

# On the self-pinning character of synchro-Shockley dislocations in a Laves phase during strain rate cyclical compressions

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Received 14 January 2008; revised 27 May 2008; accepted 12 June 2008

Available online 25 June 2008

Strain rate cyclical tests in compression, between 1350 and 1500 °C, have been employed to study the self-pinning character of thermally activated synchro-Shockley dislocations in the C15 Cr<sub>2</sub>Nb Laves phase. An average minimum effective (pinning) stress was calculated to be necessary for their propagation. The dislocation velocity cannot respond instantly to the strain rate changes and requires variations in the mobile dislocation density because the synchro-Shockleys can be pinned if the cooperating motion of their two Shockley components is hindered.

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**Keywords:** Laves phases; High-temperature deformation; Compression test; Thermally activated processes; Pinning stress

Tests employing cyclical strain rate changes have been repeatedly used in the past to deduce the parameters of thermally activated deformation in many materials. These experiments, however, are not routine when they are used for complex intermetallics such as the C15 Cr<sub>2</sub>Nb because they require dedicated facilities that are able to reach the high temperatures where such compounds are ductile. They are capable of providing valuable information about variations in the mobile dislocation density,  $\rho_m$  and the average dislocation velocity,  $\bar{v}$ , which have never before been reported for Laves phases. Cr<sub>2</sub>Nb has been considered for high-temperature service applications because it exhibits oxidation resistance superior to that of conventional Ni-based superalloys and its density is relatively low (7.7 g cm<sup>-3</sup>) [1]. It is inherently strong, maintains its strength at high temperatures and exhibits high creep resistance [2]. Cr<sub>2</sub>Nb exhibits the cubic C15 (MgCu<sub>2</sub>-type) structure below ~1600 °C and the hexagonal C14 (MgZn<sub>2</sub>-type) structure between this temperature and its melting point (between 1730 and 1770 °C) [3]. Both crystal structures are topologically close-packed

(tcp), with tetrahedra of the major element (Cr) placed within face-centred cubic (fcc) or hexagonal close-packed arrangements of the minority element (Nb). As a single-phase alloy, however, Cr<sub>2</sub>Nb is brittle below 1250 °C and exhibits poor damage tolerance at room temperature [4]. This, as well as the alloy's high-temperature strength, is associated with the complexity of the crystal structure. The 24 atoms in the highly symmetrical unit cell have an elemental radius ratio well below the ideal ( $R_{\text{Nb}}/R_{\text{Cr}} = 1.15 < 1.225$ ) and form a tightly packed structure where dislocations move only at high temperatures where thermal activation is significant.

Experimental studies, employing Z-contrast imaging in a high-resolution scanning transmission electron microscope with sub-angstrom resolution, have confirmed that slip and twinning in Laves phases occurs by synchro-Shockley dislocations [5]. These consist of two ordinary Shockley partials that are "coupled", i.e. closely spaced with each other, sweep cooperatively adjacent atomic nets and shear them along different orientations. The total Burgers vector of a synchro-dislocation is the vector sum of their two Shockley components, resulting in partial dislocations with 30° or 90° character.

The objective of the present work is to evaluate the consequences of the deformation being accomplished by

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mobile synchro-Shockley dislocations as a function of temperature. Strain rate cyclical tests are employed, under the assumption that the strain rate changes, and consequently the applied stress, affect only the dislocation velocity and not the mobile dislocation density. Consequently, the calculation of the parameters of thermal activation can provide an insight into how effectively these mobile dislocations accomplish the imposed deformation.

It is generally accepted that the plastic deformation of metals and intermetallics at high temperatures is thermally activated and that, experimentally, the strain rate  $\dot{\epsilon}$ , is related to  $\bar{v}$  according to [6,7]:

$$\dot{\epsilon} = \dot{\epsilon}_0 e^{-Q/kT} = \rho_m b \bar{v} = \rho_m b A (\sigma_f^*)^{n^*}, \quad (1)$$

where  $\dot{\epsilon}_0$  is a pre-exponential factor (related to the overall event frequency),  $Q$  is the experimental activation energy,  $\rho_m$  is presumably constant at constant temperature,  $T$ , and independent of the applied stress, and  $b$  is the magnitude of the Burgers vector of the mobile dislocations. The parameter  $\bar{v}$ , in the last part of Eq. (1), is replaced by the expression  $A(\sigma_f^*)^{n^*}$  [8], where  $A$  is a proportionality constant,  $\sigma_f^*$  is the effective flow stress which results from short-range obstacles to dislocation motion which influence strongly the thermally activated dislocation motion [9,10], and  $n^*$  is the effective stress exponent.  $\sigma_f^*$  is equal to the flow stress  $\sigma_f$  minus the athermal stress  $\sigma_a$ , which describes long-range obstacles to plastic deformation, i.e. barriers that are too high to be surmounted by thermal fluctuations and are not affected by  $T$  and  $\dot{\epsilon}$  (e.g. forest dislocations, grain boundary dislocations), except the decrease in the shear modulus, with increasing  $T$ .

Triple strain rate cyclical tests permit the calculation of the  $\sigma_a$ , since for  $n^*$  (and the effective strain rate sensitivity,  $m^*$ ) the following relation holds [8,11]:

$$\begin{aligned} m^* &= \frac{1}{n^*} = \frac{\ln(\sigma_{f2}^*/\sigma_{f1}^*)}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)} = \frac{\ln[(\sigma_{f2} - \sigma_a)/(\sigma_{f1} - \sigma_a)]}{\ln(\dot{\epsilon}_2/\dot{\epsilon}_1)} \\ &= \frac{\ln[(\sigma_{f3} - \sigma_a)/(\sigma_{f2} - \sigma_a)]}{\ln(\dot{\epsilon}_3/\dot{\epsilon}_2)} \end{aligned} \quad (2)$$

and thus

$$\sigma_a = \frac{\sigma_{f1}\sigma_{f3} - \sigma_{f2}^2}{\sigma_{f1} + \sigma_{f3} - 2\sigma_{f2}} \quad (3)$$

where  $\sigma_{f1}$ ,  $\sigma_{f2}$ , and  $\sigma_{f3}$  are the flow stresses at strain rates  $\dot{\epsilon}_1$ ,  $\dot{\epsilon}_2$ , and  $\dot{\epsilon}_3$ , respectively. The latter is valid if the strain rate ratios  $\dot{\epsilon}_2/\dot{\epsilon}_1$  and  $\dot{\epsilon}_3/\dot{\epsilon}_2$  are made equal and  $m^*$  is constant during each test.

Elemental starting materials (Cr and Nb with purity 99.5 and 99.9%, supplied by Johnson Matthey and Aldrich, respectively) were used to prepare a 500 g ingot of stoichiometric  $\text{Cr}_2\text{Nb}$ , by transferred-arc plasma melting under a slight overpressure of high-purity Ar. The ingot was remelted five times to promote homogeneity. It was then sectioned using a SiC cutting wheel into specimens with dimensions  $1 \times 1 \times 4$  cm, which were surface smoothed by conventional grinding, and polishing, and homogenized at 1400 °C for 2 days in an Ar atmosphere. The heating/cooling rate was  $1 \text{ }^\circ\text{C s}^{-1}$ , resulting in material with low residual stresses. Specimens with dimensions  $1.5 \times 1.5 \times 2.5$  mm were prepared from the homogenized material by electrodischarge machining (EDM), surface grinding and polishing in order to ensure the absence of

cracks and/or voids. The samples were mechanically compressed in an Instron 5100 frame equipped with W-mesh heating elements within a water-cooled vacuum chamber. The chamber was evacuated to  $10^{-7}$  mbar and the specimens were uniaxially compressed at temperatures of 1350, 1400, 1450, and 1500 °C. The thermal activation parameters were investigated by imposing instantaneous strain rate changes upwards, as well as downwards, at  $\dot{\epsilon}_1 = 10^{-4}$ ,  $\dot{\epsilon}_2 = 1.5 \times 10^{-4}$  and  $\dot{\epsilon}_3 = 2.25 \times 10^{-4} \text{ s}^{-1}$ . The ratios  $\dot{\epsilon}_2/\dot{\epsilon}_1 = \dot{\epsilon}_3/\dot{\epsilon}_2 = 1.5$  were made especially small, so as to avoid drastic changes in the microstructure. The applied stress was recorded with an accuracy of  $\pm 0.1$  MPa. Despite the small strain rate changes, the applied load was measured with a high sensitivity, so that the resulting calculations yielded reliable results [12]. At each  $T$  at least three specimens were successfully compressed, with the compliance of the compression facility removed from the stress–strain curves.

Detailed descriptions of the mechanical properties, the pre- and post-deformation microstructures, and composition analysis have been reported elsewhere [13]. No Cr loss was detected after deformation and the alloy was single-phase C15  $\text{Cr}_2\text{Nb}$ . Figure 1 shows representative cyclical strain rate change curves for the uniaxial compressions of polycrystalline  $\text{Cr}_2\text{Nb}$  at  $T$  from 1350 to 1500 °C. All specimens were ductile up to nominal strains of 10%. Only those compressed at 1350 °C showed stress peaks at the onset of plastic deformation, followed by softening prior to the establishment of steady-state flow. Each test included three complete cycles except that at 1500 °C which included four cycles and  $\sigma_a$  was calculated using the flow stress plateau values from each individual stage comprising a strain rate cycle. These calculations, however, produced small, alternating, negative and positive values for  $\sigma_a$  (ranging between  $-30$  and  $30$  MPa), a fact that can be interpreted in terms of the athermal stress being very small (i.e. essentially  $\sigma_a \approx 0$ ), confirmed by the absence of strain hardening in the stress–strain curves. Analogous conclusions were reported for similar tests on the C15  $\text{Fe}_2(\text{Dy},\text{Tb})$  single crystals [14].

Since  $\sigma_a \approx 0$ , one would expect that the same calculations, but using the average value of the flow stress from stages compressed with the same  $\dot{\epsilon}$  from all cycles in each test, would result more faithfully in values close to zero. These calculations, however, produced positive values, decreasing with  $T$ , from 100 MPa at 1350 °C to 65 MPa at 1500 °C, within significant error margins

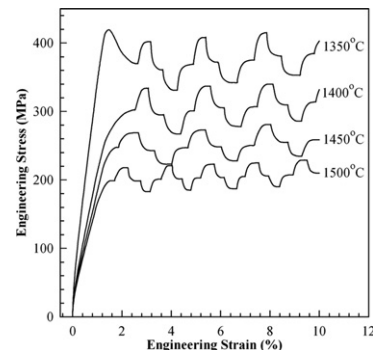


Figure 1. Stress–strain curves for the C15  $\text{Cr}_2\text{Nb}$  during triple strain rate cyclical tests.

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