

THERMOMECHANICAL STUDY OF LASER HEAT TREATED NiTi DENTAL ARCH WIRE

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ABSTRACTS

A preliminary study has been conducted to demonstrate the effect of laser heat treatment on NiTi alloy dental arch wires (0.016" x 0.022" and 0.018" x 0.026", rectangular shape).

Changes in mechanical and thermal properties and surface morphologies are investigated by using optical and scanning electron microscope (SEM), energy dispersive x-ray microprobe analysis (EDX), differential scanning calorimeter (DSC), and micro hardness tester.

The results indicate that the laser can affect the thermal equilibrium state of the localized surface. Titanium rich surface film is formed by the laser treatment. The surface film and rapidly resolidified underlying structures show better chemical resistance than the matrix material. Phase transition temperatures which are related to shape recovery temperatures are changed after laser treatment. Hardness of resolidified area and heat affected zone are lower than before treatment.

INTRODUCTION

The equiatomic nickel titanium (NiTi) alloy is one of the shape memory alloys which has an excellent spring back or super elastic properties, high degree of corrosion resistance, wear resistance and high damping properties(1). Some possible application of NiTi alloys are connectors, temperature control devices, orthodontic dental archwires, intracranial aneurism clips, a vena cava filter, contractile artificial muscle for an artificial heart, orthopedic implants and other medical devices

SME may occur in an alloy which can be changed in shape easily at below transition temperature, but it can be returned to its original configuration when it is heated to above the transition temperature. Transition temperature range(TTR) of NiTi alloy is directly related to the SME.

TTR can be controlled by changing chemical compositions and changing deformation or heat treatment histories.

Orthodontic dental archwire made of NiTi alloys has an excellent spring back properties but it does not possess SME and super elasticity(2), because it has been manufactured by a work hardening process(3,4). Therefore, the improvement of the super elasticity of NiTi alloy becomes a new contention among investigators.

In this study surface heat treatment by laser is investigated on the surface of NiTi dental arch wire for improving SME while keeping good mechanical properties and enhancing the chemical resistance.

MATERIALS AND METHODS

Commercially available rectangular shape cross section NiTi dental arch wires (0.018" x 0.026" Unitek Corp. CA and 0.016" x 0.022" Flexmedics Corp. MN) were used for this study.

Samples were cut into about 80 mm long. They were placed on a sample holding device and focused laser beam was directed on the top surface of specimen blowing argon or nitrogen gas while the device was moving at a predetermined constant speed as shown in Figure 1. Laser beam conditions and specimen moving speeds are listed on Table 1.

Laser Treated samples were cut into segments of about 4 mm long and weighed. They were put into the sample chambers of differential scanning calorimeter (Perkin - Elmer Model DSC4) and scanned from -40 °C to 120 °C with 10 °C/min. heating rate and cooling rate.

Samples for microstructural observation by scanning electron microscope (Hitachi S-570) and optical microscope were imbedded in an epoxy and surface was ground with graded sand papers

(240, 400, 600 grit) and polished with 1 μm and 0.05 μm alumina powders. The final surface was prepared by etching about 30 seconds with solution of hydrofluoric acid, nitric acid and water made of 1: 2: 17 by volume, respectively.

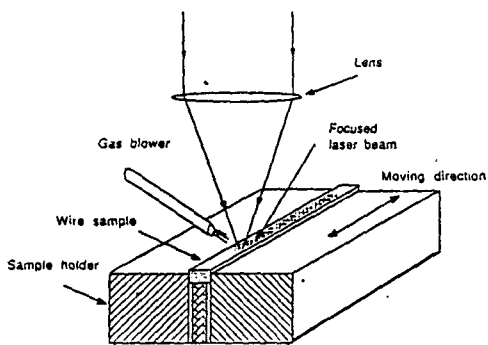


Figure 1. Schematic diagram of laser treating sample holder

Table 1. Laser treatment conditions on NiTi alloys.

Condition	Nd-Yag laser	CO ₂ laser
Laser output	0.5 J/cm ²	30 W
Lens diameter (mm)	25.4	25.4
Focal length of lens(mm)	100	25.4
Measured beam diameter(mm)*	0.3	0.6
Moving speed (mm/min)	60	90 - 210
Pulse duration time (msec)	2**	continuous wave
Shielding gas	argon	nitrogen

* Beam diameter was decided by measuring the punctured hole diameter on the transparent acrylic plate which were made by laser exposure.

** Pulse rate of Nd-Yag laser was 7/sec.

Microhardness of cross sectioned surface of laser treated samples were measured with diamond pyramid hardness tester (Leco M-400).

Semiquantitative compositional microanalysis of samples were taken with x-ray energy dispersive microanalyzers (Hitachi S-570 and EDS system Kevex 8000).

RESULTS AND DISCUSSION

Microstructural Analysis.

Figure 2 shows SEM picture of the surface of transverse cross sectional area after CO₂ laser heat treatment with 50W focused beam and 210 mm/min moving speed. It shows a newly formed surface film, resolidified zone, heat affected zone and matrix. The matrix phase shows many pores developed by the etchant.

Figure 3 is the enlarged picture of top surface film area. The newly formed surface layer thickness is about 3 μm deep. Dendritic resolidified structures are observed below the surface film. Rapidly resolidified structures are also observed below the slowly resolidified dendritic structure layer. But heat affected zone structure remains unchanged.

Studying SEM pictures, it was found that there was no etched out pores in the rapidly resolidified zone. It indicates that rapid resolidified zone has higher chemical resistance than the original matrix. The results of semiquantitative x-ray microanalysis are plotted in Figure 4. The results show that the newly formed surface film is composed of mostly titanium and it may prevent nickel ion from releasing into human body. However, titanium composition of dendritic resolidified region was slightly depleted.

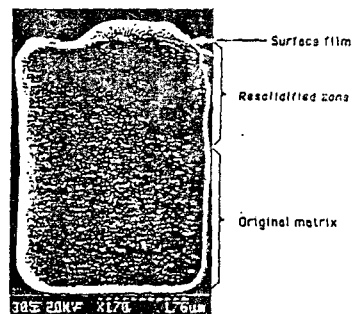


Figure 2. Low magnification (170 X) SEM picture of the transverse cross-section after CO₂ laser treatment. Note the shape of the top section due to the melting and resolidification.

Micro hardness was measured across the cross sectioned surface of laser treated samples. These results are shown schematically in Figure 5. It shows that hardness of resolidified area is about 230 in diamond pyramid hardness scale and hardness of heat affected zone is about 270 and unaffected matrix is about 420. One can deduce from this result that the surface become softer, can

be deformed more easily than matrix. The compositional analysis shows the similar results as discussed earlier.

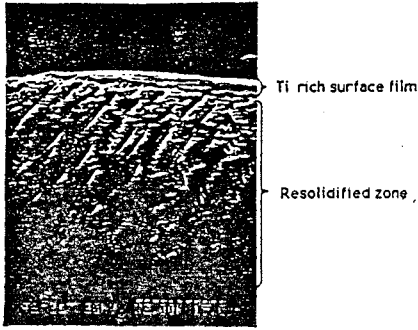


Figure 3. Higher magnification of the surface layer of Figure 2. Note the dendritic growth feature in the resolidified zone.

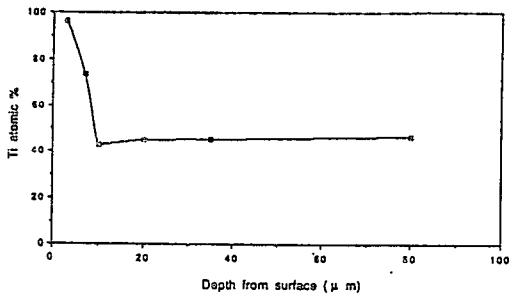


Figure 4. Ti element after CO₂ laser treatment profile along the depth of the transverse cross-section of NiTi wire.

DSC measurements

The NiTi alloys generally display two peaks which are associated with martensitic phase transformation temperature in cooling (M_s and M_f) and heating (parent phase transformation, A_s and A_f) where subscripts s and f denote the starting and finishing of transformations respectively as shown in Figure 6. The area under the specific heat versus temperature curve is the amount of thermal energy used for the phase transformation.

Results of the DSC measurements after laser treatment are illustrated schematically in Figure 7.

Previous work in our laboratory for annealed NiTi wire have shown that, increasing the annealing temperature, decreased TTR. And it did not show any transition temperatures for as-received NiTi wire samples, hence have no SME.

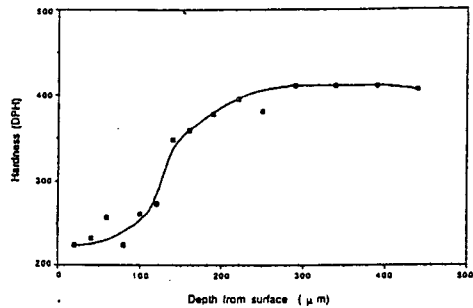


Figure 5. Micro hardness variation plotted along the depth of the wire cross-section after Nd-Yag laser treatment on the surface.

There were parent phase transformation peaks at -5°C (A_s) to 23°C (A_f) for the CO₂ laser treated samples which show relatively wide range of TTR. Because of the existence of these parent phase transformation peaks, one can deduce the laser treated samples have SME. Mechanical measurements verified these although we're trying to get more data at this time. As the scanning speed decrease, the more laser power is delivered and the martensite transformation temperature range was increased and peaks were narrowed. In effect, the more heat delivered by the laser, the higher martensite transformation temperature results.

CONCLUSION

Based on the present study one can conclude that the laser beam treatment on NiTi wire surface could produce titanium rich surface film and rapid resolidified zone. Those newly formed layers show higher chemical resistance compared to the matrix. The resolidified area and heat affected zone become softer. Laser treated samples are shown TTR's which are related to SME.

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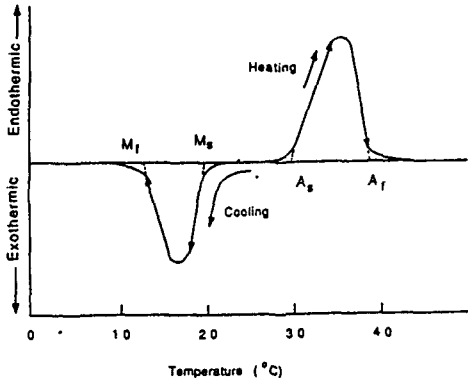


Figure 6. A typical DSC curve of the specific heat (thermal energy) versus temperature

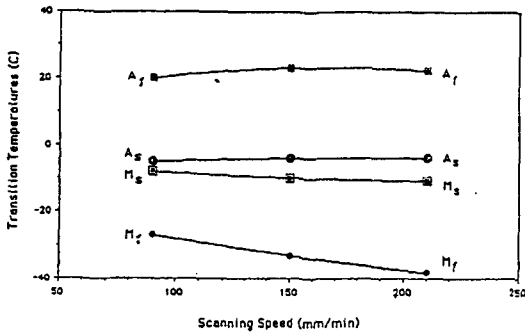


Figure 7. Phase transformation temperatures of CO₂ laser treated NiTi alloy.

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