EXPERIMENTAL INVESTIGATION OF GLASS FIBER/MATRIX INTERFACIAL DEBONDING UNDER TRANSVERSE LOAD

K. Martyniuk[1*], B.F. Sørensen[1], P. Modregger[2] and E.M. Lauridsen[3]

[1]*Department of Wind Energy, Section for Composites and Materials Mechanics, Technical University of Denmark, Risø Campus, Frederiksborgvej 399, 4000 Roskilde, Denmark*
[2]*Swiss Light Source, Paul Scherrer Institut, 5232 Villigen, Switzerland*
[3]*Department of Energy Conversion and Storage, Technical University of Denmark, Risø Campus, Frederiksborgvej 399, 4000 Roskilde, Denmark*

Keywords: single-fiber composite, in situ, interface, debonding

Abstract
Two- and three-dimensional (2D and 3D) in situ observations have been performed in order to investigate glass fiber/matrix interfacial debonding initiation and propagation. This study focuses on microscale damage of the single-fiber composites subjected to transverse load. Scanning electron microscopy and high resolution X-ray microtomography have been utilized for visual observations of damage initiation and propagation during interrupted tensile tests. Qualitative data acquired during in situ tests is presented and its usefulness for fracture mechanics is discussed.

1 Introduction

The mechanical behavior of the fibre/matrix interface controls fibre/matrix debonding, which strongly affects the microscale damage evolution and thus the macroscopic behavior of composite materials. Therefore, the determination of the mechanical properties of the fiber/matrix interface is of high importance.

A range of methods for experimental evaluation of interfacial behavior have been proposed and implemented [1]. However, most of these are meant to be used for evaluation of interfacial shear properties. There is lack of test methods, which would allow for investigating the mixed mode fracture properties of the interface. Such a test method for studying interfacial properties under load normal to the fiber/matrix interface would provide parameters needed for micromechanical modeling like e.g. fracture energy as a function of mode mixity or debond crack kinking criteria. Therefore, a more advanced experimental methodology is needed. Such a methodology could be coupled to numerical studies of interfacial debonding under transverse load reported in the literature [2].

In this study, progressive debonding was studied by means of high resolution imaging techniques like scanning electron microscopy (SEM) and X-ray microtomography. Tensile tests of single-fiber model composites were performed in order to observe damage initiation and propagation in situ. The acquired knowledge will be used for development of a numerical model for prediction of mechanical properties of composites from knowledge of basic properties of fiber, matrix and fiber/matrix interface (micromechanical modeling).

2 Materials

Single fiber-model composites were manufactured using E-glass fibers and epoxy resin Epoxydharz HT 2, manufactured by Faserverbundwerkstoffe GmbH, Composite Technology. Glass fibers with diameter of ~50 μm were provided by Ahlstrom Glassfibre. Despite the larger fiber diameters used for this model composite, the surface properties of the fibers were still consistent with those used by commercial composites manufactures. Sizing (type R338) applied on the fibers' surface ensured proper chemical and physical fiber/matrix adhesion [3]. Fibers were alignment in a rubber mould with a square cross section of 2,3mm×2,3mm and subsequently pre-strained in order to prevent any residual stresses, which arise during the curing process. The procedure was presented elsewhere [4]. Residual stresses could influence interface properties and cause debonding prior to loading which would prevent studying of interface properties. Rubber moulds were filled up with the resin and cured for 24 hours at 40° C, followed by post curing at 50° C for 10 hours. Finally, the samples were grinded and polished in order to remove any fiber/matrix microcracks from the surface. Moreover precise surface polishing was required for subsequent SEM observations.

Once polishing procedure has been completed, samples with square cross section were reshaped into dog-bone shape in order to facilitate subsequent mechanical testing (Fig.1.). The reshaping process was conducted by placing the square samples in a dog-bone shaped mould and refilling free space in the mould with the resin. In order to protect the polished surfaces, the gauge length was covered by tape before place into the dog-bone shaped moulds. Curing and post curing processes were then repeated.

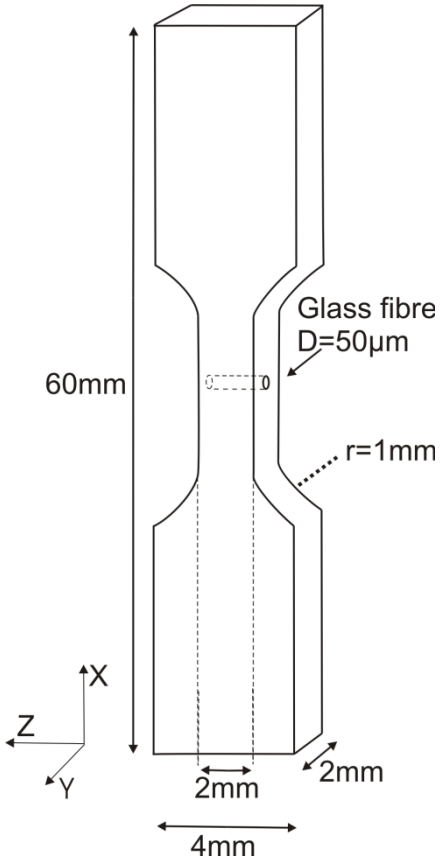

Figure 1. Specimen geometry.

2 In situ tensile tests in SEM (2D studies)

2.1 Experimental procedure

In order to observe crack initiation and propagation in situ under tensile loading, mechanical tests were conducted in the vacuum chamber of an environmental scanning electron microscope (Zeiss, EVO60) using a special custom made tensile loading fixture [5] (Fig.2), designed to be mounted in the SEM. An acoustic emission (AE) sensor was placed on the sample in order to detect damage before it becomes visible in the SEM. The tensile test was interrupted at various load levels until ultimate failure. This enabled observing and capturing images of the crack propagation using the SEM. The pixel size of the acquired SEM micrographs is ~200nm.

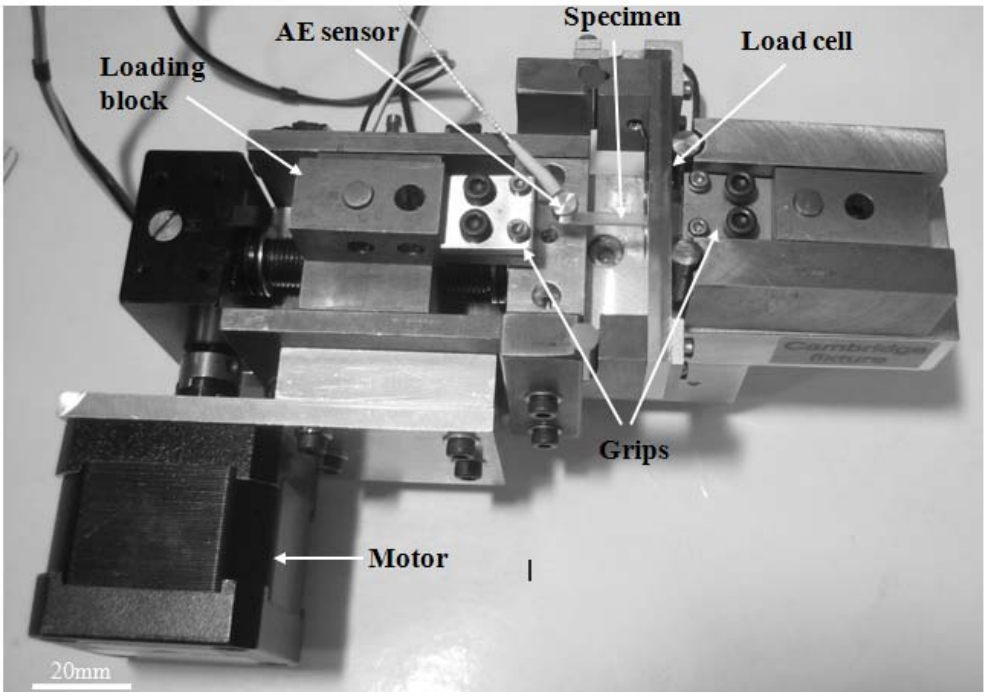

Figure 2. Test set up for tensile testing in SEM.

2.1 2D results

The fiber/matrix interfacial debonding initiation and propagation were observed in situ during the loading. Identical damage sequences were observed in several samples and thus shown to be reproducible.

The debonding initiation was observed to occur at angles 0° and 180° with respect to the applied tensile load direction at load level of ~25MPa (Fig.3.). The debonding angle θ grow with increasing load level. An example of this process is presented in Fig.4 where measured debonding angles are plotted versus applied nominal stress.

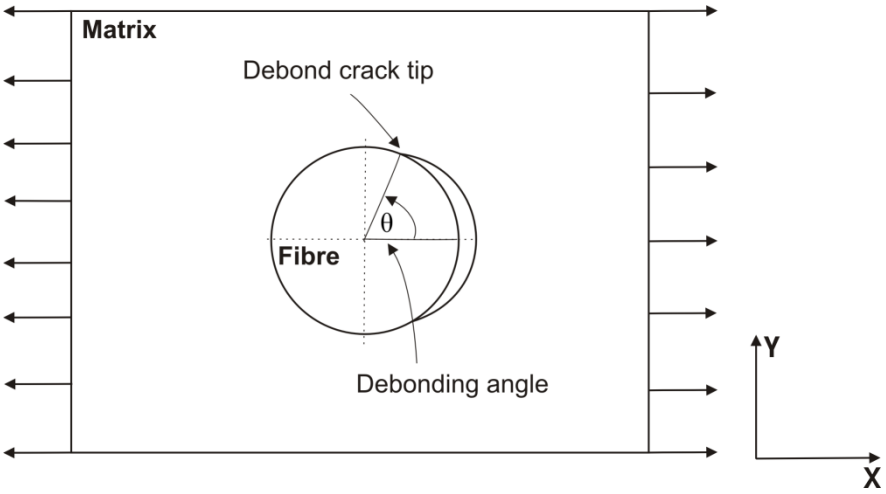

Figure 3. Cracked interface under a transverse load-close up around the fibre embedded in infinitive matrix.

Subsequently the debond crack kinks out from the interface into the matrix at a stress level of ~30 MPa. An example of the entire damage sequence for one sample is presented in Fig.5.

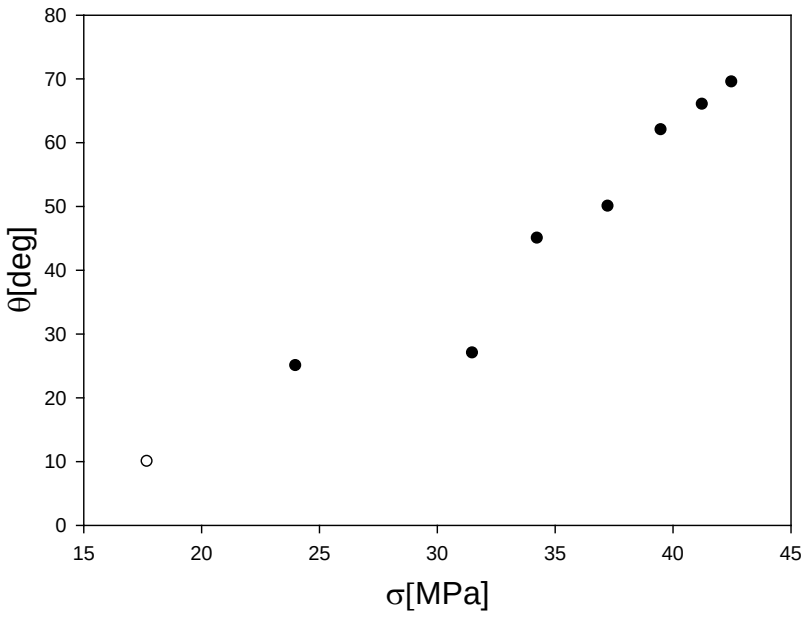

Figure 4. Debonding angle measured during in situ tensile tests as a function of applied stress.

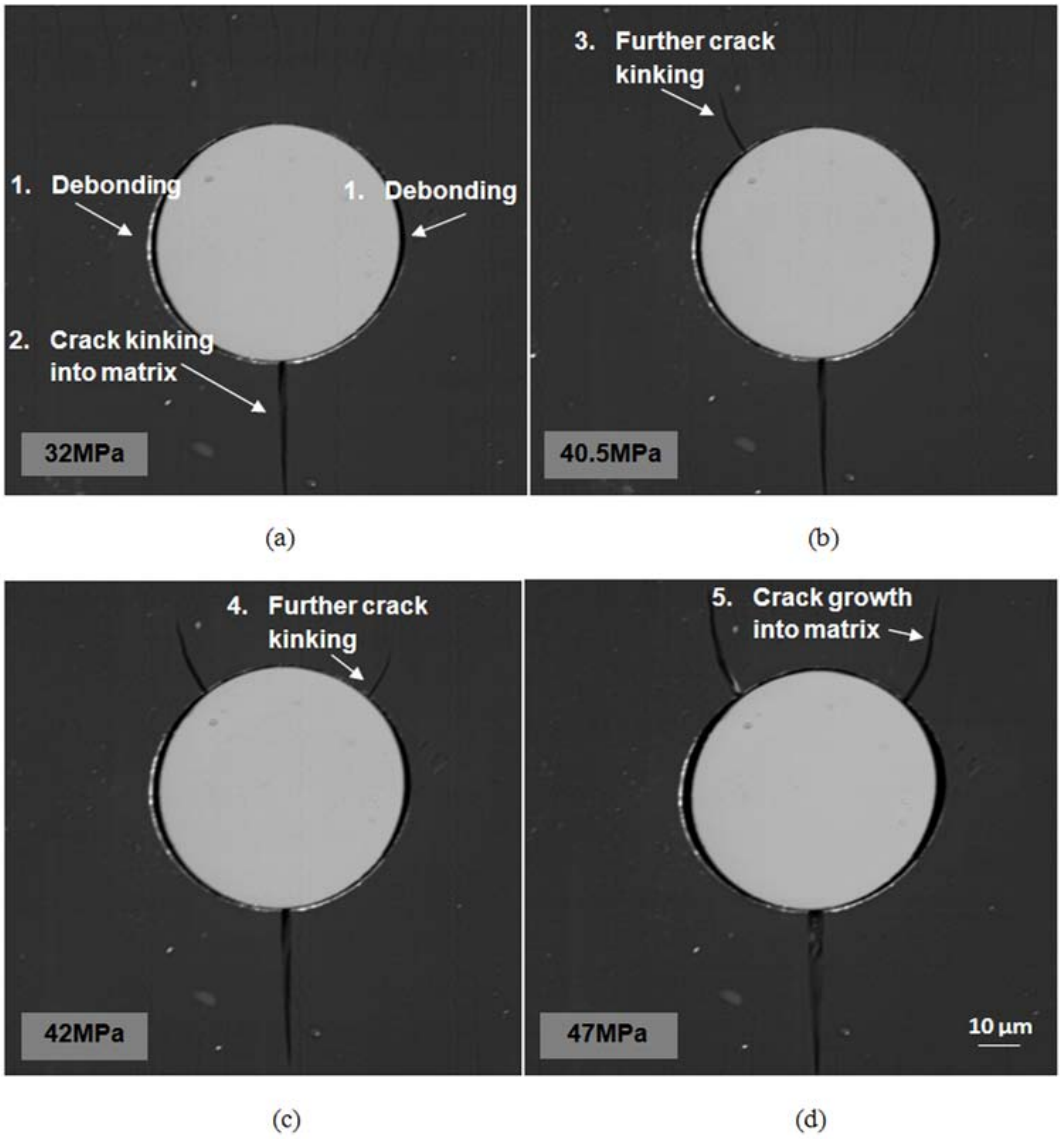

Figure 5. SEM micrographs presenting interfacial damage sequence observed on the free surface.

3 In situ tensile tests using X-ray microtomography (3D studies)

In order to investigate whether the results derived from 2D observations in the SEM are representative of what occurs inside the material, a series of 3D studies were carried out using high resolution X-ray microtomography.

3.1 Experimental procedure

The in situ microtomography tensile tests were conducted at the TOMCAT beamline located at the Paul Scherrer Institute, Swizerland. A special custom-built loading rig was utilized in order to allow for X-ray scanning during tensile loading [6]. The static tensile load was applied transversely to the fibre direction.

Samples were scanned using a 20keV X-ray beam**,** with exposure time of 350ms. In total 1601 projections were acquired during sample rotation of 180°, using standard absorption mode. From the projections a total of 2048 slices were reconstructed at each load step, with voxel size of 0.74×0.74×0.74 µm. To allow for high resolution imaging of the fiber/matrix interface, local tomography was utilized. Thus, a volume of 1.5×1.5×1.5 µm was scanned corresponding to approximately half of the thickness of the samples.

3.2 3D results

The complex test set-up was successfully applied during the experiments. Although the samples were loaded manually, compared to the automatic loading in the SEM experiments, it was possible to observe in situ the interfacial debonding in 3D.

In order to analyse the aquired data, 3D volumes as well as representative 2D projections were carefully investigated.

Interfacial debonding was observed to propagate in both directions: the debonding angle grew on the free surface as well as debond crack propagated along the fiber direction as showed in Fig. 6. The debonding angle on the free surface grow up to 2θ=136° and the debond length along the fiber reached l_d= 105µm (~2 fiber diameters) (Fig.6).

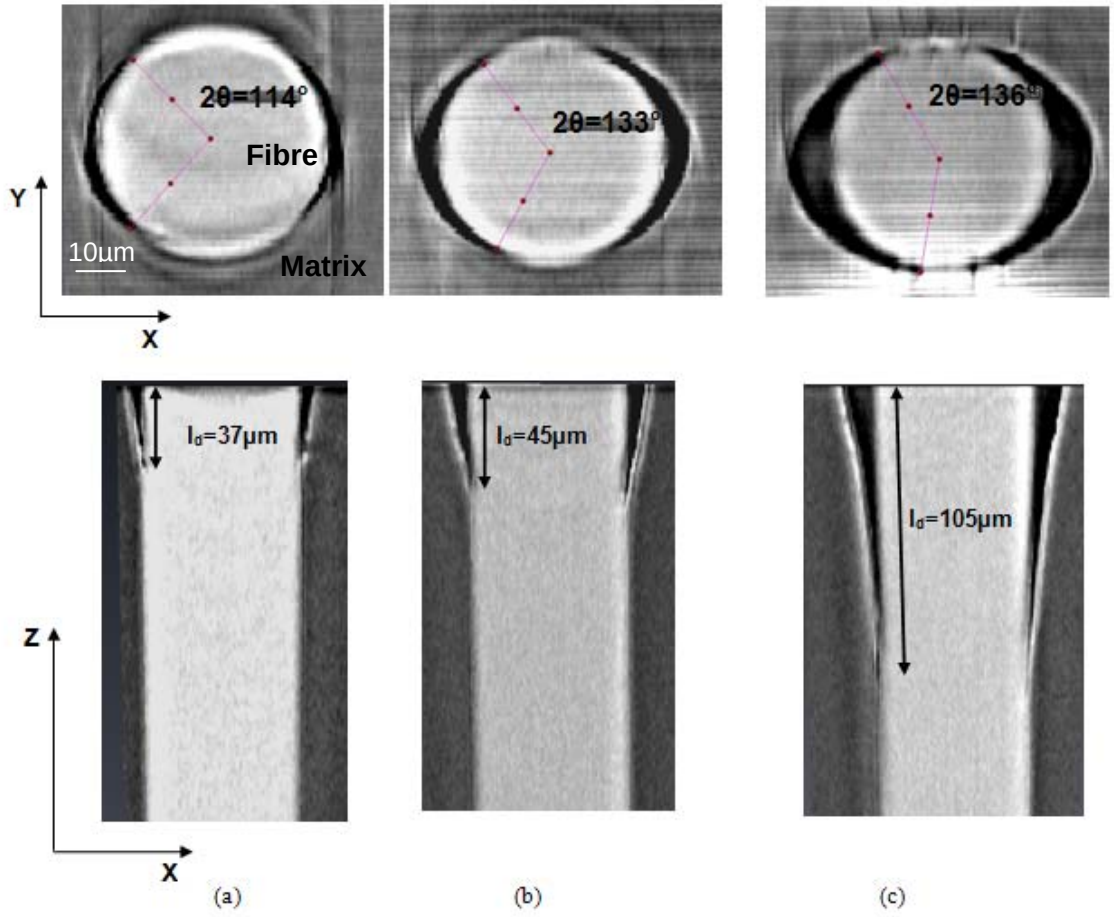

Figure 6. X-ray tomography virtual slices showing the interfacial damage evolution as function of applied load: (a) σ=34.25MPa, (b) σ=41.25MPa, (c) σ=42.5MPa. The *x*,*y*-plane corresponds to free surface and *x*,*z*- plane corresponds to the internal plane along θ=0°- θ=180° as indicated in Fig.3.

An example of 3D data presenting progressive debonding is showed in figure below.

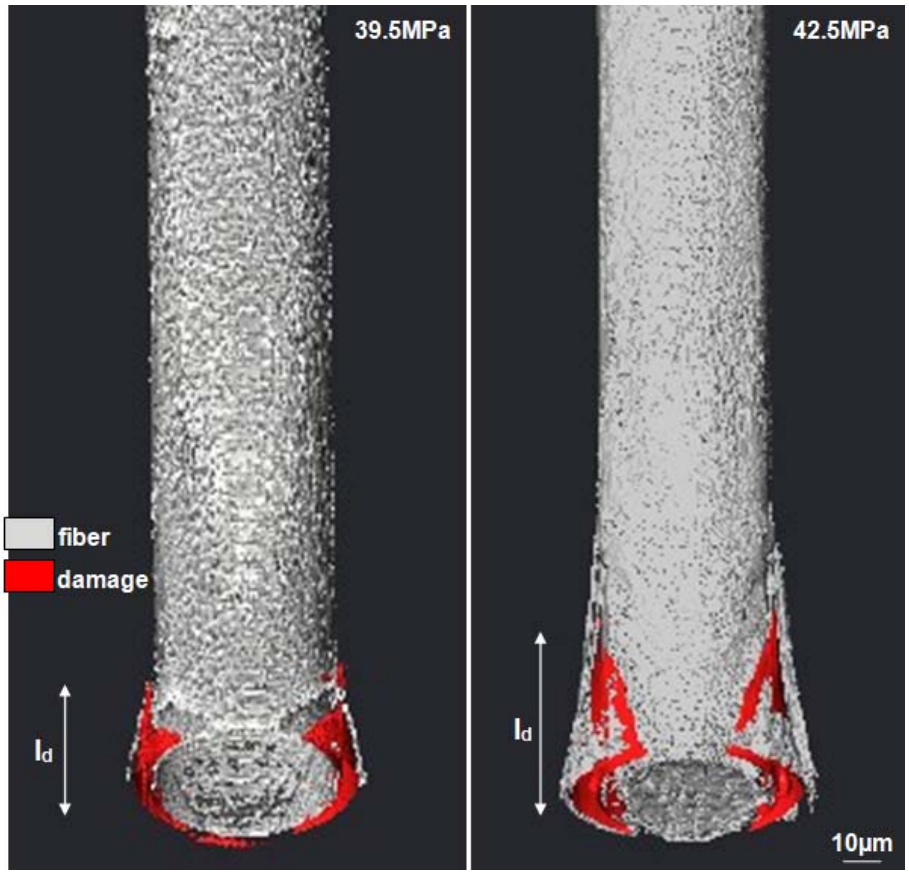

Figure 7. Interfacial damage evolution for two loads steps. For better visualisation, the matrix is rendered invisible.

High resolution 3D data allowed studying the debond shape along the fibre. The interior debonding angle 2θ for two load steps is presented as a function of the distance from the free surface in Fig.8.

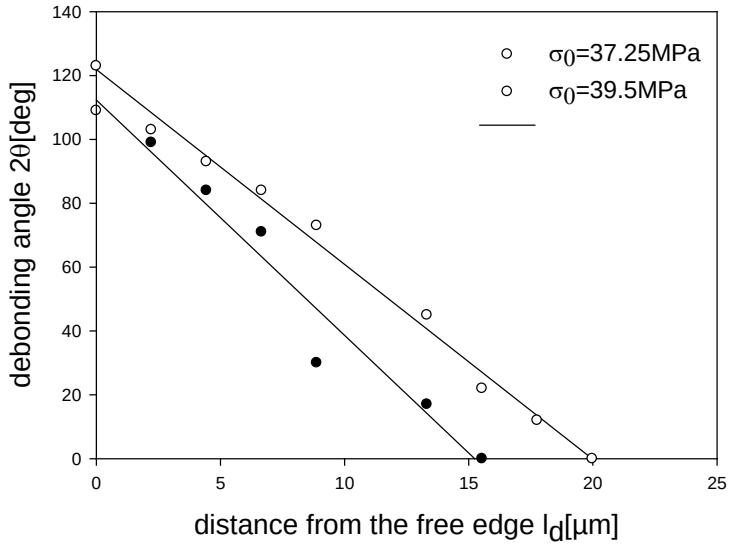

Figure 8. Debonding angle along the fibre as a function of distance from the free surface for two load levels.

4 Conclusions

A micromechanical test specimen and test methodology have been developed for in situ 2D and 3D observations of interfacial fibre/matrix debonding under transverse loading. Interfacial damage initiation and propagation were documented.
2D data aquired by means of in situ observations obtained during tests carried out in the SEM chamber allowed for studying interfacial damage initiation and propagation on free surface with high resoultion. The interfacial damage sequence observed in the SEM is in good agreement with numerical results presented elsewhere [2]. In the nearest future, the Digital Image Correlation technique will be implemented during in situ SEM experiments in order to improve data resolution.
However, 3D experiments carried out using high resolution X-ray microtomograhy indicate the importance of 3D observations of interface behavior. Although the resolution of tomography data is slightly lower than data obtained by SEM, it was possible to investigate the interfacial behavior in 3D during loading. It has been showed that damage mechanisms observed on the free surface are not necessarily representative of what occurs inside the material.
Data aquired in this study will be used for e.g. experimental determination of the critical energy release rate (fracture energy) as a function of mode mixity, which is required imput for micromechanical modelling, validation of crack kinking criteria, etc.

Acknowledgemnts

We would like to thank to Research Technicians: Jonas Heininge, Erik Vogeley, Christian H. Madsen, Jens Olsson, Steen J. Bang for assistance with specimen preparation, loading device design and test set-ups for the experiments. The Danish Research Council is acknowledged for travel support (via Danscatt). The Swiss Light Source is thanked for provision of beamtime. The authors kindly acknowledge the support from the Danish Agency for Science, Technology and Innovation.

References

[1] Zhandarov S., Maeder E. Characterization of fibre/matrix interface strength: applicability of different tests, approaches and parameters. *Composites Science and Technology*, **65**, pp. 149-160 (2005).
[2] Paris F., Correa E., Mantic V. Kinking of Transversal Interface Cracks Between Fiber and Matrix. *Jurnal of Applied Mechanics*, **74**, pp. 703-716 (2007).
[3] Larson B.K., Drzal L.T. Glass fibre sizing/matrix interphase formation in liquid composite moulding: effects on fibre/matrix adhesion and mechanical properties. *Composites*, **25**, pp.711-721 (1994).
[4] Feih S., Wonsyld K., Minzari D., Westermann P., Lilholt H. Testing Procedure for the single fiber fragmentation test. Risø Report, Roskilde, 2004.
[5] Sørensen F.B., Horsewell A.,Skov-Hansen P. In- situ observation of crack formation Bi-2223 HTS tapes. *Physica C: Superconductivity*, **372-376**, pp. 1032-1035 (2002).
[6] Wright P., Moffat A., Sinclair I., Spearing M.S. High resolution tomographic imaging and modeling of notch tip damage in laminated composite. *Composite Science and technology*,**70**, pp. 1444-1452 (2010).