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Investigating solute interactions in V–4Cr–4Ti based on tensile deformation behavior of vanadium

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Abstract

Tensile tests were carried out on annealed, unalloyed vanadium from 200 to 500 °C at strain rates ranging from 10^{-1} to 10^{-5} s^{-1} to investigate the interaction between Ti and interstitial O, C, and N solutes in the V–4Cr–4Ti alloy. Dynamic strain aging (DSA) is manifested in unalloyed vanadium at 300 and 400 °C as serrations (continuous) and discontinuous yielding in the Lüders strain and work hardening regimes of stress–strain curves with a concomitant negative value in strain rate sensitivity (SRS) for flow stresses. Diffusion data indicate that the DSA phenomenon at temperatures below 400 °C is related to the migration of carbon and oxygen while at higher temperatures nitrogen becomes increasingly important. DSA occurs in V–4Cr–4Ti within the temperature range from 300 to 750 °C (J. Nucl. Mater 283–287 (2000) 508). The data shows that alloying with Ti and Cr solutes shifts the maximum negative SRS from 300 °C in vanadium to ∼600 °C in the alloy.

1. Introduction

A vanadium alloy containing 4%Cr and 4%Ti (wt%) is currently being assessed by the US Fusion Materials Program as a potential structural material. The mechanical behavior of the V–4Cr–4Ti alloy and of BCC alloys in general is strongly influenced by interstitial C, O, and N solutes in solid solution. In a previous study [1], the deformation behavior of the V–4Cr–4Ti alloy was shown to exhibit dynamic strain aging (DSA) during tensile testing, typically at temperatures ranging from 300 to 750 °C and strain rates ranging from 10^{-1} to 10^{-5} s^{-1}. Fig. 1 shows stress–strain curves determined for temperatures ranging from 20 to 850 °C at a strain rate of 10^{-3} s^{-1} for the V–4Cr–4Ti alloy. The DSA phenomenon is manifested as serrations (continuous) and discontinuous yielding in the Lüders strain and work hardening regimes of stress–strain curves and a concomitant negative value in strain rate sensitivity (SRS) for flow stresses. The present work was undertaken to explore the DSA behavior in unalloyed vanadium, which will provide the basis for understanding the interaction between Ti and interstitial C, O, and N solutes and for developing an approach to improved microstructural control of V-based alloys.

2. Experimental

Sheet tensile specimens of the SS-3 type (nominal gage dimensions 0.76 × 1.52 × 7.6 mm) were prepared from the vanadium ingot (Teledyne Wah-Chang heat no. 820642) used in producing the 500 kg heat of V–4Cr–4Ti (Teledyne Wah-Chang heat no. 832665). The specimens were electro-discharge machined from 40% cold-rolled plate material and annealed at 1000 °C for 1 h in a vacuum of $<4 \times 10^{-10}$ bar followed by furnace cooling. The processing, resulted in a uniform microstructure with a grain size of ≃89 μm, a hardness of ≃87 DPH, and a room temperature resistivity value of 209 Ω m. The interstitial contents were 25 C, 213 O, and 153 N (wt ppm). Tensile testing was carried out under a vacuum of 3×10^{-10} bar using a screw-driven machine. Specimens were held at the test temperature for 20–30

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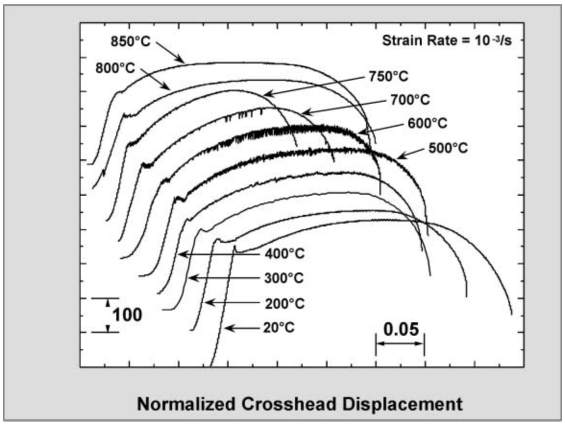

Fig. 1. Tensile curves for annealed V–4Cr–4Ti at a strain rate of 10^{-3} s^{-1} illustrating DSA regime; curves offset on stress and strain axes for clarity.

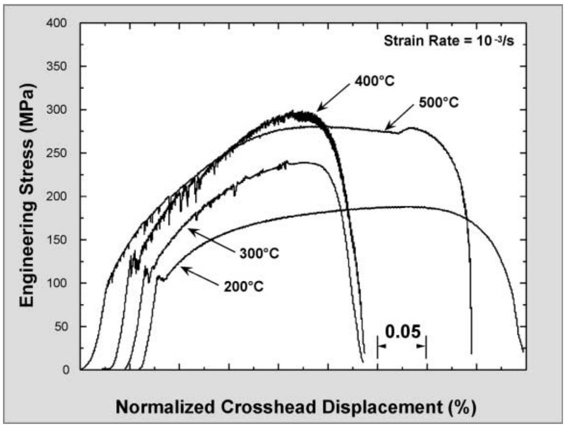

Fig. 2. Tensile curves for annealed vanadium at a strain rate of 10^{-3} s^{-1} illustrating DSA regime; curves offset on strain axis for clarity.

min before starting the test. Data were acquired digitally at rates of 20 points per second (pps) for tests conducted at 10^{-1} and 10^{3} s^{-1} strain rates and 0.2 pps at 10^{-5} s^{-1} strain rate.

3. Results

The tabulated data of tensile properties obtained from testing at temperatures ranging from 200 to 500 °C and strain rates ranging from 10^{-1} to 10^{-5} s^{-1} may be found in [2]. Engineering stress–strain curves illustrating the deformation behavior of vanadium within this temperature range at a strain rate of 10^{-3} s^{-1} are shown in Fig. 2. For clarity, the original curves are offset along the strain (normalized crosshead displacement) axis for comparison of the elastic and Lüders extension regimes.

Most of the stress–strain curves exhibited Lüders extensions after the elastic regime and continuous oscillations (serrations) and discontinuous yielding in the work hardening regime; the magnitude and spacing of these yieldings were influenced by the temperature and strain rate. Lüders extensions ranging from 0.5% to 1.0% were observed in curves at 200–400 °C at all strain rates but only at a strain rate of 10^{-5} s^{-1} at 500 °C. Serrations associated with the Lüders extension are propagated by an average stress which is defined as the lower yield stress, σ_y. Upon completion of the Lüders extension, the deformation proceeds with initially an increase in the work hardening rate on the stress–strain curve that eventually decreases until the ultimate stress, σ_u, is achieved. For temperatures between 200 and 500 °C, and at sufficiently low strain rates, deformation occurs inhomogeneously in the form of serrations and discontinuous yielding in various regions of strain in the flow curves (Fig. 2). These serrations and discontinuous yieldings are related to the diffusion of solute atoms and the locking of dislocations during the test, i.e., the phenomenon of DSA.

4. Discussion

Plastic deformation occurs in most metals and alloys by thermally activated dislocation slip processes. The flow stress during deformation is influenced by temperature and strain rate and is described by the SRS parameter as follows [3]:

$$m = \frac{1}{\sigma}\frac{\delta\sigma}{\delta \ln \dot{\varepsilon}}|_{\varepsilon,T}. \tag{1}$$

Normally, the flow stress (defined here as the stress required to produce a plastic strain of ε) increases with increasing strain rate at a constant temperature, i.e. m is positive. Deformation under this condition is homogeneous and stress–strain curves exhibit smooth flow stresses as a function of strain. However, within certain temperature and strain rate regimes, DSA may occur and is manifested by oscillations in the flow stress on stress-strain curves and concomitant negative values of m. The DSA phenomenon is related to the diffusion of solute atoms to immobile dislocations to form Cottrell atmospheres that become barriers to dislocation movement during plastic deformation. The oscillations observed in the flow stress can be attributed to repetitive localized yielding in dilute solutions involving the creation of a slip band (yield drop) and subsequent propagation until stopped once again at obstacles in the slip path (stress rise) during deformation.

The results show the addition of substitutional 4%Cr and 4%Ti solutes to vanadium changes the flow stress behavior during deformation. Fig. 3 shows the flow

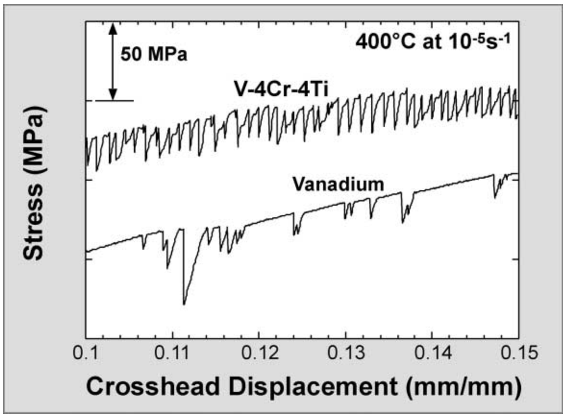

Fig. 3. Tensile curves for vanadium and V–4Cr–4Ti at 400 °C and strain rate of 10^{-5} s^{-1} illustrating serrations and discontinuous yielding in the strain regime from 0.10 to 0.15 strains; curves offset on stress axis for clarity.

stress behavior for vanadium and V–4Cr–4Ti [1] over the strain regime from 0.1 to 0.15 at a strain rate of 10^{-5} s^{-1} at 400 °C. With these conditions, large discontinuous yield drops bounding regions consisting of small serrations (continuous yield drops) are observed for vanadium. The flow curve shows no increase in stress, or yield point, prior to discontinuous yield drops, and occurs at the upper limits of stress for the serrations. There usually is a small decrease in stress for serrated flow regions following a discontinuous yield drop. This deformation behavior changes in V–4Cr–4Ti to regularly spaced serrations, which have a magnitude similar to the discontinuous yield drops observed in vanadium. The flow stress increases non-linearly after each yield drop during deformation, and often saturates at an upper stress level over small increments of strain. However, the data shows the flow behavior of V–4Cr–4Ti at 600 °C looks markedly similar to the flow curve of vanadium at 400 °C. Zaiser and Hahner [4] investigated the spatio-temporal behavior of oscillations in deformed materials and showed there may be three different types of oscillations, which are influenced by temperature and strain rate. From their analysis, the flow stress increases to a yield point where pronounced collective dislocation slip occurs that produces a large yield drop and then propagates as a localized slip band. Differences in the mode of band propagation result in three types of oscillations, which are: (1) smooth band propagation between regularly spaced load drops (type A), (2) minor oscillations in the band propagation superimposed on irregularly spaced load drops (type B), and (3) oscillations with linear load rises and drops, i.e. saw tooth shape, and no band propagation (type C). Although the flow curve observed for vanadium (Fig. 3) shows some similarities with type B oscillations, the load does not rise prior to the large load drop. Type C oscillations may possibly account for the flow behavior observed in V–4C–4Ti, but the non-linear load rise is inconsistent with this classification. Thus, the present data on serration and discontinuous yielding behavior in vanadium and V–4Cr–4Ti do not fit with any of the three types of oscillations. The reason for these differences are not clear but may be related to the tensile test conditions. In past studies the stress–strain curves have been recorded on strip charts, and may not have had sufficient resolution in strain to detect intricate shapes of the oscillations. In the present study, the stress–strain curves were digitally acquired using a normalized counting rate at strain rates of 10^{-3} and 10^{5} s^{-1}, and the data was recorded with a resolution of $\simeq 20\,000$ pts ε^{-1} (where ε is the normalized crosshead displacement) so that fine detail could be seen in the flow curves.

The SRS parameters m were determined from stress–strain curves for vanadium. The lower yield stress, σ_y, and the flow stress measured at 8% strain, σ_f, were determined for each temperature and strain rate. These stress parameters are plotted against $\log \dot{\varepsilon}$, in Figs. 4 and 5. SRS parameters were calculated from a logarithmic fit to the data and are also shown in the figures. Positive m parameters for σ_y occur at 200 and 500 °C, but are insensitive to changes in strain rate at 300 and 400 °C (Fig. 4). However, strongly negative m parameters for σ_f occur at 300 and 400 °C and are relatively insensitive to changes in strain rate at 200 and 500 °C (Fig. 5). These results indicate that DSA effects occur in the unalloyed vanadium in the temperature range from 200 to 500 °C with a maximum between 300 and 400 °C. Early work by Bradford and Carlson [5] and Thompson and Carlson [6] have shown a correlation between DSA effects and the concentration of interstitial O and N solutes in vanadium, respectively. Bradford and Carlson deter-

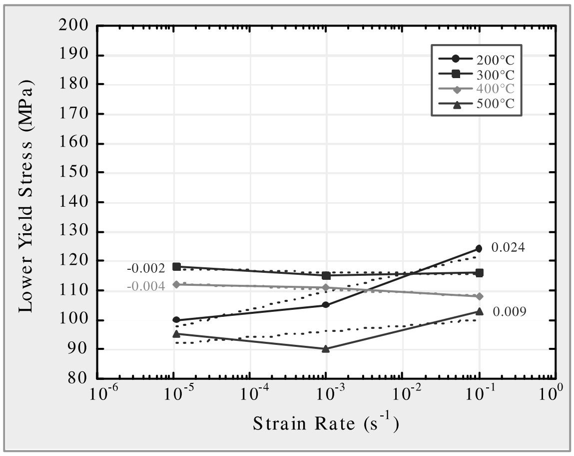

Fig. 4. Lower yield stress, σ_y, as a function of strain rate for various temperatures; SRS is positive at 200 and 500 °C, and is strain rate insensitive at 300 and 400 °C.

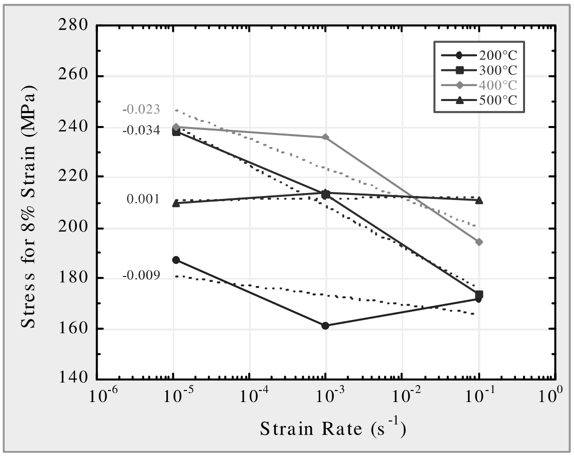

Fig. 5. Flow stress at 8% strain, σ_f, as a function of strain rate for various temperatures; SRS undergoes a transition from slightly negative at 200 °C, negative at 300 and 400 °C, and slightly positive at 500 °C.

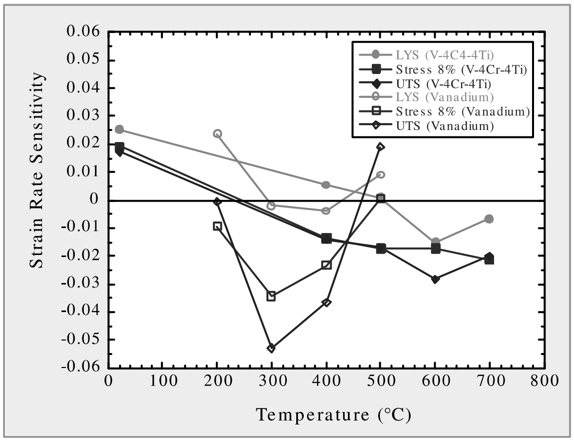

Fig. 6. SRS of lower yield and flow stresses as a function of temperature for vanadium and V–4Cr–4Ti; illustrating that as a result of alloying, the temperature at which SRS achieves a maximum negative value is increased by 300 °C.

mined the temperature range for DSA was from 175 to 425 °C in vanadium containing 265 wppm O and 150 wppm. A temperature range from 250 to 550 °C was determined by Thompson and Carlson in vanadium containing 210 wppm N. In both studies, the maximum negative SRS parameters m occurred between 300 and 400 °C; $m = -0.023$ at 340 °C [5] and $m = -0.011$ at 400 °C [6]. Thus, the present observations of DSA effects appearing in vanadium are consistent with the earlier work on pure vanadium. However, the data from the present study indicates that a maximum negative SRS parameter for σ_u of $m = -0.056$ occurred at 300 °C [2]; this value is more than twice the value measured by Bradford and Carlson [5]. Using the diffusivity data for C, O, and N in vanadium [7], the average diffusion distances ($\sqrt{Dt}$) for each solute at 300 °C for the duration of the tensile test at 10^{-3} s^{-1} ($\simeq$240 s) are $\simeq$73 nm (C), $\simeq$58 nm (O), and $\simeq$6 nm (N). These values indicate that C and O solutes play a dominant role in DSA effects in vanadium below $\simeq$400 °C, which is the temperature that N shows a similar diffusion distance of $\simeq$55 nm. Above 400 °C, all three interstitial C, O, and N solutes contribute to the DSA effect in vanadium.

The SRS parameters *m* determined from the present study on vanadium (Figs. 4 and 5) and the previous study on the V–4Cr–4Ti alloy [1] are shown as a function of temperature in Fig. 6. The DSA effect exists over a temperature range in vanadium and V–4Cr–4Ti, with a maximum negative *m* parameter for the ultimate tensile stress, σ_u, occurring at 300 °C for vanadium and at 600 °C for V–4Cr–4Ti. Thus, the data shows that alloying with Ti shifts the maximum negative SRS by 300 °C. An interaction between Ti and interstitial solutes is the primary reason for the temperature shift in the SRS data. Internal friction measurements provide evidence for a strong positive interaction between interstitial O and C and Ti atoms in V–Ti alloys [8]. This interaction may also account for the lower diffusion rate measured for O in a V–5%Ti alloy [9]. Since Ti is immobile below $\simeq$500 °C [10], the interaction should lower the mobility of interstitial solutes and reduce the local interstitial solute concentration that forms around arrested dislocations. At temperatures >600 °C, Ti possesses long-range diffusion and can form Ti(O,C,N) precipitates [11]. Since precipitation could conceivably occur during testing, especially at low strain rates, this process would remove interstitial solutes from the matrix and signify the upper temperature limit for the DSA effect in the alloy.

5. Conclusion

The data obtained from tensile tests conducted on unalloyed vanadium at temperatures ranging from 200 to 500 °C and strain rates ranging from 10^{-1} to 10^{-5} s^{-1} with those from a previous study on the V–4Cr–4Ti alloy [1] indicate that, (1) the segregation of interstitial solutes to dislocations to form Cottrell atmospheres begins at $\simeq$200 °C in vanadium and $\simeq$300 °C in V–4Cr–4Ti, and (2) the strong interaction between Ti and interstitials solutes lowers the mobility of the interstitial solutes, increases the amplitude of oscillations (barrier strength), and shifts the maximum negative SRS by 300 °C.

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