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Observing deformation in situ

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Standfirst: Marc Legros, Frédéric Momprou, and Daniel Caillard discuss the different aspects that influence the reproducibility and reliability of the characterizations with in-situ mechanical tests in transmission electron microscopes.

Mechanical in-situ transmission electron microscope (TEM) experiments consist in deforming a piece of material in a TEM while capturing the nanoscale mechanisms responsible for its plastic behavior, correlating with microstructures such as dislocations, twins and phases. The dream to see how matter deforms with measuring the contribution of each mechanism, understand how they combine, and ultimately explain macroscopic mechanical properties seems at reach. The development of new imaging tools, *micromechanical* holders and dedicated numerical methods concur to this possibility. In practice, however, beyond the gap that separates macroscopic and micro- or nanoscopic scales, and the complexity of microstructures, the reproducibility of such tests remains questionable and the caveats numerous.

One of the first concerns may be the interaction of the powerful electron beam (typically 100-300keV) with the sample and its potential changes on its structure. Despite the moderate heating generated by this interaction (at the origin of the first observation of a moving dislocation ¹), and the potential radiation damages on light metals that constituted an early concern², recent studies have reported a drastic alteration of mechanical properties of amorphous silica³ and more moderate changes in aluminum or gold⁴. The physical reasons for such lately reported variations are still largely not understood but technical and scientific advances including the ones described below should provide adequate tools to unravel them.

With the advent of focused ion beam (FIB) milling, TEM sample preparation has gained universality and precision but also generated worries regarding structural modifications as Ga ions both implant in the TEM sample and create irradiation damages. These damages can create or interact with dislocations, modifying the mechanical properties of the studied crystal⁵. They also hinder the observation of "intrinsic" defects which precludes clean characterization of a given microstructure⁶, an ubiquitous problem rarely put forward. While techniques exist to remove such preparation artifacts, electropolishing of bulk metals is usually considered as the most conservative method to avoid structural modifications.

Even with minimum modification, free surfaces may influence a given microstructure through image forces: those may favor the escape of dislocations, their dissociation or the rate of kink nucleation on their line. Their contribution depends on several factors such as surface properties (oxidation, etc.), studied phenomena, and sample thickness. Although generally weaker than the dislocation confinement effect there is no general criterium to quantify the influence of surfaces forces, which means that it should be discussed for each experiment.

The in-situ technique that first emerged consists in a miniaturized tensile test using an infinite screw type holder where no load is measured (conventional holder). These holders operate on millimeter-size TEM samples, usually electropolished, offering several micron square transparent area. Moreover, their stiffness is several order higher than that of the TEM sample so that burst-type events such as dislocation avalanches and Lüders slip bands can be only attributed to intrinsic factors of the material. Such events are frequently observed in *nanomechanical* holders such as in-situ nanoindenters, that appeared in the years 2000's. These tools include piezo-electric actuators and miniaturized load cells that grant stress measurements, sometimes at the expense of the rigidity of the whole. Strain bursts visible in the stress-strain curve of Fig. 1a for instance⁷, may be characteristic of small-scale plasticity, but also a result of the holder compliance or slow feed-back loop of its electronic.

These nanoindenter-type holders are often claimed as the first ones capable to generate stress-strain curves in a TEM, but early developments in high voltage electron microscope⁸ did so with conventional holders in the early 1970's. Such established technologies also have the advantage to allow for temperature control, spanning from liquid helium to very high temperatures⁹, capabilities currently inaccessible using piezo-based nanoindenters. Another complication inherent to compression is the very fast degradation of imaging conditions during in situ tests, partly visible in Fig. 1b. One reason is that TEM imaging conditions are very sensitive to small crystal rotations which inevitably occur during compression due for instance to buckling. Dark-field imaging, as employed in Fig. 1b is a way to overcome this, but the transformation of compression into tension using "push to pull" lithography patterned Si devices⁹ could be a more versatile option. Indeed, straining in tension helps maintaining imaging conditions by stabilizing microsample orientation after the yield. In all cases, technical improvements are needed to lift in-situ TEM tests to levels close to ASTM (American Society for Testing and Materials) standards. Micro-Electro Mechanical Systems (MEMS) may be one way to reach this goal, but again, this will be reserved to very specific samples (FIB made, nanostructures, thin films). Potential charge effects on capacitance readouts (displacement and load) should also be controlled¹⁰.

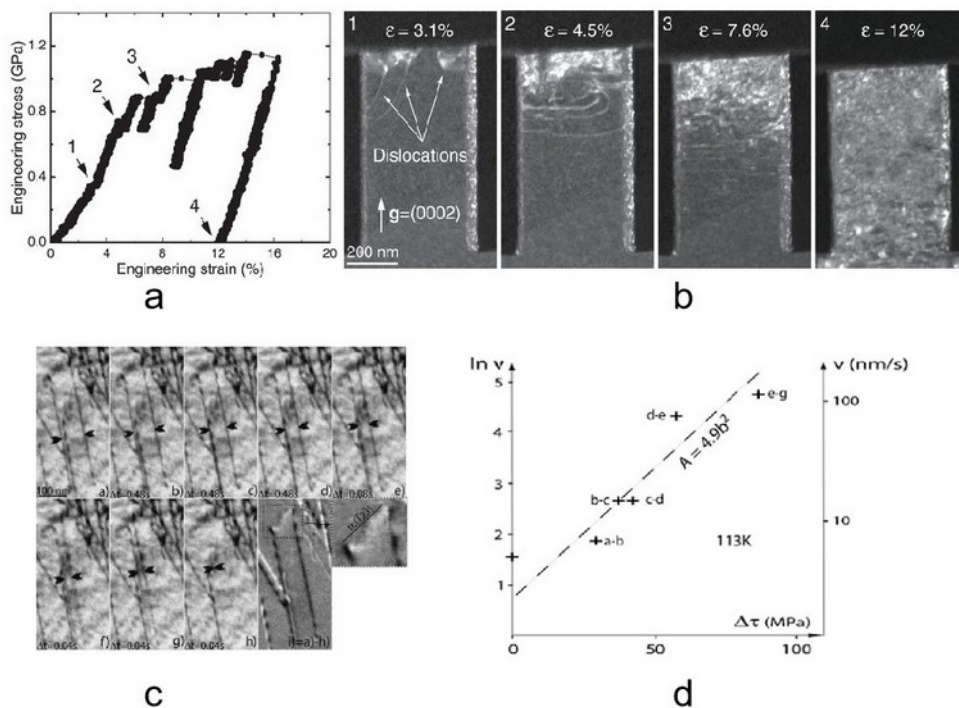


Fig. 1| Dynamical sequences captured during in-situ TEM straining or compressing experiments and associated mechanical properties. a Stress-strain curve obtained from the in-situ compression of a magnesium micropillar along its c-axis using a nanoindenter sample holder⁷, **b**) dark field TEM snapshots corresponding to arrows 1-4 in **a**, showing dislocations emitted from the top of the pillar at various stages of compression strain (3.1 to 12%) , **c** In-situ TEM sequence obtained using a

conventional straining holder on electropolished high purity Fe showing a screw dislocation dipole (black arrows) annihilation at 113 K. **d** Activation area A of the Peierls mechanism that governs the motion of dislocation in bcc crystals, directly inferred from such test (log of the velocity vs elastic interaction directly function of the dipole separation). A is obtained directly from the intrinsic properties of moving single dislocations¹⁴ (stresses and velocities marked a-h correspond to letters a)-h) in Fig.1c. **a** and **b** are reproduced with permission from Ref. 7, AAAS; **c** and **d** are adapted with permission from Ref. 14, Elsevier..

The disadvantage of not having load cells on conventional tensile straining holders may be turned positively because strain increments are perfectly controlled. Larger accessible volumes and steady imaging conditions also offer better statistics on dislocation behavior, and imaging quality allows measurements on single dislocation behavior, eventually using machine learning recipes¹¹. A common substitute to external load cell measurement is the use of dislocations curvature as local stress probes¹², an approach that matches macroscopic yield stress measurements in iron¹³ and several other metals and alloys in function of temperature, considering the small increase of stress due to confinement effect in the thin foil. Fig. 1c also exemplifies how a dislocation dipole annihilation is turned into a perfect single dislocation experiment¹⁴. There, both the elastic interaction stress and the velocity increase as the dislocations come close to each other, allowing a direct and fully local assessment of the activation volume linked to the Peierls motion of these screw dislocations in the bcc structure of Iron¹⁴. Such access to intrinsic dislocation properties is unique to in-situ TEM experiments, although very seldom exploited. They could also serve as a direct benchmark to atomistic simulations, either molecular dynamics (MD) or discrete dislocation dynamics (DDD)¹⁵.

Another common observation that may be noticed by the reader outside of the field about in-situ TEM straining publications is that they often report on single experiments, far from the scientific requirement to repeat an observation to fully validate its occurrence. In FIB-made samples or nanostructures, the limited volume accessible to dislocation, or their interaction with interfaces and obstacles, shrinks the available information even more. An obvious reason for this lack of repetition is that the success rate of such experiments is often very low. In any case, extra-ordinary behaviors should require more scrutiny from the whole community before being accepted as a fully valid statement and extrapolations on a possible macroscopic property drawn from it.

Competing interests: The authors declare no competing interests

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