DIRECT EVALUATION OF AXIAL COMpressive PROPERTIES OF CARBON FILAMENT

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SUMMARY: In order to utilize the fibers’ high performance, axial compressive properties of carbon fibers were measured. Direct compressive test of single filament and in situ observation were applied to obtain more reliable data than indirect methods. For such test, new apparatus was composed that could be operated under optical microscope.

Pitch- and PAN-based fibers with almost the same modulus were tested. Pitch-based fiber broke with shear mode when gauge length-diameter ratio was less than 10. However, almost all the tests of PAN-based fibers showed buckling mode even with the same gauge length-diameter ratio. They were very similar to failure appearances of the recoil test. Obtained compressive strength was 0.62 GPa for pitch-based fiber and 0.93 GPa for PAN-based fiber. Both were different from what was expected from single filament three point bending test results.

KEYWORDS: Axial compressive test, single filament, in situ observation, failure mode, compressive strength.

INTRODUCTION

Composite materials are now widely used in many fields such as aerospace field, sports goods, building materials, etc. It is clear that precise design of the composite takes the biggest advantage of its constituents. Properties of fiber reinforced composites are governed by those of fibers, matrices and interfaces or interphases. Carbon fibers, which are well known as reinforcement of composites, have high specific tensile strength and specific tensile modulus. Here, it should be reminded that these properties are only one aspect of the material. For the precise design, stresses in all direction should be considered. This means that even the properties of reinforcement should be known in every direction to utilize its advantages. Precise and reliable evaluation in every direction will make the precise design of composites possible and it will lead to the improvement of the performances of the composites.

During the last decade, some efforts have been made to evaluate other properties than axial tensile ones for carbon fibers. Torsional test [1], bending test [2-4], transverse compressive test [5, 6] or axial compressive test [6-10] have been carried out to evaluate each property and
understand the relation between fiber properties and their structures. Axial compressive properties, which have been said to be performance-limiting factor of carbon fibers, have been comparably well studied among them. Considerable efforts have been made for it and some unique test methods were proposed, such as composite test [7], loop test [9], recoil test [8] or apparently direct methods [6, 11]. Though every method is well considered to avoid difficulties in compressive test for very small material, there still exists a very big problem, that is, obtained results vary from one method to another. Such incoincidence may be caused by various reasons such as analytical assumptions. Therefore, we tried to reduce indirect and unknown effects as much as possible in axial compressive test for single carbon filament. In our method introduced in this report, only one filament is directly compressed without any constriction around the filament and compressive force is also directly measured by load cell. The new apparatus will be introduced and failure mode and the results will be shown.

EXPERIMENTAL

Apparatus

High performance carbon fibers’ diameter is 6-10 µm. It means that the gauge length should be much shorter than usual tensile test to get rid of fiber buckling. As long as the gauge length is short, the displacement should be also small. Even a small backlash becomes a serious problem. Therefore, smooth and accurate displacement of the head in the loading direction is necessary.

In order to satisfy such requirements, a new apparatus as shown in Fig. 1 was composed. The bases on which specimen fibers should be bonded were made of stainless steel and their edges were etching treated to be sharp. These stainless steel bases can be changed because fiber is bonded on the base with epoxy resin on each test.

One base was connected to drive actuator and the other was connected to load cell directly. In order to achieve smooth displacement and to avoid off-axis loading, 3D-positioning system (Tritor 3D 100NV of Piezoelectric jena) was used as drive actuator. As this positioning system uses piezoelectric actuators as driver parts that don’t drive mechanically, we need not worry about backlash and we can do fine adjustment because of its three dimensional movement. This actuator supports about 100 µm movement in three directions without applied force.

Carbon filament was cut in short on the stainless steel bases and was arranged to make the fiber axis direction parallel along load measuring direction. This arrangement was operated under optical microscope with micro manipulators (MO-302 and MMO-22 of NARISHIGE). Small amount of epoxy resin was put on the specimen to bond to the bases. The resin was expanded until the end of the base so that the interval of the bases should be close to the real gauge length. These operations were also done with micro manipulators under optical microscope.

By increasing the applied voltage to the actuator, which caused movement of the base, compressive strain was induced to the specimen filament. During the test, the compressive force in the fiber axis direction was measured by load cell and the specimen was observed with CCD video camera and digital camera to be recorded.
Fig. 1: Schematic of the apparatus for axial compressive test of single carbon filament. Gray arrows show the directions that adjustment of the positions is possible. This system can be set under optical microscope, which enables in situ observation.

**Fibers**

Used carbon fibers for the test were pitch-based CF-P and PAN-based CF-A which have almost the same tensile modulus. Diameter of each filament for the test was measured using laser diffraction [12].

**RESULTS AND DISCUSSION**

**Failure mode**

It is clear that fiber buckling occurs when the gauge length is long. By carrying out tests, three types of failure was observed under optical microscope. One was an apparently shear failure. Second one was bending or buckling failure. Third one was the failure in the bonding resin and could not be identified clearly. The first one was often seen for CF-P. Observed appearances under optical microscope before and after failure were shown in Fig. 2 (a) and (b). In this case, compressive force increased almost linearly with the increase of applied voltage to the actuator until the peak. The second one was often seen for CF-A. Typical failure appearances were shown in Fig. 2 (c) and (d). Here, increasing rate of measured compressive force decreased near the peak point. Fiber’s deformation could be observed under optical microscope.

Failure part was observed also under scanning electron microscope (SEM), too. Failure part of CF-P, which showed apparently shear failure under optical microscope, showed two stepwise appearances. This phenomenon reflects properties of mesophase pitch-based carbon fiber that graphite crystallite is large in the fiber direction and the longitudinal cross section will be weak. This may be the reason to cause not one flat face inclined to fiber direction but stepwise fracture surface.
Fig. 2: Optical microscope images to show fracture appearances. (a) and (b) for pitch-based CF-P are showing shear failure mode. (c) and (d) for PAN-based CF-A are showing buckling failure mode.
Almost all the tests showed buckling failure for CF-A. Fracture surface observed by SEM showed very similar one proposed by Dobb et al. [13] for recoil test. That is, one large flat surface in the tensile side and kinking portion at the compressive side. According to the three point bending test for these fibers, flexural modulus of CF-A was higher than that of CF-P though their tensile moduli were almost the same [4]. From this result, CF-A was considered to be more difficult to buckle than CF-P. However, the observation of compressive test showed the opposite manner.

In order to investigate the effect of gauge length, failure mode of each test was shown in Fig. 3 according to its gauge length-diameter ratio. According to Macturk et al. [11], pure compressive failure is achieved only in columns that have a length equal to or less than 10 times the least lateral dimension. Their tests were carried out for high performance mesophase pitch-based fibers. The failure they showed looked like shear failure. Considering that point, it can be said that almost the same result was obtained for CF-P about the effect of gauge length. However, about one third of the tests with 5-10 gauge length-diameter ratios showed buckling failure in our tests. Therefore, it is more preferable to test with less than 5 of gauge length-diameter ratio in order to avoid buckling failure for CF-P. For CF-A, almost all the tests have shown buckling failure even if gauge length-diameter ratio is smaller than 10.

![Fig.3: Failure mode and failure stress of each sample according to the gauge length-diameter ratio of its test. Closed symbols show the data of CF-P and open symbols show CF-A. Circles indicate the apparently shear failure mode and triangles indicate buckling mode. Squares show the failure in the bonding resin.](image)

**Compressive strength**

The compressive strength was evaluated by defining it as the force at failure divided by cross sectional area. It was calculated for the specimens showed apparently shear failure for CF-P and both shear and buckling failures for CF-A. Though there was much scattering as seen in Fig. 3, the averaged compressive strength was 0.62 GPa for CF-P and 0.93 GPa for CF-A. They were listed with other fibers’ properties in Table 1. CF-P data are rather similar to the reported ones with recoil method [14] or direct method [11] for a fiber data with similar tensile
modulus. As for PAN-based fiber, data vary very much and do not agree with each other [10, 14].

Table 1: Properties of used fibers. CF-P is a pitch-based fiber and CF-A is a PAN-based fiber. Compressive strength was calculated from shear failure mode samples for CF-P and shear failure and buckling mode samples for CF-P.

<table>
<thead>
<tr>
<th>Fiber</th>
<th>CF-P</th>
<th>CF-A</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diameter [µm]</td>
<td>10.0</td>
<td>6.7</td>
</tr>
<tr>
<td>Tensile strength [GPa]</td>
<td>3.12</td>
<td>3.43</td>
</tr>
<tr>
<td>Tensile modulus [GPa]</td>
<td>402</td>
<td>431</td>
</tr>
<tr>
<td>Compressive strength [GPa]</td>
<td><strong>0.62</strong></td>
<td>(0.93)</td>
</tr>
</tbody>
</table>

From the three point bending test, more than 2 GPa of compressive strength for both fibers had been expected because the evaluated flexural strength was 2 GPa for CF-P and more for CF-A. Both compression and tension in axial direction were loaded in the three point bending test. These axial stresses distribute in the transverse cross section. However, only axial compressive stress is applied in the whole transverse cross section in the compressive test. This difference of the stress distribution in the transverse cross section will affect to these large differences of compressive strengths.

CONCLUSIONS

In order to evaluate more accurately and to utilize the performance of carbon fibers in composites, direct axial compressive test was carried out for single carbon filament. New apparatus was composed for the test. It uses piezoelectric actuators to drive that ensures smooth and fine displacement. Two types of carbon fibers, pitch-based and PAN-based ones, were tested with the apparatus under optical microscope. Pitch-based fiber fractured with shear mode when gauge length-diameter ratio was less than 10. However, almost all the tests of PAN-based fibers showed buckling mode even with the same gauge length-diameter ratio. Compressive strength of pitch-based and PAN-based fibers were 0.62 and 0.93 GPa, respectively. Pitch-based fiber’s strength was similar to the obtained value by recoil method. However, both were quite different from what had been expected from the three point bending test.

REFERENCES


