

CHARACTERISATION OF DAMAGE EVOLUTION ON TEXTILE CERAMIC MATRIX COMPOSITE BY IN-SITU X-RAY COMPUTED TOMOGRAPHY TEST

Weiyu Guo¹, Yi Zhang², Daxu Zhang^{1*}, Yuqi Feng¹, Long Wang³, Yonglong Du¹

¹ School of Naval Architecture, Ocean & Civil Engineering, Shanghai Jiao Tong University

² Science and Technology on Thermostructural Composite Materials Laboratory, Northwestern Polytechnical University, Xi'an, 710072, China

³ Science and Technology on Reliability and Environment Engineering Laboratory, Beijing Institute of Structure and Environment Engineering, Beijing 100076, China

* Corresponding author (daxu.zhang@sjtu.edu.cn)

Keywords: Ceramic matrix composites (CMCs); Damage evolution; X-ray CT; Deep Learning

ABSTRACT

This paper studied the plain weave textile ceramic matrix composite's meso-structure and damage evolution with the aid of an in-situ X-CT test system. A deep learning-based image segmentation approach was applied to characterise the material's constituents, and crack initiation and propagation. It was found that the volume fractions of the carbon fibre bundle, silicon carbide matrix and voids outside the bundle were 61.3%, 27.3% and 11.4%, respectively. The volume fraction of cracks of the C_f/SiC composite was approximately 0.05% when it approached to failure. The cracks tended to initiate near the voids and in the matrix rich-region and propagated rapidly during high-level loadings.

1 INTRODUCTION

Ceramic matrix composites (CMCs) are a novel composite material which has promising mechanical performance under high temperatures. They could be widely seen on aeronautic and astronautic field, sports vehicle's brake disc, nuclear reactor cladding and so on [1]. The rapid development of ex-situ and in-situ computed tomography testing technology has played a powerful role in the direct observation of the microstructure of materials and damage evolution. Bale [2] et al. obtained the 3D microstructure, composition characteristics and statistical laws of parameters of unidirectional ceramic matrix composites with the help of micro-CT technology. Larson [3] et al. used X-ray CT scanning to observe the microstructure and internal crack evolution of SiC-based preceramic polymer during the moulding process at high temperatures. Zhang [4] et al. investigated plain weave ceramic matrix composites' tensile behavior from the dimension of damage evolution under the in-situ X-ray scanning state. Tomograms with high resolution could provide a clearer visualisation of the inner morphologies of CMCs. However, obtaining high-quality tomograms could result in longer scanning time, thus, there is a trade-off between tomogram quality and scanning time. Zwanenburg [5] investigated the CT scanning parameters that could affect the image quality and found that maximizing the source-detector distance and minimizing the voltage yielded the best image quality for observing the structures and defects of carbon fibre composites. Based on the slices of CMCs from CT scanning, various works could be conducted, such as developing segmentation approach [6], converting CT images into finite element models, i.e., IB-FEM [7], and generating 3D strain fields by digital volume correlation (DVC) analysis [8], etc. In the present study, the failure mode and damage evolution of plain weave C_f/SiC composites under in-situ X-ray CT scan and uniaxial tension load case was investigated. Deep-learning-based segmentation method was utilized to identify the materials' constituents and damages.

2 IN-SITU X-RAY CT TEST

2.1 Specimen and Test Instrument

Several plain weave C_f/SiC composite specimens, manufactured by the chemical vapor infiltration (CVI) process were tailored, with their width and thickness of 5.0 mm and 3.1 mm, respectively. The

reinforcing phase is composed of several layers of interweaved yarns, and the amount of fibres in each yarn is approximately 0.5k. Due to the CVI process's characteristic during precursor infiltration and SiC matrix deposition, a large number of pores and voids could be generated among the inner morphology of Cf/SiC composites. A lab-CT system, ZEISS Xradia 520 Versa TM X-ray microscope, with a maximum spatial resolution of 0.7 μm was applied to characterise the mesostructured and damages of the material. A Deben CT5000-H250 TM in-situ tensile/compression loading fixture with a maximum loading capacity of 5 kN was utilised to carry out tensile test under the CT scan chamber environment.

2.2 Test Schemes

Given the absorption of the X-ray energy by the loading instrument's tube wall that surrounded the specimen, the X-ray source voltage was set to 80kV. After the specimen fractured, it was carefully taken out from the loading fixture and did a rescan without the existence of the tube wall and applying a lower source voltage of 60 kV and adjusting the source-detector distance with the purpose of obtaining a better contrast of CT images. In order to see the whole specimen's width direction in the FOV, the scanning resolution was adjusted to $\sim 5.0 \mu\text{m}$ per voxel during the in-situ tensile test. All scans were 180 degrees of scan mode. The displacement control mode of loading was adopted and the loading rate was 0.2 mm/min. For the in-situ X-CT tensile test of the material, the in-situ loading system recorded the force value and the grips' displacements. Tomographic slices were collected continuously during the entire test and three-dimensional (3D) reconstruction was then performed using XMReconstructor. At each loading level, a single CT scan was carried out for a couple of hours and the loading were kept unchanged.

3 DEEP LEARNING-BASED IMAGE SEGMENTATION

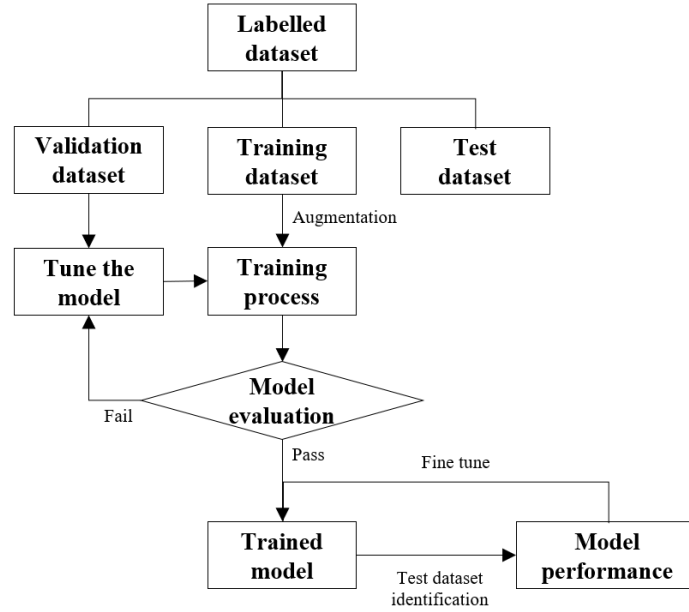


Figure 1: The workflow of the deep learning-based image segmentation.

Since the carbon fibre and SiC matrix cannot be separated from the CT images merely by threshold segmentation, the U-net deep convolutional neural network (DCNN) architecture, as a built-in algorithm of ORS Dragonfly TM, was applied and trained for segmenting the constituents of the C_f/SiC composite automatically. Fig. 1 shows the workflow of the deep learning-based image segmentation: First, several images were selected as the original dataset, then manually labelled each constituent (the background also needed to be labelled) in the images. The original images and their corresponding labels together formed the labelled dataset. Then, the dataset was augmented and input into the DCNN for model training. During the training process, the parameters of the model were updated after each training

epoch. It is worth noting that a part of the labelled dataset was extracted as the validation dataset and test dataset to help iteratively optimise the trained model to a level where the SiC fibre and SiC matrix constituents can be accurately identified. The same method was applied for detecting the internal cracks of the C_f/SiC composite. However, before labelling, the original CT images were filtered firstly using the black top hat algorithm and let the cracks, which featured long, thin and branch-shaped, be enhanced. Then, similar operations of the labelling, model training and optimisation were conducted.

4 DAMAGE EVOLUTION

4.1 Load-displacement curve

The tensile load-displacement curve of the material obtained from the in-situ X-ray test is shown in Fig. 2 where σ_u represented the ultimate stress of the material. It can be seen from the figure that the force of the scanning point (the point marked in red) has experienced a situation where it first dropped and then raised. The stress relaxation during the CT scans may be the possible reason for these zig-zag segments of the curve. Additionally, the curve demonstrates obvious nonlinearity, indicating that material undergoes damage initiation and propagation.

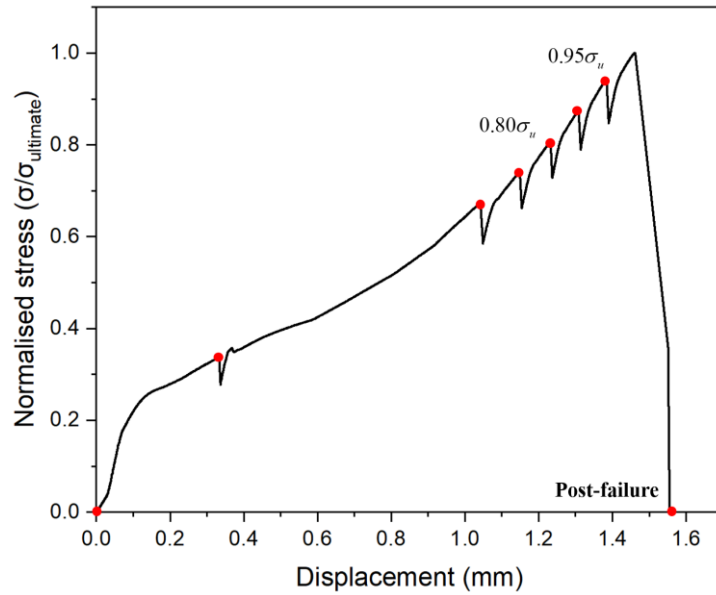


Figure 2: The normalized stress versus displacement curve of the plain weave C_f/SiC specimen.

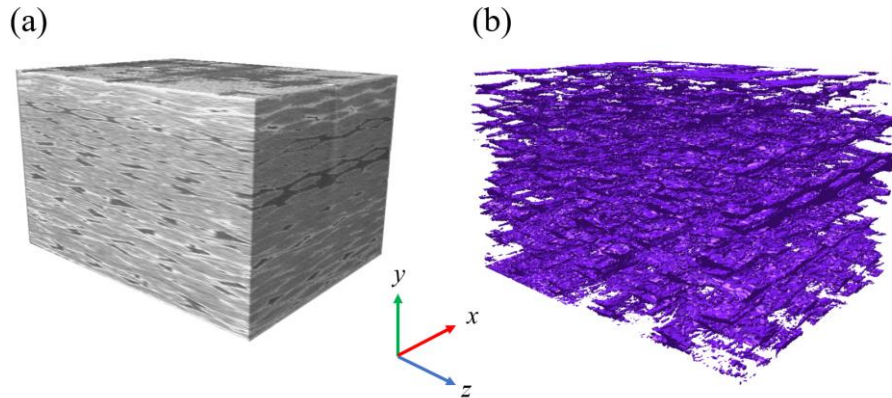


Figure 3: (a) The morphology of the C_f/SiC composite after 3D reconstruction, (b) the rendering of the voids outside the yarns.

4.2 Meso-structure Characterisation

The three-dimensional morphology of the C/SiC composite is presented in Fig. 3(a). A cartesian coordinate system was defined in this figure. Here, the loading direction was set as the Z direction while the X and Y direction were defined according to the right-hand screw rule. From the view of the YZ plane, some SiC fibres were ribbon-like and were denoted as warp yarns while others were ellipse-like and were denoted as weft yarns. The SiC matrix was not fully filled with the region of the interest' remaining area, the vast majority of the matrix adhered to the surface of the yarns. With deep learning-based image segmentation method mentioned in part 3 applied, the large voids outside the yarns were rendered and shown in Fig. 3(b). Moreover, the volume fraction of each phase was calculated based on the segmentation results where the volume fraction of the carbon fibre bundle, matrix and voids outside the yarns were 61.3%, 27.3% and 11.4%, respectively.

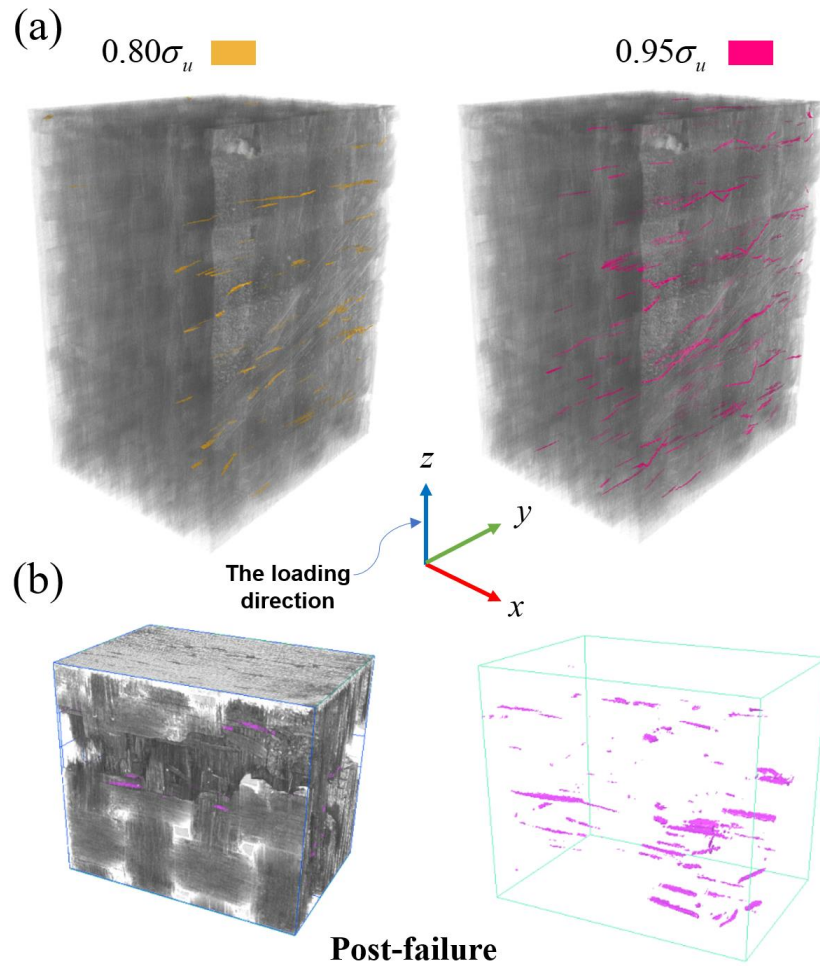


Figure 4: (a) The spatial distribution of cracks of the C/SiC composite under the two levels of load state and (b) the fractured region of the specimen and the transverse matrix cracks distribution in the region.

4.3 Failure mode and Damage Evolution

The internal cracks under different load states were identified automatically based on the well-trained deep learning-based segmentation model. The cracks under the load state of $0.80\sigma_u$ and $0.95\sigma_u$ were rendered and shown in Fig. 4(a). The image registration approach was used to align the datasets of different load states. After image registration, the newly initiated cracks were obtained by Boolean

operations between the cracks of two adjacent load states. The damage volume fraction at these two load states were 0.037% and 0.052%. It was founded that the cracks tended to initiate near the voids and in the matrix rich-region. The transverse cracks first appeared during stretching the C_f/SiC specimen. There were two ways of crack increment: one way was that new cracks formed elsewhere in the material, and the other way was the propagation of the original cracks. For the crack propagation case, when one crack propagated to the fibre-matrix interlayer, it then deflected. Such behaviour of cracks delays the penetration of them through the fibres, thereby increasing the ductility of the material.

Transverse matrix cracking, longitudinal matrix cracking, and fibre pull-outs are the main failure modes. At the low loading level, the stress-strain response is linear and no material damage is observed. At the intermediate loading level, both transverse matrix cracking and longitudinal matrix cracking initiate and propagate gradually. This results in the degradation of the slopes of the stress-strain curves. At the high loading level, both transverse matrix cracking and longitudinal matrix cracking develop rapidly.

5 CONCLUSIONS

The present work characterised the meso-structure of plain weave carbon fibre-reinforced silicon carbide composites manufactured by the CVI process. And an in-situ X-ray CT tensile test was conducted on the C_f/SiC composite to investigate its damage evolution. The constituent's separation and crack detection works were accomplished with the application of the deep learning-based image segmentation method. The failure mode and damage initiation and propagation during the tension was observed and discussed. The following are the main conclusions.

(1) The in-situ computed tomography test system, including the X-ray microscope and the in-situ loading instrument, plays a key role in characterising the meso-structure and damage evolution of C_f/SiC composites. Deep learning-based image segmentation can be an effective approach for quantifying the composite's constituents and damage growth.

(2) The volume fraction of the carbon fibre bundle, silicon carbide matrix and voids outside the bundle were 61.3%, 27.3% and 11.4%. And the volume fraction of cracks of the C_f/SiC composite was approximately 0.05% when it approached to failure.

(3) Transverse matrix cracking, longitudinal matrix cracking, and fibre pull-outs are the main failure modes. The cracks tended to initiate near the voids and in the matrix rich-region. And it propagated rapidly during high-level loadings.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the financial support of the National Natural Science Foundation of China (12072192, U1831105, 11272207).

REFERENCES

- [1] N. P. Bansal and J. Lamon, *Ceramic Matrix Composites: Materials, Modelling and Technology*, John Wiley, The American Ceramic Society, Hoboken, NJ, 2014.
- [2] H.A. Bale, A. Haboub, A.A. MacDowell, J. R. Nasiatka, D. Y. Parkinson and B.N. Cox, Real-time quantitative imaging of failure events in materials under load at temperatures above 1,600 °C, *Nature Materials*, **12**(1), 2013, pp. 40-46 (doi: 10.1038/nmat3497).
- [3] N.M. Larson and F.W. Zok, In-situ 3D visualization of composite microstructure during polymer-to-ceramic conversion, *Acta Materialia*, **144**, 2018, pp. 579-589 (doi: 10.1016/j.actamat.2017.10.054).
- [4] D. Zhang, Y. Liu, H. Liu, Y. Feng, H. Guo, Z. Hong, C. Chen and Y. Zhang, Characterisation of damage evolution in plain weave SiC/SiC composites using in situ X-ray micro-computed tomography, *Composite Structures*, **275**, 2021, 114447 (doi: 10.1016/j.compstruct.2021.114447).
- [5] E.A. Zwanenburg, D.G. Norman, C. Qian, K.N. Kendall, M.A. Williams and J.M. Warnett, Effective X-ray micro computed tomography imaging of carbon fibre composites, *Composites Part B: Engineering*, **258**, 2023, 110707 (doi: 10.1016/j.compositesb.2023.110707).

- [6] J. Bénézech and G. Couégnat, Variational segmentation of textile composite preforms from X-ray computed tomography, *Composite Structures*, **230**, 2019, 111496 (doi: 10.1016/j.compstruct.2019.111496).
- [7] S. Ai, W. Song and Y. Chen, Stress field and damage evolution in C/SiC woven composites: Image-based finite element analysis and in situ X-ray computed tomography tests, *Journal of the European Ceramic Society*, **41(4)**, 2021, pp. 2323-2334 (doi: 10.1016/j.jeurceramsoc.2020.12.026).
- [8] G. Niu, R. Zhu, H. Lei, R. Zhang, P. Wang, Z. Qu and D. Fang, Internal damage evolution investigation of C/SiC composites using in-situ tensile X-ray computed tomography testing and digital volume correlation at 1000 °C, *Composites Part A: Applied Science and Manufacturing*, **163**, 2022, 107247 (doi: doi.org/10.1016/j.compositesa.2022.107247).