# Fatigue Behavior of High-strength Fiber from Liquid Crystalline Polymer

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## 1 Introduction

Fatigue behavior of high-strength fibers from liquid crystalline polymers is considered to be completely different from that of general-purpose plastics because of the microfibrillar fiber structure of the former. This study investigates the progress of the fatigue damages for this type of fiber, poly-p-phenylene benzobisoxazole (PBO) fiber, based on the structure analysis using the synchrotron radiation small-angle X-ray scattering (SAXS) and the single-fiber axial compression test. The reason for performing the axial compression test is that the axial compressive strength is sensitive to the damages of the fiber structure. The details of the present study will be published in another paper [1].

#### 2 Experimental

A PBO fiber with the tensile strength of 5.8 GPa, the tensile modulus of 180 GPa, the strain-to-failure of 3.5 % and the density of  $1.54 \text{ g cm}^{-3}$  was used for the study.

For the specimens of SAXS measurements, tensilefatigue deformation was applied to the bundles of ten filaments of PBO fiber at the frequency 10 Hz with the maximum stress of 4.0 GPa and the minimum stress against the maximum stress ratio of 0.25. For the specimens of the single-fiber axial compression tests, tensile-fatigue deformation was applied to the single filaments of PBO fiber at the frequency 10 Hz with the maximum stress of 4.0 and 2.0 GPa and the minimum stress against the maximum stress ratio of 0.25.

For the original fiber without fatigue test and the fatigue tested fibers, the SAXS measurements were performed in the static state and during tensile deformation at the beamline BL6A of High Energy Accelerator Research Organization, Japan. The SAXS was detected using a CCD camera (C7300, Hamamatsu Photonics) attached with an image intensifier. The X-ray wavelength, the beam cross section sizes and the specimen to detector distance were 0.15 nm, 0.3 mm by 0.3 mm and 2.1 m, respectively.

The single-fiber axial compression tests were performed according to the method shown in a previous paper [2]. A single filament of the PBO fiber was bonded on a small steel plate using an adhesive and the compressive load was applied to the protruded fiber end with the length of about 16  $\mu$ m using an indenter having a mirror-finished surface.

### 3 Results and Discussion

Fig. 1 shows the SAXS pattern of the original PBO fiber without fatigue test where the fiber axis is in the upand-down direction of the figure. The scattering pattern in the form of the elongated equatorial streak indicates that the scattering bodies in the fiber have a large aspect ratio and their longitudinal directions are highly oriented in the fiber axis direction. In the spinning process of the PBO fiber, the PBO solution with nematic liquid crystalline characteristics is extruded into the solidification liquid during which the microfibrils and microvoids are formed [3, 4]. The scattering bodies in the PBO fiber which produces the SAXS pattern shown in Fig. 1 are not the microfibrils but the microvoids scince the volume fraction of the scattering bodies was measured to be as small as 1.4 %.

The azimuthal intensity distribution of the SAXS pattern is caused by the finiteness of the longitudinal microvoid length and the orientation distribution of the microvoids. The longitudinal microvoid length was determined from the dependence of the broadening of the azimuthal intensity distribution on the scattering angle.

The SAXS measurements were performed during



Fig. 1: SAXS pattern of original PBO fiber.



Fig. 2: Variation of axial microvoid length with fatigue cycle number for PBO fiber. Maximum stress applied during fatigue cycle was 4.0 GPa.

tensile deformation and the longitudinal microvoid length was determined as a function of the stress and the strain for the original PBO fiber. As a result, we found that the increase in the longitudinal microvoid length up to the stress of 3.8 GPa was 21 % which was significantly larger than the macroscopic fiber strain, 2.3 %, at the same stress. This indicates that the increase in the longitudinal microvoid length during tensile deformation is not caused by the affine-like deformation of the whole fiber but occurs due to the splitting of the microfibrils.

Fig. 2 shows the variation of the longitudinal microvoid length with the fatigue cycle number for the PBO fiber. We found that the longitudinal microvoid length is increased by the fatigue deformation.

By the single-fiber axial compression test, the PBO fiber failed accompanied by the formation of the kink band as shown in the SEM images of Fig. 3. Fig. 4 shows the single-fiber axial compressive strength of the PBO fiber fatigue tested with various fatigue cycle numbers. The PBO fiber fatigue tested with the maximum stress of 2 GPa (35 % of the tensile strength) during the fatigue cycle did not show a marked reduction in the compressive strength, while the fiber fatigue tested with the maximum stress of 4 GPa (70 % of the tensile strength) decreased 13 % at the fatigue cycle number of  $1.6 \times 10^4$ . The decrease in the axial compressive strength is caused by the increase in the longitudinal microvoid length. We concluded that for improving the fatigue resistance of the PBO fiber, the bonding strength between microfibrils needs to be increased.

### 4 Summary

The structure analysis using the synchrotron radiation SAXS and the single-fiber axial compression tests were performed for investigating the progress of the fatigue damages in the PBO fiber. The increase of the fatigue cycle number enhanced the splitting of microfibrils which increased the longitudinal microvoid length and as a result, decreased the single-fiber axial compressive strength. We concluded that for improving the fatigue resistance of the PBO fiber, the bonding strength between microfibrils needs to be increased.

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Fig. 3: SEM images of original PBO fiber (a) before and (b) after single-fiber axial compression test.



Fig. 4: Single-fiber axial compressive strength of PBO fiber fatigue tested with various fatigue cycle numbers. Maximum stress applied during the fatigue cycle was (□) 2.0 GPa and (○) 4.0 GPa.