WHAT CAN BE LEARNT FROM TEM IN-SITU STRAINING EXPERIMENTS?

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A few examples are given to demonstrate the key role of TEM in-situ straining tests in the investigation of dislocation mechanisms. This technique provides quantitative information on the kinetics of these phenomena, like dislocation velocities or mobile dislocation densities, but also in evidencing subtle mechanisms that would have left no record in post-mortem observations. In addition to individual behaviour controlled either by individual dislocation propagation or by dislocation nucleation, the collective behaviour of a large number of interacting dislocations also proved to be accessible by in-situ TEM through a statistical analysis of elementary events. These events (e.g. dislocation jump lengths) often reveal a scale invariance. Such a scale invariance is also observed at larger scales, e.g. by the measurement of dislocation avalanches using acoustic emission.

Key words: Transmission electron microscopy in-situ, in-situ straining tests, dislocation nucleation and propagation, plasticity

CO SE LZE DOZVĚDĚT Z DEFORMAČNÍCH EXPERIMENTŮ IN-SITU PROVÁDĚNÝCH V TEM?

Několik příkladů ukazuje důležitou úlohu deformačních zkoušek in-situ v TEM při studiu dislokačních mechanismů. Tato metoda umožňuje získat kvantitativní informace o kinetice jevů, jako je rychlost dislokací, hustota pohyblivých dislokací, ale zároveň poskytuje důkaz o subtilních mechanismech, které nelze metodami konvenční TEM post mortem pozorovat. Kromě chování jednotlivých dislokací, které je řízeno buď šířením jednotlivých dislokací, nebo jejich nukleací, umožňuje metoda TEM in-situ rovněž získat informace o kolektivním chování velkého počtu vzájemně interagujících dislokací pomocí statistické analýzy jednotlivých elementárních událostí. Tyto události (např. délky přeskoků jednotlivých dislokací) často vykazují rozměrovou nezávislost, která byla rovněž pozorována v makroskopickém měřítku, např. měřením velikosti dislokačních lavin metodami akustické emise.

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1. Introduction

Disregarding the case of low stresses and high temperatures where diffusion processes may be dominant, plasticity of crystalline materials is governed by Orowan's equation. This equation can be written in two different ways according whether plasticity is controlled by the average velocity V of a density ρ of pre-existing mobile dislocations (Eq. 1), or by the nucleation rate of dislocations that subsequently travel a distance Λ:

$$\dot{\varepsilon} = \rho\, b\, V, \tag{1}$$

$$\dot{\varepsilon} = \dot{\rho}\, b\, \Lambda. \tag{2}$$

Except for the dislocation mean free path Λ which is usually (though not always) related to microstructure (e.g. grain size), these microscopic parameters are only accessible through so-called dynamic observations of dislocations, which are illustrated by a few examples hereafter. We shall also show that TEM in-situ testing can provide valuable quantitative information in the more general and complex case where plastic activity results from the collective behaviour of a complex system of mutually interacting dislocations driven by an external stress.

2. Experimental techniques

TEM in-situ observations of dislocation activity require a specimen thickness large enough to avoid, for instance, thin foil artifacts related to microstructure scale or surface effects, and an acceptable penetration of electrons. The use of high voltage microscopes may be of interest to this respect, since beautiful records of dislocation activity were obtained with foil thicknesses as large as 1 or 2 μm [1]. However, radiation damage significantly limits the electron beam energy to a typical value of 300 to 400 kV, except for high threshold voltage materials like BCC metals.

Many types of specimen holders were designed in the past. In contrast with the top entry technique widely spread in Japan, the ones used in the following examples [2] are of the side entry double tilt type, and allow tensile straining and heating up to about 800 ℃.

3. Velocity controlled plasticity

3.1 Velocity measurements

It may be thought that the simplest type of measurement that can be carried out during in-situ testing is that of dislocation velocity. However, owing to the usual spatial and temporal heterogeneities of plastic deformation at the mesoscale, this task may be easily performed only in the case of a strong lattice friction responsible for sluggish dislocation motion. In the case of diamond cubic structured semiconductors like Si or Ge for instance, and owing to covalent bonding, dislocation loops consist of screw and 60° segments, both of them experiencing a strong lattice friction. The mobility of such dislocation segments is controlled by i) the formation energy of a kink pair, and ii) by the kink migration energy [3]. Their velocity therefore depends on whether their lengths are smaller or larger than the mean free path of kinks. In the first case, a single kink pair nucleation brings the whole segment into the next valley, which results in a dislocation velocity proportional to the number of possible nucleation sites, i.e. to its length. In the second case, kinks of opposite signs meet and annihilate, which corresponds to a saturation in the length dependence of the dislocation velocity. Temperature and stress dependence of dislocation velocities in Si was measured by X-ray topography [4] but the spatial resolution of this technique did not allow any measurement in the short length regime. In contrast, TEM in-situ measurements of dislocation velocities in the two length regimes (Fig. 1) could be performed, and allowed a direct determination of these two activation energies [5].

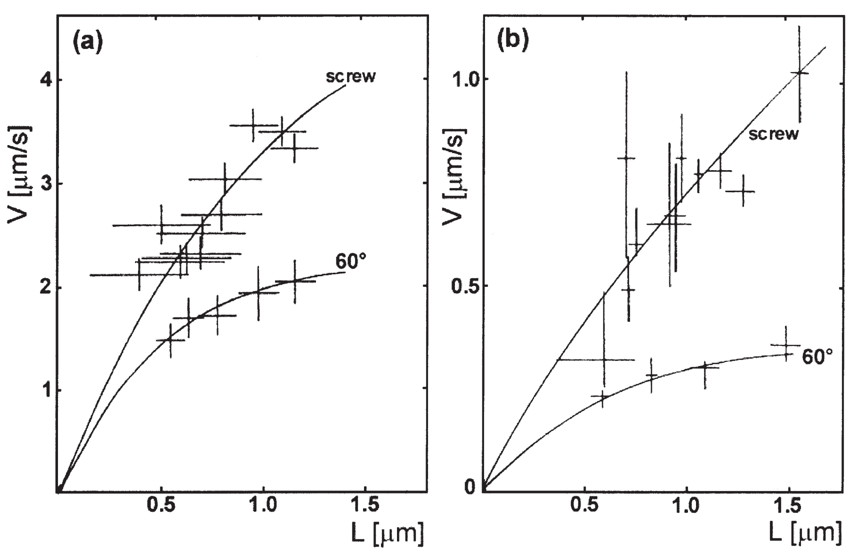

Fig. 1. Dependence of dislocation velocities V on their length L in Ge, as measured from high voltage electron microscopy in-situ experiments: (a) T = 703 K, σ = 40 MPa, (b) T = 678 K, σ = 35 MPa.

3.2 Mobile dislocation density estimates

During straining, the mobile dislocation density ρ may significantly differ from the total dislocation density, which makes post-mortem measurements (etch pits, static TEM observations, etc.) somewhat hazardous, but TEM in-situ tests promising. Indeed, in some simple cases the mobile dislocation density may be directly measured from in-situ records, for instance for viscous motion resulting from a strong lattice friction. However, owing to heterogeneity of plastic deformation, such a direct measurement is hardly applicable to other cases. Since the mobile dislocation density results from a balance between multiplication and annihilation (or locking) mechanisms, ρ may instead be theoretically determined (even if absolute estimates of ρ cannot be obtained) if the kinetics of these mechanisms are known. In this respect in-situ TEM straining provides a powerful means of investigation. We shall give hereafter two examples of multiplication and locking mechanisms that should not have been probably discovered without the help of in-situ testing.

The first one is a particular multiplication mechanism evidenced in pearlitic steels (Fig. 2). So-called scolopendra sources operate through dislocation propagation in ferrite around cementite islands, yielding dislocation multiplication [6]. When a dislocation rotating around a cementite island comes at the island tip, it turns back into the adjacent channel, leaving a new dislocation segment that propagates in the facing corridor. It can be easily imagined that, in the bulk, dislocations propagating in the ferrite matrix and meeting the cementite island actually overcome it through a kind of Orowan by-passing process. Yet, owing to the BCC nature of the ferrite lattice, the main difference with such an Orowan mechanism is that the two dislocation segments propagating on both sides of the island have a screw character and experience a series of cross slip events. They do not lie any more in the same slip plane at the exit, and since they cannot recombine, they pass over each other, and move back in the opposite channels, leaving two new segments that propagate into the facing channel. This is equivalent to the combination of the clockwise mechanism shown in Fig. 2 with the symmetrical (anticlockwise) one. The operation of this type of source requires sufficiently wide channels in order to propagate dislocations at reasonable stress levels.

The second example is a locking mechanism responsible for exhaustion of superdislocations in FeAl (B_2) alloys. Fig. 3 shows a superdislocation cross-slipping into a plane in which the remaining segment is essentially of edge character. This edge segment eventually develops a screw part that cross-slips again in the primary plane in which it keeps a screw character. It can be easily seen that edge segments are very often pinned, and subsequently drag defects that are left in the dislocation wake, but which disappear after a few seconds. This mechanism has never been observed for screws. The defects produced by edges are therefore understood as follows: in this material, the thermodynamic vacancy concentration varies quite

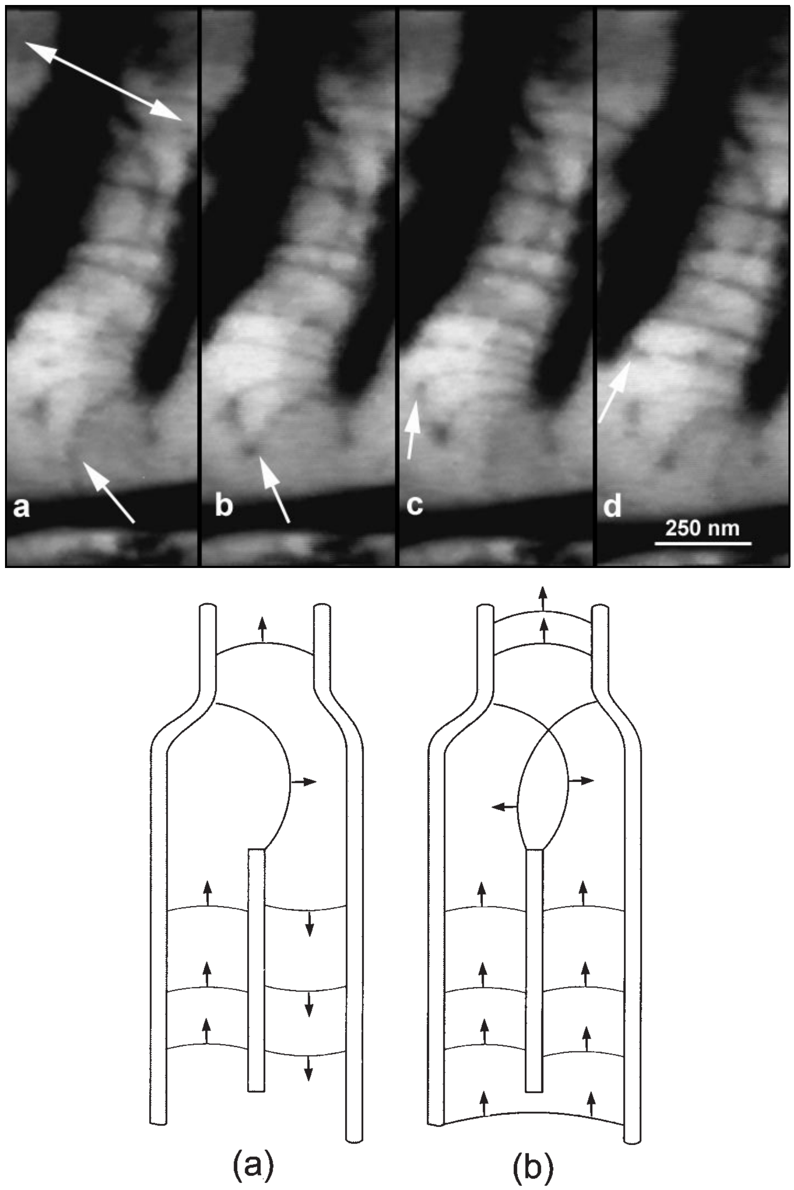

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Fig. 2. (i) In-situ observation of a scolopendra source: dislocations (marked with white arrows) rotate around the bottom end of the cementite island and pile up into the channel. In the bulk, such a mechanism leads to dislocation multiplication. The applied stress is marked with the white double arrow. Dislocations are elongated along the $\langle 111 \rangle$ screw direction. (ii) Scolopendra source mechanism: (a) as observed in situ, (b) probable mechanism acting in the bulk, producing two new dislocations at a time (see the text).

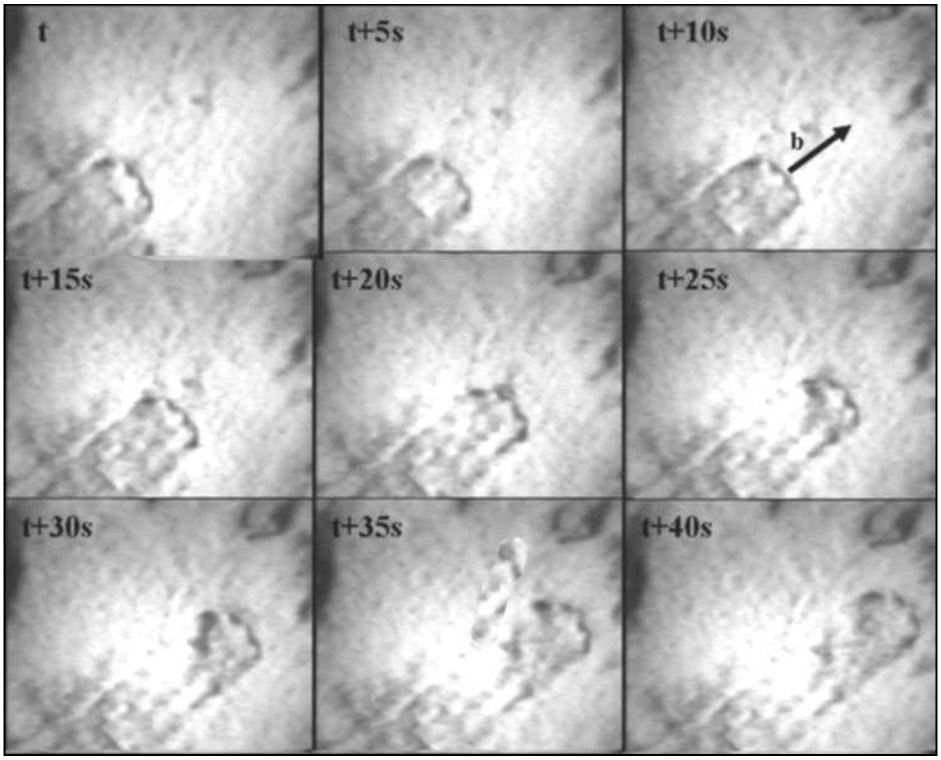

Fig. 3. In-situ observation of dislocation motion in a FeAl (B2) alloy: screw dislocations segments move easily, but when they turn to edge character, sluggish motion is observed, associated with production of defects left in the dislocation wake. These defects, that are likely to be APB tubes (see text), disappear after a few seconds, which excludes any post-mortem characterisation.

rapidly with temperature. At high temperatures, the leading edge dislocation absorbs a large number of vacancies lying randomly in its slip plane, which results in local climb. The trailing partial cannot follow the same path, since no vacancy has been left in the plane. Its motion cannot erase the APB, and an APB tube is left in the dislocation wake. The corresponding dragging force can be estimated from the APB energy and turns out to be quite large. This mechanism is therefore thought to be responsible for both the exhaustion of superdislocations and the associated stress anomaly in FeAl (B_2) [7].

4. Nucleation controlled plasticity

Plasticity of nanostructured materials is characterised by a drastic change in the plastic properties for lamellae thicknesses or grain sizes lower than a critical value of a few tens of nanometers. The flow stress, which normally is a decreasing function of this size, saturates or sometimes softens as the characteristic scale is decreased below such a critical value [8]. As the stress necessary to bow out dislocations, i.e. to operate conventional dislocation sources and to propagate dislocations in a layer or within a nanograin, is expected to drastically increase when the layer thickness or grain size goes down [9], other mechanisms than conventional dislocation processes are likely to operate beyond this stage.

It seems probable that grain boundaries, which contain a significant proportion of atoms in these fine scaled structures, participate in the overall deformation through grain boundary sliding, especially, when the structure scale becomes smaller. These views seem to agree with the fact that post-mortem TEM observations carried out at the nanometric scale are scarcely able to show dislocations, which may indeed support the idea that dislocations are not involved in mechanisms responsible for plastic flow. However, such a deformation may generate plastic incompatibilities between grains that have to be accommodated (and possibly controlled) either by diffusional deformation of grains or by some shearing of grains in order to preserve crystal cohesion. At sufficiently high stresses, which is the case considered here, shearing of grains should be preferred to diffusional deformation. Atomistic simulations by Schiøtz [10], also performed at high stresses, suggest that grain shear may proceed through a succession of dislocation nucleation events at grain boundaries, followed by a rapid propagation through the grain and an annihilation on the opposite grain boundary, as proposed by Li [11] a long time ago. Though involving dislocation motion across grains, this mechanism is not expected to leave any visible dislocation in grain interior. A dislocation in a small grain, if not stabilised by interactions with other defects within the grain or at grain boundaries, is unstable, and is expected to move to the nearest grain boundary and to annihilate there in order to reduce its line energy. Therefore, the fact that dislocations are not observed inside nanograins does not mean that they do not participate in strain in a significant way.

This assumption of dislocation nucleation at interfaces was still to be validated experimentally. It has been done recently by TEM in-situ observations of nucleation and expansion of half loops in the ferritic phase of pearlitic wires [6] (Fig. 4). Somewhat similar observations were reported by Anderson et al. [12]. However, in this latter case the observed phenomenon clearly represents a dislocation transmission through a coherent interface occurring several times at the same place, rather than a random nucleation from the interface toward the softer phase. It must be emphasized that the above-mentioned nucleation mechanisms are not classical multiplication processes as they do not restore the initial configuration, and hence cannot resume the same process forever (this also means that plastic flow is likely to be controlled by nucleation rather than by propagation). However, they

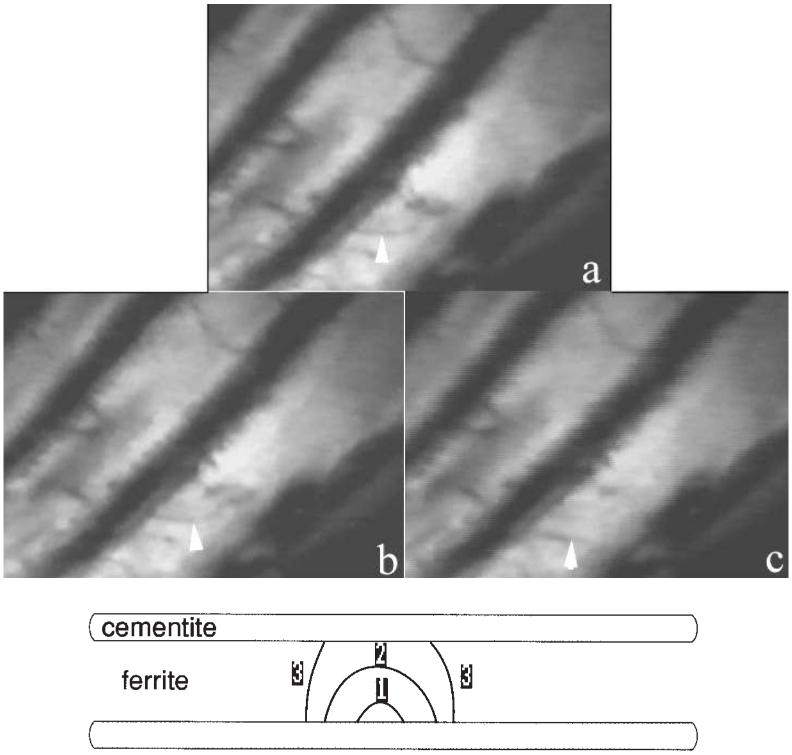

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Fig. 4. Nucleation of a dislocation half loop at a ferrite/cementite interface in a pearlitic steel (arrow), (i) in-situ observation, (ii) schematic drawing of the mechanism. After nucleation (1), the loop crosses the ferrite channel and impinges onto the opposite interface (2). In some cases the generated dislocations may propagate along channels (3).

might account for a specific dependence of the yield stress on lamellar thickness as discussed now.

If deformation proceeds by the propagation across lamellae of dislocations nucleated at interfaces, the type of dependence of yield stress on the lamella thickness can be obtained through a simple dimensional argument: the nucleation rate is proportional to the number of interfacial sites, which scales as $1/d$, as previously noted by Spitzig [13]. In turn, each nucleation and propagation event provides a strain increment proportional to the lamella thickness d. The strain rate proportional to the product of the nucleation rate by the corresponding strain increment is thus independent of d, and so is the yield stress in the lamellar thickness range where the present mechanism controls plastic flow, as experimentally observed, e.g. in CuCr multilayers [8]. In this case again, TEM in-situ experiments were able to give a key information about the basic mechanisms responsible for this particular behaviour.

5. Plasticity as the critical behaviour of a complex ensemble of interacting objects

Orowan's description of dislocational plasticity is essentially a mean field approach. If examined more closely, the plasticity actually consists of a slowly driven motion of a large number of elastically (i.e. long range) interacting objects, in some way similar to the sand pile problem on which the concept of self organised criticality (SOC) [14] was founded. According to the SOC theory, such a complex

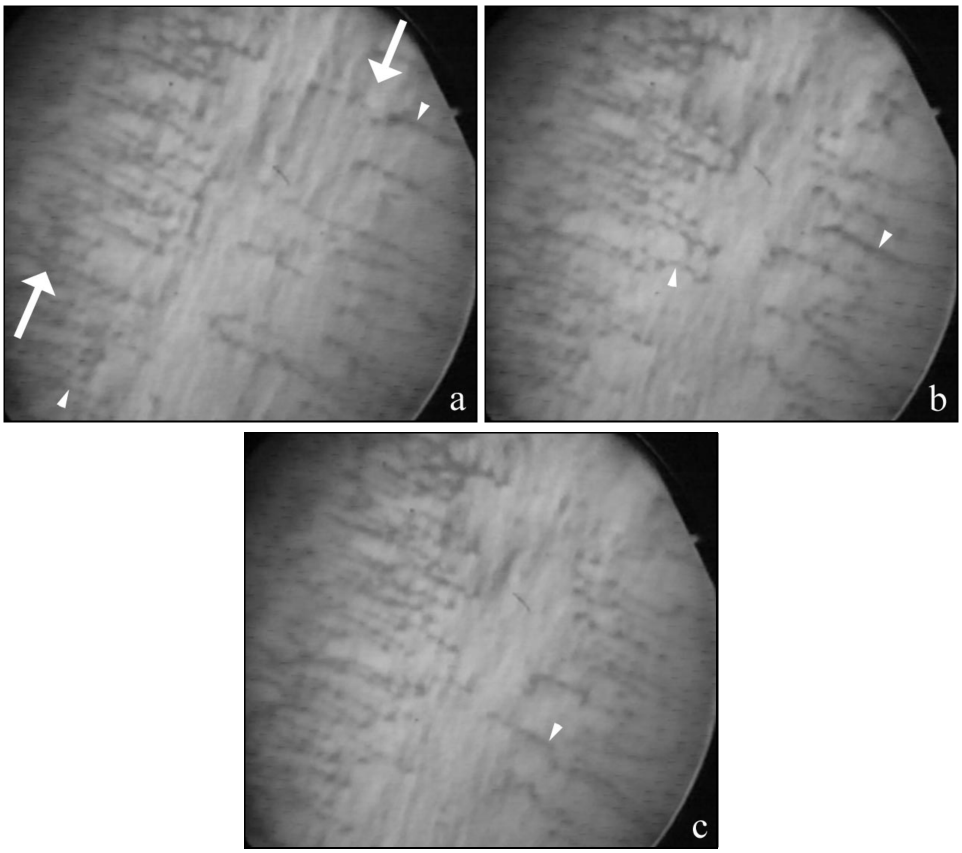

Fig. 5. Slip localisation in a Fe50%Cr alloy aged 40 h at 400 °C. Screw dislocation motion proceeds in a jerky way, and reveals strong elastic coupling (in a, b, and c three successive snapshots of in-situ recording are shown). The direction of dislocation motion in the band is marked with white arrows, the position of the same dislocation in each band is marked with small white pointers.

system reacts to a slow loading rate through a series of avalanches whose sizes range on a rather wide scale, and the statistical distribution of avalanche sizes is expected to be scale invariant, which means that the occurrence frequency $f(s)$ of an avalanche has a power-law dependence on the avalanche size s,

$$f(s) = s^{\alpha}, \tag{3}$$

where α is called critical exponent.

Such a critical behaviour was already evidenced in plasticity, at the centimetric scale, using acoustic emission during deformation of ice crystals [15]. It was therefore interesting to check whether such a behaviour could also be observed at the micrometric scale. For that purpose, we chose the FeCr system that combines a strong hardening due to spinodal decomposition with a significant Peierls friction characteristic of BCC metals [16, 17]. Local disordering of concentration modulations due to dislocation shearing results in a strong slip localisation, that enhances dislocation elastic interactions. We observed a jerky motion of screw dislocations during TEM in-situ straining (Fig. 5), with obvious coupling between jump events. The statistics of dislocation jumps were extracted from these observations. The Pickering procedure [18] was used to correct the possible artefacts of the relatively small number of recorded events, and it converged after a single iteration. Fig. 6 shows that the data align on a straight line in a log-log scale, which confirms the expected scale invariance. Further work is needed to relate the measured critical exponents with the physics of the observed phenomena.

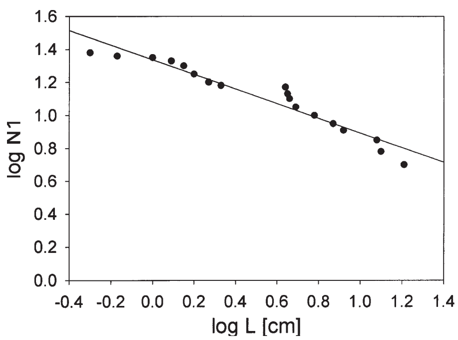

Fig. 6. Double logarithmic plot of dislocation jump length frequency $N1$ vs. dislocation jump length L. The straight line reflects a power-law characteristic of scale invariance.

6. Conclusion

Dislocational plasticity results from a complex interplay between several competing or complementary elementary mechanisms ranging from the atomic scale up to the mesoscale.

Apart from classical concepts like dislocation pair annihilation or Frank-Read sources, the wide variety of more or less specific individual dislocation mechanisms involved in the plastic behaviour of crystals is far from being explored, both qualitatively and quantitatively. Though TEM post-mortem observations of prestrained samples provide a considerable amount of valuable information on these mechanisms, some of them remain inaccessible by these methods. The few examples given above show the role of TEM in-situ tests in measuring the kinetics of the explored phenomena, but also in evidencing subtle mechanisms that would have not left any record of their operation in thin foils used for static observations. These phenomena range between two extreme cases of individual behaviour, controlled either by individual dislocation propagation or by dislocation nucleation. Moreover, the more general case of the collective behaviour of a large number of interacting dislocations also proved to be accessible by in-situ TEM through a statistical analysis of elementary events. The observed statistics of dislocation jumps was shown to be scale invariant, which agrees with more global information gathered at a much larger scale, using for instance acoustic emission.

New and different possibilities in the exploration of microscopic dislocation mechanisms are also offered by 3d mesoscopic simulations [19, 20]. We strongly believe that future progress in the knowledge of basic plasticity processes will result from a clever combination of these two complementary techniques.

Acknowledgements

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