New dedicated methods for the improvement of quantitative in-situ TEM nanomechanical testing

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Recently, the development of a new generation of advanced instruments for quantitative in-situ TEM nanomechanical testing has allowed establishing a one-to-one relationship between load-displacement characteristics and stress-induced microstructure evolution in the transmission electron microscope (TEM). In the present work, it will be demonstrated that a step forward in the investigation of the small-scale plasticity mechanisms and size effects can be made by using original sample preparation methods. Recently, we used an original method to revisit the plasticity of olivine single crystals at low temperature. Microscopic tensile samples with pre-defined crystallographic orientation were prepared by focused-ion-beam (FIB) from macroscopic olivine single crystals oriented with X-rays. This allowed preparing samples with ideal conditions for the activation of dislocations and for the imaging of these dislocations. Furthermore, in order to facilitate the nucleation of dislocations in such hard and brittle material, the samples have been prepared on small scratches (from the last polishing stage of the bulk sample) located at the surface of the macroscopic sample. Cyclic deformation was thus applied in the load control mode using the PI-95 TEM picoindenter holder and the Push-to-Pull (PTP) device (Bruker.Inc). Load was increased to a given value, which is maintained constant for several minutes before unloading. During the plateau, dislocation motion is observed and characterized (hence, under a known and constant applied stress). Using this method and dislocation dynamic (DD) simulations, we found that the intrinsic ductility of olivine at low temperature is significantly lower than previously reported values which were obtained under strain-hardened laboratory conditions. More generally, we demonstrated the possibility of characterizing the mechanical properties of specimens which could be available in the form of sub-millimetre sized particles only. More recently, very clean single crystal and bi-crystal Ni samples with desired crystallographic orientations and microstructures have been prepared for quantitative in-situ TEM tensile tests. The preparation included twin jet electro-polishing, FIB as well as an extra annealing step in TEM to remove FIB damages. The results revealed that, this method could significantly improve our understanding of the relationship between the intrinsic deformation mechanisms and the mechanical behaviour of small-sized systems.

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