

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

<http://193.49.43.2:8080/smis/servlet/UserUtils?start>

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.



	Experiment title: Development of a method to investigate the damage structure near a crack tip in metals by means of SM-SAXS.	Experiment number: TC131
Beamline:	Date of experiment: from: 03.04.2002 to: 04.04.2002	Date of report: Renewed: 06.09.2002
Shifts:	Local contact(s): Stephan Roth, Manfred Burghammer and Christian Riekel	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Kristel Van Ouysel*, Gudrun Müller, Jürgen Böhmert (head of dept.) Fz-Rossendorf e.V., Dept. Material Behaviour and Component Safety, Institute of Safety Research, Bautzner Landstraße 128, 01328 Dresden		

Report:

In order to study the defect gradients around the crack-tip (nucleation, growth and coalescence of microvoids) in an Al-2024 alloy without anisotropic grain-boundary interference, small regions ahead of the crack-tip were scanned using a 5 μ m by 5 μ m collimated beam (Figure 1).

Three scans were made, the first scan (step size 0.2mm) covered a region in y (parallel to the crack) from 0 to 2mm and from -1.5 to 2.1mm in z and was carried out to map the damage zone around the crack-tip (0,0). The second scan (step size y:18 μ m, z: 24 μ m) was more detailed to investigate the defects in a smaller region close to the crack-tip (y: 0 to 200 μ m; z:-60 to 60 μ m) using a smaller step size. A third, linear scan (from 0 to 150 μ m in y, step size: 7 μ m) was run to look inside the grains (20-25 μ m in diameter).

All scans revealed more or less isotropic (figure 2, lower) and to a lesser extent diamond-shaped (figure 2, upper) scattering images, occasionally adorned with single streaks. An important results from this test experiment is that the typical anisotropic streaks with hexagonal symmetry attributed to grain-boundary scattering were no longer observed¹. The number of streaks were on the whole much less frequent and occur most frequently in closer vicinity of the crack which points to tiny cracks in the material (which do not cut the surface of the specimen). There is an indication of tiny through-cracks from SEM and TEM-images which supports this explanation.

From these two-dimensional distributions of the scattered intensity, the azimuthally averaged intensity was determined (Figure 3). The azimuthally averaged intensity tends to decrease with increasing distance from the crack tip [1]. In an attempt to map out the damage zone, the integral intensity was then determined and the values plotted for every scan point of the scanned region within the damage region (Figure 4 for a small scan around the crack tip and Figure 5 for a bigger scan covering most of the expected damage zone). Figure 4 shows a distribution of stronger scattering, but the strongest scattering is about 100 μ m from the crack tip. This may be due to scattering of nano-sized precipitates which were observed in the TEM (Figure 6).

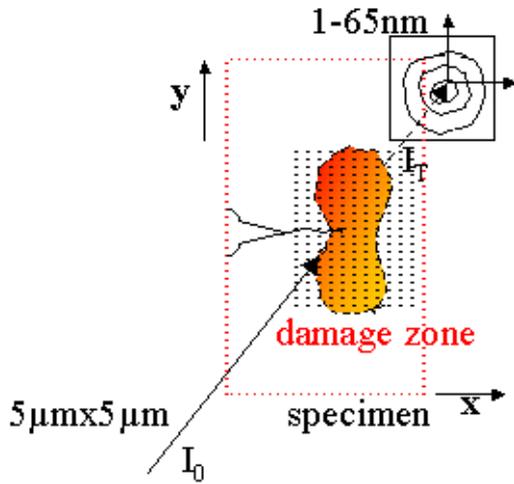


Figure 1: Schematic diagram of the scanning set-up.

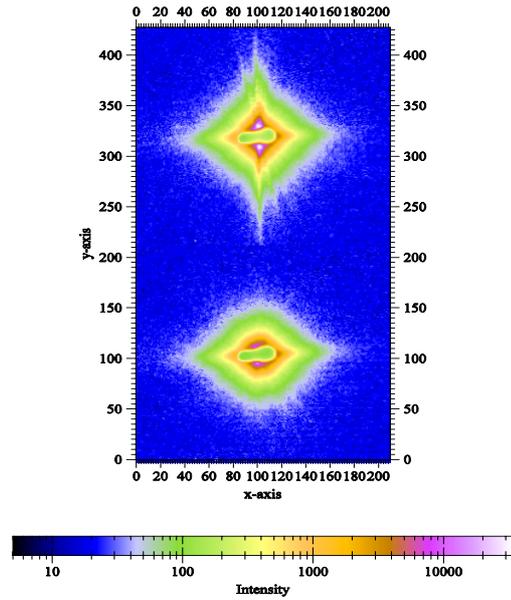


Figure 2: Typical scattering images, diamond shapes with streaks and rounder shapes.

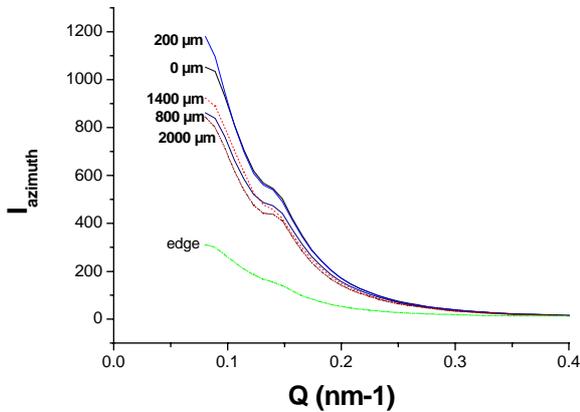


Figure 3: The azimuthally averaged intensity tends to decrease with increasing distance from the crack tip.

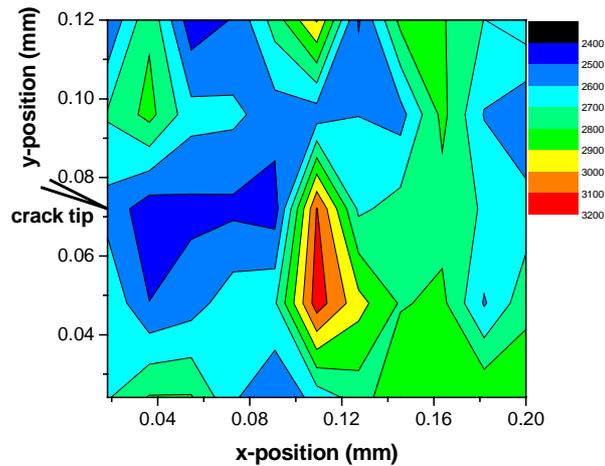


Figure 4: The integral intensity reveals the distribution of the scatterers over a small range across the damage region.

Figure 5 reveals the integral intensity over most of the expected damage zone around the crack tip. A stretched zone is observed which compares with a stretched damage zone. The intensity, however, decreases in a direction almost perpendicular to the crack tip. The reason for this is not yet clear and further investigations are under way.

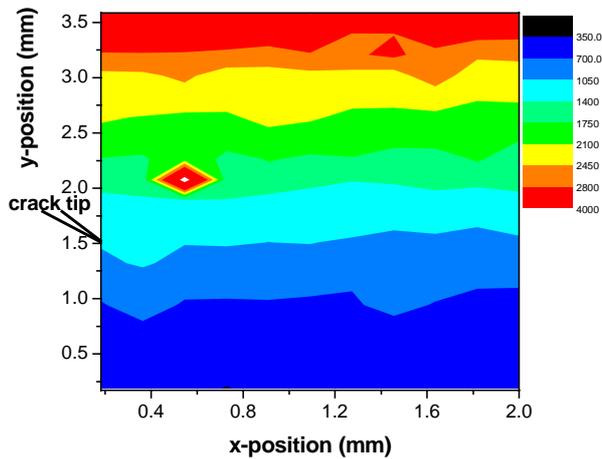


Figure 5: The integral intensity reveals a stretched damage region which, however, gradually decreases in intensity in the direction perpendicular to the crack tip.

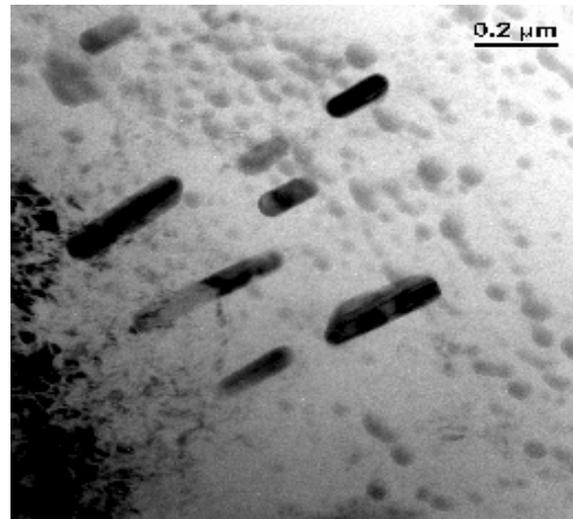


Figure 6: Rod-like precipitates ranging from 10-400nm in length were observed in the TEM.

SEM (Scanning Electron Microscopy) investigations have revealed a stretched shape of the damage zone around the crack tip, which has been attributed to grain-boundary reorientation during deformation, for a different alloy and further experiments to investigate also alloy 2024-T351 are under way; unfortunately no dislocation patterns could be visualized with this technique due to the large dislocation networks present. FEM analyses revealed a somewhat larger plastic strain region for the 2024-T351 alloy and stretching of the region with crack growth. A stretched shape is achieved only at crack extensions beyond 2mm, but this may be related to the limited thickness (1.6mm) of the sheet specimens, which has not yet been taken into account in the calculations; further improvements are being made for an accurate comparison of the shape of the damage zone with SAXS, SEM and PAM.

Conclusion:

A first experiment was carried out at the ESRF, MICROFOCUS beamline. One important observation was that the disturbing influence due to grain boundary scattering was no longer present. This allowed us to evaluate the azimuthal intensity without grain-boundary interference which has shown that the intensity tends to decrease moving away from the crack tip. A plot of the integral intensity for all the scan positions reveals a stretched shape within the damage region and this stretched shape is also found in the SEM photograph, for a different alloy though, but with comparable crack extension, and predicted from FEM calculations for both alloys, but at larger crack extensions. Improvements for accurate comparison will be completed. The gradual decrease of the intensity in a direction perpendicular to the crack, however, has not been clarified yet.

Abstract to the paper contribution to the 12th International conference on small angle scattering in Venice, 25-29 August, 2002. To be published in a special issue of the Journal of Applied Crystallography.

Title: Investigations of the damage structure near the crack tip in Al-alloys by means of Small Angle X-Ray Scattering. K.Van Ouytsel, G.Müller, J.Böhmert (Fz-Rossendorf, Dresden), S.Roth, M.Burghammer (ID13, ESRF)

Structural materials, such as bainitic and austenitic steels and aluminium alloys as used in the nuclear and aircraft industry are subject to external stresses in different environments: water, air, ...

Understanding the growth of an assumed preexisting crack under these conditions is of prime importance to prevent extensive crack propagation and failure of the component.

Preliminary SAXS investigations at Hasylab (Hamburg) and Esrf (Grenoble) on deformed Al-samples revealed microvoids, dislocations and anisotropic grain-boundary scattering.

The ongoing research aims to investigate the early stages of ductile fracture, the nucleation, growth and coalescence of microvoids and the evolution of the spatial dislocation distribution. A first step consisted of the selection of two suitable commercial Al-alloys, alloys 2024-T351 and 6013-T6 used in the aircraft industry and well-characterized in the literature, based on information about the grain size, amount of

precipitates and on tensile data. Different crack lengths were subsequently introduced into M(T)-type specimens through precracking and deforming in tension.

Within the above framework, a small region around the crack tip of a 2024-T351 specimen was scanned using a microfocused beam of 5 μ m in diameter (ESRF, ID13-Microfocus).

Basic analysis has indicated the presence of spherical and rectangular shaped scatterers/precipitates and hardly any grain boundary influence, which represents an important improvement. Information from fracture toughness tests, from the simulation of the distribution of stresses and strains by means of finite element analysis (FEM), from SEM and TEM and positron annihilation experiments, will support the SAXS investigations.

