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Application of femtosecond laser shock peening in Nitrogen gas for improvement of corrosion resistance of NiTi alloy in Hank's solution

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Abstract

Femtosecond laser shock peening(FsLSP) in Nitrogen gas was performed on the NiTi shape memory ally without protective coating. In this article X-ray diffraction (XRD), Energy Dispersive X-Ray (EDX), Scanning Electron Microscope (SEM), three dimensional surface morphologies, wettability and corrosion properties were tested. The XRD results showed that TiN and Ni_4Ti_3 coating can be found on the surface after femtosecond laser shock peening and the sample Bragg diffraction peak became broader than that of the sample without FsLSP treatment, which also showed that the surface residual stress could be generated by FsLSP. The wettability measurements showed that the FsLSP decreased the contact angle of Hank's solution. The electrochemical corrosion property and immersion corrosion behaviour were tested in Hank's solution, which was prepared in our laboratory. The corrosion results demonstrated that the corrosion inhibitive properties of NiTi sample could be improved with FsLSP treatment.

Keywords: Femtosecond laser shock peening, NiTi shape memory alloy, wettability, corrosion inhibitive properties ;

1. Introduction

Laser shock peening (LSP) is a new surface treatment technology, which have more advantages compared with other surface techniques(Gao, 2011). LSP can generate residual compressive stress and induce harden-layer in the surface of target metal. Normally the LSP is operated with nanosecond laser system and the target is coated by protective layer such as black paint and Al foil, which can be vaporized into plasma with high temperature and pressure(Wang et al., 2017). When the laser beam irradiated on the surface of target it can create a high-pressure shock wave, which can induce plastic deformation in the surface layer of metal.

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Lots of researches have been done and show that laser shock peening with nanosecond laser system can improve the mechanical properties such as fatigue life, wear resistance and corrosion resistance(Zhang et al., 2010)(Zhang et al., 2010). However, few investigations of laser shock peening with femtosecond laser system were studied. The femtosecond laser shock peening (FsLSP) can induce higher shock wave pressure of up to 100-1,000 GPa than that by nanosecond laser which is only 1-10 GPa(Hoppius et al., 2018). And FsLSP without protective layer in air condition produces smaller heat-affected and melted zones due to its ultrashot pulse duration.

In our previous studies, we found that the FsLSP treatment can enhance the wear property of NiTi shape memory alloy and 0.4% C steel(Wang et al., 2019)(Majumdar, Gurevich, Kumari, & Ostendorf, 2016). The purpose of the research is to study the effect of FsLSP with protective layer in Nitrogen gas on the corrosion resistance of NiTi biological shape memory alloy in simulated body liquid.

2. Experiment and method

The NiTi shape memory alloy sample of 15 mm × 15mm × 1 mm with chemical composition of 50.80%Ni-49.20%Ti from Memory-Metalle GmbH, Germany, was used during this experiment and the samples were not polished. The samples were not coated with any protective layer during laser processing. All the samples were cleaned by distilled water and ethanol before and after FsLSP treatment. Fig. 1 shows the setup and schematic diagram of femtosecond laser shock peening (FsLSP) in Nitrogen gas without confinement medium and protective layer. The femtosecond laser shock peening was performed with a femtosecond pulse laser system (Spitfire Ace produced by Spectra-Physics). The wavelength and pulse width of the laser are 800 nm and 35 fs respectively. The FsLSP treatment used the following parameters: laser pulse energy of 800 µJ; diameter of laser-spot of 45 µm; laser scanning speed of 45 mm/s. The flow speed of the nitrogen gas is 10 L/min.

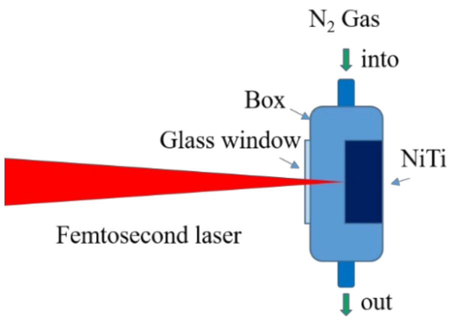

Fig. 1. The schematic diagram of the femtosecond laser shock peening in Nitrogen gas.

The surface morphology of the before and after FsLSP treatment NiTi shape memory alloy was observed by Nikon Eclipse LV100 microscopy and scanning electron microscopy (Zeiss EVO MA 15). A contact angle tester was used to measure the surface hydrophobicity of the NiTi alloy before and after FsLSP treatment at room temperature. The surface roughnesses of samples were tested with laser scanning microscopes (Polytec TMS 1200 and VK-X100). The change of phases on the surface of treated and untreated NiTi alloy was identified with the X-ray diffraction (XRD). In order to compare the difference of the NiTi alloy with and without FsLSP treatment, the electrochemical corrosion testing and immersion corrosion testing were carried out in Hank's solution.

3. Experimental results

Fig. 2 shows the surface optical and SEM images of NiTi alloy after FsLSP treatment in Nitrogen gas. From the optical-image it can be found that the light yellow layer appeared on the surface of NiTi alloy. The SEM image showed that there were no cracks and periodic surface structures (LIPPS) on the surface. The laser energy was absorbed, which can induce laser ablation and shock wave with high pressure (up to 100-1000 GPa), when the femtosecond laser beam irradiated on the NiTi shape memory alloy directly. The golden colour layer appeared on the surface of NiTi after FsLSP treatment in Nitrogen gas as shown in Fig. 2, which is the formation of TiN. From the X-ray diffraction patterns (XRD) testing it can also be found that TiN peaks were observed as shown in Fig. 3. What's more, the full width at half maximum of B2 phase becomes wider, which suggests that the FsLSP treatment induced residual stress on the surface of NiTi alloy.

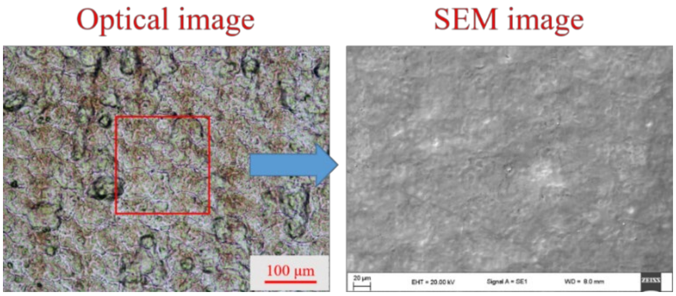

Fig. 2. The surface morphologies of NiTi shape memory alloy after FsLSP treatment in N_2.

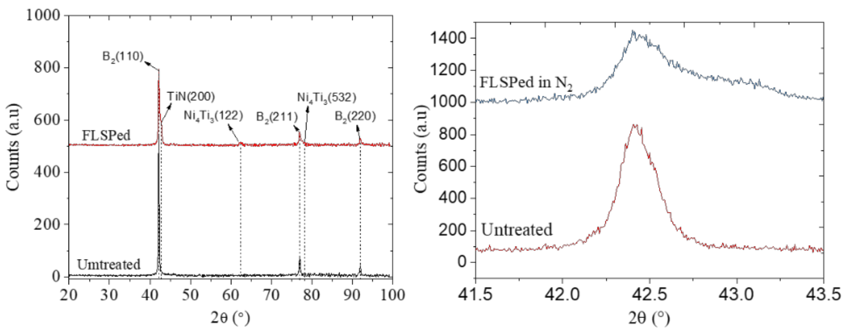

Fig. 3. The XRD patterns of NiTi shape memory alloy with and without FsLSP treatment.

From the Fig. 4 and Table 1, it can be found that the roughness Sa and Sp of untreated NiTi alloy decreased from 510 nm and 710 nm to 480 nm and 610 nm of FsLSPed NiTi alloy respectively. The roughness did not change very obviously after FsLSP treatment. The contact angle also decreased from 78˚ to 71˚ after the same treatment, which means that the NiTi alloy becomes more hydrophilic after FsLSP treatment. The laser ablation is the main reason for the decreased roughness and more hydrophilic, which can generate thin TiN layer on the surface of NiTi alloy. Ng et al. also found the similar results when they processed the NiTi with 100 W CW fiber laser(Ng, Chan, Man, Waugh, & Lawrence, 2017)(Ng et al., 2017). During their study, they also found that the laser treatment in nitrogen gas may be an effective technology to improve the tribological property of NiTi and enhance its the hydrophilicity and biological performance.

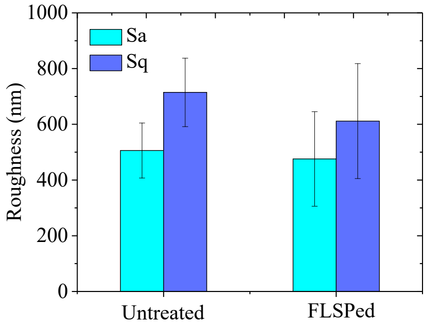

Fig. 4. The roughness on the surface of NiTi alloy before and after FsLSP treatment.

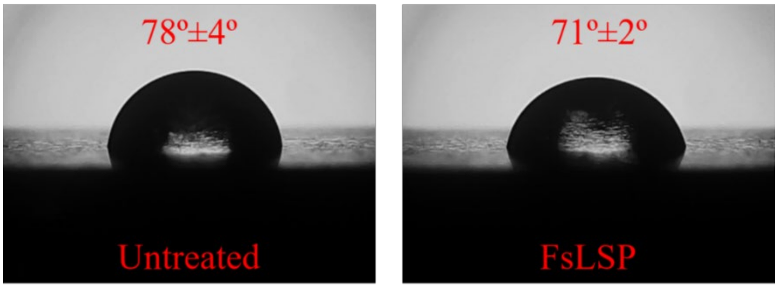

Fig. 5. The images of contact angle before and after FsLSP treatment.

Fig. 6 shows the electrochemical polarization curve of NiTi alloy in Hank's solution and Table 1 gives the corrosion current density and corrosion potential of NiTi alloy with and without FsLSP treatment. The corrosion potential of NiTi alloy decreased from -263±27 mV to -346±15 mV and the corrosion current density becomes lower from 0.99±0.96 A/cm^2 to 0.02±0.005 A/cm^2 when the sample was treated by FsLSP in nitrogen gas. The lower corrosion current density means normally better corrosion resistance, so the FsLSP treatment is an effective method to reduce the corrosion current density, which is due to the surface TiN layer and residual stress induced by the shock wave with high pressure. Fig. 7 shows the surface SEM picture of samples after electrochemical corrosion testing in Hank's solution, from which the corrosion pits can be found on the surface of untreated sample. But the surface of FsLSPed sample was smoother and had less corrosion pits.

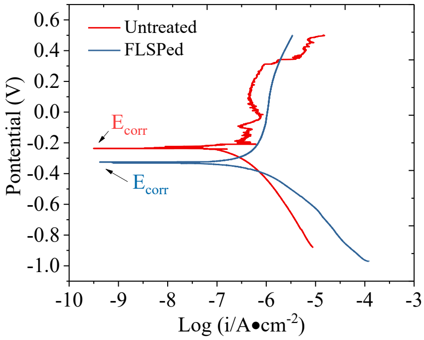

Fig. 6. Electrochemical polarization curve of NiTi alloy in Hank's solution

Table 1. Surface characteristics and corrosion results.

	Untreated NiTi alloy	FsLSPed NiTi alloy
Roughness Sa (nm)	510±100	480±170
Roughness Sq (nm)	710±120	610±210
Contact angle (°)	78±4	71±2
Corrosion potential (mV)	-263±27	-346±15
Corrosion current density (A/cm^2)	0.99±0.96	0.02±0.005

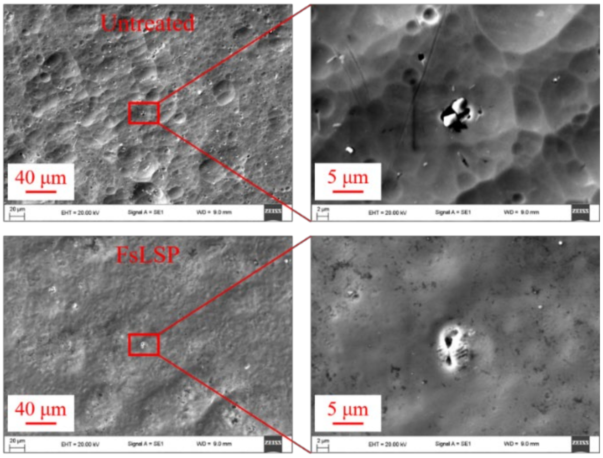

Fig. 7. The SEM images on the surface of NiTi alloy after electrochemical corrosion testing in Hank's solution.

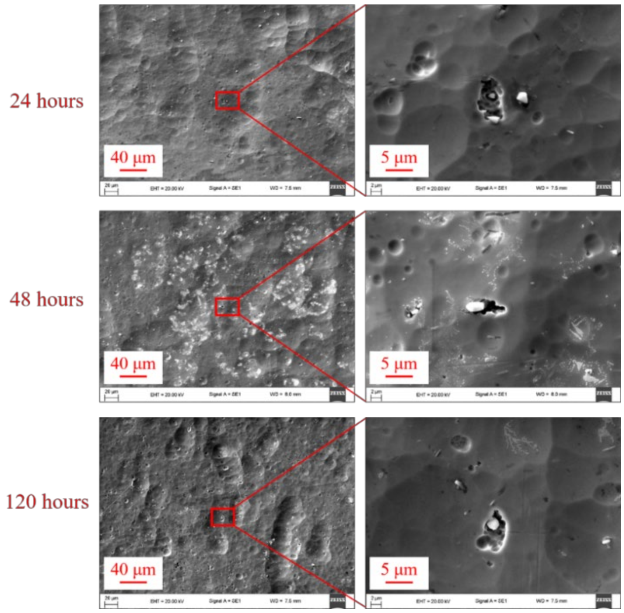

Fig. 8. The SEM images of untreated NiTi alloy after immersion corrosion testing for different time in Hank's solution.

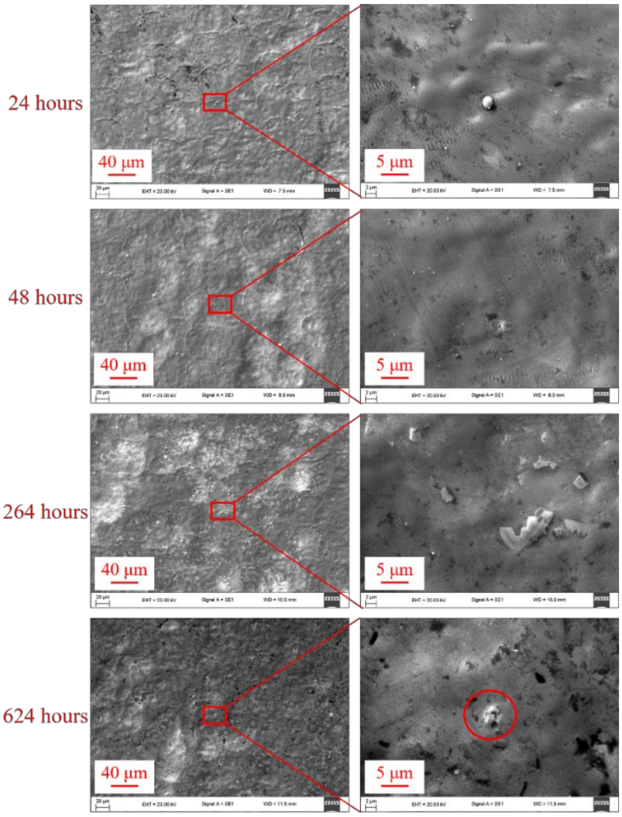

Fig. 9. The SEM images of FsLSPed NiTi alloy after immersion corrosion testing for different time in Hank's solution.

The Fig. 8 and Fig. 9 give the surface SEM images of untreated and FsLSPed after immersion corrosion testing for different time in Hank's solution respectively. According to the present immersion corrosion results, we can guess the immersion corrosion mechanism and the schematic diagram. The NiTi alloy will be vulnerable to attack by Cl^- and other ions when NiTi the sample is putted in the Hank's solution. And the small corrosion pits would take place on the surface and the ions attack can destroy the Ni-Ti bond, which can induce that broken particles fall down from the corrosion pits. It could be clearly seen from the Fig. 8 that corrosion pits appeared on the surface of untreated NiTi sample after immersion corrosion testing in Hank's solution for about 24 hours. Fig. 9 shows the surface images of FsLSPed NiTi sample after immersion testing for 24 hours, 48 hours, 26 hours and 624 hours, from which no corrosion pits and corrosion cracks can be found. The surface corrosion pits are very dangerous when the NiTi alloy is used in body as implant biomaterial, which can induce fatigue cracks very easily under the action of stress. So, it is very important to prevent the appearance of corrosion pits and cracks on the surface of NiTi alloy. The present study found that the FsLSP treatment in Nitrogen gas may be an effective method to improve the corrosion resistance of NiTi alloy, which may be due to the surface residual stress and TiN layer induced by FsLSP.

4. Conclusions

Femtosecond laser shock peening (FsLSP) treatment was used to improve the corrosion resistance of NiTi shape memory alloy, which was performed without any protective layer and absorbing coating in Nitrogen gas. The TiN and Ni_4Ti_3 coating can be found on the surface of FsLSPed NiTi sample. The contact angle of Hank's solution on the surface of NiTi decreased after FsLSP treatment. And the results of electrochemical corrosion and immersion corrosion show that the FsLSP treatment in Nitrogen gas can enhance the corrosion of NiTi alloy because of the surface residual stress and TiN and Ni_4Ti_3 coating.

Acknowledgements

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