DIRECT MEASUREMENT ON FRACTURE TOUGHNESS OF CARBON FIBER

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ABSTRACT

For the direct measurement of the fracture toughness of the carbon fiber, a new technique was proposed and examined its applicability. At first, machining condition of the notch was examined. The notch was introduced using focused ion beam (FIB). The ion beam can be electronically scanned to introduce a sharp notch on the carbon fiber. Notches with various notch width and length were introduced by changing beam and scanning conditions. Tensile tests on notched carbon fibers were carried out following the test method for carbon fiber monofilaments. Fractured specimens were successfully corrected without secondary damage using protection films. SEM observations revealed that a crack propagated from a notch-tip, and notch size was able to be determined successfully. Effect of notch root radius was also examined to investigate the validity of the fracture toughness obtained by this method.

1. INTRODUCTION

Fracture toughness of carbon fiber is of great interest as well as its strength, because it is essentially important when fracture behavior of composites was investigated. For example, fracture mode of brittle matrix composites are strongly affected by the fiber fracture toughness as well as its interface toughness [1]. The measurement of fiber fracture toughness is, however, thought to be difficult due to its small diameter. One of the practical ways to estimate fracture toughness is determination of the initial crack size by observing fracture mirror size on a tensile fracture surface of un-notched specimens [2]. Since the mirror size measurement involves a considerable error, estimated fracture toughness is usually scattered significantly. Accuracy of the measured fracture toughness will be significantly improved, if a notch is introduced intentionally by controlling its size and shape. In this study, therefore, a new measurement technique was proposed to estimate fracture toughness of the carbon fiber.

At first, applicability of the focused ion beam (FIB) system was examined as a machining tool of notches. The FIB is regarded as a useful machining tool to prepare samples for transmission electron microscope observations. Recent FIB systems have less than 10 nm beam diameter, achieving micro-machining by scanning the beam electronically. Various sizes and types of notches were tried to be introduced on the carbon fibers. Tensile tests were carried out on the notched carbon fibers to examine the validity of this technique.

2. EXPERIMENTAL PROCEDURE

2.1 Specimens Preparation

The carbon fiber tested in this experiment is a PAN based high strength type carbon fiber (IM-600; 6000 filaments) manufactured by *Toho Tenax Co. Ltd*, *Japan*. A carbon monofilament was extracted from a bundle, and was attached on a holder as shown in Figure 1. An aluminum holder and carbon tapes were used since electrical conductivity was required to introduce a notch by the FIB.

The notch was introduced using the FIB system (JFIB-2300; *JEOL Co. Ltd., Japan*) on a carbon fiber. Ga^+ ion beam was extracted from an ion source and accelerated with 30kV accelerating voltage. Various types of notches were introduced by changing a beam diameter and other machining conditions. Details of the conditions were shown in Table 1.

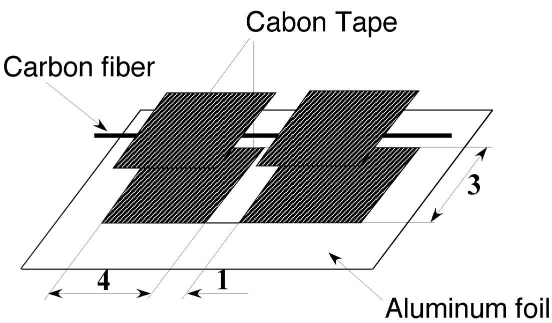

Figure 1: Schematic illustration of facture

Table 1: FIB machining conditions

Acceleration Voltage	30 kV
Extraction Voltage	5.58 kV
Emission Current	1.26 μA
Ion Beam Current	0.6 μA
Filament Current	1.62 A
Filament Voltage	0.58 V
Prove Current	1 pA
Beam Diameter	7 ~ 18 nm

2.2 Fracture Toughness Test

Figure 2 schematically shows a cross section of the carbon fiber on a notched plane. A notch was introduced by the FIB system on one side surface of the carbon fiber with various notch depths (a) as indicated by gray area in Figure2. Notched carbon fibers were tested in tension with crosshead speed of 0.1 mm/min. following the tensile test method of carbon fiber monofilaments. Plastic films were set on both sides of a carbon fiber specimen, and liquid detergent was filled between them to avoid secondary damage of the carbon fiber. After tensile tests, liquid detergent was removed by dipping in water. Fracture surface was observed by SEM to confirm a fracture position and to determine the notch length, a.

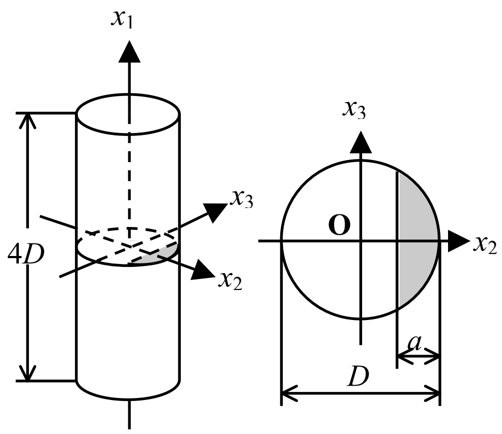

Figure 2: Schematic illustration of notched carbon fiber

3. Finite elements analysis

Energy release rate, G_I, at the center of the crack front was examined by the finite elements method using the virtual crack closure method (VCCM). Figure 2 shows the model of a carbon monofilament which has a straight edge crack from the surface located at its center. The model was analyzed as both an isotropic and an orthotropic materials. In the case of the isotropic material, Young's modulus (E) is set to be 285GPa, Poison's ratio (ν) 0.3. Table 2 shows the material properties used for the analysis in the case of the orthotropic material. In the case of isotropic material, the energy release rate (G_I) is converted to the stress intensity factor (K_I) by the following equation

$$G_I = \frac{K_I^2(1-\nu^2)}{E} \tag{1}$$

where assumption of plain strain condition in the neighborhood of the crack front is made. In the case of orthotropic material, following equation is used [3].

$$G_I = K_I^2 \sqrt{\frac{a_{11}a_{22}}{2}} \left[\sqrt{\frac{a_{22}}{a_{11}}} + \frac{2a_{12} + a_{66}}{2a_{11}} \right]^{1/2} \tag{2}$$

where

$$a_{11} = \frac{1}{E_{11}},\ a_{22} = \frac{1}{E_{22}},\ a_{66} = \frac{1}{G_{12}},\ a_{12} = a_{21} = -\frac{\nu_{12}}{E_{11}} = -\frac{\nu_{21}}{E_{22}} \tag{3}$$

Table 2 : Material properties used for orthotropic analysis

Young's modulus [GPa]	Poisson's ratio	Shear modulus [GPa]
E_{11} 285	ν_{12} 0.300	G_{12} 20.0
E_{22} 30.0	ν_{23} 0.400	G_{23} 10.7
E_{22} 30.0	ν_{31} 0.0316	G_{31} 20.0

The dimensionless stress intensity factor which is described by

$$f = \frac{K_I}{\sigma\sqrt{\pi a}} \tag{4}$$

where σ is the far field stress and a is the crack length [4]. Figure 3 shows the comparison between f calculated by equation (1) (isotropic case) and that in [4]. A good agreement is obtained which implies the accuracy of the present calculation. Figure 4 shows the comparison of f between isotropic and orthotropic cases. The dimensionless stress intensity factor is approximated as

$$f = \frac{K_I}{\sigma\sqrt{\pi a}} = 28.3\left(\frac{a}{D}\right)^3 - 15.0\left(\frac{a}{D}\right)^2 + 4.09\left(\frac{a}{D}\right) + 0.773 \tag{4}$$

in the isotropic case and as

$$f = \frac{K_I}{\sigma\sqrt{\pi a}} = 9.19\left(\frac{a}{D}\right)^3 - 2.87\left(\frac{a}{D}\right)^2 + 0.859\left(\frac{a}{D}\right) + 0.511 \tag{5}$$

in the orthotropic case. Fracture toughness was calculated by these approximations.

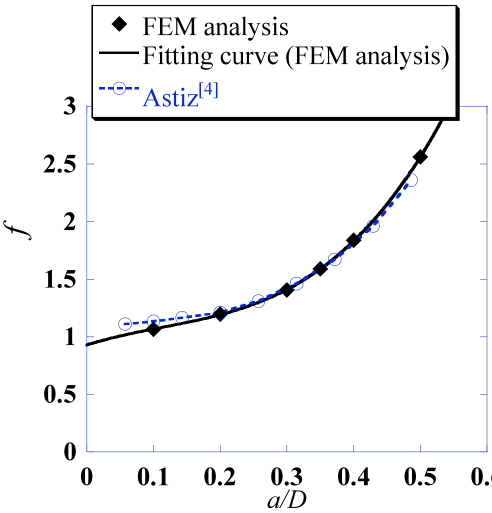

Figure 3: Comparison of the dimensionless stress intensity factor (isotropic)

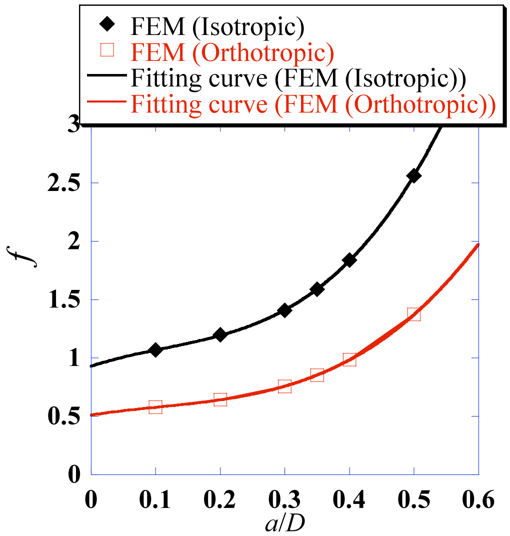

Figure 4: Comparison of the dimensionless stress intensity factors between isotropic and orthotropic cases

4. RESULTS AND DISCUSSION

4.1 Notch machining

Figure 5 shows typical outlooks of notched carbon fibers introduced with different beam condition. When the beam diameter of 7nm was used, a sharp notch was successfully introduced with notch-tip width of less than 50 nm as shown in (a). On the other hand, 360nm notch-tip width was obtained by changing a beam diameter (18 nm) and scan area as shown in (b). Various kinds of notches were introduced with different notch-width and notch-length.

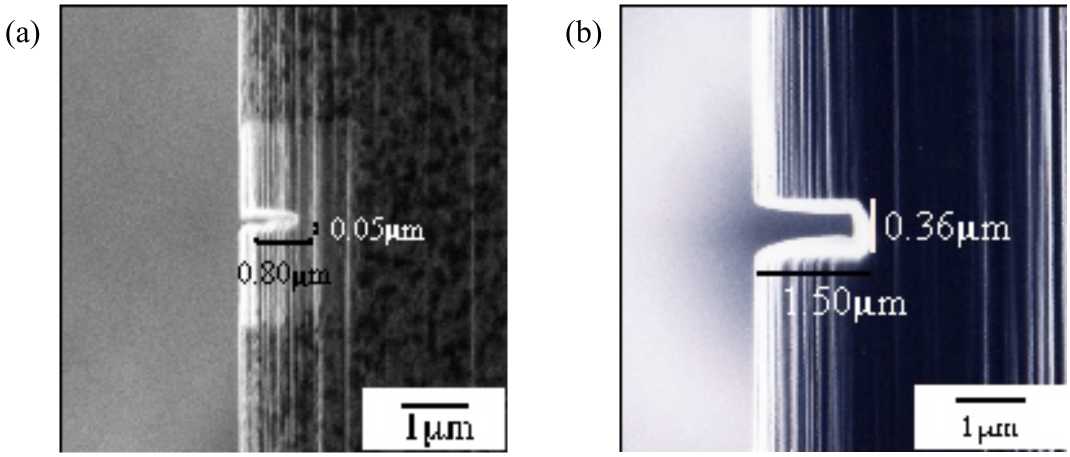

Figure5: Typical outlooks of notched carbon fibers
(a) introduced by the beam with 7 nm diameter,
(b) by the beam with 18 nm diameter

4.2 Tensile tests

A typical load-displacement (P-δ) curve was shown in Figure 6. The P-δ curve was linear up to the final fracture. In all tests, same behavior was observed. After tensile tests, specimens were successfully corrected without secondary damage and fracture surface could be observed as shown in Figure 7. A flat notch surface was clearly observed as indicated by a circle on the fracture surface. It was also observed that the notch front was straight, indicating machining by the FIB was applicable to the notch introduction. Notch length, a, was measured from the micrograph for every specimen.

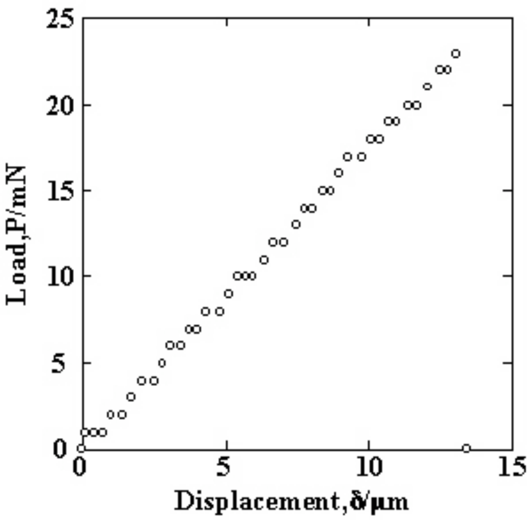

Figure 6: Typical Load-displacement curve of notched carbon fiber in tension test

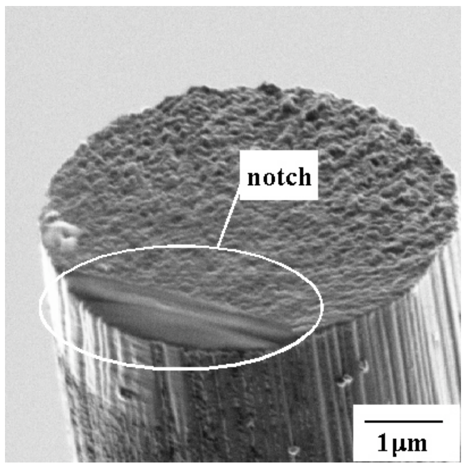

Figure 7: Typical Fracture surface of notched carbon fiber tested in tension.

On the fracture surface, hackle pattern was clearly observed from the notch tip. This means that the fracture of the notched carbon fiber originated from a notch-tip. It was confirmed with these observations that fracture toughness tests were successfully carried out with the present technique.

Based on the fracture toughness tests with various widths of notches, effect of notch width was examined as shown in Figure 8. Although K_C was somewhat scattered, it showed almost constant value of 1.6 $\text{MPa}\sqrt{\text{m}}$ (isotropic) and 1.0 $\text{MPa}\sqrt{\text{m}}$ (orthotropic) on average, when the notch-tip width was less than 0.2 μm. Since the fracture toughness of the PAN based carbon fibers were reported to be in the rage from 1~2 $\text{MPa}\sqrt{\text{m}}$ [2], present results is expected to give the reasonable value in this range of notch width. On the other hand, K_C linearly increased with notch-tip width, if the notch-tip width was more than 0.2 μm. These results suggest that the fracture toughness tests should be carried out with small enough notch-tip width to which calculated fracture toughness is insensitive.

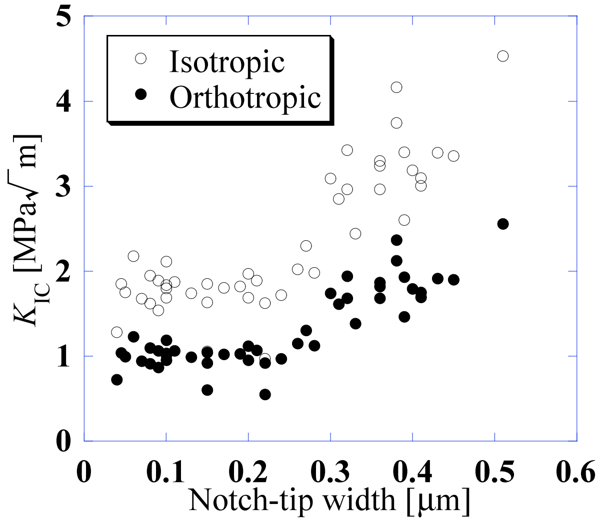

Figure 8: Effect of notch-tip width on fracture toughness of carbon fibers.

5. CONCLUSION

(1) Various types of notches with straight notch front could be successfully introduced on carbon fiber monofilaments using a focused ion beam machining system.

(2) Fracture surface observation revealed that fracture of notched carbon fibers originated from notch tip with hackle pattern, indicating present technique is applicable for fracture toughness tests of the carbon fiber.

(3) Effect of notch tip width was clearly shown that the fracture toughness tests should be carried out with small enough notch tip width to which calculated fracture toughness is insensitive.

(4) Stress intensity factor is calculated by using the VCCM technique as a function of

crack length for both isotropic and orthotropic carbon fibers. The fracture toughness of IM600 was estimated as 1.6 $\mathrm{MPa}\sqrt{\mathrm{m}}$ (isotropic), 1.0 $\mathrm{MPa}\sqrt{\mathrm{m}}$ (orthotropic) on average.

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