

Functional Materials

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Electron-beam-hardening of nanoscaled amorphous silica

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Materials are traditionally strengthened by controlled creation of internal defects and boundaries, which inhibit dislocation motion [1]. Such strategies unalterably compromise ductility, the ability of a material to plastically deform without fracture. Due to absence of long-range periodicity in amorphous materials dislocations are not available and therefore plasticity has to rely on other mechanisms that govern the irreversible deformation, such as local plastic rearrangements [2]. It was shown by in situ mechanical testing in a transmission electron microscope (TEM) that nanoscaled amorphous silica exhibits enormous ductility even at or near room temperature [3]. By means of molecular dynamics simulations the authors revealed an interesting mechanism, involving electron-beam-induced generation of structural and bonding defects that facilitate bond-switching events in the silica network, the rotation and reorientation of SiO₄ clusters, finally accommodating viscous flow.

Here we report on an experimental approach for hardening of nanoscaled amorphous silica, achieved by electron-beam irradiation inside of a TEM. The silica spheres investigated here were synthesized by the Stöber-Fink-Bohn (SFB) method [4], being monodisperse, amorphous and without any obvious impurities. The in situ compression in the TEM was carried out using a Hysitron Picoindenter™ (PI-95), equipped with a flat punch. Before every in situ compression experiment, the silica spheres were irradiated with the electron beam (e-beam), using a specific beam current density and afterwards compressed either with or without e-beam irradiation. Displacement-controlled in situ compression under e-beam irradiation (beam on) confirmed severe ductility, without any evidence of shear banding or fracture, as shown in figure 1. These results agree well with the results reported previously by Zheng et al. [3], who used an identical experimental set-up and comparable in situ compression experiments of sol-gel derived silica spheres. Beyond that, we carried out in situ compression experiments, where the silica spheres were compressed without e-beam, after e-beam irradiation with different beam current densities (see figure 2). After specific e-beam irradiation the e-beam was switched off and the compression was carried out at different times (beam-off 30 and 300 s) after switching off the e-beam. In particular, by using such experiments, the aim was to show the impact of specific beam current densities and the e-beam effect on time scale on the deformation behavior of the silica spheres. As can be seen in figure 2 the load-displacement curves for beam-off experiments clearly show that the silica spheres exhibit more elasticity and less plasticity, compared with beam on experiments (see figure 1). This is not surprising, since the e-beam softens the silica spheres and thus enables more plastic flow. More interesting is the comparison of the maximum loads for different beam-off experiments and for low and high beam current densities (see bottom in figure 2). Namely, using a low beam current density during e-beam irradiation, followed by a compression with beam off (A in figure 2) significantly lower maximum loads are observed, compared to the experiments, where the silica spheres are irradiated with a higher beam current density (B in figure 2) and compressed with beam off. Even if the compression was carried out after 300 s beam off (C in figure 2) the maximum loads were in a comparable range as in the case for 30 s beam off. This clearly indicates that the e-beam makes the silica permanently stronger. Moreover, our results suggest that the hardness of nanoscaled amorphous silica spheres can be tuned selectively by using a specific e-beam irradiation, by a selective choice of the beam current density.

The results in the present study may help for tuning and improvement of the mechanical stability of amorphous silica, where functionality and mechanical resistance is needed at the same time.

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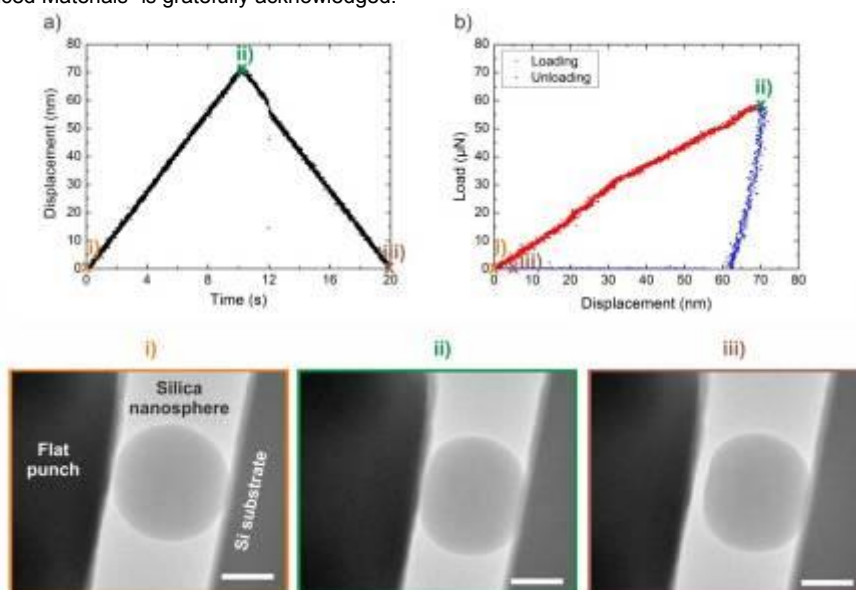


Figure 1. Displacement-controlled in situ compression of silica nanospheres under electron beam irradiation (beam on). In a) the used displacement profile is shown, while b) shows the simultaneously acquired load-displacement data. TEM images i-iii) are extracted from the in situ movie at different stages of the compression experiment, as marked in a) and b), respectively. The scale bars are 200 nm.

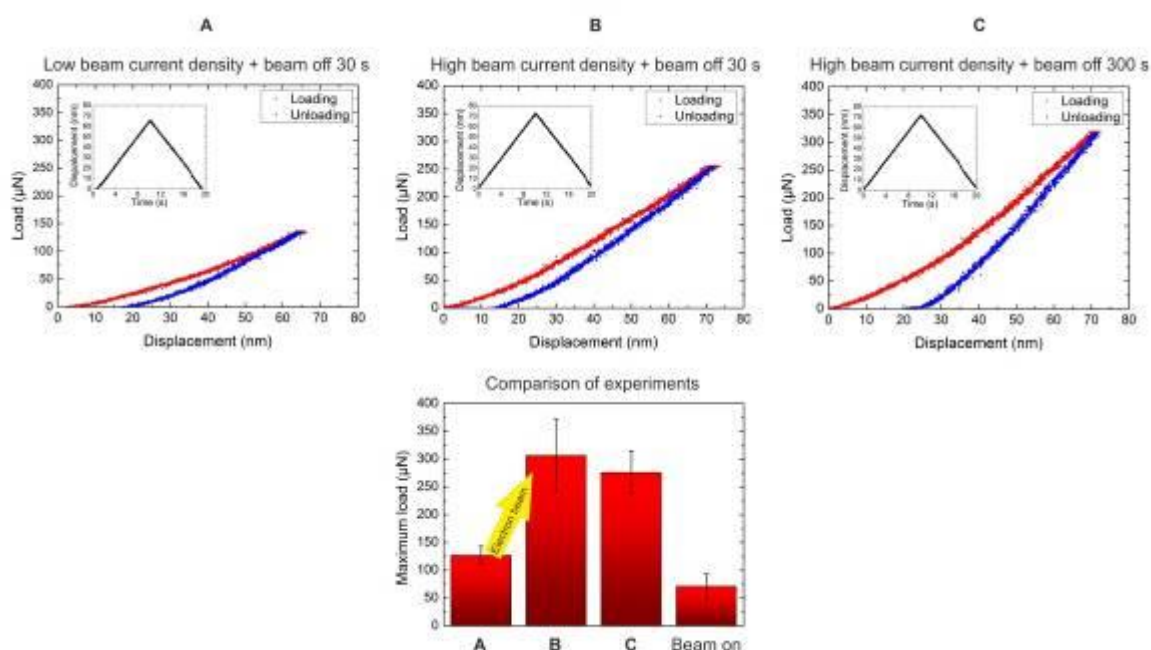


Figure 2. Displacement-controlled in situ compression of silica nanospheres without electron beam irradiation (beam off), after illumination with different beam current densities: A) illumination with low beam current density and compression after beam off 30 s; B) illumination with high beam current density and compression after beam off 30 s; C) illumination with high beam current density and compression with beam off 300 s. The used displacement profiles are shown as insets in A, B and C. The figure at bottom compares the different experiments (A, B and C), while also the beam on experiments are plotted as reference.