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Extending 4D-STEM based strain mapping to polycrystalline materials

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Background incl. aims
Four-dimensional scanning transmission electron microscopy (4D-STEM)-based techniques for elastic strain mapping have advanced significantly, offering a robust tool to investigate deformation mechanisms in materials at the nanoscale. In single crystalline materials, measurements of the reciprocal lattice vectors allow to accurately determine the full in-plane elastic strain tensor [1]. This technique exhibits high sensitivity, enabling the detection of stress fields associated with individual dislocations. In amorphous materials, like metallic glasses, 4D-STEM based strain mapping techniques have also been successfully utilized to determine nanoscale strain states in materials [2]. In contrast, for polycrystalline microstructures, lying between the two extreme cases of single crystals and amorphous materials, a technique for local nanoscale strain mapping is lacking. Although global strains can be inferred from the ellipticity of diffraction rings in selected area diffraction (SAD) patterns, this method provides only average values across multiple grains, lacking insights into the stress state within individual grains or at grain or phase boundaries. This study aims to close this gap and develop a new routine to map the nanoscale strain in polycrystalline materials, facilitating the measurement of strain fields and grain rotations during in-situ deformation of polycrystalline materials with a resolution of 2 nm.

Methods
To demonstrate the technique a 50 nm thick nanocrystalline gold (Au) thin film is used. The sample was produced by DC magnetron sputtering and subsequently underwent heat treatment at 360 °C to reduce the dislocation density present after deposition. The thin film was mounted on a push-to-pull MEMS device and in-situ tensile testing was conducted using a Hysitron PI 95 nanoindenter holder capable of recording the load-displacement data during deformation. The tensile test was intermittently halted during deformation to acquire full 4DSTEM datasets. Precession electron diffraction was employed to enhance the quality of diffraction patterns. By precessing the electron beam the experiment is shifted towards more kinematical diffraction conditions vastly improving the quality of the diffraction patterns. On the one hand, inner intensity distributions are reduced, making peak detection more precise, and on the other hand, more diffraction disks are revealed thus improving on the crystal orientation determination. The utilization of a direct electron detector and an in-column energy filter further refines the dataset quality.

Results
The resulting strain and orientation maps, obtained during in-situ deformation, affirm the feasibility of this study's objectives. Only small changes in crystal orientation were observed, consistent with the low plasticity exhibited by the nanocrystalline thin film. The limited thickness of the Au film, consisting of a single layer of grains, might also lead to less pronounced grain rotation. Nonetheless, orientation changes within individual grains resulting in subgrain structures could be detected and quantified. At the same time, the precise determination of elastic strain states in polycrystalline materials during in-situ deformation was demonstrated. Under loading, a discernible shift towards higher tensile strains was observed, with a histogram analysis revealing a gradual increase in the tensile strain spread with increasing applied load. This clearly indicates, that even in the elastic regime dislocations are generated. Following fracture, the average strain go back to zero, however an even larger spread is measured. This shows that the increase in dislocation

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density and thus the broader strain distribution is not due to a measurement artifact but an actual mechanism in the material that can be quantified.

Conclusions

Atomic-level elastic strains in a nanocrystalline thin film were determined by combining concepts from single crystalline strain mapping and automated crystal orientation mapping techniques. This paves the way for future in-situ deformation experiments on industrially relevant polycrystalline structural materials, thereby enhancing our understanding of their deformation mechanisms.

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In-situ, strain mapping, 4D-STEM, ACOM-TEM