APPLICATION OF X-RAY MICROTOMOGRAPHY TO THE STUDY OF POLYMER COMPOSITES

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SUMMARY: The ability to make precise X-ray attenuation measurements on very small volume elements is a developing technology. This technology is known as X-ray microtomography to delineate the method as a form of X-ray microscopy that uses tomographic reconstruction techniques to build three-dimensional images of microstructure. Since the morphological data of a microstructure are usually collected from plane cross sections taken through an opaque material then this necessary step results in a great loss of information regarding spatial features of the dispersion. In order to overcome this difficulty X-ray microtomography is applied to extract the 3-D information that may be used for micromechanical modelling purposes.

KEYWORDS: X-ray microtomography, serial sections, image reconstruction, microstructure, X-ray projections.

INTRODUCTION

The physical properties of a material are strongly influenced by the microstructure which is designed during processing. Amount of constituent elements forming the material is in many cases known beforehand as for example the volume fraction of reinforcing phase in composite materials. However, the final architecture of a microstructure is controlled only to a limited extent, and on the microscale the geometrical arrangement of second-phase inclusions is a result of complex and interacting processing micromechanisms rather than a design variable. This seems to create an obstacle in modelling the relation between microstructure and overall material properties.

In order to overcome this difficulty it is customarily assumed that the geometrical features of a microstructure are randomly distributed without any precise explanation of what randomness means and how it can be quantified. On the other extreme lies the assumption that the dispersion of fillers is regular. It significantly simplifies calculations, especially with the finite element method, and provides satisfactory results as far as highly nonlinear phenomena are not concerned. For strongly localized effects such as initiation of microcracks and plastic zones which subsequently propagate at different scale lengths, the regularity assumption may lead to seriously erroneous results. Therefore, it is important to describe dispersion characteristics of the microstructure in an unambiguous way by quantitative factors which eventually could be related to physical properties of the material [1-3].

Developments of X-ray microscopy and computer tomography provide a new tool for the assessment of materials morphology. X-ray microscopy is a relatively new technique that has not been applied to any significant extent in materials science [4-7]. Most X-ray microscope development has so far been made using large synchrotron sources. This has limited X-ray microscopy to a research tool available only at the major synchrotron facilities. The use of X-ray tubes with a very small focus and an energy in the order of 20-100 keV together with a very sensitive recording devices enable the design of a bench-top X-ray microscope with a spatial resolution less than 8 μm [8]. A common problem in morphological analysis of non-homogeneous materials is that three-dimensional information of microstructure is required, but its images are two-dimensional. Monitoring materials´ microstructure using X-ray micro-tomography allows us to begin to bridge this gap since a three-dimensional image of the specimen can be reconstructed from non-destructive, serial sections and can be processed to show and measure three-dimensional features.

The main objective of the present study is to establish that X-ray microtomography is a measurement technique that is quite appropriate for the qualitative and quantitative analysis of microstructures in polymer based composite materials.

X-RAY MICROTOMOGRAPHY TECHNIQUE

In X-ray microtomography, the object is rotated so as to obtain radiographic projections from different viewing angles. The projections are the measured values of the overall attenuation that X-rays undergo when they travel through the object, Fig. 1. During a radiography the rotation of the object is carefully controlled with a minimal rotation step of 0.45^o. The object can be moved up and down depending on the axial position of the volume element to be irradiated. An image magnification is achieved by moving the object horizontally between the source-detector set. The X-ray microtomographic scanner used is a commercially available high-resolution system Skyscan 1072.

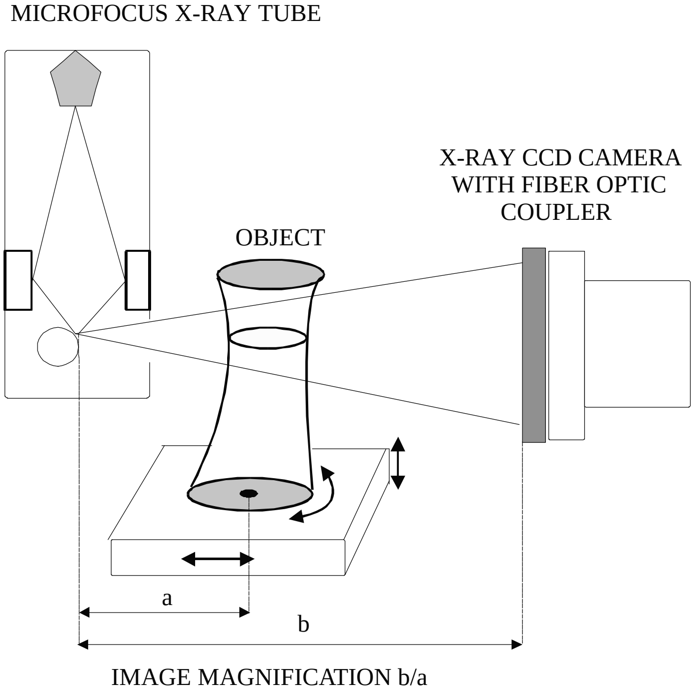


Fig. 1: Schematic diagram of the experimental equipment.

The attenuation of each ray in a beam result from interactions between the radiation used for imaging and the substance of which the object is composed. X-rays passing through material are absorbed according to a linear attenuation coefficient that has some spatial variation depending on the local composition and the density. These differences in the linear attenuation coefficient provide the contrast necessary to form an image. In an idealised case of parallel X-ray illumination a profile of the attenuation through the sample is obtained, Fig. 2. Practically, the attenuation measurement is averaged over a finite-sized volume element in the sample since the detector has finite spatial resolution to discriminate between closely spaced ray paths.

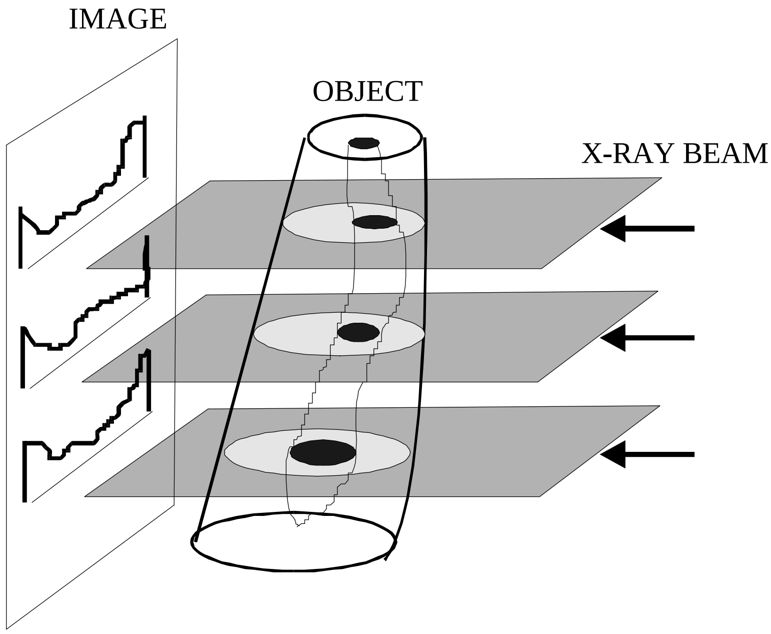


Fig. 2: A set of projection lines through an object.

By rotating the sample in discrete angular increments through 180^o, sufficient data are obtained from the projection images to reconstruct slices of the three-dimensional object. A reconstruction procedure is based on the backprojection principle. The attenuation recorded in each projection is due to the structure of the object along the individual lines. It is not possible to know from one projection where along the line the attenuation occurs. However, it is possible to distribute the measured attenuation evenly along the line. If it is done along projections from several views, the superposition of the attenuation values should correspond to the features present in the structure.

Figure 3 illustrates this result for the slice through a cylindrical object. Data from several views overlap to delineate the cylinder's slice in the reconstruction image.

There are many factors influencing the precision and accuracy of any density gradient quantification. Image quality depends on real density variation within the object, and the energy of the X-rays has to be chosen so that the differences in resulting linear attenuation coefficients between the contrasting detail and surrounding material increase the contrast more than it increases the image noise. Control parameters such as X-ray tube potential and current, filter material and thickness and exposure time all affect the final image quality. Also, the reconstruction parameters are very important for the image quality.

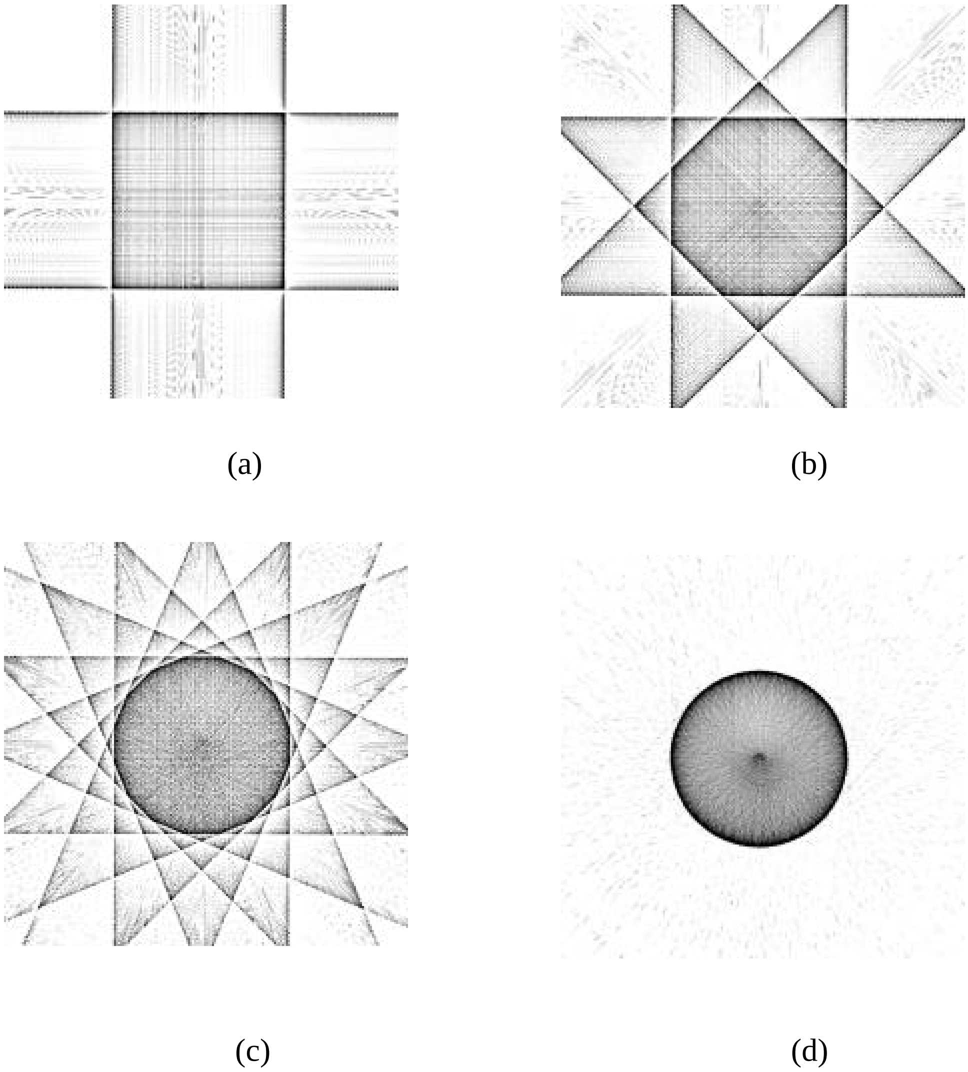


Fig. 3: Backprojection procedure: a) 2 views, b) 4 views, c) 8 views, d) 200 views.

APPLICATION EXAMPLES

A problem of reconstruction of spherical particle sizes from profile sizes has been frequently treated in the literature since mid twenties [9]. The concept of an inclusion size is inherently connected to the notion of shape. The concept of size becomes critical if one wants to compare inclusions of unequal shape. In this situation, statement defining a size of one type of shape not necessarily holds true for another shape of inclusions. In any case, the reconstruction technique may be used to overcome these difficulties. The radiographic view and consecutive reconstructed sections through the compound of glass particles are shown in Fig. 4. The glass spheres were embedded in an epoxy matrix. The mean diameter of the spheres was 75 μm, and the distance between slices is equal to 25 μm. With an appropriate 3D algorithm which creates spatial visualisation from reconstructed microtomographic images it is possible to measure such microstructural features as distances, particles' volume and particle numbers per volume.

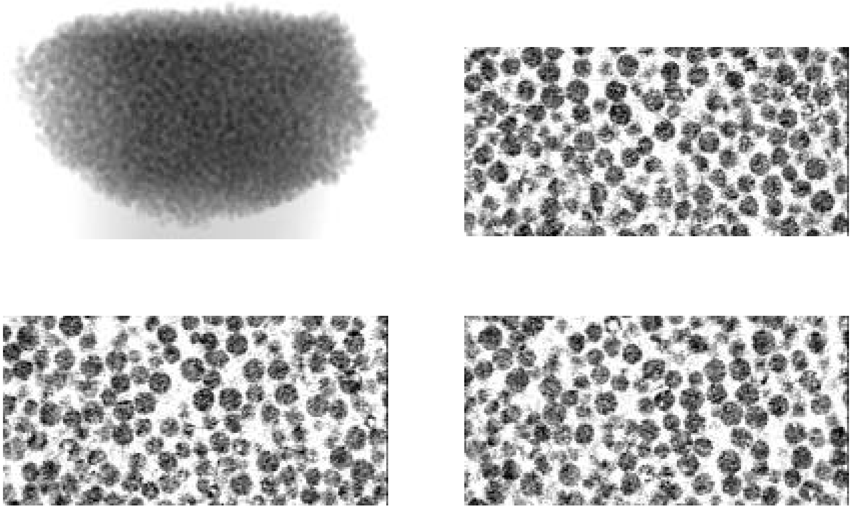


Fig. 4: Glass spheres embedded in an epoxy matrix.

The short range interactions between microstructural entities play a dominant role in non-homogeneous local variations of field quantities giving rise to potential sites of a microfailure. The interaction effects produced by fibres or microcracks are highly sensitive to their exact positions. The fracture behaviour is highly non-linear and is therefore far more sensitive to local variations of the microstructure than any other mechanical phenomena. Local stress variations at the interfaces between the matrix material and the fibres are strongly influenced by the distribution pattern of fibres [10]. In unidirectional composites with continuous fibres creation of matrix rich areas and clustering of fibres may lead to significant magnification of the local stress field triggering an early microfailure. Thus an assessment of fibres dispersion may help to estimate a load bearing capacity of the material [2].

Figure 5 presents one recostructed cross section through the unidirectional glass fibre epoxy composite. This nondestructive image allows to determine the centre position of all fibres to characterize a dispersion pattern by quantitative term, simultaneously giving a visual impression about the homogeneity of fibres dispersion.

Fig. 5: Reconstructed transverse section of unidirectional composite.

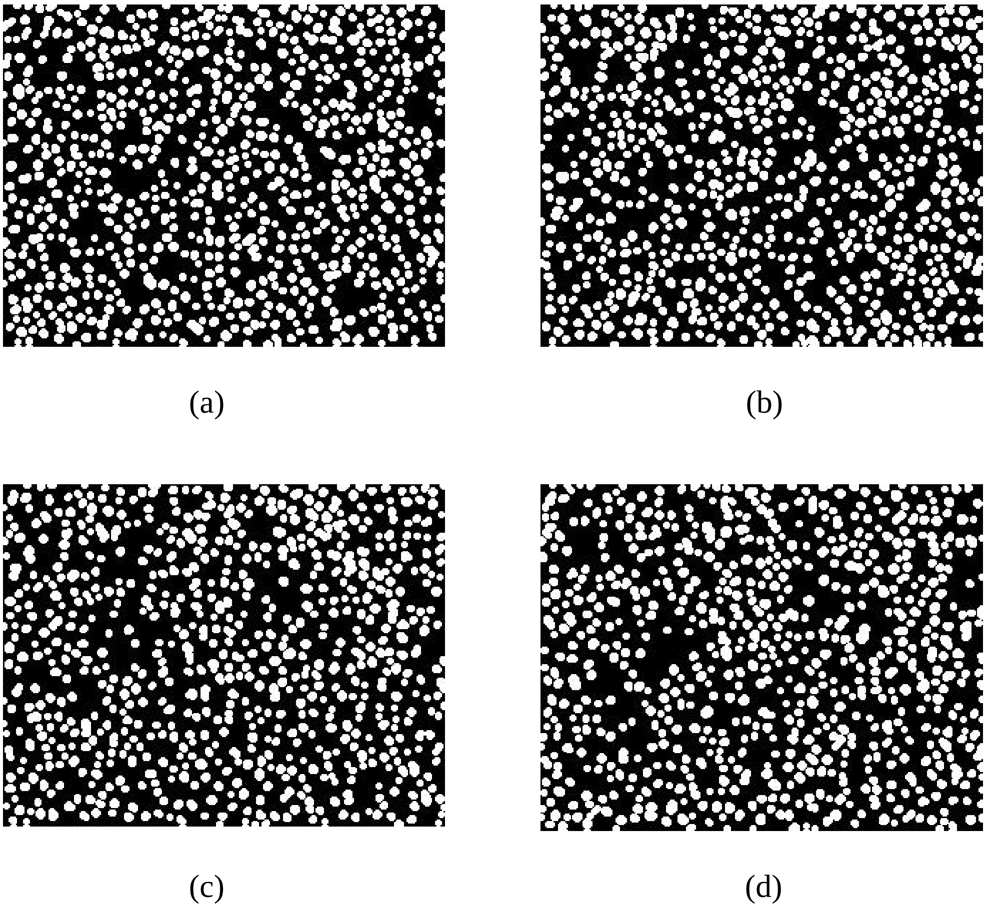


Fig. 6: Consecutive cross sections: a) reference cross section, b) 71 µm apart, c) 153 µm apart, d) 235 µm apart.

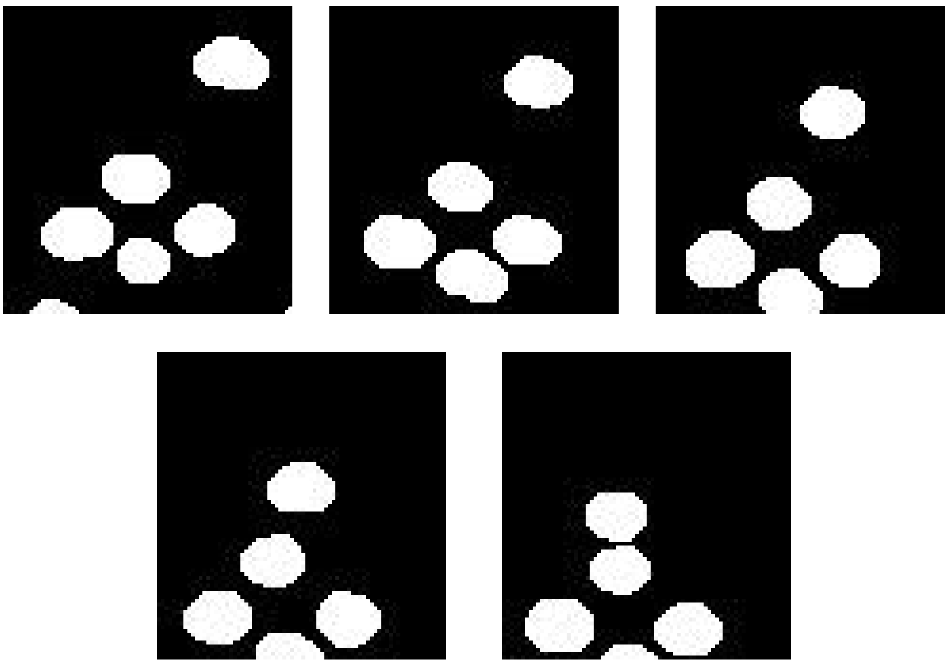
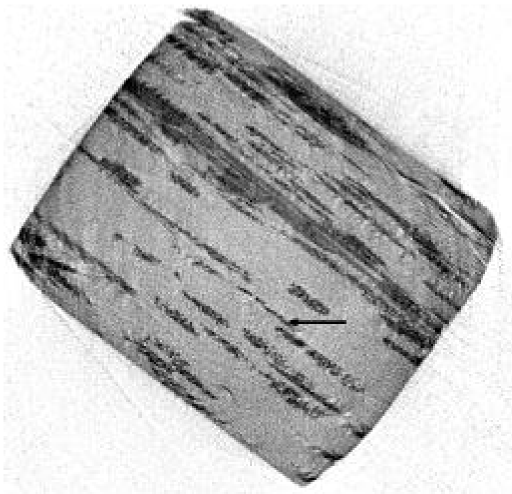


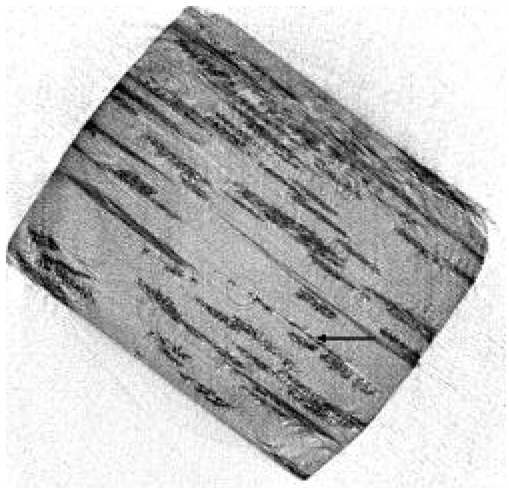
Fig. 7: Enlarged images of selected fibres indicating that fibres are not parallel.

A factor decreasing the precision and accuracy of the fibres` image is statistical noise which cannot be eliminated from microtomographic scans. However, the post-reconstruction filtering can to a large extent eliminate its influence. This is illustrated in Fig. 6 where a central part of the image from Fig. 5 has been subjected to different image transformations together with the associated images from parallel serial slices. In the post-reconstraction filtering procedure a major part has been played by a proper selection of morphological filters, image enhancement and thresholding. The nondestructive slices taken at relatively small intervals of approximately 80 μm clearly indicate that the fibres are not parallel. That is to say, that the dispersion characteristics of fibre centres vary with the position of the cross section. This effect is detailed in Fig. 7 where a few fibres have been enlarged to show changing configuration as one goes with 20 μm steps from the one section to another. Presented images may be used to disclose a fibre waviness, a geometrical factor of the reinforcing fibres which controls compressive properties of the composite.

Detection of voids and microcracks induced either during the processing or under the load is



(a)



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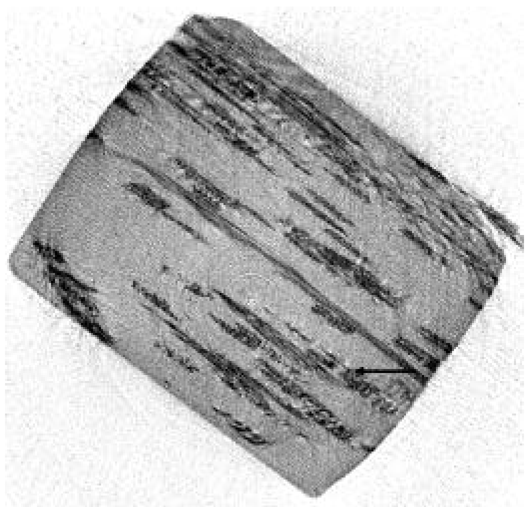

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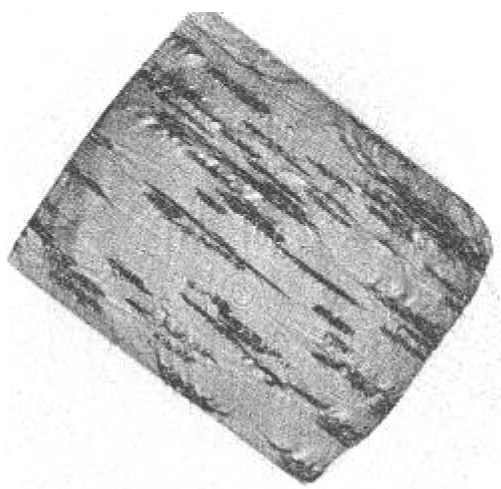

(d)

Fig. 8: Microcrack detected in glass mat polyester composite.

an another area where the microtomographic scans can provide an information not available by other means. An example of monitoring internal microcracks is shown in Fig. 8. The material was glass mat polyester and the glass mat layers are easily visible on reconstructed cross sections. The sections were taken at the distance of 100 μm. The arrows indicate the position of the microcrack which traverses the sections toward the sample boundary, Fig. 8c. The microcrack terminates there since it is not detected on the sebsequent cross section.

Textile structural composites represent an important class of materials for structural applications. In particular, the textile composite preforms technology opens a new opportunity for designing composite materials and structural components in a unified way. A textile preform architecture can vary from a simple plate to a complex three-dimentional shape. Simultaneously, the fabric structure may be very complicated including different woven, knitted or braided structures. Three-dimensional configuration of textile unit cells makes them unsuitable for any observation technique that needs physical sectioning such as the optical and scanning electron microscopy. Recostruction of nondestructive serial sections and eventual three-dimensional visualisation seems to be the only rational solution for microstructure characterization of these materials. Figure 9 shows reconstructed images of glass fibre plane weave vinylester composite. The sections are 224 μm apart showing how the individual yarns that create the plane weave structure, change their thickness and shape. The X-ray microtomographic reconstruction technique might be able to visualise and measure geometrical parameters of the complicated unit cell architecture. These measurements may be crucial for the description of a resin transfer moulding technique.

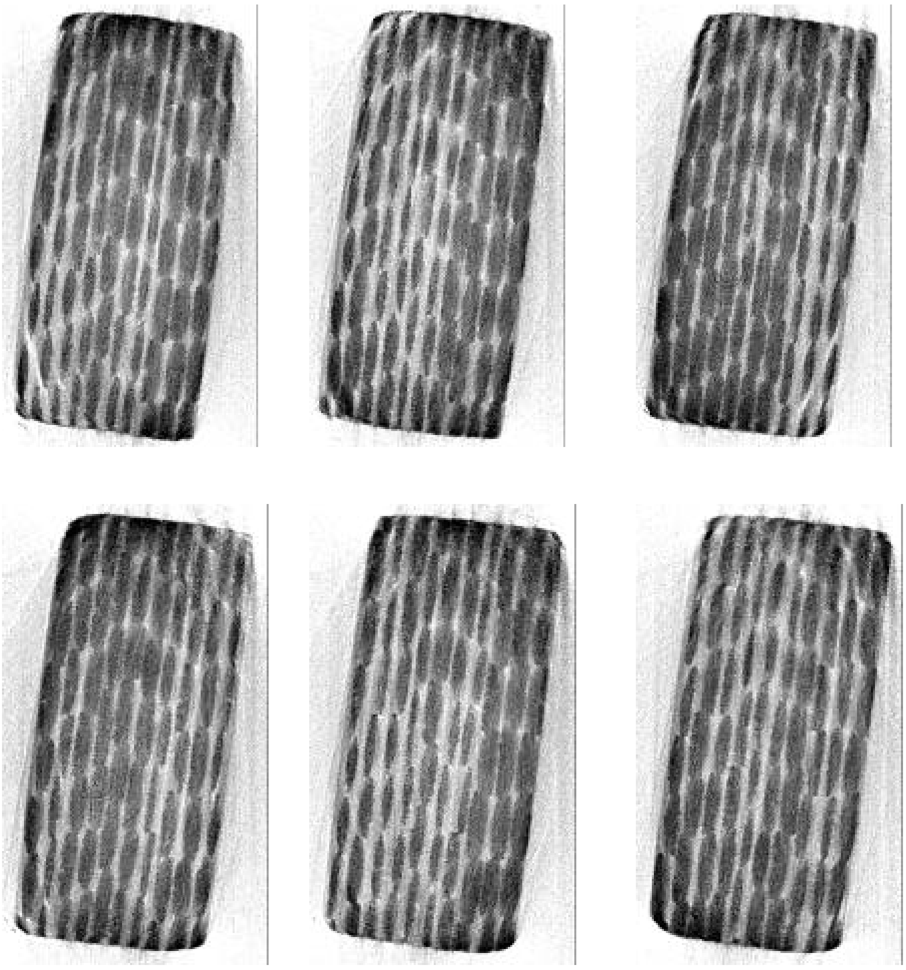

Fig. 9: Reconstructed cross sections of glass plane weave vinylester composite.

CONCLUSIONS

The application of the X-ray microtomographic technique to characterize advanced composite materials has been presented. With commercially available scanners the smallest detail that migh be detected is close to 2 μm. The most important aspect of X-ray microtomography is that the method is nondestructive. Furthermore, it is possible to characterize specimens in situ and observe how the microstructure evolves under the influence of mechanical loading and/or aggressive environments. This technique can make a valuable contribution to studies that require a three-dimensional understanding of non-homogeneous microstructures.

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