ESRF	**Experiment title:** In-situ crystal growth studies of inorganic materials using double crystal X-ray topography and laser interferometry	**Experiment number:** HS 260
Beamline: . ID19	**Date of experiment:** from: 21/6/97 to: 26/6/97	**Date of report:** 1.2.98
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Names and affiliations of applicants (* indicates experimentalists):

J. N. Sherwood University of Strathclyde, UK
D. B. Sheen* University of Strathclyde, UK
R. M. Vrcelj * University of Strathclyde, UK
P. J. Halfpenny University of Bath, UK
G. S. Simpson* University of St. Andrews, UK

Report: This experiment showed the feasibility of in-situ diffraction topography experiments of crystal growth from solution. A non-chopped white beam was utilised to illuminate the surface of a growing (111) face of Potassium Aluminium Sulphate (Potash Alum). The sample was mounted in a cell, specially designed such that topographic images could be taken. For ease of observation, the images taken are of the (222) symmetric reflection. The temperature control was maintained by a double bath system, enabling the main body of the growth solution to be kept saturated, whilst allowing a small variation in the temperature (thence saturation) of the solution passing through the growth cell. This is standard procedure to encourage crystal growth only in the cell, rather than any nucleation and growth throughout the rest of the system.

Two images of the crystal surface are given in figures 1 and 2. Figure 1 shows the crystal within the cell but prior to any crystal growth. Very little detail can be seen at the surface due to incoherent scatter, which is presumed to be caused by damage at the surface of the crystal.Figure 2 shows the same area of the crystal after 4 hours growth. The image is much clearer than in figure 1. The hexagonal nature of the surface can clearly be seen , as can the original crystal surface. Also plainly visible are sets of defects emergent at the surface of the crystal. These defects are unusual in that their contrast is not as expected. It is expected that a defect yielding a dark contrast image will lose contrast and disappear as it reaches the surface where the crystal is in a more relaxed state. However it is clear from this image that some of the defects create a dark fan of contrast as they approach the surface. It is believed that this is caused by a combination of relaxation phenomena and the geometry of the diffracted image, but as yet these images are not fully understood.

It is clear from these images that the concept of in-situ crystal growth observations using X-ray imaging topography is a feasible proposition. The intensity of the beam at station ID19, means that even with a very soluble material (such as Potash Alum) enough diffracted beam intensity is available such that images can be obtained in a reasonable space of time. The absorption of X-rays through the mother solution does not cause problems. However three problems still exist with the current set-up; firstly only partial imaging of the crystal surface is achieved, secondly photographic resolution is poor and lastly the angle of reflection gives highly compressed images. The first two of these problems are easily remedies. The use of a vibrating monochromator will give rise to a slightly divergent beam, which will enable the whole of the crystal surface to be imaged. The poor resolution of the photographic images will be solved by the use of nuclear emulsions, which, though less easy to handle than the high resolution film currently available at the ESRF, require much shorter exposure times (equivalent to the low resolution film used for the images in Figures l-3). The third problem is less tractable, ideally the resultant images would be of a highly asymmetric reflection, to yield less compressed images, but it appears that with further consultation with the station managers, much clearer and less compressed images will be obtained.

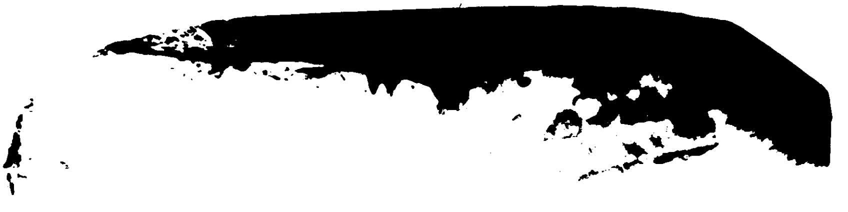

Figure 1 Damaged crystal surface prior to growth.

Figure 2 Crystal Surface after 4 hours growth