Single fiber pull-out test of nitinol-silicon-textile composite

L. Horný, J. Veselý, H. Chlup, K. Janouchová, and M. Vyšanská

Abstract—**This is preliminary report describing results of the single fiber pull-out tests conducted with newly developed composite material based on integration of NiTi filaments into the silicon-polyester matrix. Nominal shear stress at debonding, debonding force, and spring constant characterizing fiber-matrix interface prior to a failure of the adhesion, were derived from experiments. It was concluded that current data does not support a hypothesis of rate-dependent adhesion limit and mechanical response before the debonding.**

Index Terms—**Debonding, Hybrid composite, Interface, Nitinol, Pull-out test.**

I. INTRODUCTION

SHAPE memory alloys (SMA) are successfully applied in aerospace and automotive engineering especially in a design of actuators, manipulators and regulating elements [1]–[4]. They are also frequently used in cardiovascular engineering as a material for self-expandable stents, stent-grafts or wired scaffolds for heart valve prostheses [5]–[8]. In all these applications, superelasticity (recoverable deformation up to 10%), thermomechanical shape memory effect or high damping capacity are utilized to endow a final structure with properties unachievable by common metals. Physical principle underlying these phenomena is thermoelastic austenite ↔ martensite phase transformation achievable around room temperature [9]–[11].

In the past decade, theoretical and experimental treatment of a possibility to implement SMA wires into polymeric matrix has received wide attention. These materials are usually referred to as hybrid composites and were obtained mostly by reinforcing thermoplastic or resin matrix with SMA filaments [12]–[15]. An attention has also been paid to a development of composites based on elastomer matrix [16]–[19].

Present study is focused on determining the mechanical properties of the hybrid composite manufactured from silicon–polyester matrix reinforced with nitinol filaments (NiTi alloy developed in Naval Ordnance Laboratory in 1962; probably the most frequently used SMA worldwide). Since a key to successful composite is fiber–matrix interface, we will limit ourselves to the investigation of an adhesion between the components of the composite. In what follows, methodology and results of the single fiber pull-out test are described with respect to the effect of pulling velocity.

II. METHODS

In this study, single fiber pull-out tests conducted with newly developed composite material, NiTiSilTex, will be described. First of all, NiTiSilTex fabrication should be explained. It is the material developed cooperatively at the faculty of Textile Engineering of the Technical University in Liberec and at the Institute of Physics of the Czech Academy of Sciences [16], [20].

NiTiSilTex. The material was obtained in two steps: (1) braiding thermoplastic-coated NiTi filaments into knitted polyester (PES) textile, and (2) embedding NiTi-PES network into silicon elastomer (trademark ZA4LT) which was cross-linked at room temperature.

Samples. Rectangular strips were cut manually by a scalpel from a block of NiTiSilTex. This block had edges with protruding NiTi filaments. Protruding parts of the filaments were utilized in pull-out experiments. Both protruding end of a filament and composite block were glued by cyanoacrylate adhesive to linen cords which were used upon mounting a specimen into a testing machine (see Fig. 1).

NiTi wires (0.075 mm in radius) were purchased form Fort Wayne Metals Ltd. They were delivered in so-called straight-annealed state and exhibited superelastic behavior at temperatures above -10 °C.

Experiments. All experiments were conducted in the Laboratory of Biomechanics at the Faculty of Mechanical Engineering of the Czech Technical University in Prague. The samples were mounted into the custom-specific tensile testing machine (Zwick/Roell) equipped with ± 50 N force transducer (U9B, HBM) and built-in videoextensometer (Messphysik). A movement of the electro-mechanical actuator (resolution in positionig 0.001 um) was transferred onto the sample via linen cords. Pull-out tests were performed at two loading velocities, 0.05 mm/s and 0.2 mm/s. Acting force, actuator's position and the distance between marks made on the sample (one mark at the end of the composite block and one on the protruding filament, see Fig. 1) were recorded during experiments.

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L. H., J. V. and H. C. are with Faculty of Mechanical Engineering of the Czech Technical University in Prague, Technicka 4, 166 07, Prague, Czech Republic (L.H. is corresponding author: +420-224352690; e-mail: lukas.horny@fs.cvut.cz; J. V., H.C., and R. Ž. have email adresses as follow: jendavesely@gmail.cz, chynek@seznam.cz, rudolf.zitny@fs.cvut.cz).

K. J. and M. V. are with Faculty of Textile Engineering of the Technical University of Liberec, Studentska 2, 461 17, Liberec, Czech Republic (emails: katerina.janouchova1@tul.cz, monika.vysanska@tul.cz).

Adhesion properties. Pull-out tests are widely used to quantify properties of a fiber-matrix interface. Parameter usually used to this end is the nominal shear stress at debonding (1), τ_d. It is the ratio between the force F_{max} acting at a failure of adhesion and nominal embedded surface of the fiber.

$$\tau_d = \frac{F_{\max}}{2\pi RL} \qquad (1)$$

Additionally to debonding shear stress τ_d, spring constant k of NiTi-silicon-PES interface was computed. It corresponds to the model in which elastic response of the interface is considered to be similar to one-dimensional spring (see Fig. 2). In this case, NiTi filament is presumed to be rigid (due to elastic modulus, E, which is expected to be several orders of magnitude higher than elastic modulus of silicon-PES matrix, specifically NiTi has 10 GPa $< E <$ 100 GPa [16], and silicon-PES is estimated to be $1 < E < 10$ MPa [21]), thus mark glued to the filament serves as a fixed point from which displacement of the edge of NiTiSilTex block, Δl, is measured. Spring constant is then obtained via (2).

$$F = k\Delta l \qquad (2)$$

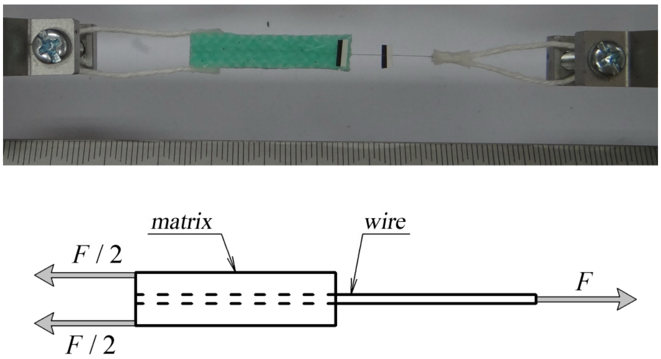

Figure 1. Top – The sample of NiTiSilTex composite with protruding NiTi filament mounted into the testing machine upon pull-out test (before testing). Two paper marks were glued on the specimen to measure a change in the distance between the edge of the composite block and (arbitrary) point on the NiTi fiber. It allows us (1) to measure pull-out of the fiber, and (2) considering the fiber as approximately rigid (acting force was not higher than 1N), to estimate spring constant of the fiber-matrix interface. The ruler in the photo has the scale in millimeters. Bottom – schematic view.

III. RESULTS

Total number of seven pull-out experiments was performed (three at 0.2 mm/s and four at 0.05 mm/s). They are summarized in Table 1 where the loading velocity, width and thickness of NiTiSilTex block, embedded length of the filament, debondnig force, spring constant, and nominal shear stress at debonding are presented.

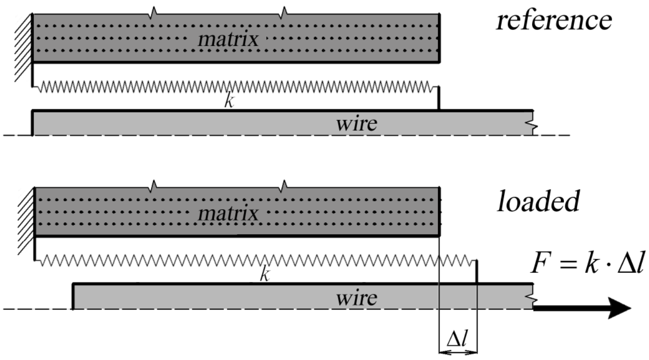

Figure 2. Schematic view of the spring-like behavior of fiber-matrix interface.

Mean debonding force $F_{max} = 0.673 \pm 0.239$ N was obtained at loading velocity 0.2 mm/s, and the velocity 0.05 mm/s gave $F_{max} = 0.832 \pm 0.119$ N (± standard deviation, SD). See Figure 3 for $F - \Delta l$ relationships. According to (1), F_{max} implies shear stress at debonding $\tau_d = 90.45 \pm 32.93$ kPa and 110.3 ± 18.63 kPa at 0.2 mm/s and 0.05 mm/s, respectively (Fig. 4). Spring constant of the interface was $k = 8.307 \pm 1.734$ N/mm at 0.2 mm/s, and $k = 9.065 \pm 2.153$ N/mm at actuator's velocity 0.05 mm/s (Fig. 5).

Figure 3 shows recorded force during the tests with respect to the change in the distance between marks on the sample. $F - \Delta l$ relationships were characterized with initial nonlinear (convex) increase of the force which was followed by the interval of proportional response characterized with k. Short concave interval preceded the maximum force in all cases. The maximum force (failure of the adhesion) was always followed by sudden decrease approximately to the same value of the force. Frictional response, sliding of the filament along the matrix, was observed after debonding of the fiber.

Table 1. Results and experimental conditions. W, T, L, k, F_d, and τ_d respectively denote width and thickness of NiTiSilTex block, embedded length, spring constant, debonding force, and nominal shear stress at debonding.

Sample	***loading velocity*** [mm/s]	***W*** [mm]	***T*** [mm]	***L*** [mm]	***k*** [N/mm]	F_d [N]	τ_d [kPa]
1	0.2	6.35	1.61	31.87	10.35	0.487	64.91
2	0.2	6.56	1.64	31.30	8.46	1.010	136.94
3	0.2	6.17	1.69	31.85	6.11	0.522	69.50
4	0.05	6.07	1.70	32.89	8.76	0.679	87.57
5	0.05	6.51	1.72	32.23	7.40	0.844	111.20
6	0.05	7.25	1.63	32.51	12.67	0.793	103.49
7	0.05	6.05	1.60	30.83	7.43	1.010	139.02
Mean ± SD	—	6.42 ±0.39	1.66 ±0.04	31.93 ±0.65	8.74 ±2.02	0.764 ±0.197	110.80 ±27.56

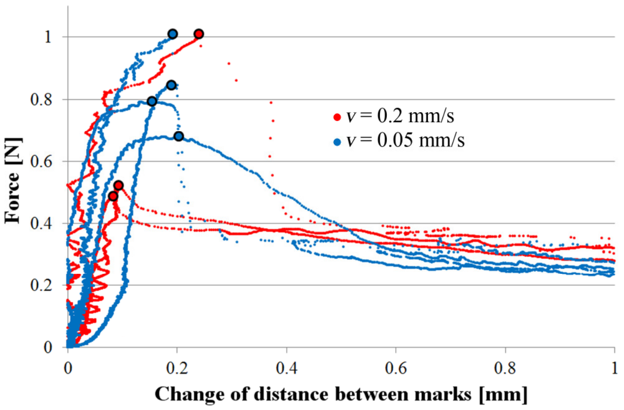

Figure 3. Force recorded during pull-out test. Recorded force showed steeply increasing response before adhesion failure, sudden decrease after the failure and followed by slowly decreasing response (corresponding to the friction between NiTi fiber and matrix) up to final disconnection between the wire and block. Different loading velocities did not show systematic deviation in debodning force.

IV. DISCUSSION

In this report, we briefly presented the results obtained in the pull-out test of the newly developed composite material NiTiSilTex which integrates nitinol SMA into silicon-PES matrix. A debonding, similarly to a delamination in laminates, is the main functional limit which one expects designing composite structure. Our attention was focused on the effect of pulling velocity. It could be of importance due to viscoelastic nature of the silicon elastomer which creates the main bulk material in the matrix.

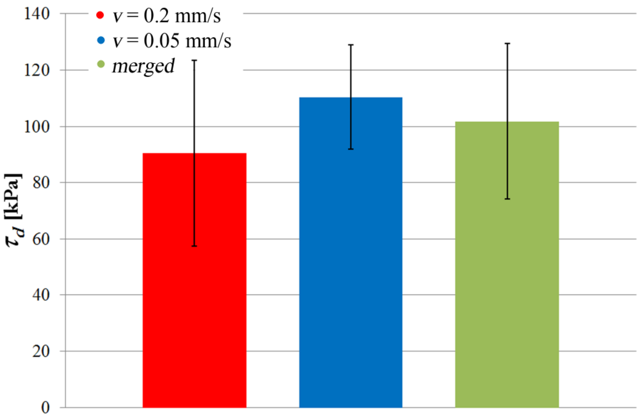

Figure 4. Nominal shear stress at debonding (mean ± SD). Although lower velocity gave higher mean shear strength, it was concluded that results suggest the failure of adhesion between the matrix and fiber does not differ significantly with respect to used velocity. It is due to rather large variability in 0.2 mm/s (±SD column in 0.2 mm/s covers mean value of 0.05 mm/s).

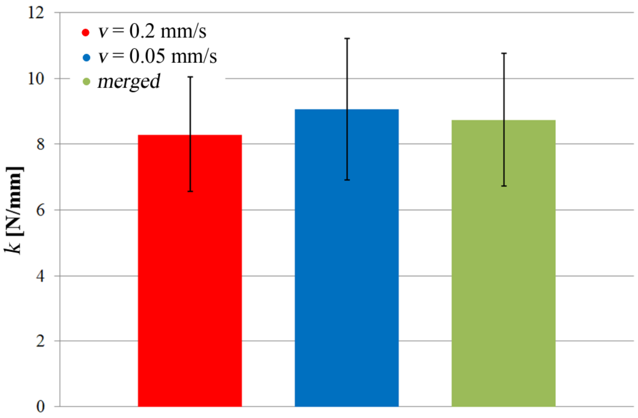

Figure 5. Spring constant of the interface (mean ± SD). *k* characterizes the behavior of interface prior to the failure. In this study, we did not found significant differences with respect to loading velocity.

This is the preliminary report involving rather small number of observations with respect to a pulling velocity. Nevertheless, despite this fact, the measurement revealed that intra-class variance may reach up to 35% of the mean value (SD/mean for F_d and τ_d in 0.2 mm/s). This dispersion covers the expectation based on mean value derived form experiments at 0.05 mm/s (Fig. 4). It suggests that rather large number of observations would be necessary to prove statistically significant differences between classes (pulling velocity 0.2 vs. 0.05 mm/s). In this situation we concluded that our data does not support the hypothesis that pulling velocities 0.2 mm/s and 0.05 mm/s could lead to differences in the debonding limit. The same conclusion is suggested by the data in case of spring constant k. It motivated us to present mean ± SD for F_d, τ_d, and k in Table 1, as well as in figures, also for merged groups.

In our study obtained shear stress at debonding, which can be considered as shear strength of the fiber-matrix interface, is somewhat smaller in comparison with results available in the literature. Several surface treatments were studied in [17]. The authors found mean shear strength approx. 700 kPa for straight-annealed NiTi filament in silicon matrix. They, however, also shown that the shear strength significantly depends on the used method of surface finishing (scarping, cleansing and washing led only to 250 kPa of the shear strength). The difference between our results (110 kPa) and [17] can be attributed to this fact.

Finally, it has to be noted that debonding shear stress presented in our experiments corresponds to nominal stress (averaged value presuming uniform stress distribution over embedded length of a filament). There are numerous studies showing that finite length of the fiber induces significant stress concentration at the entry of the fiber to the composite [22],[23]. It has to be born in mind when composite structures are designed. Based on [24], we can roughly estimate true maximum shear stress to be 2 – 4 times higher than nominal one.

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