EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

ESRF

# 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.

| ESRF | **Experiment title:**<br>Development of ID15A for crack tip strain imaging | **Experiment number**:<br>ME 452 |
|---|---|---|
| **Beamline**: | **Date of experiment**:<br>from: 27-11-02 to: 2-11-02 | **Date of report**: |
| **Shifts:** | **Local contact(s)**:<br>Thomas Buslaps | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

**Axel Steuwer* & M Preuss**

**Manchester Materials Science Centre, University of Manchester, Grosvenor Street, Manchester M1 7HS, England UK**

**Javier Santisteban***
**Department of Materials Engineering, The Open University, Walton Hall, Milton Keynes, MK7 6AA, England, UK**

## Report:

The beam line ID15A is a highly versatile beam line with a multitude of potential applications in materials engineering. In particular, the broad energy spectrum and high intensity enables the non-destructive investigation of residual stresses in large engineering components at high spatial resolution, as well the monitoring of processes in controlled environment. Furthermore, the energy-dispersive X-ray diffraction spectra (EDXRD) allow the application of the powerful and versatile Rietveld method. Where previously only single peak strain measurements have been undertaken, we have developed the framework on ID15A at which the information of the whole diffraction pattern is now available for investigations. These has been undertaken by carring out a series of proof-of-concept studies. This development will be beneficial for a variety of industrial and academic users, and has already stirred the interest of the UK aerospace, automotive and nuclear energy industries amongst others where a fundamental understanding of the lifing of safety critical components play a critical role. This technology is particularly useful for large (>4cm path length) titanium based components for which neutron diffraction strain measurements are notoriously difficult, and for which other synchrotron beam lines might not deliver the penetration required, or for high spatial resolution measurements on steel components & testpieces.

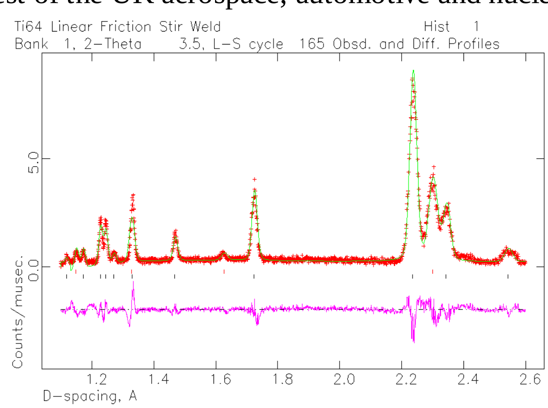


**Figure 1: An example of GSAS Rietveld refinement of the spectrum of Ti-6Al-4V showing the ability to resolve the two phases.**

### Rietveld Refinement

A typical Rietveld (Pawley) fit is shown in Figure 1. The refinement has been done using GSAS. Special code has been developed by the Manchester Material Science Centre for the porting of ID15A data into GSAS format, which allows the instant refinement of collected data. Although

GSAS is capable of fitting EDXRD spectra directly, we found that converting the measured spectra from energy to a fictitious reciprocal scale (similar to time-of-flight data) proportional to the d-spacing, and subsequent logarithmic rebinning ($\Delta d/d$ = const) improved the statistial results. Indeed, this allowed us to use many software routines developed for ENGIN-X diffractometer at the spallation source ISIS in the UK. Strain accuracy at very high spatial resolution ($0.15 \times 0.15 mm^2$ at $2\Theta=3.5^o$) was found to be of the order $10^{-4}$ for both phases in Fig. 1, which can be improved using larger gauge volumes or longer counting times.

**Some Examples and Proof-of-Concept Studies**

i) ***Powders***: we have measured & succesfully refined the following materials: α-iron, γ-iron, duplex steel (α+γ), molybdenum, nickel, titanium-6al-4v (α+β) with a strain accuracy consistently better than $10^{-4}$ for individual phases.

ii) ***Titanium Linear Friction Weld***: Linear Friction Welding (LFW) is seen as a key technology for the aerospace industry as it enables the joining of difficult to bond materials, and can be used both as a repair process, as well as to build the complex structures required for today's gas turbines. Essentially, friction welding is a non-melting fusion process with potentially widespread use in the automotive and aerospatial industry. Before LFW parts can be put into application, sensitivity of the mechanical and microstructural properties of the joints to the weld process parameters have to be assessed. We have been able to measure the strains across the weld (~60points) in a 3cm thick Ti-6Al-4V linear friction weld (see Figure 4) in less than 1h with an accuracy of 80με at 0.15×0.15 $mm^2$ spatial resolution.

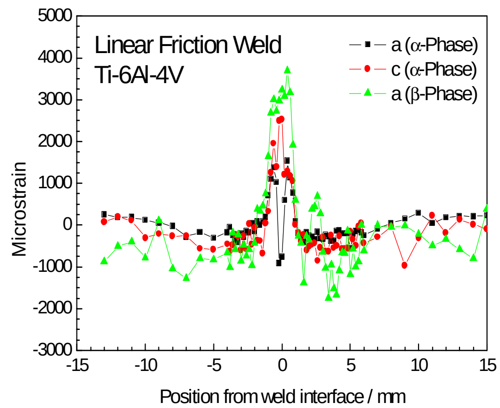


**Figure 4: The residual strains across the weld in a Ti-6Al-4V linear friction weld**

iii) ***Cold expanded fasterner holes:*** This technique involves placing a longitudinally split-sleeve within the hole to be expanded and then drawing an oversized tapered mandrel through the assembly, so that the material undergoes permanent plastic deformation. Upon removal, a self-equilibrating residual stress field is produced owing to the spring-back of the surrounding elastic material, which gives rise to improved fatigue resistance. Fig. 2 shows the residual hoop strains around a cold-expanded hole measured on ID15A using EDXRD. The gauge volume used was 0.15×0.15mm at $2\Theta=3.5^o$. Counting times for measuring 240 points were in total ~8h at an accuracy of 60με.

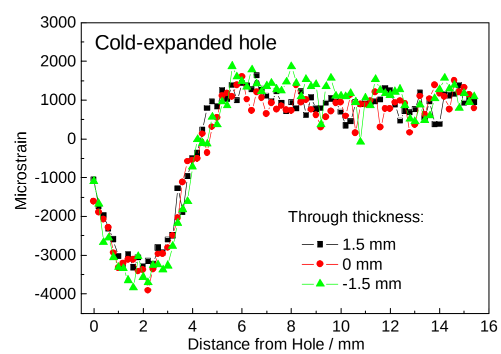


**Figure 2: The longit. strain around a alum. CE hole against poposition.**

iv) ***Crack tip strain imaging***: This has been the most succesfull of all proof-of-concept studies, whose results were immedeatly applicable to ME 451, and allowed the improved analysis of data collected during ME 279. The (large) gauge volume was $0.4\times0.4 mm^2$ at $2\Theta=3.3^o$ in a 2.5cm thick steel and counting times were 40s per point giving a very high strain resolution of 20με. Total counting time for this map of 800 points, *including* positioning were only ~10h. Unfortunately we have not been able to undertake the proposed in-situ straining due to a shortage of time.

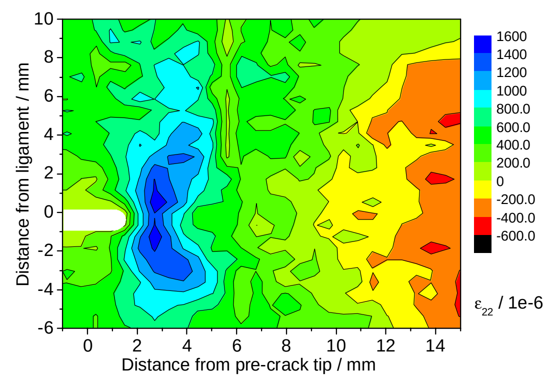


**Figure 3: The longitudinal strain around a fatigue crack-tip after plastic deformation measured using ID15A. The crack tip is located at the approximate coord.: (2,0)**

**Technology Transfer**

The knowledge gathered from this experiment has already stirred considerable interest in the UK engineering community and academia, and will be part of a forthcoming long-term proposal on crack characterisation and visualisation. This ability of accurate strain mapping at high spatial resolution in large engineering components promises to be a major break-through, and exceeds by far the capabilities of neutron techniques. The code is available upon request, and has been installed at the FaME38 facility of the ESRF/ILL.