3D Mapping of Dislocation-Crack Tip Interactions in SrTiO by Dark-Field X-ray Microscopy

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Crack formation and propagation remain major limitations for the reliability of structural and functional ceramics. Dislocation-based toughening, highly effective in metals, could in principle compensate for the intrinsically poor fracture toughness of ceramics, yet its role remains poorly understood because conventional techniques such as TEM and chemical etching cannot access the three-dimensional defect structures around cracks in bulk material.

Dark-Field X-ray Microscopy (DFXM) [1-3] addresses this challenge through non-destructive 3D imaging of dislocations, strain fields and orientation gradients in bulk single crystals, with spatial resolution down to ~100 nm, angular resolution down to ~10-3 ° and strain sensitivity of ~10-5. Here, we apply DFXM at the ID03 beamline of the ESRF to investigate dislocation arrangements around crack-tips in mm-sized single-crystal SrTiO as a model perovskite oxide. Recent advances in room-temperature dislocation engineering enable tailoring of dislocation density across five orders of magnitude (~1010 to ~1015 m-2) in this oxide [4], yet the resulting increase in fracture toughness remains marginal (~20%) [5], contrary to analytical models that predict crack-tip shielding by dislocations [6,7,8].

Specimens with (001) surface orientation were prepared by cyclic Brinell scratching [4], producing dislocation-rich tracks (~1012 m-2) embedded in the pristine material (~1010 m-2), with cracks pre-induced on {110} planes that traverse both regions. DFXM resolved individual dislocations and their associated strain fields around the crack-tip in 3D from layer-by-layer rocking and mosaicity scans, allowing for direct comparison of crack-tip behaviour with/without pre-engineered dislocations.

The result provides the first 3D experimental view of dislocation-crack tip interactions in a bulk perovskite oxide and clarifies why pre-engineered dislocations confer only limited crack shielding in SrTiO3. Beyond this material, the DFXM workflow demonstrated here offers a transferable framework for the quantitative mesoscale characterisation of defect-crack interactions in other semi-brittle crystals [9] in a 3D manner.

References

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