	Experiment title:	Experiment number:
ESRF	An Investigation of the interface in composite materials by use of µSAXS and µWAXS	SC-1099
Beamline: ID-13	**Date of experiment:** from: 05-Jul.-04 to: 11-Jul.-04	**Date of report:** 2-March-05
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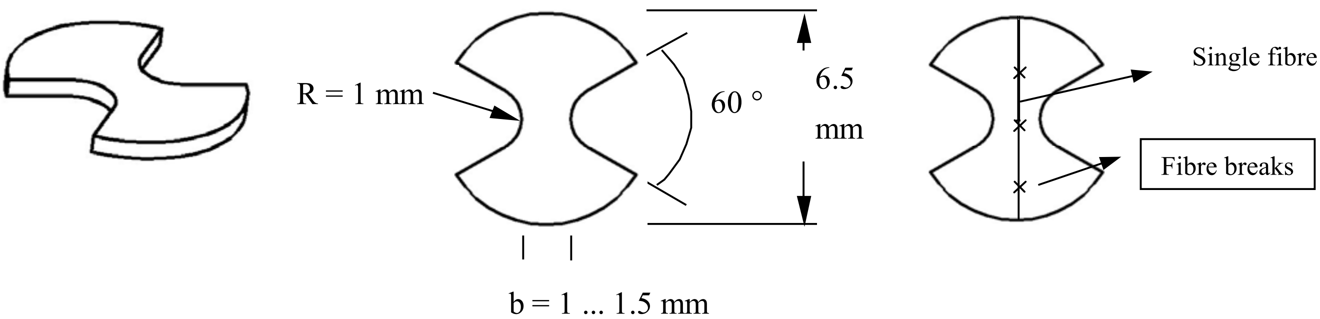
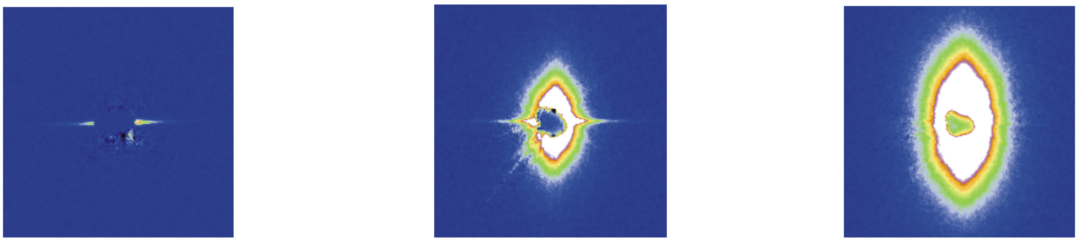
The interface in composite materials is one of the key determining factors for the overall performance of the final material. As a result a great deal of effort has been dedicated in understanding and engineering the interface to obtain the optimal performance out of composites. Yet a number of key questions still remain ananswered. In our experiment we attempted to answer some of these quyestions, and in particular how damage proceeds at the interface under load, and what is the structure of highly oriented transcrystalline interfaces in thermoplastic composites with a semicrystalline matrix.

In the first experimental setup composite materials with the geometry shown in figure 1 were prepared out of glass fibres and an epoxy matrix, and subsequently tested off-line. As the load on the samples increased over time, the fibre started to fragment into smaller pieces and debond from the matrix due to the increased load, through a phenomenon termed as fibre fragmentation [1].



<u>Figure 1:</u> The geometry of the samples

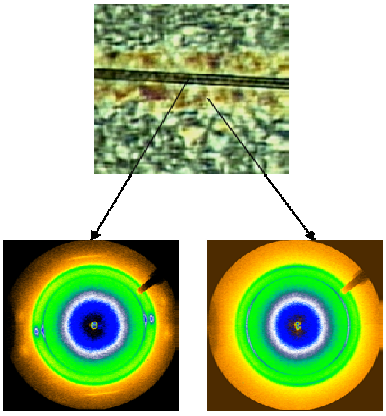
The predamaged sample was then mounted on the ID13 beamline, and a y-z SAXS scan using a 5µm beamspot was carried out around the interface. The results revealed the presence of a very sharp streak from the interfacial area (Figure 2a) while in the area between two fibre breaks a highly elliptical distribution of intensity could be seen on the detector (figure 2c). Modelling of the interfacial streak via a rod form factor of infinitely long length, yielded a diameter of c.a. 25 nm for the interfacial crack. The large increase of the intensity in the fibre broken regions is due to extensive generation of voids, denoting that contrary to what it was generally believed before the matrix inside these zones has undergone extensive local deformation in comparison with the matrix at the interface and the bulk. Hence, it may be that these zones are the ones that probably control the catastrophic fracture in composites. On the other hand, scans at the position of the interface in the fibre break zone, revealed that the interfacial crack is present along with the generated voids from the energy released during the crack, as it may be seen from Figure 2b, where both the characteristic narrow streak and the elliptical intensity distribution are present.



(a) (b) (c)

Figure 2: µSAXS patterns from three different positions. (a) at the interface, (b) at the same level of the interface but in the fibre break region, (c) in the centre of the fibre break region.

A second experiment was also performed on kevlar 49/nylon 66 microcomposites that were isothermally treated under different conditions. As a result of these treatments, a highly oriented transcrystalline layer was developed and the samples were mounted on the ID13 beamline to carry out WAXS scanning microscopy using a 5µm beamspot. The geometry of the test and two typical WAXS patterns from the transcystalline layer and the fibre are shown in figure 3.



a h

Figure 3: X-ray patterns of TC layer in kevlar 49 reinforced nylon 66 microcomposite: a) typical reflections of aramid fiber and nylon; b) [100] and [010]/[110] reflections of tc layer

The preliminary results from Synchrotron experiments showed, that TC layer is obviously oriented and there is no "sheaflike" structure, as proposed in previous works [3-4]. Every scanned sample showed the consistent lamellae orientation of TC layer. Figure 1 presents two main reflections of nylon 66 (triclinic unit cell): [100] (inner circle) and [010]/[110] (outer circle). It is clearly seen that two arcs of [100] reflection on TC layer equatorial relatively to the fiber axis. While [010]/[110] reflections are meridian relatively to the fiber axes. On the base of this data, the microstructure of transcrystallinity will be decoded in short time. In all cases, the microstructure of transcrystalline layer is still under investigation.

The performed experiment shed more light in the phenomena associated with interfacial failure in composites and has provided more understanding as to what happens at the interface on the nanometer scale.

References

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