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Laser Annealing on the Surface Treatment of Thin Super Elastic NiTi Wire

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Abstract. Here the aim of this research is annealing the surface of NiTi wire for shape memory alloy, super-elastic wire by solid state laser beam. The laser surface treatment was carried out on the NiTi wire locally with fast, selective, surface heat treatment that enables precisely tune the localized material properties without any precipitation. Both as drawn (hard) and straight annealing NiTi wire were considered for laser annealing with input power 3 W, with precisely focusing the laser beam height 14.3 % of the Z-axis with a spot size of 1 mm. However, straight annealing wire is more interest due to its low temperature shape setting behavior and used by companies for stent materials. The variable parameter such as speed of the laser scanning and tensile stress on the NiTi wire were optimized to observe the effect of laser response on the sample. Superelastic, straight annealed NiTi wires (d: 0.10 mm) were held prestrained at the end of the superelastic plateau (ϵ : 5 ~6.5 %) above the superelastic region by a tensile machine (Mitter: miniature testing rig) at room temperature (RT). Simultaneously, the hardness of the wires along the cross-section was performed by nano-indentation (NI) method. The hardness of the NiTi wire corresponds to phase changes were correlated with NI test. The laser induced NiTi wire shows better fatigue performance with improved 6500 cycles.

1. Introduction

NiTi based shape memory alloys are used in a wide variety of engineering applications [1] due to their unique thermomechanical functional properties. Shape memory alloys (SMAs) belong to the category of smart materials offers the unique properties of super-elasticity and shape memory effect [2]. The near equi-atomic NiTi alloy is emerging as one of the most demanding among SMAs alloys due to the stable performance in fatigue performance. Fatigue limits the long term safe use of the material and reduces the durability of the application. Generally, fatigue occurs in the cycle of loading and unloading of the sample during application, at various operating environments (specially temperature as one of the crucial parameters during application stage) with proper surface conditions. According to theoretical prediction the solid state conversion in NiTi alloy martensite (monoclinic, B_{19}') daughter phase stable at low temperature derived from austenite (B_2) parent phase (cubic) stable at high temperature towards could deliver millions of mechanical or thermomechanical cycles as the function of strain or stress [3]. However, in practice to achieve the structural fatigue performance for NiTi alloy is a challenge to researchers. The main reason for the obstacles is the surface oxide layer formation during conventional heating method [4] of NiTi alloy for the phase transformation during heat treatment in a furnace at the range of 400 to 500 °C for 30 minutes. To improve the heating process, the conventional method shifts towards laser technology that not only improves the temperature profile also avoids the oxide layer formation on the surface of NiTi wire. To improve the surface



treatment process, earlier researchers were used a various alternative process such as electric pulse heating under mechanical constraint [5] method in replace of the conventional heating method. Although electric pulse treatment is a faster process and improves the functional properties of NiTi alloy, however the upscaling of this technology is an obstacle.

Laser technique offers a lot of promising factors in surface treatment such as high throughput, rapid technology, flexibility in process technology, quick and reproducibility process with better quality in the product. Laser heating is emerging a new innovative approach for the surface treatment of NiTi wire rather than the application limited in areas of melting and cutting technology [6]. Thin films of amorphous NiTi alloys were annealed with a laser beam to improve the fatigue life with small spots of SMA [7]. However, there is lack of work presented in the areas of laser surface annealing of NiTi wire at constant strain by inducing homogenous functional properties.

In the present work, the laser annealing was carried out on the surface of NiTi wire on martensite phase (straight annealed) at constant strain. Superelastic, straight annealed NiTi wires (d: 0.10 mm) were held prestrained at the end of the superelastic plateau (ϵ : 5 ~6.5 %) above the superelastic region by a tensile machine (Mitter: miniature testing rig) at room temperature (RT). The laser plasma creates much larger pressure that resulted from the momentum impulse generated by surface heating rapidly by the laser pulse. As a result, laser pulse interaction generates stress waves intensity from the interaction point to the core of the alloy. The laser annealing of super-elastic, straight annealed wire was performed with the various speed of laser scanning. As a result, it can improve the annealing behavior of the sample during laser treatment and improve the fatigue cycle. The laser annealed NiTi wire (as drawn, hard) was investigated by the tensile machine. Thin NiTi wire was investigated in cyclic tensile tests beyond the end of the transformation plateau at RT until a failure happens. The aim was to generate compressive stress on the NiTi wire by the laser low temperature shape setting process to improve fatigue life. The hardness, Young's modulus and remnant depth of the laser treated NiTi wire were measured by the NI test.

2. Experimental method

2.1. Materials

NiTi superelastic wires, Ti-50.9 at.% Ni (both as drawn and straight annealing, 100 μm of diameter) were supplied by Fort Wayne Metals company.

2.2. Laser Technique

The laser experiments were carried out on the Lumonics Laser JK701H, 1064 nm solid state laser (Nd: YAG) from the Research Center for Engineering Production Technology and Technology at Czech Technical University. The continuous laser scanning was performed at a frequency of 500 Hz with a residence time (Δt) of 0.5 ms, input power 3 ~4 W.

2.3. Methods

Laser experiments were performed on the surface of NiTi wire at the tensile machine (miniature tensile machine, Metter, Max. Load: 10 N) strained with a fixed value (5~ 6 %) after plateau region. Tensile stress response of NiTi wire without and with laser treatment was tested as the function of strain. The hardness measurement was performed on laser treated NiTi wire mounted on the epoxy resin with capillary fixed at the end point by nano indentation method. Fatigue tests were carried out in strain controlled mode (strain rate: 0.01 s^{-1}) for laser annealed samples at constant temperature 20 $^{\circ}\text{C}$. The limit values were chosen with stress value of σ_{min} : 10 MPa and σ_{max} : 700 MPa and strain values are ϵ_{min} : 0 % for both NiTi wire and laser annealed NiTi wire. The upper limit ϵ_{max} for NiTi wire was chosen 6.5 % and 6 % for laser treated NiTi wire considered in the tensile mode of fatigue cycle until a failure happens.

3. Results

3.1. Surface treatment of as drawn sample from hard to super elastic behavior with laser modification

The hard samples of NiTi wire were fixed at Metter tensile machine with 220 MPa applied stress at a fixed position. Laser scanning was carried out on the upper surface of hard wire at a constant speed. The super-elastic behavior was observed after laser treatment on the hard wire. This experiment confirms the functional behavior of hard wire after laser shape setting. Fig. 1. displays the stress versus strain curve for hard NiTi wire before and after laser treatment. The plateau behavior explains the existence of super-elastic behavior in NiTi wire. Based on this laser surface treatment parameter were chosen for super-elastic straight annealed NiTi wire samples.

3.2. Laser annealing of super-elastic, straight annealed wire at martensitic phase by laser heating

The straight annealed, super-elastic sample was strain up to 6 % and fixed above the plateau region in the tensile machine before laser treatment. Laser heating was performed across the wire in martensitic phase in tension stage and the effect was studied carefully to induce the temperature gradient zone across the cross section from laser annealed point from the edge towards the core of the NiTi wire. With the target maintain the core of the wire unaltered as same as raw NiTi wire. Only induce the localized laser heating effect in the extended wire, as a result, to create compressive stresses from the surface region. Fig. 2. shows the similar mechanical response of NiTi wire at speed of 450 mm/min at 800 MPa and stress versus strain curves coincide with each other.

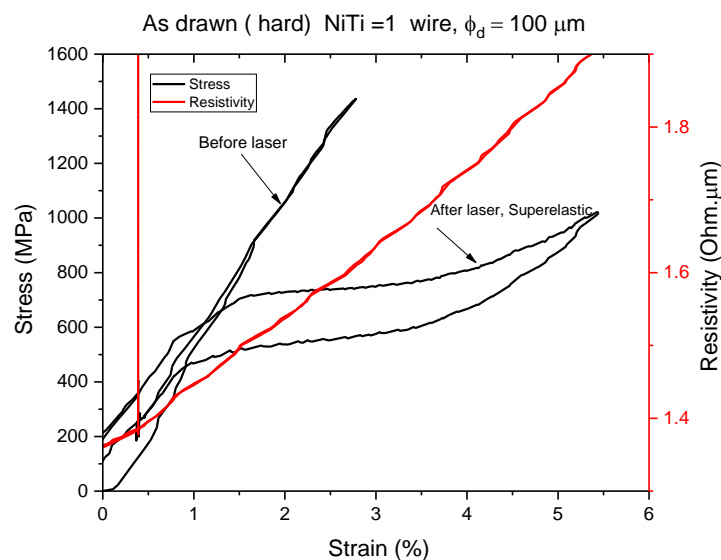


Fig. 1. Stress versus strain for hard NiTi wire before and after laser surface treatment shows response before (straight*, hard) and after laser heating (plateau, super elastic*).

However, on decreasing the speed of 370 mm/min, the stress curve reduces and hysteresis curve lowers with decreasing trend from the original response. This behavior of laser annealing sample is depicted in Fig. 3.

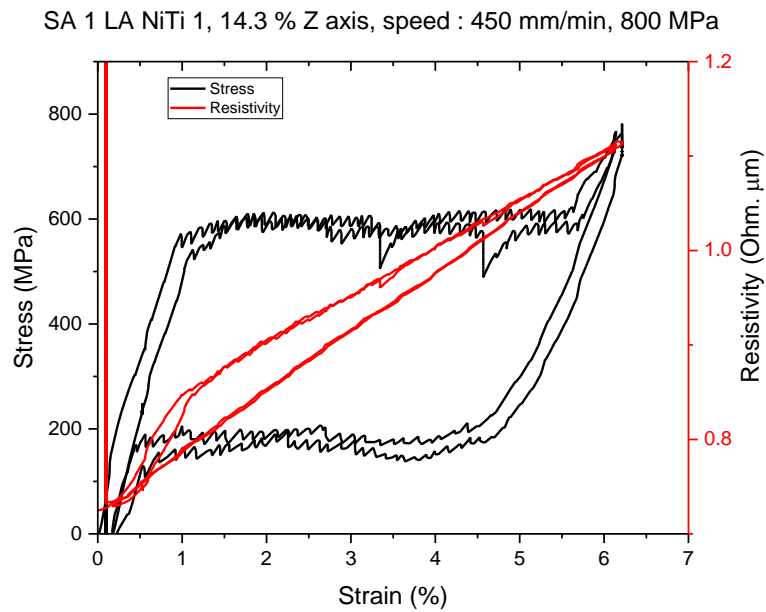


Fig. 2. Laser annealed straight annealed wire at 800 MPa with speed 450 mm/min*.

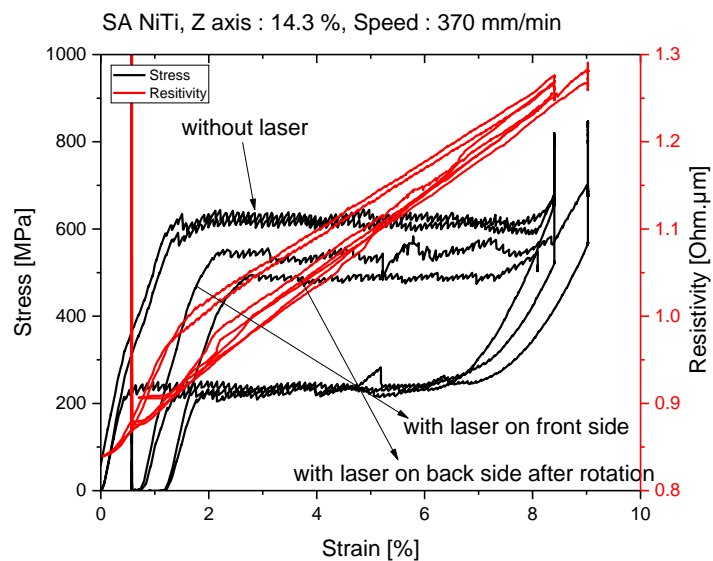


Fig. 3. Laser annealed straight annealed wire at 800 MPa with speed 370 mm/min*.

Fig. 4 represents the stress strain behavior of the sample at a lower speed of 230 mm/min. This shows laser annealed sample accumulates transformation strain more of 2 %. To interpret this mechanism of laser annealing is investigated by NI method of laser annealed NiTi wire along the cross section of the sample during loading and unloading at maximum load.

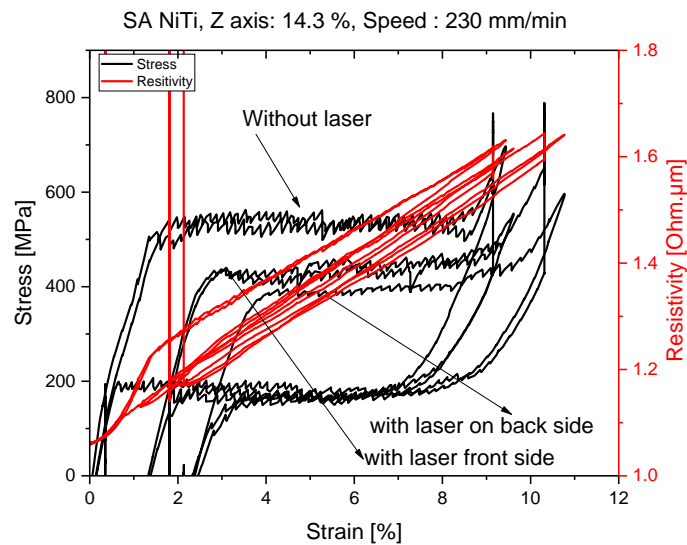


Fig. 4. Laser annealed straight annealed wire at 800 MPa with speed 230 mm/min*.
(* Noise in the curve is due to the error of manual tensile machine)

3.3. NI method of the NiTi wire without and with laser annealed samples

Table 1 shows the summary of NI results for hard NiTi wire, SA wire, and the respective laser annealed samples. Hard NiTi wire shows the higher hardness of 5.5 GPa, however, SA wire is softer shows 3.4 GPa. Laser annealed hard wire shows super-elastic behavior from the Fig. 1, reveal the hardness of 4.6 GPa, however laser annealed SA wire shows slightly increase in hardness 4.5 GPa than the raw SA (without laser) wire. On decreasing the scan speed to 370 mm/min, the annealed of SA wire shows the better result of hardness value of 3.9 GPa. On going to more lower speed of 230 mm/min, the sample shows the slightly lower value of hardness decrease the value of 3.8 GPa.

Table 1. Nano indentation test on the summary of the samples both hard and straight annealing and their respective laser treated samples.

NiTi wire / Property	Hard	SA	HARD + LA, 450 mm/min	SA + LA, 450 mm/min	SA + LA, 370 mm/min	SA + LA, 230 mm/min,
Hardness (H,GPa)	5.46 ± 0.30	3.40 ± 0.11	4.6±0.3	4.52 ±0.22	3.89 ±0.19	3.84 ±0.16
E (GPa)	78.41± 3.30	49.51 ± 2.11	59.61±3.19	50.13 ± 4.69	56.89 ± 4.98	53.76 ±1.6
Remnant depth, H eff(nm)	211.57± 5.64	273.69 ±3.64	NA	244.39 ± 8.19	250.65 ± 5.48	254.21 3.63

3.4. Fatigue life of Straight annealed NiTi wire before and after laser treatment

Fatigue life of SA, NiTi wire shows 3150 cycles until a failure happens (Fig. 5). However, laser treated the sample with a similar response as SA, raw wire from the stress-strain response from Fig. 1 shows the slightly lower value of 3073 cycles of fatigue life until rupture. The laser annealed NiTi wire at speed of 370 mm/ min with reduced stress value shows improved fatigue life of 6500 cycles (Fig. 6). However, the functional behavior of the cold work wire after electrical pulse heating shows compare result on the previous work by the researchers [8-10].

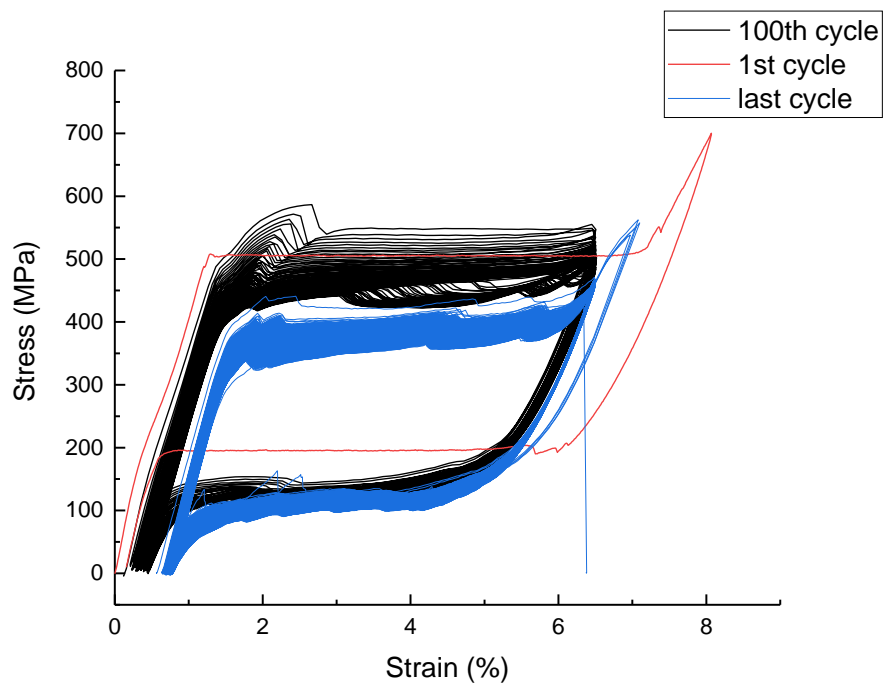


Fig. 5. SA wire (raw, without laser) shows fatigue of 3150 cycles until failure.

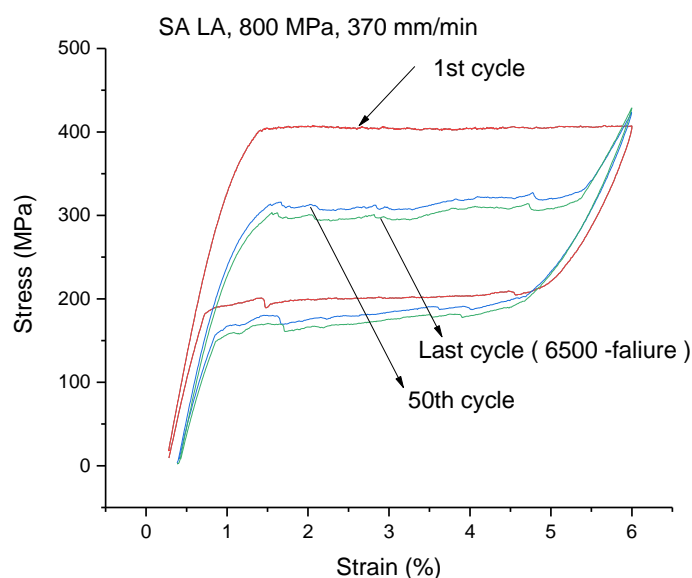


Fig. 6. The laser annealed SA wire corresponds to Fig. 4 shows the fatigue of 6500 cycles until failure.

4. Conclusion

Laser annealing improves the functional properties of super elastic NiTi wire by surface treatment. The phase transformation of martensite to austenite phase during laser heating generates compressive stress on the surface of NiTi wire. Surface annealing of super-elastic, SA wire was achieved by solid state laser beam using both scan speed and applied external stress condition. Both factors have a significant contribution to annealing behavior of the sample. The lower value of the hardness 3.8 GPa of NiTi wire signifies surface annealing of SA sample. The annealed sample shows higher fatigue strength of 6500 cycles in compare to SA sample (3100 cycles). The wires with large residual strain show less fatigue cycle and vice versa.

5. References

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Acknowledgments

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