Coherent Diffraction Imaging as a tool to investigate polycrystal mechanics

Methodology for studying strain inhomogeneities in polycrystalline thin films during in situ thermal loading using coherent x-ray diffraction

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Supported polycrystalline thin films are ubiquitous in many applications ranging from micro-devices to hard coatings. The Grain size generally scales with the film thickness and both inter and intra-grain strain heterogeneities are expected [1]. The origins and magnitudes of these strain heterogeneities are of great interest in technology because many manufacturing and reliability problems are stress related. But measuring local strains in sub-micron size grains remains a real experimental challenge.

The recently developed Coherent X-ray diffraction technique [2,3,4] is very promising for measuring strain in small dimensions. Indeed this technique has proved its ability to evaluate the displacement field in direct space with a high resolution down to 8nm thanks to phase retrieval methods. Strain heterogeneities may hence be deduced from the retrieved displacement field.

Previous experimental studies using coherent diffraction have been made on objects which are isolated in direct space. In the case of a polycrystalline film, the experiment is more complex and has required a specific strategy. The first issue was to know exactly which grain was diffracting. For this, a small square area (~10µm Figure 1a) was isolated in the film using a focused ion beam. The mosaicity of the grains (dispersion in the orientation of the individual crystals) allows the signal to be collected from one single grain in reciprocal space without any overlapping. In addition, because the microstructure is well known, it is possible to compute realistic FEM simulations (here anisotropic elasticity with the Z-SeT software suite) to evaluate the displacement field within the grains [5] and then calculate the diffracted intensity in reciprocal space (Figures 1 b,c,d,e).

These methods have been applied to a 400nm thick Au polycrystalline film (111 fiber textured) deposited on a glass substrate. Coherent diffraction experiments were carried out on the CRISTAL beamline at SOLEIL. This beamline is well suited for coherent diffraction measurements. 3D mapping of the 111 Bragg reflection in a coplanar geometry from a single Au grain was carried out during a thermal cycle between 20 and 450° C (Figure 2). The mechanical loading was performed via heating up and cooling down the sample. Because glass and gold thermal expansion coefficients are different, a strain field is induced in the thin film during heating.

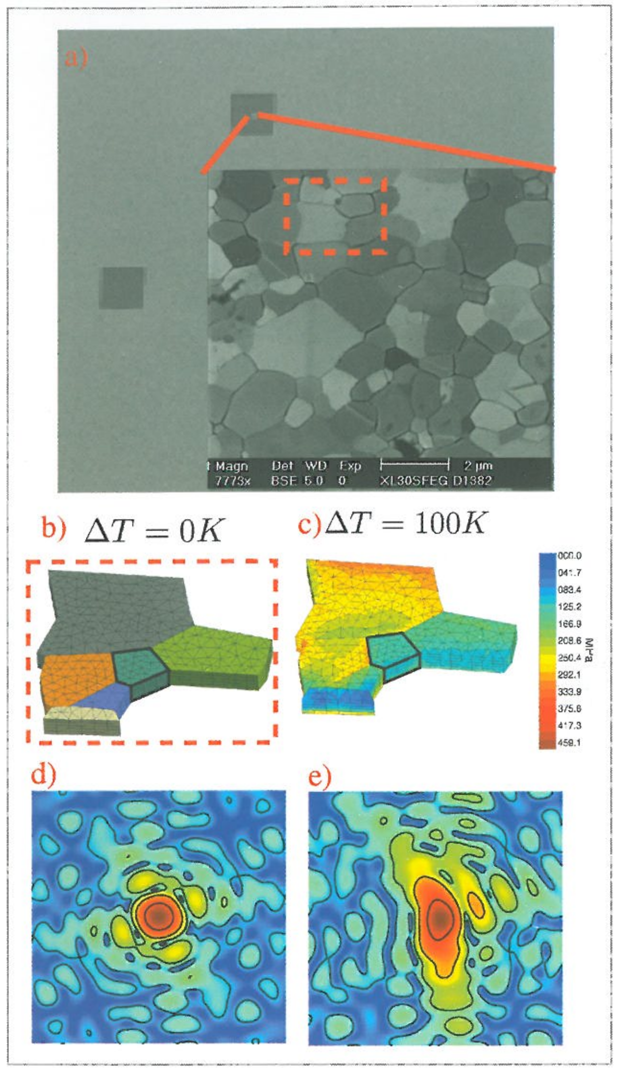

Figure 1: (a) A special setup was built in order to collect the diffraction signal from one grain in a specific region of the sample. Hence it was possible to compute realistic FEM simulations (b) to calculate diffraction intensity in the vicinity of the 111 Bragg reflection from one grain during a thermal loading (c).

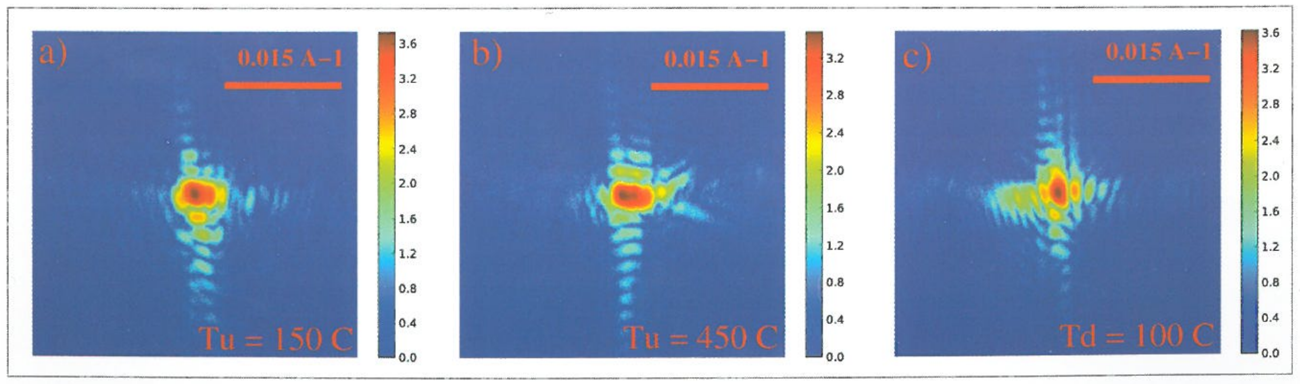

Figure 2: Experimental 2D slice of the 111 Bragg reflection (log scale) from a single grain at different temperatures (a,b,c). The fringes are related to the shape and the displacement field within the small grain (~1 µm).

The experimental signal is complex and analysis by phase retrieval is not straightforward. Indeed, algorithms do not always converge with highly strained samples. Thus alternative techniques are currently proposed and tested to overcome these algorithm stagnations. Finite element modeling with an elastic model shows qualitatively similar behavior during the thermal cycle. But investigating the experimental Bragg reflections in more detail indicates that plasticity probably plays a role even at low temperatures. Finite element plasticity will clearly be needed in order to get a better understanding of the coherent diffraction patterns.

In-situ experiments are very instructive for monitoring changes in strain heterogeneities and understanding the complex mechanical behavior of these polycrystalline samples (grain interactions, grain boundary homogenization, plasticity). More generally, this kind of experiment offers unique perspectives for the study of the mechanical properties of nano-objects.

References:

[1] R. Spolenak et al. Phys. Rev. Lett. 2003 90, 096102
[2] J. Miao et al. Nature 1999 400, 342
[3] M.A. Pfeifer et al. Nature 2006 442, 63
[4] A.A. Minkevich et al. Phys. Rev. B 2007 76, 104106
[5] F. Šiška et al. Modelling Simul. Mater. Sci. Eng. 2007 15, S217