MICROBOND TEST FOR THE FIBER/MATRIX INTERFACIAL SHEARING STRENGTH

Atsushi Wada* and Hiroshi Fukuda**

Department of Materials Science and Technology
Science University of Tokyo
Yamazaki, Noda, Chiba 278-8510, Japan
*Graduate student, **Professor

SUMMARY: Fiber reinforced plastics are essentially heterogeneous materials where plastic matrix is reinforced by fibers. Therefore, it is important to know the properties of fiber/matrix interaction in addition to the properties of fiber and matrix themselves. To measure the fiber/matrix adhesive strength, various kinds of tests such as fiber pull-out, fragmentation, microbond, etc. have so far been tried. But the correlation of the strength among these test methods is not necessarily clear although the argument of Ref.[1] has a point. In the present study, we tried microbond tests with various test conditions to get reliable interfacial shearing strength.

KEYWORDS: microbond, fiber/matrix interface, shearing strength, meniscus, debonding, carbon monofilament, epoxy resin.

INTRODUCTION

Fiber-reinforced plastics (FRPs) are nowadays used in various engineering fields primarily due to their high strength-to-density and/or stiffness-to-density ratios. When discussing these materials from a micromechanical view point, it will easily be understood that the properties of their components, fiber and matrix, will directly affect the properties of their products, composites. In addition, the properties of the fiber/matrix interface also play great role on the composite properties. For example, if the interfacial strength between fiber and matrix is weak, a so-called pull out of fiber will take place and the failure pattern will become like a zigzag manner as schematically shown in Fig.1(a). On the other hand, if the bonding is strong, the fracture surface will be flat like in Fig.1(b). This means that the interfacial characteristics are important to evaluate both the strength and the failure pattern.

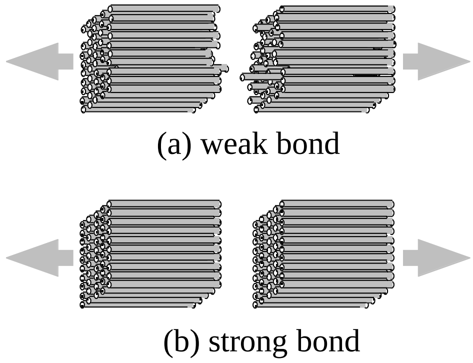


Fig.1 Typical fracture patterns of unidirectional composites.

In the past studies, the measurement of fiber/matrix adhesion was conducted using many kinds of examinations, for example, fragmentation test, microbond test, pull-out test, short-beam shear test, and so on.

Among these tests, the interfacial shearing strength measured by the microbond test is lower than that by other tests[1,2]. For example, Ref.[1] reports the interfacial shearing strengths of 68.30MPa (fragmentation) and 50.30MPa (microbond). Our results [2] were 52.4MPa (fragmentation) and 48.0MPa (microbond). This is probably due to a pair of meniscuses which are inevitably formed at both ends of the microdrop. Due to these meniscuses, the measurement of the embedded length becomes somewhat inaccurate. Another reason might be a stress concentration generated by the contact of a microvice to the microdrop.

In the present study, we focused on these two causes to measure the shearing strength more accurately.

EXPERIMENTAL PROCEDURE

Raw materials used for the present microbond test are Torayca T300 carbon fibers and Epicote 828 epoxy resin by Yuka Shell with the hardener of Epomate RD1.

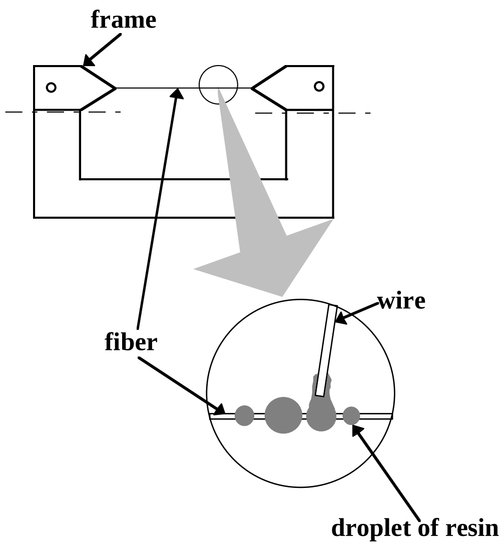


Fig.2 Method of making a microbond specimen.

Figure 2 shows the procedure to make a specimen. A carbon monofilament was first picked up from a bundle of fibers and it was set up straightly to a frame. Then a very small amount of the epoxy resin with hardener was dropped on the monofilament by which several microdrops of 50-90μm diameter were made on the monofilament. This specimen was kept about 24 hours at the room temperature and then after-cured in the oven for 12 hours at 85˚C.

Figure 3 is a test device for the microbond test, which was designed by ourselves. The specimen was set so that one end of the monofilament was connected to a micro load cell of the device and it was inserted into a small gap of a pair of microvises as shown in Fig.3. The small gap between these microvises was controlled by rotating the micrometers manually.

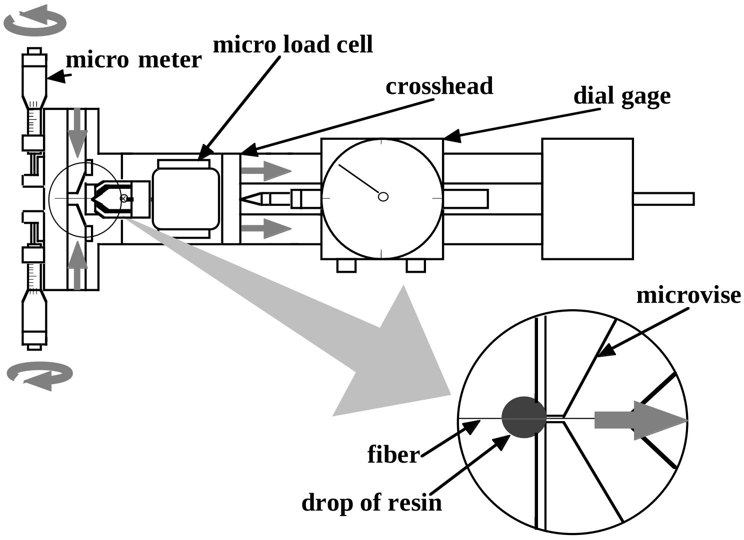


Fig.3 Test devicce for the microbond test.

The apparent interfacial shearing strength may be affected by the gap width between a pair of microvices and by the shape of the microvices. Then we tried several conditions. Figure 4(a) shows two kinds of gap width. One is that the gap width is fixed to 10µm which is fairly small considering the diameter of carbon monofilament, 7µm. The other is that the gap width is adjusted so that the contact angle, measured from the center of the microdrop, becomes 60˚. In the latter case, the gap width varies in accordance with the size of the microdrop.

Figure 4(b) shows, on the other hand, two types of microvise configuration. That is, the contact edge of the microvise is either the right angle (sharp-type) or blunt (blunt-type).

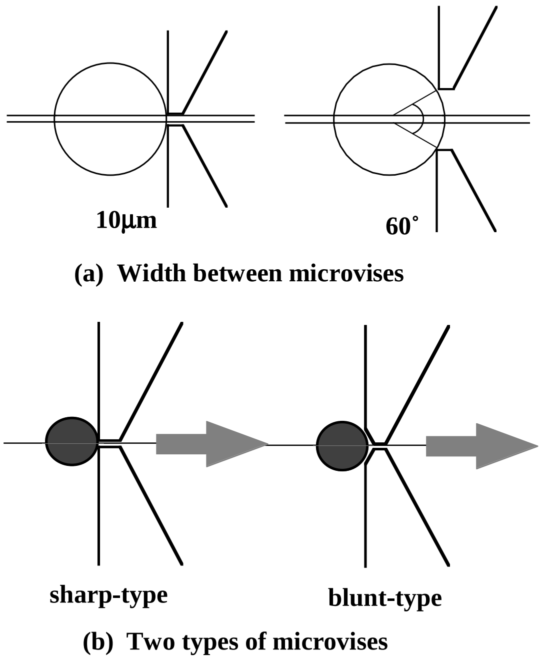


Fig.4 Location and shape of microvices.

These conditions may affect the stress distribution in the microdrop and hence the strength. In the present study, the interfacial shearing strengths under the above 4 conditons were measured.

The test device of Fig.3 was designed so that the crosshead speed could be controlled by an electric motor and the crosshead speed was fixed to 1µm/s throughout the test. Due to this movement, the microdrop is pulled out from the monofilament. During the test, the load was measured by the micro load cell and the displacement (crosshead movement), by the dial gage.

These data were saved in a personal computer with the sampling time of 1s. This microbond test was carried out on an optical microscope to observe the in-situ behavior of the microdrop. The fiber/matrix interfacial shearing strength τ was calculated by the following equation [3].

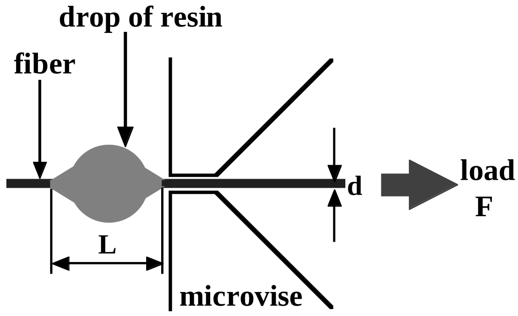


Fig.5 Schematic view of microbond test.

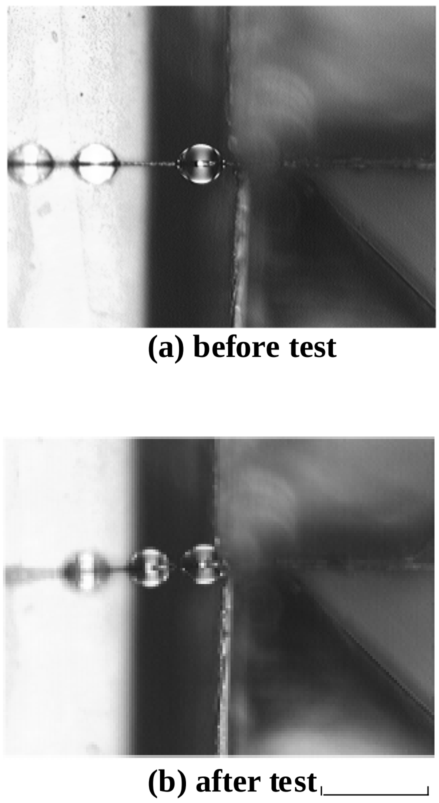
$$\tau = \frac{F}{\pi d L} \tag{1}$$

where F is the pull-out force, d is the diameter of the monofilament, and L is the adhesive length between fiber and matrix as shown in Fig.5.

RESULTS AND DISCUSSION

An example of the observed picture before and after the test of the same specimen is shown in Fig.6. It is clearly seen that the rightmost microdrop was moved relatively to the left during the test which indicates that the debonding took place. Figure 7 demonstrates the load-displacement diagram.

The interfacial shearing strengths, derived from the microbond test of four patterns, are shown in Fig.8 where the abscissa is the adhesion length defined in Fig.5. Each mark corresponds to each experimental value. As is shown



(a) before test

(b) after test

200µm

Fig.6 Microscope observation of microbond test.

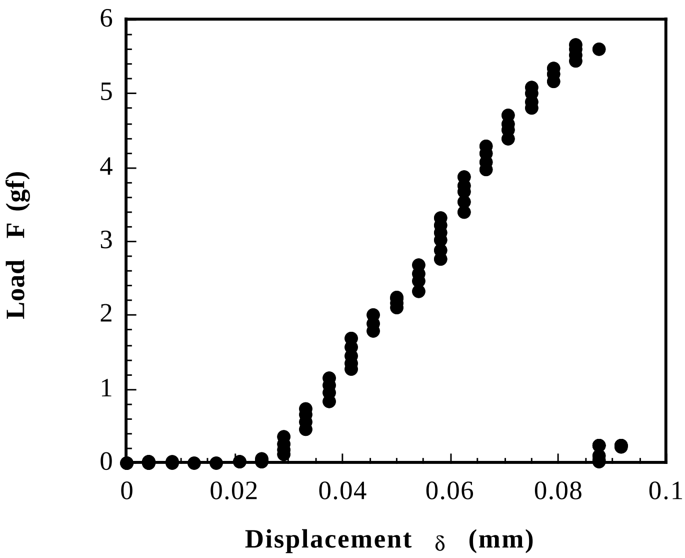


Fig.7 Typical load-displacement diagram of microbond test.

in this figure, the scatter of data is fairly large. But if we dare conduct line fitting by means of a least square method, we get four lines in Fig.8. The correlation coefficients were 0.61 for sharp-10μm, 0.75 for sharp-60˚, 0.64 for blunt-10μm, and 0.73 for blunt-60˚.

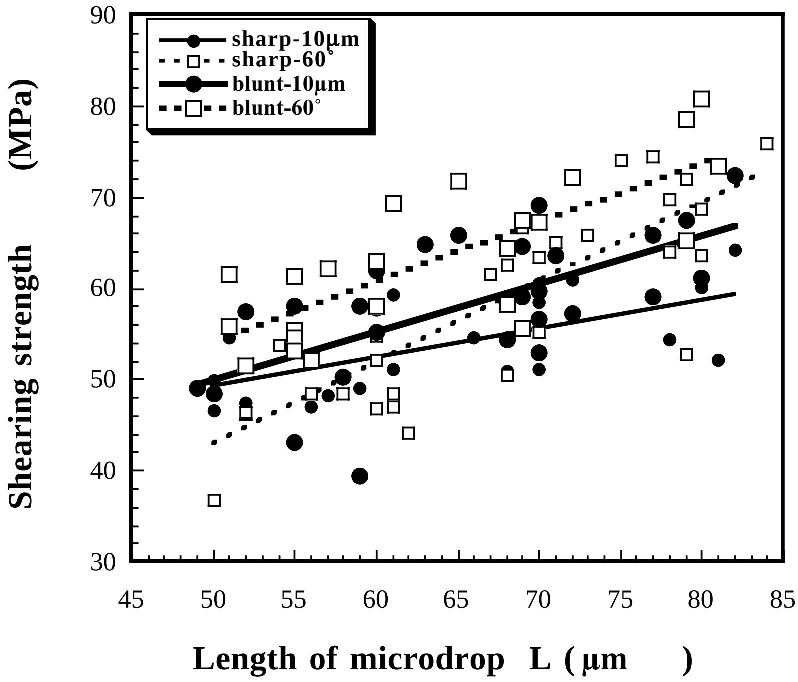


Fig.8 Shearing strength vs. length of microdrop.

First of all, for all cases, the shearing strength increased as the length of the microdrop increased. This point is discussed first. Figure 9 qualitatively shows that the size of meniscus doesn't depend on that of microdrop although the quantative discussion is not easy. As will be discussed later, the part of the meniscus may carry little load. Because the ratio of size of the meniscus to the microdrop is relatively large for small microdrop, the shearing strength of the small microdrop was measured as lower value.

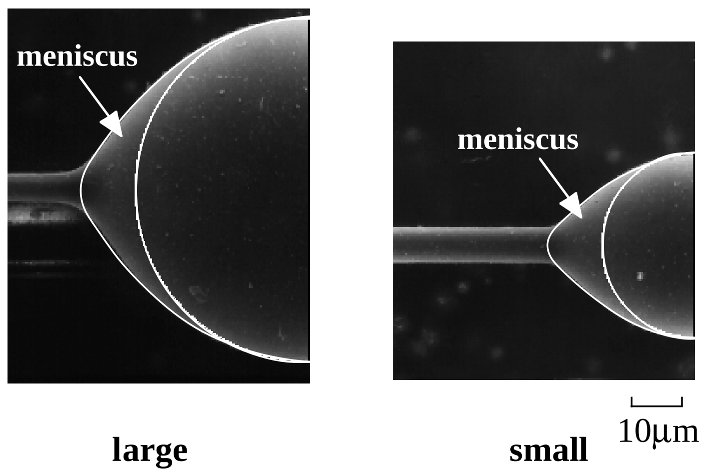


Fig.9 Size of meniscus.

Secondly, the shearing strength under the sharp-type microvices is entirely smaller than that under the blunt-type ones. As is seen in Fig.10, the sharp-type microvises sink into the microdrop whereas the microdrop using blunt-type microvises is almost intact. This suggests that blunt-type microvises are desirable in order that a stress concentration doesn't take

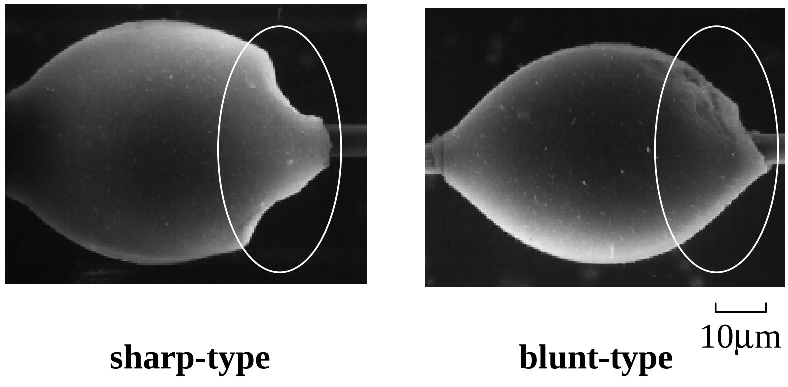


Fig.10 Comparison of failure pattern between sharp-type and blunt-type microvises. (Width between microvises 10μm)

place to the microdrop, hence in order to measure more exact shearing strength by the microbond test.

The third point suggested in Fig.8 is that the shearing strength at 10μm width between microvises is smaller than that at 60˚ width between microvises. The microvises of 10μm width directly contact to the meniscus whereas those of 60˚ width contact to the body of microbond rather than the meniscus. That is, the microbond test with microvises of 60˚ width can be examined without a failure of meniscus. Figure 11 shows that meniscus was not damaged due to the microvises of 60˚ width. Instead, it seems to have been broken at the final stage of pull-out. Therefore, the microbond test should be conducted at 60˚ width between microvises to measure the shearing strength accurately.

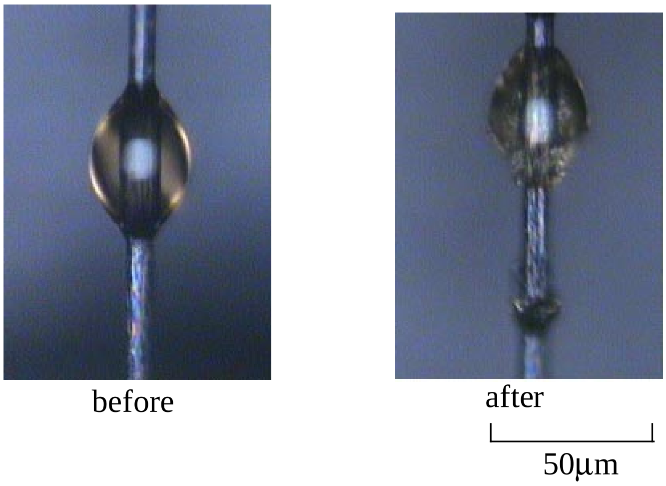


Fig.11 Damage of meniscus.

To examine by calculation the above experimental results, we conducted a finite element analysis using a commercially available LUSAS software. In the FEM, an axisymmetric modelling of linear elasticity was assumed although this assumption is different from the actual test where the microvise was not axisymmetric. The microdrop was modelled as a sphere plus meniscus. The total element number was 2632. The applied load was 5gf which was a representative value at failure.

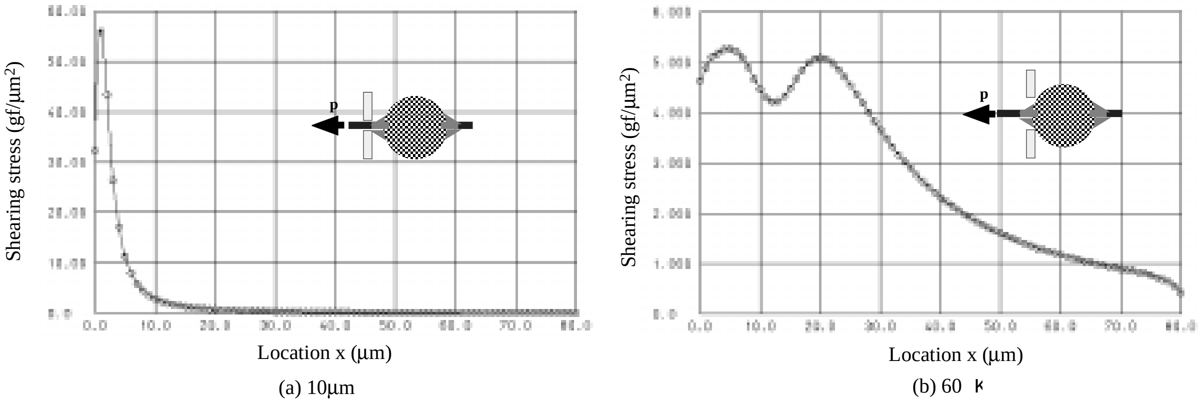


Fig.12 Interfacial shearing stress distribution, FEM.

Figure 12 demonstrates the interfacial shearing stress distributions for (a) 10μm gap width and (b) 60˚ gap width. This example is the case of L=80μm and the abscissa is the location from the end of the meniscus. Since we assumed the size of each meniscus to be 10μm, the parts of 0<x<10μm and 70<x<80μm correspond to the meniscuses. In the case of small gap width of (a), the stress concentration is very large especially along the part of the meniscus. The shearing stress in the meniscus is almost 10 times larger than the expected shearing strength. This may lead the failure of the meniscus followed by the interfacial debonding between the fiber and the main body of the microdrop. In the case of 60° gap width of Fig.11(b), on the other hand, the shearing stress is small and moderate even though the stress is not constant throughout the interface.

As was discussed above, the meniscus may not have large contribution to the debonding load. If we discard the meniscus to which the microvise contacts, the bonding length, L, decreases, which results in the increase of the interfacial shearing strength from eq.(1). Figure 13 was re-drawn from Fig.8 assuming the contact-side meniscus is missing.

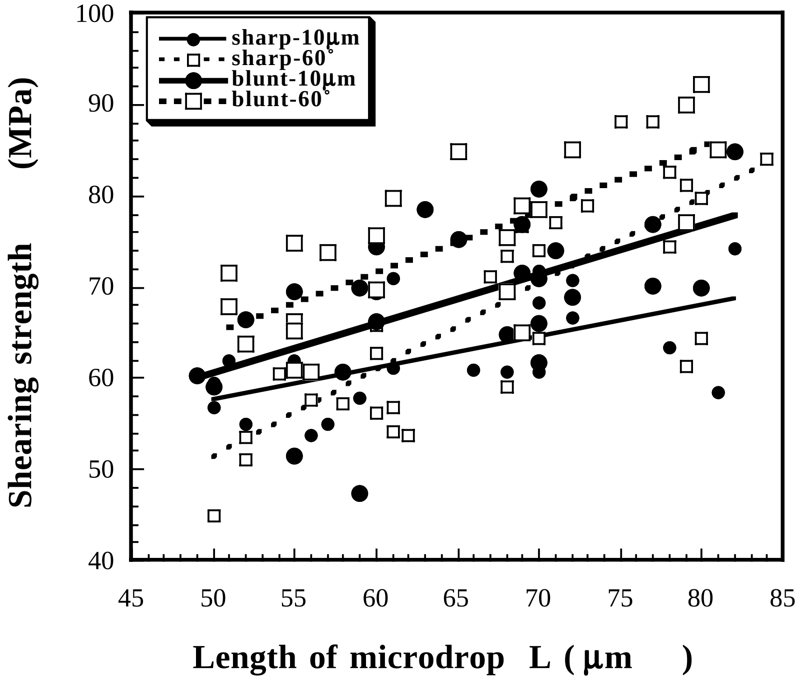


Fig.13 Modification of Fig.8 considering the fracture of meniscus.

Comparing to Fig.8, the interfacial shearing strength increased by 10-20%. This may give an answer why the interfacial strength measured by the microbond test is lower than that by the fragmentation test. The shearing strength was, nevertheless, not constant with respect to the size of microdrop.

It is very difficult to conduct in-situ observation of the failure process of the meniscus and the debonding process between the microdrop and the fiber. In that sense, Fig.13 still includes some assumptions. Nevertheless, we may conclude that the true shearing strength of the present material is in between the values of Fig.8 and Fig.13, that is, τ = 55 MPa (reprentative value of Fig.8)-65MPa (reprentative value of Fig.13).

CONCLUSIONS

In the present paper, we measured the fiber/matrix interfacial strength by the microbond test of four patterns to discuss the effects of the meniscus and the contact condition of the microvice to the microdrop. We made clear the cause that the shearing strength measured by the microbond test is lower than that by other test methods. We also proposed a desirable shape of microvice to get more reliable interfacial strength.

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