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Laser Surface Processing of Hot Rolled Ni-45.0at.%Ti Shape Memory Alloy

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Both oxide removal and low surface roughness was achieved at hot rolled Ni-45.0at.%Ti shape memory alloy (SMA) surface using a nanosecond pulsed Nd:YAG laser. The oxidized and polished surfaces were analyzed by White-Light Interference (WLI), Focus ion beam (FIB) and energy dispersive spectrometer (EDS). Special attention was paid to investigate phase transformation evolution of SMA before and after laser irradiation using Differential scanning calorimeter (DSC) and X-ray diffraction (XRD). Results revealed that the as-received oxides with thickness of 10 micron had been removed completely after laser treatment, and surface roughness of laser-treated surface was less than 1 μ m. Moreover, no obvious change occurred for phase analysis and transformation temperatures of the SMA, indicating that the influence of laser irradiation on shape memory effect of hot rolled Ni-45.0at.%Ti SMA was negligible.

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Keywords: shape memory alloy, one-stop laser processing, oxide removal, surface roughness, transformation temperature, phase

1. Introduction

Ni-Ti shape memory alloy (SMA) have a wide range of applications due to shape memory effect (SME) and superplastic property, such as orthodontics, actuators and nanotweezer [1-3]. Hot working processes including forging and rolling have been recognized as crucial forming techniques for SMAs [4-5]. As an active element, Ti is always oxidized during hot working processes [6], and the oxides layer can be easily found at the initial surface of SMAs after hot treatment, which usually affect the SME of SMA. Therefore, it is required to remove oxides layer of the SMA for further applications. At the same time, surface roughness of the SMA should be maintained at low level to keep mechanical properties of workpieces or fit further processing.

Laser surface processing has been considered as a promising method to enhance surface properties of materials without altering or affecting bulk properties in numerous applications [7-8]. For example, laser cleaning has been developed to replace mechanical polishing and aggressive chemicals due to serious drawbacks of being expensive, poor quality control or a source of environmental pollution caused by these conventional cleaning methods. Particularly for metallic objects in industrial settings, laser is considered as an attractive tool for removal of particulate contaminants, oxide layers, oil, grease, etc. from the surface, as they are highly controllable and can be selectively applied [9-10]. Moreover, in recent years, laser polishing had been demonstrated to be an effective polishing method for metal materials such as iron, steel, and titanium alloys. Compared with manual mechanical polishing, laser polishing have advances in automation and surface integrity. Laser polishing is mainly based on melting caused by laser

irradiation. When molten pool formed, flowing material tend to redistribute to a same horizontal level due to surface tension and gravity, then after quick solidification of molten pools, surface roughness would be reduced [11-13]. Laser polishing could be achieved by pulsed laser, continuous wave (CW) laser [14-16]. CW laser melt materials continuously, while pulsed laser melts material surface during each pulse and forms ripple structure. By adjustment of laser beam overlapping, pulsed laser could also produce a relatively smooth surface. Compared with CW laser, pulsed laser heat materials discretely and cause less heat accumulation. However, recent researches of laser polishing have not investigated about SMAs, which have some particular characteristics. Due to heat sensibility of SMAs, pulsed laser is more suitable for polishing this kind of material.

In this paper, we carry out a one-stop method to remove oxides layer of Ni-Ti SMA surface and achieve low surface roughness by nanosecond pulse laser. To study the effect of laser treatment on properties of SMA, by using SEM, EDS, XRD and FIB, we discuss how laser treatment affects surface phase compositions and of Ni-Ti SMA, DSC is used to investigate transformation temperatures. The aim of this study is to understand the effects of laser processing for Ni-Ti SMA, and indicate how it applies for potential applications.

2. Experimental Procedures

2.1 Materials

Commercial Ni-Ti plate with a nominal composition of 45.0 Ti and 55.0 Ni with 0.6 mm thick was chosen, the alloy plate was hot rolled and without aging treatment. Specimens

were cut to size of 10mm × 10mm and 4mm × 5mm for different measurements.

2.2 Laser processing

A nanosecond pulsed Nd:YAG laser (wavelength 1064 nm, pulse duration 34 ns, repetition rate 30 000 Hz, spot size 50μm) was used in this study. The optimized average power density was 6.01×10^4 W/cm², and the optimized scanning speed was 500 mm/s with 67% overlapping rate on scan direction. The irradiated area was as big as specimens using hatched scanning mode with 80% overlapping between scan lines in the program. Laser beam scanned three times to remove oxide layer and another two times for surface polishing. When laser was turned on, the specimen was placed under Ar gas protection.

2.3 Surface Characterization

Morphology and was observed by Carl Zeiss EVO MA15 scanning electron microscopy (SEM) with energy dispersive spectrometer (EDS) and KLA-Tencor MicroXAM-100 3D Surface Profilometer.

Cross section views of local area were prepared by FEI Helios 650 with focused ion beam (FIB) technology. The main parameters for ion cutting were chosen as 30 kV, 47 nA-0.79 nA, and 1.5×10^{-4} Pa pressure. Average dimension of each hole was (100-200μm) × (40-80μm) × (20-30μm).

Surface crystal structure of the Ti-Ni alloys was examined by Bruker D8 ADVANCE X-ray diffractometer (XRD) with Cu K α radiation ($\lambda = 0.15418$ nm) filtered by nickel. A part of specimens are manual polished to substrate for measuring original state phase.

2.4 Transformation temperatures measurement

Transformation temperatures of Ni-Ti SMA was measured by NETZSCH DSC 204F1 Phoenix differential scanning calorimeter. Size of specimens for DSC measurement was 4mm × 5mm.

3. Experimental results and discussions

3.1 Effect of laser treatment on oxides removal

Different from previous work, separate beam scanning during laser treatment was chosen to examine local effect of laser on oxides removal. After laser treatment, surface chemistry and cross-section view of laser-treated area at the SMA surface was examined carefully, as shown in Fig. 1 and Fig. 2. Fig. 1 shows a photo of laser treated region at the as-received SMA surface.

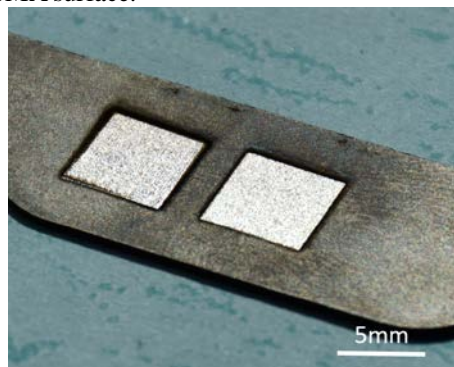


Fig. 1 Laser-treated region at as-received Ni-Ti SMA surface.

It can be found that oxygen content was dropped significantly at the irradiated area on basis of EDS element mapping data in Fig. 2. Oxygen content was more than 40% at untreated surface, and reduced to less than 3% at treated surface, as shown in Table 1. Results of EDS indicates that laser treatment has effectively removed the original oxide layer from as-received Ni-Ti surface.

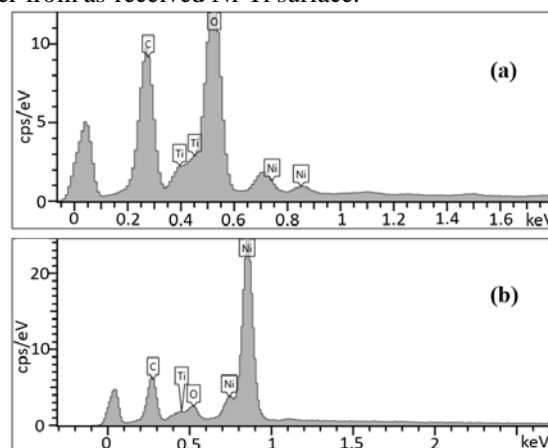


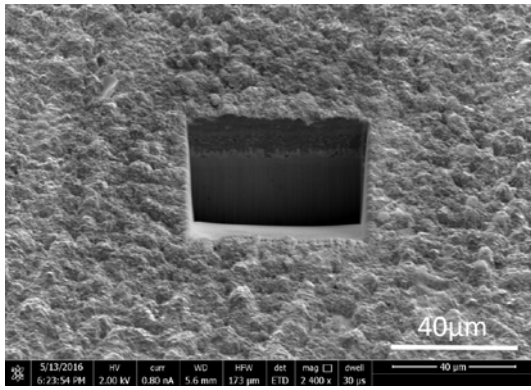
Fig. 2 EDS analysis of Ni-Ti SMA. (a) As-received surface (b) and laser-treated surface.

Table 1 Element contents of surfaces before and after laser treatment

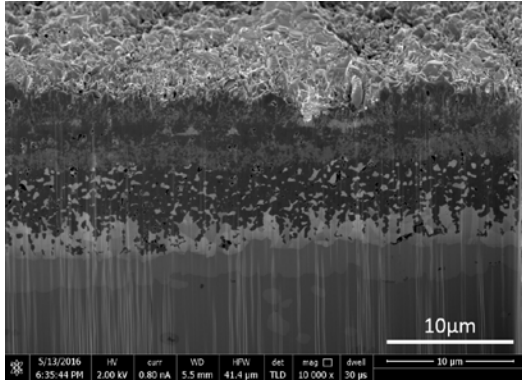
Concentration(wt %)	Ti	Ni	O	Possible composition
Original	53.28	3.78	42.95	TiO ₂
Laser treated	18.06	79.10	2.85	NiTi metal compound, Ni

Continuous beam scanning for practical laser treatment was further performed, the top surface and cross section view of both as-received surface and laser-treated area are shown in Fig.3. Fig. 3(c) and (d) show that no oxides or contaminants layer was observed at laser-treated area, while melting and solidification occurred at the top surface. Fig. 2(b) suggests that the thickness of original oxides and contaminants layer at as-received surface was nearly 10 micron.

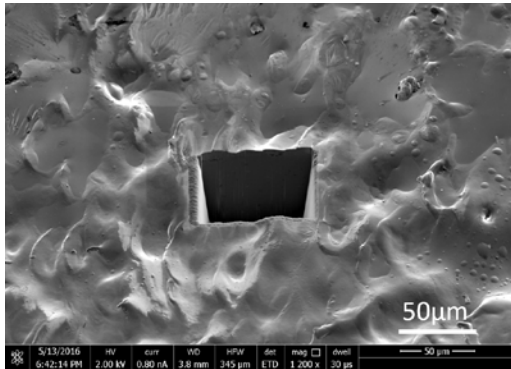
Fig. 4 shows XRD results of as-received and laser-treated Ni-Ti SMA alloy. In theory, original phase compositions of Ni-45.0at.%Ti SMA in low temperature are NiTi (B2) and Ni₃Ti. Compared with the phases of substrate, phases of surface have increased in Ni₃Ti and have a new phase of Ni. According to oxidation behavior theory of Ni-Ti SMA, oxidized surface of Ni-Ti has a multilayered structure, because of deposition of Ni during the oxidation process of surface NiTi phase [6]. There are 4 layers from surface to substrate, the first layer is probably pure TiO₂, the second layer may be composed of TiO₂, Ni and a little Ti, the third layer could be composed of Ni and Ni₃Ti, and the substrate is the same as as-received material, as shown in Fig. 5. Considering phase compositions after laser treatment, we speculate that laser treatment removed the first two layers and the remained surface layer is the third layer which contain more pure-Ni phase. Meanwhile, it means laser treatment had not change phases of the remelting third layer.



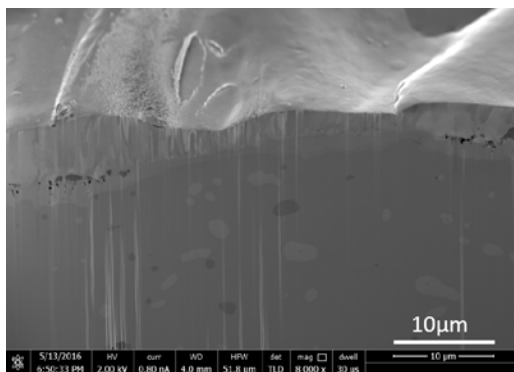
(a)



(b)



(c)



(d)

Fig. 3 Cross section of Ni-Ti SMA surface cut by FIB (a)(b) As-received surface (c)(d) laser-treated surface.

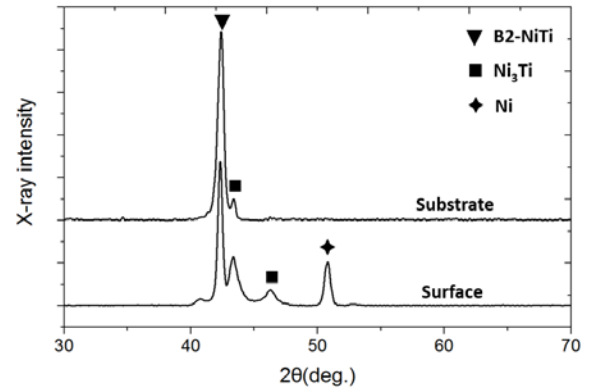


Fig. 4 XRD patterns of Ni-Ti SMA

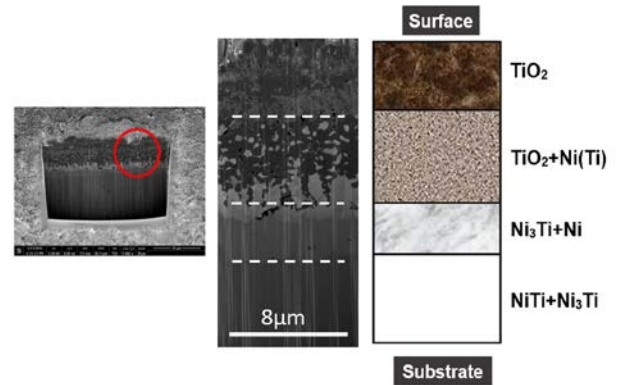


Fig. 5 Multi-layer structure of as-received oxidized surface

3.2 Effect of laser treatment on surface morphology

In this work, to reach a balance between oxides removal efficiency and keeping surface roughness, laser scanning model was hatched to be 80% overlapping and multiple scanning. As shown in Fig. 6, melting tracks are observed on surface of Ni-Ti SMA. Moreover, white-light interferometer was employed to measure surface topography and surface roughness of laser treated Ni-Ti SMA. Fig. 7 shows that after laser treatment, on most of the surface, peak-to-valley height of ripple structure was about 4-5 μ m, and average roughness of the laser treated surface was measured as 0.9 μ m.

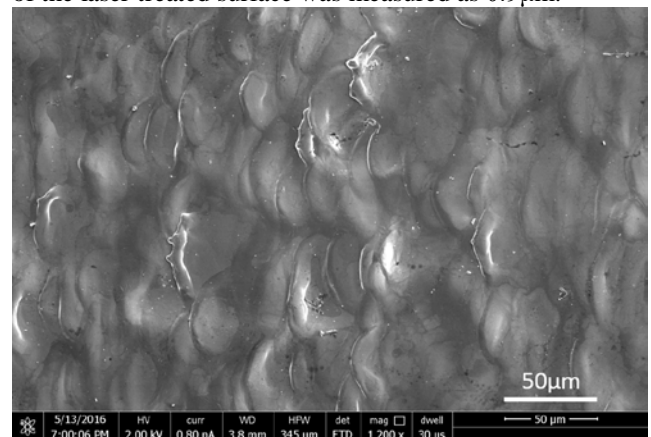


Fig. 6 Surface of Ni-Ti SMA after laser treatment.

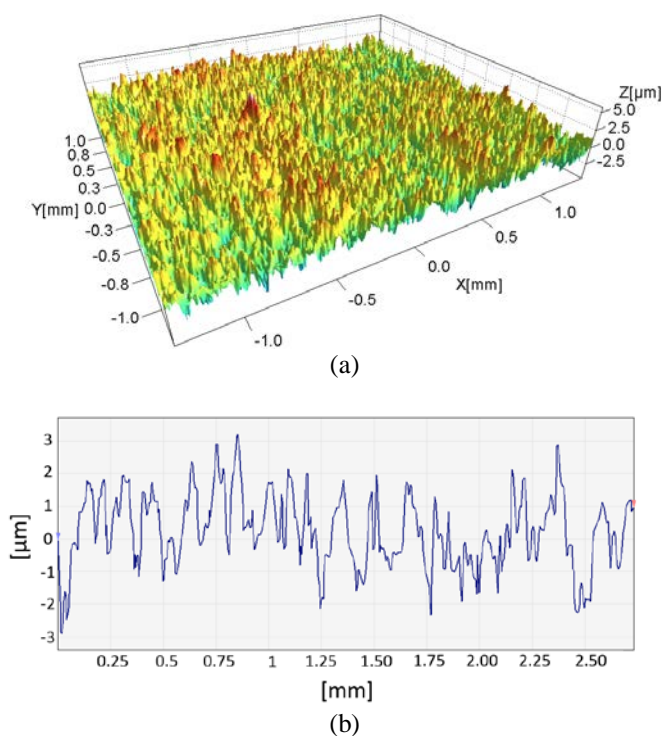


Fig. 7 (a) 3D surface profiles and (b) section profile of Ni-Ti SMA.

3.3 Effect of laser treatment on transformation temperatures of Ni-Ti SMA

In order to investigate the influence of laser treatment on transformation temperatures of Ni-Ti SMA, as-received and laser treated Ni-Ti SMA sheets were aged at 450°C for 30min and then measured transformation temperatures of the aged Ni-Ti sheets. DSC results obtained as a function of temperature for as-received and laser treated Ni-Ti SMA is shown in Fig. 8. For both cooling and heating circles, only slight shifts of the transformation temperatures are observed, and the differences are less than 5°C. Therefore, it can be deduced that laser treatment of SMA surface have not changed transformation temperatures of the Ni-Ti SMA, and main phase of Ni-Ti SMA is also not changed after laser processing.

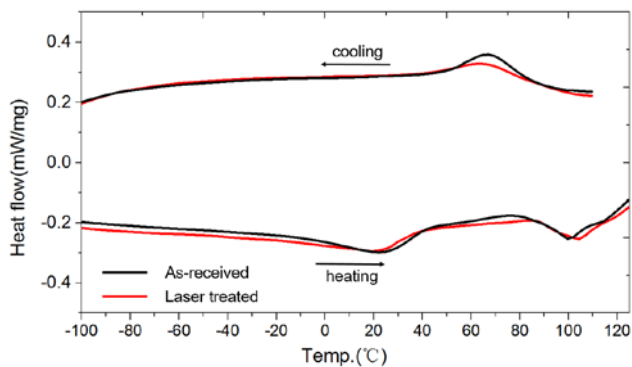


Fig. 8 DSC graph of Ni-Ti SMA before and after laser processing.

4. Conclusion

Analyses of Ni-45.0at.%Ti shape memory alloy before and after laser processing was carefully examined. Thickness of as-received oxides has observed to be 10 μ m. Oxide layer was completely removed and oxygen content was decreased significantly after laser treatment. Moreover, surface roughness of laser treated surface was measured as less than 1 μ m. Phase compositions of the SMA sheet had no obvious change after laser treatment. In addition, there was no significant change on transformation temperatures of Ni-Ti SMA sheets.

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