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Characterization of both adhesion and interfacial interaction between optical fiber coating and structural adhesives

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Abstract. Optical fiber sensors are by now broadly accepted as an innovative and reliable device for structural health monitoring, to be used either embedded into or bonded on structures. The accuracy of the strain measurement achievable by optical fiber sensors is critically dependent on the characteristics of the bonding of the various interface layers involved in the sensor bonding/embedding (structure material and gluing agent, fiber coating and gluing agent, fiber coating and fiber core). In fact, the signal of the bonded/embedded optical fiber sensor must correspond to the strain experienced by the monitored structure, but the quality of each involved interface can affect the strain transfer. This paper faces the characterization, carried on by both mechanical tests and morphological analysis, of the strain transfer function resulting with epoxidic and vinylester gluing agent on polyimide and acrylate coated optical fibers.

Keywords: smart materials; interfaces; strength.

1. Introduction

Optical fiber sensors can be successfully employed for monitoring the stress of civil and mechanical engineering works. The underlying technique is by now established and applications for monitoring a large variety of civil and mechanical structures such as bridges, aircrafts, railways and dams were already reported in recent past (Doornik, *et al*. 2004, Kressel, *et al*. 2005, Voet, *et al*. 2005, Auflrger, *et al*. 2004). Moreover embedding fiber optic sensors in composite materials allows physical simulation models devoted to better understand traditional experimental methods used to study the interaction of composite material fibers with their surrounding material (Peters, *et al*. 2002).

Optical fiber sensors are embedded into or more simply stuck on structures by use of commercial gluing agents. In order to guarantee effective stress monitoring, the deformation of the structure must be efficiently transferred from the structure to the sensor, but the strain transfer process is complex because

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several interfaces are involved. Strain must be transferred from the structure itself to the gluing agent, and from the gluing agent to the optical fiber. The critical role of the interfaces involved in the strain transfer from the monitored structure to the monitoring sensor was already pointed out with respect of composite structures with embedded optical fibre sensors (Dasgupta and Sirkis 1992, D'Acquisto, *et al.* 2002). The sensing part of the optical fiber stays in the glass central part of the fiber, surrounded by a relatively thick polymeric coating, necessary for mechanical protection and to allow safe handling. Since strain must be transferred through the outer polymeric coating to the inner glass sensing part of the optical fiber, the efficiency of the strain measurement relays on the integrity, strength and toughness of the coating material. Thus, experimentally characterization of the interfacial properties of the polymeric coating through reproducible mechanical tests and accurate morphologic analysis becomes important, since there is no effective strain transfer from the structure to the sensor if the inner glass part of the fiber slides inside the outer coating (Peters, *et al.* 2002, Le Blanc 2005a,b).

In this paper we compare the efficiency of the strain transfer obtained with different polymeric coatings of the optical fiber and different gluing agents, analyzing the morphological aspects of the interfaces and measuring the values of strain in correspondence of which sliding occurs at the interfaces. Experimental measurements are conducted with optical fibers embedded in the gluing agent just for a short length (< 5 cm), referring to the experimental set-up commonly adopted with Fiber Bragg Grating (FBG) sensors, due to their unrivalled outstanding role gained in practical applications of structural monitoring by fiber optic sensors.

2. Experimental set up and procedure

In order to study the strain transfer efficiency, four commercial gluing agents and two commercial optical fiber with different coatings have been used. Vinylester and epoxidic gluing agents have been employed: epoxidic structural adhesive M-Bond AE-10, epoxidic structural adhesive Araldite 2011 (both adhesives suitable for on surface optical fiber sticking); epoxidic resin Mapewrap 31 and vinylester resin Norpol Dion 9800 (both resins widely used as matrix in fiber reinforced polymeric composites). The two structural adhesives and the epoxidic resins cure at room temperature, while the vinylester resin needs hot curing (180 °C). The optical fibers are: a double layer acrylate coating; a single layer polyimide coating. Both optical fibers have the same core and cladding type (monomodal SMF28 compliant glass fiber) and are commercially available, thus differing only for the external coating type and dimension (external diameter: 145±10 μm acrylate coated; 155±10 μm polyimide coated). Moreover, a 'bare' optical fiber was also considered: the 'bare' fiber has no coating; it was obtained by removing the acrylate coating from the acrylate coated fiber, using a standard mechanical stripper. The 'bare' fiber was used as a reference, in order to evaluate strain transfer with no (neither acrylate, nor polyimide) coating interface.

Experimental measurements have been carried out with specimens produced with all possible combination of fiber type and gluing agent. Specimens are 200 mm long optical fibers with the ends glued on two aluminum plates accurately degreased. For each combination of fiber type and gluing agent, 8 specimens were prepared, arranged in 4 pairs with different length of the glued segments (20 mm, 30 mm, 40 mm, 50 mm). Fig. 1 shows a sketch of the specimen geometry. Gluing has been performed spreading a thin layer of the gluing agent on the aluminum plate and merging the fiber end in the layer for the desired length. Structural adhesives and epoxidic resin Mapewrap 31 have been cured at room temperature for longer than 48 hours before testing; Norpol Dion 9800 vinylester resin has been cured

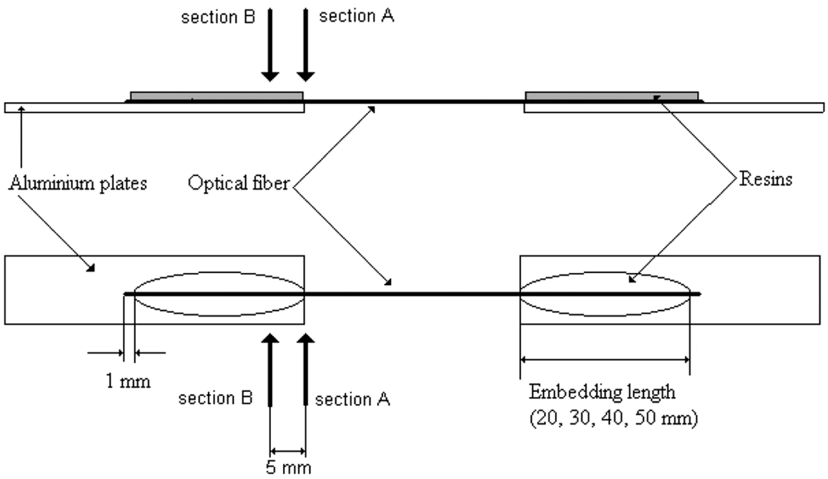


Fig. 1 Tensile test specimen geometry

at 180 °C for few minutes.

Experimental results are in the following presented grouping specimens according to fiber coating:

a) Acrylate group, i.e. fiber coated with double acrylate coating;

b) Bare group, i.e. bare fiber;

c) Polymide group, i.e. fiber with a single polyimide coating.

Specimens were all subjected to traction tests by tensile machine (INSTRON IX 3360 Series) with crosshead speed 0.5 mm/min and in controlled room condition. After tensile test, specimens have been subjected to a morphologic analysis by stereomicroscopy and successively by scanning electron microscope (SEM). This analysis was devoted to observe the state of the several involved interfaces: fiber coating and gluing agent; gluing agent and aluminum surface. In the morphological analysis, special attention was paid to verify the effect of the traction test on the interface between the fiber coating and the fiber glass inner part: in fact, if the sensing part of the fiber slides inside the coating tube, strain measurement becomes unreliable.

3. Results and discussion

Fig. 2 shows the results of tensile tests carried out on the bare group. It can be observed that during tensile test bare fibers don't slide, but simply they show a typical brittle behavior (linear outline until they break). Bare fiber breaks in correspondence of an applied force ranging from 20 up to 24 N, corresponding to values of σ_R comprised between 1600 and 2000 MPa. Breaking point is independent of both embedding length and kind of resin or structural adhesive employed, and it is lower than the nominal value declared by the manufacturer. This low breaking value can be considered dependent on microcracks produced during the mechanical stripping operation.

Test carried out on acrylate group show a different behavior respect to those observed in bare fiber group. Similar trends have been obtained with all kind of resins employed in this experimentation. As examples Fig. 3 reports the results of acrylate coated fiber coupled with M-Bond AE-10 structural adhesive at several embedding lengths. Tensile tests show a first almost linear behavior until load

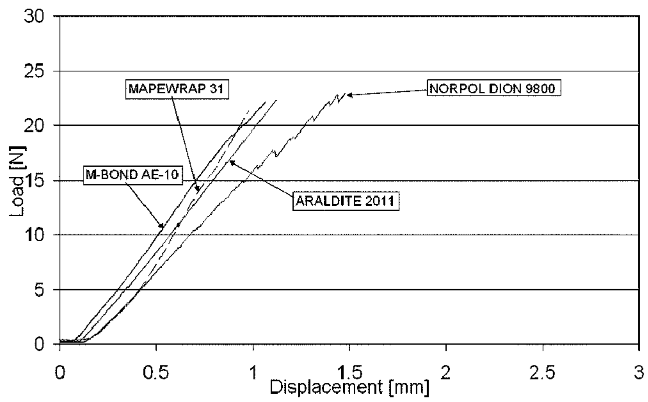


Fig. 2 Bare fiber-resin coupling. Comparison between the behavior of the four resins considered (embedding length 30 mm)

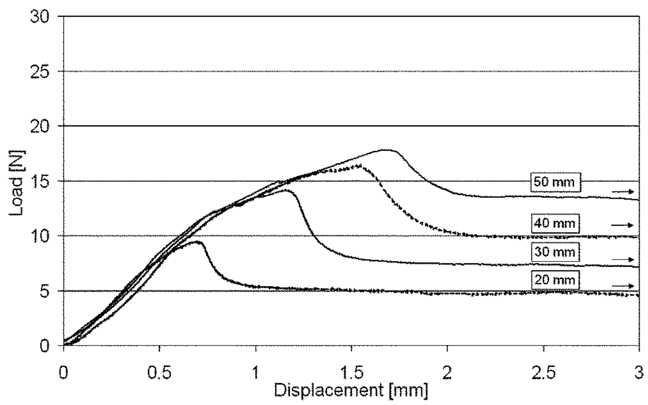


Fig. 3 Tensile test result about acrylate group coupled with M-Bond AE-10 structural adhesive at several embedding lengths

reaches its highest value comprised between 8 and 18 N; this peak value increases proportionally with the length of the fiber embedded into the polymer. After this peak the load decreases rapidly up to a value comprised between 5 and 14 N. Also this value increases proportionally with the length of the fiber embedded into the polymer. Then the load decreases slowly up to the end of the test. It was observed by visual inspection of the specimen during the tensile test that, after the peak, the fiber pull out from the polymer. In particular the coating remains linked to the resins/adhesives, while the glass fiber slides. At the end of the test we always obtain a glass fiber stripped out from its acrylate coating. This behavior has been confirmed by the morphological observation carried out on the specimens after tensile test. Fig. 4 is a steromicroscopy photo of a broken specimen taken in correspondence of section A of figure 1. It is clearly visible the acrylate coating without glass fiber inside. Fig. 5 shows a SEM photo of a section of the same specimen taken 5 mm from the previous section (section B of Fig. 1). In this photo the two acrylate coatings can be easily identified, while in the centre glass fiber is not present. acrylate coating seems well bonded to the resin.

The peak value is the detachment strength between glass fiber and coating and it is an useful index of the intensity of the surface forces which assure the adhesion between glass fiber and acrylate and allow

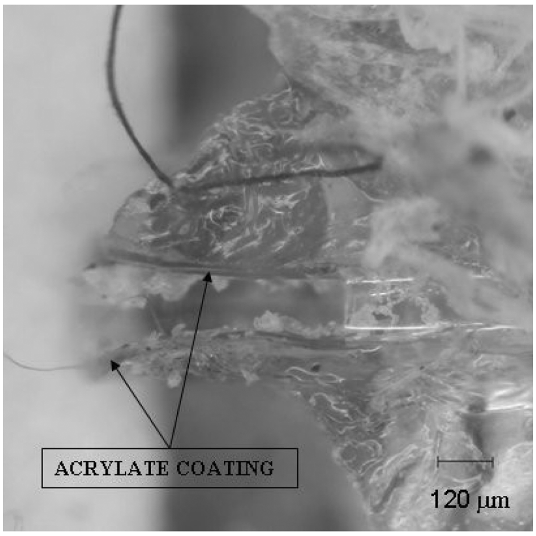


Fig. 4 Acrylate coating which remains attached to the resin after glass fiber sliding

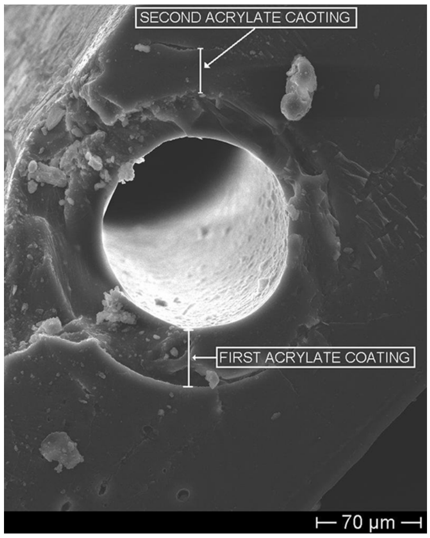


Fig. 5 SEM micrograph of the section of a acrylate-group fiber-resin specimen after glass fiber sliding

the strain transfer through this interface. Because of the different nature of the involved materials (glass fiber is a silicate while coating is a polymer), these surface forces are due to weak bond like electrostatic or Van der Waals interaction between molecules of the surfaces into contact. The peak value depends from the gluing length. The detachment between glass fiber and acrylate coating is due to the shear stress at the interface glass fiber-coating. For this reasons an higher gluing length, which correspond to an higher glued surface, will produce an higher detachment load. In any case the detachment shear stress is almost independent from the gluing length. Furthermore it can be observed that this peak load is lower than the breaking load reached with bare fiber.

After the glass fiber-coating detachment, the load concentrates on the acrylate coating which breaks in the much weak section. In all specimen this section is always in proximity of the point where fiber come out from the resin (section A of Fig. 1).

The second value of the load identified in tensile tests, is the sliding friction strength and it is related to the force that must be applied in order to maintain the relative movement between glass fiber and coating. Obviously this strength is proportional to the interface surface and then it increases with the length of the fiber embedded into the polymer and it reduces as the glass fiber is pulled out.

Similar results have been obtained with the other resins. The only observed difference regards the peak load which sensibly changes with the employed resin. Fig. 6 shows the different behavior of acrylate coated fiber coupled with several polymeric matrices. acrylate coating-Norpol Dion 9800 resin is the worst coupling because there is a poor wettability between acrylic coating and vinylester resin. Furthermore acrylate coating, during hot curing at temperature around 80 °C, is damaged (Kalamkarov, *et al*. 1998).

A completely different behavior is shown by the polymmide group. Also in this case no sensible different behaviors have been recorded changing the gluing agent.

As example, Figs. 7(a) and (b) show tensile test results of the polyimide fiber coupled with M-Bond AE-10 structural adhesive at several embedding lengths. Several differences between the polyimide and the acrylate group can be observed. First, the polyimide group doesn't show the load drop which

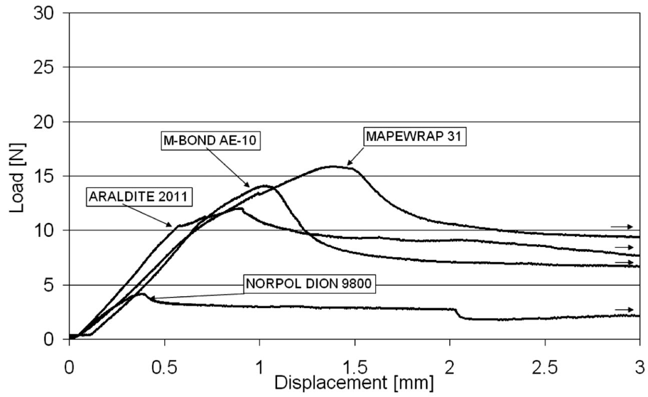
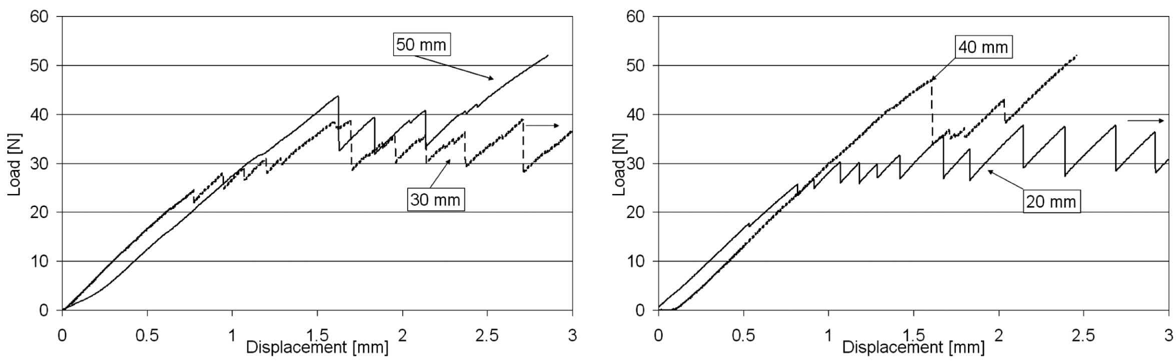


Fig. 6 Acrylate coated fiber-resin coupling. Comparison between the behavior of the four resins considered (embedding length 30 mm)

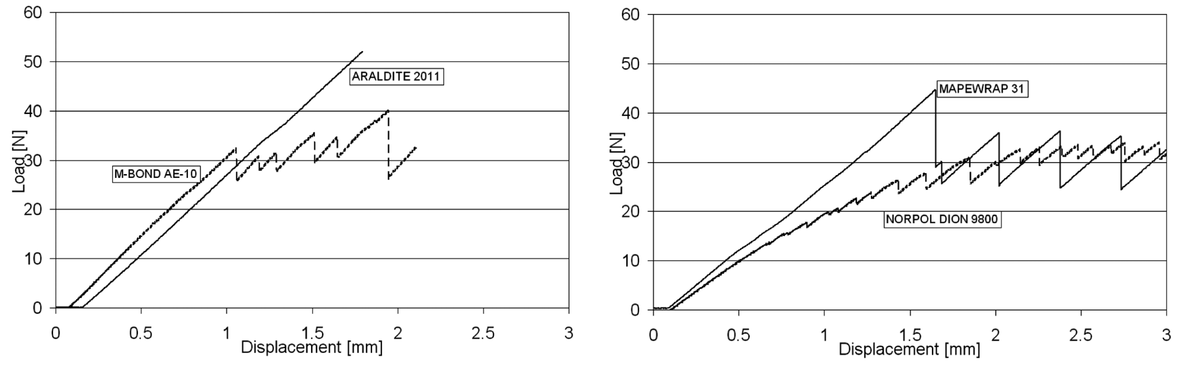


(a) (b)

Fig. 7 (a) Tensile test result about Polyimide group coupled with M-Bond AE-10 structural adhesive at several embedding lengths, (b) Tensile test result about Polyimide group coupled with M-Bond AE-10 structural adhesive at several embedding lengths

characterizes tensile test of the acrylate group. The load reaches the peak and then remains almost constant, showing a particular tooth outline. Moreover there isn't any dependence between embedding lengths and the nonlinearity limit load that has been reached during tensile test.

Figs. 8(a) and (b) show the behavior of polyimide coated fiber coupled with several polymeric matrices. In this case all the couplings show the elastic linear behavior until the value of the first detachment strength. This value is changes with the employed resins. In particular The Vinilesther resin Norpol Dion 9800 shows the worst behavior: the minimum detachment load is approximately 18 N and furthermore, unlike the other gluing agent, it loses the linear behavior at approximately 10 N. The maximum load is comparable with those of the other resins (about 30 N). However, despite the other, this coupling shows a strong dependence of the maximum load on the embedding length. Maximum load increases with the embedding length, like observed in the acrylate group system. Visual inspections of specimens during and after tensile tests reveal that the whole optical fiber (glass fiber + polyimide coating) pulls out from the vinylester resins. Fig. 9 is a macrophoto of Norpol Dion 9800-Kapton specimen after tensile test. It is clearly visible that the coating is still adherent to the whole glass fiber. In the same photo, an empty channel left by the fiber pull out can be identified. The low first



(a) (b)

Fig. 8 (a) Polyimide coated fiber-resin coupling. Comparison between the behavior of the four resins considered (embedding length 30 mm), (b) Polyimide coated fiber-resin coupling. Comparison between the behavior of the four resins considered (embedding length 30 mm)

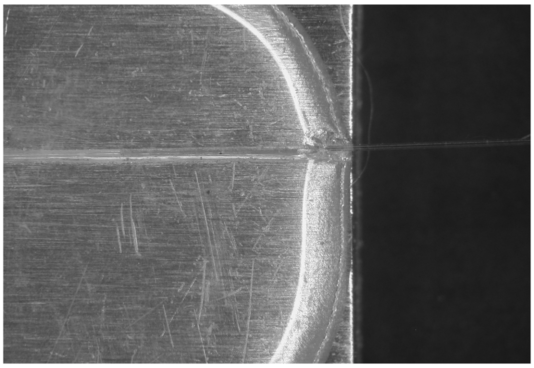


Fig. 9 Macrophoto of polyimide coated optical fiber coupled with Norpol Dion 9800

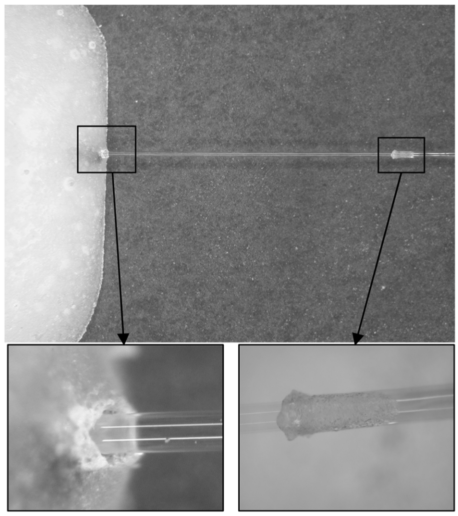


Fig. 10 Macrophoto of polyimide coated optical fiber coupled with Mapewrap 31

detachment load values and the morphology of sliding phenomena indicate that vinileshter and polymmide are low chemically compatible.

The epoxidic resins Mapewrap 31 and the epoxidic adhesive M-Bond AE-10 show the same behavior with a linear outline until the first detachment load (ranging from 30 up to 45 N). After the first detachment all these polymers show the tooth outline with continuous load drops followed by steps where load increases. In particular it can be observed that the slope of the increasing step load is always the same. During tensile test a sound emission has been recorded in correspondence of load drop. Visual inspections of the specimens during and after tensile test show pull out phenomena between polymmide coating and glass fiber. Fig. 10 is a macrophoto of a Mapewrap 31- Kapton specimen after tensile test. It can be observed that: the coating brakes in proximity of section A, the fiber pulls out from its coating, the coating is strongly etched by the resin. The same braking morphology has been detected

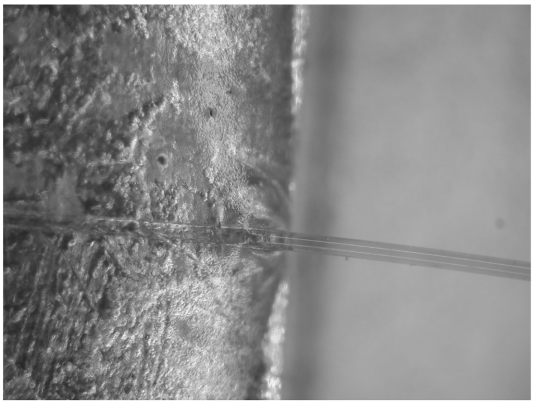


Fig. 11 Macrophoto of polyimide coated optical fiber coupled with Araldite 2011

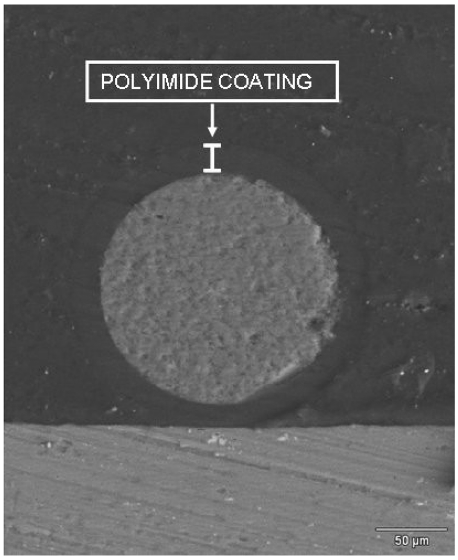
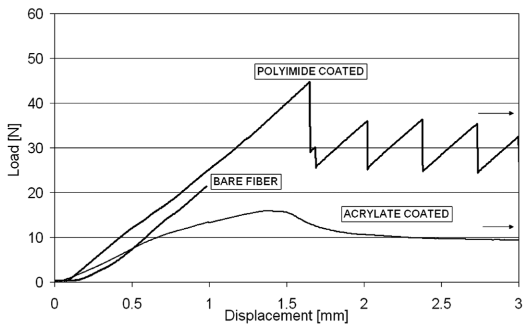
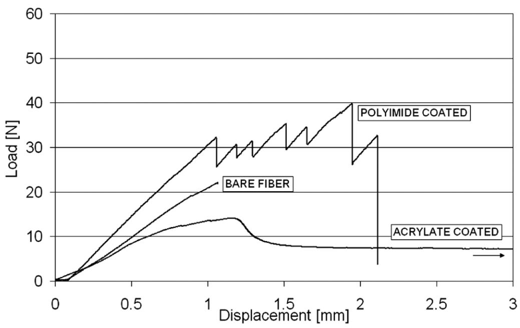


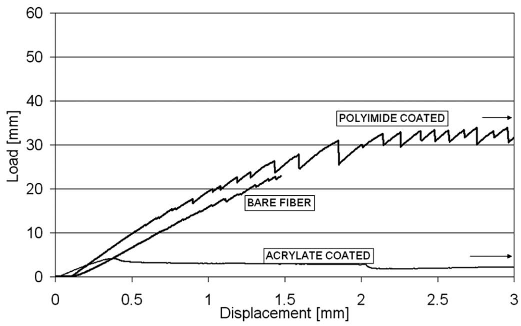
Fig. 12 SEM micrograph of the section of a polyimide coated optical fiber coupled with Araldite 2011



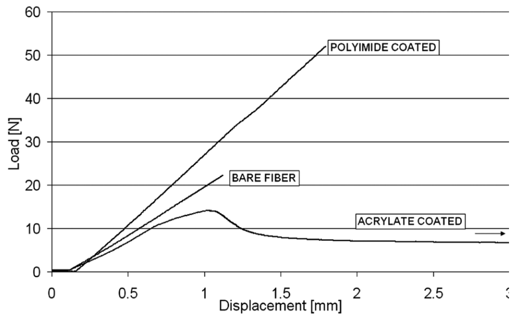
(a)



(b)



(c)



(d)

Fig. 13 (a) Epoxidic resin Mapewrap 31, Comparison between the behavior of the three kind of fibers considered (embedding length 30 mm), (b) Epoxidic structural adhesive M-Bond AE-10, Comparison between the behavior of the three kind of fibers considered (embedding length 30 mm), (c) Vinylester resin Norpol Dion 9800, Comparison between the behavior of the three kind of fibers considered (embedding length 30 mm), (d) Epoxidic structural adhesive Araldite 2011, Comparison between the behavior of the three kind of fibers considered (embedding length 30 mm)

Table 1 Non-linearity limit load for the several studied system

	Kind of fiber		
	Acrylate coated	Bare fiber	Polyimide coated
Structural adhesive			
M-Bond AE10	10.7 N	22.2 N	32.3 N
Araldite 2011	11.7 N	16.7 N	52.1 N
Resins for FRP			
Mapewrap 311 (epoxidic resin)	11.1 N	21.4 N	44.7 N
Norpol Dion 9800 (vinylester resin)	4.2 N	15.2 N	25.0 N

Note: Table 1 Maximum load reached during tensile test

for M-Bond AE10-Kapton system. The only difference regards a lower coating etching degree.

Pull out phenomena has been recorded also in the Araldite 2011- polymmide system, even if several times fibers broke outside the embedding before starting to pull out. Fig. 11 shows a macrophoto of an araldite 2011-polymmide specimen after test. It is clearly visible that the whole optical fiber pulls out form the epoxidic resins. Fig. 12 shows a SEM photo taken in section B of Fig. 1 of a section of a specimen which didn't show pull out. Polyimide coatings seems still well bonded to the resins and glass fiber is still present and it is well bonded to its coating.

Fig. 13(a-d) show a comparison between different kind of fiber embedded into the same resin (embedding length 30 mm). It can be noted that the acrylate group reaches the lowest load values; Polyimide group reaches the highest values, whereas Bare group reaches intermediate values, as it can also be seen in Table 1 where maximum loads of the different couplings have been compared (embedding length 30 mm).

4. Conclusions

With this experimental study, useful informations about work range of the coating of fiber optic sensors have been obtained.

All kinds of fiber optic sensors (based either on intensity or interferometry or wavelength shift) work correctly when there is a perfect transfer of the loads from the structure/material to the glass fiber. Polymeric coating allows to keep physical integrity of the glass fiber during its handling and installation, but this coating introduces a third material between glass fiber and structure/material. So stress/strain must be transferred from the structure to the material not through one interface (material-glass fiber), but through two interfaces (material-coating and coating-glass fiber). It is important that the strength of both these interfaces is high enough to allow the stress/strain transfer from the material to the glass fiber without the interface breaking. For this reason several properties of the coating material, like wettability and chemical compatibility with the host material, must be considered.

It was observed that all kinds of epoxidic resins studied react with both coatings. Resins chemically reacts with coatings creating a strong bond between these two materials. Only in the case of the epoxidic adhesive Araldite 2011 coupled with polymmide coated fiber no reactions signs have been detected. Vinilesther resins is not compatible with both coating. No chemical reaction sign has been observed, furthermore fiber-vinilesther resin repulsion phenomena has been recorded during acrylate group specimen manufacturing.

In general the weak point of the stress/strain transfer system is the glass fiber-coating interface. The strength of this interface could be modified during optical fiber embedding process by residual tension or coating damaging produced by resins-coating chemical interaction and/or resins curing.

The best results have been obtained employing the polyimide coated optical fiber. Probably this is due to the better mechanical properties of this material or by a low chemical interaction which can reduce the damaging effect of the manufacturing process on the glass fibre-coating interface strength.

In absolute the best behavior has been shown by the Araldite 2001-Kapton (polymmide coated) optical fiber. This system shows a completely different braking morphology. No epoxidic resin-polyimide coating interaction has been detected. The weak point is the resin-coating interface. The absence of any chemical coating damagement probably the optical fiber maintain its original properties. Furthermore the araldite-polyimide interface strength is comparable to those of the optical fiber. In fact fiber pull out has been observed as much as fiber braking.

Polyimide coating can guarantee higher level of stress/strain transfer from the material structure to the sensorized optical fiber. However this coating is more expensive then acrylate coating; furthermore acrylate is easy to manipulate, to weld during connectorization process and to remove both chemically or mechanically. So it must be evaluate both the technical and the economic aspect of the particular application in order to choose for a kind of fiber and not for the other.

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