INTERFACIAL STRENGTH OF OPTICAL FIBRES

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SUMMARY: The interfacial strength of three different optical fibres were evaluated using a push-out test method. Polyimide coated optical fibres either without or with a silane sizing on the silica glass, and optical fibres with aluminium coating were tested. A significant increase in the interfacial strength was achieved with the silane sizing. The failure mode shifted from the silica glass/polyimide interface to the polyimide/composite. For the two other fibres the interfacial strength was similar. Pre-conditioning of samples with polyimide coated optical fibres indicates that moisture can result in a significant reduction in interfacial strength for fibres without sizing whereas the sizing has a protective effects on the interfaces to moisture.

KEYWORDS: Smart structures, Optical fibre, Interfacial strength, Polyimide coating, Silane, Aluminium coating, Push-out testing

INTRODUCTION

For aerospace applications, fibre optic systems are developed for operational load monitoring. Contrary to conventional resistive strain gauges, fibre optic sensors (Bragg grating sensors) can be multiplexed in a large number which may reduce the weight of such systems. Especially, embedded optical fibre sensors are viewed as potentially more cost-effective and reliable than surface attached sensors. To be effective in an embedded environment, the optical fibres require a coating which maintain their integrity over the lifetime since repair of embedded sensors is not possible. Therefore an issue is the sensor reliability of embedded sensors as only limited data exist. Embedded Bragg grating sensors change the microstructure of the composite and perturb the strain field [1,2]. The resulting stress concentrations give rise to large interfacial stresses at the interfaces of the sensor. The stresses make the interfaces susceptible to debonding. If debonding occurs at the interface, this may lead to unacceptable stress redistribution in the sensor. The effect may be that the stress state in the fibre core is changed and as a result the accuracy in sensor response is reduced.

Numerous studies of the effect of the fibre/matrix-adhesion in composite materials on the material behaviour have been performed, whereas only limited number of studies on embedded optical fibres exists [3]. Interfacial adhesion has a large effect on many of the mechanical properties of composite materials. Besides that the effect of the adhesion on overall strength and stiffness properties, also the test methods for characterisation of the interfacial adhesion has been thoroughly studied. Basically, in the literature three kinds of methods [4,5,6], the push-out and the pull-out techniques, and single fiber fragmentation testing are described which are used to determine interfacial strength on single embedded fibres. One problem with the push-out and pull-out tests is the clamping stresses at the top surface as an effect of the loading and from mismatch in the thermal expansion coefficients of the fiber and matrix. This gives rise to frictional forces at the interface when the fibre is partially debonded. To include the effect of friction in the results have shown to be difficult. However, one advantage with the push-out test is that samples using this test method are sections of real composites, which may eliminate the uncertainty concerning differences that might arise in preparing samples with no reinforcement as compared to embedding optical fibres into the actual composites.

Silanes are used as coupling agents for fibre in polymer composites to improve the bonding between the fibre and the matrix, to obtain composites with better mechanical properties [7]. The silane reacts with silanol groups present at the fibre surface to form strong siloxane bonds. The fibre/matrix interface is further strengthened as an effect of the polymer penetrating and reacting with the silane layer. However, an increase in interfacial strength can only be achieved by a tuned balance of type of silane and silane concentration.

In this study, three different optical fibres were evaluated in terms of interfacial shear strength. The objective was to evaluate high temperature coatings protecting the silica glass in the optical fibres from moisture. The method allowed tests on sections of real composite with an embedded optical fibre. One of the polyimide coated optical fibres had a silica glass/polyimide coating interface treated with a silane sizing expected to improve the interfacial adhesion, while the other polyimide coated fibre was a standard type of fibre without any surface treatment. The third optical fibre had an aluminium coating. Attention in the present study was paid to the effect of absorbed moisture in the composite on the strength of the interface. The effect that absorbed moisture may lead to a irreversible reduction in interfacial strength by weakening the bond between the coating and fiber was evaluated.

EXPERIMENTAL APPROACH

Optical fibres were embedded in the midplane of laminates made from carbon fibre/epoxy matrix composite HTA/6376C. The laminates were cured at 180°C and 0.6 MPa in an autoclave. The optical fibres were embedded along the direction of the carbon fibres. The glass in the fibre was 125 µm in diameter; the polyimide coating was approximately 7 µm thick and the aluminium coating was 1 µm. The polyimide coated fibre was tested with or without an silane sizing between the polyimide and the silica glass of the optical fiber.

In order to determine if moisture absorption has any effect on the interfacial strength of the optical fibres, samples were pre-conditioned. Two different type of samples were tested; optical fibres which had a silica glass/polyimide coating interface without silane and the same fibre with silane sizing. The effect of moisture on fibres with silane sizing was studied in a cross-ply laminate, $(0_4/90_4/0_4/90_4/0_2)s$, rather than in a unidirectional laminate $(0)_{24}$ as in the rest of the study.

The push-out technique was used in the present study to determine the strength of the optical fibre interface. The embedded optical fibre was loaded with a small indenter which pushed the fibre out of the sample under a monotonically increasing displacement. The load and displacement were monitored during the test.

The samples were cut from the laminates using a low speed cutter with a diamond blade. The polishing after cutting was performed using two different diamond particle sizes (6 µm and 1 µm) in order to remove the damage from the cutting around the glass fibre. A number of different sample thicknesses were chosen to obtain results over a large range of embedded optical fibre lengths. The laminates were cut into the thin samples only a few days before the testing to avoid damage from moisture at the edges of the sample. The pre-conditioned laminates were kept in a chamber at 70°C and 95 % RH for approximately one year before being tested. This allowed the laminates to be saturated with moisture. The absorbed moisture in the laminates was measured to be 1.0 % by weight. Samples were examined using scanning electron microscopy to check if the polishing had damaged the interface or not. In some pre-conditioned samples with optical fibres without silane sizing, it was observed that partial debonding occurred at the polyimide/glass interface in a few cases, caused by the polishing. These samples were discarded.

The laminates were examined by X-ray radiography to determine the location of the optical fibres after manufacturing. Further, the misalignment of the embedded optical fibres were estimated from the X-ray radiographs. The measured misalignment angles were determined to be less than 0.3°. These angles were considered to have no influence on the result.

Figure 1 Test set-up

The test set-up is shown in Figure 1. A support which held the sample during the experiment was carefully designed with a hole to accomodate the fibre being pushed out. In testing of thin samples, an important problem to be avoided was the bending of the sample, so the diameter of the hole in the support was chosen to be only 0.25 mm. The sample was fixed to a X-Y stage which allowed translation of the sample in the horizontal plane, with an accuracy of a few microns in each direction.

An indenter with flat surface was selected to facilitate a smooth load introduction into the fibre and thus avoid any premature failure of the fibre. The indenter had a diameter of 0.1 mm, slightly smaller than the diameter of the silica glass, avoiding in this way contact with the coating. The positioning of the indenter was such that a minimum distance of 5 μm between the edge of the indenter and the coating was accepted. Each sample had to be carefully centred to the hole in the support before running the test.

In the experiments, a load cell for a maximum load of 100 N was used. The noise in the signal from the load cell was approximately 0.04 N. The sampling frequency was 1 Hz. The loading rate corresponding to the rate at which the indenter pushed the optical fibre had to be chosen as small as possible, was 0.01 mm/min to obtain a full set of data. The displacement data was provided by an extensometer. The accuracy of the extensometer was approximately 0.1 μm.

RESULTS

The initial experiments were performed using the push-out test method on samples which were not pre-conditioned. These samples included embedded optical fibres with polyimide and aluminium coatings, respectively, in unidirectional laminates.

An example of a load-displacement curve for an embedded optical fibre is shown in Figure 2. In the first stage, the slope of the curve is approximately constant. This is a result of elastic deformation. At a particular load level, the load-displacement curve exhibits a sharp change in the slope. Bechel and Sottos [8] have experimentally shown that this load level directly corresponds to the load where the initiation of the debonding occurs. This load is often referred to as the initial debonding load. At further increase in load the debonding is further driven along the interface at different stages of debonding and sliding until a maximum load is reached. At this load, total debonding of the fibre has occurred.

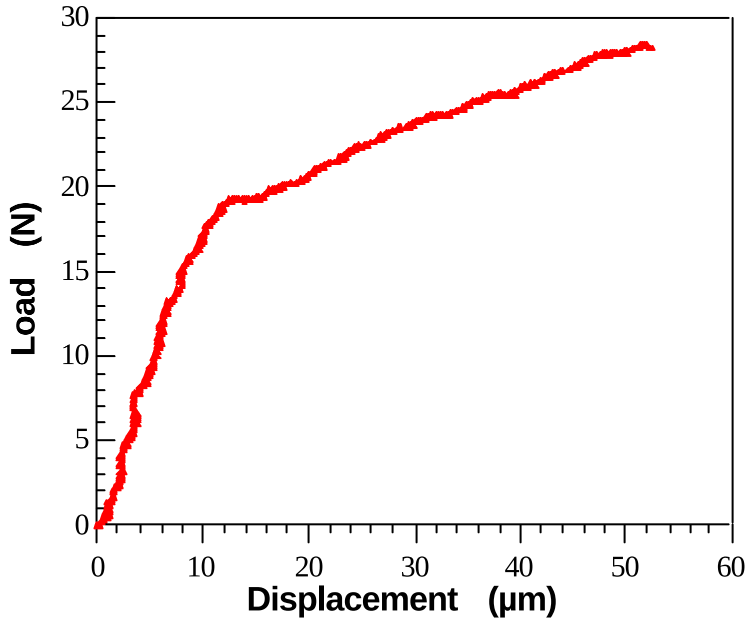


Figure 2 Load-displacement curve up to maximum load for a 2.17 mm thick sample with an aluminium coated fibre

The results in terms of maximum load for the samples with the three different optical fibres are shown in Figure 3, where the maximum load is plotted as a function of sample thickness. The maximum load was directly determined from the load-displacement curve of each test. The figure shows that the optical fibres with silane sizing have the highest maximum load of the three optical fibres. The polyimide coated optical fibres without silane had significant lower maximum load. The testing of the polyimide coated fibre without the silane sizing and the aluminium coated fibre resulted in similar maximum loads for these two optical fibres.

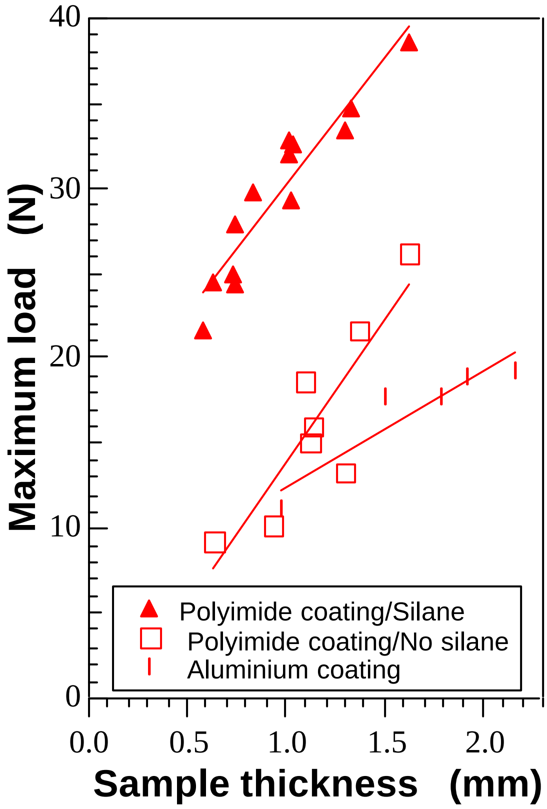


Figure 3 The maximum load as function of sample thickness for polyimide and aluminium coated optical fibres

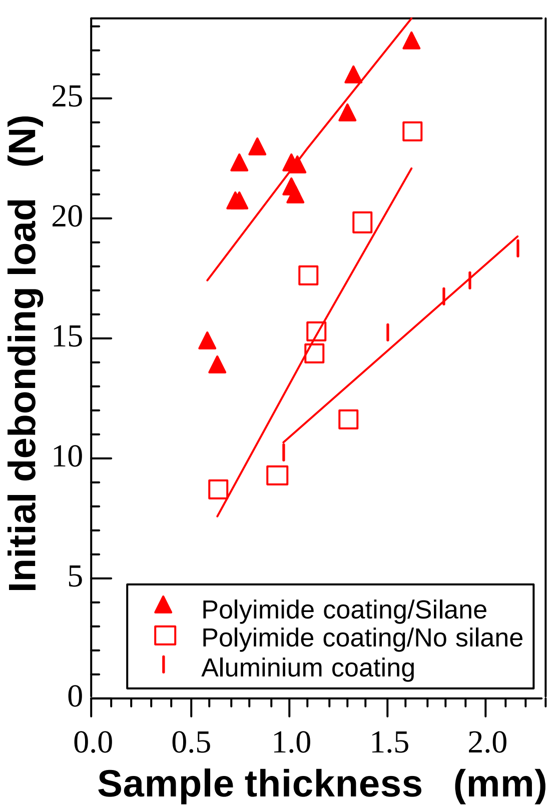


Figure 4 The initial debonding load as function of sample thickness for polyimide and aluminium coated optical fibres

The initial debonding load is plotted as a function of the sample thickness in Figure 4. Similar behaviour was observed as in the results for the maximum load. The silane sizing improved the interfacial strength significantly indicating a clear effect of the surface treatment of the silica glass. The figure shows that the debonding load of the aluminium coated fibre was slightly smaller than that for the polyimide coated fibre without silane sizing. However, for the thinnest samples no significant difference between the aluminium coated fibre and the optical fibre without silane was observed.

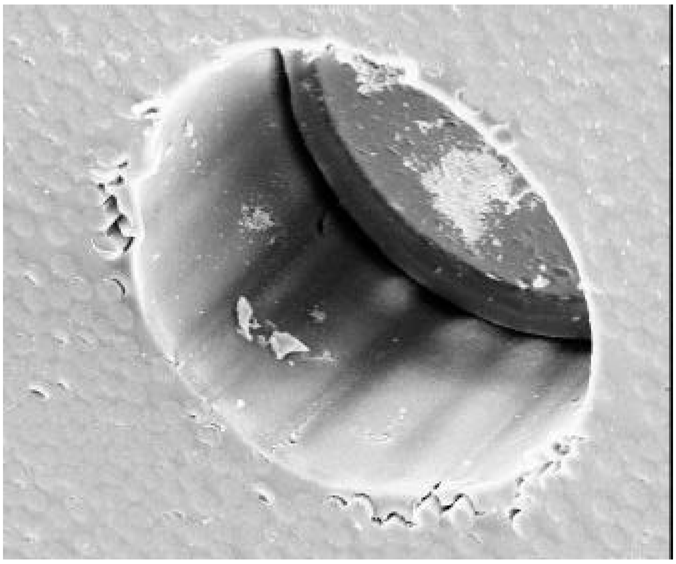
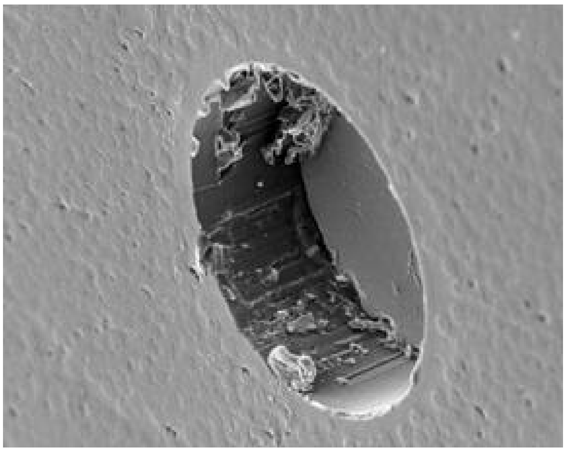


Figure 5 Micrograph of the indentation surface of the polyimide coated fiber with silane sizing (sample thickness was 0.84 mm)



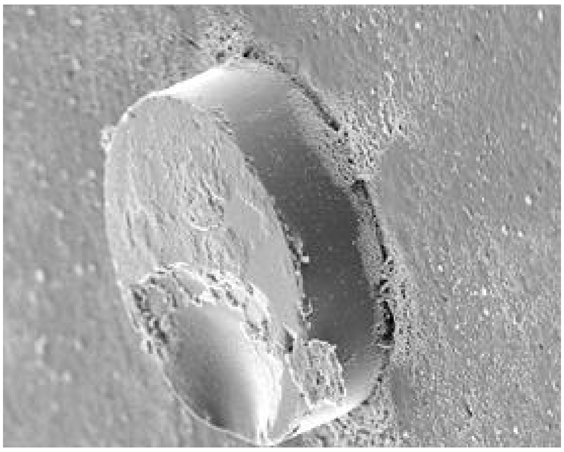


Figure 6 Micrographs of a sample with silica glass without silane sizing (sample thickness : 1.36 mm) (a) Indented surface of the sample (b) Rear surface of the sample

The samples were further examined in a scanning electron microscope to determine the failure modes. In Figure 5 an indented polyimide coated optical fibre with silane sizing is shown. It may be clearly seen that the failure location did not appear at the silica glass/polyimide coating interface, but along the coating/composite interface. A typical feature was that some damage occurred between carbon fibres and epoxy matrix close to the optical fibre. In contrast the polyimide coated fibre without silane sizing failed between the polyimide and the silica glass as seen in Figure 6. The figure indicated that the debonding mainly grew along the silica glass/polyimide interface. The micrograph of the failure surface of the aluminium coated fibre is shown in Figure 7. The figure also includes a closer view at higher magnification of the same fibre. At the failure surface some areas had residues of the coating. This indicated that debonding did not only occur along the silica glass interface but also at the aluminium and composite interface. Generally it was observed that the debonding was more prone to fail at the silica glass interface. The failure through the aluminium coating also indicated that the strength of the coating was relatively low.

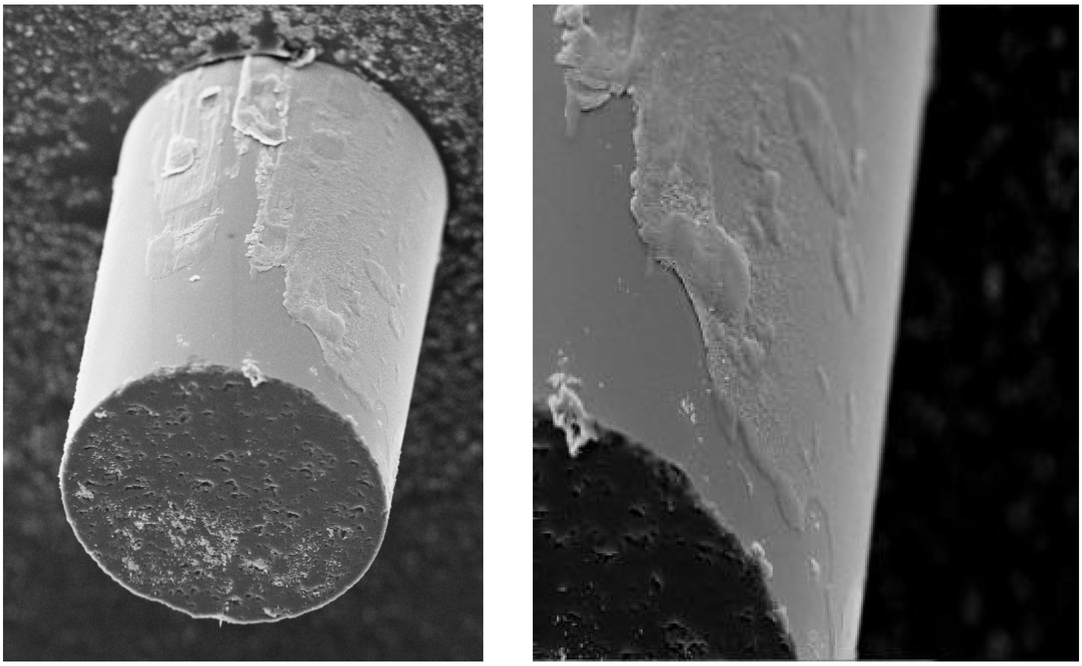


Figure 7 An aluminium coated fibre viewed from the rear side of the sample (sample thickness; 1.75 mm)

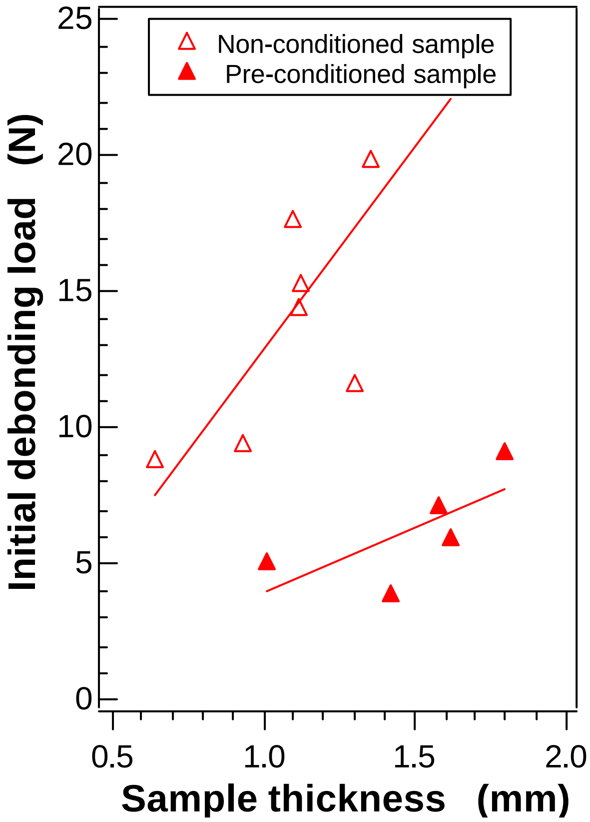
The average shear strength, t, were calculated from the initial debonding load, P, by the relationship,

$$t = \frac{P}{2p\,rt} \qquad (1)$$

where r is the distance from the centre of the fibre to the failed interface and t is the sample thickness. The use of the average shear strength for comparing the results for different coatings was justified by the simplicity. Although the shear stresses are not uniform along the interfaces, the differences of thermal residual stresses and stress distribution as a result of the loading are similar in the different samples [9]. The average strength values were 26.4±4.8 MPa, 16.6±3.0 MPa and 12.2±1.0 MPa for the samples with polyimide coated fibres with and without silane, and aluminium coated fibre, respectively.

Pre-conditioned samples

In order to evaluate the effect of moisture on the interfacial strength, samples with the two polyimide coated fibres were tested. Figures 8 and 9 summarises the results for the pre-conditioned samples with optical fibres with non-silane and silane samples respectively, in terms of initial debonding load. The initial debonding load is plotted as a function of the sample thickness. The experiment of the optical fibre with the silane sizing was run on samples with a cross-ply lay-up and therefore these load values can not be directly compared with those for non-silane fibre. The load where debonding was initiated in the interface was significantly lower for the pre-conditioned optical fibre without silane than in the non-conditioned sample. The failure location was identical to that observed in the normal samples, i.e. it occurred between the glass and the polyimide coating. Results from the tests on the fibre with silane did not indicate any effect of moisture, although the number of samples with no pre-conditioning were limited to two samples. The failure mode was identical in all samples; they failed between the polyimide and the composite indicating a strong bond between the silica glass and the polyimide.



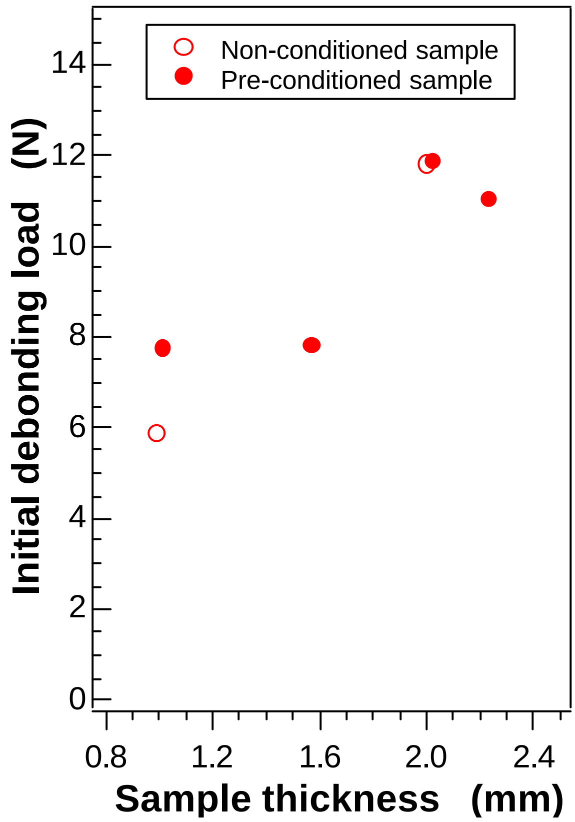


Figure 8 The effect of preconditioning on maximum load for polyimide coated optical fibre without silane. Unidirectional laminate

Figure 9 The effect of preconditioning on the initial debonding load for polyimide coated optical fibres with silane. Cross-ply laminate

The absorbed moisture in the composite and the polyimide coating changes the residual stresses in the samples [9]. In the unidirectional laminates the moisture absorption gives rise to uniform tensile radial stresses at the interfaces. On the other hand, in a cross-ply laminate, both tensile and compressive radial stresses act along the interfaces. The magnitude of the tensile radial stress is similar to those in the unidirectional case. Although the change in the residual stress makes the results for the case of moisture absorption more difficult to interpret the large difference in debonding load for the non-silane fibre indicated that moisture may damage the silica glass/polyimide interface. In the same way, the unchanging debonding load and the same damage location for the fibre with silane sizing in the pre-conditioned sample indicated that the degradation, if any, was small.

CONCLUSIONS

It was found that the coating material and surface treatment for polyimide coated optical fibres had a significant effect on the interfacial strength. The results showed that the polyimide coated fibre with silane had significantly higher interfacial strength than the other tested fibres. The aluminium coated fibre had similar interfacial strength as the standard polyimide coated fibre.

Fractographic studies showed that in the samples with the aluminium coated and the polyimide coated fibre without silane sizing, the failure was initiated at the interface between the glass and the coating, and preferably grew along the same interface to the rear surface of the sample. A different failure mode was observed for the polyimide coated fibre with silane. In this case, the debonding occurred between the polyimide coating and the composite, which indicated a strong bonding between the silica glass and the coating.

The embedded standard optical fibres in the pre-conditioned samples had lower interfacial shear strengths compared to the non-conditioned fibres. For the pre-conditioned samples with the polyimide coated fibre with silane sizing this reduction was not significant. In the both cases, the failure location was identical to that observed in non-conditioned samples. These results indicate that the silane can improve the moisture protection.

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