ULTRA-FAST TIME-LAPSE SYNCHROTRON RADIOGRAPHIC IMAGING OF COMPRESSIVE FAILURE IN CFRP

Y. Wang[1]*, S. C. Garcea[1], T. Lowe[1], E. Maire[2], C. Soutis[3] and P. J. Withers[1]

[1]Henry Moseley X-ray Imaging Facility, School of Materials, University of Manchester, Manchester M13 9PL, United Kingdom
Email: ying.wang-4@manchester.ac.uk, Web Page: http://www.mxif.manchester.ac.uk/
[2]INSA-Lyon, 69621 Villerbanne Cédex, France
[3]Northwest Composites Centre, School of Materials, University of Manchester, Manchester M13 9PL, United Kingdom

Keywords: Kink band, Ultra-fast X-ray computed tomography, Compressive failure, CFRP

Abstract
In this study, the compressive failure of unidirectional (UD) carbon fibre/epoxy composite rods is studied using time-lapse synchrotron X-ray imaging. Ultra-fast two-dimensional (2D) radiography (0.1 ms per frame) and three-dimensional (3D) X-ray computed tomography (CT) (0.46 s per tomograph) were used to follow the catastrophic damage evolution under axial compression loading. Radiographs demonstrate the sequence of events leading to the formation of multiple kink bands. Fibre buckling occurs in accompany with splitting, and excessive fibre bending results in fibre failure and the formation of kink bands. Multiple splits at maximum fibre bending points are opened with large matrix shear deformation. The initiation and full propagation of a kink band (across the specimen) is found to occur in less than 1.2 ms. Tomographs just before failure reveal that fibre micro-buckling/kinking due to fibre misalignment proves to be the incipient damage mechanism, the extent of which is small until the sudden formation of a kink band.

1. Introduction

Although the first report on the low compressive strength of fibre composites resulting from fibre micro-buckling (kinking) and kink bands appeared in 1960 [1], this subject is still an active field of research in recent years. This is because a full understanding of the damage mechanisms has not been achieved yet due to the sudden and uncontrolled nature of compressive failure and the need to understand the instabilities that give rise to buckling in 3D over time. As carbon fibre reinforced plastics (CFRPs) are being increasingly employed in aero-industries [2], the catastrophic compressive failure of CFRP needs to be better understood to avoid unexpected failure of such structures. In addition, in contrast to tensile failure, compressive failure can be very sensitive to small amounts of fibre misalignment as well as imperfections introduced during manufacturing [3]. Therefore, the aim of this project is to study the damage mechanisms resulting in kink bands in the catastrophic compressive failure in CFRP, which requires advanced damage characterisation capability.

X-ray CT can provide 3D images of the structure of materials across a range of scales. As a non-destructive technique X-ray imaging can be applied repeatedly either continuous streaming or to provide time-lapse sequences. In high-speed *in situ* tomography, the same sample is scanned continuously without interrupting the heat treatment or the mechanical loading. In order to obtain an artefact-free tomographic reconstruction the sample should not change over the time needed to collect all the radiographs (projections) to reconstruct the 3D volume. If this is not satisfied, motion artefacts can occur, making it difficult to perform quantitative analysis of the data. There are two methods to

Excerpt from ISBN 978-3-00-053387-7

ensure the image quality. The first is to slow down [4] or interrupt the deformation of the sample and the second is to acquire the tomographic dataset faster [5]. Generally the higher the resolution the slower the fastest acquisition rate possible and the current state of the art is given in [6]. Frame rates as fast as 10,000 frames per second can be achieved by ultra-fast synchrotron radioscopic imaging at TOMCAT beamline based in Swiss Light Source (SLS). Although several ultra-fast imaging experiments have been explored so far [7] until now no one has started to look at the fast imaging of the failure of CFRPs. In view of the very rapid rate of kink-band formation fast X-ray imaging may be able to explore the kink band formation in 2D over time.

In this project, ultra-fast X-ray imaging is used to capture the catastrophic compressive failure of UD CFRP sample with the aim of better understanding the damage mechanisms associated with kink-band formation.

2. Materials and Methods

2.1. Fabrication of UD composite

A small-scale resin infusion (SSRI) method, as reported in [8], was used here to fabricate UD composite rods consisting of Torayca T700 carbon fibre yarns and Huntsman Araldite LY 564/XB 3486 epoxy resin. Figure 1 shows the experimental set-up of the SSRI method. After infusion, the composite was cured at 80 ºC for eight hours.

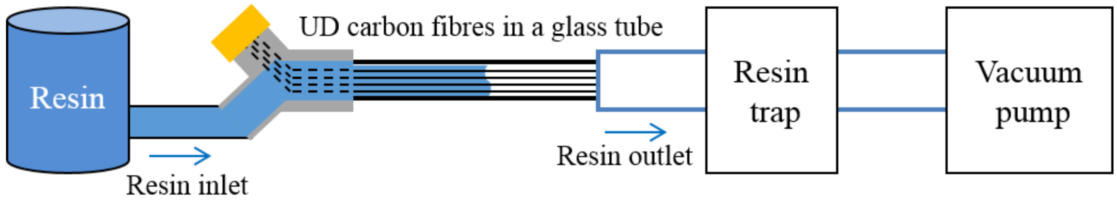

Figure 1. Schematic of the SSRI manufacturing method developed to fabricate UD rods. [8]

Following our previous post-failure X-ray tomography study [8], specimens with a groove in the centre of the reduced gauge section were used for the tests here. The gauge section was ground down to a diameter of around 1.5 mm over 3 mm in length, with smooth radii at the transition regions. A groove around the circumference was made using a razor blade giving a notch depth around 100-200 µm. The two ends of the specimen were then glued with an epoxy adhesive (Araldite® Strength in Bonding 2000+) into chamfered steel end caps having small holes drilled to remove any surplus epoxy.

2.2. In-situ compression test

Specimens were end-loaded in a tension-compression *in situ* loading rig developed at INSA-Lyon, which could be accommodated on the ultra-fast imaging TOMCAT beamline (see Figure 2). The compression tests were conducted under displacement control at the rate of 1 µm/s.

Excerpt from ISBN 978-3-00-053387-7

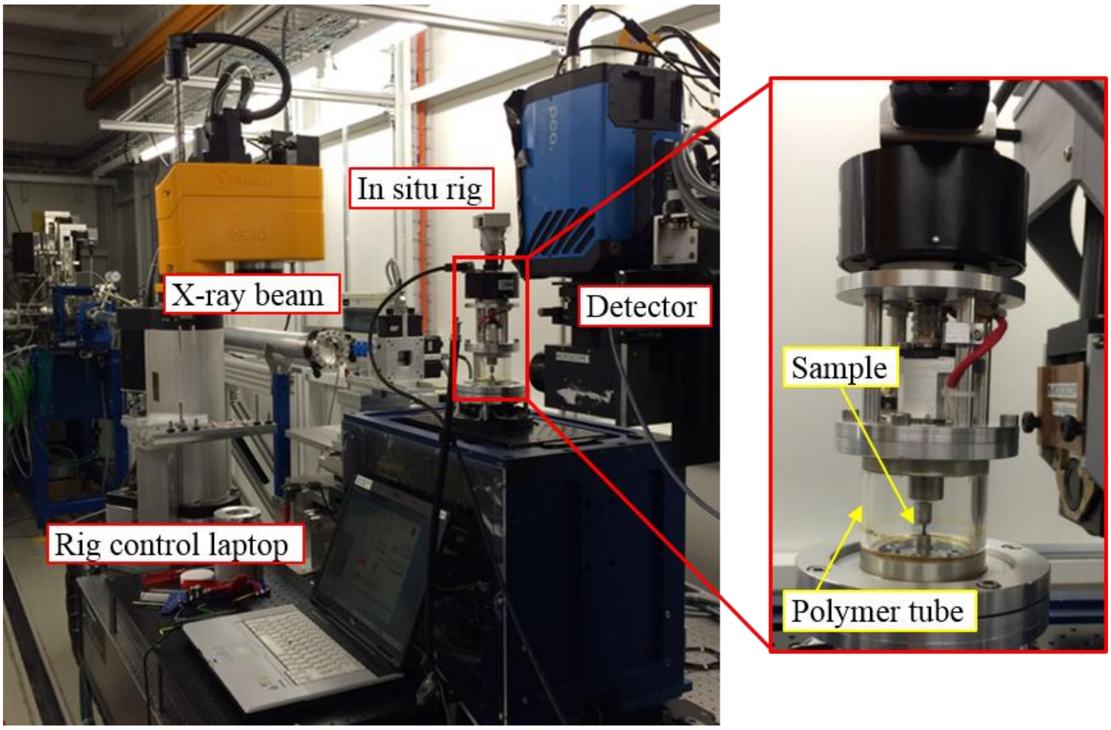

Figure 2. *In situ* experiment set-up at TOMCAT beamline.

2.3. Ultra-fast synchrotron X-ray imaging

The continuous *in situ* X-ray CT imaging was conducted at the TOMCAT synchrotron beamline based at SLS, where ultra-fast X-ray imaging could be performed at relatively high resolution. Figure 2 shows the in situ experiment set-up at synchrotron beamline with sample mounted in the *in situ* loading rig. For the sample presented in this paper, an initial static scan at higher resolution (voxel size 1.1 µm) and lower speed was performed before loading, followed by interrupted fast static scans (voxel size 3 µm) at relatively low load levels and continuous dynamic scans (voxel size 3 µm) while approaching the expected failure load. Table 1 summarises the scanning parameters. The datasets were reconstructed using the Pagnin algorithm at TOMCAT and analysed in Avizo 8.0 software (FEI Visualisation Sciences Group).

Table 1. Scanning parameters for synchrotron radiation experiments at the TOMCAT beamline.

Data acquisition rate	1 dataset/second	22 datasets/second
X-ray beam	Monochromatic	White
Mean beam energy (keV)	20	15.5
Exposure time (ms)/projection	2	0.1
Number of projections/dataset	501	461
Voxel size (µm)	1.1	3

Excerpt from ISBN 978-3-00-053387-7

3. Results and Discussion

3.1. Radiographic imaging of the sequence of events leading to failure

In our previous studies, it was found that in un-notched samples kink bands tend to develop near either of the two transient regions at the ends of the waisted gauge section, but the field of view (FOV) could not cover the whole gauge length if reasonable resolution is to be assured. Thus a circular notch around the circumference was added to localise kink band formation within the FOV.

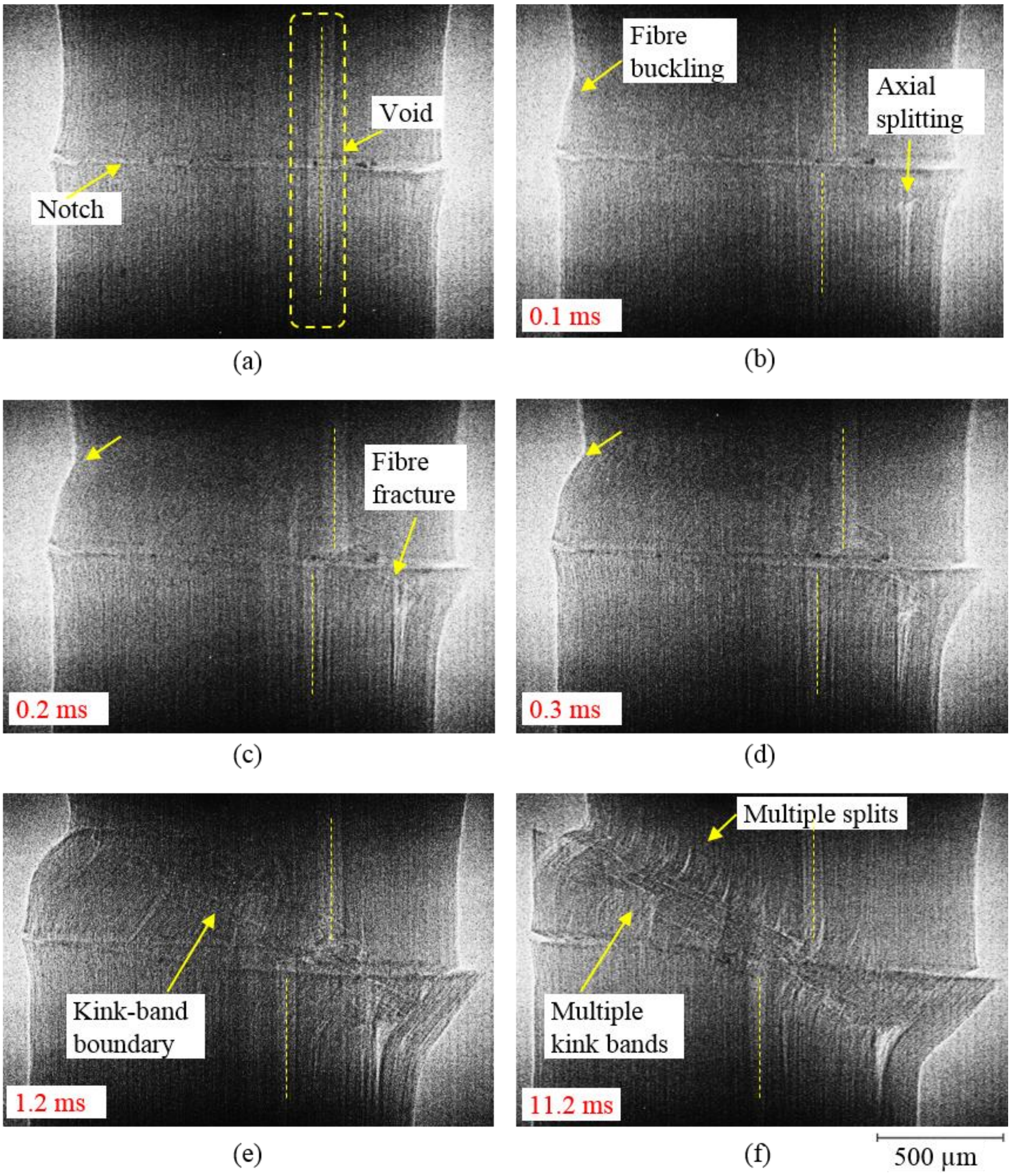

Figure 3. Radiographs showing the evolution of damage leading to kink failure.

The development of kink bands in UD fibre reinforced composite is a rapid catastrophic process, which is challenging to be observed by X-ray imaging. Due to limited beam energy, micron resolution scans on lab-based CT scanners take ～3 hours each in order to achieve reasonable image quality [6]. Even in the

Excerpt from ISBN 978-3-00-053387-7

continuous datasets obtained at TOMCAT using a synchrotron beam to acquire a tomograph at 1 dataset per second, the initiation and propagation of kink bands were observed to occur within the time to acquire just one projection (exposure time 2 ms).

With the ultra-fast imaging capability at TOMCAT, the structural and fibre architectural evolution of kink bands was captured in radiographs of the CT dataset at failure. Figure 3 shows the radiographs taken at 10,000 frames per second (exposure time 0.1 ms), showing the sequence of events leading to final failure. As the 2D radiographs were captured while the specimen was rotating to get a 3D CT dataset, the angle between each successive frame was 0.78º. In Figure 3(a) the specimen is intact with an elongated void lying along the fibre direction (manufacturing defect), while in the next radiograph (b) fibre buckling has occurred accompanied by the opening of an axial splitting. The curvature of the buckling fibres then increased along with the broadening of the split, as shown in (c). In (d) and (e) the buckled fibres broke from the split and formed the boundaries of the kink band across the sample. Sometime later (～10 ms) multiple kink bands formed with the opening up of multiple splits due to the buckling of fibres at the boundaries (maximum fibre bending points). Multiple splits at maximum fibre buckling points are opened while large matrix shear deformation occurred. The initiation and full propagation of a kink band (across the specimen) were found to occur in less than 1.2 ms.

The damage mechanisms can be roughly proposed based on observations in 2D radiographs. The buckling of fibres and the splits were the incipient damage modes that occurred at the initial stage of damage in less than 0.2 ms. Excessive rotation of fibres resulted in fibre fracture, which then propagated to form the boundary of the kink band in less than 1 ms. The formation of multiple kink bands is not accidental and has been observed in a number of studies [9, 10]. The progressive adding of narrow kink bands could not be observed from the radiographs here. This might be attributed to the poor image quality of the radiographs for the observation of fibre breakage within the sample or the sudden nature of multiple fibre breaks within the damage zone.

3.2. Tomographic imaging of the effect of fibre misalignment on compressive failure

Some degree of fibre misalignment is inevitable in the manufacturing of long fibre reinforced composite materials, especially during the manual handling of fibres. Figure 4 shows virtual CT longitudinal sections of the same location in the sample taken from different CT datasets. Fibre micro-buckling/kinking of misaligned fibres can be observed prior to the sudden matrix shear deformation and the formation of a kink zone across the sample with no fibre fractures at boundaries. This feature is localised in a region near the sample surface, while in other parts of the sample fibre kinking cannot be observed prior to the formation multiple kink bands (boundaries defined by fibre breaks) as demonstrated in the radiograph image of Figure 3(f). The wavelength of fibre micro-buckling (see Figure 4(c)) is small compared to the final fibre wave pattern at failure (see Figure 4(d)).

Excerpt from ISBN 978-3-00-053387-7

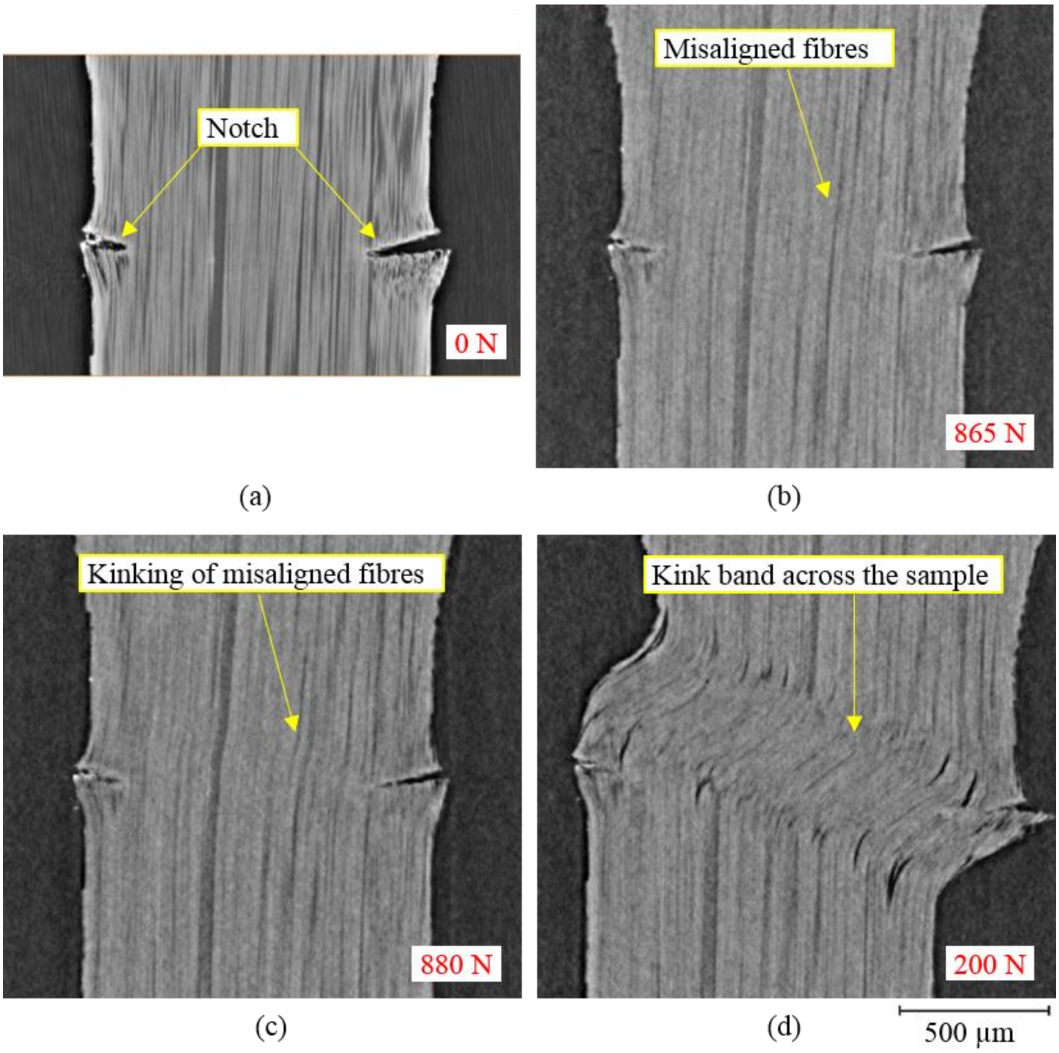

Figure 4. **V**irtual X-ray CT slice images of the same position in the sample (a) before loading, (b) and (c) before failure and (d) after failure showing the initiation of fibre micro-buckling/kinking from misaligned fibres.

4. Conclusions

In this study, the introduction of a circular notch at the centre of the waisted gauge section has been used to successfully constrain the location of a kink band in the vicinity of the notched region. The rapid catastrophic kink-band formation has been captured by ultra-fast X-ray imaging. The initiation and propagation of kink bands in UD fibre composite under axial compressive loading occur in a very short time (less than 1.2 ms). The general sequence of events leading to failure can be drawn from radiographs: (1) fibre buckling and splits, (2) fibre breakage forming kink-band boundary and (3) multiple kink bands. Fibre micro-buckling/kinking of misaligned fibres has been observed preceding the formation of kink bands.

Acknowledgments

The authors would like to thank Swiss Light Source for the beamtime and kind help from the TOMCAT beamline scientist Rajmund Mokso. The authors would like to acknowledge EPSRC funding for the

Henry Moseley X-ray Imaging Facility under grants EP/M010619, EP/K004530, EP/F007906, EP/F028431

References

[1] N. F. Dow and I. J. Gruntfest. Determination of most needed potentially possible improvements in materials for ballistic and space vehicles. *TJS R 60 SD*, 389, 1960.
[2] C. Soutis. Fibre reinforced composites in aircraft construction. *Progress in Aerospace Sciences*, 41(2):143-151, 2005.
[3] M. R. Wisnom and J. Atkinson. Fibre waviness generation and measurement and its effect on compressive strength. *Journal of reinforced plastics and composites*, 19(2):96-110, 2000.
[4] Y. Wang, C. Soutis, and P. J. Withers. Time-lapse X-ray microtomographic imaging of compressive failure in carbon fibre-epoxy composites. *Proceedings of the 20th International Conference on Composite Materials ICCM-20, Copenhagen, Denmark*, July 19-24 2015.
[5] R. Mokso, F. Marone, D. Haberthür, J. C. Schittny, G. Mikuljan, A. Isenegger, and M. Stampanoni. Following dynamic processes by X‐ray tomographic microscopy with sub-second temporal resolution. *Proceedings of the 10th International Conference on X-ray Microscopy, Chicago, USA*, August 15-20 2010.
[6] E. Maire and P. J. Withers. Quantitative X-ray tomography. *International materials reviews*, 59(1):1-43, 2014.
[7] J. Adrien, E. Maire, N. Gimenez and V. Sauvantmoynot. Experimental study of the compression behaviour of syntactic foams by in situ X-ray tomography. *Acta Materialia*, 55(5):1667-1679, 2007.
[8] Y. Wang, C. Soutis, and P. J. Withers. X-ray microtomographic imaging of kink bands in carbon fiber-epoxy composites. *Proceedings of the 16th European Conference on Composite Materials, Seville, Spain*, June 22-26 2014.
[9] S. Pimenta, R. Gutkin, S. Pinho, and P. Robinson. A micromechanical model for kink-band formation: Part I—Experimental study and numerical modelling. *Composites Science and Technology*, 69(7):948-955, 2009.
[10] J. Hapke, F. Gehrig, N. Huber, K. Schulte, and E. T. Lilleodden. Compressive failure of UD-CFRP containing void defects: In situ SEM microanalysis. *Composites Science and Technology*, 71(9):1242-1249, 2011.

Excerpt from ISBN 978-3-00-053387-7