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# 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 via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

***Reports supporting requests for additional beam time***

Reports can 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:** Crack formation and propagation in a novel tungsten-fibre reinforced tungsten composite | **Experiment number**: MA-1119 |
|---|---|---|
| **Beamline**: ID 15A | **Date of experiment**: from: 28.11.2010 to: 01.12.2010 | **Date of report**: 26.02.2015 |
| **Shifts:** 9 | **Local contact(s)**: Mario Scheel | *Received at ESRF:* |


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

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## Report:

In the MA-1119 experimental campaign tungsten fibre-reinforced tungsten composites ($W_f$/W) were investigated by means of in-situ tomographic observation during mechanical bending tests. In these composites the intrinsic brittleness of tungsten is overcome by extrinsic mechanisms of energy dissipation. Besides the bridging of cracks by intact fibres the dominating effects are the ductile fibre deformation and fibre pull-out respectively depending on the ductility of the fibres. A novel upright 4-point bending possibility was designed to allow the in-situ observation by tomography. Various Samples with different interfaces in the as-produced and in a heat treated state were tested.

*1. Specimen preparation*

Various $W_f$/W composites samples were investigated: single fibre samples (as-produced and heat treated), multi fibre samples (as-produced and hot pressed) and samples of pure matrix material (as-produced and heat treated). The fibres are drawn tungsten wire with a diameter of 150µm. The interface is produced by magnetron sputtering. The matrix is fabricated by chemical vapour deposition where $WF_6$ is reduced by $H_2$ in a surface reaction to form solid tungsten. Cylindrical specimens with a diameter of 1-2 mm and a length of 20 mm were thinned in the central measuring area to 1 mm. An artificial notch (wire saw + FIB cut) is produced to emphasize crack initiation in the measuring area. Single fibre samples with two types of interfaces were investigated $Er_2O_3$ with a thickness of 1 µm and $WO_x$ with a thickness of 670 nm. The heat treatment was done at 2000K for 30 min which leads to an embrittlement of the single fibre samples.

*2. Experimental*

The tomographic setup built up on our previous experiment MA-859. We used white beam in the energy range of 60-240 keV. 600 projections with an exposure time of 300ms each over a rotation angle of 180° were collected for one tomogram. As scintillator a LuAG:Ce (Screen) with a thickness of 25µm was used. Two step optical lenses (total magnification 10 times) and a Frelon 2k camera (2048x2048 px) were used to detect a picture. As the transmission for 1 mm tungsten was only 3% a special aperture, in this case made of

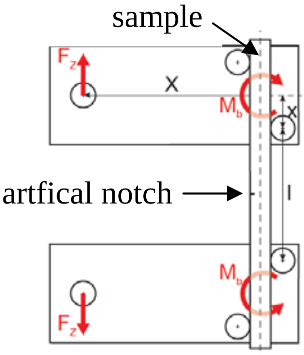


*Fig. 1 Schematic drawing of upright 4-point bending test*

silver; was used to avoid premature camera saturation on the peripheral domains of the samples (as in MA-859). In addition 40mm of Si and 10mm of water were used to regulate the beam energy. Altogether the spatial resolution was improved to 1.5 µm (voxel size 0.5 $\mu m^3$) compared to 5 µm in previous experiments.
For the in-situ bending a mechanical testing machine used before for tension tests was combined with the newly designed upright 4-point bending equipment. The machine was capable of a maximum load of 5 kN with a minimum displacement rate of 0.1 µm/s. The load was measured by a 500N range load cell and the displacement was calculated using the displacement rate of the machine head. To allow upright 4-point bending test on that machine specially designed specimen holders were used to alternate the direction of the force transmission into a vertical position (Fig.1). By applying a vertical load, the rotatable lever arms move apart and tilt applying a bending force on the specimen. The test was performed step wise in a displacement controlled mode. A tomogram was taken at each bending increment.
Experiments were done in the allocated 9 shifts from 28.11 till 01.12 and in two additional shifts on the 02.12. (spare time before next experimental campaign).

## *3. Results*

Of 46 prepared samples 24 were tested during the campain. Succesful tomograms could be produced for all samples. The multi-fibre samples as well as the pure matrix samples however showed no controlled crack propagation due to very brittle behaviour and/or lack of sample quality (caused by the production process itself not by the preparation).

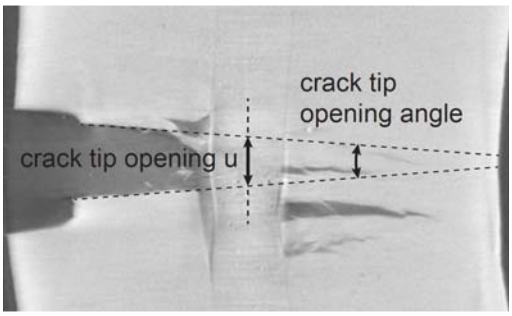


*Fig. 2 Plastic bridging*

8 out of 21 single fibre composite samples allowed the in-situ observation of crack propagation and various mechanisms of extrinsic toughening like crack bridging, crack deflection or fibre pull out. For both interface types in the as-produced state briging by the fibre was detected after crack intitation followed by an intese plastic deformation (plastic bridging). The crack tip opening as well as the crack tip opening angle could be determined using the tomograms (Fig.2). The stress-displacement measurement allowed a quantitative determination of the toughness contribution by this effect. For the heat-treated and therefore embrittled samples the behaviour was different depending on the interface. The samples with $Er_2O_3$ interface which was stable during the heat treatment showed (elastic) crack bridging as well as pull-out for some samples. For the first time the elastic bridging contribution could be determined quantitatively (Fig.3). For samples with $WO_x$ interface, which is lost during the heat treatment, no toughening mechanisms could be observed.

## *4. Summary*

The campaingn was very successful especially regarding the single fibre composite samples. The bending possibility worked well and allowed the observation of stable crack propagation not only for the as-produced samples (due to the ductile fibre quite easy) but also for the embrittled samples. Thus it was possible to proof for the first time a key feature of $W_f/W$ composites which is that the toughening also works after embrittlement. The high resolution tomography together with mechanical stress-strain data allowed in addtion a quantification of the effects.

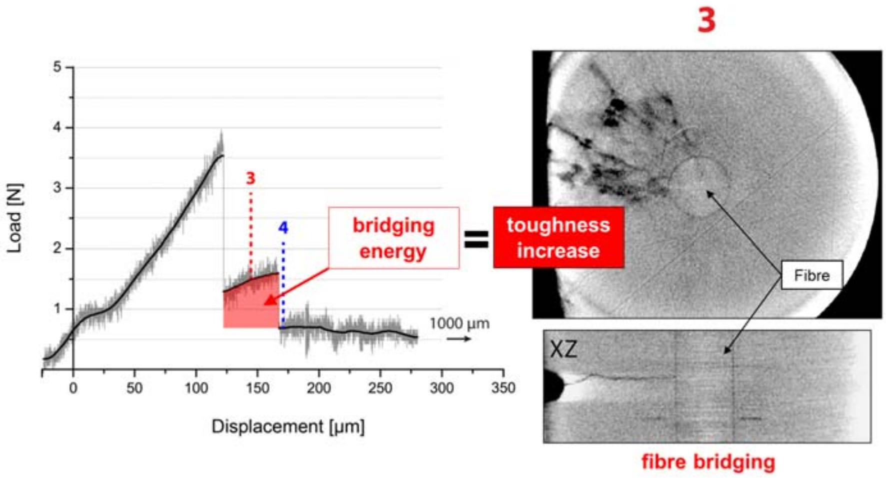


*Fig. 3 Qualitative and quantitative measurement of elastic bridging*