	Experiment title: Investigation of the elastoviscoplastic behaviour of halite (rock salt) by combined use of Digital Volume Correlation and X-ray Diffraction Contrast Tomography	Experiment number: MA1091
Beamline: ID11	Date of experiment: from: 5 nov. 2010 to: 9 nov. 2010	Date of report: <i>Received at ESRF:</i>
Shifts: 12	Local contact(s): Wolfgang Ludwig	
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Report:

Context

This project aimed at an exhaustive micromechanical investigation of the viscoplastic behaviour of halite (polycrystalline NaCl) in order to develop multi-scale crystal plasticity finite element method (CPFEM) taking into account the elastic and viscoplastic anisotropy of individual grains, specific microstructures (grain size and distribution...) and micro-physical creep mechanisms (crystal slip plasticity (CSP), grain boundary sliding (GBS)...). Such powerful method provides the opportunity to simulate the deformation behaviour of polycrystalline materials and structures on the basis of well identified physical mechanisms, and thus, to be virtually able to investigate any experimental conditions, for various industrial applications (Héripé et al., 2007). However, CPFEM needs first to be developed and tuned on the basis of experimental data providing the constitutive equations (macroscopic behaviour), local scale heterogeneity (by Full Field Measurements (FFM) and Digital Image Correlation (DIC), Doumalain et al., 2003, Bornert et al., 2009) and the active mechanisms. To date, such data are parcellar, often obtained separately, most often by “post-mortem” (destructive) characterization, or sometimes “in-situ” (during mechanical testing), but only by surface observations. However, it is still a crucial challenge to be able to demonstrate how representative surface informations may be, with respect to in depth microstructure.

Nowadays, progress in x-ray imaging and image analysis techniques allows for simultaneous investigations of microstructures and mechanical fields in the bulk of some polycrystalline and granular materials (Lenoir et al., 2007). Unfortunately, most of the industrial metallic materials present too strong X-ray attenuation. Therefore, we proposed to perform a simultaneous and complete 3D characterization of the elastoviscoplastic behaviour of Halite (analogue FCC polycrystalline material, little absorbing to X-rays), by performing 3D FFM (with 3D-DIC techniques, Bornert et al., 2004, Lenoir et al., 2007) on incrementally deformed specimens during ACT and DCT acquisitions, providing respectively 3D kinematic (displacement field) and microstructural (cristallographic orientations) data.

Samples

Practically, we prepared by hot isostatic pressing (200°C, 100 MPa) polycrystalline salt samples with coarse grains (200 – 500 µm) adapted to the DCT technique. The material contained either Cu or KCl fine grained (10 - 30 µm) particles, in proportion of 5 vol. %, which are more absorbing than NaCl and act as kinematic markers for the ACT-DIC technique. The approach mimics the usual 2D investigation method, where surface markers are deposited onto the sample surface to be observed during testing. Figure 1 shows the microstructure of the tested material and the marking techniques we applied for 2D (SEM-DIC) and 3D (ACT-DIC) investigations of kinematic fields during in-situ loading.

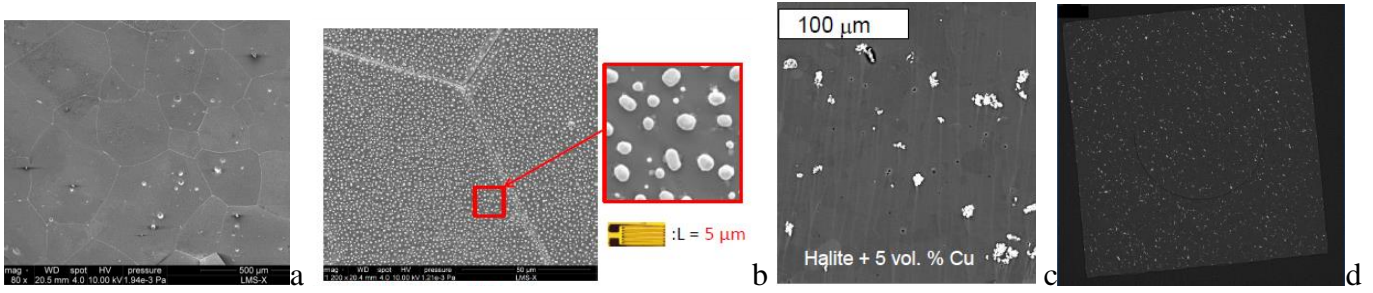


Figure 1: a) SEM micrograph at the microstructure scale (c.a. 25 grains). b) Surface marking by gold micro spheres (1 μm sized), c & d) volume marking by dispersed Cu micro particles (10 μm sized) observed by SEM (c) and ACT (d).

2D Results obtained in the lab

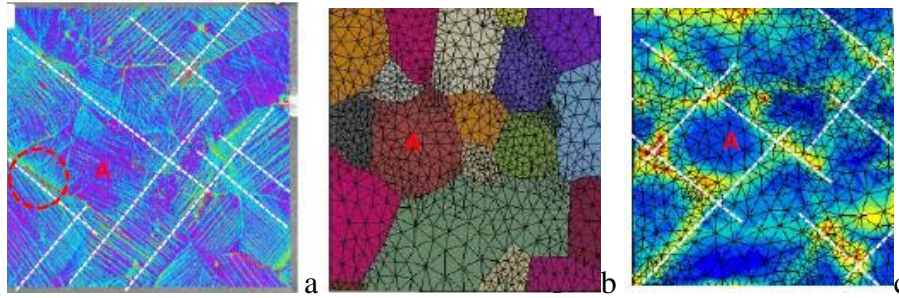


Figure 2: a) Equivalent (Von Mises) strain map superimposed onto the microstructure presented in Fig. 1a after 6 % global shortening (horizontal), showing localization bands of CSP (at c.a. 45° of the loading direction: highlighted by the white lines). Note that the strongest localization (an ex. is circled in red) corresponds to GBS (see text). b) FE meshing of the microstructure. c) CPFE modelling of equivalent stress distribution in the mesh (in simple compression, with respect to crystallographic orientations measured by EBSD), showing alternation of localization bands along interfaces and more relaxed grain interiors (for ex. grain A). Note that the strongest localizations correspond to interfaces where GBS was experimentally evidenced.

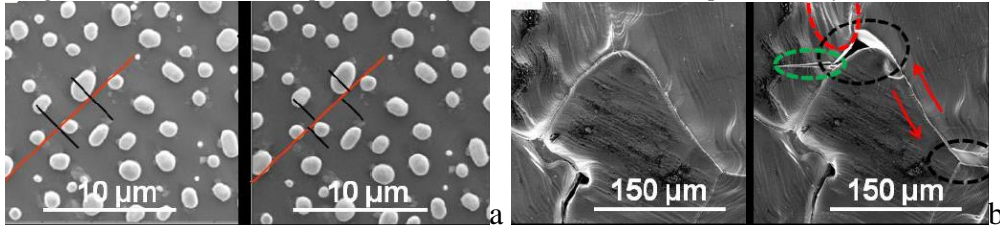


Figure 3: SEM micrographs characterizing the active micro-physical mechanisms of the NaCl viscoplasticity. a) CSP evidenced by intracrystal slip lines (markers offset between two loading stages). b) grain boundary sliding along an interface of two plastically incompatible grains. Note the micro-damage related to the GBS (which in itself represents inter-granular de-cohesion): triple point cavities and intra-granular micro-fractures.

2D full strain field measurements were realized at the LMS at the scales of the sample and the microstructure by optical and SEM observations during uniaxial loading. Figure 2 shows a 2D strain mapping obtained at the scale of the microstructure (a few tens of grains) after uniaxial loading, and corresponding CPFE modelling (Zebulon code, Onera). Localization bands at 45° of the loading direction are observed both experimentally and numerically, and some of these bands are matching. The local interaction length scale is of 2 - 3 grains. But, the correspondance between experiment and modelling remains qualitative. The discrepancies might be partly related to the influence of the underlying real microstructure. Conversely, the identification of the operational micro-physical mechanisms (Fig. 3) shows that in addition to crystal plasticity (Fig. 3a), limited amount of grain sliding (Fig. 3b contributing < 10 % to total strain) was often activated along interfaces between grains presenting strong plastic incompatibilities with respect to their active slip systems (depending on their crystallographic orientations and the loading direction). For polycrystals constituted of plastically anisotropic grains (which is the general case), the grain boundary sliding (GBS) is a necessary local accomodation mechanism. The latter is however not accounted for in classical CPFE modelling, which might contribute to the discrepancies between observed and calculated strain fields.

3D Results obtained at ID11 and ID19

Samples of < 1mm to 3 mm asize were first tested on ID11, using two miniaturized and manually screw-driven compression rigs (Fig. 4a). The facility specifications imposed < 1 mm sized samples for

combined ACT and DCT investigations. Hence, at first only samples 800 μm large and 1.5 mm long were incrementally loaded. Unfortunately, these samples were far too small (a couple of grains aside) and the manual loading proved fairly uncontrolled to allow for measurable ductile deformation before failure. Accordingly, priority was given to ACT investigation of larger specimens and the facility configuration had to be modified (with substantial loss of time), but in spite, the tested 3 mm-sized samples also failed in a brittle manner (Fig. 4d). Yet, these experiments allowed to test and tune the experimental conditions for the ACT and DCT techniques. Crystallographic reconstruction of specimen slices could be achieved, though complete 3D reconstruction was not attempted because of the lack of complete 3D ductile strain field for comparison.

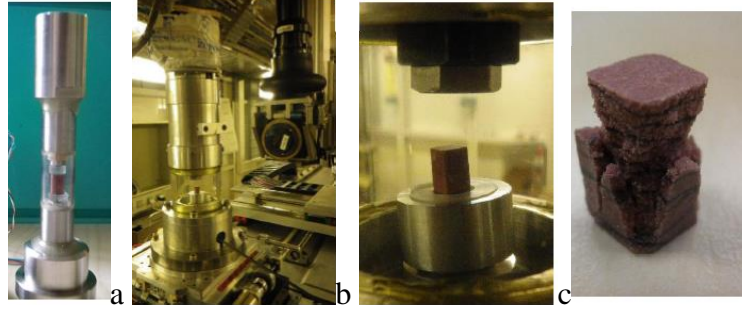


Figure 4: a) manual driven loading frame. b & c) MATEIS (INSA) traction machine mounted on ID19 with a 5 mm sized specimen. d) failed 5mm sized sample (samples appear red-brown due to the Cu particles).

In house time (2 shifts) could be obtained on ID19 for ACT investigation (DCT was impossible due to monochromator deficiency) during testing of larger specimens (5 mm aside) with an electromechanical press from INSA-Lyon (Fig. 4b). Figure 4c shows the experimental assembly, with a 5 mm sized specimen. The machine is designed for traction, so we had to adapt and glue rough pistons for compression. The poor piston flatness was partly compensated by the use of a ball-joint (see the tilted lower spacer before loading Fig. 4c). But, the minimum loading rate was about 10 times faster than usually performed for halite samples with similar dimensions in the SEM, and thus, the samples failed at fairly low macroscopic strains (Fig. 4d), though we could compute local 3D strain fields (Fig. 5).

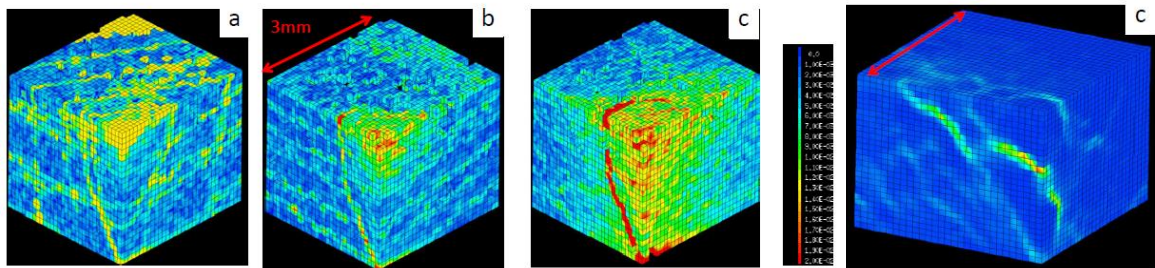


Figure 5: a), b) and c) 3D equivalent strain fields computed at three consecutive steps of loading. One can see the development of localized ductile deformation (yellow/orange) in the front corner of the reconstructed volume, followed by fracturing along the whole corner (in red). d) coalescence of localized micro-fractures prior to fracture propagation and sample ruin (Fig. 4d).

The unexpected premature failure of these larger specimens (similar to those successfully tested in SEM) precluded the development of extensive ductile deformation. The problem was primarily attributed to the improperly controlled loading. Defectuous piston flatness and fast loading rates triggered localization and intense grain sliding, with corresponding interfacial de-cohesion, micro-damage (cavitation development) and micro-fracturing (see Fig. 3b).

Perspectives

After the half success of the above preliminary work, we spent a year to improve both the materials and the mechanical testing condition. With the double perspective to investigate solely crystal plasticity and to avoid premature sample damage, we elaborated a new type of material, which presents a nearly double mechanical strength and well equilibrated polycrystalline microstructure, with clearly distinguishable faceted interfacial morphology and very low porosity ($< 0.5\%$, Fig. 6). In addition, we developed our own uniaxial compression rig (Fig. 6d), which is specifically adapted to mechanical testing of mm- to cm-sized samples on

the laboratory X-ray tomograph of Navier, and which allows for very slow loading rates ($< 0.1 \mu\text{m/s}$), as needed to avoid microcracking of the NaCl material. The new material presented in Fig. 6 was tested with the new machine and was conclusively shortened by 15 % before showing the premises of strength levelling related to damage. The machine (c.a. 15 kg) could be easily adapted to the ESRF facilities at ID19. The plexiglass frame will be modified in order to allow for both MCT and DCT scanning.

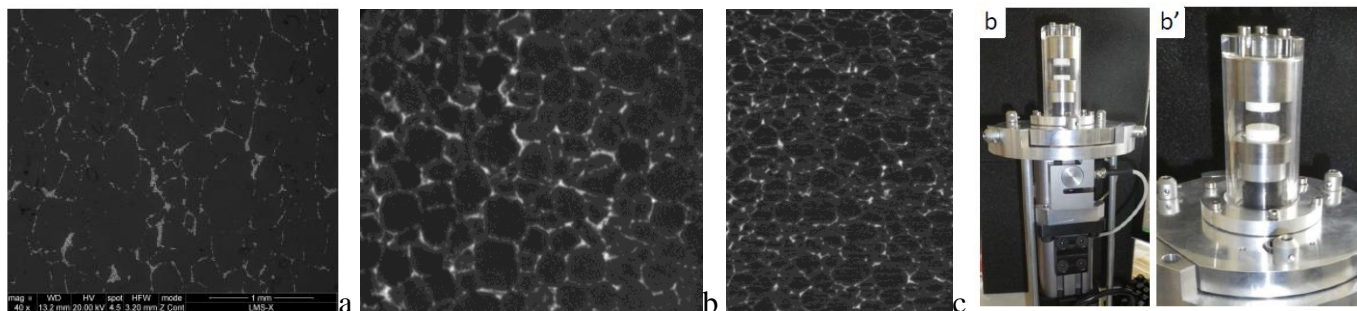


Fig.6. Microstructure of NaCl polycrystals with grain boundaries decorated by Cu particles. a) BSE-SEM micrograph showing Cu particles distributed along NaCl grain boundaries. b and c) ACT transverse and longitudinal slices from a parallelepiped shaped specimen (4 mm aside and 8 mm high) reconstructed at Navier. d) Newly developed uniaxial compression machine.

Publications and communications

Workshop on Alpine Evaporites and Tectonomechanics Colloquium. 2011. Saltzborg (Austria). Viscoplastic behavior of synthetic salt : In-situ SEM full field measurement, a micromechanical approach. Bourcier, M., Dimanov, A., Héripré, E., Bornert, M., Raphanel, J., Ludwig, W.

Congrès Français de Mécanique. 2011. Besançon (France) Etude expérimentale du comportement viscoplastique d'un polycristal de sel-gemme synthétique. Bourcier, M., Dimanov, A., Héripré, E., Bornert, M., Raphanel, J., Ludwig, W. **Peer Reviewed Proceeding**

European Geosciences Union. 2011. Vienna (Austria). Viscoplastic behavior of synthetic salt : In-situ SEM full field measurement, a micromechanical approach par Mathieu Bourcier, Alexandre Dimanov, Eva Héripré, Michel Bornert, Wolfgang Ludwig, and Jean Raphanel.

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