

Novel In-situ TKD Nano-tensile Testing: Insights into Nanoscale Crystal Plasticity and Grain Boundary Mechanics

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Background

Advancements in nanoscale characterization techniques are crucial for understanding the complex deformation mechanisms of metal alloys, coatings and nanolaminates. Traditional post-mortem Transmission Kikuchi Diffraction (TKD) and Transmission Electron Microscopy (TEM) analyses, although powerful, often fail to capture the complete sequence and nature of deformation mechanisms as they occur [1]. This limitation is due to their inability to observe the initiation, progression, and interaction of deformation features such as dislocations or twins, leading to potential misinterpretations involved in material deformation.

In-situ TEM and in-situ EBSD nano- and micromechanical testing are both well-established techniques that offer detailed characterization of evolving crystal plasticity [2-3]. However, in-situ TEM is technically challenging and is limited in its scale. In contrast, In-situ EBSD is more feasible and allows larger field of views, yet its spatial resolution is typically limited to ~50 nm, which can miss finer structural details critical in nanoscale materials. In-situ TKD, on the other hand, would offer a significantly enhanced spatial resolution down to ~5 nm [4], while still performed inside a relatively accessible SEM.

Given these challenges and opportunities, this study introduces in-situ TKD integrated into nano-tensile testing as a novel methodology designed to provide comprehensive insights into the evolution of twinning, dislocation plasticity, and grain boundary mechanics under applied loading. This method will bridge the gap in understanding the intricate behaviors of complex nanolaminate metal/ceramic structures, while also providing an ideal platform to study phase transformation and twinning in advanced alloys.

Methods

A specialized experimental methodology was devised, combining (i) focused ion beam (FIB) processing for specimen preparation, (ii) a push-to-pull device for in-situ tension application, and (iii) an SEM configured for in-situ TKD with an Alemnis indenter. As proof of concept, we investigate deformations of advanced Cu_{1-x}Al_x (X=0-12 at.%) multi-layers with interlayer interfaces of 2-5 nm amorphous Al₂O₃ for improved microstructure control and strength. These were synthesized in a novel deposition chamber from Swiss Cluster AG (<https://swisscluster.com/>), combining physical vapor and atomic layer depositions (PVD and ALD) without breaking the vacuum.

Specimens were prepared through site-specific FIB lift-out, subsequent fixation to the push-to-pull device using Pt deposition [5], a dedicated procedure for thinning to ~150 nm, and creation of a precise gauge section of several μm². We designed and employed a custom configuration of an Alemnis nanoindenter frame inside a Tescan SEM, configured to fit with an Oxford Instruments Symmetry 2 EBSD detector for in-situ TKD and simultaneous STEM detection. Specimens are loaded with the push-to-pull device under continuous in-situ STEM observation, with intermittent loading pauses for detailed TKD mapping.

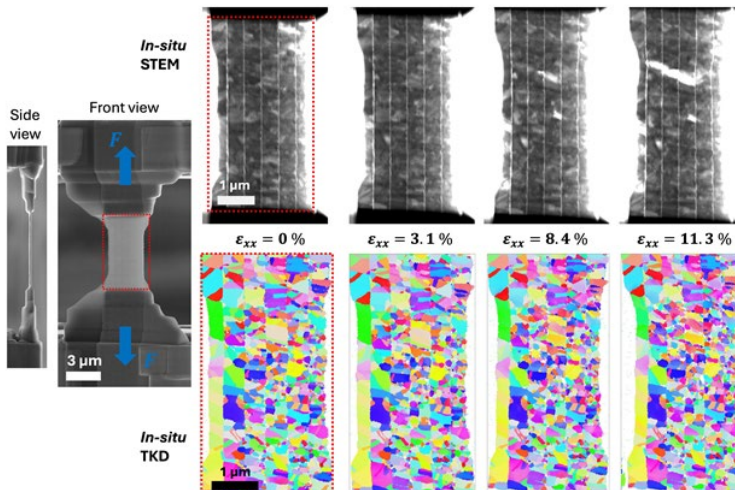
Results

In-situ STEM and TKD nano-tensile testing provides detailed and rich insights into the microstructural behavior of nanolaminate CuAl/Al₂O₃ films. We observe the development of twinning, dislocation plasticity and interactions at grain boundaries from the undeformed state all the way to the formation of a localized shear band. Thereby, we demonstrate the capability of this methodology to analyze complex mechanical interactions at the nanoscale on a specimen of several micrometers in size.

Conclusions

This study establishes in-situ TKD nano-tensile testing as a valuable approach for analyzing nano- and micromechanical deformation mechanisms of complex metallic specimens. The integration of TKD with nano-tensile testing offers a new perspective for exploring deformation mechanisms, providing insights essential for the design and improvement of nanolaminated metallic materials, as well as advanced alloys that show phase transformation and twinning. The findings emphasize the utility of this method in advancing materials science and engineering.

Graphic:



Keywords:

TKD, STEM, twinning, micromechanics, nanolaminates

Reference:

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