ESRF	**Experiment title:** Transformation-induced plasticity in zirconia-based ceramics: an *in situ* microLaue experiment during compression of micropillars	**Experiment number**: MA-5162
Beamline: BM32	**Date of experiment**: from: 29/06/2022 to: 01/07/2022	**Date of report**: 03/02/2023
Shifts: 6	**Local contact(s)**: MICHA Jean-Sebastien, PURUSHOTTAM RAJ PUROHIT Ravi Raj Purohit	*Received at ESRF:*

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Report:

This experiment aimed to study the strain, crystal orientation and phase, and dislocation nature in ceria stabilized tetragonal zirconia (CSTZ) micropillars during microcompression using in-situ Laue microdiffraction. This experiment was performed at the BM32 beamline where the incident polychromatic X-ray beam was focused down to a size of 300 nm x 300 nm using a pair of Kirckpatrick-Baez mirrors. The CSZT micropillars with 12 mol% Ce were obtained according to the following methodology, illustrated in Fig. 1(a): First, 12 mol% ceria-doped zirconia powder was pressed uniaxially, followed by cold isostatic pressing to form green pellets for sintering. Then, the pellets were sintered at 1600 ºC for 10 hours dwell time. These temperature and sintering time were selected to facilitate the growth of large grains with sizes of up to 8 µm, needed for the subsequent micro-machining of single-crystalline micropillars. The sintered samples were cut into triangular shapes with a diamond wire cutting machine, followed by surface preparation using a broad Argon ion beam (Ilion II, Gatan). The low-energy milling allows for reduced surface damage, which prevents premature phase transformation. Sequentially, electron back-scattered diffraction (EBSD, Symmetry, Oxford Instruments) was performed for selecting grains with specific crystalline orientations before the pillar's milling. Finally, 6 micropillars were milled out of the bulk material using a focused ion beam microscope (FIB, Zeiss NVision 40), with diameters around 0.5-0.9 µm, and height of 2-2.5 µm. The pillars, named C1-6, are presented in Fig. 1(b).

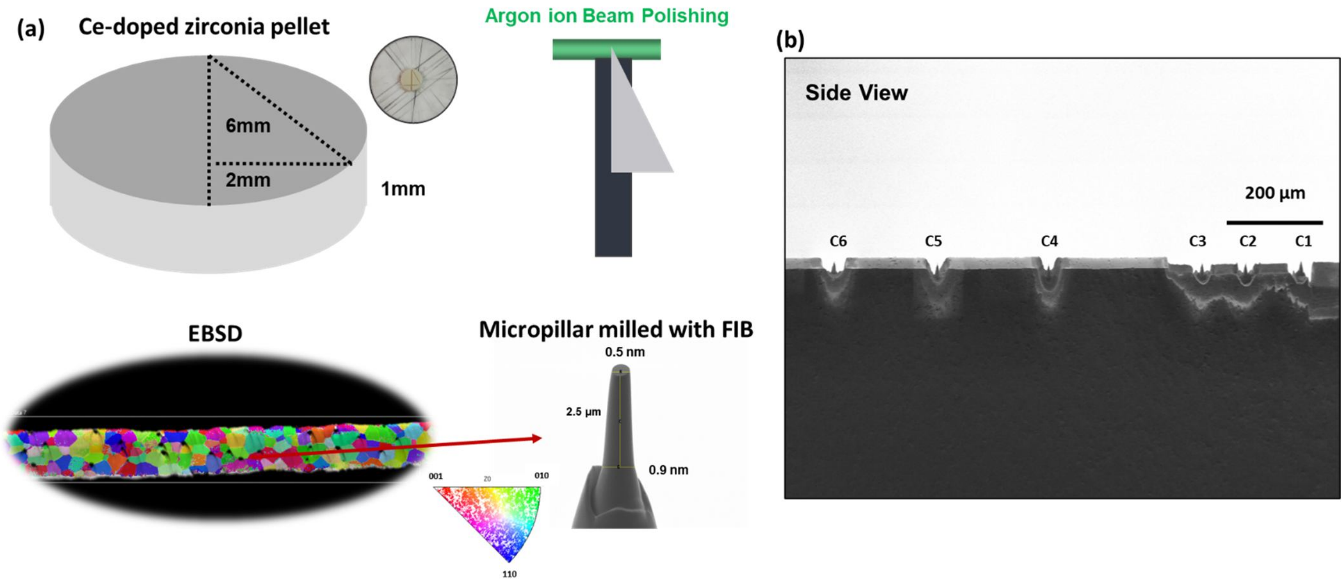

Figure 1 – (a) Processing steps for micropillars processing, starting from bulk material, followed by broad beam polishing and EBSD grain selection previous to FIB milling. (b) Side view of micropillars milled by focused ion beam.

In this experiment, we aimed to mill single-crystal pillars, where three main orientations with low indexes were selected for the *in situ* compression tests. These orientations of the MPs are according to Tab. 1:

Table 1 – Miller indices orientations indexed by electron backscattered diffraction, of micropillars composed of 12 mol.% Ce-doped zirconia.

Sample	Miller indices [uvw]
C1	[110]
C2	[100]
C3	[001]
C4	[100]
C5	[001]
C6	[110]

E*x situ* Laue microdiffraction maps were acquired for each pillar with a stp size of 0.3 µm corresponding to the X-ray probe size. The Laue microdiffraction patterns were indexed using LaueTools software, whch eventually allowed to confirm the crystalline orientation that was previously obtained by EBSD.

For the *in situ* microcompression tests, the nanoindenter FT-NMT04 (FemtoTools) equipped with a diamond flat punch was installed on the goniometer of the BM32 beamline. The micropillars and the flat punch were located and aligned with respect to each other using the optical microscope installed at BM32 (Fig. 2).

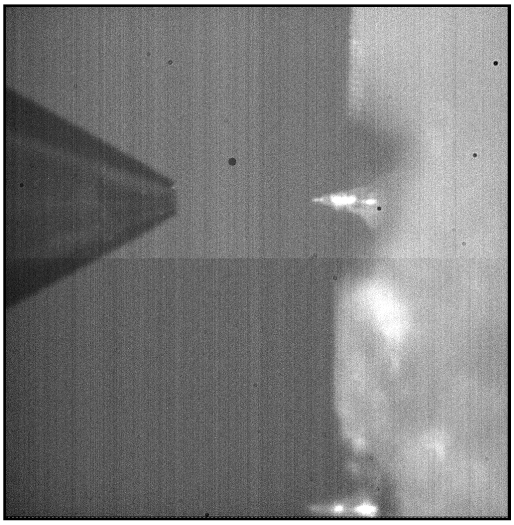

Figure 2 – Optical microscope image of diamond flat punch (left) approaching a micropillar (right)

During compression in displacement-controlled mode with a displacement rate of 30 nm/s, Laue microdiffraction patterns were recorded using a sCMOS camera installed at 90° with respect to the incident polychromatic X-ray beam. Cyclic loading-unloading curves with increasing maximum load were applied to each micropillar. 2D Laue microdiffraction maps were recorded from the pristine micropillars, as well as after each loading-unloading cycle. The Laue diffraction patterns were indexed using the LaueTools software [1]. Not all 6 pillars could be tested, as we could not locate them by mapping the yield of the Zr Ka fluorescence. Hence, only pillars C1 and C2 were mechanically evaluated.

All loading-unloading curves are displayed in Fig. 3 for both pillars with their calculated engineering stresses and strains.

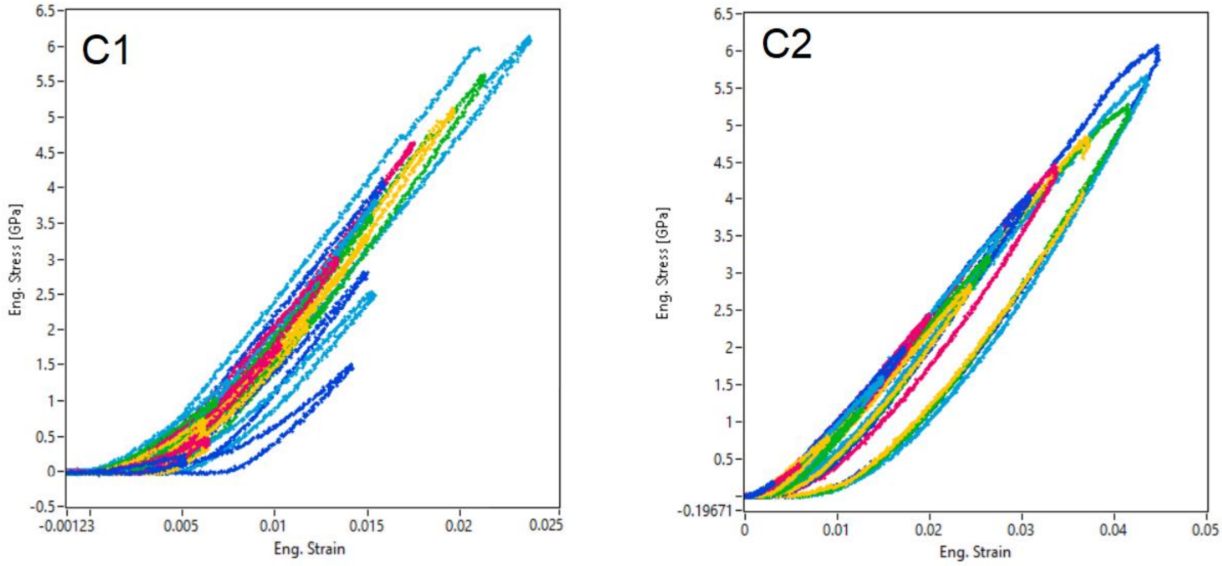

Figure 3 – Calculated engineering stresses and strains for micropillars C1 and C2. Cyclic loading and unloading compression tests were performed until a load drop was visible on the curve.

After analyzing the microdiffraction maps, no phase change from tetragonal to monoclinic could be observed. Fig. 4 displays the Laue diffraction patterns of the C1 and C2 micropillars before and after compression illustrating that they both exhibit a tetragonal crystalline phase even after compression. Reasons for no transformation could be related to misalignments of the setup, or crystal orientations that do not favor it. In this case, higher loads might be needed. For pillar C2, signs of cracks and bending were observed by *post-mortem* scanning elecron microscopy (see Fig. 6 in Annex).

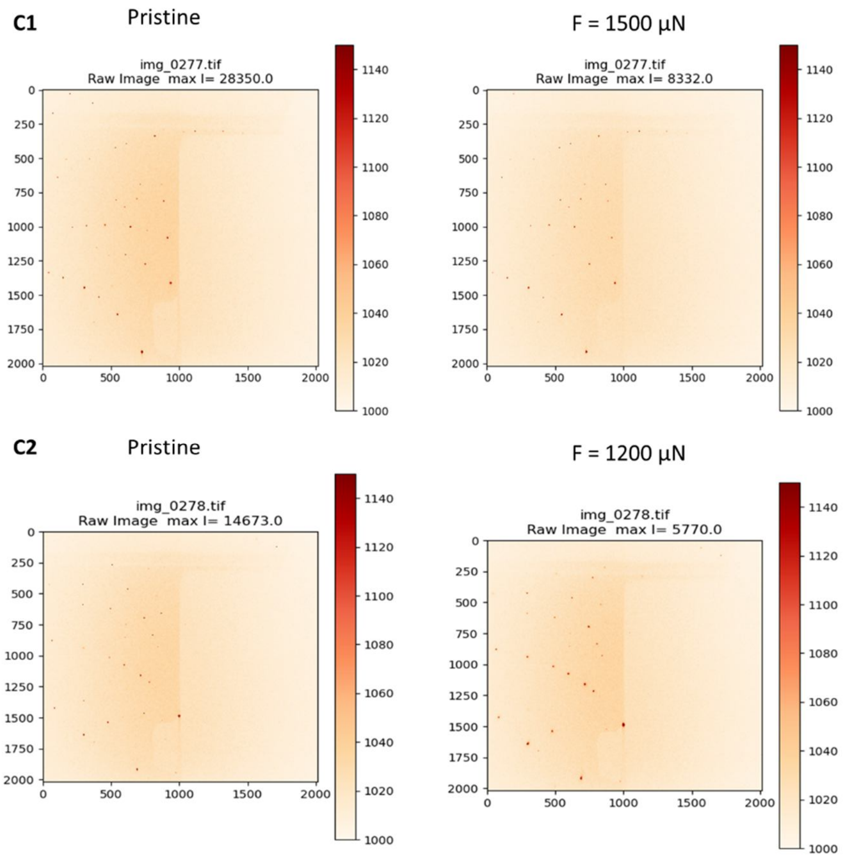

Figure 4 – Laue microdiffraction maps of the pristine (left) and compressed (right) state of pillars C1 and C2.

Future experiments will focus on CSZT micropillars with different crystalline orientation that favor more the t-m phase transformation in contrast to brittle cracking.

References:

[1] Micha, J.-S. & Robach, O., (2010). LaueTools laue x-ray microdiffraction analysis software.

Annex:

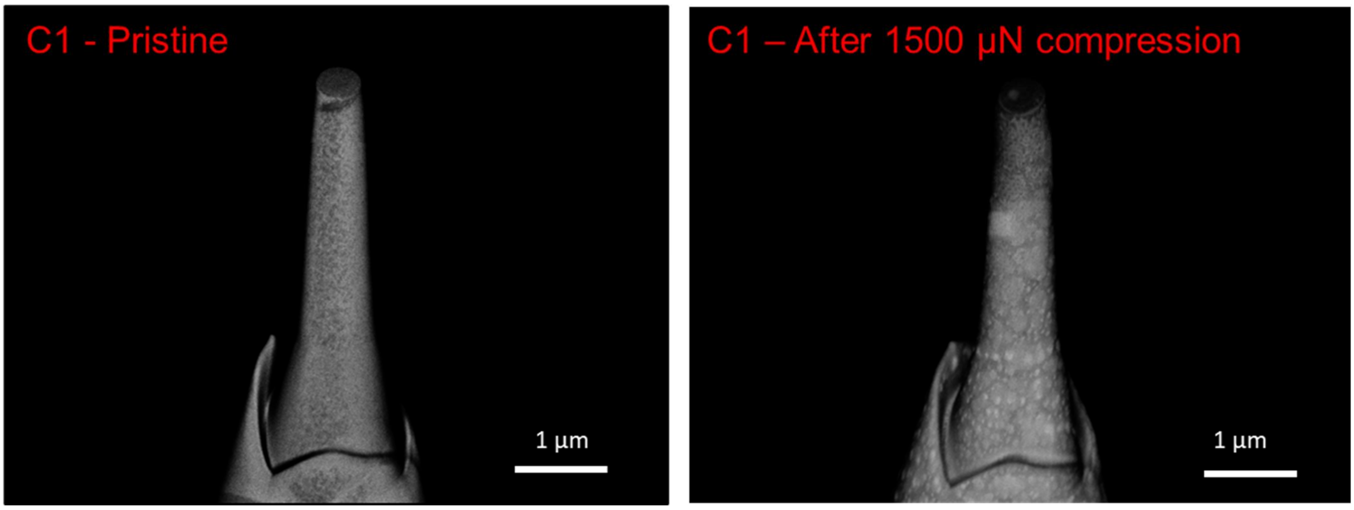

Figure 5 – Scanning electron microscope image of the pristine (left) and compressed (right) state of pillar C1.

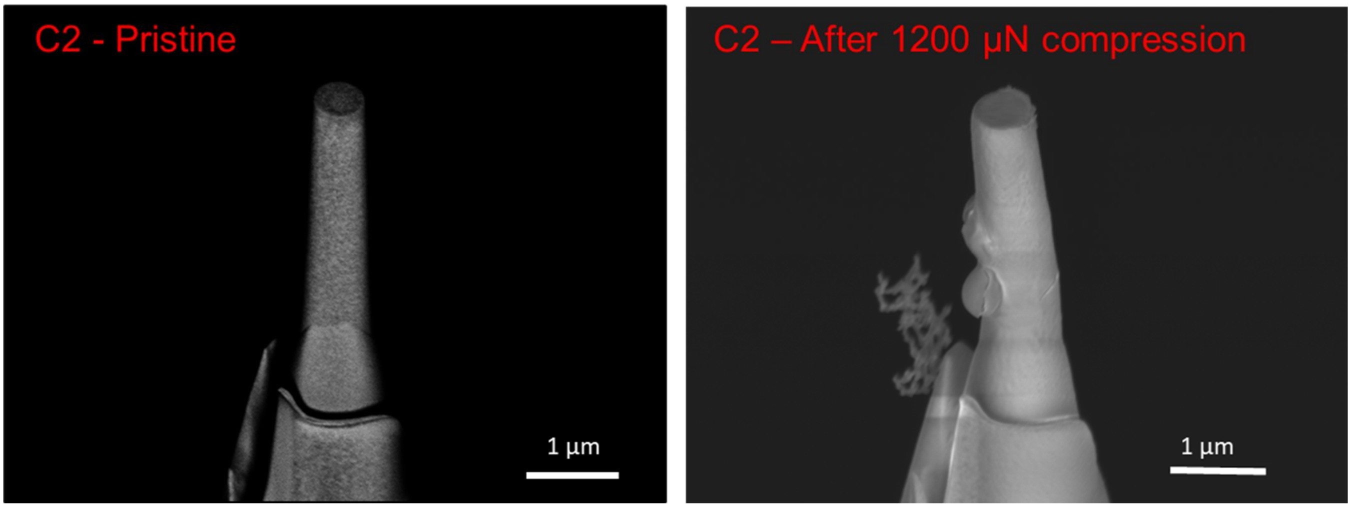

Figure 6 – Scanning electron microscope images of the pristine (left) and compressed (right) state of pillar C2, with signs of cracking and bending.