Characterization of the interface between composites and embedded Fiber Optic sensors or NiTiNOL wires

P. Bettini*, L. Di Landro, A. Airoldi, A. Baldi and G. Sala

Abstract

The interface between constitutive elements always plays a crucial role on the global performances of composites. Among smart materials, FO and NiTiNOL are the most popular to be used as embedded sensors and actuators in structures. Nevertheless, their adhesion properties with the host materials are not still well known and few experimental data can be found in the literature also due to the lack of the specific testing methods. The aim of this work is to give a contribute to the study of the behavior of such interfaces. Two different FO having poly-acrylate and poly-imide coating were embedded in epoxy resin blocks and their interface was characterized adopting a Pull-Out tests. In the same way the adhesion between resin and NiTiNOL wires was evaluated both in austenite and in martensite phase.

Keywords: Smart materials; Interface analysis; NiTiNOL actuators; Fiber Optic sensors; Pull-Out test.

1. Introduction

The efficiency of a smart structure is strongly dependent on the interface between the sensors/actuators and the host materials. Their adhesion is an essential requirement to ensure high load transfer capability, to guarantee high levels of accuracy of the sensors and to improve the activation efficiency of the actuators. The design of embedded SMA actuators, for instance, is now limited to the maximum level of pre-strain that can be induced on the material before failures occur in the structure [1].

For these reasons the study of the interface assumes a fundamental importance and the work described in this paper gives a contribute to this goal.

*Corresponding author. Tel.:+39-02-2399-8033; fax:+39-02-2399-8028.
E-mail address: bettini@aero.polimi.it (P. Bettini)
Similar considerations have always driven the advance of composite materials. As a matter of fact, the interface between reinforcements and matrix plays a crucial role on the global performances of the material, in terms of mechanical properties, damage tolerance as well as fatigue behavior. In absence of dedicated methods, the characterization of the interface between composites and embedded Fiber Optic (FO) sensors or NiTiNOL wires can be done adopting one of the several methods developed to study the interface between reinforcing fibers and matrix in the composites (like microtension, fragmentation, microcompression, pull-out tests) [2]. These methods can be independently used to evaluate the interfacial shear strength (IFSS) and to understand failure mechanisms. Pull-out test was selected for this work due to its simple set-up and specimens manufacturing [3].

Specimens are made up of small resin blocks with a single fiber embedded for a length \( L \) that must be less than a critical value \( L_C \); the critical length is defined as the embedded length that gives a pull-out force equal to the fiber failure one. In such a way debonding occurs instead of fiber failure (Fig. 1-a). The average value of maximum shear stress is obtained from the tests by using an equilibrium relationship based on a simplified stress distribution at the interface [2]:

\[
\tau_{IFSS} = \frac{F_{MAX}}{\pi \cdot d \cdot L}
\]

where \( F_{MAX} \) is the maximum recorded force corresponding to the debonding and \( d \) is the fiber diameter. Two observations can be done. First, this formula underestimates the actual stress levels since it assumes a uniform state-of-shear-stress along the interface in the fiber direction. In fact, the failure propagation mechanism implies much different stress conditions as shown in Figure 1-b.

The fracture nucleates and propagates due to a peak of stress localized at the propagation front. Second, the friction forces between resin and fiber are neglected. Their evaluation could be done considering both friction coefficients and pressure at the interface due to technological cycles but they are very difficult to be obtained [4].

Fig. 1. Standard Pull-Out test to evaluate the InterFacial Shear Stress between fiber and resin of the composite materials: (a) specimen scheme and (b) failure mechanism.

2. Characterization of the SMA

Before the production of SMA specimens, mechanical and physical properties of the NiTiNOL are needed for the setting up of the manufacturing stage. As a matter of facts, the adhesion between wires and resin has to be done in the austenite phase of SMA (the same phase present during high temperature curing cycle of host composite materials).
Moreover, to avoid every working change of SMA actuators it is important to eliminate other minor phases that could be present [5]. DSC analyses were performed and the transition temperatures were evaluated. Results showed a multistep forward transition due to the presence of the R-phase that can be removed by an annealing treatment [6-7].

Figure 2-a shows the results obtained with the SMA wire as-received. The superposition of all the scanning cycles (after the first one) confirms the high-quality of the analyses. The scanning n.4 (dashed line) underlines that Austenite Start and Finish Temperatures ($A_S$ and $A_F$) do not change even if the reverse transition begins in the R-phase condition.

3. Specimens Manufacturing and Tests Set-up

Three different kinds of specimens were made and tested. In the first one NiTiNOL wires were embedded in blocks made of Araldite LY5052/Aradur 5052 epoxy resin for a length of about 2÷3 mm. SMA was embedded in its austenite phase. A curing cycle of 3h at 100°C was chosen in order to have the glass transition temperature (130°C) higher than $A_F$.

By the same curing process, the other kinds of specimens were produced embedding two types of FO with different polymeric coatings. During the manufacturing phase a particular attention was used to eliminate voids in the resin and to avoid its rising up along the wires and FO (Fig. 3-a). After a degassing process, the resin was poured in a special aluminum mold equipped with a drilled PTFE plate for the wires and FO crossing. Moreover, the mold had a flexible wall to prevent possible residual stresses due to curing cycles (Fig. 3-b). At the end of curing all the SMA specimens were cooled down to ensure martensite phase.

FO required the adoption of dedicated tabs to clamp it on the testing machine (Fig. 3-c) due to their high fragility. A specific testing equipment was also designed and manufactured to apply the restrained top loading method so that pre-load stresses on the resin blocks due to the test machine clamping could be avoided (Fig. 3-d).

SMA specimens were tested at low and high temperature to evaluate the IFSS in martensite and in austenite phase, respectively. A hydrofluoric acid (HF) surface treatment was also performed on the embedded wires to evaluate the possible improvement of the adhesion between resin and wires.

Fig. 2. (a) DSC analysis of the Dynalloy SMA actuators: determination of transition temperatures without stress applied; (b) Stress dependence of the Dynalloy SMA transition temperatures obtained using a Dynamo Mechanical Analyzer.

FO specimens were tested at Room Temperature and only the influence of polymeric coatings was considered.
All the tests were conducted using a monoaxial electro-mechanical testing machine (INSTRON 4302) in displacement control mode with crosshead speed of 0.5mm/min equipped with a 1KN cell load and a thermal chamber.

![Image](a)

![Image](b)

![Image](c)

![Image](d)

Fig. 3. (a) Detailed view of a SMA specimen showing the absence of resin rising; (b) Special mold for the Pull-Out specimens manufacturing; (c) Dedicated tabs for clamping FO; (d) Testing equipment for applying the restrained top loading method on the specimens.

4. Results

4.1. SMA/Epoxy interface

All the pull-out tests (performed both on martensite with and without HF surface treatment as well as Austenite phase) led to complete debonding. Results are summarized in Figure 4-a that reports the force versus displacement curves. The load always rises until debonding and then a sudden drop occurs; afterward the effects of friction between wire and resin are evidenced by a slightly varying force.

In the martensite tests, the debonding occurs at higher loads than those leading to stress induced martensite (SIM) underlined by the presence of a plateau. The comparison between the curves referred to HF treated and not treated wires does not show appreciable differences.
On the other hand, in austenite tests, the debonding loads are higher than in martensite ones. This behavior could be related to the thermal mismatch between resin and metal which affects the mechanical constraint on the wires, or to a variation of wire roughness consequent to phase transformation. Figure 4-b shows IFSS obtained from all experimental tests and it underlines the SMA phase influence on the interface.

Even if experimental data are limited for a reliable statistical analysis, the low standard deviation obtained permits to evaluate a meaningful average of IFSS reported in Table 1. It can be seen that austenite interfacial shear stress (20.05 MPa) is three times larger than the martensite one (7.59 MPa) while the interface is not significantly influenced by the HF treatment (6.67 MPa).

Table 1. Interfacial Shear Stress obtained from Pull-Out tests on SMA specimens.

|                  | Martensite - HF treated | Martensite | Austenite |
|------------------|-------------------------|------------|-----------|
| Number of data   | 15                      | 10         | 15        |
| Average IFSS (MPa) | 6.67                    | 7.59       | 20.05     |
| Standard Deviation | 1.35                    | 1.32       | 1.43      |

4.2. FO/Epoxy interface

FO specimens exhibited a critical length of about 2 mm. Figure 5-a shows the representative curves referred to the two different coatings. It is clear the difference in terms of load levels failure. However, all the specimens show a sudden yielding even if friction phenomena were noticeable for the poly-acrylate ones.

A microscopic analysis on tested FO also detected a different damage mechanism (Fig. 6). In the case of poly-acrylate FO a mixed adhesive/cohesive fracture occurred: the coating failed meanwhile both epoxy/coating and coating/cladding interface fail. Therefore, due to the impossibility to uniquely individuate a broken interface, the calculation of IFSS was done assuming in the formula (1) the value of an average surface.

On the other hand, the collapse of poly-imide specimens involves only adhesive phenomena and the IFSS could be located at the surface of the cladding/coating interface. Figure 5-b reports all experimental
data that are summarized in the Table 2. It can be seen that poly-imide IFSS (32.71 MPa) is much larger than the poly-acrylate one (2.69 MPa).

![Graph showing force vs displacement and interfacial shear stress vs embedded length for poly-acrylate and poly-imide coatings.](image)

Fig. 5. (a) Comparison among the interfaces Epoxy resin/FO with the 2 different coating (Poly-acrylate and Poly-imide): (a) Pull-Out curves; (b) InterFacial Shear Stress obtained.

![Images of FO with damaged and perfectly cleaned cladding.](image)

Fig. 6. Microscopic analysis on tested FO: (a) damaged poly-acrylate coating; (b) perfectly cleaned cladding of poly-imide coated FO.

|          | FO with poly-acrylate coating | FO with poly-imide coating |
|----------|-------------------------------|---------------------------|
| Number of data | 13                            | 10                        |
| Average IFSS (MPa) | 2.69                          | 32.71                     |
| Standard Deviation | 1.01                          | 3.49                      |

### Table 2. InterFacial Shear Stress obtained from Pull-Out tests on SMA specimens.

5. Conclusion

NiTiNOL pull-out tests have shown that the highest load transfer capability is obtained when the wires are in the austenite phase (three times higher than martensite). This is a good result considering that the activation of NiTiNOL actuators is driven by the austenite phase. Nevertheless, being NiTiNOL wires also a discontinuity inside the host materials, values of interfacial shear stress have to be still increased.
both for martensite and austenite phase. It should never be neglected that the interfaces always are a weak point of the structure. The HF acid surface treatment seems doesn’t work.

FO Pull-Out tests underlined the superiority of the poly-imide coating. Besides the higher average IFSS (that resulted to be about 10 times larger than the poly-acrylate one) other aspects indicate poly-imide FO as the first choice for applications on Smart Structures. In fact, the strength of the material and the thin thickness of the coating (only 6\(\mu\)m instead of 60\(\mu\)m for poly-acrylate) could give to more accurate measures of sensors. Finally, being smaller poly-imide FO are less invasive on the host material.

Acknowledgements

The contribution of FIRB project RBIP06AWF9 is gratefully acknowledged. The authors wish to thank Mrs. M.R. Pagano and Mr. A. Maggiolini for their assistance during specimens production and testing.

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