ESRF

Experiment title:

Determination of the interface structure of $SrTiO_3$ bicrystals with x-ray diffraction

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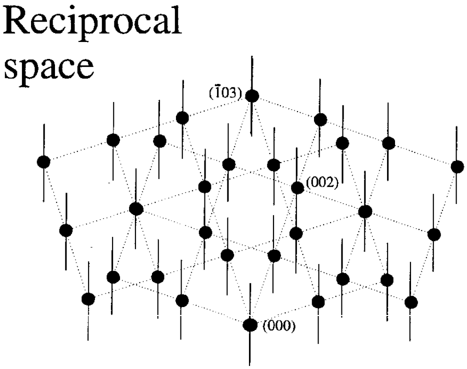
Report:

Grain boundaries in materials are technologically of great interest. However, it is difficult to obtain accurate structural information down to the atomic level in particular in a non-destructive way. We have studied the interface in $SrTi0_3$(103) bicrystals. This interface can be regarded a model system for grain boundaries. The bicrystals were formed by fusing two $SrTi0_3$(103) crystals together at high temperature and pressure. Transmission electron microscopy images and earlier XRD measurements at the ESRF[1] had suggested flat interfaces allowing structural studies by x-ray diffraction. A sketch of the symmetry of the $SrTiO_3$(103) bicrystal interface is shown in figure 1. The x-ray diffraction experiments were performed at the large kappa diffractometer at ID-11, the material science beamline. In order to be able to access the interface by x-ray scattering the upper crystal was thinned down to 25 pm. In order to further minimise the attenuation, the x-ray energy was chosen to 15.7 keV, just below the Sr K-edge. Because the lattice of the upper and lower crystal were rotated +/– 18 deg. with the respect to the (00l) direction, the reciprocal lattices and hence the crystal truncation rods (CTR)'s of the two were easily distinguishable. The conventional cubic Miller indices were used as reciprocal lattice coordinates. The intensity of a number of CTR's as a function of momentum transfer in the direction normal to the surface were measured. Scans along high symmetry directions parallel to the bicrystal interface were also performed. Surprisingly, satellite peaks were found in the k-direction, the direction perpendicular to the plane shown in figure 1. Typical scans in the vicinity of the (002) Bragg peak of the lower crystal is shown in figure 2, where satellites clearly are present at k = +/– 0.02 (and slightly indicated also at +/– 0.04). Further k-scans through the CTR at different momentum transfer in the direction normal to the surface, show that the



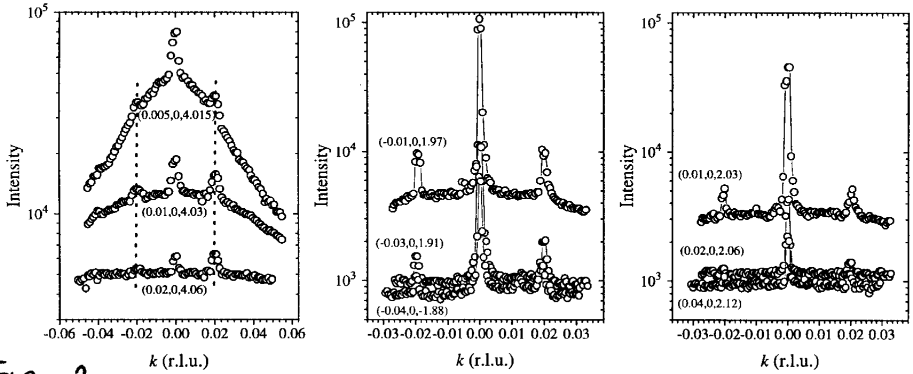


FIG. 2

satellites are due to rods of scattering parallel to the CTR. Similar satellite rods were found around the (004) Bragg peak and also around the (002) peak from the upper (thinner) part of the bicrystal. The displacement k = +/– 0.02 away from the main CTR was the same in all cases, showing the satellites neither are" due to mosaic spread nor facetting at the interface. The satellites must arise from reconstructions or relaxations at the interface. The satellite positions at multiples of k = 0.02 and the sharpness of the satellites show that they originate from modulations with a well defined period of about 50 lattice units. Furthermore, the intensity of the satellites decays fast as a function of distance away from the (002) Bragg peak, which means that the structural defects must have nearly the same crystal structure as $SrTiO_3$, and are hence supposively due to a well-ordered array of dislocations. The value of k = 0.02 corresponds exactly to the misalignment of the upper and the lower crystal with respect to each other and the dislocations must arise from the strain imposed onto the crystals during the fusing.

Fig 1. Sketch of a (103) bicrystal interface.

Fig 2. Scans along the k-direction around the CTR crossing the (002) Bragg peak. The satellites at k = +/— 0.02 are due to an ordered array of interface modulations.

[1] A. Kazimirov, J. Zegenhagen, I. Denk, J. Maier, D.-M. Smilgries, and R. Feidenhans'1, Surface Science, in print.