Evaluation of Digital Volume Correlation (DVC) Applicability in Silicon dioxide (SiO_2) particle-doped Carbon Fibre Reinforced Polymers using *In Situ* Synchrotron Radiation Computed Tomography (SRCT)

Y.Lee[1a], E. Schöberl[1], K. Ball[1], C. Furtado[2,3], P. Patel[2], A. Arteiro[3], M. Majkut[4], L. Helfen[5], B. Wardle[2], T. Tsuneo[6], F. Yasuhiro[6], S. Naoki[6], M. Jalalvand[1], M.N. Mavrogordato[1], S.M.Spearing[1], I. Sinclair[1]

[1] University of Southampton, Southampton, UK [2]Massachusetts Institute of Technology, Cambridge, MA, USA [3]University of Porto, Porto, Portugal [4]European Synchrotron Radiation Facility, Grenoble, France [5]Institut Laue-Langevin, Grenoble, France [6]Mitsubishi Chemical Corporation, Aichi, Japan

[a]yeajin.lee@soton.ac.uk

Abstract. Digital Volume Correlation is a powerful non-intrusive technique capable of full-field strain mapping of internal structures via displacement tracking. The principles of DVC have been successfully applied to unidirectional (UD) Carbon Fibre Reinforced Polymers (CFRPs) by doping with trackable particles (i.e. fiducial markers), thereby enabling strain mapping of materials with an inherent self-similar microstructure [1]. In this paper, the utility of Silicon Dioxide (SiO_2) particle-doped CFRPs for DVC analysis is investigated. Compared to previous use of $BaTiO_3$ particles, SiO_2 is investigated on the basis of having established commercial use in CFRPs, whilst from an imaging perspective it will be less strongly attenuating. In this paper, DVC combined with in situ Synchrotron Radiation Computed Tomography (SRCT) is applied to SiO_2–doped UD CFRPs under quasi-static tensile loading to explore the evolution of individually fractured 0° fibre into clusters of breaks. DVC strain uncertainties are quantified through stationary and rigid body displacement tests, with results being compared for $BaTiO_3$ and SiO_2 particle-doped materials.

Introduction

A fundamental understanding of the fibre break process is essential for a complete interpretation of composite tensile failure, alongside the various other composite damage mechanisms (e.g. fibre-matrix interfacial debonding, matrix microcracking, delamination, sub-laminate, etc.) [1]. Under tension, local load transfer capability deteriorates as the initial fibre break evolves into the ultimate tensile failure of composite materials. This principally induces shear loads in the matrix around individual fibre breaks, followed by load transfer into adjacent fibres and back to broken fibres [2]. The process is also accompanied by stress redistribution into the broken fibres, during which stress and fibre length are repeatedly recovered. With ongoing loading, critical cluster is subsequently formed in an unstable and self-sustaining manner when sufficient fibre breaks are undertaken, which may lead to a catastrophic failure [3,4]. In other words, a key approach to controlling stress redistribution around fibre breaks and the length over which it occurs is critical when predicting tensile failure of UD composites. Recent advancements in X-ray Computed Tomography (XCT) combined with in-situ mechanical testing have allowed for the identification of detailed sequences of damage accumulation down to the fibre level, in 3D, within the bulk of real engineering materials under load [F]. DVC combined with the XCT, is a powerful non-intrusive technique capable of quantifying full-field strain mapping of internal structures between different load states by extracting local displacements and strains [2]. However, it should be noted that the noise and sensitivity of CT-based DVC measurements highly relies on the imaging system, the nature of the tested materials, and imaging results. The use of synchrotron radiation source may outperform micro-focus CT in terms of imaging results (signal- and contrast-to noise-ratio, spatial resolution, artefacts) and acquisition time due to the nature of the beam (e.g. monochromatic) [1,2].

The main intrinsic DVC challenges associated with conventional unidirectional CFRPs are attributed to its highly anisotropic and somewhat self-similar/regular microstructures, particularly along the fibre direction. The filaments with cylindrical structures and relatively featureless surfaces lack a well-defined and trackable contrast pattern, resulting in potentially false correlation peaks (and displacement inaccuracies) in fibre loading directions. In response, Brault *et al*. devised a method of inserting 150 µm metallic particles (Copper) between each carbon plies to create individual unique features providing [5]. These particles provided the necessary contrast for DVC, however they were limited from an imaging perspective to a voxel size of 52 µm. Subsequently, Schoberl et al. demonstrated the principle of exploiting DVC at fibre-level by embedding a sparse population of 400 nm-sized Barium Titanium ($BaTiO_3$) particles directly within the matrix phase of carbon fibre-epoxy composites. Strain distributions were quantified under in-situ SRCT tensile testing, imaged at a voxel resolution of 0.65 µm. However, there is an increasing need for alternative markers that can increase their real engineering applications with higher strength.

Materials and Methodology

In this paper, Silicon Dioxide (SiO_2) particle-doped CFRPs are newly developed as a DVC-optimised material considering two facts aspects: (1) SiO_2 particles are closer to the type of hard particles that would be used in composites. (2) They are routinely utilised in the real world as a ‘filler’ to increase the effective modulus and strength. Following a similar approach taken by Schoberl et al [1-2], particle dispersion is carried out within a matrix phase of the CFRPs. From an imaging perspective, a limitation exists in that the proposed material undergoes lower X-ray attenuation than $BaTiO_3$-doped materials due to the atomic number differences (O<Si<Ti<Ba), which eventually makes it challenging to apply DVC. However, it may reduce artefacts associated with a high contrast mismatch between the particles and the other constituents of the composites. This paper, a combination of DVC and in-situ SRCT, first assesses the DVC applicability for a new particle doping (SiO_2) based on the DVC performance successfully measured in $BaTiO_3$ particles. Herein, a double edge notched tension (DENT) testing was performed on the SiO_2-doped CFRPs to explore the development of individually fractured fibres evolving into the cluster of breaks. DVC strain mapping is then performed within 0° fibre plies using the commercial Davis v10.2.0 software (LaVision) to understand corresponding stress redistribution between broken and unbroken fibres. DVC measurement errors (i.e. uncertainties) in strain mapping are also quantified by stationary (i.e. repeated scans such as 'zero-strain') and rigid body displacement tests. This paper will also present a comparison of the DVC traceability between SiO_2 and $BaTiO_3$ particles according to particle distribution, particle size, X-ray contrast, and uncertainties.

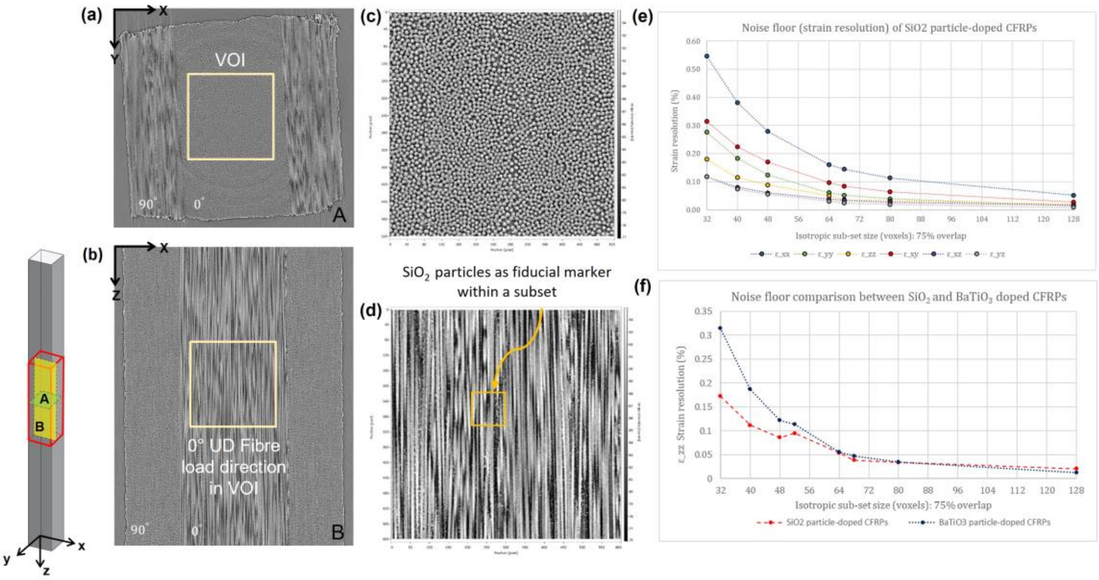

Fig. 1. (a) XY view at SiO_2 doped CFRPs, Volume of Interest (VOI) is chosen for DVC processing (b) XZ view where 0° UD fibre can be seen. (c) XY view of VOI in Davis V10.2.0 (d) View for longitudinal fibre. 500nm sized SiO_2 particles are well-dispersed along the fibre load direction (e) Noise floor according to different subset sizes. The 'ε_{zz}' component of the strain tensor denotes the longitudinal (fibre) strain, while 'ε_{xx}' and 'ε_{yy}' represent in-plane transverse and axial strain, respectively. (f) Comparison of noise floor between SiO_2 and $BaTiO_3$ particles along the fibre load direction.

Conclusion

This paper demonstrates the procedure to evaluate the DVC applicability of SiO_2 particle-doped CFRPs compared to the use of $BaTiO_3$ particles. Quantification of noise floors according to different DVC subset sizes has significance in assessing whether local strain measurements via DVC strain mapping are achieved within satisfactory accuracy. It has been shown in this paper that SiO_2 materials have a lower or similar noise floor along the fibre load direction compared to $BaTiO_3$ particles, which also indicates that low attenuation may no longer be a limiting factor in the DVC utility of SiO_2 particles as fiducial markers. Work is ongoing to discover effects of particle characteristic and evaluate the DVC traceability of SiO_2 particles in predicting the evolution of individual fibre breaks into the cluster of breaks via strain mapping.

References

[1] Schöberl E, Breite C, Melnikov A, Swolfs Y, Mavrogordato MN, Sinclair I, Spearing SM. Fibre-direction strain measurement in a composite ply under quasi-static tensile loading using Digital Volume Correlation and in situ Synchrotron Radiation Computed Tomography. Composites Part A: Applied Science and Manufacturing. (2020) Oct 1;137:105935.

[2] Schöberl E, Mavrogordato MN, Sinclair I, Spearing SM. Fibre direction strain measurement in a composite ply under pure bending using Digital Volume Correlation and Micro-focus Computed Tomography. Journal of Composite Materials. 2020 Jun;54(14):1889-912.

[3] Rosini S, Mavrogordato MN, Egorova O, Matthews ES, Jackson SE, Spearing SM, Sinclair I. In situ statistical measurement of local morphology in carbon-epoxy composites using synchrotron X-ray computed tomography. Composites Part A: Applied Science and Manufacturing. (2019) Oct 1;125:105543.

[4] Swolfs Y, Morton H, Scott AE, Gorbatikh L, Reed PA, Sinclair I, Spearing SM, Verpoest I. Synchrotron radiation computed tomography for experimental validation of a tensile strength model for unidirectional fibre-reinforced composites. Composites Part A: Applied Science and Manufacturing. 2015 Oct 1;77:106-13.

[5] Brault R, Germaneau A, Dupré JC, Doumalin P, Mistou S, Fazzini M. In-situ analysis of laminated composite materials by X-ray micro-computed tomography and digital volume correlation. Experimental Mechanics. 2013 Sep;53(7):1143-51.