

An empirical correlation between temperature and activation energy for brittle-to-ductile transitions in single-phase materials

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Abstract

The strain rate dependence of the brittle-to-ductile transition (BDT) temperature gives the activation energy controlling the BDT. Until recently, data were only available for a limited number of materials. Experimental data on the BDT of tungsten and other bcc metals have recently become available. We have compared all the data from different materials and sources, finding a distinctive relationship between the BDT temperature (T_{BDT}) and the activation energy for BDT (E_{BDT}) which holds over a wide range of materials, temperatures and activation energy values. The ratio $E_{\text{BDT}}/kT_{\text{BDT}}$ gives approximately the value 25 for all the materials considered.

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1. Introduction

At high temperatures, a solid material is likely to deform plastically. The generation and subsequent motion of dislocations, which control the bulk plasticity of crystals, are thermally activated processes and for this reason, plastic deformation of crystals is a sensitive function of temperature. Thus, some characteristic parameters such as the activation energy of a given deformation process may be deduced experimentally by varying the temperature of mechanical tests. For example, the plastic strain rate in a metal, $\dot{\epsilon}$, follows the relationship [1]

$$\dot{\epsilon} = \dot{\epsilon}_0 \exp\left(-\frac{\Delta H_s(\tau^*, T)}{kT}\right), \quad (1)$$

where $\dot{\epsilon}_0$ is a pre-exponential factor (sometimes called frequency factor), k is Boltzmann's constant, T the absolute temperature, τ^* is the thermal component of the resolved shear stress, τ , and $\Delta H_s(\tau^*, T)$ is the activation enthalpy for slip. If the dependence of $\dot{\epsilon}_0$ on T and on τ is weak, then the value of ΔH_s can be estimated from the relationships between stress, temperature and strain rate obtained experimentally by conventional mechanical tests. Interestingly, the

ratio $\Delta H_s/kT$ was found to be approximately 25 in several body-centred-cubic (bcc) metals [1, 2]. In some cases, such as for example in silicon and germanium, an activation enthalpy can also be deduced directly from dislocation velocity measurements [3–5]. The relationship between strain rate and dislocation velocity $V(\tau, T)$ can be written as [1, 3, 6]

$$\dot{\epsilon} = \rho b V(\tau, T), \quad \text{with } V(\tau, T) = V_0 \left(\frac{\tau}{\tau_0}\right)^{m(T)} \exp\left(-\frac{\Delta H_d}{kT}\right), \quad (2)$$

where ρ is the density of mobile dislocations with Burgers vector b , V_0 and τ_0 ($=1$ MPa) are constants, $m(T)$ is a monotonic function of temperature and ΔH_d is the activation enthalpy for dislocation mobility. If $m(T)$ does not vary significantly with temperature, such as for example in silicon [3], then a value for ΔH_d can be experimentally deduced. In various semiconductors (for example in Si, Ge, GaAs, InP) the correlation between the activation energy deduced from stress–strain curves, ΔH_s , and that obtained from dislocation velocity measurements, ΔH_d , is well-established [6]. For metals, this relationship is unfortunately not as clear as in semiconductors due to the lack of experimental data on dislocation velocity.

Another, indirect, approach to investigate dislocation activity in some classes of materials (bcc metals, ceramics, semiconductors, ordered intermetallic compounds) is the study of their brittle-to-ductile transition (BDT) [7–11]. At low temperatures, such materials generally fail by cleavage and exhibit completely brittle behaviour. At high temperatures, as dislocation activity becomes important, these materials show ductile behaviour, characterized by a marked plastic deformation. A BDT temperature (T_{BDT}) can be measured for each specific material and test condition. In previous studies of BDT behaviour in pre-cracked single crystal materials, such as silicon [7], germanium [8] and alumina [9], it has been established that the process controlling the BDT is dislocation glide in the region near the crack tip, rather than dislocation nucleation at or near the crack tip. Experimental studies on these materials have shown that the relationship between the applied strain rate $\dot{\epsilon}$ and T_{BDT} is given by [7]

$$\dot{\epsilon} = A \exp\left(-\frac{\Delta H_{\text{BDT}}}{kT_{\text{BDT}}}\right), \quad \text{with } A = \text{constant}, \quad (3)$$

where ΔH_{BDT} was found to be equal to the activation energy for dislocation glide, ΔH_{d} . Moreover, in Si, Ge and Al_2O_3 it has been found that models of the elastic ‘shielding’ of the crack tip by mobile dislocations fit the experimental results to high accuracy [11]. In other materials such as TiAl [12], NiAl [13], zirconia [14] and SiC [15], a well-defined ΔH_{BDT} was found, but no data exist for comparison on the thermal activation parameters of dislocation glide. In the case of molybdenum [16], dislocation-dynamics modelling was able to reproduce the variation of fracture stress intensity with strain rate and temperature below the BDT, but failed in predicting the BDT temperature itself. Although T_{BDT} depends on strain rate and structure, it is found for the above materials that the ratio between the activation energy for BDT and kT_{BDT} gives on a relatively coarse scale $\Delta H_{\text{BDT}}/kT \sim 25$ [17], which is the same ratio as that found for $\Delta H_{\text{s}}/kT$ in yield stress experiments with bcc metals [1, 2]. This is a further strong indication that the BDT process is controlled by dislocation activity at the crack tip and that the mobility of dislocations plays a major role in shielding and/or blunting the crack. In this paper, we present new data on tungsten, vanadium and iron which are here compared with data from other previously investigated materials. An unambiguous trend in the relationship between temperature and activation energy for BDT is found.

2. Experimental

The BDT of tungsten has been investigated using four-point bend testing of pre-cracked single-crystal rectangular bars (1 mm × 1 mm × 11 mm) with sides cut along the [100] directions from an as-grown pure tungsten rod. Similar tests were performed on polycrystalline tungsten samples containing grains elongated along the rod axis with size of approximately 3 μm (cross-section). Pre-cracks were introduced on the tensile faces of the bars by using a sharp edge mounted on a spark erosion machine. In the single-crystals, the observed pre-crack planes were (100) and (010), whereas in the polycrystalline specimens a mixture of

inter- and intra-granular pre-cracks with different orientations was observed. The temperature range investigated was 77–550 K and the strain rate ranged from 4×10^{-5} to $5 \times 10^{-2} \text{ s}^{-1}$. The fracture tests were performed in an argon atmosphere for temperatures above 300 K and in a (cooling) nitrogen atmosphere below room temperature. Fracture surfaces of all specimens were examined by optical and scanning electron microscopy. Specimens which fractured with no plastic strain were considered to be fully brittle; those which did not fracture after >5% of plastic strain were considered to be fully ductile; those which fractured in an apparently brittle manner after a limited amount of plastic strain were designated as ‘semi-brittle’. The technique described here has also been used to test other bcc metals, particularly vanadium and iron (the full experimental details for these materials are described elsewhere [18]).

3. Results

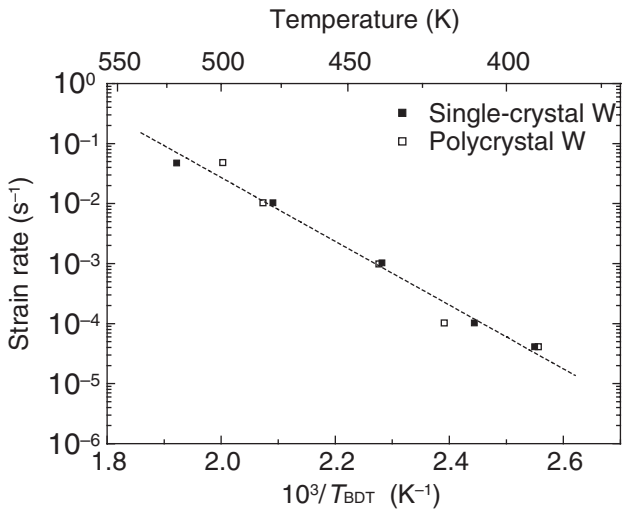
The experimentally deduced fracture toughness of tungsten and its variation with temperature and strain rate are described in detail elsewhere [19]. Figure 1 shows an Arrhenius-type plot of five different strain rates and the respective BDT temperatures for single-crystal and polycrystalline tungsten. Data for single-crystal vanadium and poly- and single-crystal iron are shown in figure 2. In the case of tungsten, all the data points in figure 1 are reasonably aligned along a straight line, giving an activation energy $\Delta H_{\text{BDT}} = 1.05 \pm 0.05 \text{ eV}$. Unfortunately there are no dislocation velocity data available for comparison at the BDT temperatures of interest. The only study on dislocation velocity in tungsten was carried out at room and liquid nitrogen temperatures [20] and gave data for edge dislocations; it is to be expected that the BDT in bcc metals is controlled by the motion of the slower screw dislocations. This is verified in tungsten by comparing ΔH_{BDT} with the kink-pair formation energy, $2H_{\text{k}}$, which is known to control the mobility of screw dislocations in bcc metals. From the analysis of flow-stress measurements in tungsten [2], it was deduced that at temperatures between 220 and 600 K, the kink-pair formation process (at zero stress) requires an energy $2H_{\text{k}} \cong 1.75 \text{ eV}$, which is much larger than the activation energy for edge dislocation mobility ($\sim 0.3 \text{ eV}$ [20]) and perhaps more comparable with the activation energy for BDT reported here for tungsten.

In single-crystal vanadium, the best fit to the data shown in figure 2 gives $\Delta H_{\text{BDT}} = 0.27 \text{ eV}$, whereas for single and polycrystal iron the deduced activation energy is respectively $\Delta H_{\text{BDT}} = 0.33 \text{ eV}$ and $\Delta H_{\text{BDT}} = 0.21 \text{ eV}$. Table 1 shows ΔH_{BDT} values found in Si [7], GaAs [21], Ge [8], Al_2O_3 [9], Mo [16], SiC [15], TiAl [22], diamond [23] and Fe-3%Si [24]. The four different values reported in table 1 for GaAs are related to four crack systems in which 60° dislocations with different character (i.e. with either Ga or As atoms at the dislocation core) are emitted from the crack tip [21].

In tungsten, averaging over the experimental temperature range investigated, we have calculated $\Delta H_{\text{BDT}}/kT_{\text{BDT}} \approx 26.8$, which is in good agreement with other materials where the BDT is known to be controlled by dislocation mobility [7]. The $\Delta H_{\text{BDT}}/kT_{\text{BDT}}$ ratio calculated for vanadium and

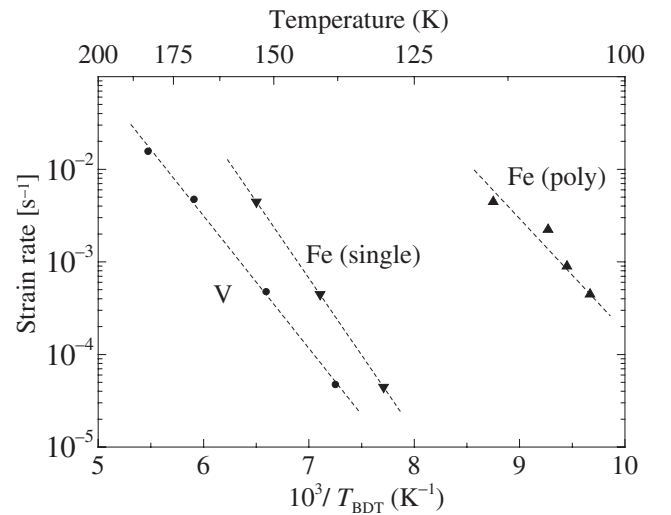
Table 1. ΔH_{BDT} values for various materials.

Material	ΔH_{BDT} (eV)	$\langle \Delta H_{\text{BDT}}/kT_{\text{BDT}} \rangle$	ΔH_{d} (eV)
Intrinsic Si [7]	2.1	23.71	2.2
n-type Si [7]	1.6	22.02	1.7
Sapphire [9]	3.2	27.36	3.2
Intrinsic Ge [8]	1.54	25.65	1.58
GaAs (Ga-1) [21]	1.3 (+0.8/−0.4)	37.05	1.1 ± 0.1
GaAs (Ga-2) [21]	1.09 (+0.04/−0.15)	22.59	1.1 ± 0.1
GaAs (As-1) [21]	1.7 (+0.5/−0.1)	35.24	1.6 ± 0.1
GaAs (As-2) [21]	1.7(+0.5/−0.1)	35.24	1.6 ± 0.1
Diamond [23]	3.06 ± 0.25	23.03	2.6 ± 0.5 (ΔH_{s})
SiC [15]	2.47 ± 0.2	20.65	4.5 ± 0.6 (ΔH_{s})
TiAl [22]	1.4	17.64	–
Mo [16]	0.49	24.77	0.49
W	1.05	26.83	–
V	0.27	19.09	–
Fe (single)	0.33	27.08	–
Fe (poly)	0.21	21.80	–
Fe-3%Si [24]	0.5	31.17	–

**Figure 1.** Arrhenius plot of the strain rate versus $1/T_{\text{BDT}}$ for single and polycrystal tungsten.

iron (investigated using the same experimental method as tungsten) is also shown in table 1.

Figure 3 shows a plot of ΔH_{BDT} as a function of T_{BDT} for different materials; the solid line is a fit to the data giving the ratio $\Delta H_{\text{BDT}}/kT_{\text{BDT}} = 25$. The large error bars in figure 3 are due to the strain rate dependence of T_{BDT} which results in a temperature band rather than a well-defined transition temperature. Although in figure 3 the scatter in the data is quite large (this could possibly be due to the different strain rate range investigated in each material), it is clear that for a variety of materials including metals, semiconductors and insulators, there is a nearly constant ratio (~ 25) between ΔH_{BDT} and T_{BDT} over a wide range of temperatures and activation energy values. The similarity with known $\Delta H_{\text{s}}/kT$ values deduced from flow stress measurements (where dislocation mobility is certainly the controlling factor) is a strong indication that the BDT process

**Figure 2.** Arrhenius plot of the strain rate versus $1/T_{\text{BDT}}$ for single and polycrystal iron and single-crystal vanadium.

is controlled by dislocation velocity in at least the materials reported in table 1 and figure 3. If we assume that dislocation motion, especially in bcc metals [2], is by formation and propagation of kink-pairs, a fixed ratio of $\Delta H_{\text{BDT}}/kT_{\text{BDT}}$ may suggest that the number of atoms involved in the formation of a kink-pair is constant (~ 25 or less) in all the materials reported here. However, understanding the physical meaning of this number certainly requires further study and analysis.

4. Conclusions

The activation energy controlling the BDT in tungsten, vanadium and iron has been recently determined. The comparison of these results with other BDT data available in the literature for different materials (obtained using various techniques) suggests that the ratio between ΔH_{BDT} and T_{BDT} gives approximately the value 25 in all the materials

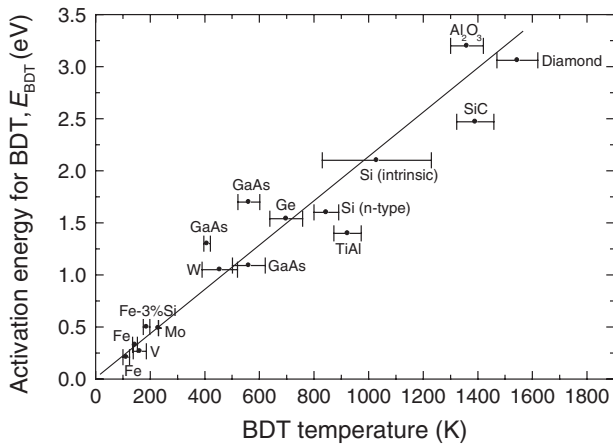


Figure 3. The activation energy for BDT in different materials plotted as a function of BDT temperature. Data points indicate the average BDT temperature taken within the temperature range investigated. Data for W, V, Fe are from this study. Other data are from: Si [7], GaAs [21], Ge [8], Al_2O_3 [9], Mo [16], SiC [15], TiAl [22], diamond [23] and Fe-3%Si [24].

investigated. The explanation for this number is still unclear; however, the empirical relationship $\Delta H_{\text{BDT}}/kT_{\text{BDT}} \approx 25$ could be used to estimate the mechanical behaviour of materials yet to be investigated in terms of T_{BDT} and/or thermal activation of dislocations.

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