EVALUATION OF THE INTERFACIAL PROPERTIES IN AN ARAMID FIBER/EPOXY MODEL COMPOSITE USING MICRO-RAMAN SPECTROSCOPY

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ABSTRACT

A single-fiber pull-out model composite for an Aramid/epoxy system was specially designed to measure the stress distribution of the Aramid fiber embedded in the matrix using micro-Raman spectroscopy. The stress transfer length of the fiber was about 300 μm. Stress distribution in the fiber changed after the onset of the fiber/matrix interfacial debonding and the fiber axial stress showed the maximum at a certain point in the debonded region, which was larger than the applied fiber stress. As the applied stress was decreased step by step after the debonding, this larger stress peak was considered to be attributed to the residue of the previous stress distribution caused by the fiber/matrix interfacial friction on the debonded interface.

1 INTRODUCTION

Investigation of the fracture strength and fracture mechanism of the fiber/matrix interface is extremely important, because the mechanical properties of the fiber reinforced composites depend strongly not only on the properties of the fibers and the matrix but also on the fiber/matrix interfacial ones. We carried out the single fiber pull-out tests to evaluate the influence of water absorption on the interfacial properties of Aramid/epoxy composite (Tanaka [1]). In this study, however, the pull-out load, at which the unstable crack propagated through the total embedded fiber length, was used for the measure of the interfacial strength and the stress distribution along the fiber in the matrix was not clarified. Moreover for the study of interfacial crack propagation under fatigue loading, it is extremely important to understand the stress distribution in the fiber (Minoshima [2]).

Raman spectroscopy is a new technique to directly measure the strain or stress distribution along the fiber embedded in a matrix (e.g. Galiotis [3], Patrikis[4], Cervenka[5]). In this work, a single-fiber pull-out model composite for an Aramid/epoxy system was specially designed and micro-Raman spectroscopy was used to clarify the difference of stress distribution along the fiber before and after the interfacial debonding.

2 EXPERIMENTAL PROCEDURES

The technique to measure the stress of the fiber using laser Raman spectroscopy is based on the fact that the Raman frequencies, which is obtained when the laser is introduced to the fibers, are strain (stress) dependent. Therefore the calibration curve of the peak wavenumber of Raman spectrum vs stress of a fiber has to be obtained. Using this calibration curve, the measured Raman

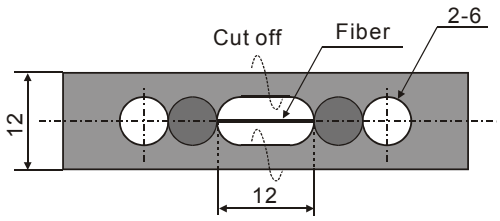
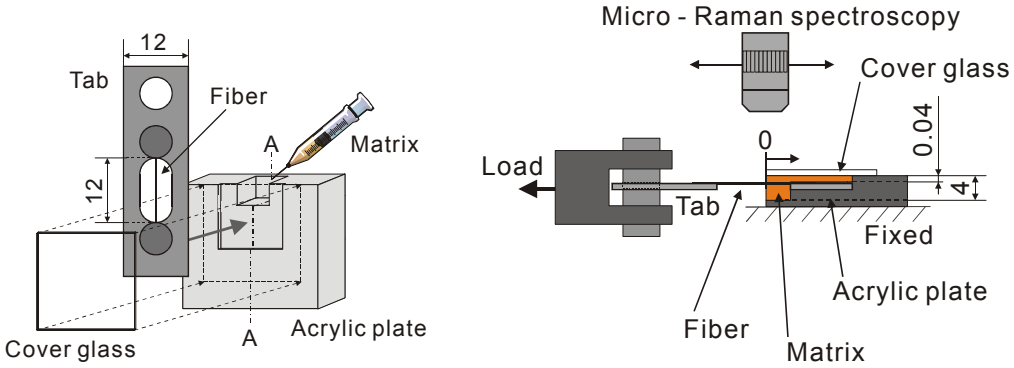


Fig.1 Single fiber tensile test specimen. All dimensions are in mm.



(a) Specimen construction. (b) A-A cross section in (a).

Fig.2 Pull-out specimen. All dimensions are in mm.

peak wavenumber is converted to axial stress. In this study, the Raman spectra were obtained by means of a laser Raman micro spectrometer (Japan Spectroscopic Co. Ltd, NRS-2000). The 514.5nm line of an argon-ion laser was used and the laser beam was focused to a 1.5μm spot on the fiber by an optical microscope.

The Aramid fiber used for single fiber tensile tests was Kevlar 49 manufacture by Du Pont, USA. The fiber had an average diameter of 12 μm. Specimens were prepared by following the recommended testing procedure as descried in ASTM D3379/JIS R7601. Polyester thin film was used for a tab and a single fiber was glued to it, giving a gauge length of 12 mm, as illustrated in Fig.1. Quasistatic tensile test was carried out using a tensile testing machine with a load cell (Capacity: 1N), which can be installed in the laser Raman micro spectrometer. After chucking a specimen, the tab was cut and the load was applied to the fiber.

The specimen construction for a single fiber pull-out specimen is shown in Fig.2. The polyester tab with a fiber, which was manufactured following the procedure of the single fiber tensile test specimen, was glued to the machined acrylic plate shown in Fig.2(a) and covered with cover glass. An epoxy resin Epikote 828 (Yuka-Shell Epoxy Co. Ltd.), curing agent Rikacid MH-700 (New Japan Chemical Co. Ltd.) and accelerator U-CAT 18X (SAN-APRO Co. Ltd) were used in 10:8.6:0.5 weight ratio. The resin was injected into the cavity between the acrylic plate and the cover glass and the specimens were placed in an oven for 2 h at 100°C and then for 15 h at 150°C. Pull-out tests were performed using the tensile testing machine in the laser Raman micro

spectrometer. Raman spectrum was measured along the fiber for both the embedded fiber and the free fiber at several stress levels. In this study the stress of the fiber adopted was the nominal stress, which was calculated from the value of the load cell.

3 EXPERIMENTAL RESULTS AND DISCUSSION

Raman spectra of a Kevlar 49 single fiber without stress and with 2.85GPa tensile stress are shown in Fig.3. The increase in tensile stress results in a clear shift of the spectrum to a lower wavenumber. In this study, a strong band around 1620cm^{-1}, which corresponds mainly to the phenyl ring/C-C stretching, was used to evaluate the stress.

The influence of the applied load on the Raman peak position for a Kevlar 49 single fiber is shown in Fig.4. Five specimens were tested and each result was shown by the same symbol. The approximately linear decrease in the Raman peak position with stress was found, irrespective of the specimens. Therefore, the liner regression line for the measured results shown in the figure was used to estimate the tensile stress of the embedded fiber.

The stress distribution of the axial tensile stress of the fiber during the pull-out test was shown in Fig.5. Zero of the distance along fiber is the meniscus point from which the fiber was embedded in the epoxy matrix and the fiber at the positive value of the distance along fiber was embedded in the resin matrix; the fiber axial stress at the negative value of the distance shows the results of the free fiber. For specimen A, shown in Fig.5(a), the stress distribution was measured at the applied stress of 0, 0.81, 1.75 and 2.27 GPa. When the applied stress was increased after measuring the stress distribution of the fiber at 2.27 GPa, the fiber was fractured. For specimen B, shown in Fig.5(b), after measuring the stress distribution at the applied stress for the fiber of 0 and 1.52 GPa, the applied stress was increased. When the applied stress was about 2.5 GPa, the fiber/matrix interfacial debonding occurred. To avoid the propagation of the interfacial crack, the applied stress was step by step decreased and the stress distribution was measured, as shown in the figure.

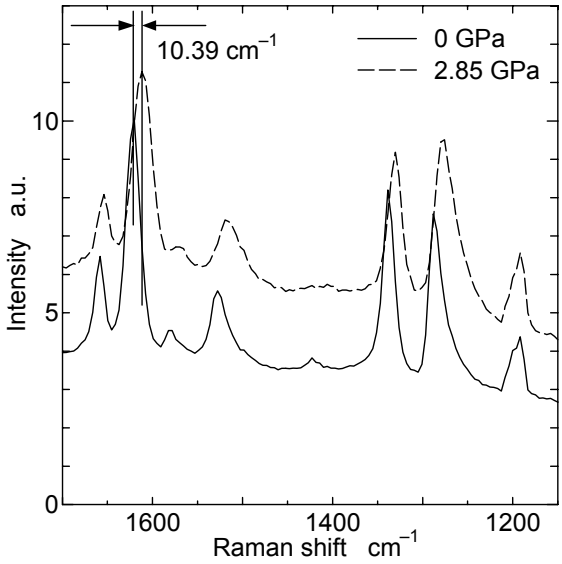


Fig.3 Examples of the Raman spectrum of Kevlar 49.

Before the initiation of the fiber/matrix interfacial debonding, the stress of the embedded fiber decreased gradually along the fiber from the meniscus point, which means that the stress was transferred from the fiber to the matrix. Considering from the fact that the fiber axial stress was almost 0 GPa at the distance of about 300 μm, the stress transfer length was about 300 μm for the Aramid/epoxy system adopted in our study. The stress distribution after the fiber/matrix interfacial debonding differed from that before the debonding. The fiber axial stress in the debonded region had the maximum at a certain point, which was higher than the applied stress. This higher stress was measured during the pull-out test while the applied stress was decreased step by step after the fiber/matrix interfacial debonding. This larger stress peak was considered to be attributed to the residue of the previous stress distribution caused by the fiber/matrix interfacial friction on the debonded interface. This speculation was supported by the fact that the friction stress plays an important role in the propagation of the fiber/matrix interfacial debonding and the propagation rate of interfacial debonding slowed down and retarded when the a constant fatigue load was applied to the specimen (Minoshima [2]).

4 CONCLUSIONS

Stress distribution along the fiber of an Aramid fiber/epoxy model composite during the pull-out test was measured using micro-Raman spectroscopy. The stress transfer length of the fiber was about 300μm. Stress distribution of the fiber changed after the fiber/matrix interfacial debonding and the fiber axial stress showed the maximum at a certain point in the debonded region, which was larger than the applied fiber stress.

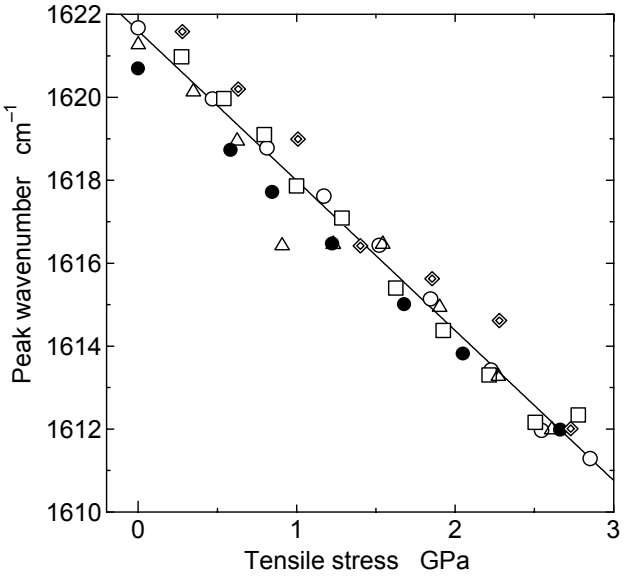
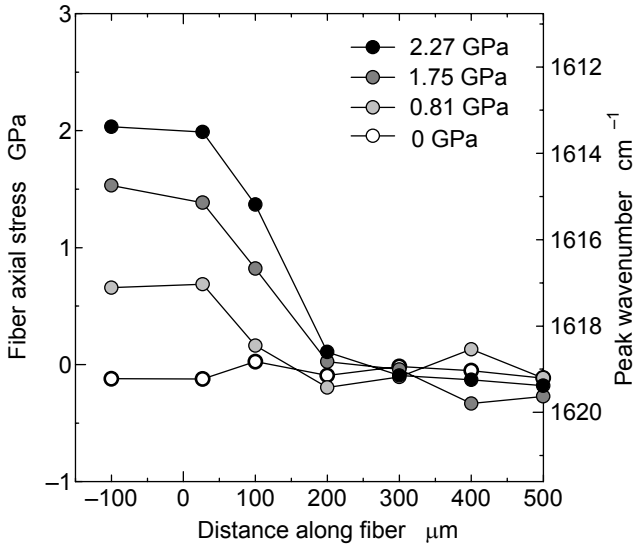
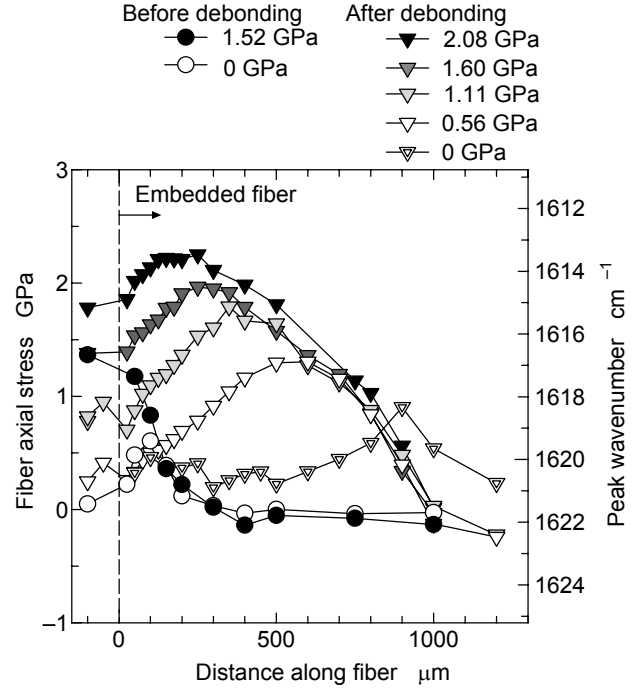


Fig.4 Relationship between tensile stress of the Aramid single fiber and peak wavenumber.



(a) Specimen A.



(b) Specimen B.

Fig.5 Axial fiber stress distribution during the pull-out test.

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