APPLICATION NOTE

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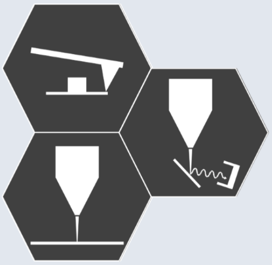
Correlative EBSD, SEM and AFM analysis of ZrO2 ceramics

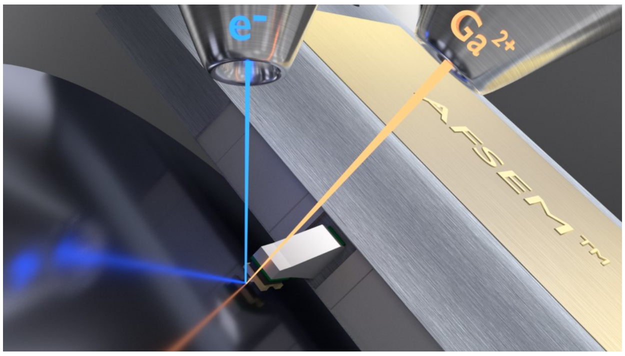


In situ analysis of martensitic transition in ZrO_2 ceramics with AFSEM®

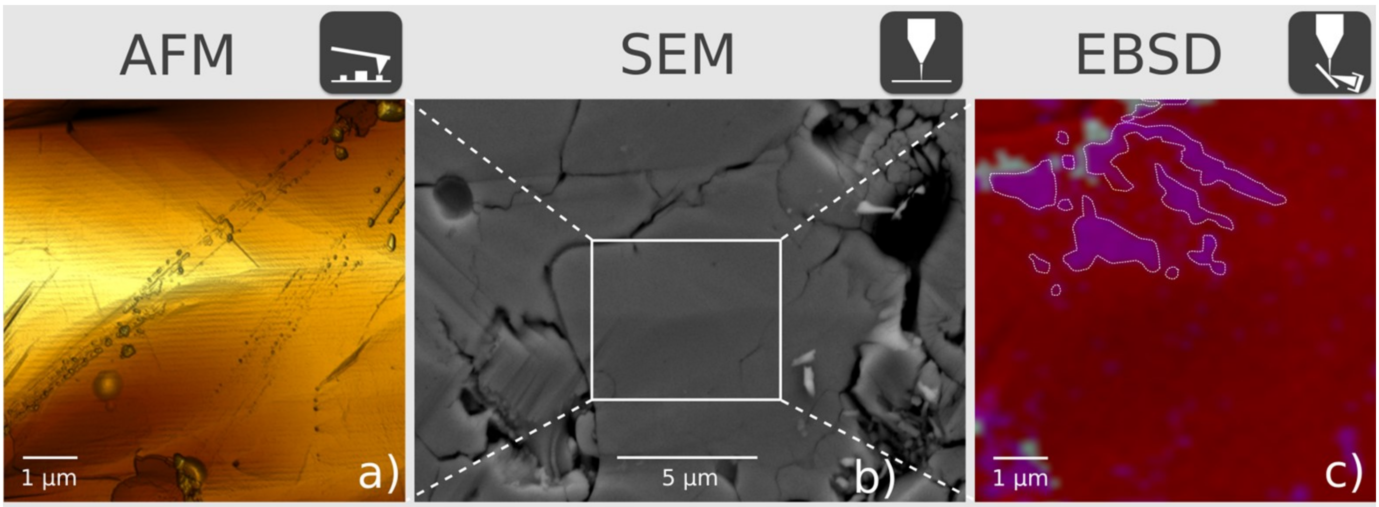
Zirconia (ZrO_2)-based ceramics are strong, hard and inert and have low thermal conductivity and good biocompatibility. Due to these properties they have found wide applicability in the fields of optical communication, thermal barrier coatings, automobile industry and femoral implants. However, phase transformation processes in zirconia-based ceramics, which can either strengthen or weaken the material are barely understood due to the lack of correlative crystallographic phase and morphology information.

A unique atomic force microscope (AFM) -the AFSEM®- designed for seamless integration into scanning electron microscopes (SEM) or dual beam systems is used for the correlative in-situ AFM/SEM/EBSD analysis at the exact same sample position without the need of air exposure or sample transfer. Its compact design and the use of self-sensing cantilevers with electrical readout allows for simultaneous operation of SEM, FIB and AFM inside the vacuum chamber.

For the first time, the martensitic transition in zirconia coatings was investigated by combination of in-situ electron backscatter diffraction and in-situ atomic force microscopy. The results were recently published by the group of Prof. Zeng [1].

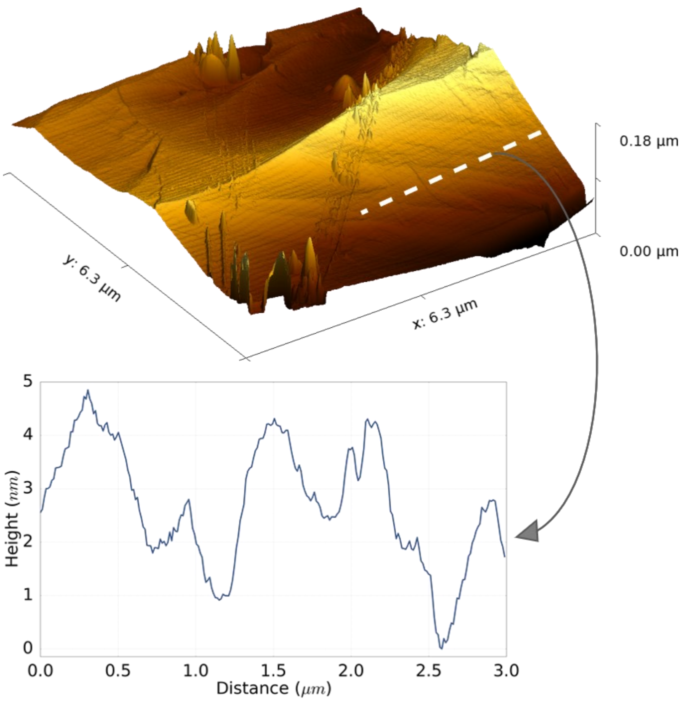


Schematics of correlative analysis in a dual beam system using SEM, FIB, and AFSEM® in an interactive experiment .



AFSEM™ enables correlative EBSD and AFM topography analysis. AFM image (a), correlative SEM image (b) and EBSD crystallographic analysis of ZrO_2 sample (c). Sample courtesy : Prof. Y. Zeng (SICCAS, China)

Correlative EBSD, SEM and AFM analysis of ZrO2 ceramics



3D topography showing phase transformation-induced changes of topography up to 5 nm as shown by the line cross section in transformed areas.

The direction of residual stress was revealed by the periodic corrugation patterns of AFSEM® images, matched precisely with the 10° deflection of the basal texture. This finding was further confirmed by an in-situ reverse transformation from the monoclinic phase to tetragonal phase and the formation of vertical microcracks after stress release during heat treatment.

In summary, while EBSD allows for locally identifying areas where the phase transformation has occurred, in-situ AFSEM® images can now be utilized to analyze phase-transformation-induced topographic changes with sub-nm resolution, enabling correlative EBSD and AFM topography analysis.

References:

[1] Wang, Yongzhe et al. “Investigation on the habit plane of martensitic transformation in zirconia coatings.” Journal of the Australian Ceramic Society 56 (2020): 257 - 264.