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On the stress discrepancy at low-temperatures in pure iron

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Abstract

We present a complete analysis of the velocity of individual screw dislocations in pure iron, as a function of stress and temperature, by means of in situ straining experiments in a transmission electron microscope. The results show a very pronounced deviation from the classical laws of thermodynamics, which is at the origin of the discrepancy between experimental and calculated deformation stresses at low temperature. This strongly supports the occurrence of large quantum effects proposed in a recent theoretical study.

Keywords: Dislocations; Transmission electron microscopy; In situ experiments; Iron

1. Introduction

The plasticity of body-centred cubic (bcc) metals is achieved, as in most crystals, by the glide motion of dislocations, which, at low temperatures, obeys several fundamental properties of the crystal structure:

- Because of the lattice periodicity, dislocations parallel to dense directions are subjected to periodic variations of core configuration and corresponding core energy.
- Because of the rotational symmetry around the $\langle 111 \rangle$ densest direction of slip, the glide resistance is the highest for screw dislocations which have a threefold non-planar core structure.

These properties, first described by Peierls [1] and Nabarro [2], are now part of all models of low-temperature plasticity. They are well described by the so-called Peierls periodic potential, of height ΔE and periodicity h, in which screw dislocations move either by nucleation and propagation of kinks (the kink-pair mechanism) or by other, closely related mechanisms [3]. Screw dislocation cores and Peierls potential shapes have subsequently been computed by various methods [4–9].

Classical models are, however, still unable to predict the correct value of the deformation stress extrapolated to absolute zero temperature (0 K), which indicates that some unexpected phenomenon has not been taken into account [10–12]. Indeed, in pure iron, the computed Peierls stress necessary to overcome the Peierls potential in the absence of any thermal activation [4–9] is 2.3–4.3 times larger than the experimental critical resolved shear stress measured down to liquid helium temperature [13–17], depending on the stress orientation, method and potential used.

This stress discrepancy is a major difficulty in the physics of dislocations and plastic deformation of metals. Two types of interpretations have been proposed so far. The first one is based on the presumably easier collective motion of dislocations in networks [18] or at pile-up heads [11]. The second one is based on quantum effects taking place either below 30 K, according to Kuramoto et al. [14] and Brunner and Diehl [15], or in a larger temperature range below half the Debye temperature (namely 235 K), according to the recent article of Proville et al. [12]. This second kind of interpretation is extremely attractive because it can account for a substantial softening in a fairly large temperature range consistent with experimental observations.

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Quantum (or other unexpected) effects can be identified through a thermodynamic analysis of the velocity of screw dislocations. According to the classical laws of thermodynamics, this velocity can be expressed as [19,20].

$$v = v_0 \exp - \frac{G(\tau)}{kT}$$

where $v_0 = \alpha v_D b$ (v_D is the Debye vibrational frequency, b is the Burgers vector and α is slightly higher than unity for Peierls-type mechanisms), k is the Boltzman constant, T is the temperature, G is the Gibbs activation energy and τ is the local shear stress. For a constant dislocation velocity v of the order of 10 nm s^{-1}, this equation can also be written as

$$G = kT \ln \frac{v_0}{v} \approx 26kT \tag{1}$$

This equation is approximately verified in iron at the athermal temperature of 330 K, where G is twice the kink energy, which is 0.65 eV according to atomistic calculations [21].

The stress dependence of G is described by the activation area

$$A = -\frac{1}{b}\frac{\partial G}{\partial \tau}\bigg|_T = \frac{kT}{b}\frac{\partial \ln v}{\partial \tau}\bigg|_T \tag{2}$$

At a constant dislocation velocity, we have

$$d \ln v = \frac{bA}{kT} d\tau + \frac{G}{kT^2} dT - \frac{1}{kT}\frac{\partial G}{\partial T}\bigg|_\tau dT = 0, \text{whence}$$

$$G = -TbA\frac{\partial \tau}{\partial T}\bigg|_v + T\frac{\partial G}{\partial T}\bigg|_\tau = H - TS \tag{3}$$

where H is the activation enthalpy. In the classical approximation, the entropy $S = \frac{\partial G}{\partial T}\big|_\tau$ mainly corresponds to the temperature variation of the elastic constants, which is negligible at low temperature. Under such conditions, combining Eqs. (1) and (3) yields

$$H = -TbA\frac{\partial \tau}{\partial T}\bigg|_v \approx G = 26kT \tag{4}$$

Since A and $\frac{\partial \tau}{\partial T}\big|_v$ can be measured, Eq. (4) can be checked experimentally. If verified, the classical laws of thermodynamics apply. In the alternative case, explanations must be found for an unusual large entropy, or for an anomalous pre-exponential factor v_0.

This procedure was used by Kuramoto et al. [14] and later by Brunner and Diehl [15], though not through direct dislocation velocity measurements. Indeed, the conventional mechanical tests used by these authors yielded only the strain rate $\dot{\varepsilon}$, which is related to the dislocation velocity v by the Orowan equation $\dot{\varepsilon} = \rho b v$, where ρ is the mobile dislocation density. Since ρ is usually dependent on stress and temperature, the results can be significantly altered and difficult to interpret unambiguously. In particular, the "apparent" activation area

$$A_a = \frac{kT}{b}\frac{\partial \ln \dot{\varepsilon}}{\partial \tau}\bigg|_T \tag{5}$$

can be substantially different from the real one given by Eq. (2).

Following this procedure, an unusually low enthalpy has been found below 30 K, and interpreted by quantum effects [14,15]. However, since the corresponding softening is too weak to account for the stress discrepancy at 0 K, possible quantum effects across a larger temperature range (as predicted by Proville et al. [12]) must be checked avoiding the above approximations.

In order to determine real activation areas, measurements must be made at the scale of individual dislocations. Here we present a complete set of experimental dislocation velocities, measured at the nanoscale and as a function of stress and temperature. All data (except at 300 K) are issued from a single grain of a polycrystalline microsample of pure iron, strained in situ in a transmission electron microscope between 95 and 300 K, where two dislocation families have been observed under stress. Other aspects of the motion of dislocations can be found in Refs. [22,23].

2. Experimental procedure

Polycrystalline high-purity iron (less than 15 ppm-wt. of C, N, O and Si as a whole) was prepared at the Ecole des Mines of St Etienne (France). Rectangles of 3 × 1 mm size, with a thickness of 50 μm, were cut by spark erosion machining, then mechanically and electrolytically polished to perforation at their center. They were glued to a GATAN nitrogen-cooled straining holder and observed in a JEOL 2010HC transmission electron microscope operated at 200 kV. Dynamic sequences were recorded by a Megaview III camera operating at 25 images s^{-1}. As discussed in Ref. [24], deformation starts in the two zones where the stress is concentrated, which is where the tensile axis is tangential to the thin edges. The local stress direction remains at ±10° from the external one in these zones, in such a way that the activated slip systems can be roughly predicted by the Schmid's law. Artifacts like kink nucleation at the free surfaces have been avoided by selecting suitable orientations of foil plane and stress direction [25].

Observations were carried out in a grain with a $(\bar{1}15)$ sample surface, and $[\bar{2}\bar{1}0]$ strain direction. Dislocations with $1/2[111]$ and $1/2[\bar{1}\bar{1}1]$ Burgers vector were identified through conventional $\mathbf{g} \cdot \mathbf{b}$ analysis, and observed in two-beam conditions with the diffraction vector $[110]$ at a tilt of −6°. Image distortions due to projection effects have been corrected assuming that dislocations lie in their average slip plane. Under such conditions, the stress and velocity measurement accuracy is of the order of ±20%, as a result of (i) the difference in position between the real dislocation and its image, (ii) the complex projection effects when slip planes are highly wavy, (iii) the inhomogeneous internal stress due to neighbouring dislocations and (iv) the irregular instantaneous velocity resulting from the jerky