Elastic FEM Analysis of Fiber Push-Out Test for C/C Composites

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To study mechanical properties of fiber, matrix and their interface in C/C composites, fiber push-out test has been carried out. Where, to decompose the deformation, measured from indenter displacement, into local deformation is essential for mechanistic understandings. But up to now, there has been no systematic study on this effect. In this study, as the preliminary analysis of the push-out tests, those with a variety of groove width on specimen holder were performed and the elastic deformations of specimen were analyzed by a finite-element method (FEM). Materials used were unidirectionally reinforced C/C composites. Fiber push-out tests were carried out by utilizing dynamic ultra micro indentation test machine.

For the cases of 20 and 100 μm groove, plateaus which indicate the occurrence of fiber push-out could be seen in Load-displacement curves (indentation curves) and there was a difference in the duration of plateau. On the other hand, for all the cases, any difference could not be seen before plateaus. Now by using axis symmetrical FEM model, elastic deformation of C/C composites before push-out was calculated. From the results of calculation, the effect of groove width on indentation curves within elastic regime was considered to be little. But there was a large difference in the distribution of the stress transverse to carbon fiber direction on fiber-matrix interface between 20 and 100 μm , and this was considered to be the origin of the difference of the duration of plateau.

Keywords: C/C composite, micro indentation, elastic modulus, finite element method, push-out, carbon fiber, carbon, Young's modulus, composite

I. Introduction

Mechanical properties of interface between fibers and matrices are known to be quite influential in determining properties of fiber reinforced composites. Some of the previous reports have indicated the effectiveness of controlling fiber-matrix interface on improving properties of composite materials⁽¹⁾. So the effects of surface treatment or coating on fibers for several composites have been studied⁽²⁾ and many experiments have been carried out to measure the interfacial mechanical properties by various methods, those were pull-out test⁽³⁾, push-out test⁽⁴⁾, protrusion test⁽⁵⁾ and multiple fracture test⁽⁶⁾. And most of these studies have done by using simple model composites reinforced with single thick fiber. But practical carbon reinforced carbon (C/C) composites are reinforced with a large number of very thin fibers and have many pores and cracks in matrix⁽⁷⁾. Therefore, to measure the mechanical properties in the micro area is very difficult and the micro indentation test is considered to be one of the suitable test method.

The ultra micro indentation test machine, which allows

to locate the indenter with an accuracy of 0.5 μm , was newly developed⁽⁸⁾. This accuracy makes this apparatus possible to indent at the center of fibers with their diameters of about 10 μm . Fiber push-out tests were successfully performed with the apparatus under the condition with sufficiently thin specimen thickness. And in this test, to decompose the deformation measured from displacement of indenter into local deformation is essential for mechanistic understandings. But up to now, there has been no systematic study on this effect. So in this study, as the preliminary analysis of the fiber push-out tests for C/C composites, those with a variety of groove width on specimen holder were performed and the elastic deformations of specimen were analyzed by a finite-element method (FEM).

II. Experimental

1. Material and specimen

Material used was unidirectionally reinforced C/C composite, heat treated at 1873 K. Carbon fibers used were mesophase pitch-based fibers and their characteristics were shown in Table 1. Matrix precursor was a mixture of green coke and phenolic resin (80/20 in volumetric ratio). And a fiber volume fraction and a density of C/C composites were

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Fig. 1 Schematic flow of specimen preparation and shape of indenter tip.

Table 1 Characteristics of fibers used.

Diameter (μm)	9.8
Tensile Modulus (GPa)	542
Tensile Strength (GPa)	2.92
Heat Treatment Temperature (K)	2473

45.0 % and 1.69 Mg/m^3 , respectively.

Schematic flow of specimen preparation for fiber push-out test is shown in Fig. 1. C/C specimen, whose size was $20^l \times 5^w \times 1^t \text{ mm}$, was fixed at the center of acrylic pipe by resin. And next, the fixed specimen was sliced perpendicular to the fiber direction with about $200 \mu\text{m}$ thickness by low speed diamond saw. Then the thickness of sliced specimen was reduced $80 \mu\text{m}$ by mechanical grinding and the pipe and resin were removed in acetone. Finally the size of specimen for fiber push-out test became $5^l \times 1^w \times 0.08^t \text{ mm}$.

2. Fiber push-out test

Fiber push-out tests were carried out by utilizing the dynamic ultra indentation test machine. The specimen was placed across the groove, which was engraved by laser, on a specimen holder made of SUS and fibers located at the center of groove were loaded by diamond trigonal pyramid indenter tip. The groove width on specimen holder were chosen to be 0, 20 and $100 \mu\text{m}$ to inspect groove width effect on indentation tests. The shape of diamond trigonal pyramid indenter tip is shown in Fig. 1. The inclination angle of slide triangle was 68° , and the radius of curvature at ends was under $0.1 \mu\text{m}$.

This machine measures the displacement of indenter tip with controlling loading speed. In this study, constant displacement speed tests were applied from 0 load to presenting maximum load by controlling loading speed. Under this setting, a rapid increase of displacement of indenter tip is supposed to indicate a rapid increase of displacement without load change on a load-displacement curve (indentation curve), and the indentation curves of actual pushed out fiber showed such aspects.

III. Fiber push-out test

Typical examples of indentation curves under fiber push-out test are shown in Fig. 2. For the cases of 20 and $100 \mu\text{m}$ groove, plateaus which indicate the occurrence of fiber

Fig. 2 Indentation curves with several grooves.

push-out could be seen and there was a difference in duration of plateau. On the other hand, for all the cases, any difference could not be seen before plateaus.

If mechanical properties of specimen are near isotropic, the total flexural displacement of the specimen is supposed to increase with increasing the groove width at any load. So the displacement of indenter with $20 \mu\text{m}$ groove seems to be larger than that with $100 \mu\text{m}$ groove at same load, but the results did not show that phenomenon. The origin of this is considered to be a large anisotropy of mechanical properties of carbon materials. Especially, the longitudinal Young's modulus of carbon fiber is about fifty times of the radial Young's modulus⁽⁹⁾. Therefore, for the exact understanding of the indentation curves and the deformation of C/C composites under fiber push-out test, a FEM analysis seems to be needed.

IV. FEM analysis

1. FEM model

By using the axis symmetrical FEM model shown in Fig. 3, the deformations of C/C composites before fiber push-out were calculated. For the analysis of practical composites, the effect of adjacent fibers must be taken into account, but in this study, two dimensional models were used for the first step of FEM examination. So C/C composites reinforced with only single carbon fiber were used for this calculation. And in the most previous analysis of micro indentation, the extra interphase, which was made

Fig. 3 FEM model of indentation test.

from reaction of fiber and matrix, was set up independently⁽¹⁰⁾. But for C/C composites the interphase should be as same as matrix, so in this model the interphase was included into matrix phase and the matrix phase was assumed to be carbon layer. The width of this layer was assumed to be 2 μm , that reflects a fiber volume fraction of specimen for fiber push-out test correctly.

The indentation curve with 0 μm groove shown in Fig. 1 showed a large hysteresis loop, that indicated the existence of some energy loss under indentation test without fiber push-out. So some plastic deformation or production of micro cracks were supposed to occur before fiber push-out. But any traces were not shown on specimen surfaces after indentation tests with and without groove⁽⁸⁾, that means no occurrence of plastic deformations in C/C composites under these indentation tests. So in this FEM analysis, only the elastic deformations of specimen were considered. And from the indentation curves, fiber push-out started over 2.5 μm displacement of indenter, so elastic FEM analysis were carried out until 2.5 μm displacement.

Indenter tip was assumed to be a cone with 136 degrees of the apical angle and the 0.1 μm radius of curvature at ends. The specimen holder and the indenter tip were presumed to be rigid. The condition of FEM analysis is as shown in Table 2. The longitudinal Young's modulus of carbon fiber and the Young's moduli of C/C composite were determined from the measured values⁽¹¹⁾. The radial Young's modulus of fiber and the isotropic Young's modulus of matrix was assumed to be same as the transverse Young's modulus of unidirectional C/C composites. And the Poisson's Ratio and the shear modulus were determined from the previous researches⁽¹²⁾ except for ν_{xy} and ν_{zx} of fiber, which was decided in reference of the Poisson's Ratio of composite and fiber.

Table 2 Condition of FEM model.

Analysis Model	Axis Symmetrical FEM Model
Specimen Size :	Radius 2500 μm , Thickness 80 μm
Radius of Fiber :	5 μm
Indenter Tip :	Cone with 136° of the Apical Angle (0.1 μm radius of curvature at ends)
Radius of Groove :	0 or 10 or 50 μm
Thickness :	2 μm (from Fiber Volume Fraction) of Carbon Layer
Elastic Properties of Each Element	
Fiber :	E_x :500GPa ν_{xy} :0.01 G_{xy} :10GPa E_y : 8.9GPa ν_{yz} :0.20 G_{yz} :20GPa E_z : 8.9GPa ν_{zx} :0.01 G_{zx} :10GPa Density 2.08 Mg/m ³
Carbon Layer :	E : 8.9GPa ν : 0.30 Density 1.70 Mg/m ³
C/C :	E_x :150GPa ν_{xy} :0.20 G_{xy} :8.9GPa E_y : 8.9GPa ν_{yz} :0.30 G_{yz} :15GPa E_z : 8.9GPa ν_{zx} :0.20 G_{zx} :8.9GPa Density 1.68 Mg/m ³

Fig. 4 Indentation curves with several grooves calculated by FEM analysis.

2. Analysis of indentation curve

The indentation curves with 0, 20 and 100 μm grooves calculated from FEM model are shown in Fig. 4. The absolute values of load were very different from the experimental data. One of the reason of this difference is considered to be a non-linear Young's modulus of carbon fiber⁽¹³⁾. In this analysis tensile Young's modulus of fiber was used as E_{xx} , but for indentation test the fibers were mainly received the compressive stress. So E_{xx} should be decided as the compressive Young's modulus of fiber which has been reported to be smaller than tensile Young's modulus⁽¹⁴⁾. Another considerable reasons are differences of shape on indenter tip and grooves. In experiment, the indenter tip was trigonal pyramid and the shape of groove was linear. However, in two dimensional FEM analysis, the former was a cone and the latter was circular. And more important reason is supposed to be the absence of plastic deformation or cracks in this analysis. As written in section IV-1, the experimental indentation curves showed

Fig. 5 Effect of groove width on displacement- x at center of fiber with 2 μm depth of indenter.

hysteresis loops that means the existence of some energy loss. So for more exact analysis, some improvements, that are, for examples, the considerations of the debonding at interface, some initial cracks or pores and the residual stress, would be necessary.

On the other hand, the results of groove width effect on the indentation curves showed a good agreement between the experimental and calculated data. The effect of groove width on displacement- x at center of fiber with 2 μm depth of indenter is shown in Fig. 5. This figure seems to indicate that only the deformation of fiber adjoin indenter tip was very large and the total deformation of C/C composite was little. So the effect of groove width on the indentation curves is considered to be little.

3. Distribution of stress under fiber push-out test

The distributions of stress transverse to carbon fiber direction (denoted as stress- yy) with 2 μm depth of

indenter are shown in Fig. 6. The left end of each figure is the center of carbon fiber and indenter tip. The bright area indicates the positive stress- yy to y -axis direction, the dark area suggests the negative stress- yy . So compressive stress transverse to fiber direction was applied on the interface at the upper of specimen, and on the other hand, tensile stress at the lower. And there was a little difference in the distribution of stress- yy between 0 and 20 μm groove, but there was a very large difference between 20 and 100 μm .

Concerning to fiber push-out, the distribution of stress on fiber-matrix interface is very important, so the distributions of stress parallel (denoted as stress- xx) and transverse to fiber direction on interface with 2 μm depth of indenter are shown in Fig. 7 and Fig. 8, respectively. The changes of stress- yy under 10 μm depth from specimen surface were very large. But in this analysis the shape of indenter tip was not trigonal pyramid, so the deformation of specimen adjoin indenter tip was supposed not to be calculated correctly. Therefore in this study the distributions of stress over 10 μm depth from specimen surface were noticed.

The difference between the tendencies of stress- xx with 20 and 100 μm groove was little. But in the case of stress- yy over 70 μm depth from specimen surface, the very large difference could be seen. To put it concretely, the stress- yy on interface with 20 μm groove decreased from 70 to 75 μm depth and increased to 80 μm , but this change was very small in comparison with the change with 100 μm groove. And the positive stress- yy on interface is considered to operate on breaking the bond between fiber and matrix. So over 70 μm depth from specimen surface, the debonding of fiber-matrix interface with 100 μm groove seems to occur earlier than with 20 μm groove, and when fiber push-out starts the real length of the bonding interface with 100 μm

Fig. 6 Distribution of stress- yy (displacement of indenter = 2 μm)

Fig. 7 Distribution of stress-xx on interface (displacement of indenter = 2 μm)

groove is supposed to be shorter than with 20 μm. Therefore, the frictional resistance of 100 μm groove on interface in the sliding of fiber seems to be smaller than 20 μm and this difference of the friction resistance is considered to be one of the origin of the difference on the duration of plateau in Fig. 2.

V. Conclusions

As the preliminary analysis of the fiber push-out tests for C/C composites, those with 0, 20 and 100 μm groove on specimen holder were performed and the elastic deformations of specimen were analyzed by FEM. The conclusions could be summarized as follows.

(1) For the cases of 20 and 100 μm groove, plateaus which indicate the occurrence of fiber push-out could be seen in the indentation curves, and there was a difference in the duration of plateau. On the other hand, for all the cases, any differences could not be seen before plateaus.

(2) The results of elastic axis symmetrical FEM analysis seems to indicate that only the deformation of fiber adjoin indenter tip was very large and the total deformation of C/C composite was small. So the effect of groove width on the indentation curves within elastic regime is considered to be little.

(3) From the results of FEM analysis, there was a large difference in the distribution of the stress transverse to carbon fiber direction on fiber-matrix interface between 20 and 100 μm groove, and this difference is considered to be

Fig. 8 Distribution of stress-yy on interface (displacement of indenter = 2 μm)

one of the origin of the difference on the duration of plateau.

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