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

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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 Cr₂Nb 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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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 Cr₂Nb 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, ρ_m and the average dislocation velocity, \bar{v} , which have never before been reported for Laves phases. Cr₂Nb 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⁻³) [1]. It is inherently strong, maintains its strength at high temperatures and exhibits high creep resistance [2]. Cr₂Nb exhibits the cubic C15 (MgCu₂-type) structure below ~1600 °C and the hexagonal C14 (MgZn₂-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, Cr₂Nb 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, ρ_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, σ_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. σ_f^* is equal to the flow stress σ_f minus the athermal stress σ_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 σ_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 σ_{f1} , σ_{f2} , and σ_{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 Cr_2Nb , 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°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 ± 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 Cr_2Nb . Figure 1 shows representative cyclical strain rate change curves for the uniaxial compressions of polycrystalline Cr_2Nb 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 σ_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 σ_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

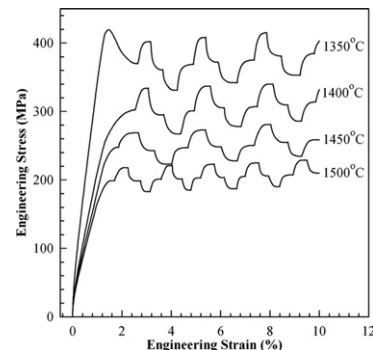


Figure 1. Stress–strain curves for the C15 Cr_2Nb during triple strain rate cyclical tests.

(Table 1). These positive values are more likely related with the average minimum effective stress required for the dislocations to retain their mobility, at each T , i.e. the average pinning stress, σ_p , the synchro-Shockleys have to overcome in order to remain mobile. Table 1 also shows the average σ_f at each T and $\dot{\epsilon}$, and the m^* resulting from the calculations, which was slightly higher than 0.30. Since σ_p is related to the minimum average force required to maintain the coupled configuration of the mobile synchro-dislocations at each constant T , it is not related to thermal activation phenomena and is subtracted from σ_f in the calculations that follow.

The experimental activation volume, V , is given by:

$$V = kT \left(\frac{\partial \ln(\dot{\epsilon}/\dot{\epsilon}_0)}{\partial \tau_f^*} \right) \Big|_T \approx kT \left(\frac{\ln(\dot{\epsilon}_1/\dot{\epsilon}_2)}{\Delta \tau_f^*} \right) \Big|_T = kT \left(\frac{\ln(1.5)}{\Delta \tau_{fT}^*} \right) \Big|_T, \quad (4)$$

where $\Delta \tau_{fT}^*$ is the value of the flow stress increments extrapolated to zero strain, after they have been divided by a Taylor factor of 3 (so as to convert the normal stresses to shear stresses). This approximation is valid if the dislocation velocity changes “instantly” by the ratio of the applied $\dot{\epsilon}$ which means that the pre-exponential term $\dot{\epsilon}_0$, and consequently, ρ_m remains constant. Using Eqs. (1), (4) takes the form:

$$V = kT \left(\frac{\partial \ln(\dot{\epsilon}/\dot{\epsilon}_0)}{\partial \tau_f^*} \right) \Big|_T \approx kT \left(\frac{\partial \ln \dot{\epsilon}}{\partial \tau_f^*} \right) \Big|_T = \frac{kT}{m^* \tau_f^*}. \quad (5)$$

The variation of V with T is presented in Figure 2a, whereas Figure 2b shows the variation of V with τ_f^* , (given by Eqs. (4) and (5), respectively). They are expressed in units of $(b_{\text{perf}})^3$ and $(b_{\text{part}})^3$, i.e. the Burgers vectors of a perfect and a partial dislocation of the C15 Cr₂Nb ($b_{\text{perf}} = 0.4946$ and $b_{\text{part}} = 0.2856$ nm, respectively). Deformation by slip involves relatively small V varying between 6 and 16 $(b_{\text{perf}})^3$, whereas in twinning, V increases to values between 30 and 80 $(b_{\text{part}})^3$. For comparison, the activation volume for Nb ranges from 100 $(b_{\text{perf}})^3$, when σ^* approached zero, to below 5 $(b_{\text{perf}})^3$ as σ^* exceeded 400 MPa [8]. Since $(b_{\text{perf}})^3$ in Cr₂Nb is approximately five times as large as that in Nb, one can easily consider that these values of V in the former are larger than those in the latter.

Using Eq. (1), Q becomes:

$$Q = -kT^2 \frac{\partial \ln(\dot{\epsilon}/\dot{\epsilon}_0)}{\partial \tau_f^*} \Big|_T \frac{\partial \tau_f^*}{\partial T} \approx -\frac{kT^2}{m^* \tau_f^*} \frac{\Delta \tau_f^*}{\Delta T}. \quad (6)$$

The monotonic decrease of Q with increasing σ_f^* , from 600 to 200 kJ mol⁻¹ (Fig. 3) suggests that more than one process controls the deformation rate. Furthermore, the dependency of V on τ_f^* does not permit a unique identification of the deformation-controlling mechanism [11].

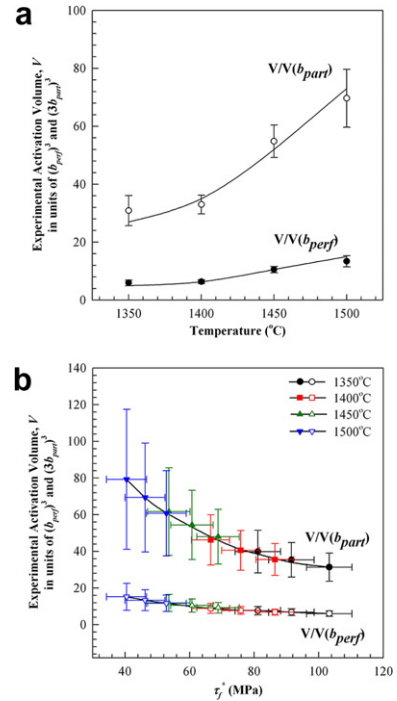


Figure 2. Variation of the experimental activation volume with (a) T and (b) τ_f^* .

Post-mortem TEM observation revealed that at these $\dot{\epsilon}$ twinning by autocatalytic nucleation dominated in grains oriented so as to have two coplanar twinning systems with comparable resolved shear stresses, and consequently, twinning and slip may occur concurrently in different grains [13]. The values of V suggest that slip is controlled by the Peierls–Nabarro mechanism, whereas twinning is controlled by the cross-slip of screw dislocations. These were extended and thus twinning may be accomplished by Hazzledine’s model of Frank partial [15].

Cyclical strain rate experiments are carried out under the assumption that ρ_m remains constant and strain rate changes affect only \bar{v} . Thus, the pre-exponential term $\dot{\epsilon}_0$ in Eq. (2) should be insensitive to strain rate and temperature (i.e. the expression Q/kT should be constant with T) [16]. This plot, however (Fig. 4), shows an increasing trend, indicating that ρ_m increases with $\dot{\epsilon}$ and T . The stages corresponding to a higher $\dot{\epsilon}$ are normally accompanied by high σ_f^* , and consequently, deformation should be accomplished by the dislocations acquiring higher velocity. Analogously, at low $\dot{\epsilon}$, the velocity of the mobile dislocation should decrease. At high T , Q and V increase, i.e. the number of successful events per dislocation increases, and deformation requires low σ_f^* .

Table 1. Average σ_f at each T and $\dot{\epsilon}$, the calculated σ_p and the average m^*

T (°C)	Average σ_f at each strain rate			σ_p (MPa)	m^*
	$1.00 \times 10^{-4} \text{ s}^{-1}$	$1.50 \times 10^{-4} \text{ s}^{-1}$	$2.25 \times 10^{-4} \text{ s}^{-1}$		
1350	342.00 ± 0.12	373.17 ± 0.11	408.33 ± 0.12	98.51 ± 20.99	0.30 ± 0.04
1400	277.00 ± 0.12	304.89 ± 0.11	336.67 ± 0.12	77.37 ± 17.52	0.32 ± 0.04
1450	228.53 ± 0.12	250.00 ± 0.11	274.33 ± 0.12	67.78 ± 19.61	0.31 ± 0.04
1500	186.25 ± 0.12	203.50 ± 0.11	223.20 ± 0.11	64.80 ± 18.45	0.33 ± 0.05

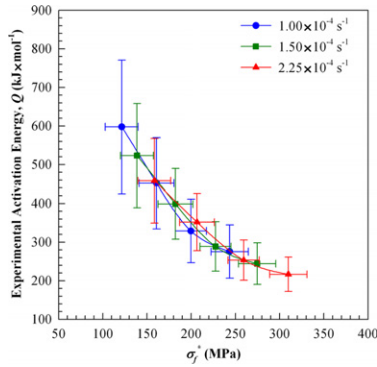


Figure 3. Variation of the experimental Q with σ_y^* .

The increase in ρ_m with $\dot{\epsilon}$ and T indicates that \bar{v} is unable to adapt instantly to changes in $\dot{\epsilon}$, and, consequently, additional mobile dislocations are nucleated to facilitate deformation. Furthermore, upon decreasing $\dot{\epsilon}$, sufficient time has to elapse until the excess mobile dislocations annihilate, or vacate the crystal at the grain boundaries (i.e. the variations of \bar{v} and ρ_m are complementary to each other). V and Q , therefore, are appreciably lower since an increase in ρ_m would result in a decrease in the average activation area and fewer successful activation events per dislocation. Evaluation of the behaviour of this compound using higher strain rate ratios is not possible because at 1350 and 1400 °C, Cr_2Nb is brittle above 5×10^{-4} and 10^{-3} s^{-1} , respectively [13]. Thus the customary strain rate ratios of 10 cannot be employed. In addition, at higher $\dot{\epsilon}$, the variations in ρ_m would almost certainly result in the appearance of stress peaks, which in turn would necessitate longer times within each cycle before steady-state flow is established. These longer stages would in turn require larger total strains, which may be detrimental to the ductility of the Cr_2Nb and/or change considerably the microstructure.

Our experimental results of the response of this Laves phase can be understood if one considers the synchro-Shockley propagation inside the individual atomic nets of the C15 structure. These are stacked as $A\alpha\zeta\beta B-\beta\gamma C\gamma b\alpha$, where uppercase letters correspond to “3636” kagomé Cr nets, Greek characters to “3⁶” Nb nets fitted in the holes of the underlying/overlying kagomé Cr nets, and lowercase letters represent “3⁶” Cr nets occupying the only available sites between the Nb atoms. The cooperative propagation of a synchro-

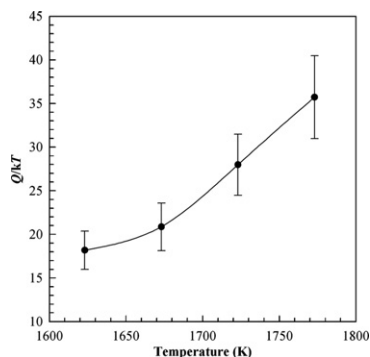


Figure 4. Variation of Q/kT with T .

Shockley within the layers between the A and B nets occurs when the advancing Shockley forces the c atoms over the saddle points of the α net to the b position while its trailing component forces the β atoms (and the crystal above) over different saddle points of the α net, along the trailing Shockley's orientation to the γ sites [5]. This changes the stacking into $A\alpha\uparrow b\uparrow\gamma C\gamma b\alpha A\alpha\zeta\beta$ and requires two consecutive successful thermal fluctuations per synchro-Shockley. If either fluctuation is unsuccessful, then the total dislocation becomes temporarily immobile. The probability of this occurring is twice that of an ordinary dislocation in ordinary fcc crystals and results in its self-pinning nature [17]. This paper shows that the response of the material to strain rate changes requires variations in the mobile dislocation density because the synchro-Shockleys are pinned if the cooperating motion of their two Shockley components is hindered. The decrease of σ_y with T is most likely related to the degree of softening of the crystal structure (i.e. the decrease of the elastic constants with T). Thus deformation occurs only when the interatomic distances have increased, thus enabling the synchro-Shockleys to overcome easily their self-pinning resistance and respond, alleviating the imposed stresses.

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