In situ high-temperature 3D-reciprocal space mapping as a new way to study solid state phase transition

R. Guinebretière¹, D.P. Fowan¹, E. Thune¹, R.R.P. Purushottam Raj Purohit², S. Arnaud³, G. Chahine⁴, N. Blanc⁵, O. Castelnau⁵

¹Université de Limoges, IRCER, UMR CNRS 7315, 12 rue Atlantis, Limoges 87068, France
²Université Grenoble Alpes, CEA, IRIG, MEM, CNRS, 17 avenue des Martyrs, Grenoble, 38000, France
³Université Grenoble Alpes, CNRS, Institut Néel UPR CNRS 2940, 38000 Grenoble, France.
⁴Université Grenoble Alpes, SIMaP, Grenoble INP, CNRS, 38000 Grenoble, France
⁵Laboratoire PIMM, UMR CNRS 8006, ENSAM, CNAM, 151 Bd de l'Hôpital, 75013 Paris, France
rene.guinebretiere@unilim.fr

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Solid-state phase transitions (SPT) are fundamental processes that are driving a large part of the effective physical properties of single crystals as well as polycrystalline materials. In addition to the structural evolutions that can be described by space group transitions, SPTs are often associated with transformations that occur at the mesoscale and generate structural defects such as dislocations, twinning, stacking faults, strain fields, etc. Such defects can be detected and quantitatively studied in the reciprocal space through high-resolution x-ray diffraction experiments. Originally developed to study defects in epitaxial layers, the Reciprocal Space Mapping (RSM) method has been extended to cover everything from imperfect single crystals to complex polycrystals. Over the past decade, the widespread use of 2D solid-state detectors at synchrotron radiation sources has promoted the development of 3D-RSM on timescale that enables to follow the sample evolutions as a function of external stimuli. In the case of polycrystals, it has been shown that this approach allows to select the crystals of interest in reciprocal space from all the crystals constituting the probed volume. We have recently shown that this method can be used even at very high temperature (over 1000 °C) [1, 2]. We will show during the conference that high temperature 3D-RSM can be extended to in situ observation of solid-state phase transition allowing to evidence the formation of crystallographic variants and twins.

The potential of this approach will be illustrated by the in situ study of two successive phase transitions in pure zirconia dense polycrystal. Under atmospheric pressure, pure zirconia solidifies into a cubic crystal structure (c) (space group Fm3m). Upon cooling it transforms first to tetragonal (t) (space group P4(2)/nmc) and then to monoclinic (m) (space group P2(1)/c). We will show that the method allows following the temperature induced splitting of an initially unique Reciprocal Lattice Node (RLN) into 24 RLNs generated by the loss of symmetry axis through two successive phase transitions. Moreover, since the SPT occurs under huge stresses [3], the RLNs are embedded in a large, 3-dimensional, diffuse scattering signal that is very clearly evidenced (see Fig. 2). The crystallographic interpretation of RLN splitting and the diffuse scattering signal associated with the phase transitions in pure zirconia polycrystal will be discussed during the talk according to [2] and another article that we published in October 2022 [4].

Figures 1. in situ high-temperature observation of the splitting of the tetragonal 111 RLN during the tetragonal to monoclinic phase transition in pure zirconia.