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Enhancing the adhesive bonding strength of NiTi shape memory alloys by laser gas nitriding and selective etching

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Abstract

Laser gas nitriding process (LGN) was applied on NiTi shape memory alloy to obtain an alloyed surface consisting of TiN dendrites in NiTi matrix. By applying subsequent selective etching process, the matrix material in the alloyed layer can be selectively removed and a three-dimensional network of TiN dendrites is left on the surface protruding from the metal substrate. The 3D dendritic network provides extra surface area and locking mechanism for the adhesion joint. The microstructures of such textured surface were examined. The adhesion jointing characteristics of the surfaces were studied. A 150% increase in the lap-joint strength was achieved in the laser gas nitrided and etched specimen as compared with the sandblasted and etched ones.

Keywords: Laser gas nitriding; NiTi alloy; Shape memory alloy; Adhesive bonding; Textured surface

1. Introduction

The unique superelastic and shape memory characteristics of shape memory alloys (SMA) have drawn immense interests across a wide spectrum of industries. The properties of SMA are utilized and explored in products such as biomedical components, sensors, actuators, etc. The combination of SMA and other metals or polymers would enable special properties of a material system to be tailor-made [1–3]. Adhesion between SMA and other materials is therefore an important issue to be addressed for successful combination of SMA and other materials.

Most published work dealing with the interfacial adhesion of NiTi SMA and polymers are those concerned with NiTi wires and polymer matrix as in the form of reinforced composites. Paine et al. [4] used acid etching, hand sanding, polymer coating and sandblasting techniques to improve the adhesion of NiTi wires to epoxy matrix and concluded that sandblasting was the most effective method as the debonding stress has been increased by 70% as compared with the untreated wires. A similar study carried out by Jonnalagadda et al. [5] also recorded a 190% increase in debonding stress using sandblasted wires in the NiTi epoxy system. Smith et al. [6] applied silane-coupling agents to NiTi wires and achieved a 100% improvement in the adhesion strength between the NiTi wires and polymer matrix. Jang and Kishi [7] used various chemical etchants to treat the NiTi fibres and recorded a 3–18% improvement in the interfacial adhesive strength as compared with the untreated ones.

On the other hand, published work on the investigation of improvement of adhesive strength of large bonded area of SMA to other materials was not found. This paper will address a novel method to create a textured surface for the improvement of the adhesive strength of NiTi plates using epoxy resin as the adhesive.

Our previous work on surface engineering of NiTi by laser gas nitriding [8,9] have shown that it is possible to create a unique surface with hard TiN dendrites protruding from the NiTi metal matrix. This three dimensional dendritic network structure on the NiTi metal matrix allows a large increase in surface area for mechanical anchoring between the adhesive and adherend. This paper reports our investigation on the effect of dendritic height on the adhesive strength of the adhesive bond between two NiTi plates.

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2. Experimental procedures and methods

2.1. Materials

The material used for the experiment was hot-rolled Ti-50.8 at.% NiTi plate metal with a thickness of 3 mm. The specimens were cut into strips with the dimension of 23 mm × 6 mm × 3 mm. The specimens were than sand-blasted and cleaned in alcohol.

2.2. Laser gas nitriding (LGN)

A continuous wave 2 kW Nd–YAG laser was used to irradiate the specimen surface with power intensity of 500 W. The laser beam was defocused using a 100 ml focal length zinc selenide lens to a 2 mm spot size. High purity nitrogen gas (99.9%) was discharged onto the specimen molten metal pool through a nozzle. Nitrogen gas flow rate was 50 l/min. The beam scanning speed was kept as 5 mm/s. The individual tracks were overlapped with an overlapping interval of 0.5 mm to create a large laser gas nitrided (LGN) surface.

2.3. Etching

The specimens with LGN surface were then polished using 1 μm diamond paste to achieve mirror finish. About 20 μm was polished off from the surface. The specimens were then etched at 10% HF, 40% HNO_3 aqueous solution at room temperature from 5 to 40 min to remove loose phases such as impurities and the matrix phase. This process was expected to leave the TiN dendrites in a three-dimensional structure protruding from the metallic substrate. These samples are denoted as LGN-etched samples.

For comparison purpose, controlled specimens were prepared by sandblasting and etching in the same reagent for 10 min. This specimen is denoted as sandblasted-etched specimen in the text below.

2.4. Characterization methods

JEOL JSM-6335F field emission scanning electron microscope (FE-SEM) and Leica Stereo Scan 440 scanning electron microscope (SEM) were used for the microstructure and morphology observation.

The microhardness of the cross-section of LGN treatment specimen was measured with Shimadzu micro hardness tester with the load of 300 g.

The roughness of the sample surface was measured using Form Talysurf Series 2.

In order to estimate the effect of the textured surface on the adhesion strength, lap-joint tensile test specimens were prepared. Only the adhesive jointing surface was treated by the processes mentioned above. A two-part slow-cured epoxy resin (Araldite ''Standard'', by Ciba-Geigy) was used as the adhesive. The overlap jointing area was 6 mm × 7 mm and the adhesive bond thickness was controlled to be 100 μm. A compressive load was applied on each joint in order to ensure consistent adhesive thickness and pressure in the joint during the curing stage. The joined specimens were cured for 24 h at room temperature. The dimensions and joint assembly of the specimens are shown in Fig. 1. The joint strengths were determined by tensile tests using an Instron Machine Model 4301 at a cross-head speed of 1 mm/min and 22 °C. The experimental lap-joint strength σ was calculated as:

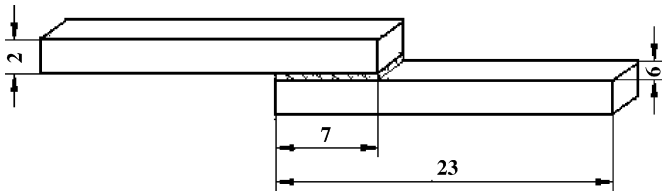

Fig. 1. Lap joint specimen for tensile test.

$$\sigma = F/wl$$

where F is the failure force, w is the joint width, and l is the joint length.

3. Results and discussion

3.1. Microstructure and hardness

Fig. 2 shows the cross-section microstructure of the LGN specimen. Laser surface nitriding of NiTi produce a close packed TiN layer at the outmost surface and a TiN dendrite/NiTi matrix composite layer beneath this. The total thickness of the nitrided layer is 400–500 μm. The distribution of TiN can be divided into three different zones, as shown in Fig. 2. Zone A is the outmost surface in which the TiN is close-packed, as shown in Fig. 3. Zone A is the hardest and is about 3 μm thick. Zone B is immediately beneath Zone A and has a thickness of about 10–20 μm. It is interested that Zone B is almost free from TiN. Zone C is the TiN dendrite/NiTi matrix composite region. Zone C is the region of interest in this work as some of the matrix is subsequently etched away chemically and the

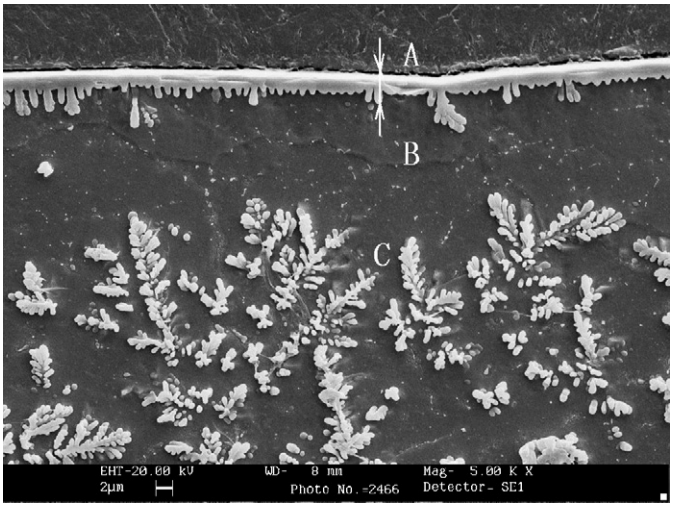

Fig. 2. Cross-section microstructure of LGN NiTi.