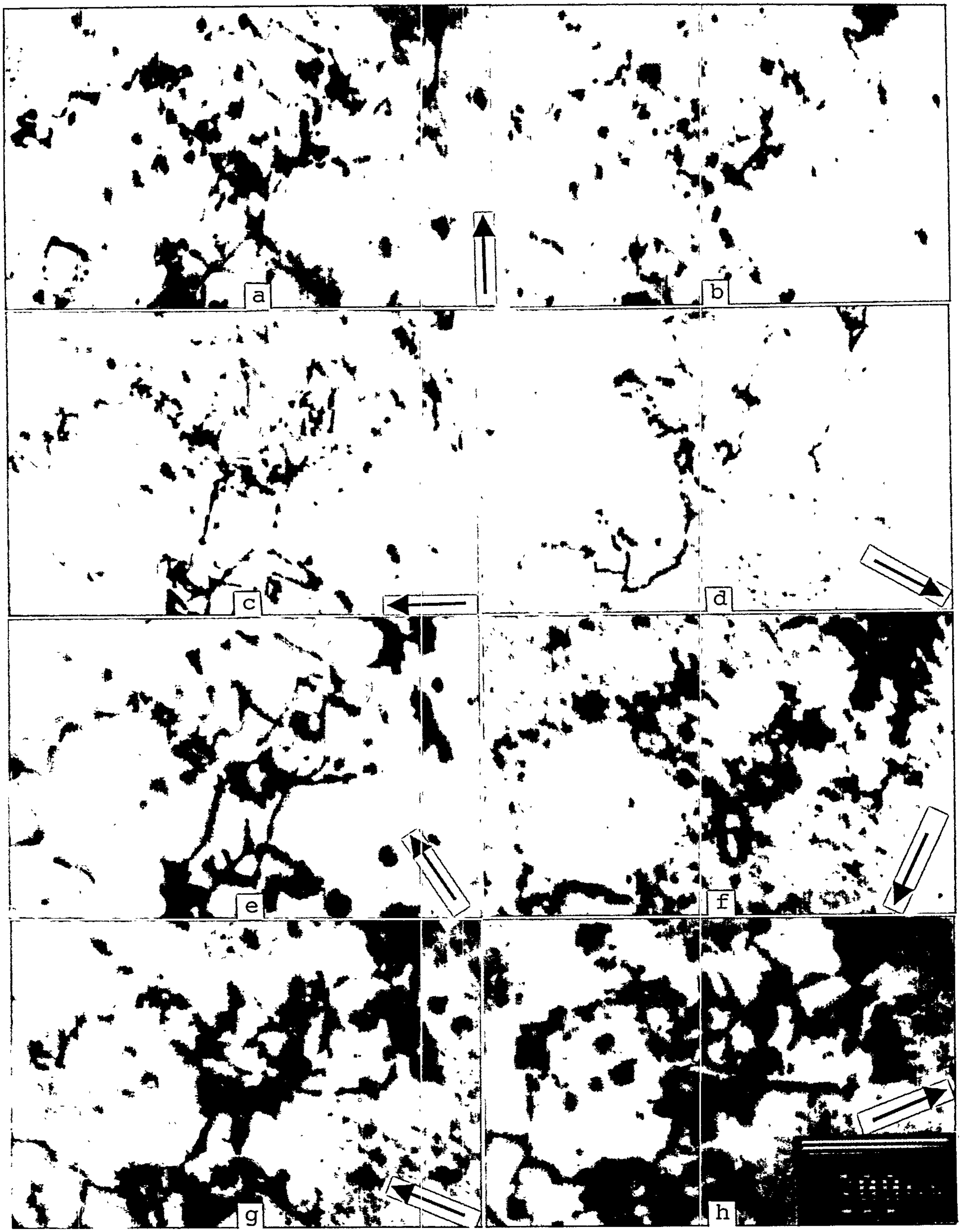

Figure 3. Dislocations in pure vanadium strained 5% at room temperature under the following contrast conditions: a) 200 near (011), b) 200 near (013), c) $01\bar{1}$ near (011), d) $\bar{1}10$ near (112), e) $21\bar{1}$ near (011), f) $2\bar{1}1$ near (011), g) $1\bar{2}1$ near (013), and h) $12\bar{1}$ near (013).

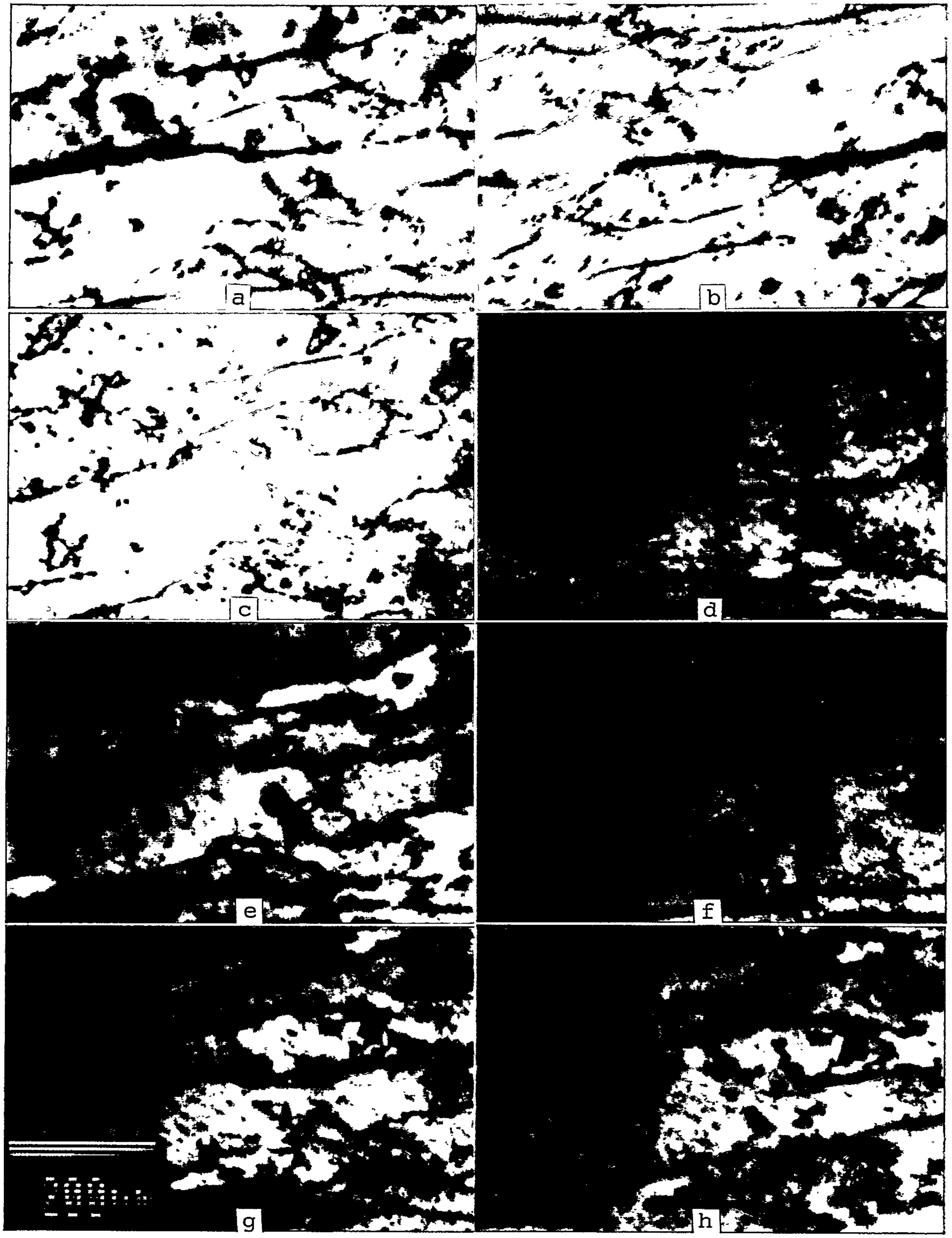

Figure 4. Dislocations in V-5Cr-5Ti strained 5% at room temperature under the following contrast conditions: a) 200 near (011), b) 200 near (013), c) $01\bar{1}$ near (011), d) $\bar{1}10$ near (112), e) $21\bar{1}$ near (011), f) $2\bar{1}1$ near (011), g) $1\bar{2}1$ near (013), and h) $12\bar{1}$ near (013).

Table 3. Results of quantitative dislocation analyses.

Alloy	Dislocations (cm^{-2})					Loops	
	$\frac{a}{2}[111]$	$\frac{a}{2}[\bar{1}11]$	$\frac{a}{2}[1\bar{1}1]$	$\frac{a}{2}[11\bar{1}]$	Total	# Density (cm^{-3})	Mean Diameter (nm)
Pure Vanadium	$1.2x10^8$	$1.4x10^9$	$1.2x10^8$	$2.7x10^9$	$4.3x10^9$	$6.2x10^{14}$	11.9
V-5Cr-5Ti	N.M.	$2.3x10^9$	N.M.	$4.2x10^9$	$6.5x10^9$	$1.8x10^{15}$	8.1

N.M. = not measured, too small to measure given the limited statistics

Discussion

Slip modes in vanadium and its alloys

Dislocation development in vanadium and its alloys has been reported a number of times. Edington and Smallman showed typical microstructures as a function of test temperature in polycrystalline vanadium.[3] The predominant slip response was later documented by private communication[4] as follows: both at room temperature and at low temperature, {110}(T) and {112}(T). Russian work was on slip in single crystals of vanadium for the temperature range 150 to 400K oriented to favor $(112)[\bar{1}\bar{1}1]$ slip in Stage I, with $(\bar{2}11)[111]$ coming into play in Stage II.[5] As a function of temperature, slip began on $(1\bar{1}0)[111]$ at 150K, but on $(112)[\bar{1}\bar{1}1]$ and $(\bar{2}11)[111]$ at higher temperatures. The Stage II dislocation structures were similar to those found in the present study in V-5Cr-5Ti (Figure 4) whereas the Stage III dislocation structures were similar to those for pure vanadium (Figure 3). In contrast, Belgian work on pure vanadium showed that slip in single crystals of vanadium in the temperature range 77 to 298K was on {101} at low temperatures and was noncrystallographic at 298K, deviating towards the $(\bar{1}01)$ plane.[6] Slip on {112}-type planes was rarely observed. The analysis was based on slip trace analysis. Most recently, the Russian results were reviewed,[7] including results for two vanadium alloys: at low temperature, the $(011)[11\bar{1}]$ system operated in the range 77 to 150K for vanadium, in the range 77 to 270K for V-5Ta and in the range 77 to 300K for V-25Ta whereas the $(112)[11\bar{1}]$ system operated in the range 120 to 650K for vanadium, and in the range 250 to 650 for V-5Ta and V-25Ta. The work also assesses the transition temperature to form a cellular dislocation structure and notes that this occurs at 320K in vanadium, but at 350 and 560K in V-5Ta and V-25Ta, respectively.

Explanation for notch sensitivity in V-5Cr-5Ti

The present effort has provided several results that may be relevant to the question of increased notch sensitivity in V-5Cr-5Ti. Those results will be summarized, and then the results will be considered in light of their effect on notch sensitivity.

Tensile tests to 5% deformation show that in comparison to pure vanadium, V-5Cr-5Ti has a higher yield strength and a lower work hardening rate, but no yield drop. A higher yield strength should correspond to a smaller crack tip plastic zone size, and the lower work hardening rate can be expected to provide higher toughness. The lack of a yield drop can also be expected to enhance toughness.

Microstructural examinations demonstrate that Stage II deformation is maintained to 5% strain in V-5Cr-5Ti, whereas in pure vanadium, Stage III behavior is found at 5% strain. Also, the density of loops is found to be higher in V-5Cr-5Ti. The observation of Stage II response in V-5Cr-5Ti is likely a consequence of a lower work hardening rate and may be a measure of a tendency to avoid cross slip, but the increased loop density is expected to be a measure of strain centers arising from the presence of increased titanium levels and the resultant tendency for formation of Ti(C,N,O) complexes.

It can be noted that the tendency for reduced cross slip and delay in the transition to Stage III can be expected to be related to a lower stacking fault energy.[8] Noskova[9] has shown that as a function of increasing tantalum in vanadium, the stacking fault energy is reduced, from 80 to 100 MJ/m^2 for pure vanadium, from 73 to 80 MJ/m^2 for V-5Ta and from 37 to 70MJ/m^2 for V-15Ta. Similar reductions in stacking fault energy can be expected to arise with additions of titanium and chromium, although no measurements are available to support such an assumption. However, it can also be noted that the work hardening rate is generally inversely related to stacking fault energy so that high work hardening rates arise when stacking fault energies are low, in opposition to the present situation. Also, Stage III work hardening rates are lower than Stage II. Therefore, the lower work hardening rate found in V-5Cr-5Ti must be a consequence of some other factor such as reduced interstitial hardening due to the presence of titanium gettering.

Increases in notch sensitivity are likely to arise from three effects: interstitial impurity hardening, grain boundary segregation, and large precipitate particles ahead of the crack tip. The present work only points to an effect due to the interaction of a higher stress field (associated with a sharper crack) with crack nucleation sites ahead of the propagating crack. If sufficient potential nucleation sites for cavity formation exist, then a higher stress state will be more likely to activate cavitation ahead of the propagating crack. Conversely, notch insensitivity can be expected when the crack tip plastic zone size is small compared to the spacing of potential crack nucleation sites. Therefore, key factors affecting notch sensitivity can be expected to be plastic zone size and spacing between potential cavity nucleation sites.

The present work does not provide clear guidance on the effect of alloying on plastic zone size. Strength is increased due to alloying, but stage II is prolonged to higher strains, and work hardening rates are reduced. The relative increase in strength is small due to the avoidance of yield drop behavior. Therefore, it is likely that the plastic zone sizes for pure vanadium and V-5Cr-5Ti are similar.

The most likely reason for increased notch sensitivity in V-5Cr-5Ti can be traced to potential cavity nucleation site density. Alloying is expected to promote the formation of Ti(C,N,O) complexes, and microstructural observation of enhanced loop density following deformation is interpreted as evidence supporting this assertion. However, the sites promoting loop formation are probably too small to be potential cavity nucleation sites. A more reasonable feature for a cavity nucleation site is larger Ti(C,N,O) precipitate particles, approximately 100 nm or larger in diameter, that can easily be found in V-5Cr-5Ti. The distribution of these larger precipitate particles can be expected to be altered by heat treatment in the temperature range 900°C and above. Therefore, it can be tentatively concluded that enhanced notch sensitivity in V-5Cr-5Ti is a consequence of the scavenging effect of titanium additions and the resultant increase in the density of potential cavity nucleation sites. This density of sites can be altered, at least to some degree, by suitable heat treatment.

CONCLUSIONS

Deformation response of V-5Cr-5Ti has been compared with that of pure vanadium. It is found that alloying increases yield strength, reduces work hardening rate, and avoids yield drop response. Following deformation to 5% strain, Stage II response is maintained in the alloy, whereas Stage III is reached without alloying. Alloying also increases the density of dislocation loops formed during deformation. These results are interpreted to conclude that alloying does not significantly alter crack tip plastic zone size, whereas it can significantly increase Ti(C,N,O) precipitation and therefore potential sites for cavity nucleation.

FUTURE WORK

This work will be continued when warranted.

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