2343
doi:10.1017/S1431927615012490 Paper No. 1170 Microsc. Microanal. 21 (Suppl 3), 2015

In-situ AFM and SEM Investigation of Slip Steps Evolving during Nanoindentation

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Since plasticity in metallic materials is usually attributed to the motion of dislocations, the knowledge of the evolution of the dislocation structure during deformation is the key to understanding the mechanical properties on all length scales. Especially for samples in the micrometer regime, the evolution of the slip-step pattern can be directly linked to the active dislocation sources. Depending on the number of operating dislocation sources on the different slip systems (slip planes) a characteristic slip-step pattern forms at the surfaces. The collective motion of dislocations and the formation of complex dislocation structures have been studied with nanoindentation using slip-step analysis. Scanning electron micrographs can give only information on slip-step orientation and slip-step distribution with no quantitative information on their heights. The heights are needed to estimate the number of dislocations that have reached the surface. Therefore, new correlated microscopy techniques are needed to further understand the origins of plasticity.

The advent of integrated atomic force microscopes (AFMs) in stand-alone scanning electron microscopes (SEMs) has opened new possibilities due to quantitative 3D information on complex surface morphologies. This does not only reduce experimental times but also expands the information for both techniques in a comprehensive manner due to the quasi-simultaneous correlation within one setup. In material science, the investigation of slip steps structures and the correlation to the active dislocation sources during nanoindentation of metallic single crystals is necessary to examine size effects related to deformation. The collective density of dislocations and the formation of spatially complex dislocation structures have been studied with GETec's combined AFM and SEM (AFSEM™). This system uses a highly flexible, in-situ tip scanning system with a wide range of scan-head modules. While the SEM acts as the high resolution navigation tool and can provide structural information via electron backscatter diffraction analyses, the integrated AFM solution provides laterally resolved, quantitative step height information with nm and even sub-nm resolution in XY and Z, respectively. This is an enormous advantage as it is very complicated to distinguish between real surface features on the lower nanoscale and coverage layers via SEM due to the electron beam penetration depth. By that, both techniques are complemented by each other in a straightforward manner providing comprehensive insights practically impossible or extremely complicated via individual technique or two separate instruments, respectively.

Nanoindentation is a common measurement technique used to measure mechanical properties and plastic deformation events. However, difficulties arise when investigating size effects due to the size of the resulting indent imprints. With the combined imaging techniques nano- and micro-sized indents were located with the SEM while the AFM was employed to resolve the actual depth and resulting deformation with nanometer resolution (Figure 1). The AFM-SEM technique will be demonstrated through the evolution of slip steps emanating around nanoindentation imprints in a single crystal brass (Figure 2). It will be shown how the load-displacement curve corresponds to the resulting deformation

[1-2], what slip planes are activated [3], and the number of dislocations will be estimated using the heights of slip steps and pop-in events [4].

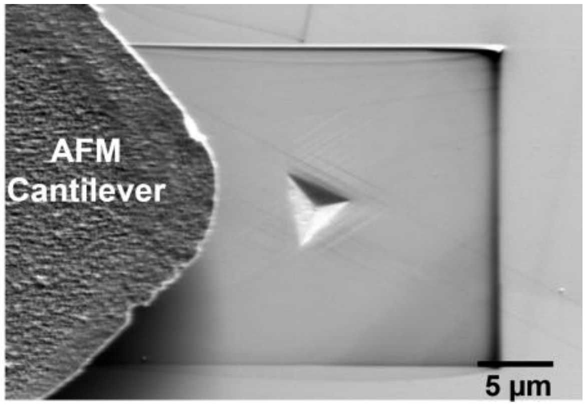

Figure 1. SEM micrograph demonstrating how the AFM cantilever approaching a nanoindent.

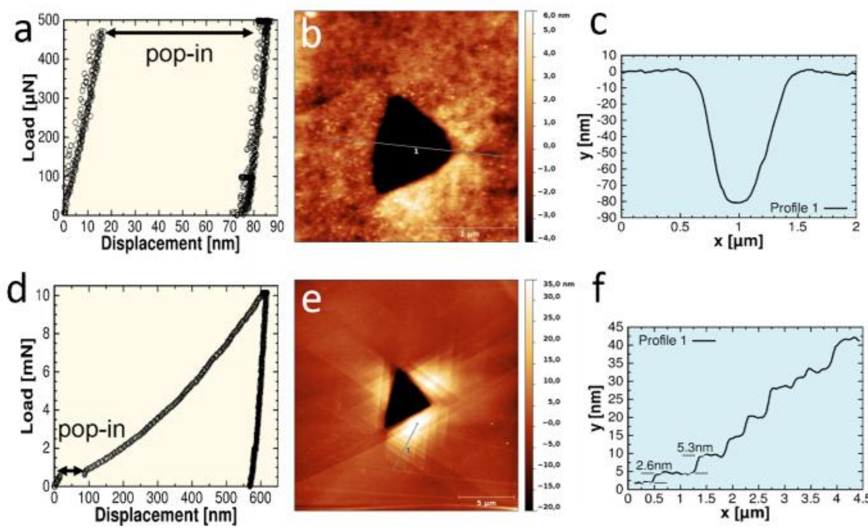

Figure 2. (a) Load-displacement curve, (b) AFM height image, (c) and height profile of a 500 µN indent. (d) Load-displacement curve, (e) AFM height image, and (f) height profile of slip steps that formed with a 10 mN indent.

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