Non-Destructive Evaluation of 3D Microstructure Evolution during Sintering of Strontium Titanate

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Following the evolution of a three-dimensional (3D) microstructure during processing is a long sought desire of materials science. Based on synchrotron radiation several non-destructive techniques which, in principle, allow such time-dependent microstructure characterization have become available in recent years. Here we apply diffraction contrast tomography (DCT) [1] to the investigation of microstructure evolution during sintering of strontium titanate ceramics.

Strontium titanate (SrTiO3) is a well-established model system for perovskite ceramics due to its stable cubic structure. Nevertheless, the growth kinetics of strontium titanate shows unusual deviations from the expected Arrhenius behavior [2]. To study correlations between grain boundary properties and microstructure evolution, and to allow for modelling of this behavior, truly 3D microstructural information needs to be acquired at various stages during grain growth from pure stoichiometric SrTiO3 [3]. Specimens prepared by the mixed oxide route were sintered at 1600°C in an oxygen atmosphere yielding a material with an average grain radius of 14.1±1.5 µm [2,4]. Cylindrical samples with a diameter of approximately 300 µm were prepared for DCT analysis at ID11 of the European Synchrotron Radiation Facility (ESRF) in Grenoble, France. The microstructure reconstruction is described in [5]. Between two scans, the sample was annealed for 1h at 1600°C in air. DCT reconstructions of two successive microstructures are shown in Fig.1 which also displays the shrinking of the porosity from 2.6 vol% in the initial stage and 1.2 vol% in the post annealing stage.

The final DCT microstructure has been reinvestigated by means of mechanical serial sectioning and electron backscatter diffraction (EBSD) characterization as described in [6]. Corresponding two dimensional grain maps from both characterization methods are aligned by adjustment of the small intragranular pores. The average grain size for these sections was measured by the linear intersect method and found to be 30.1±0.4 µm (DCT) and 29.7±0.4 µm (EBSD). Overall agreement of morphology and crystallographic orientation is extremely good. However, the accuracy of the precise location of the grain boundaries was only of the order of ±1.5 µm. Nevertheless, the concept of DCT measurements is applicable to studying the sintering behavior of perovskite ceramics.

DCT investigations of microstrcutures during sintering result in 3D grain structures with reasonable resolution at the grain boundaries and high-accuracy orientation detection. The ability of acquiring multiple such structures from the same specimen will for the first time allow precise 3D validation of sinter and grain growth simulation against experiments. In the future, we aim at inverse modelling of the grain growth process to independently determine orientation specific grain boundary energy and mobility.

References

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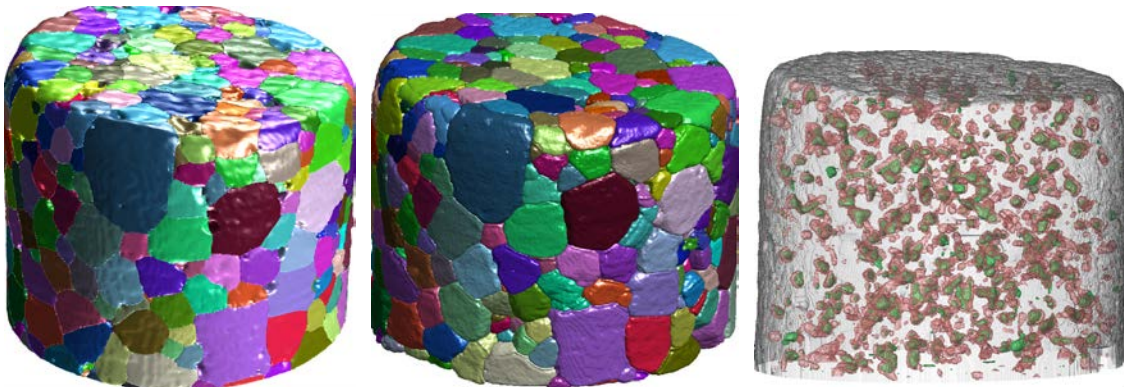

FIG. 1. 3D microstructure reconstruction (left) before and (middle) after annealing. (right) Collective pore ensembles in the microstructure reconstruction before (red) and after (green) annealing.

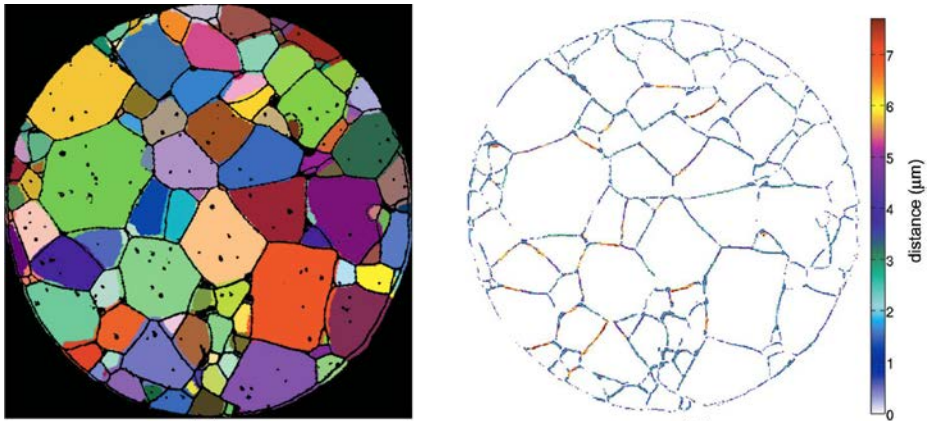

FIG. 2. (Left) Cross section of the DCT reconstruction (color) superimposed with the corresponding EBSD data (wireframe). (Right) Same cross section colored by Euclidean distance to the EBSD data.