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Microyielding in alpha titanium

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The microflow parameters of two grades of polycrystalline alpha titanium have been determined from compression tests at temperatures in the range 77 to 335 K. Specimens were tested in either annealed or prestrained conditions, and the elastic limit, microyield stress, activation volume and activation energy were determined. The data indicate that dislocations can move at the elastic limit without strong influence from interstitial solutes, except at 77 K where some solute atoms provide a barrier to motion. As the plastic strain is increased, solute obstacles play an increasing part in determining the flow stress, and the microyield stress shows a strong dependence on solute concentration. There is conflicting evidence as to the role screw dislocations have in the microflow region.

1. Introduction

The characteristics of plastic flow in alpha titanium have been studied extensively in recent years, so that, as reported in the reviews of Reed-Hill *et al.* [1, 2] and Conrad *et al.* [3], the dependence of phenomena such as yielding and work hardening on parameters such as impurity content and temperature are reasonably well understood. Like the other close-packed-hexagonal metals which slip predominantly on the prism system, the yield stress of titanium shows a strong dependence on temperature and interstitial-solute concentration, and this is generally interpreted in terms of a model in which yielding is controlled by dislocation–solute interactions.

In some cases, the experimental data on yielding have not been in agreement with a simple solute-interaction mechanism. Levine [4], for example, interpreted his yield-stress and activation-parameter data obtained from high-purity titanium in terms of several mechanisms, each one being of importance in different temperature ranges. He concluded that below 220 K, double-kink nucleation over the Peierls–Nabarro barrier controls prismatic slip, whereas a different, unidentified mechanism is controlling between 220 and 300 K. Reed-Hill [5], however, pointed out the danger of placing great reliance on the activation parameters under the conditions employed. Evans [6] also deduced that two controlling mechanisms occur below 500 K, the temperature of transition varying with impurity content from 200 to 450 K, with the low-temperature mechanism being a dislocation–solute interaction. Tung and Sommer [7] undertook stress relaxation tests on commercial purity titanium, and demonstrated that a process with activation energy 0.3 eV controls dislocation motion between 300 and 380 K, and another with energy 1.1 eV operates between 380 and 500 K. The actual mechanisms involved could not be identified. More recently, Sastry and Vasu [8] measured the activation parameters in three grades of titanium, and concluded that double-kink nucleation is the sole mechanism controlling the motion of dislocations at the yield stress at temperatures below 700 K, whereas Akhtar and Teghtsoonian [9] found prismatic slip in single crystals to be controlled by dislocation–solute interactions below 250 K and a different mechanism, which could not be identified, above 250 K. Thus, although the mass of evidence favours a model in which interstitial solutes control dislocation motion, this view

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is not held unanimously and there are strong indications that several mechanisms may be involved.

In many respects, the temperature- and impurity-dependence of yielding in titanium resembles that of the b c c transition metals. There has also been debate as to the relative roles of the intrinsic-lattice and impurity-associated mechanisms (see, for example, the review by Christian [10]), although these mechanisms are complicated in the b c c lattice by the distinct differences in the core structures of the screw and non-screw dislocations. Considerable assistance in understanding the mechanisms which can occur in a metal of given purity under the various conditions of strain and temperature has been gained in the b c c case by carrying out measurements in the microflow region. (This subject has been reviewed by Seeger and Wüthrich [11]). Microflow here refers to plastic strain levels below about 10^{-3} and down to $\sim 10^{-6}$ in the case of uniaxial, microstrain tests and possibly two orders of magnitude lower in the case of conventional, internal-friction tests. The former are perhaps easier to relate to macroyield experiments, and have been employed in the present investigation to study the microyielding behaviour of two grades of alpha titanium. The aim was to shed light on the mechanisms which control the earliest stages of dislocation motion.

2. Experimental procedure

The less-pure titanium employed was a commercial-purity grade supplied by Metals Research Ltd. The material was supplied in the form of 4 mm diameter rod, and a 1 h anneal at 700° C at a pressure $< 10^{-5}$ Torr produced equiaxed grains of mean diameter $25 \pm 5\,\mu$m. Subsequent analysis for interstitial-gas content by I.M.I. (Kynoch) Ltd yielded O:1850, N:75, C:200 and H:25, all in wt ppm. The other material used was based on an ex-iodide purity bar of 10 mm diameter supplied by Alpha Metals Inc. After a single pass of a molten zone in an electron-beam zone refiner, the rod was swaged to 4 mm diameter in 1 mm steps without intermediate anneal, and then annealed for 9 h at 570° C. This treatment produced equiaxed grains having a mean diameter of $23 \pm 5\,\mu$m. The interstitial solute content of material prepared in this way was O:400, N:820, C:150, H:25. In order to compare the results from different materials, Conrad [12] has suggested, and extensively used, a total interstitial content given in terms of an oxygen equivalent thus: $[O]_{eq} = [O] + 0.75\,[C] + 2.0\,[N]$. For the specimens based on the commercial-purity (CP) and iodide-purity (IP) metals used here, this yields $[O]_{eq}$ values of 6654 and 3714 at. ppm, respectively.

The specimens were tested in compression using the jig described by Cowling and Bacon [13] and used by those workers in a study of microyielding in tantalum [14, 15]. The rods were lightly polished for a few seconds in a solution of 25 ml HF and 75 ml HNO_3, and 10 mm long compression specimens were prepared by grinding and polishing the end faces in the manner described elsewhere [13, 16]. The strain within a specimen was measured by two Micro-Measurements type SK resistance gauges of the temperature-compensating type bonded diametrically opposite each other. The M-Bond 610 adhesive used was cured by annealing in an argon atmosphere at 150° C for 1 h. Specimens prepared in this way are referred to here as "annealed". Some specimens were compressed by 0.5% before microflow testing, and these will be referred to as "prestrained".

The compression jig used avoids the difficulties often encountered in microyield testing in compression [13], and the system was capable of a resolution of 1 kgf load and 2×10^{-6} strain per 10 mm of chart on an x–y recorder. A second, time-based pen on the recorder measured total strain rate, which was constant within one test and fell in the range 10^{-5} to $8 \times 10^{-5}\,\text{sec}^{-1}$. Compression tests were carried out at four fixed temperatures, namely 77, 203, 295 and 335 K, by placing the jig in a simple cryostat [16].

The parameters determined in the course of the testing were the elastic limit σ_e, the microyield stress σ_{my}, the activation volume v^*, the activation energy ΔH and the damping $\Delta W/W$. The stress values quoted here are for normal stress and can, of course, be compared with resolved shear stress values by using the appropriate Taylor factor of 5 [3]. The usual testing procedure was to take the specimen through a series of load–unload cycles at successively higher stress amplitudes. The stress amplitude at which the loading and unloading curves on the recorder form a lenticular loop with an associated plastic strain ϵ_p (measured as loop width) of 1×10^{-6} was defined to be σ_e. An alternative procedure in which σ_e was taken to be the slope of a plot of energy dissipated per cycle ΔW (= loop area) versus ϵ_p at an ϵ_p value of 1×10^{-6} gave similar values, but a sufficient num-

ber of load–unload loops for this method to be used could not be obtained for all specimens. The stress required to produce a permanent strain (equivalent to a closure failure of a loop at zero load) of 5×10^{-6} was defined to be σ_{my}. Once this stress had been determined, the testing procedure was repeated at two other strain rates, and then one of the 0.5% prestrains was carried out.

The activation parameters were determined from the following equations:

$$v^* = kT(\Delta \ln \dot{\epsilon}_p / \Delta\sigma)_T, \quad (1)$$

$$\Delta H = -v^* T(\Delta\sigma/\Delta T)_{\dot{\epsilon}_p}, \quad (2)$$

where k is Boltzmann's constant and T the absolute temperature. It is almost impossible to make direct, strain-rate-change tests in the stress range $\sigma_e \leqslant \sigma \leqslant \sigma_{my}$ used here, and v^* was therefore determined using the indirect method first used by Kossowsky [17]. It involves plotting ln σ versus ln ϵ_p for the series of tests carried out at each strain rate on a specimen at one temperature. (Each plot was found to be linear in the present investigation.) A plot of σ versus ln $\dot{\epsilon}_p$ can then be constructed for a given ϵ_p level, and the gradient of this graph is that required to calculate v^* and hence ΔH. The errors inherent in this method are bound to be large – possibly as high as ± 30% – but it seems to be the only one available for estimating v^* and ΔH for deformation below the microyield point. These parameters could not be determined for the annealed specimens or the prestrained CP specimens tested at 77 K as they did not produce load–unload loops of sufficient width for stresses less than σ_{my}; i.e. the loop width did not exceed approximately 5×10^{-6} for these conditions, whereas it could reach 5×10^{-5} or more for the others.

3. Results

The variation of the elastic limit σ_e with temperature T for annealed specimens and those prestrained 0.5% at either 77 or 295 K is shown in Fig. 1. The total strain rate used was $4 \times 10^{-5}\,\text{sec}^{-1}$ and it is estimated that the possible error in the data points is ± 15 MPa; changing the strain rate by a factor of 8 produced changes in σ_e which were within the experimental error. The σ_e values for the CP material at 77 K are some 30 to 50% higher than those of the IP titanium, but the values at 203 and 295 K are very similar. The temperature dependence of σ_e is clearly largest in the annealed state for both materials.

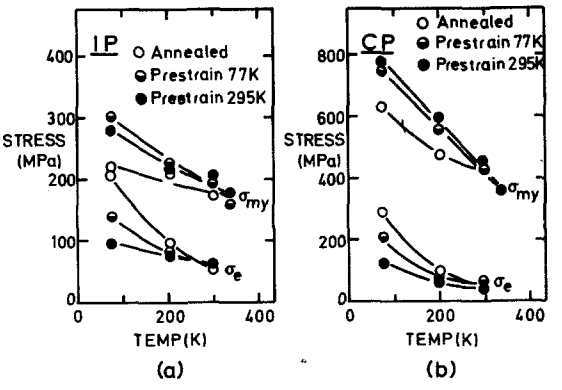

Figure 1 Elastic limit (σ_e) and microyield stress (σ_{my}) versus temperature for (a) iodide purity (IP) and (b) commercial purity (CP) titanium tested in the conditions indicated.

Results for the microyield stress, with possible error ± 30 MPa, are also plotted in Fig. 1, from which it can be seen that σ_{my} is consistently between two and three times higher in the less-pure, CP material. The temperature dependence is also more marked for this alloy, and, unlike σ_e, the annealed state for both materials has the lowest microyield stress and this is raised by equal amounts by the 77 and 295 K prestrains. The (micro)flow stress versus plastic strain curves obtained during the 0.5% prestrains at 77 and 295 K are shown in Fig. 2. The plastic strain for values $> 10^{-4}$ was measured from the elastic line obtained for cycles with $\sigma \leqslant \sigma_e$. From a comparison of these and the σ_e and σ_{my} data of Fig. 1, it is clear that only a small amount of strain hardening occurs over the range between σ_{my} and the (macro)yield stress measured in conventional tests. Rapid hardening occurs between σ_e and σ_{my} in the less-pure material, however, and this is even more marked after the prestrains, which lower σ_e and raise σ_{my}. The hardening within this range in

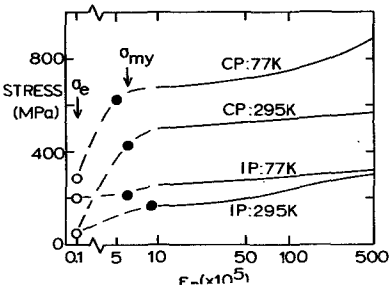

Figure 2 Flow stress versus plastic strain ϵ_p for the iodide (IP) and commercial (CP) grades at 77 and 295 K. The elastic limit and microyield points are indicated.

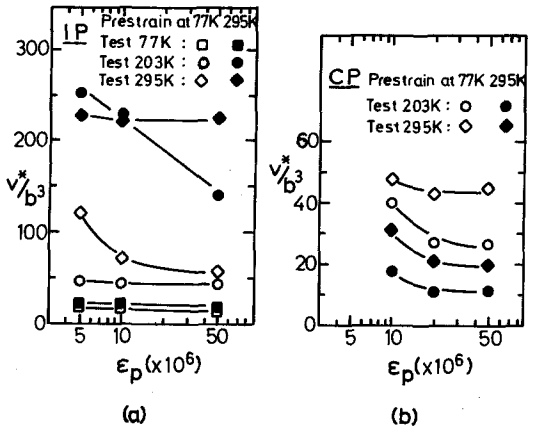

Figure 3 Activation volume, in units of (Burgers vector)^3, at different temperatures versus plastic strain for (a) IP and (b) CP specimens prestrained at either 77 or 295 K as indicated.

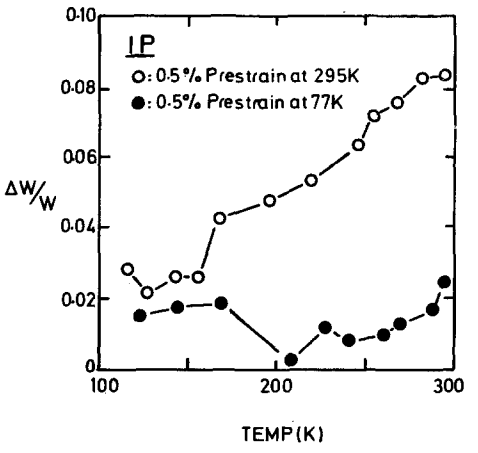

Figure 5 Damping capacity versus temperature for the iodide-purity specimens prestrained at either 77 or 295 K. The maximum stress for each cycle was 144 MPa.

the IP material is much smaller, particularly at 77 K, but is again increased by the prestrains.

The activation volumes, in units of Burgers vector cubed, are presented in Fig. 3. The plastic strain ϵ_p was determined from loop width, and, as indicated above, analysis was not possible for the annealed specimens and the 77 K tests on the CP specimens. Bearing in mind the possible errors referred to above, v^* shows only a small dependence on ϵ_p for $\epsilon_p \gtrsim 10^{-5}$ and tends to increase with increasing temperature. The values are larger for the IP material, particularly after the 295 K prestrain. The activation energies calculated for the same specimens and conditions are shown in Fig. 4. ΔH increases with increasing strain in the microyield region, and the dependence on T may be taken to be linear to within the errors discussed above. The differences between the two materials also fall within the possible error.

The damping capacity $\Delta W/W$ for stresses below σ_{my} was calculated from the stress–strain loops which were of sufficient size for meaningful measurements to be made. This precluded analysis for the CP specimens and the annealed IP material. For the IP specimen prestrained at 77 K, $\Delta W/W$ was small ($<4\%$) and amplitude independent. Following the 295 K prestrain, however, it showed a strong amplitude dependence at 203 and 295 K increasing to approximately 7% at σ_{my} [16]. In a further experiment, an IP specimen of each prestrain was allowed to warm up slowly from 100 to 295 K, and the damping was repeatedly measured at a constant maximum stress of 144 MPa, which lies between σ_e and σ_{mys}. The variation of $\Delta W/W$ with T obtained in this way is shown in Fig. 5. The difference between the two specimen conditions is marked in this kind of test, although their σ_e values are the same over most of the temperature range.

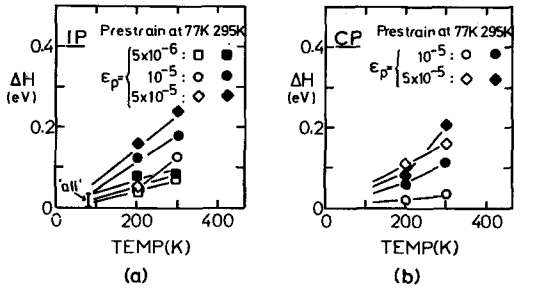

Figure 4 Activation energy (eV) at different strain levels versus temperature for (a) IP and (b) CP specimens prestrained at either 77 or 295 K as indicated.

4. Discussion

Consider first the effects of the prestrains on the materials used. Although transmission electron microscopy was not undertaken, a good indication of the dislocation structures produced can be gained from the observations of Williams *et al.* [18]. They noted a transition from a planar structure consisting of dense bands of near-screw dislocations on the prism planes to one of a more random, wavy nature as the temperature of deformation rises. The transition temperature increases with increasing interstitial-solute content. From these observations, the dislocation structure in the IP specimens prestrained at 295 K is expected to be the random configuration, whereas that produced

by the 77 K prestrain should be mainly random with some planar bands. In the CP specimens, the 77 K deformation should produce the planar structure and that at 295 K should form a mainly planar configuration with some random structure.

The prestrains reduce σ_e, particularly at 77 K, with the 295 K prestrain having the stronger effect. The greater dislocation mobility which this reduction reflects must arise from dislocations moving over large distances (on the atomic scale) and multiplying during the prestrain. The resulting mobility is less in the planar, near-screw structure produced at 77 K, presumably because of a greater resistance to movement of screw dislocations. The dislocation density in the specimens is not known, but if it is assumed to lie between 10^5 and 10^7 mm^{-2}, then the average distance moved by a dislocation at σ_e would be between $100\,b$ and $1\,b$, where b is the magnitude of the Burgers vector. This may be compared with the average distance between the interstitial solutes ($\simeq b[O]_{eq}^{-1/2}$) of $16\,b$ and $12\,b$ for the IP and CP materials, respectively. Thus, it may be possible for the plastic strain at σ_e to be achieved without dislocations passing the solute atoms, but the fact that σ_e is significantly higher (by 30 to 50%) at 77 K in the CP specimens than in the IP ones suggests that σ_e at that temperature is strongly affected by the presence of impurities. That does not appear to be the case at 203 or 295 K.

The plastic strain during a cycle to σ_{my} can be as high as 5×10^{-5}, even though the permanent set produced is only 5×10^{-6}. At the former value, the average distance moved by a dislocation for the range of density considered above would fall between $60\,b$ and $600\,b$. It is inconceivable, therefore, that dislocations can move without overcoming solute atoms at σ_{my}, and the experimental data show σ_{my} in the CP specimens to be much higher (by factors of 2 to 3) at all temperatures than that of the IP counterparts. From the microflowstress curves of Fig. 2, it can be anticipated that σ_{my} for the annealed specimens should be close to the yield stress measured in conventional uniaxial testing, and, using a Taylor factor of 5, the σ_{my} values are indeed similar to the critical-resolved-shear-stress values measured by Akhtar and Teghtsoonian [9] in alloys of similar impurity content. Furthermore, they fall close to the straight-line plot of critical resolved shear stress versus $[O_{eq}]^{1/2}$ prepared by Tanaka and Conrad [19] and Conrad *et al.* [3] from the results of several investigations; this square-root dependence is a strong indication that plastic flow is controlled by dispersed, localized barriers to dislocation motion of concentration $[O]_{eq}$. When the σ_e data are fitted to a relation of the form $\sigma_e \propto [O_{eq}]^n$, the best fit is obtained with n approximately equal to 0.5 at 77 K and zero at 203 and 295 K, reflecting the difference in mechanisms discussed above.

Most of the values of the activation volume obtained here are consistent with those measured at higher strain levels by previous workers and with the values of 40 to $60\,b^3$ calculated from stress relaxation tests [7]. The anomalously high values found in the microflow region of the b c c metals are absent here, although insertion of a Taylor factor in Equation 1 would increase v^* somewhat. The value of v^* increases with increasing temperature and purity, as expected but the values in the IP specimens prestrained at 295 K and tested at either 203 or 295 K are noticeably higher than the others. It is not known whether changes in mobile dislocation density during the tests at 203 and 295 K lead to erroneous v^* values, or if dislocation motion is controlled by a different mechanism at these temperatures in the structure produced by the prestrain. The latter interpretation is supported by the damping data, however, and even the former possibility could be consistent with a change in mechanism between 77 and 295 K in the prestrained condition. The activation energies are smaller than those measured in earlier, macrostrain tests. Conrad *et al.* [3] found that data from several sources, which included the low-temperature data of Levine [4] and Evans [6], could be fitted by $\Delta H = 28\,kT$, where k is Boltzmann's constant, and the data of Sastry and Vasu [8] give $\Delta H = 16\,kT$. The values measured here fall within the ranges 2 to $8\,kT$ at $\epsilon_p = 10^{-5}$ and 6 to $10kT$ at $\epsilon_p = 5 \times 10^{-5}$. Similarly, small values ($\sim 12\,kT$) where found by Tung and Sommer [7] from stress-relaxation tests. It is possible, therefore, that the barriers to dislocation motion for stresses below σ_{my} are not the same as those studied in macroflow tests; dislocations of predominantly edge character could be involved in the microyield region, for example, whereas macroyielding could require the large-scale motion of screw dislocations, which, as in other structures, are probably less mobile in titanium. Certainly the dislocation-damping peaks observed below room temperature in conventional internal friction experiments [20] have activation energies which

are much smaller than the absolute barrier heights (~ 1.2 eV[3]) associated with macroflow.

From the evidence at hand, it may be concluded that at σ_e dislocations move over distances comparable with the intersolute spacing, and, on the grounds of both elastic, line-tension calculations for titanium [16] and computer simulation of stressed model h c p crystals [21] which show edge dislocations to be more flexible and mobile than screws, these are predominantly edge in character. The effect on σ_e of change in $[O_{eq}]$ indicates that these dislocations can produce the observed strain by bowing mainly between the solute atoms and possibly only overcoming the weakest barriers in the obstacle spectrum. The origin of the concentration dependence observed at 77 K is unexplained. The prestrains produce mobile dislocations, and that at 295 K, which results in a more random structure, has the more pronounced effect on σ_e, as would be expected. On increasing the stress from σ_e to σ_{my}, long-range dislocation motion occurs by dislocations breaking away from the stronger pinning points provided by the solute elements. The strain-dependence of v^* and ΔH indicates a gradual change in mechanism with increasing strain, but whether or not it involves a change from edge to screw motion as well as unpinning from solute atoms is uncertain. Whilst the notion that screw dislocations are mobile at σ_{my} is in accord with the lack of strain hardening between σ_{my} and the macroyield stress, it leaves unexplained the small ΔH values measured in the microflow region. Although the data obtained point to a gradual change in mechanism with increasing temperature at σ_e and with increasing strain at all temperatures, there is no evidence of a change with increasing temperature at stress levels $\gtrsim \sigma_{my}$, as could be expected from some of the earlier studies referred to in Section 1. Finally, it has been observed from tests on the IP material prestrained at 295 K that changes in substructure can affect some parameters, namely v^* and the damping, and this indicates a change in mechanism which is not reflected in other parameters, e.g. σ_e and σ_{my}. This points to one of the problems inherent in experiments in the microflow region.

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