Evaluation of Interface Strength Properties in Vicinity of Fiber-Matrix Interface in Ceramic Matrix Composites

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The strength properties of ceramic matrix composites (CMCs), which have been expected to be applied to high-temperature section parts of aircraft engines, are greatly affected by the fiber-matrix interface strength properties. However, there are few reports on the interface strength properties of small diameter fibers used in CMCs. In this paper, we investigated the appropriate device, indenter shape and thickness of the test specimen for the push-out test, one of interface strength property tests, to establish an interface strength property evaluation method applicable to CMCs consisting of small diameter fibers approximately 10 µm. As a result of the test, it was found that the interface strength properties of CMCs with a thickness of 116 µm or less can be evaluated by using a nano-indenter with a round end cone shape indenter. However, the push-out test is not enough to be a quantitative test method so far in that the plumbness of fibers to specimen thickness cannot be considered.

1. Introduction

Replacing metallic materials with composite materials in aircraft parts can contribute to weight saving in the aircraft because composite materials are superior to metallic materials with respect to specific strength and specific stiffness. This weight saving is expected to improve fuel economy and reduce environmental load. Therefore, starting from the early 1980s, carbon fiber reinforced plastic (CFRP) has been proactively used in the airframes and low-temperature section engine parts of civilian aircraft$^{(1), (2)}$. Furthermore, in 2016, ceramic matrix composites (CMCs), which are lighter and have higher heat resistance than current nickel (Ni)-based alloy, were used for the first time in the high-temperature section parts of the engines of civilian aircraft$^{(3), (4)}$. There has been active movement towards expanding the application of CMCs to a wider range of parts in order to further improve aircraft fuel economy.

CMCs are made up of three components, i.e., fiber, interface coating layer, and matrix. **Figure 1** shows schematic images of the structure of a CMC. Although each of the three components is a brittle ceramic, the CMC as a whole has high toughness. This is because, as shown in **Fig. 1**, the brittle interface coating layer on the surface of the fibers deflects cracks, which are propagating from the matrix, along the surface of those fibers, thereby inhibiting continuous crack development$^{(5)-(8)}$. In this way, the interface coating layer has a large effect on crack development behavior in the vicinity of the fiber-matrix interface, and therefore constitutes an important factor controlling the strength properties of CMCs. Therefore, accurately assessing strength properties in the vicinity of the fiber-matrix interface is very important to understanding the fracture and strength properties of CMCs.

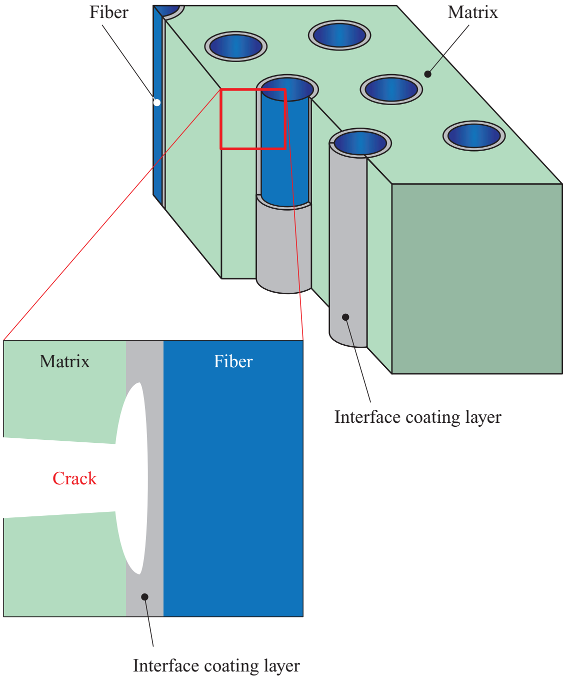


Fig. 1 Schematic images of micro-structure of CMC and crack deflection behavior

Figure 2 shows schematic images of test methods for measuring interface strength properties$^{(9)}$. There are several methods for evaluating the interface strength properties of composite materials$^{(9)-(14)}$. The multiple fracture and push-in tests evaluate them indirectly using the volume fraction and Young's modulus values of the respective phases. However,

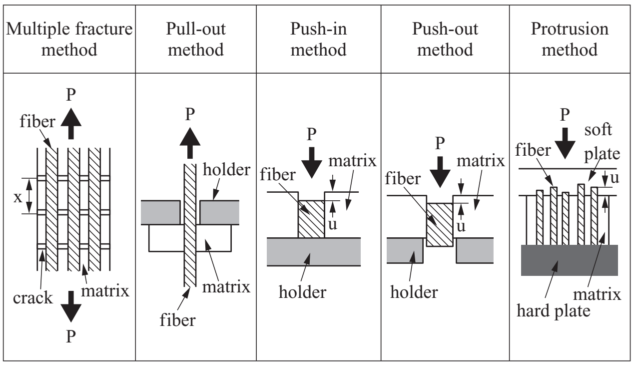


Fig. 2 Schematic images of test methods for measuring interface strength properties⁽⁹⁾

as there is wide variation in the physical property values of CMC fibers and matrixes, it is difficult to quantitatively discuss the interface strength obtained. The pull-out test has many problems, such as the necessity of preparing a special test specimen, separate to the part being examined, in order to evaluate the interface strength properties. The protrusion test is almost the only method that enables the evaluation of interface strength properties even in a high temperature but lacks quantitativeness due to the need for assuming that the interface strength is constant regardless of the position. In contrast, the push-out test can use the product or other part being evaluated to test the interface strength properties, and enables the interface shear debonding stress in the radial direction of the fibers to be studied using only the actual measured values obtained in the test. The push-out test is therefore considered to be a promising method for evaluating the interface strength of CMCs. Hitherto, there have been reports on push-out tests using Vickers hardness testers that were conducted on CMCs consisting of large diameter fibers of 100 to 200 μm(15), (16). In recent years, studies have also been performed which use these tests on CMCs consisting of small diameter fibers of approximately 10 μm, which are expected to be used in aircraft engines(10), (17)–(19), but the number of such reports is small and the reliability of such tests is still deemed to be insufficient. In addition, when testing CMCs with fibers approximately 10 μm in diameter, the influence of the thickness of the interface coating layer on the calculation result for the interface shear debonding stress is not negligibly small. It is therefore necessary to confirm the position of debonding.

In order to establish an interface strength evaluation method using a push-out test for CMCs consisting of small diameter fibers of approximately 10 μm, this study aimed to identify appropriate push-out test conditions by investigating the influence of the test device, indenter shape, and test specimen thickness on the interface shear debonding stress. In addition, by identifying the debonding position, a more accurate calculation of interface shear debonding stress was attempted.

2. Test Method

2.1 Preparation of test specimens

First, an orthogonal 3-D fabric reinforcing structure was prepared using silicon carbide (SiC) fiber bundles, with boron nitride (BN) film being formed as the interface coating layer. Next, a matrix was formed in the voids of the reinforcing structure through chemical vapor infiltration (CVI) and low temperature melt infiltration (LMI)(20), forming CMC. Following this, the CMC was processed into two types of test specimens for the push-out test, with their thickness reduced to 87 or 116 μm by resin embedding and grinding.

2.2 Dimension measurement

The thicknesses h of the test specimens were measured with a laser microscope. In addition, using a scanning electron microscope (SEM), SEM images were taken such that each shows the entirety of the interface coating layer around a single fiber. Using these images, the fiber radius R_f or the fiber radius R_{f+BN} including the interface coating layer was measured as required.

2.3 Push-out test

Figure 3 shows a schematic illustration of the push-out test. In the test, the test specimen was placed on a stainless-steel jig. The jig on which the test specimen was placed has a slit of width 30 μm to prevent contact between the fiber and jig during the test. Next, a load was applied to the fiber above the slit in order to obtain a load-displacement curve. Two types of indenters were used, i.e., a Berkovich indenter with a triangular pyramid tip and a round end cone indenter with a spherical tip, as shown in **Fig. 4**. **Table 1** shows the other test conditions.

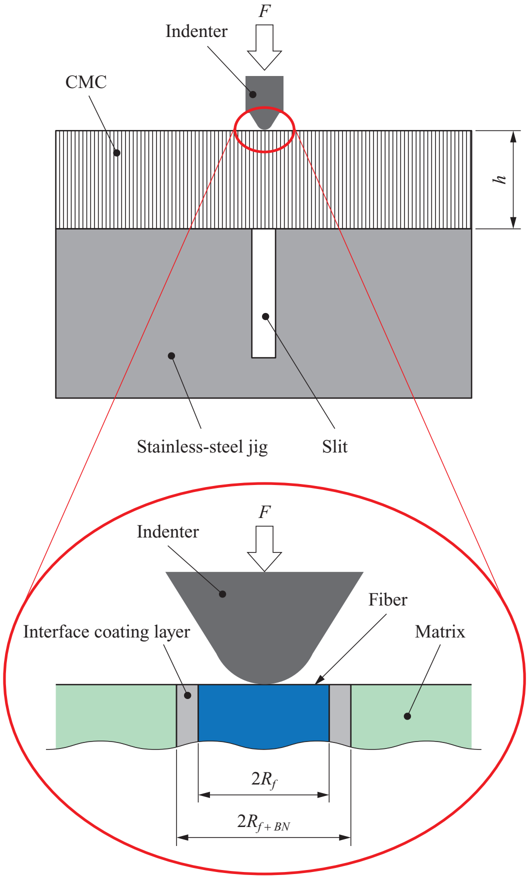


Fig. 3 Schematic illustration of push-out test

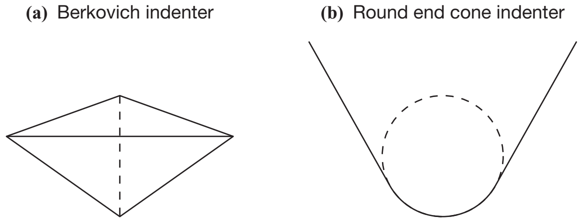


Fig. 4 Shapes of indenter for push-out test

Table 1 Push-out test conditions

Item	Unit	Condition
Control	—	Loading control
Test temperature	—	Room temperature
Atmosphere	—	Air
Maximum load	mN	300
Loading rate	mN/s	1

3. Test Results

3.1 Selection of a test device

In order to select a test device suitable for the push-out test, the two following types were chosen as candidates: a Vickers hardness tester, which has been used in past reports$^{(10), (15)–(19)}$; and a nano-indenter, which has high positioning accuracy and allows the shape of the indenter to be changed. When applying load to the central part of a fiber in a CMC consisting of small diameter fibers of approximately 10 μm, a deviation of even a few micrometers from the center may cause the indenter to come into contact with the interface coating layer or matrix, resulting in possible failure of the push-out test. It is therefore preferable to use a test device having a positioning accuracy of 1 μm or less. Since the positioning accuracy of the nano-indenter is of the order of a few nanometers in contrast to that of the Vickers hardness tester of approximately 2 μm, the nano-indenter was selected as the appropriate test device for the push-out test.

3.2 Influence of shape of indenter on push-out test results

The push-out test was conducted using two types of indenters with different tip shapes, i.e., a Berkovich indenter, and round end cone indenter. **Figure 5** shows SEM images obtained after the push-out test. For both indenters, it is believed that the effect of uniform expansion of the fiber in the radial direction due to compressive stress was superimposed on the effect of local deformation of the fiber, resulting in increased stress at the interface causing shear failure. When a Berkovich indenter was used, a clear impression was observed on the fiber after the push-out test. This is considered to be because the fiber, being made of brittle material, allows local fractures to leave permanent deformation. There is a concern that the load values produced when obtaining the load-displacement curve are affected by an increase in the frictional force between the fiber and interface coating layer due to the local permanent deformation on the fiber. In contrast, no impressions were found on the fiber when using a round end cone indenter, suggesting that there was only a slight increase in frictional force due to local permanent deformation. The round end cone indenter, for which local permanent deformation on the fiber was not observed, was therefore determined to be the one suitable for the push-out test.

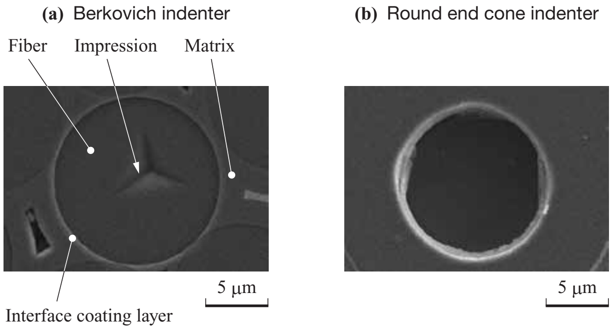


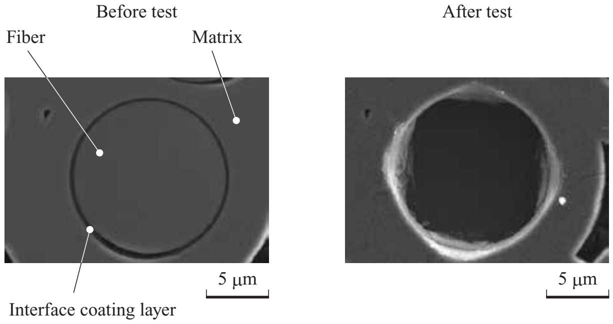
Fig. 5 SEM images of test specimens after push-out test

3.3 Calculation of interface shear debonding stress using test specimens with different thicknesses

The push-out test was conducted using test specimens of thickness 87 and 116 μm. **Figure 6** shows SEM images of the respective test specimens before and after the push-out test. After the test, both the 87 and 116 μm-thick test specimens showed debonding of the fiber from the matrix. **Figure 7** shows an image of typical debonding of the fiber after the push-out test. This image clearly shows that the debonding of the fiber during the push-out test occurred between the fiber and the interface coating layer.

Figure 8 shows load-displacement curves obtained from the push-out test. When the load was increased to 79.2 mN for the 87 μm-thick test specimen and 118.1 mN for the 116 μm-thick test specimen, the displacement increased instantaneously. This is considered to be because the whole

(a) 87 μm



(b) 116 μm

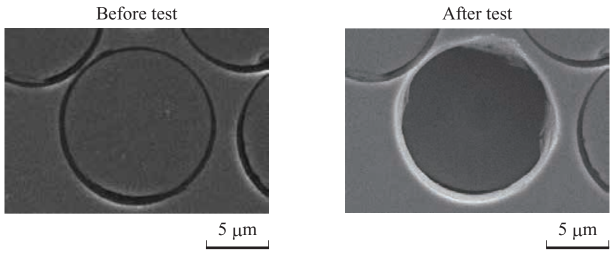


Fig. 6 SEM images of specimens with different thickness before and after push-out test

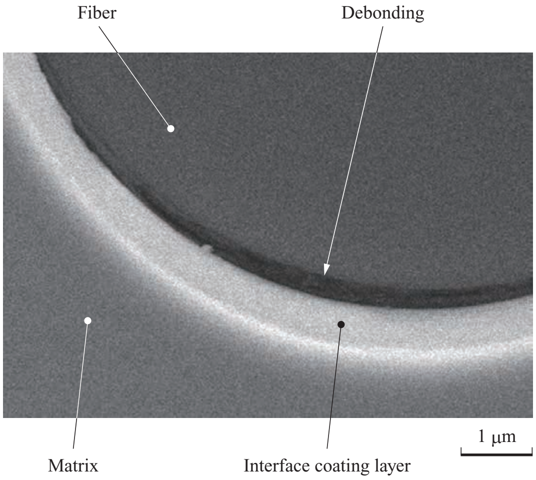


Fig. 7 SEM image of typical debonding position

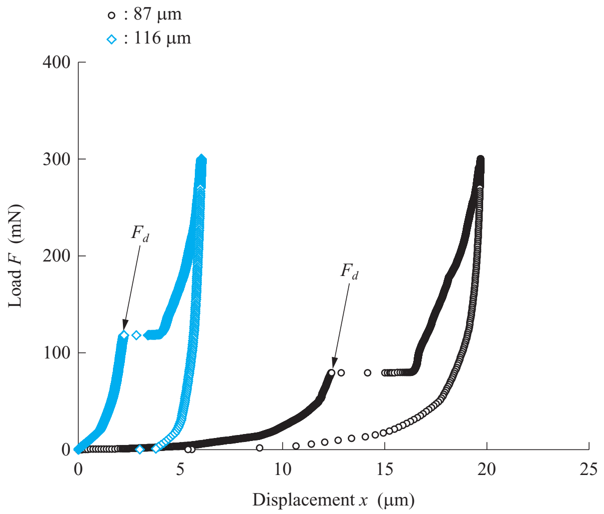


Fig. 8 Load-displacement curves for different specimen thickness in push-out test

surface of the fiber debonded from the interface coating layer, and the indenter moved abruptly in the loading direction, which is consistent with reports in the existing literature(10), (15)–(19). Because no inflection points were identified in the load-displacement curves until the fiber underwent whole surface debonding, it was considered that initial and whole surface debonding occurred almost simultaneously, and so no differentiation is made between them in this material system. Therefore, it was judged that interfacial debonding occurred when the displacement abruptly increased, and interface shear debonding stress τ_d was obtained by Equation (1) using the load F_d at that time.

Additional increases in load were observed even after debonding of the fiber because the indenter moved in the loading direction and came into contact with the matrix. As previously mentioned, because the position of debonding of the fiber in the push-out test was between the fiber and interface coating layer, the radius of the fiber R_f was used as R in Equation (1).

$$\tau_d = F_d / 2\pi R h \quad \cdots\cdots (1)$$

The values for τ_d were 23.7 MPa for the 87 μm-thick test specimen and 27.4 MPa for the 116 μm-thick test specimen. These correspond relatively well with the values obtained in the push-out test conducted using a Vickers hardness tester on fibers of diameter 144 μm reported by Honda et al.(15) Based on the above, it was discovered that, in order to use a push-out test to measure the interface shear debonding stress of a CMC consisting of small diameter fibers of approximately 10 μm and having a thickness of up to 116 μm, it is useful to utilize a nano-indenter having positioning accuracy of the order of a few nanometers and a round end cone indenter, which does not cause the local permanent deformation associated with fractures in the fiber.

4. Conclusions

In this study, the push-out test was selected as the method for evaluating the interface properties of a CMC consisting of small diameter fibers of approximately 10 μm. After investigating the influence of the test device, shape of indenter and thickness of the test specimen, the following conclusions were obtained.

(1) In the push-out test, it is necessary to apply a load accurately to the central part of the fiber, so it was found that a nano-indenter with positioning accuracy of the order of a few nanometers was suitable as the test device.

(2) A round end cone indenter with a spherical tip is useful as the indenter in a push-out test because it does not cause the local permanent deformation associated with fractures in the fiber.

(3) The position of debonding was identified from SEM observation of the test specimens performed after the push-out test. Based on this identification, the radius of the fiber R_f was selected as the value of R used in the calculation of interface shear debonding stress shown in Equation (1), thereby increasing calculation accuracy.

(4) No clear differences in interface shear debonding stress were found between the test specimens of thickness 87 and 116 μm. However, in order to constitute a quantitative method for evaluating the interface strength of CMCs, there are still challenges to be resolved, such as the inability to incorporate the verticality of the fibers with respect to the thickness direction of the test specimen.

The push-out test studied and implemented in this paper is useful as a method for evaluating the interface properties of CMCs consisting of small diameter fibers of approximately 10 μm. With deeper understanding of the fracture mechanism of CMCs obtained through the improvement of this technology, we will develop CMCs with improved strength properties and expand their applicability to aircraft, thereby contributing to improvement in the fuel economy of aircraft engines, and eventually to achievement of carbon neutrality.

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