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By invitation only: overview article

Overview on micro- and nanomechanical testing: New insights in interface plasticity and fracture at small length scales

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Micro- and nanomechanical testing has seen a rapid development over the last decade with miniaturized test rigs and MEMS-based devices providing access to the mechanical properties and performance of materials from the micrometer down to the tenths of nanometer length scale. In this overview, we summarize firstly the different testing concepts with excursions into recent imaging and diffraction developments, which turn micro- and nanomechanical testing into "quantitative mechanical microscopy" by resolving the underlying material physics and simultaneously providing mechanical properties. A special focus is laid on the pitfalls of micro-compression testing with its stringent boundary conditions often hampering reliable experiments. Additionally, the challenges of instrumented micro- and nanomechanical testing at elevated temperature are summarized. From the wide variety of research topics employing micro- and nanomechanical testing of materials we focus here on miniaturized samples and test rigs and provide three examples to elucidate the state-of-the-art of the field: *(i)* probing the "strength" of individual grain boundaries in metals, *(ii)* temperature dependent deformation mechanisms in metallic nanolayered and -alloyed structures, and *(iii)* the prospects and challenges of fracture studies employing micro- and nanomechanical testing of brittle and ductile monolithic materials, and materials containing interfaces. Proven concepts and new endeavors are reported for the topics discussed in this overview.

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1. Introduction

Micro- and nanomechanical testing methods have evolved with the invention of nanoindentation [1] and scanning probe microscopy (SPM) techniques [2] permitting to perform mechanical experiments at small length scales on any kind of material. While SPM related techniques quickly entered the field of mechanics of nano-objects (1, 2, and 3-dimensional nano-objects) [3], nanoindentation was applied to surfaces of bulk materials and surface coatings. The invention of micro-compression testing by Uchic et al. [4] using focused ion beam (FIB) milling to structure micron-sized pillars of different dimensions in Ni single crystals which were subsequently deformed by using a nanoindenter equipped with a flat punch boosted the era of experimental micro-/nanomechanical testing. Initially targeting on understanding size-effects of single crystal plasticity as reviewed in the companion overview “Microplasticity” [5] the field has evolved into studying the interplay between plastic deformation and microstructure components such as second phase inclusions [6–9], grain boundaries [10–15], and interfaces [16–21] as well as fracture [22–28].

In parallel to the above-mentioned route, MEMS (micro-electro-mechanical system) technology triggered an alternative path to create miniaturized mechanical testing devices. In this case, lithography techniques initially developed by the semiconductor industry for large scale integration of semiconductor devices is employed to fabricate structures which integrate the material to be tested as well as displacement and load sensors [29] and in some cases even the actuation [30,31]. Both testing approaches will be briefly reviewed in section 2 with an in-depth discussion of micro-compression testing as the most prominent technique. Section 2 ends with recent advances in electron microscopy and synchrotron X-ray diffraction relevant for *in situ* micro-and nano-mechanical testing to unravel the underlying deformation mechanisms.

Material development occurs at various geometrical and microstructural length scales with “nano- and micromaterials” often referring to materials with dimensions spanning from a few atoms up to the micrometer length scale. This includes objects in the form of particles, sheets and thin films, as well as wires representing 3 dimensional (3-d), 2-d and 1-d structures, respectively, fabricated by a wealth of “bottom-up” methods including wet chemical processing, physical and chemical vapor deposition techniques. While it is obvious that micro- and nano-sized materials require mechanical testing methods at the relevant length scale to quantitatively determine their mechanical properties such as elastic modulus, flow stress, and fracture toughness, the same also holds true for bulk materials. Bulk materials underwent significant developments in the last decades to enhance their structural and functional performance often rendering them into “nanomaterials” from a microstructure perspective. This includes bulk nanocrystalline materials, bulk nanostructured composites, and hierarchical multiphase materials like advanced steels with grain sizes and/or chemical modulations below ~100 nm and thus a high fraction of atoms located at internal interfaces. The numerous closely spaced interfaces significantly influence the global mechanical behavior and may open design strategies to combine usually self-excluding property combinations such as high strength and high ductility. Also new materials such as high entropy alloys or chemically complex alloys (both consisting typically of 5 or more principle elements and being single- or multiphase, respectively) have emerged where micro- and nanomechanical testing sheds light on deformation mechanisms such as twinning and dislocation glide and provides a tool to measure critical resolved shear stresses or fracture toughness [32–34]. Furthermore, mechanical failure of any bulk material starts with the local formation and accumulation of defects finally leading to fracture by an advancing crack. As a consequence, any bulk material profits from an in-depth understanding of mechanical phenomena at the nano- and micro-meter length scale.

In this light, the overview will address the materials physics of mechanical phenomena at small length scale and the corresponding challenges encountered in testing and data interpretation for three selected topics: testing the mechanical performance of *(i)* individual grain boundaries in metals (section 3), *(ii)* nanolayered film systems with their abundance of interfaces with a focus on deformation at elevated temperatures and the corresponding instrumental challenges (section 4), and *(iii)* fracture of crystalline materials with and without interfaces (section 5) probed by micro- and nanomechanical testing including the current state-of-the-art in elasto-plastic fracture mechanics at small length scales.

2. Overview on experimental methods

This section summarizes first the miniaturized deformation