ESRF	**Experiment title:** **In situ** observation of the dynamics of extended crystals defects by X-Ray Topography	**Experiment number:** HC 226
Beamline:	**Date of experiment:** from: to:	**Date of report:** 19/08/96
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Report:

The aim of the present group of experiments is twofold: to build and test, with the help of the beamline staff, an apparatus allowing in situ X-Ray Topography observations at ID 19 during mechanical testing, and to observe in situ high temperature displacement and multiplication of dislocations in silicon. When ready, the system will be accessed freely by the users of ID19. The mechanical part of the apparatus is now ready, and the allotted beamtime was used to three main purposes:

- Insertion of the apparatus in the ID19 environment:

The apparatus was placed on the IDl9 specimen holder. The dimensional constraints (position of a specimen in the beam, and at the rotation center of the goniometer) were checked, as well as the external connections (water pipes, measurement, command and power electrical connections). The weight and size of the apparatus do not impede the translation and rotation movements of the goniometre. Under the current configuration, no difficulty was met in loading a Si specimen up to 500 N, and getting good images in white beam conditions. (the use of a Si monochromator makes imaging in monochromatic beam conditions very sensitive to minor distortions of the specimen. A vibrating monochromator will be very useful in this purpose).

preparation of further developments

The choice of materials used to make heating resistors, thermal screens and large size windows able to sustain a one atmosphere pressure difference, while remaining transparent to X-Rays is still open. The simplest solution is to use high density graphite (windows using pyrolithic graphite are currently under development at CHESS). The main drawback of this material is internal porosity, which has roughly the same effect as surface roughness, i.e. it introduces phase shifts in the beam, giving rise to a random contrast on X-Ray Topographic images. Images of Si specimen were recorded using one, two or no 11 mm thick windows placed respectively before and after the specimen. A strong diffusion was observed, especially due to the rear window. More, the phase contrast superimposed on the images is very clear on high resolution films.

A possible alternative material for windows is Aluminium, alloyed to low atomic weight elements (Si, Ti...), The operating conditions of such a window have not been tested yet, especially it's ability to sustain the thermal load due to the heating furnace during experiments. Good replacement materials for making resistors and screens limiting the heat losses from the furnace are not easy to find. The simplest solution seems to be the substitution of pyrolithic graphite, which is more homogeneous than high density one, and to reduce the thickness of screens and resistors as much as possible. (the phase contrast intensity varies as the square root of the thickness). This solution, and possible alternative ones, should be tested during the current months.

Imaging of various specimen

Two different types of specimen were used: i;elemental or compound semiconductors (Si, Ge, GaAs, InP), to test the beamline practical capacity with a monochromatic or white beam for in situ experiments by compression on thick specimen and ii; preparing compression experiments to be done on Fe-4 % Si bicrystals (an application for beamtime is currently written).

The time necessary to record a monochromatic image at 35.5 keV on a SR film was in the 0.2 s range for Si specimen 0.8 mm thick, i.e. was limited by the shutter mechanism. (The typical exposure times at LURE-DCI at 17.5 KeV are 120 s (monochromatic) and 5 s (white beam). In the present conditions, it is not possible to record a complete monochromatic image of a slightly distorted specimen, because of the geometrical properties of the beam. An oscillating monochromator will be necessary, at the expense of the exposure time. The white beam image of a thick and absorbing specimen (Ge 4 mm thick) can be recorded in 5 s.

Series of section white beam tomographs were performed on 0.2 mm thick Fe 4% Si specimen, to test the possibility of such experiments on thicker specimen containing subgrain boundaries. There is no apparent difficulty, except for the necessity of thick edge slits at short wavelengths. As the beam is not completely confined by thin edge ones, the expected image is bounded by a fading one on either side. This problem should be solved by the use of the tungsten carbide precise slits now installed at ID 19.