Improved interfacial strength of SMA composite with nanotreated fibre surface

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Introduction

Shape memory alloy (SMA) composites have attracted considerable interest for their unique functions, such as shape and stiffness controls, damage detections and repairs [1-2]. However, these functions must be on the premise of the well bonded interface between SMA fibre and its surrounding matrix, which can ensure the sufficient strength of the interface to transfer the stresses and strains from SMA fibre to matrix material. It is well known that the interface between SMA fibre and matrix is also a potential source of cracks due to the relatively weak interface [3]. Therefore, interfacial adhesion between SMA fibre and matrix materials is an important issue to be investigated.

Over the last decade, various types of surface modification techniques have been developed aiming at improving the interfacial bonding of the SMA and its host materials [4,5]. In this study, three different surface treatment methods are employed to improve the bonding strength between SMA fibres and epoxy matrix, namely, sandpaper polish, anodization in HF acid and H_3PO_4 acid. The fibre pull-out test and the scanning electron microscopy (SEM) analysis are conducted to evaluate the interfacial properties of SMA composite.

Experimental

Materials

Pseudoelastic NiTi wires supplied by Shenzhen SuperLine Technology Co., Ltd., China, with diameter 1mm were used. The phase transformation temperature M_s, M_f, A_s and A_f of the wires were determined to be 7.08°C, 21.18°C, 12.32°C and 25.13°C, respectively. The liquid epoxy resin D.E.R.331 (Dow Chemical Co.) and the curing agent triethylene tetramine (Guangzhou Chemical Reagent Factory) were used to fabricate single SMA fibre pull-out samples.

Surface treatments

Mechanical polish and electrochemical anodization were carried out to modify the surface conditions of SMA wires. In order to remove the native surface oxide layer and increase the surface roughness of the SMA wires, 2000 CW sandpapers were used to polish the wires by hand. Before anodization, the polished SMA wires were immersed in deionized water and cleaned in an ultrasonic bath for 15 minutes. The anodization was carried out in a two-electrode system. The NiTi wire was used as working electrode and a platinum foil was used as the counter electrode. In the present study, the electrolytes of 0.075mol/L hydrofluoric acid and 0.5mol/L phosphoric acid were used respectively. The anodizing voltage or current was controlled by a program-controlled Agilent DC power source (N5751A). For the anodization in phosphoric acid, the constant voltage 20V was applied between working electrode and counter electrode. A magnetic stirrer was used to keep the electrolyte uniform throughout the whole anodization process. The anodization was carried out at room temperature for 30 minutes. For the anodization in phosphoric acid, constant current 1.5A was applied and the anodization was carried out in water bath to keep the electrolyte at about 25°C during the whole process.

Characterization methods

The morphologies of SMA wires with different treatment methods were observed with field emission scanning electron microscopy (FE_SEM). To estimate the effect of surface treatments on the interfacial strength of the SMA composite, single fibre pull-out test was carried out. The sample for pull-out test is a fibre-matrix cylinder with a diameter of 20mm and a length of 60mm, which was prepared by embedding a single pseudoelastic SMA wire into epoxy resin and cured (epoxy: curing agent = 100: 13) at 100°C for 2h. MTS test machine was used to carry out the fibre pull-out tests at room temperature and the test speed was 1mm/min.

Results and discussion

Surface morphologies of treated SMA wires

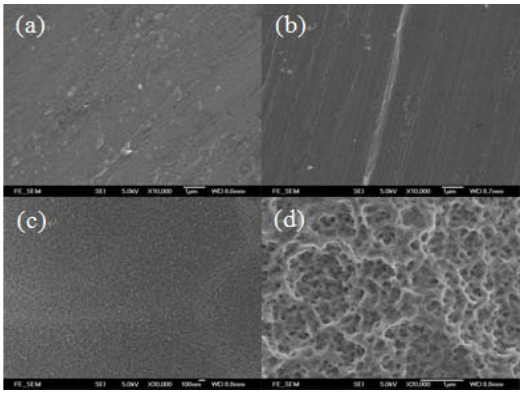

Fig.1 Morphologies of untreated (a), polished (b), anodized in H_3PO_4 (c) and HF (d) NiTi wires

Fig. 1 shows the SEM results of the SMA wires treated

with different methods. For comparison purpose, the morphology of untreated wire is presented in Fig. 1a. The conspicuous scratches can be found on the surface of polished SMA wire (as shown in Fig. 1b). For the wires anodized in hydrofluoric acid, nano-scale porous structure is obtained as shown in Fig. 1d, while it is relatively smooth for the wire anodized in phosphoric acid.

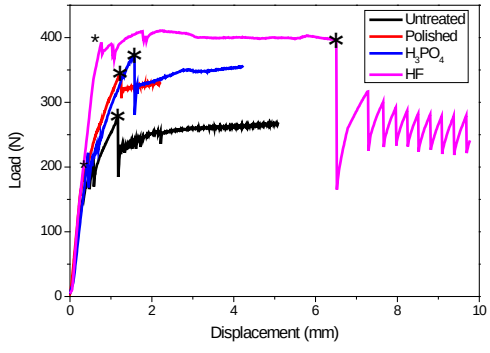

Fig. 2 Typical load vs. displacement curves of pullout test

Fibre pull-out test

The interfacial strength of SMA composite was quantified by single fibre pull-out test at room temperature. Fig. 2 shows the typical load-displacement curves of single SMA fibre composites. Initial debonding occurred at the points marked with '☆' and followed by the slope change of load-displacement curves. A sudden drop in the load can be found at the points marked with '*', which indicates that the interface between SMA fibre and epoxy matrix was debonded completely. The improvement of interfacial strength over the sample with embedded untreated wire can be observed. It is found that the load-displacement curve for the sample with the wire anodized in HF solution is quite different from others. A pretty long plateau state, which is the result of stress induced martensite phase transformation, appears and the interfacial strength is highest among the other surface treatment methods utilized in the present study.

Mechanism analysis

To analyze the debonding mechanism, the fracture surface of SMA wire pulled out from epoxy was microscopically observed by using SEM as shown in Fig. 3. It is found that the fracture surface of SMA wire anodized in HF is very different from the others, which is almost totally covered by epoxy, while very little epoxy was left on the wire surface treated by other methods. That is why the samples with embedded SMA wire anodized in HF have stronger interface. It demonstrates that the epoxy resin penetrated into the nano-porous structures and results in a large increase in contact surface area for mechanical anchoring between the SMA wire and epoxy matrix.

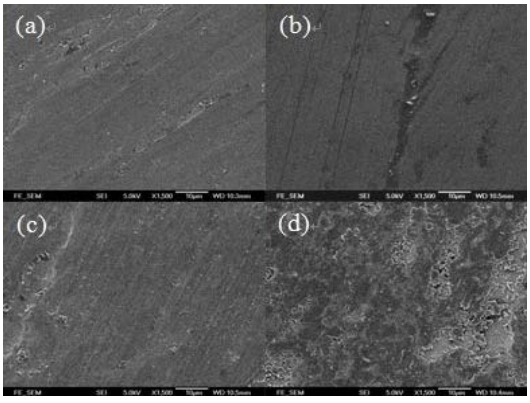

Fig. 3 The fracture surface of fibre pullout test: (a) untreated; (b) polished; (c) anodized in H_3PO_4; (d) anodized in HF

Conclusions

In the present paper, different surface treatement methods were employed to enhance the interfacial strength between the pseudoelastic NiTi fibres and epoxy matrix. The SEM and fibre pull-out test results show that the electrochemical anodizing in HF is the most effective method to improve the interfacial strength of SMA composites. The nano-porous structures formed on the surface of the SMA fibres provide extra contact surface area and strong locking mechanism.

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