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# Progress of *in situ* synchrotron X-ray diffraction studies on the mechanical behavior of materials at small scales

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In recent years, the mechanical behavior of low-dimensional materials has been attracting lots of attention triggered both by the ongoing miniaturization and the extraordinary properties demonstrated for nanostructures. It is now well established that mechanical properties of small objects differ fundamentally from their bulk counterpart and in particular that "smaller is stronger" but many questions on the deformation mechanisms remain open. Most of the knowledge obtained on small- scale mechanics is based on ex-situ and in-situ characterizations using electron microscopy. However, these techniques suffer from the fact that imaging or scattering information is either limited to the surface or from a 2D projection of a thin foil of material. Within the last two decades tremendous progress was achieved at 3rd generation synchrotrons making it possible to focus hard X-ray beams down to the 100-nm scale. Modern synchrotron X-ray diffraction methods may thus provide structural information with good spatial resolution and fully 3D. In this review, we discuss the progress achieved on in-situ micro- and nano-mechanical tests coupled with different synchrotron X-ray diffraction techniques to monitor the elastic and plastic deformation, highlighting the advantages of these approaches, which offer at the same time versatile sample environments and extreme precision in displacement fields.



**Contents**



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## 0. Introduction

The investigation of mechanical behavior in small dimensions and the influence of nanostructuring on mechanical properties constitute very fundamental and active research topics [1,2]. It is now well established that the mechanical behavior of small objects differ fundamentally from bulk material and more specifically that "smaller is stronger", i.e. nanomaterials exhibit higher yield strengths. Many unsolved questions arise, however, concerning the strength of small (below 1000–100 nm) objects. The role of dislocation nucleation and annihilation as well as the influence of atom diffusion at surfaces are still being debated. Within this review article we restrict ourselves mostly to monocrystalline metal and semiconductor micro- and nanostructures containing a limited number of defects while e.g. ultrafine grained materials are out of the scope.

The question of the influence of size on mechanical properties is certainly not new [3–5] but the development of micro and nanotechnologies together with very advanced characterization and fabrication techniques has allowed harvesting a wealth of detailed information and exploring smaller scales. In the 1980s the field of mechanical properties of coatings was first pioneered by Nix and his co-workers in Stanford [6] where mechanical properties of thin films have been evaluated as a function of film thickness and microstructure. The fundamental issues raised by these studies as well as the imperious need to master the thermomechanical reliability of electron devices has been recognized by other research groups worldwide. We shall not try being exhaustive here but one should at least quote the groups of Arzt in Stuttgart [7], Harper in IBM Yorktown Heights [8], Freund at Brown University [9] and Thompson at MIT [10]. More recently, many research works focused on testing the mechanical properties of nanostructures (islands, nanowires, etc.). The development of focused ion beam (FIB) microscopes has allowed to fabricating sub-micron pillars out of bulk materials [11,12]. Compression tests performed on such objects [11,12] have shown most of the time size effects. Tensile tests have also been reported [13]. The influence of defects induced by FIB-machining on these measured properties has been debated [14]. Mechanical testing of pillars prepared by other techniques such as direct deposition into pores [15] or wet etching [16,17] indicate that similar size effects can be observed even in the absence of any FIB-machining depending on the internal dislocation content [18]. Tensile testing performed on single-crystal Cu nanowires [19] evidenced tensile strengths close to the theoretical limit. The majority of studies reported in the literature are performed on simple FCC metals (Cu, Ni, Au, Al). More recently, BCC metals have been studied [16] and also semi-conductors such as GaAs [20] or Si [21]. Because of deep Peierls valleys semi-conductors are brittle at room temperature. Surprisingly this is not true anymore in small dimensions: small diameter semiconductor pillars exhibit ductile behavior [20].

The mechanical properties of nano-objects are being investigated by various ways. Tensile testing of Cu nanowires has been reported by Richter et al. [19]. Suspended nanowires have been bent with an AFM tip [22–24]. MEMS structures may also be used to perform tension tests [25]. These difficult experiments produce sometimes conflicting results and it is still difficult to decide whether this comes from the material under test or the test itself. The general trend is clear: smaller is stronger. On the other hand defect-free wires show weak size dependence and can be stressed until the theoretical shear strength. A single parameter like the flow stress does not, however, capture the full mechanical behavior of these objects: when the size decreases the number of defects (dislocations, twins, etc.) is reduced and a stochastic behavior is observed