The micro-mechanics of strength, durability and damage tolerance in composites: new insights from high resolution computed tomography

S Mark Spearing and Ian Sinclair
Faculty of Engineering and the Environment, University of Southampton, SO17 1BJ, UK

Abstract. Recent work, led by the authors, on impact damage resistance, particle toughening and tensile fibre failure is reviewed in order to illustrate the use of high-resolution X-ray tomography to observe and quantify damage mechanisms in carbon fibre composite laminates. Using synchrotron and micro-focus X-ray sources resolutions of less than 1 µm have been routinely achieved. This enables individual broken fibres and the micromechanisms of particle toughening to be observed and quantified. The data for fibre failure, cluster formation and overall tensile strength are compared with model predictions. This allows strategies for future model development to be identified. The overall implications for using such high-resolution 3-D measurements to inform a “data-rich mechanics” approach to materials evaluation and modeling is discussed.

1. Introduction

It has been more than fifty years since the development of carbon fibres and the introduction of high performance composite materials [1]. Material properties have developed remarkably over that period, with a more than doubling in each of the useful tensile strength, strain to failure and functional impact damage tolerance since the first generation of carbon fibre composite materials. Perhaps surprisingly this has been achieved without a strong physically-based understanding of the underlying mechanisms and also an ability to predict the resultant structural response, particularly with regard to failure, damage resistance, damage tolerance and durability. This is exemplified by the work done over the past twenty years in a series of “World wide failure exercises” [2] which have been conducted in order to assess the predictive capability for the failure of composite materials. The results have indicated that the overall modeling capability continues to relatively poor, at least compared to that for the metallic materials which composites are often used to replace. The difficulty in predicting failure is that composites typically fail by multiple interacting damage mechanisms, which occur in three dimensions. Furthermore it is usually difficult to infer the underlying damage mechanisms solely by inspection of the surface of the structure or test coupon. The response of the modeling and design communities has been to rely on extensive testing for any safety critical parts in order to supplement, or in some case, completely replace any reliance on modeling. These issues are further compounded when issues of durability are to be addressed, and where comprehensive test programmes are particularly time consuming and expensive.

Significant work has been conducted over the past fifty years to understand the impact damage tolerance [e.g 3,4], role of particle toughening e.g [5,6] and tensile fibre failure e.g [7,8], however there has been a lack of detailed experimental data to allow direct validation of the assumptions and
predictions of these models. In order to address this deficiency we have been conducting research over the past ten years into the use of high-resolution computed tomography (HRCT) to elucidate damage mechanisms in composite materials [9-20]. This is motivated by the strong belief that in order to make real progress in developing superior materials and to be able to predict the material and structural response it is essential to understand the underlying mechanisms and to incorporate these into predictive models. In particular we have found the ability to identify features in composites under load and to measure their relative displacements in order to infer strains or to calculate crack opening and shear displacements is a very powerful means by which to interrogate and validate mechanics models for composite failure processes. The use of HRCT, in combination with other techniques allows a few careful experiments to yield large, very valuable data sets which can be used to inform, validate and challenge modeling approaches. This approach has the potential to change radically the approach to composite modeling and mechanical testing. This paper provides an overview of our current and recent work in this area and an assessment of the potential for HRCT to contribute further to the understanding and modeling of composite failure.

2. Testing methods
The details of the testing and imaging procedures are described more fully elsewhere [9, 10, 17, 19]. High resolution tomographic imaging was conducted on beam line ID19 at the European Synchrotron Radiation Facility (ESRF) in Grenoble and the TOMCAT beamline at the Swiss Light Source in Villigen, Switzerland. In these facilities resolutions down to 0.3 µm can be achieved. In all cases a compromise must be considered between the resolution and field of view. The synchrotrons produce a highly coherent, monochromatic X-ray source, which allows for both absorption and phase contrast imaging, which is particularly important for allowing crack surfaces and damage feature to be identified. Lower resolution imaging was conducted using the microfocus CT beamlines available at the µVis facility at Southampton.

For in situ tensile loading one of two fixtures was used, and these are described elsewhere [9, 19]. These have load capacities of up to 5kN and are designed to allow 360° X-ray access with isotropic absorption. The fixtures are particularly suited to use in synchrotrons, with their high resolution capabilities, but can be used in conjunction with micro-focus sources. The load frames allow damage development to be tracked as a function of load, as well as allowing damage to be imaged in the loaded and unloaded states, which allows for the separation of the effect of residual stresses and applied loads on crack opening and shear displacements. In situ loading has been performed to generate tensile damage in double edge notched specimens with a total width of 4 mm, with two semi-circular notches of nominally 1mm radius/depth. This leaves a net section between the notches of 2 mm. In other work, wedge-opened double cantilever beam specimens have been used to study mode 1 fracture and end notched flexure tests loaded in three point bending to study mode 2 fracture [17].

3. Results

3.1. Damage tolerance
Quasi-isotropic laminates, approximately 4 mm thick, cut into panels 150 mm x 100 mm were tested under low velocity impact conditions. Compression after impact tests were subsequently conducted. The experimental details are described elsewhere [13-15]. Five material systems were investigated, identified as UT and T1 to T4, with UT referring to an untoughened epoxy matrix and T1 to T4 representing toughened systems with increasing levels of damage resistance. Figure 1 shows the damage area as a function of the incident impact energy. The untoughened material exhibits about three times the damage area as material T4 for a given impact energy. Figure 2 shows two rendered computed tomography images of the damage in the untoughened material and one of the toughened systems. It is clear that the principal effect of toughening is to reduce the extent of delamination under the impact site. There is no noticeable effect on the extent of intralaminar damage.
Figure 1. Impact damage resistance as a function of incident impact energy after [14].

Figure 3 shows higher resolution CT-generated cross sections of interlaminar cracks in each of the materials studied. Analysis of the full three dimensional image file allows the performance of the material to be correlated with aspects of the crack microstructure, such as crack path tortuosity, crack surface roughness, area fraction of bridging ligaments and bridging ligament size. Figure 4 shows the data for bridging ligament interconnectivity (area fraction) and bridging ligament size for the five materials as a function of position, measured from the crack tip. It is clear that the order of toughness of the materials correlates strongly with the bridging ligament interconnectivity close to the crack tip. The evidence from this study indicates that toughening strategies, which promote near-tip ligament formation are likely to have a greater effect on improving impact damage resistance.

3.2. Micro-scale toughening and durability
The observation that bridging ligament formation is important for determining the functional toughness and damage resistance of toughened composite systems motivates a closer look at the micro-mechanisms of toughening. This requires higher resolution imaging, and benefits greatly from in situ loading. SRCT was used to obtain the images shown in Figure 5, which show the behaviour observed in a relatively tough material system. Toughening is achieved by the incorporation of an interply region containing polymer particles. These particles partially debond ahead of the crack tip. This debonding process has the effect of maintaining the crack in the interply region and of promoting the creation of bridging ligaments in the crack wake. Figure 6 shows a delamination propagating at the ply interface, following individual fibre-matrix interfaces. In this case the particles provide no
direct contribution to the toughness of the material, and there is greatly reduced opportunity for the formation of bridging ligaments.

![Figure 2](image.png)

**Figure 2.** A three-dimensional rendering of impact damage in a) an untoughened material and b) a toughened material. The impact energy in b) is twice that in a). Note the reduced extent of delamination (blue) in the toughened material. After [13].

The dependence on bridging ligaments for toughening and damage resistance raises questions as to the effect of fatigue loading on such toughened systems. Work conducted by Garcea et al [18, 19] has shown that the mechanisms of matrix crack advance are microscopically similar in fatigue as for quasi-static loading. Figure 7 shows two SRCT cross-section of the same region of a split emanating from a notch under macroscopic tensile loading, with an increment of 100 cycles between the two images. Overall crack advance occurs by the linking together of discrete debond events ahead of the continuous crack tip, as for the quasi-static case. In addition, ligaments in the rear of the crack tip fail, removing this load transfer mechanism between the crack faces and facilitating further crack advance. In this particular case, fatigue loading, even at quite high loads, is not detrimental to the overall specimen residual strength and the resulting S-N curves are quite shallow or even flat.
Figure 3. CT cross sections of delaminations in the five material systems. Bridging ligaments are visible to varying extents in all five materials (e.g region “v”) after [13].

Figure 4. Data obtained by analysis of CT image volumes for a) bridging ligament interconnectivity and b) bridging ligament size as a function of position behind the crack tip. After [15].
Figure 5. SRCT cross-sections of a delamination in a particle toughened interlayer. The same region is viewed at two different load levels, the crack has grown between the load steps. After [17].

Figure 6. Mode 1 fracture at the interface between the ply and the toughened region after [17].

3.3. Tensile strength and fibre fracture
Fibre fracture is the key failure mode in tensile loading of 0° dominated laminates. Perhaps surprisingly, more than 50 years after the creation of the first continuous carbon fibre composites [1], it is still unclear as to how the tensile strength of individual fibres, in combination with the matrix and fibre-matrix interface, affects the strength of a composite. Figure 8 shows data for composites made with several similar intermediate modulus carbon fibres, combined with epoxy matrices to form composites. Despite very little variation in fibre strength, very significant variation in composite tensile strength results. High-resolution CT imaging combined with in situ loading provides a unique means to observe the development of broken fibres as a function of load. In particular the technique offers the opportunity to correlate broken fibres with microstructural features as well as the tendency for broken fibres to develop as clusters. Figure 9 shows images of clusters of broken fibres in specimens loaded in tension [20]. Using such images, combined with in situ loading capabilities, it is possible to obtain data such as that shown in Figure 10, for the evolution of broken fibres with applied tensile load, for the same set of materials examined in Figure 8. Very wide variation in broken fibre accumulation is apparent between ostensibly quite similar materials systems. In order to understand these variations, with a view to providing guidance for improving material strength, existing models for tensile strength have been applied [21, 22]. In both modeling strategies applied to date, comparisons similar to that shown in Figure 11 are obtained. The prediction of fibre break density and of isolated broken fibres is reasonably accurately modeled. However, the formation of clusters consisting of multiple broken fibres (as shown in figure 8) is not well modeled, as presented in figure 12. It is expected that cluster formation plays a critical role in determining the point of failure, so this suggests that further work is required to develop a capable modeling framework.
Figure 7. Fatigue damage propagation with 100 cycles between image a) and image b), after [19].

Figure 8. Plot of composite tensile strength vs. mean fibre strength for seven material systems all with intermediate modulus fibres but different matrices and fibre/matrix interfaces. After [22].
Figure 9. SRCT images of a) a diffuse cluster of broken fibres and b) a coplanar cluster of broken fibres after [20].

Figure 10. Data obtained via SRCT observations of fibre break density as a function of the nominal fibre stress for the same set of materials examined in Figure 8. After [22].
Figure 11. Comparison of experimental data for fibre break density with applied strain, and a model [22]. A comparison with the Weibull prediction based on individual fibre tests is provided.

Figure 12. Fibre fracture evolution with applied strain compared to model predictions. After [22]
4. Discussion

In the three areas of investigation described in this paper, a number of common themes can be identified. It is clear that the very high resolution imaging capability afforded by computed tomography, particularly when using synchrotron generated radiation, allows for insights to be obtained that were not possible more than a decade ago. As the equipment, the image processing and data handling becomes more routine, the accessibility of these techniques will continue to grow. When applied to composite materials these techniques allow the interaction of the relevant damage mechanisms to be understood in 3-D, which is often of critical importance. There is particular value when looking at the mechanisms affecting toughening, fatigue and damage tolerance, and fibre fracture and tensile strength. These areas have been particularly amenable to investigation due to the match between the relevant length scales of the mechanisms and the resolution and field of view available by high resolution computed tomography. The extension via in situ loading to examine the micro-mechanisms of damage formation, particularly in fatigue offers a high degree of novelty and great potential to advance our understanding in the field. Even a 100 cycles of loading at an intermediate load level results in a few 10’s of µm of crack advance, which would be difficult to detect using conventional techniques such as radiography, ultrasound or visual inspection. However, at SRCT resolution it allows the individual micro-mechanisms to be identified very clearly. This capability also offers the potential to provide a high quality benchmark for structural health monitoring and non-destructive evaluation techniques. Computed tomography is unlikely to be useful for anything but the highest criticality and highest value inspection of manufactured and in service parts, but as a means of calibrating and verifying lower cost techniques to be used in such situations, it has considerable potential.

The most important element of the work described in this paper, and often conducted with collaborators over the past decade is the use of the extensive data files obtained from high resolution computed tomography and other techniques to inform, calibrate and challenge models. Elsewhere this has been termed “Data Rich Mechanics”, the particular example of fibre failure and tensile strength has been highlighted in this work, but other examples have been explored for damage propagation from notches [12, 23]. The ability for high resolution CT, often in combination with in situ loading rigs, to yield large, comprehensive data sets, of damage, displacements and strains is unparalleled. When combined with models there is a real opportunity to develop fundamental, mechanism-based understanding and predictive capabilities. Such models have an important role to play in guiding material development and material and structural design. There is a need and an opportunity to continue to improve the predictive capability for composite materials and to move away from the empirical approaches to material development and structural design, as well as to achieving better material utilization and increased economic and environmental sustainability as a result. In order to achieve this ambition of a data rich mechanics framework to enable an improved model-based predictive capability for composites a number of challenges remain to be overcome. The highest resolution imaging is necessarily restricted to limited fields of view. This results in a challenge of ensuring that observations made at small scales are representative and relevant to those in larger components and structures. Multiscale imaging, using micro-focus sources as well as synchrotron sources has been important in this regard, but there is still considerable progress required. The extraction of damage from data sets is still a largely manual/visual process defined by human operators. There is considerable scope for improved automated feature recognition software to allow automated data reduction and comparison with models. For damage such as fibre failure and impact damage formation, the processes are inherently dynamic. Imaging hardware, data acquisition and image reconstruction software has developed significantly over the past decade to allow for relatively fast scanning, with useful CT images being obtained within durations of 1-10s. This is still many times longer than the timescale for some of the critical damage events, so either further development is needed, or more likely, augmentation with other experimental and modeling techniques. The data sets provided by CT imaging are relatively large; a typical image file is about 4 Gb. This creates significant challenges for long term data storage, manipulation and provision of access to other users for
comparison with models. While computer speeds, data storage capacity and transmission bandwidth are all improving, it is not clear whether current approaches will prove to be sufficient. Finally, while good progress has been made in using CT data sets to inform model development, hitherto the full 3-D potential of the data sets available has not been exploited. This will require increased sophistication in the comparison of models with experimental data in 3-D, including developing statistical techniques to evaluate the quality of agreement between experiments and models and visual techniques for representing the results.

5. Conclusions
In situ and ex situ observations have been made using high-resolution computed tomography of damage mechanisms in carbon fibre composites. Work has been conducted on impact damage resistance and damage tolerance, interlaminar fracture toughness and fibre fracture and tensile strength. These experimental studies have allowed key damage modes to be identified and quantified, which has allowed novel insights to be achieved regarding the relative importance of the individual damage modes and their relationship to microstructural features. Such data sets are particularly powerful when used to inform, calibrate and challenge models for composite performance. It is critical that there is an open and honest dialogue between those conducting experiments, those developing models and the user community.

Acknowledgements
The authors wish to thank many collaborators, postdoctoral researchers and doctoral students who have contributed to the work described herein. Funding from multiple sources, including the UK’s Engineering and Physical Sciences Research Council is gratefully acknowledged.

References
[1] Philips L N 1987 Carbon fibre comes of age Endeavour, 11 127-132
[2] Hinton M J, Kaddour A S Soden P D 2013 The background to the third world-wide failure exercise”, J.Comp. Mater. 47 2417-2426
[3] Cartie D D R Irving P. E. 2002 Effect of resin and fibre properties on impact and compression after impact performance of CFRP Composites Part A 33 483-493
[4] Cantwell W J Morton J 1989 Comparison of the low and high velocity impact response of CFRP Composites 20 545-551
[5] Evans A G Ahmad Z B Gilbert D G Beaumont P W R 1986 Mechanisms of toughening in rubber toughened polymers Acta Metallurgica 34 79-87
[6] Sela N and Ishai O 1989 Interlaminar Fracture-Toughness and Toughening of Laminated Composite Materials – A review Composites 20 423-435
[7] Rosen B W 1964 Tensile failure of fibrous composites, AIAA Journal 2 1985-1991
[8] Harlow D G Phoenix S L 1981 Probability distributions for the strength of composite materials 2. A convergent sequence of tight bounds International Journal of Fracture 17 601-630
[9] Wright P Fu X Sinclair I Spearing S M 2008 Ultra high resolution computed tomography of damage in notched carbon fiber-epoxy composites J. Comp. Mater. 42 1993-2002
[10] Moffat A J Wright P Buffiere J Y Sinclair I Spearing S M 2008 Micromechanisms of damage in 0 degrees splits in a [90/0]s composite material using synchrotron radiation computed tomography Scripta Mater 59 1043-1046
[11] Moffat A J Wright P Helfen L Baumbach T Johnson G Spearing S M Sinclair I 2010 In situ Synchrotron Computed Laminography of damage in Carbon Fibre Epoxy (90/0)s Laminates Scripta Materialia, 62 97-100
[12] Wright P Moffat A Sinclair I Spearing S M 2010 High-resolution tomographic imaging and modeling of notch tip damage in a laminated composite Comp. Sci. Tech. 70 1444-52
[13] Bull D J Spearing S M Sinclair I Helfen L 2013 Three-dimensional assessment of low velocity impact damage in particle toughened composite laminates using micro-focus X-ray computed tomography and synchrotron radiation laminography Composites Part A 52, 62-69

[14] Bull D J Scott A E Spearing S M Sinclair I 2014 The influence of toughening-particles in CFRPs on low velocity impact damage resistance performance Composites Part A 58 47-55

[15] Bull D J Spearing S M Sinclair I 2014 Observations of damage development from compression-after-impact experiments using ex situ micro-focus computed tomography Comp. Sci. Tech. 97, 106-114

[16] Bull D J Spearing S M Sinclair I 2015 Investigation of the response to low velocity impact and quasi-static indentation loading of particle-toughened carbon-fibre composite materials, Composites Pt. A, 74, 38-46

[17] Borstnar G Mavrogordato M N Helfen L Sinclair I Spearing S M Interlaminar fracture micro-mechanisms in toughened carbon fibre reinforced plastics investigated via synchrotron radiation computed tomography and laminography”, Composite Pt. A, 71, 176-183, 2015

[18] Garcea S C Mavrogordato M N Scott A E Sinclair I Spearing S M 2014 Fatigue micromechanism characterisation in carbon fibre reinforced polymers using synchrotron radiation computed tomography Comp. Sci. Tech. 99 23-30

[19] Garcea S C Sinclair I Spearing S M 2015 In situ synchrotron tomographic evaluation of the effect of toughening strategies on fatigue micromechanisms in carbon fibre reinforced polymers Comp. Sci. Tech. 109 32-39

[20] Scott A E Mavrogordato M Wright P Sinclair I Spearing S M 2011 In situ fibre fracture measurement in carbon fibre-epoxy laminates using high resolution computed tomography Comp. Sci. Tech. 71 1471-77

[21] Blassiau S Thionnet A and Bunsell A R 2009 Three-dimensional analysis of load transfer micro-mechanisms in fibre/matrix composites Comp. Sci. Tech. 69 33-37

[22] Swolfs Y Morton H Scott A E Gorbatikh L Reed P A S Sinclair I Spearing S M Verpoest I 2015 Synchrotron radiation computed tomography for experimental validation of a tensile strength model for unidirectional fibre-reinforced composites Composites Pt. A, 77 106-113

[23] Yang Q D Schesser D Niess M Wright P Mavrogordato M N Sinclair I Spearing S M Cox B N 2015 On crack initiation in notched, cross-plied polymer matrix composites J.Mech. Phys. Solids 78 314-32