In situ X-CT observation of crack initiation and propagation in CFRP with a full-field X-ray microscope

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Abstract
We have developed and installed a new X-ray microscope using synchrotron radiation at the NW2A beamline of PF-AR in IMSS, KEK. Using phase-contrast imaging technique, we have succeeded in nondestructive and three-dimensional (3D) observation of the initiation and propagation of cracks with a resolution down to 50 nm under an applied stress. Fibers, matrix polymer, and cracks were clearly identified in reconstructed 3D images. This first observation has successfully shown that cracks are initiated by (a) de-bonding at the fiber/polymer interfaces and (b) void formation in the polymer, and these factors may determine the propagation path.

1. Introduction

Carbon fiber-reinforced polymer (CFRP) composites are increasing in use in aircrafts owing to their high specific strength and stiffness. The micromechanism of damages and the microscopic chemical properties of CFRPs are key to understanding the mechanical properties and durability of these materials [1]. Recent reports pioneered the micromechanical investigation of fractures under quasi-static stress [2] and fatigue failures [3] in CFRPs by analyzing the three-dimensional (3D) dataset obtained using synchrotron X-ray computed tomography (X-CT).

2. Experiments

We have developed and installed a new X-ray microscope (XRM) using synchrotron radiation (SR) at the NW2A beamline of PF-AR in IMSS, KEK. The outline of its X-ray optics is illustrated in Figure 1. A monochromatic X-ray beam from the undulator is focused onto the sample using an elliptical glass capillary and the image is projected onto the CCD detector by means of a Fresnel zone plate lens. A standard test pattern confirmed a high spatial resolution of less than 50 nm for the system. The sample is mounted on an X, Y, Z, θ stage, and X-CT measurements were performed by rotating the sample for a specific X-ray energy.

It was difficult to obtain clear images of the interfaces between the fibers, resin, and dispersed particles in resin owing to their small density difference. Hence, we used the imaging technique of phase contrast that enhances the image of the interfaces by the scattering effect of X-rays at the interfaces. We could successfully obtain the 3D images of voids and cracks in the CFRP fibers with a resolution of less than 50 nm.

The [90/0]s laminate plates were fabricated from Toray carbon fiber prepreg. Columunar specimens, with a size of ~60 μm in diameter and 1 mm in length, were mechanically cut along the fibers from the plate. A pyramid-shaped diamond tip was indented on to the top of the specimen using a nanomechanical test stage (Fig. 2), which features a high-precision piezo actuator and an integrated load cell up to 5 N, enabling the load-displacement curve to be measured and related to the evolving microstructure observed in the corresponding 3D tomographic reconstructions. Using this stage, primarily a tensile stress perpendicular to fibers and partially a shear stress were applied to the specimen and X-CT measurements were performed (Fig. 2). Typically, transparent images were obtained at each 1° in a range of −75° to 75° with an exposure time of 1 s.

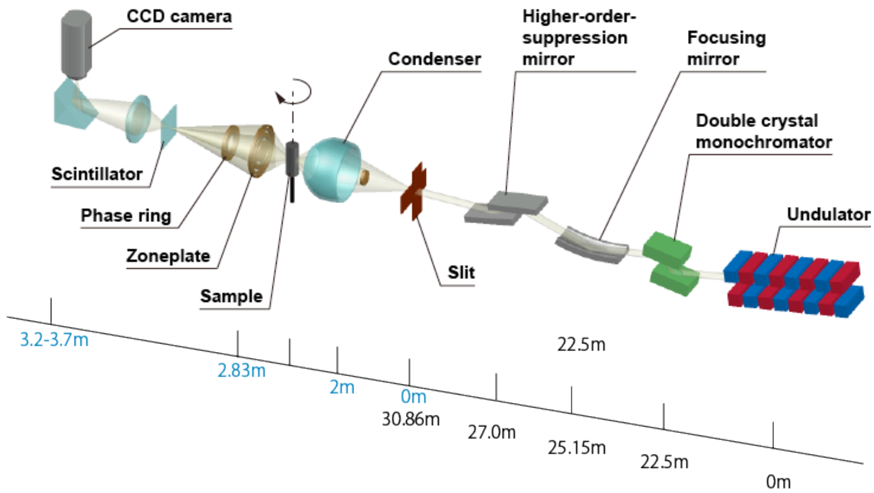

Figure 1. Outline of SR-XAFS-CT microscope.

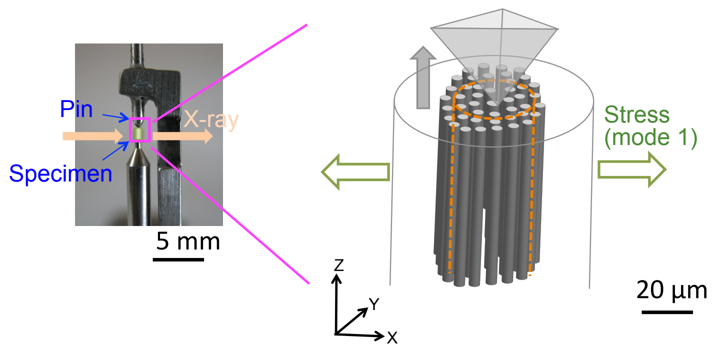

Figure 2. Picture of nanomechanical test stage around the specimen, and a schematic showing how stress is applied to the specimen.

3. Results and discussion

Initially, we performed the challenging high-resolution time-lapse study of *in situ* crack growth in the CFRP by increasing the insertion amount of the tip. High-resolution snapshots of showed that cracks initiate at the top and they propagate down to the bottom of the specimen.

Subsequently, we maintained the insertion amount of the tip at a specific value, and performed X-CT measurements. Figure 3 shows cross-section images of the CFRP under an applied stress by indentation, where the typical crack initiations are shown by triangles. It is noteworthy that the images show the microstructure inside the specimen, which is different from that obtained at the surface. Further, the field of view size is much smaller than the specimen, and the X-CT images represent the initiation and propagation of cracks inside the specimen, free of surface effects.

Owing to the phase imaging technique, the boundaries of the fibers, matrix polymer, and crack were clearly observed. At the small insertion, cracks initiate along the boundaries of fibers and matrix polymer (shown by red triangles in Fig. 3 (b)). This corresponds to the de-bonding at the fiber/polymer interfaces. When the amount of insertion was increased, the cracks propagate along the boundaries and migrate with each other. Some of them traverse in the matrix polymer and form “transverse cracks” in the matrix (shown by blue triangles in Fig. 3 (a)). This corresponds to void formations in the polymer, and their topological and energetic interactions determine the propagation path.

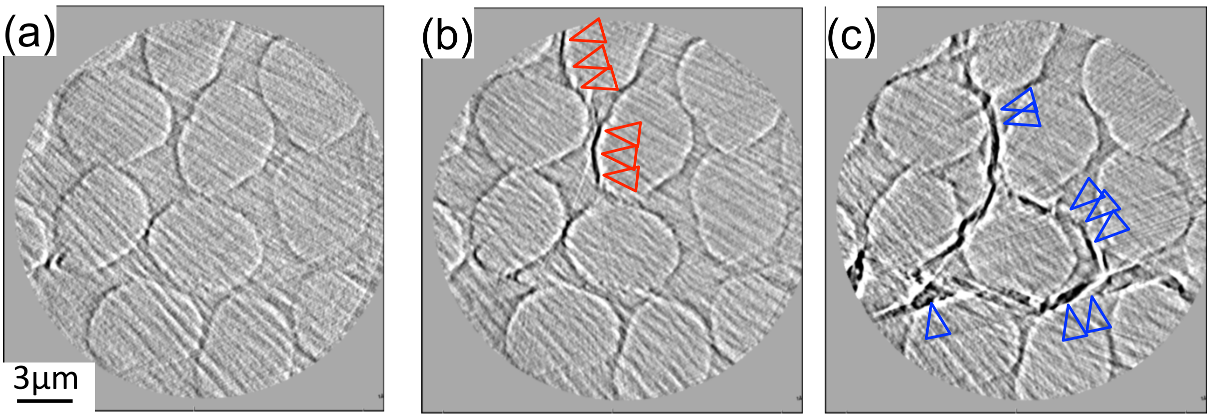

Figure 3. Cross-section images of CFRP under an applied stress by indentation, where typical crack initiations are shown by triangles. The insertion amount was increased from (a) to (c).

Macroscopically, the failure mechanism of CFRP has been primarily investigated based on mechanics and micromechanics, where the interactions of the constituent, carbon fiber, and polymer in the CFRP are examined on a microscopic scale.

Microscopically, for the first time, the new XRM using synchrotron radiation successfully showed the mechanisms of crack initiation and propagation. It showed that cracking was formed by (a) de-bonding at the fiber/polymer interfaces and (b) void formation in the polymer. The former factor is determined by the bonding energy between fiber and polymer, and surface treatments (sizing) of fibers as well as surface morphology may have large effects on the factor. The latter one is determined by localized stress, and it may largely effected by cross-section shapes of fibers and their alignments as well as the bonding energy between fiber and polymer. Nondestructive and 3D X-CT observations of crack initiation and propagation using XRM with synchrotron radiation will provides crucial information in order to understand these mechanisms.

4. Conclusions

We have succeeded in attaining nondestructive and 3D observations of their initiation and propagation with a resolution down to 50 nm under an applied stress. This first observation successfully showed that cracks are initiated by (a) de-bonding at the fiber/polymer interfaces and (b) void formation in the polymer and these factors may determine the propagation path.

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