SYNCHROTRON-BASED 3D IMAGING OF CURING IN CARBON FIBRE PREPREG

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ABSTRACT

Imaging techniques have long been an invaluable tool for characterizing the microstructure and monitoring processes in carbon fibre composites. These techniques include everything from basic photography with a visible-light microscope to more advanced modalities like digital image correlation (DIC), ultrasound (US), and x-ray-based techniques. Only one x-ray-based technique in particular provides a complete image of internal microstructure, and that is computed tomography (CT).

Laboratory-based Micro-CT has been used extensively to this end to quantify high-contrast features like porosity and damage. During a CT scan, the sample is slowly rotated so that projections can be captured from thousands of angles and reconstructed into a 3D volume dataset. Using a conventional x-ray source, such a scan can take many hours or even days, during which the sample must remain completely static in order for the scan to reconstruct. Using a synchrotron-based x-ray source (which is based on a large-format particle accelerator) provides an x-ray beam that is several orders of magnitude brighter, allowing for micro-CT scans to be completed in a matter of seconds or minutes. Synchrotron CT also provides greatly improved contrast using propagation-based phase imaging, allowing for the resolution of very similar materials (such as carbon fibre and resin).

This presentation describes preliminary work undertaken towards the use of synchrotron CT for the study of woven carbon fibre composite materials. The development of an apparatus for time-resolved imaging of the curing process in an out-of-autoclave (OOA), active vacuum bag assembly is discussed and results from proof-of-concept experiments are presented. The result is an OOA apparatus that is capable of producing scans down to a demonstrated resolution of 4 microns while under active vacuum.

KEYWORDS: *curing, in-situ, computed tomography, synchrotron*

1 INTRODUCTION

A wide variety of imaging techniques has historically been available to composites researchers as part of a broader effort to understand the behaviour and evolution of microstructure during manufacturing processes. This toolset includes optical microscopy (Farhang *et al.*, 2015), ultrasonic imaging (Thomas *et al.*, 2008), infrared thermography (Ray *et al.*, 2007), and x-ray computed tomography (CT) (Stamopoulos *et al.* 2016). Optical microscopy provides the advantage of high resolution and rich morphological information of the surface, but cannot be used to probe the internal microstructure in a non-destructive manor. Ultrasonic imaging and thermography are very useful non-destructive techniques, but are limited

in resolution to a scale of hundreds of microns (Farhang *et al.*, 2015). Computed tomography is capable of non-destructively providing full internal microstructure at high resolution, but conventional CT measurements can be very slow, with typical micro-CT and nano-CT scan times on the order of several hours, and contrast is generally insufficient to resolve materials of similar composition/density (Singhal *et al.*, 2013). Although conventional CT has been used extensively to characterize static composite samples, it is poorly suited to time-resolved studies of dynamic processes like curing.

One option for overcoming these limitations is using synchrotron x-ray source in place of conventional x-ray tubes present in laboratory CT scanners. A synchrotron is a particle accelerator that is used to generate high-flux, broad-spectrum, and highly collimated x-ray beams for materials research applications (Dallin *et al.*, 2003). Synchrotron radiation can be filtered using x-ray monochromators to deliver highly monochromatic beams that provide significantly improved contrast in CT scans compared to conventional sources. The high photon flux of synchrotron sources also provides data acquisition times that are several orders of magnitude shorter than for conventional CT scans, down to a scale of seconds or minutes (Bond *et al.*, 2017). This provides an ideal tool for time-resolved imaging of the internal microstructure in carbon fibre composites at micron-scale resolution (Moffat *et al.*, 2008).

Synchrotron micro-CT thus provides a means of potentially imaging the evolution of microstructure during the curing process. To this end, we present a feasibility demonstration of an in-situ apparatus designed to carry out synchrotron CT scans of a woven carbon fibre prepreg during out-of-autoclave (OOA) in a vacuum bag assembly. This work shows that CT scans of an uncured coupon can be collected down to high resolution (4.5-µm pixel size) under applied active vacuum with almost no imaging artefacts related to motion or vibration.

2 METHODS

2.1 Imaging

All CT scans were acquired at the Canadian Light Source synchrotron facility using the Biomedical Imaging and Therapy Beamline Bending Magnet beamline (BMIT-BM) (Wysokinski *et al.*, 2007). Scans were collected using monochromatic beam set to an energy of 20 keV. The x-ray detector used consists of a 10-µm-thick Gadox scintillator paired with optical Hammamatsu C9300 camera. Using this setup, most scans were carried out using an effective resolution of 4.5 µm (pixel dimensions) except where otherwise noted. Experiments were carried out in "step-and-shoot" mode, where the sample is moved in discrete increments between exposures. Reconstruction of CT datasets was carried out using the NRecon software package and visualizations were produced using the Avizo software package.

These experiments are optimized for maximum contrast and image quality, yielding scan times on the order of one hour for 4.5-µm scans. Much faster scan times can be achieved on the same beamline using filtered white beam and continuous rotation of the sample, with total scan times typically on the order of 1-2 minutes (and sometimes as low as tens of seconds). For the purposes of this study, it was desirable to test the setup using quality-optimized scan conditions, since these conditions are the most sensitive to vibration from the attached vacuum pump. For the purposes of future time-resolved tests, shorter scan times are likely more desirable so as to best capture transients during the curing process, though it is important to retain the option of quality-optimized scans in order to study low-contrast features (such as the boundaries between carbon fibre and resin).

2.2 Materials

For these proof-of-concept scans, a single coupon was used with a width of 10 mm and a thickness of 2 mm. The material used was a 0-90 woven prepreg that contained uncured resin. This was used for these tests, as the uncured resin would be the most prone to motion artefacts as a result of vibrations from the vacuum pump. As long as motion-related artefacts can be managed in the uncured state using the longest-possible scan times, all other sample states and scan conditions should not present any issues.

2.3 In-Situ Apparatus

In order to carry out imaging of a coupon during the curing process under active vacuum, it was necessary to construct an apparatus that would allow for a vacuum assembly to be connected to a vacuum pump without obscuring the coupon with any x-ray-opaque materials. Another design requirement is that the coupon not move or vibrate appreciably over the length of a CT scan, which can cause image artefacts, or even failure of CT reconstruction. Several designs were attempted and tested that involved fixing a coupon to an aluminium block that can be fixed to the sample stage of the CT beamline.

Figure 1 shows the design that yielded the first successful in-situ CT scans. The vacuum port on the side of the block connects to a simple scroll pump using flexible plastic tubing. The coupon itself is positioned at the top of the assembly with the vacuum bag so that it is not occluded by the large aluminium block. For these tests, a coupon with a width of 10 mm was used. During CT scans, the entire assembly is fixed to a rotational stage as shown in Figure 2. During a scan, the sample is rotated through a range of 180 degrees about the vertical axis of the assembly (the laser indicates the position of the beam on the coupon). During the scan, the vacuum pump is connected and the vacuum bag is actively pumped through the entire duration of the scan.

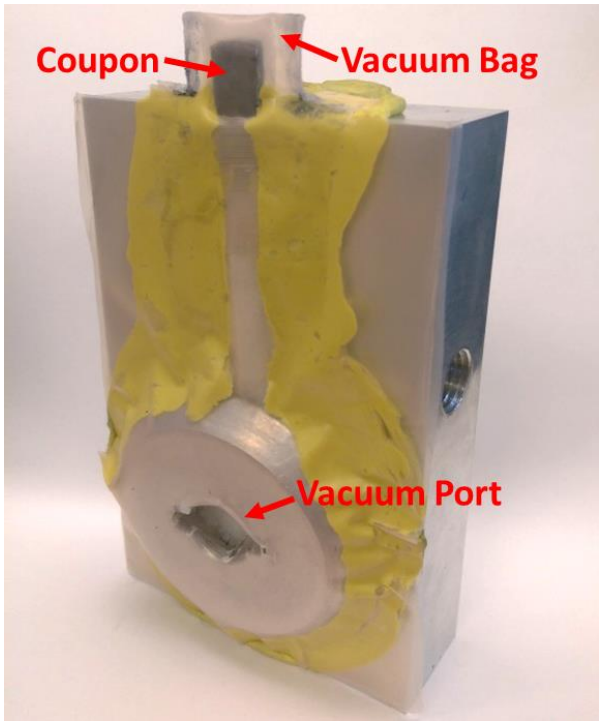

Figure 1: Vacuum bag apparatus for in-situ, CT imaging. Labels indicate position of coupon to be imaged as well as the vacuum port and vacuum bag affixed to the aluminium block.

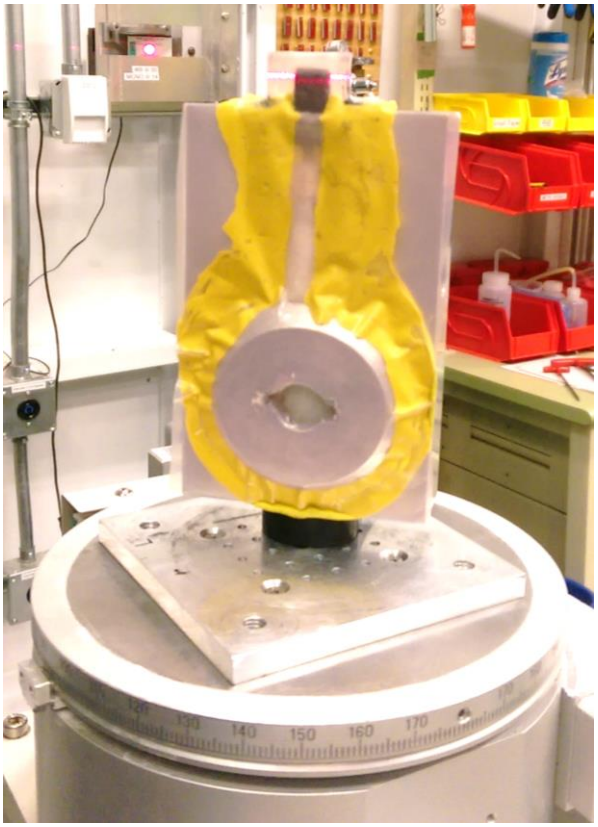

Figure 2: In-situ CT apparatus mounted on rotation stage of BMIT-BM beamline.

3 RESULTS

In order to determine a target resolution for the in-situ system, ex-situ scans of a pre-cured coupon were first carried out using different detectors with different effective pixel sizes. Figure 3 shows cross sections taken from two of these scans, which were acquired using detectors with resolutions of (a) 8.9 µm and (b) 4.5 µm. The darkest regions of both images represent the pore space in the uncured coupon. Dark and bright fringes are visible near pore boundaries due to propagation-based phase contrast, where destructive and constructive interference (respectively) of the beam with itself results from interaction of materials with different refractive index. Although the 8.9-µm data does reasonably resolve the intra-tow pore space, this level of porosity is much better captured in the 4.5- µm scan. 4.5 µm was thus used as the target resolution for the in-situ system.

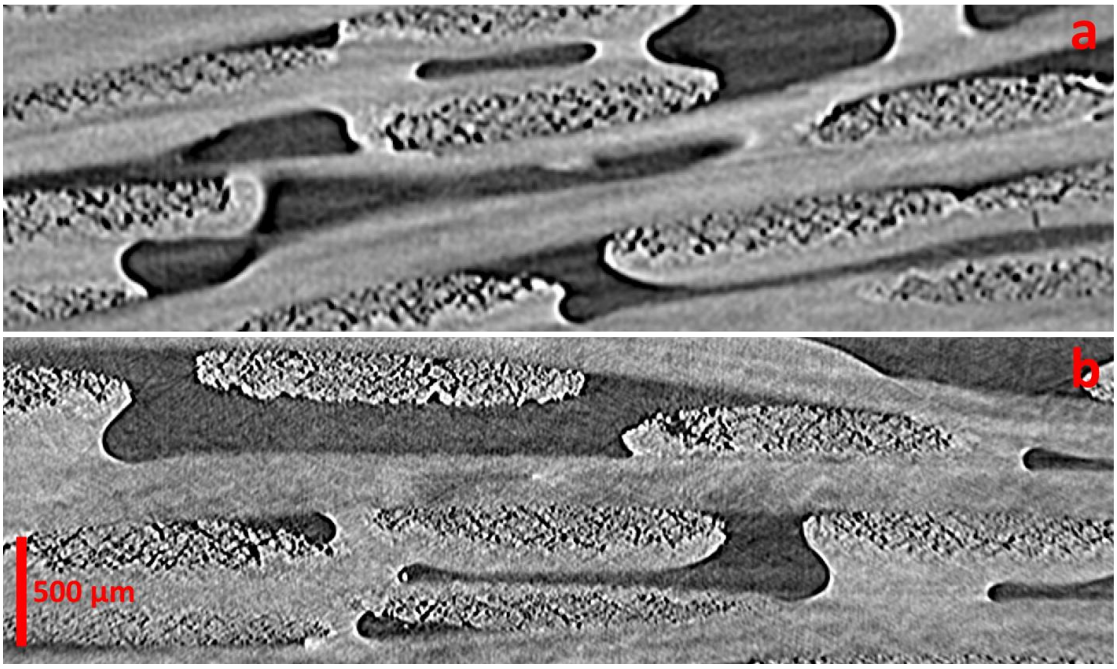

Figure 3: 2D cross sections taken from ex-situ CT scans of an uncured coupon collected at resolutions of (a) 8.9 µm and (b) 4.5 µm, respectively.

In order to test the in-situ apparatus, it was mounted on the beamline and two CT scans of the coupon were carried out at 4.5-µm resolution: one scan of the coupon inside the apparatus at ambient temperature (no vacuum applied) and one scan where the vacuum was connected and actively pumping during the full duration of the scan. Figure 4 shows 3D renderings of CT scans showing the coupon and vacuum bag that were acquired (a) without vacuum and (b) with applied vacuum. The contraction of the bag is clearly visible and the features of the bag and coupon appear reasonably well-resolved. Figure 5 shows a close-up 2D cross section of the coupon (a) without vacuum and (b) with vacuum. This visualization provides a more detailed comparison of the two scans. In these images, the coupons were aligned using a 3D image registration algorithm so that the same area could be compared. The overall image quality of the two scans is quite similar, indicating that efforts to supress the vibration of the pump during the scan were highly successful. Upon close inspection, there are very small artefacts present around the edges of some pores in Figure 5(b), which are likely the result of slight motion artefacts. These artefacts are manifested as small streaks and “half-moons” that are typical of reconstruction errors due to vibrational sample motion. Considering the significant vibration produced by the connected vacuum pump, the small scale of these artefacts is an encouraging result.

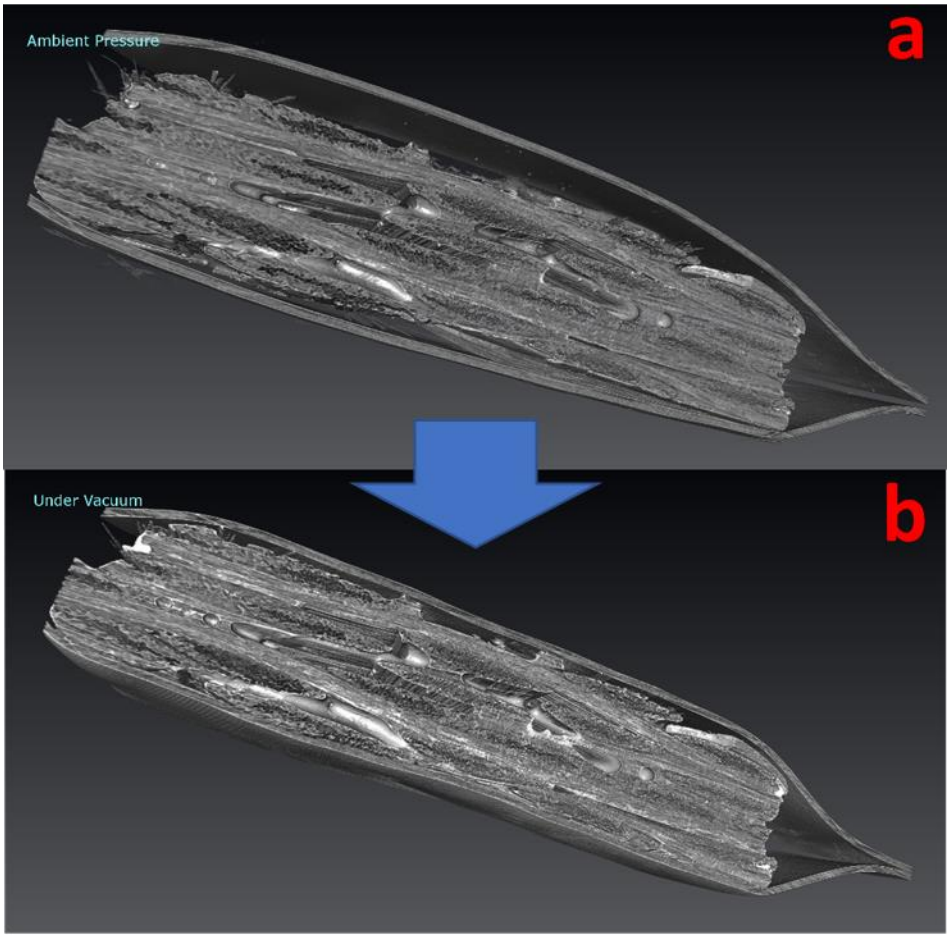

Figure 4: 3D renderings of the vacuum bag and carbon fibre sample (a) without vacuum and (b) with applied vacuum.

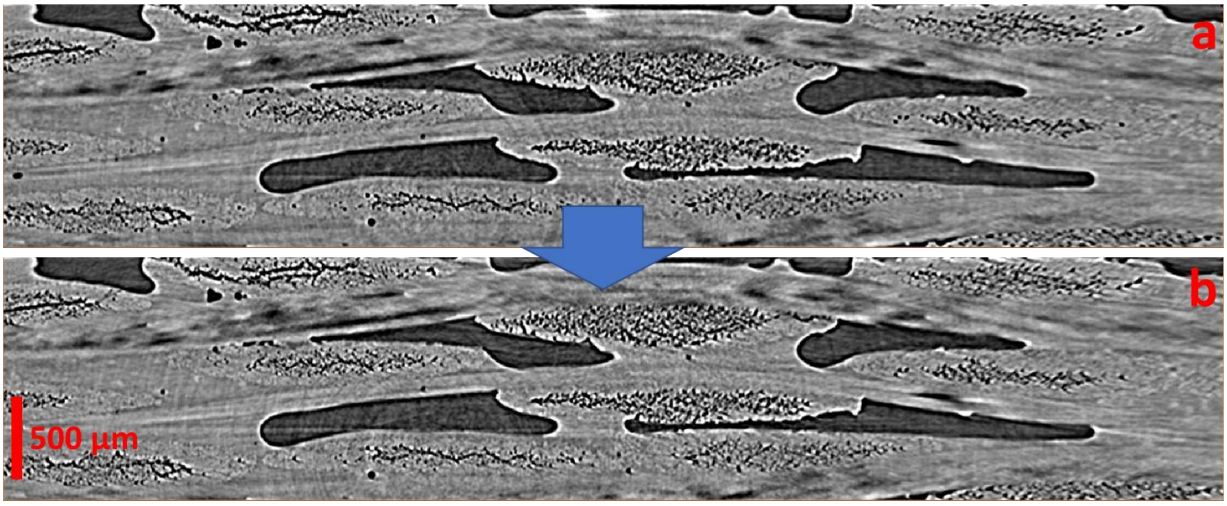

Figure 5: 2D cross sections taken from the same section of the coupon (a) without vacuum and (b) with applied vacuum.

4 CONCLUSIONS

This work demonstrates that a carbon fibre coupon can be successfully imaged at high resolution (4.5 µm pixel size) inside a vacuum bag while under active applied vacuum with almost no motion-related artefacts or other loss of image quality. Moreover, these scans were collected under contrast- and signal-to-noise-optimized conditions, which result in relatively long scan times that are highly sensitive to any motion. Real time resolved experiments are likely to be much faster and less sensitive to motion, but these results show that even the most motion-sensitive scan conditions can successfully be used. Although this has only been demonstrated in this work using an uncured coupon, this work paves the way for time-resolved imaging of an OOA curing process, which is the ultimate goal of the project.

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