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Bimodal nanocrystallization of NiTi shape memory alloy by laser shock peening and post-deformation annealing

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Abstract

In this paper, surface nanocrystallization of NiTi intermetallic alloy by a novel method is reported. The NiTi alloy is processed by laser shock peening (LSP) and controlled annealing. The microstructure of the NiTi alloy after processing is characterized by transmission electron microscopy. At the top surface of the material, a nanostructure with bimodal grains is obtained. The mechanism of the formation of the bimodal microstructure is discussed. At the material subsurface, deformation twins are generated by LSP and retained after controlled annealing. Tensile test results showed that both strength and ductility are significantly improved through LSP and controlled annealing.

Keywords: Laser shock peening (LSP); Post deformation annealing; Shape memory alloys (SMAs); Amorphization; Nanocrystallization

1. Introduction

NiTi shape memory alloy (SMA) is one of the most widely utilized materials in medical devices due to its good biocompatibility, shape memory characteristics and extraordinary ability to accommodate large strains. NiTi SMAs are also used as actuators in microelectromechanical systems (MEMS) due to their high work output per unit volume [1]. However, the fatigue failure of NiTi components has been a major concern [2]. Various techniques have been proposed to improve the strength and fatigue performance of this material. For example, NiTi alloys have been processed by high-pressure torsion (HPT) [3–5], cold rolling [6,7] and shot peening [8]. However, most of these processing techniques are not suitable for very small or thin film samples, which are common in medical devices and microsystems.

Laser shock peening (LSP) has been widely used to improve component fatigue performance [9]. Like most surface processing techniques, LSP improves material strength by inducing grain refinement and work hardening in the surface of the component. As well as surface hardening, LSP also creates beneficial compressive residual stress on the component surface. In addition, LSP processing is surface specific, so the shape memory characteristic of the bulk materials can be preserved. Furthermore, the laser beam can be easily adjusted to a small size (microscale) and conveniently directed by optical mirrors, which makes LSP very suitable for treating components with complex structures both for medical applications and for MEMS devices. However, LSP of NiTi alloys has not been reported in the literature to date. Recently, surface nanocrystallization of aluminum alloys [10], titanium alloys [11] and steel [12,13] by LSP has been reported. It has been shown that LSP causes beneficial microstructural changes in the material surface and thus improves component fatigue performance. Thus, it is of great interest to investigate

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LSP of NiTi alloys and see how it affects the microstructure and strength of these SMAs.

In this study, a unique methodology to control the nanocrystallization of NiTi alloy is reported. The NiTi alloy was processed by LSP followed by post-deformation annealing (PDA) [14–16]. The crystallization temperature and volume fraction of the amorphous phase after the process were evaluated by differential scanning calorimetry (DSC). The microstructure before and after processing were characterized by transmission electron microscopy (TEM). The mechanisms of amorphization, nanocrystallization and the formation of bimodal nanocrystals will be discussed.

2. Experimental methods

A 0.3 mm thick 50.26 at.% Ni wrought NiTi alloy strip from Special Metals Corporation (WV, USA) was used in this study. The NiTi alloys were heat treated in a vacuum furnace at 800 °C for 30 min followed by water quenching, which results in a initial grain size of around 80 μm according to Prokofyev et al. [17]. Before LSP, the specimens were ground with 1200 grit sandpaper followed by final polishing with 50 nm colloidal silica. In the LSP experiment, aluminum thin foil (30 μm) was used as an ablative coating to protect the sample from thermal effects and BK-7 glass was used as confinement media. The pulsed laser was delivered by a Surelite III Q-switched Nd-YAG laser from Continuum Inc. (Santa Clara, CA, USA) with a wavelength of 1064 nm and a pulse width (full width at half maximum, FWHM) of 5 ns. A focusing lens was used to adjust the beam size to 1 mm. The laser intensity was 4 GW cm^{-2}. The overlap ratio was 75%, controlled by an x–y table.

The microhardness of the sample before and after LSP was measured by a Leco M-400-H microhardness test machine with a load of 200 g and a holding time of 10 s. An average of five measurements was used for each data point. DSC was carried out between 250 and 500 °C with a heating rate of 50 °C min^{-1} using a DSC 1 Star-e System from Mettler Toledo (Columbus, OH, USA) to determine the crystallization temperature and enthalpy of the amorphous NiTi after LSP. The DSC sample was prepared from the top surface (20 μm) of the laser-peened sample by polishing away the material on the non-treated side of the sample. Five DSC measurements were carried out for each condition. Having thus established the crystallization temperature, the sample was annealed at a specific temperature (360 °C) for 3 h to induce controlled nanocrystallization.

The TEM samples were prepared from the top surface and from 30 and 125 μm below the top surface of the peened sample by the focused ion beam (FIB) lift-out method in an FEI NovaLab 200 FIB system. Finally thinning was carried out at low voltage (10 kV) and low current (30 pA) to decrease the degree of damage. TEM was performed with an FEI Titan operated at 300 kV.

Tensile testing was carried out to evaluate the strength and ductility of the samples after LSP and LSP + annealing. The tensile test sample is dog-bone shaped. The cross-section of the gauge area is 6 mm × 0.25 mm. There are approximately 230 grains across the gauge sections in the sample before LSP, which is large enough for a bulk material response. Both sides of the gauge area were processed by LSP. The tensile test was carried out at room temperature with a strain rate of 10^{-4} s^{-1}. For each condition, an average of five measurements was reported.

3. Results and discussion

3.1. Top surface amorphization by LSP

After LSP, dense dislocation tangles were observed as in Fig. 1. Fig. 1A shows the microstructure viewed from the [0 0 1] direction and Fig. 1B shows the microstructure viewed from the [0 2 3] direction. From both Fig. 1A and B, a non-uniform dislocation arrangement can be observed. Fig. 1C and D present the bright-field and dark-field images obtained by tilting to create a two-beam condition. Fig. 1C and D have complementary contrast: the regions of dark contrast in Fig. 1C and the regions of white contrast in Fig. 1D are associated with dislocations generated by LSP.

The hardness of the sample increases from 225.6 ± 5.94 kg mm^{-2} before LSP to 333.2 ± 7.85 kg mm^{-2} after LSP, i.e. a 47.7% increase. This is caused by work hardening, i.e. dense dislocations generated by LSP leads to an increase in hardness. This is consistent with the LSP work on aluminum alloy [10] and steel [12], i.e. after LSP, high densities of dislocations were generated due to high-strain-rate plastic deformation.

3.1.1. Amorphization at the top surface by LSP

In the sample after LSP, halo rings can be observed in the diffraction pattern of Fig. 1A and B. According to Williams and Carter [18], these rings could be caused by either very fine grains or amorphous phases. To clarify this, high-resolution TEM work was carried out as shown in Fig. 2. Fig. 2B shows a representative TEM image taken from region B of Fig. 2A. It can be observed that the microstructure is fully amorphous in Fig. 2B, which can also be confirmed by the halo rings in the corresponding fast-Fourier transform (FFT) pattern [19,20]. Fig. 2C shows a representative TEM image of region C of Fig. 2A. Unlike Fig. 2B, the microstructure in Fig. 2C is composed of both crystals and amorphous phase. This is also confirmed by the corresponding FFT pattern in Fig. 2C, in which both the halo ring and diffraction spots are present.

3.1.2. Volume fraction of the amorphous phase

The overall degree of amorphization can be estimated by comparing the crystallization enthalpy of a partially amorphized material to that of a completely amorphous alloy with the same composition [21]. A representative DSC curve of the NiTi alloy (top 20 μm) after LSP is shown in Fig. 3. It should be noted that the DSC curve