Microscopic Examination of Embedded Optical Fibres with Polymer Coating after Fatigue Loading

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ABSTRACT

An experimental investigation was carried out to characterise the interface behaviour of embedded optical fibres (OF) within a carbon/epoxy composite after fatigue loading. The test specimens contained OFs with polyimide (PI) and epoxy coating, respectively. They were loaded in a tension to tension mode up to one million cycles at two strain levels. Microscopic inspection was then carried out to examine the bonding performance of the embedded OF within the host structure. While the investigation showed disbonding at the inner interface between the PI coating and the silica glass, it suggested that an epoxy coating could provide a better interface material.

INTRODUCTION

Optical fibre Bragg grating (FBG) sensors have emerged as one of most important sensing technologies for structural health monitoring (SHM) applications [1-4]. Their small size and light weight, allowing surface attachment on, or embedment within a composite structure, are of great interest, along with their capability of multiplexing and electronic magnetic interference (EMI) immunity.

Optical fibres (OF) are generally coated with a thin polymer for protection due to their fragile nature and poor environmental resistance. Subsequently, this polymer coating has to be designed appropriately, not only for the FBG to sense the state of the host structure (through a good strain transfer mechanism), but also for robust and durable operation within the design life of the host structure under monitoring. If a disbond occurs between the host and the coating, or between the coating and the silica glass, the strain measured by the sensor will differ from the strain experienced by the host structure. This essential requirement has led to the durability of the interfacial bonds becoming an important research area in OF smart structures.

Currently, coatings produced from polyimide (PI) polymer are commonly used for high temperature applications due to the ease of coating, processing (liquid form) and the high temperature properties of PI. It has been reported that the interface between the coating and the silica glass is the weaker interface [5-10] based on single-fibre pull-out and push-out tests. However, very few studies have been done on the durability performance of the complete sensor within a composite structure. Some testing programs have showed no significant signal degradation after one-million cycles for the embedded OF in composite laminates. However, tests such as the one carried out by

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Jang et al. [11] were based on measurement of signal intensity, which could not provide information on the bonding behaviour of the embedded OF within the host structure.

The present experimental investigation used a different approach to characterise the bonding performance of embedded OFs when coated with different polymers, within a carbon/epoxy composite. After one-million cyclic loads under two different strain levels, the tested specimens were cross-sectioned and microscopically examined.

EXPERIMENTAL

Optical Fibre Form

Three forms of OFs were embedded in a carbon/epoxy composite and tested in the present investigation. These included a standard telecommunication PI-coated (SPI) single mode OF with a diameter of 150μm, a special small-diameter PI-coated (SSDPI), and optical fibre tape (OFT) in which the OF was coated with epoxy. The second sample had an outside diameter of 52μm (the cladding is 40μm in diameter and the polyimide coating thickness is 6μm), and was provided by the University of Tokyo. The last sample was fabricated in-house, based on a standard telecommunication OF with a diameter of 125μm and with the coating removed.

Manufacture of Fatigue Test Specimens

Fatigue test specimens were manufactured using 12-ply plain-woven prepreg material, BMS8-256, according to ASTM D3479 specification. The specimens comprised of the three forms of OFs described above. The test matrix is shown in Table 1. In Group A, two SPI and two SSDPI optical fibres were placed in two directions, longitudinal (0), parallel to loading direction, and transverse direction (90), respectively, as shown in the semi-circular brackets in the following lay-up.

[±45/0/±45/0/(**90, 90**)/±45/0/(**0, 0**)/0/±45/0/±45/0/±45]

Figure 1 shows the locations of the embedded OF for Group A specimens.

Table 1: Test Matrix

Specimen Group	Numbers of Specimens	Specimen Details	Fatigue Test Details Nominal strain levels
Control	2	Contains no OF, only for a static tension test	—
	1	Contains SPI/SSDPI, only for microscopic analysis	
	1	Contains OFT, only for microscopic analysis	
A	4	Contains SPI and SSDPI in both longitudinal and transverse directions	Two tested at 1000με/5000με and two at 600με/3000με.
B	3	Only contains OFT in longitudinal direction.	Two tested at 1000με/5000με and one at 600με/3000με.

SPI – standard PI-coated optical fibre, SSDPI - special small-diameter PI-coated OF, OFT – optical fibre tape

Group B contained only one OFT at the mid-plane of the lay-up in the longitudinal direction due to the relatively large dimension of the OFT and the limited size of the test specimen.

For comparison, three types of control specimens were also prepared: the first one contained no OF (for static tension test); the second and the third contained OF for microscopic analysis.

After curing inside an autoclave (under the condition of high pressure and high temperature), the composite laminates were cut into a number of tensile test specimens for fatigue test. The dimensions were 25mmx250mmx2.5mm

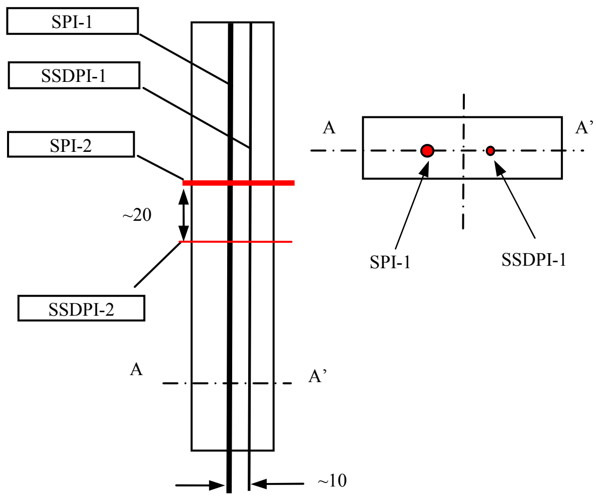


Figure 1: Locations of the embedded OFs in Group A specimens (unit:mm)

Fatigue Test

Tension-tension fatigue testing was used to determine the durability of the embedded OFs with different polymeric coatings. A hydraulic test machine was used under load control with a sinusoidal load (R-ratio = 0.2), up to one million cycles.

Before the fatigue test was started, control specimens (without embedment of OF) were tested to failure under static loading to establish the loading levels required to achieve required strain levels in the subsequent fatigue test which were 600με/3000με and 1000με/5000με. All specimens were tested with linear strain gauges back-to-back, orientated with the loading direction. Loads and strains were measured regularly throughout the test.

TBE-Enhanced X-Ray Radiography

Tetrabromiethene (TBE) dye-enhanced X-ray radiography was used to detect any possible damage (such as breakage) in the embedded OF after fatigue test. This was done on Group A specimens only. The gripping areas on the tested specimens were initially removed and then the cut specimens were immersed in TBE solution for 20 minutes at room temperature before being exposed to X-ray under the condition of 20kV at 5mA for one minute at a distance of one meter.

Microscopic Analysis

Microscopic examination of the bonding behaviour of the embedded OF was performed on the cross-sections of the fatigued-tested specimens.

Multiple cross-sectioning was carried out by a diamond cutter. Up to eleven sectioned samples were prepared with surfaces perpendicular to the OF direction. These sectioned specimens were then mounted using a room-temperature-cure resin and polished with 1μm paste. The polished specimens were then examined under an optical microscope. The optical examination was later confirmed with a scanning electron microscope (SEM).

DURABILITY OF EMBEDDED OPTICAL FIBRES

Observations showed that no degradation was observed in the measured strain profiles during the fatigue testing and the specimens remained intact after one-million cyclic loading (one was tested up to 1.6 million cycles, still without obvious damage). TBE-enhanced X-ray radiography also indicated that no breakage occured in the embedded OF (Figure 2). Table 2 summarises the test results.

Table 2: Fatigue Test Results

Test Specimens	Cyclic Load Levels	Cyclic Strain Levels	Total Cycles	Comments
A-1	2.5kN-13kN	1000με-5000με	1.6 million	No external damage was observed
A-2	1.5kN-8.0kN	600με-3000με	1.0 million	
A-3	1.5kN-8.0kN	600με-3000με	1.0 million	
A-4	2.5kN-13kN	1000με-5000με	1.0 million	
B-1	2.5kN-13kN	1000με-5000με	1.0 million	
B-2	1.5kN-8.0kN	600με-3000με	1.0 million	
B-3	2.5kN-13kN	1000με-5000με	1.0 million	

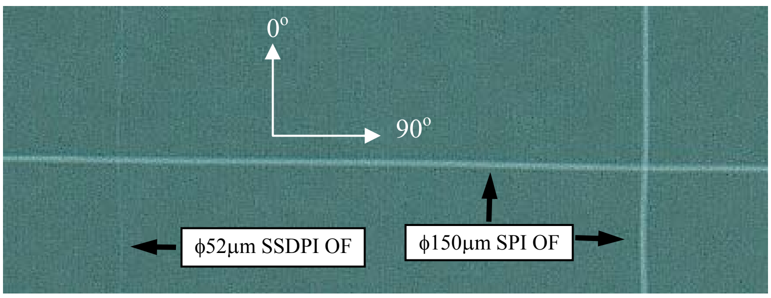


Figure 2: TBE-enhanced X-Ray radiography image for Group A specimen

Control Specimens: It appears that the outer interface between the epoxy matrix and the PI coating has a good bonding performance after cure with both SPI and SSDPI OFs.

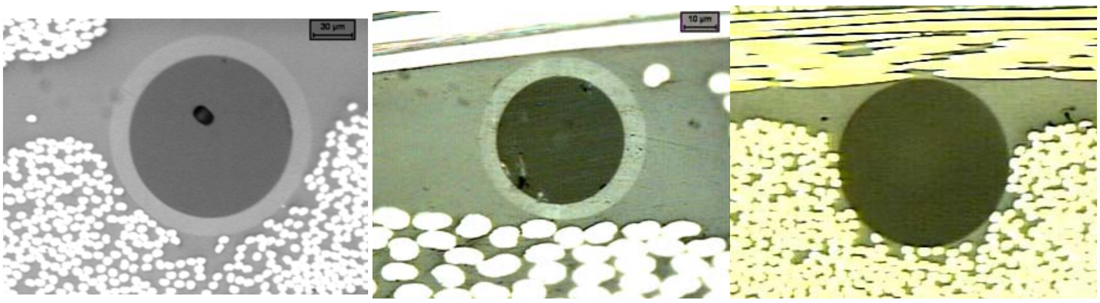
No disbonds were observed. However, microscopic examinations on Group A specimens showed some disbonds at the inner interface between the silica glass and the PI coating particularly for the embedded SPI OF, even though the samples were prepared carefully. Figure 3a is an example of showing no disbond.

This was partially due to the very low interfacial strength at the inner interface, and partially due to the sample preparation process (cutting and polishing). This observation is in agreement with that made by Gosh and Mittal [5] and DiFrancia et al. [7-9].

No disbond was observed in SSDPI OF and Group B specimen before fatigue testing was commenced. Typical micrographs showing good bonds at those interfaces are presented in Figure 3b and Figure 3c.

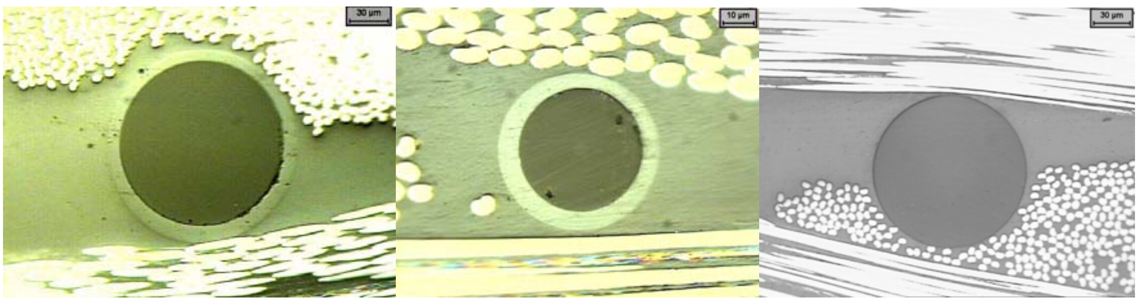
Fatigue-Tested Specimens: Significant disbonding was observed at the inner interface of SPI OF in Group A specimens. Nearly 80 % of the sectioned samples showed disbonds, including those OFs embedded in both directions (longitudinal or transverse) and at both strain levels used in the tests. Less than 20% of sectioned SSDPI OF samples showed a sign of disbonding while no disbonding was observed in the OFT samples.

Figure 4 shows the cross-sectional views of embedded OFs after one-million load cycles at the low strain level (600με~3000με). Figure 4a is one of the SPI OF specimens in Group A, in which significant disbond is observed, while Figure 4b is one of the SSDPI specimens in Group A, which also shows disbond. Figure 4c is one of the OFT specimens in Group B. No disbond is observed.



a. SPI b. SSDPI c. OFT

Figure 3: Cross-sectional views of embedded OFs before fatigue test



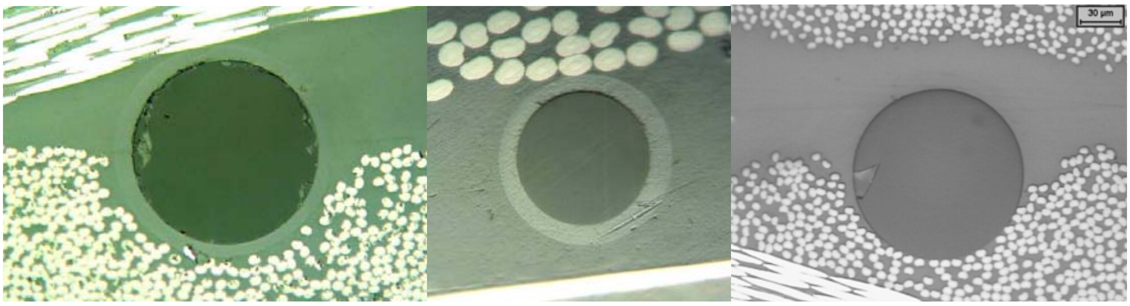
a. SPI b. SSDPI c. OFT

Figure 4: Cross-sectional views of embedded OFs after fatigue testing at low strain levels

Figure 5 shows the cross-sectional views of embedded OFs after one-million load cycles at the high strain level (1000με~5000με). Figure 5a is one of the micrographs for SPI OF in Group A, significant disbonding occurred. Figure 5b is one of the micrographs for SSDPI in Group A specimen, where a minor disbond was observed, while Figure 5c is one of the micrographs for OFT in Group B specimen and no disbond was observed.

Clearly, SSDPI performed better than SPI, with only minor disbonds being observed. It was not known what surface treatment the OF received before the PI-coating. Meanwhile, it is noted that the embedment of SSDPI OF in the interface between 0^{o} ply and the 90^{o} ply did not form such a significant matrix rich region as that formed by the SPI due to its smaller diameter [4]. As a consequence, it is likely that they would experience less thermal stress after curing and present less disruption to reinforcement fibre alignment.

It is also noted that no matrix cracking was evident due to the incorporation of PI-coated OF either after manufacture or after fatigue loading. This indicates that good adhesion exists between the PI coating and the epoxy matrix.



a. SPI b. SSDPI c. OFT

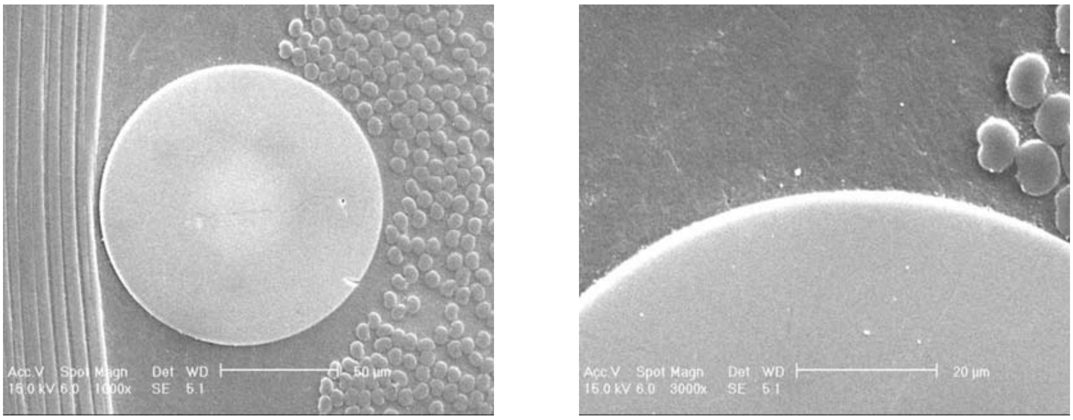
Figure 5: Cross-sectional views of embedded OFs after fatigue test at high strain levels

Cross-sectional examination revealed that the integrity of the interface between the silica glass and the epoxy matrix remained reasonably well for Group B specimens. There is some cracking observed within the silica glass (Figure 5c), which is believed to be due to the preparation process. This was confirmed later by a burning test, in which the matrix in the samples were burnt away (at $550^{o}C$) and the collected OFs showed no damage under microscopic examination. This indicated the cracks in the silica glass observed in Figures 5a and 5c were caused by the preparation process.

However, it is interesting to note that the cracks introduced by the preparation process did not promote disbonds between the silica glass and the epoxy matrix, indicating the adhesion at the interface is reasonably good for Group B specimens.

Some of the cross-sections were further examined by SEM. Good bonding was observed, as seen in Figure 6. This confirms the previous optical microscopic examination.

It was assumed that no damage was caused by the loading conditions to the embedded OF if no disbond was observed in those multi-sectioned specimens. This is assumed even though a disbond might not be continuous, and might only exist within a short length (less than the section length, ~10mm) and not propagate under the loading conditions. This appears to be realistic though simplistic and further understanding is required.



a b

Figure 6: SEM images showing good bonding between the silica glass and the epoxy matrix after one-million-cycle fatigue cyclic loading at high strain levels (a: x1000; b: x3000)

CONCLUSIONS

The conclusions that can be drawn from the preliminary investigation are:

1. Microscopic examination indicates that bonding at the inner interface between the polyimide coating and the silica glass is a major problem particularly for the standard PI-coated OF when embedded in a composite laminate. On the other hand, the specially made SSDPI OF showed improved bonding performance although minor disbonds were observed at both interfaces after one-million cyclic loads at both low and high strain levels.
2. The microscopic examination also revealed that the OFT performed well within the composite laminate. Good adhesion exists between the silica glass and the epoxy matrix and no disbond was observed after one-million cyclic loads at two strain levels. This OFT could be a useful alternative coating for OF surface-mounting and embedment applications.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the following organisations for supply of the standard and small-diameter PI-coated optical fibres:

- Redfern Optical Components, Australia
- Diamond Australia and
- Tokyo University

REFERENCES

1. “Fibre Optic Smart Structures”, ed. E. Udd, John Wiley and Sons, New York, 1995

2. P. D. Foote "Fiber Bragg grating strain sensors for aerospace smart structures", *Proc. SPIE* Vol. 2361, 1994, pp. 290
3. "Health Monitoring of Aerospace Structures: Smart Sensor Technologies and Signal Processing", ed. W. Staszewski, C. Boller and G. Tomlinson, John Wiley & Sons Ltd, 2004
4. N. Takeda, Y. Okabe, S. Yashiro, S. Takeda, T. Mizutani and R. Tsuji "Towards smart sensing in fabrication and maintenance of composite laminates with embedded small-diameter fibre Bragg grating sensors" Proc of the 10th US-Japan Conference on Composite Materials, ed. Fu-Kuo Chang, Sept. 16-18 2002
5. Gosh, M. K. and Mittal, K. L. (eds) "Polyimides: Fundamentals and Applications", Marcel Dekker, New York 1996
6. Plueddemann, E. P. "Silane Coupling Agents", 2nd edition, Plenum Press, New York1991
7. C. DiFranca, R. Claus, J. W. Hellgeth, T. C. Ward, "Structure/property correlations of several polyimide optical fiber coatings for embedding in an epoxy matrix", *Fiber Optic Smart Structures and Skins II*, Proc. SPIE Vol. 1170, 1989, pp. 505-512
8. C. DiFranca, R. Claus, J. W. Hellgeth, T. C. Ward, "Discussion of pull-out tests of polyimide coated optical fibers embedded in neat resin", *Fiber Optic Smart Materials and Structures*, ed. R. Claus, 1991, pp. 70-82
9. C. DiFranca, T. C. Ward, R. Claus, The single-fibre pull-out test. 1: Review and interpretation, *Journal of Composites Part A*, Vol. 27, 1996, pp.597-612
10. K. Levin "Interfacial strength of optical fibres", *Proc. 12th International Conference on Composite Materials*, ICCM-12, Paper 640, 1999
11. T. S. Jang, J. J. Lee, D. C. Lee, D. C. Seo, H. Kim "Fatigue behaviour of optical fiber sensors embedded in smart composite structures", *ICCM 11*, Vol. VI, Gold Coast, 14-18 July, 1997, pp. 582-589