TEM in situ straining experiments in Fe at low temperature

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Abstract. In situ TEM straining experiments were carried out in pure Fe, to investigate the origin of the discontinuity observed at 250K in the temperature variation of the deformation activation parameters. The results show that the motion of screw dislocations is steady at 300K, in agreement with a kink-pair mechanism, but jerky at 110K. This change has been attributed to a transition from a kink-pair mechanism to a locking-unlocking mechanism, similar to that observed previously in Ti.

1. Introduction
The low-temperature mechanical properties of body centred cubic (BCC) metals exhibit all the characteristic features of the kink-pair mechanism, controlling the motion of screw dislocations with extended cores (see reviews by Kubin [1], Christian [2], Suzuki et al [3], Duesberry [4], Taylor [5]). In particular, there is a rapid decrease of the flow stress at increasing temperature, and activation volumes are fairly small.

In iron single crystals, the investigations of Quenel et al [6], Kuramoto et al [7], and Brunner and Diehl [8], show that the stress versus temperature curve exhibits a discontinuity at around 250K, to which corresponds a peak in the variation of the activation volume. These discontinuities indicate a change of controlling mechanism at 250K. Such a behavior is not specific to pure iron, as it is also observed in Nb and Nb alloys, Mo, as well as in hexagonal close packed metals deformed in prism slip (see a review in [9]).

In BCC metals, discontinuities have been ascribed to several possible effects: i) the occurrence of anomalous slip (in Nb and Mo, not in Fe where anomalous slip has never been reported), ii) a change in the kink-height, provided the Peierls potential has a "camel hump" shape (Guyot and Dorn [10], Takeuchi [11], Aono et al [12], Koizumi et al, [13]), iii) a transition between the low-stress and high-stress regimes of the kink-pair mechanism (Statham et al [14], Kubin and Jouffrey [15], Brunner et al [16], Groger and Vitek [17]), iv) a change of the kink-pair plane from {110} to {112} (Brunner and Diehl [18, 19]). These interpretations are however not fully convincing, because they have never been verified experimentally, and because (i) is restricted to Nb and Mo, (iv) cannot be transposed to hexagonal close-packed metals, the camel-hump potential assumed in (ii) is not reproduced by recent ab initio calculations [20], and the sole modification of kink-pair mechanism, proposed in (ii)-(iv), seems incompatible with the huge variation of pre-exponential term.

The aim of the present study is thus to perform in situ TEM experiments in pure Fe at different temperatures, in order to detect potential changes in the dynamical behaviour of dislocations that could be related to the discontinuity at 250K.
2. Experimental procedure
A large-grain polycrystal of pure iron, containing 2.5 to 4 ppmwt (ppm in weight) of C, N, O, and Si (15 ppmwt as a whole), and less than 2 ppmwt of other elements, has been provided by J. Le Coze, Ecole des Mines of St. Etienne, France. Microsamples were prepared as described in [21], and strained in a JEOL 2010HC transmission electron microscope. Two Gatan straining holders were used, one working at room temperature, and the other (lent by R. Schaeublin, PSI, Switzerland) between 110K and room temperature. The microsamples were deformed by series of constant strain-rate deformations followed by relaxations, at an average strain-rate of the order of $10^{-6}$ to $10^{-5}$ s$^{-1}$. The videos were recorded by a Megaview III camera, at a frame rate of 25 images per second, and analysed frame by frame.

3. Results
Fig. 1 shows a dislocation source anchored at one extremity, in S, in a microsample strained at room temperature. The screw segment labelled 1 moves to the top-left between $t = 0$ and $t = 3.60s$ (note the anchoring in P). When both opposite screw segments 1 and 2 are sufficiently widely separated, the curved edge portion moves rapidly to the top, emerges at the foil surface, and leaves the slip trace tr ($t = 3.60s$ to $t = 3.64s$). The velocity of this motion is too high to be measured with the time-resolution of the camera (0.04s between two successive frames). Dislocation 1 moves on to the top-left, whereas dislocation 2 moves in the opposite direction ($t = 3.80s$ to $t = 6.68s$). Then, the second curved edge portion escapes rapidly at the opposite free surface, and the rotation resumes. Several tens of successive rotations of the dislocation source have been observed at 300K, with the same characteristics, summarized as follows:

i) the velocity of screw dislocations is considerably lower than that of edge and other non-screw segments, which results in a large density of moving straight screw dislocations.

ii) slip traces are generally wavy, and do not correspond to any low-index slip plane. In some cases, however, cross-slip is less abundant, and slip occurs preferentially in \{110\} planes.

iii) screws move steadily, at a velocity which increases with their length.

At low temperature, screw dislocations are much straighter than at room temperature, and the edge parts are more highly curved. Their kinetics is also very different from the above one. Fig. 2 shows a...
group of dislocations under stress at 110K. One dislocation (noted d) moves very quickly over ~ 20nm between the two first frames of the sequence. Then, nothing occurs during almost one second, till the same dislocation jumps again. Other dislocations have the same jerky motion in the following of the sequence (not shown).

4. Discussion

Except for the activation of anomalous slip, which has not been observed in Fe, the explanations proposed for the discontinuity in the mechanical properties at 250K are all based on a change in the properties of the kink-pair mechanism. This mechanism is characterized by jumps of screw dislocations between adjacent Peierls valleys, which results in the steady motion of rectilinear dislocations at the scale of in situ TEM observations.

The observations at 300K are fully consistent with the kink-pair mechanism. The jerky motion observed at 110K is, however, difficult to describe in the same way. Indeed, a simple evaluation shows that it cannot be due to any variation of the local stress, e.g. due to elastic interactions with neighbouring dislocations. It is not either due to interactions with extrinsic obstacles like impurity atoms, because the jump length decreases with decreasing temperature. Under such conditions, the change of kinetics occurring between 110K and 300K reveals a change of controlling mechanism, which probably corresponds to the discontinuities discussed in the introduction.

A similar change in the kinetics of screw dislocations occurs in titanium deformed in prismatic slip around 300K, in correlation with a similar peak in the activation area versus stress or temperature. It has been ascribed to a change in the mechanism of dislocation motion, from the kink-pair one above 300K, to the locking-unlocking one below 300K [22, 23]. The locking-unlocking mechanism is a variant of the kink-pair mechanism, where dislocations extracted from Peierls valleys can glide freely over a large number of inter-atomic distances, before being locked again. Since the experimentally measured free-glide distance decreases with increasing temperature, there is a transition from the locking-unlocking mechanism to the kink-pair one, when this distance reaches the kink-pair height. As shown in [9], this transition naturally induces a peak in the stress or temperature dependence of the activation area, and a discontinuity in the stress versus temperature curve.

In Fe, we propose that the discontinuities measured at 250K have the same origin as in Ti, i.e. they are related to a transition between the locking-unlocking mechanism, below 250K, and the kink-pair one above.

Fig. 2: Dislocation motion in pure Fe at 110K. The subtractions of the different successive images underline the jerky motion of dislocation d.
5. Conclusions
In situ straining experiments in pure Fe have revealed different kinetics of screw dislocations at 300K and 110K:
- At 300K, screw dislocations move steadily, at a velocity which depends on their length, as expected from the kink-pair mechanism
- At 110K, screw dislocations have a jerky motion, with jumps longer than several tens of nanometers, separated by locked positions that last up to several seconds. This jerky motion is inconsistent with the kink-pair mechanism.
This change of behaviour has been compared to that studied previously in titanium, and ascribed to a transition from a kink-pair mechanism to a locking-unlocking one. It is likely to be responsible for the discontinuities in the activation parameters of the deformation, observed at 250K.

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