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ESRF

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Texture changes during phase transformations: In situ observations at high temperature.

In spring 2004 and following op on exploratory experiments during a sabbatical leave at ESRF in 2001 (Puig-Molina et al. 2003, Wenk and Grigull, 2003), we had the opportunity to conduct a series of new experiments on beamline ID 15B. The goal was to investigate texture changes during phase transformations to better understand the texture memory and variant selection when a polycrystalline material returns to its original state. With experiments in a vacuum furnace on iron and zirconium we could document rapid recrystallization around 550ºC (Figure 1) and then a phase transformation around 850ºC. At that temperature diffraction patterns suddenly became very diffuse, characteristic of a highly stressed material.

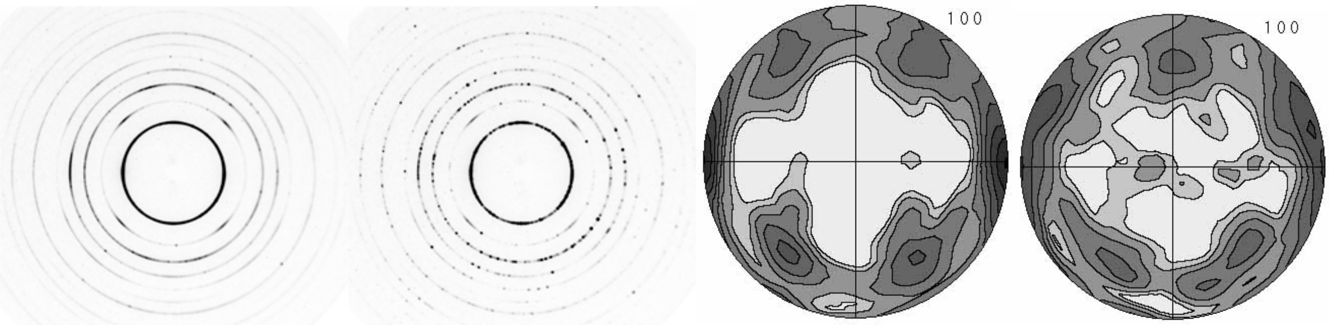

Figure 1. Recrystallization of zirconium measured in situ. Left 531ºC, right 560ºC. Reconstructed 100 pole figures are also shown (Ischia et al. 2005).

A first task was to develop new methods of image analysis to extract quantitative texture information. Previously we have shown that in principle it is possible to determine the orientation distribution from a single image (Wenk and Grigull, 2003), but the method of individual peak extraction was cumbersome and unsatisfactory. The images such as those of Figure 1 clearly document texture and it was for us a challenge to modify a new Rietveld method, developed largely for TOF neutron diffraction (Lutterotti et al. 1998), to analyze synchrotron diffraction images for texture. First the method was introduced on a combination of images at different tilts (Lonardelli et al. 2005), but subsequently we were able to use only a single image to obtain a full orientation distribution function, without imposing any sample symmetry (Ischia et al. 2005). Reconstructed (100) pole figures in Zr even capture such details as a change from a 100 maximum in the rolling direction to a 110 maximum (Figure 1, right side) that conforms with previous findings (Puig-Molina et al. 2003). We are satisfied that we have developed a method that can now be applied routinely to synchrotron diffraction images, including diamond anvil cell experiments (Wenk et al. 3004).

With these experiments we used two furnaces: The older furnace that was developed by Puig et al. (2003) and a newer much more sophisticated furnace that just became available for the experiment. The advantage of the old furnace is that it was simple to use, quick in response to heating and cooling but unfortunately we reached the temperature limit and just at the phase transformation (900C) the resistance heating element melted. With the new furnace we were able to go to higher temperature but this was just when our beamtime ended. One recurring problem was daily thunderstorms that paralysed ESRF for extended periods, which was particularly critical for kinetic experiments that rely on real time. A second problem was the small beam (200 µm). We started with fine-grained material but experienced rapis grain

growth above 500C. This was interesting in itself, particularly the high strain experienced at transformation but unfortunately it precluded us from a quantitative analysis of the high temperature texture. The qualitative results compare favourably with complementary neutron diffraction studies (Wenk et al. 2004, Wenk and Huensche, 2005). In the future it will be necessary to oscillate the sample in situ to provide better grain statistics. This will require beam attenuation to have more time for such averaging (exposure times were around 1 sec). A second unexpected problem was the saturation of the CCD imaging system with artefacts appearing in the images. Unfortunately all this became only apparent during the data analysis and is the basis for a new proposal that will be submitted to take account of these deficiencies. Nevertheless we learnt considerably and these pilot experiments made us ready for a new round.
We were very impressed by the efficient operation of the beamline by instrument scientists. It was extraordinary that the new furnace worked on its first trial.

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