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

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Summary: Nanoscopic crack initiation and propagation in CFRP under applied stress was observed *in-situ* using X-ray microscopy with synchrotron radiation. It was shown that (a) de-bonding along the carbon fiber/plastic interfaces and (b) cracking within plastic are competing and the features are largely location-dependent.

1. INTRODUCTION

Carbon fiber reinforced polymer (CFRP) composites are of growing use in aircraft owing to their high specific strength and stiffness. The micro-mechanism of damages and microscopic chemical properties of CFRPs are key to understanding the mechanical properties and durability of these materials. In particular, the initiation and propagation of cracks along fiber/polymer interfaces is of great importance. However, most experimental observations have been carried out for surfaces (cross sections) of CFRP specimens typically using an electron microscope, which cannot observe crack propagation along the fibers, i.e. along the thickness direction of a specimen. Recent reports pioneered non-destructive and three-dimensional (3D) observation using X-ray computed tomography (X-CT), but their spatial resolution was limited to several μm or just under 1 μm.

We have succeeded in non-destructive 3D observation of crack initiation and propagation with a resolution of less than 50 nm under an applied stress with a new X-ray transmission microscope (XRM) using synchrotron radiation.

2. EXPERIMENTAL METHOD

We utilized the transmission X-ray microscope (TXM) recently installed at the PF-AR NW2A beamline in IMSS, KEK, Japan [1-4]. The microscope (Xradia Ultra, Carl-Zeiss X-ray Microscopy, Inc.) [5] is a full-field type and is designed to work at the photon energy range of 5–11 keV. The X-ray beam from synchrotron radiation (undulator) is focused on the specimen by the condenser capillary. The zone plate and optical magnification allow the projection of the field of view (FOV) of 20–40 μm onto the 12-bit 2k × 2k CCD detector. The spatial resolution was confirmed to be <30 nm in 2D observation.

The nanomechanical testing apparatus features a high-precision piezo actuator and an integrated load cell, enabling the load-displacement curve to be measured during the X-CT measurements (Fig. 1(a))[6]. A cylindrical CFRP specimen (approximately 60 μm^D × 1000 μm^L) was indented with the diamond apex to initiate and propagate cracks, and X-CT measurements were carried out under the stress. The FOV was small enough to avoid the effects of stress release at the specimen surface. X-ray images were collected using Zernike phase contrast with a phase ring to obtain clear phase boundaries between fiber and polymer. Typical conditions were: rotation from −75° to +75° with a step size of 1°, exposure time of 10 s at each angle, and total measurement time of 25 min.

The [90/0]s laminate plates were fabricated from Toray carbon fiber prepreg. First, *in-situ* observation of crack formation was performed at macroscopic scale using the in-house X-CT with a spatial resolution of ~0.7 μm. A specimen with a gauge part of 1 mm^D × 1 mm^L was used. Then, after identifying macroscopic behaviors, *in-situ* observation of crack initiation was performed at nanoscopic scale using the TXM.

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Columnar specimens, with a size of ~60 μm in diameter and 1 mm in length, were mechanically cut along the fibers from the plate.

3. RESULTS

Figure (b) shows typical cross-sectional (on the X-Y plane) images of the 3D volume data obtained by X-CT at different locations on the Z-axis, corresponding to different strains. At a low strain (small Z), no clear crack formation was found. Further increase in strain (large Z) caused crack initiation at the fiber/polymer interfaces and/or inside the polymer. The initiated cracks traversed into the neighboring interfaces and/or within the polymer.

Macroscopic observation showed that transverse cracks were first initiated in 90° plies at ~40–50% of the fracture strength and their propagation was accelerated by increasing tension. Subsequently, transverse cracking in 90° plies induced splitting in central 0° plies. Then, the crack initiation in 90° plies was investigated at nanoscopic scale using TXM[2]. The *in-situ* observation showed that cracks were initiated according to two competing mechanisms which were largely location-dependent. In some regions, cracks were initiated and propagated by de-bonding along the fiber/polymer interfaces (shown by red triangles in Fig. 1(b)). In other regions, cracks were initiated and propagated within plastic (shown by blue triangles in Fig. 1(b)).

In-situ microscopic observation can improve our understanding of crack/void initiation and propagation mechanisms on multiple scales, which should facilitate the fabrication of CFRPs with reasonable safety margins for use in airplanes.

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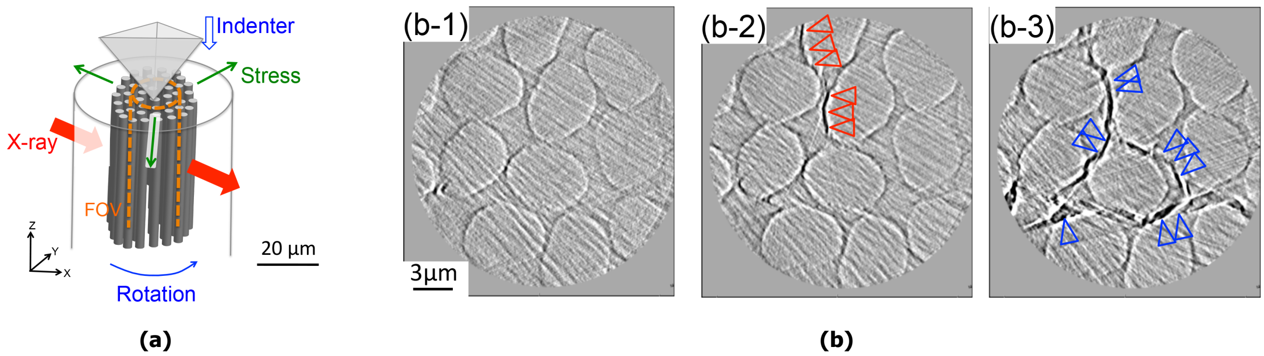

Figure 1: (a) Schematic of nanomechanical testing. (b) Typical cross-sectional (on the X-Y plane) images of the 3D volume data obtained by X-CT at the different locations on the Z-axis corresponding to (a) low, (b) middle, and (c) high strains. Red and blue triangles exemplify the crack initiation at the fiber/polymer interface and within the polymer, respectively.