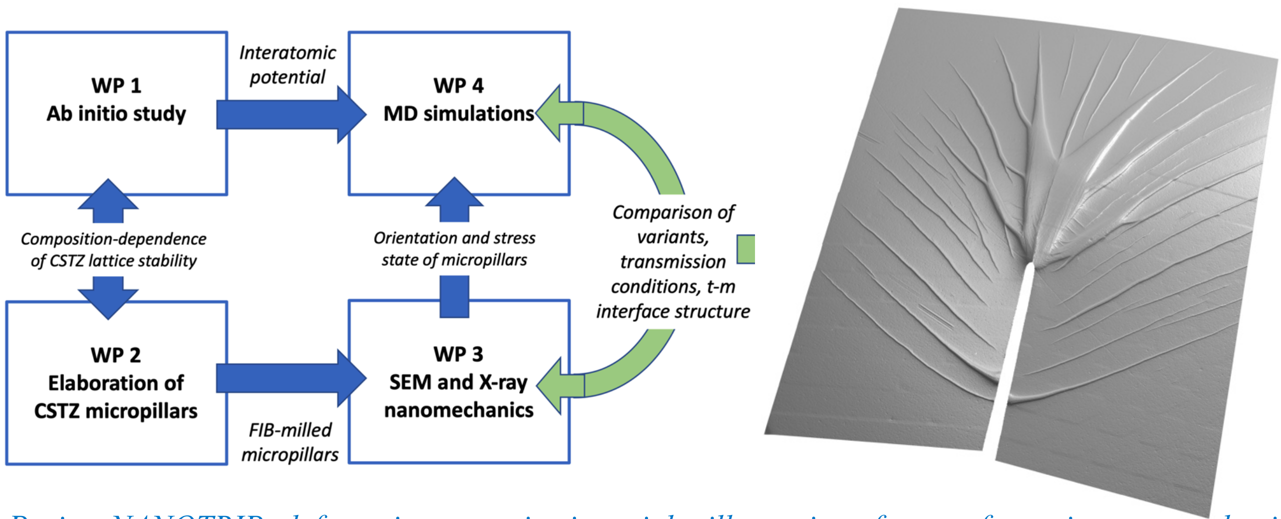
PhD thesis subject:

Transformation induced plasticity in zirconia: characterization at small scales by X-ray diffraction and in-situ electron microscopy during mechanical loading

Thesis proposed by MATEIS (INSA-Lyon) and IM2NP (AMU), in the framework of the project ANR NANOTRIP

General context:

The vast majority of ceramics exhibit, as a first very good approximation, an elastic-brittle behavior at room temperature. They therefore break before reaching their elastic limit. This is linked to the very low mobility of dislocations at room temperature, which compromises any ductility by movement of dislocations as is observed in metals. However, some zirconia-based ceramics can exhibit real plasticity at room temperature, thanks to a stress-induced martensitic phase transformation mechanism which is similar in several ways to the TRIP ('TRansformation Induced Plasticity') effect of certain steels and/or the 'shape memory' effects of certain metal alloys. This behavior is particularly remarkable in zirconia doped with cerium oxide, which exhibit plastic deformations of the order of one percent before rupture, high toughness (for ceramics), and lower sensitivity to the presence of defects than classic ceramics. The NANOTRIP project proposes a study of the physical processes that control this TRIP effect at the nanoscale in these zirconia, with the aim of designing ceramics with improved ductility and toughness, as well as a "shape memory" effect. The methodology proposed in the NANOTRIP project bases both on modeling approaches (ab initio, molecular dynamics) and on *in-situ* experiments, with the latter being the subject of this thesis.



Project NANOTRIP : left, project organisation; right, illustration of a transformation zone at the tip of a crack in a cerium oxide doped zircone

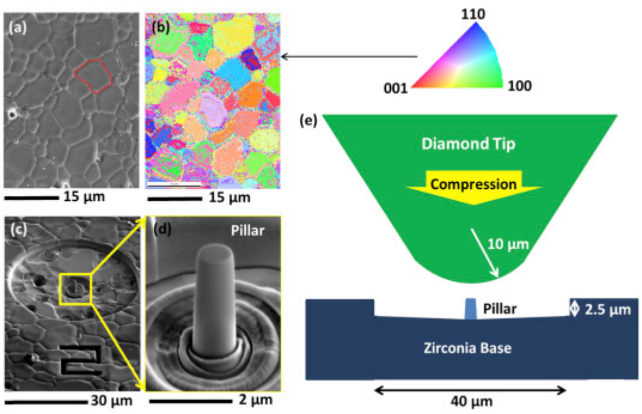
Objective of the thesis:

The objective of this thesis is to better understand the tetragonal-monoclinic phase transformation mechanism at the origin of this transformation-induced plasticity. For this, *in situ* mechanical tests on micro-pillars in Scanning Electron Microscopes and in combination with synchrotron X-ray diffraction will be performed.

The thesis project therefore aims to:

- Develop cerium oxide doped zirconia exhibiting a TRIP effect, with grain sizes (microstructures) compatible with the fabrication of micro-pillars and small samples for micromechanical tests in the broad sense,
- Perform micrometric-sized samples (micro-pillars, but also potentially micro-beams or other suitable geometries) by focused ion beam (FIB) micromachining,
- Carry out *ex-situ* mechanical tests followed by microstructural analyzes. Later on, *in-situ* tests in a scanning electron microscope, in order to obtain the constitutive laws of micro-pillars cut from a single grain or presenting interfaces to better understand the criteria for the transformation and transmission from grain to grain,
- Develop *in situ* synchrotron X-ray diffraction experiments on micro-pillars (Laue micro-diffraction, coherent diffraction imaging and phase mapping, etc.), in particular within the framework of tests carried out on synchrotron beamlines.

In general, the thesis will provide a better understanding of the conditions / criteria for nucleation of the monoclinic phase and its transmission across grain boundaries, the constitutive laws associated with the transformation at small scales or the effects of the level of cerium on the transformability (critical transformation constraint).



Fabrication of a micropillar within one grain with a pre-selected orientation (Figure taken from Zeng et al. Acta Materialia, Volume 116, 2016, Pages 124-135)

Profile of the candidate:

The PhD candidate should have:

- Either a training in “material science”, with proven experience (internship, end-of-study project) in the field of design and / or use of advanced characterization methods like X-ray diffraction and electron microscopy, with a passion for the understanding of the underlying physics of phenomena.
- Or training in "Condensed Matter Physics", with experience in the field of relationships between microstructures and material properties.
- Experience in *in situ* testing at the microscale would be a plus.

Applications (Cover letter + CV) to be sent exclusively to:

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