



**Experiment title:**  
 Scanning Microbeam Small Angle X-Ray Scattering  
 Investigations of Materials with Structural Gradients

**Experiment number:**  
 HC414

**Beamline:** ID13      **Date of Experiment:** from: 22.5.      to: 27.5.96      **Date of Report:** 27.8.96

**Shifts:** 15      **Local contact(s):** Dr. C. Riekell      *Received at ESRF:*  
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**Report:**

The identification of spatial gradients of the defect structure in materials are often needed to understand the properties of these materials. Scanning Micro-Beam Small Angle Scattering (SM-SAXS) with a lateral resolution in the order of magnitude of some microns can be an important tool for the characterization of structural gradients in materials, In a first tests it was already shown that a Bragg-Fresnell-Lense (BFL) can be applied for SM-SAXS investigations of the damage structure in the plastic zone ahead of an crack tip in a AlMg alloy.

The objective of these experiments shall be the application of SM-SAXS with BFL arrangement for the investigations of the differences in the carbide structure in a welding seem as well as of the gradient of the nitride structure in nitrided layers in tempered steel.

'The experiments were performed at the "MICROFOCUS" beamline ID13. An X-ray wavelength of  $1.25 \text{ \AA}$  was used. The first order interference of the BFL with an aperture of  $200 \text{ }\mu\text{m}$  has reduced the X-ray beam cross section at sample position to a diameter of about  $2 \text{ }\mu\text{m}$ . The SAXS intensity should be measured with a gasfilled detector. Tests with a radioactive source showed that the detector was able to neglecting the iron fluorescence radiation when it was optimized for the used energy.

Due to the low efficiency of the BFL at the used energy it was needed to exclude the unfocused zero order interference by a pinhole of a diameter of  $10 \text{ }\mu\text{m}$ . In order to align the pinhole a "photonic science" CCD camera was placed at sample position. With the help of this configuration the alignment of the pinhole was possible. The removal of the "Photonic Science" camera from the beam effected the loss of the previous alignment of the pinhole. A number of attempts to remove the camera without misalignment were not successful. Attempts to find the beam without the CCD camera on the basis of moving the pinhole in a trial and error modus did not provide satisfying results as well. Due to this problem the proposed experiments could not be performed. The proposed experiments should be performed with a focusing mirror/monochromator optics which can reduce the cross section of the beam to  $10 * 10 \text{ }\mu\text{m}$ . This beam cross section seems to be small enough to perform the proposed experiments. The focusing mirror/monochromator optics is much less sensitive relating to misalignment than the BFL optics,