Martensite Stabilization During Superelastic Cycling of Laser Welded NiTi Plates

J.P. Oliveira, F.M. Braz Fernandes, N. Schell, R.M. Miranda

PII: S0167-577X(16)30263-4
DOI: http://dx.doi.org/10.1016/j.matlet.2016.02.107
Reference: MLBLUE20396

To appear in: *Materials Letters*

Received date: 7 November 2015
Revised date: 17 February 2016
Accepted date: 21 February 2016

Martensite Stabilization During Superelastic Cycling of Laser Welded NiTi Plates

J. P. Oliveira[1,*], F. M. Braz Fernandes[1], N. Schell[2], R. M. Miranda[3]

[1]CENIMAT/i3N, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Portugal

[2]Institute of Materials Research, Helmholtz-Zentrum Geesthacht, Max-Planck-Str. 1, D-21502 Geesthacht, Germany

[3]UNIDEMI, Faculdade de Ciências e Tecnologia, Universidade Nova de Lisboa, Portugal

[*]Corresponding author: jp.oliveira@campus.fct.unl.pt

Abstract

Superelastic behavior of laser welded NiTi is significantly different from the original base material due to microstructural changes introduced during welding. These are responsible for a higher accumulated irrecoverable strain during mechanical cycling. In order to clarify the mechanisms responsible for the evolution of the accumulated irrecoverable strain on superelastic laser welded NiTi, detailed synchrotron X-ray diffraction analysis was performed. Welded samples were analyzed as-welded, after 4 cycles at 10% strain and after 600 cycles at 10% strain. As-welded sample had thermally stabilized martensite in the heat affected and fusion zones due to the welding procedure. It was observed that after 4 cycles, stabilization of the stress induced martensite occurred in a massive way in the thermal affected regions, due to the introduction of defects which prevented the reverse transformation upon unloading. After 600 cycles no significant changes were observed in the thermal affected regions. However, evidence of martensite stabilization in the base material, which was fully austenitic, near the heat affected zone was observed.

Keywords: NiTi shape memory alloys; martensite stabilization; laser welding; phase transformation; X-ray techniques; synchrotron radiation.

1. Introduction

Laser welding has proven to be a suitable joining technique for NiTi shape memory alloys [1–5]. The functional behavior, either by superelasticity or shape memory effect, of the laser welded NiTi joints is dependent on the laser welding procedure.

Both superelasticity and shape memory effect are affected by structural characteristics of the material, such as the martensite stabilization. Martensite stabilization can arise from either the thermal martensite [6] or from the stress-induced martensite [7]. This stabilization is usually studied separately, that is, studies are based on the stabilization of thermal martensite or in the stabilization of stress-induced martensite. In the former case, this stabilization occurs by reorientation of martensite variants [8]. In the latter case, introduction of dislocations during or after the superelastic plateau, makes the reverse transformation require an increased driving force [9]. In either case, the higher the applied stress/strain the higher should be the stabilization effect. As a consequence of this stabilization, the strain recovery after the stress-induced transformation decreases.

Recently, the present authors analyzed the functional fatigue by superelasticity in laser welded NiTi plates [10], by performing cycling tests up to 10% for a total of 600 cycles. The evolution of the accumulated irrecoverable strain was found to increase significantly at the early stages of cyclic deformation, followed by a tendency to stabilize at a fixed value. The presence, at room temperature, of both austenite and martensite in the thermal affected regions, while the base material was fully austenitic, may justify this sharp increase of the accumulated irrecoverable strain. In particular, the existing martensite in the as-welded material should have undergone detwinning during solicitation up to 10%, contributing to the irrecoverable strain of the welded joint. As for the austenite, it is expected that, aside from the introduction of dislocations which are known to occur during cyclic solicitation of NiTi [11,12], some retained martensite is found to occur due the blockage of the reverse martensitic transformation upon unloading [13,14]. In either case, the same phenomenon occurs: martensite stabilization.

In order to understand which phenomena were taking place during cyclic solicitation of these NiTi laser welds, detailed structural characterization by X-ray diffraction using synchrotron radiation was performed in three different welded NiTi joints. These samples were welded with the same parameters to keep consistency between the observed results. The use of a high energy synchrotron radiation is extremely suitable for such laser welds for two main reasons: (i) it allows a finer discretization of the analyzed regions due to the reduced beam spot used; (ii) it is a non-destructive technique which gives information on the microstructure of the material. In this work, stabilization of martensite (either thermal and stress-induced) is discussed based on the superelastic behavior presented by laser welded NiTi sheets complemented with synchrotron analysis performed in the welded samples after a pre-defined number of superelastic cycles

NiTi plates, with a nominal composition of 50.8 at. % Ni - 49.2 at. % Ti, supplied in the flat annealed condition, 1 mm thick, were used. Laser welding was performed using a DY033 Nd:YAG laser power source from Rofin-Sinar, operating in a continuous wave mode, with a gas protection of Argon injected in both face and root of the weld. The laser welding parameters for the analyzed sample in this work are as follows: power of 990 W; welding speed of 20 mm/s; heat input of 495 J/cm.

All samples were removed from the same welded plate and subjected to different mechanical solicitations: one sample as-welded (sample reference W0, where W = weld, 0 = number of mechanical cycles); one sample with a total of 4 load/unload cycles up to 10% strain (sample reference W4); one sample with a total of 600 load/unload cycles up to 10% as reported in [10] (sample reference W600).

X-ray diffraction analysis, using synchrotron radiation, was performed at P07 beamline at PETRA III/DESY, with a wavelength of 0.1426 Å (87 keV). The distance between the sample and the detector was kept at 1090 mm. The beam spot dimension was of 200 x 100 µm. A distance of about 6 mm was probed starting in the base material going through the thermal affected regions and finishing on the other side of the base material. The distance between shots was of 200 µm. Fit2D [15] was used to analyze the raw diffraction images. X-ray diffraction was performed at room temperature as well as the cycling tests.

2. Results and Discussion

The diffratograms for samples W0, W4 and W600 are depicted in Figure 1 a), b) and c), respectively. For the as-welded sample W0, a clear microstructural distinction between the base material and the thermal affected regions is observed. While the base material is fully austenitic, with no evidence of martensite, the thermal affected regions present martensite with a corresponding decrease in the austenite peak intensity. The formation of this martensite is related to two distinct phenomena which occur during welding: (i) precipitation of Ni_4Ti_3 in the heat affected zone [16] and (ii) preferential Ni volatilization in the fusion zone [17].

Analyzing the superimposition of the diffractograms after 4 cycles at 10% strain (Figure 1 b), which had an accumulated irrecoverable strain of 2.85%, a notorious microstructural evolution is observed: the intensity of the martensite peaks, in the thermal affected regions, greatly increases when compared to the as-welded sample. As a result of the augmented intensity of the martensite peaks in these regions, the austenite peak intensity decreases. Additionally, the base material starts to present some evidence of martensite peaks.

After 600 mechanical cycles (Figure 1 c), there are no significant differences in the microstructure of the heat affected and fusion zones when compared to the sample cycled 4 times. Only a slight increase in the martensite peaks intensity was observed. The most significant change occurs in the base material near the heat affected zone, where martensite is clearly visible in this region. This evidence is more clear analyzing the evolution of the net height of the austenite and martensite peaks (the latter located at d = 2.17 Å) along the welded material, as depicted in Figure 2.

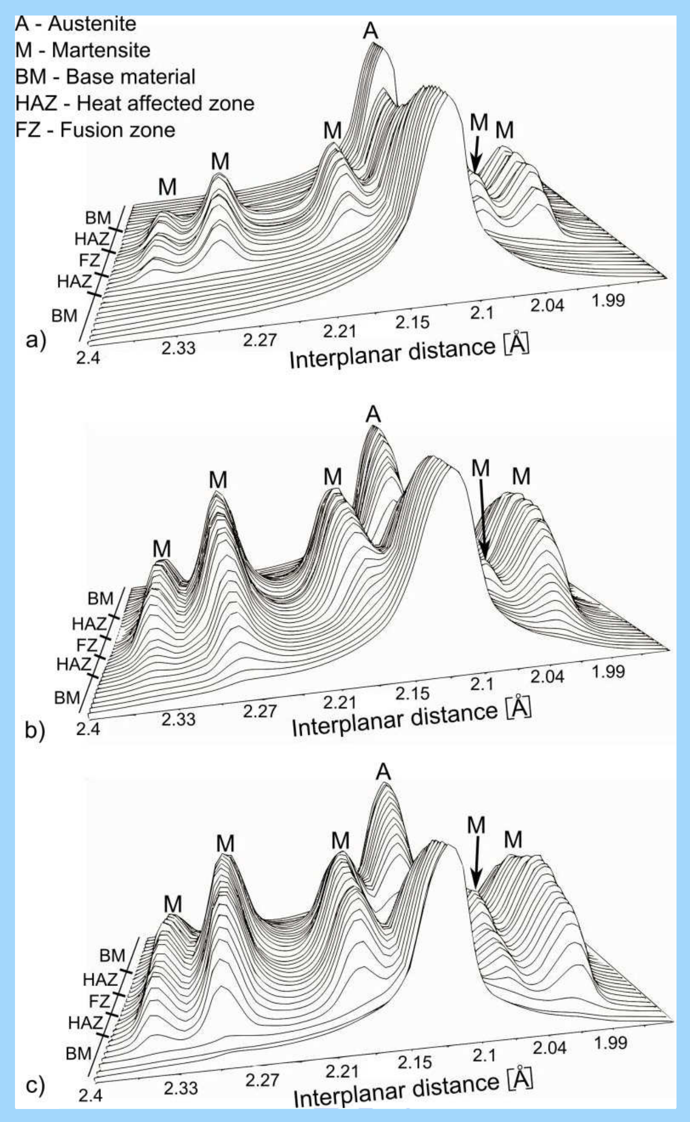

Figure 1 – Superimposition of the X-ray diffractograms for laser welded NiTi samples: a) as-welded; b) after 4 cycles at 10% strain; c) after 600 cycles up to 10 %. Intensity in log scale.

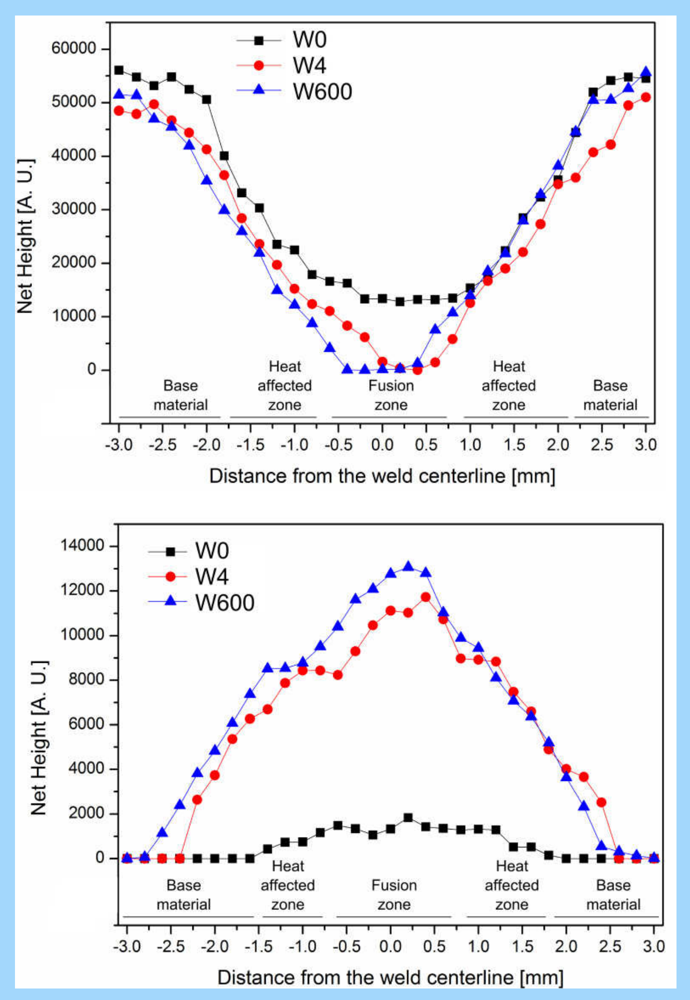

Figure 2 – Evolution of the net height from the austenite peak (top) and for the martensite peak located at d = 2.17 Å (bottom) along the NiTi laser welds.

From the analysis of the aforementioned Figures, it is seen that prior to any mechanical solicitation, the welded material presents a mixture of both martensite and austenite in the heat affected zone and in the fusion zone. This martensite is thermally stabilized at room temperature as a consequence of the weld thermal cycle. While austenite can undergo a reversible stress-induced transformation up to 10% strain [18], the same does not occur with martensite. When in the martensitic state, any applied stress/strain after the onset of the detwinning plateau will generate a given irrecoverable strain [6]. However, it must be noticed that this deformation can be recovered by heating the material up to its A_f temperature in order to trigger the shape memory effect [19]. Cycling solicitation of these laser welds was performed up to 10% at room temperature, so, as far as thermal martensite is concerned, no recovery upon unloading would be expected.

Aside from the stabilization of thermal martensite during mechanical cycling, another martensite stabilization phenomenon occurs: stabilization of stress-induced martensite [6,20]. Delville *et al*. [21] showed that, during superelastic cycling of NiTi, significant accumulated irrecoverable strain could occur due to the introduction of defects along cycling. Later, Sedmak *et al*. [14] performed in-situ X-ray analysis using synchrotron radiation on the same material as in Delville's work. It was observed that, although the accumulated irrecoverable strain increased at each load/unload cycle, only after the 4th cycle, a clear evidence of retained martensite was found. In an ideal superelastic behavior, upon unloading, stress-induced martensite would return to austenite, with no irrecoverable strain. As such, the irrecoverable strain in the early cycles was due to increasing dislocation density in the material. As residual martensite starts to accumulate (or being stabilized), build-up of the residual stress and generation of dislocations during mechanical cycling starts to decrease. In the laser welds studied in this work the same must occur: if after 4 load/unload solicitations the limit of the martensite stabilization is reached, then, after 600 cycles, no significant differences should be encountered. In fact, this can be observed comparing Figures 1 b) and c). The only difference between these two Figures is that some martensite was found to be stabilized in the base material as a consequence of the cycling tests performed. However, this martensite stabilization occurs in the base material closer to the heat affected zone. As we move into the base material, far away from the fusion zone, no evidence of martensite stabilization is observed.

It must be noticed that the length of about 6 mm probed by X-ray diffraction analysis is only part of the gauge length used for the mechanical tests, which was kept at 30 mm. Nonetheless, it is evident that, within this 6 mm length, it is possible to obtain detailed information of the microstructural evolution of the welded joints as a result of the cycling tests performed. This structural information made it possible to highlight the two different mechanisms of stabilization of the martensite, which are relevant to understand the cycling behavior of the welded joints.

It is important to note that the stabilization effect can be reversed by heating the sample up to a temperature that enables the martensite to austenite transformation. After this, it is expected that the transformation temperatures decrease significantly, when compared to the stabilized material [20]. Further studies are planned to analyze the effect of an intermediate reheating on the transformation temperatures of the different regions of the laser welded NiTi plates.

3. Conclusions

Laser welding of NiTi shape memory alloys promotes the formation of martensite in the thermal affected regions, at room temperature, when the base material is fully austenitic. The reason for the occurrence of martensite is related to Ni depletion in those regions leading to an increase of the transformation temperatures. As such, the martensite of the as-welded samples is thermally stabilized. After 4 cycles at 4%, the martensite peaks intensity in the thermal affected regions increase significantly with a correspondent decrease in the superelastic recovery. The reason for the increase of the martensite peaks intensity is the stabilization of stress-induced martensite which cannot return to austenite upon unloading due to the introduction of dislocations during mechanical cycling.

After 600 mechanical cycles up to 10% strain, no significant change in the martensite peak intensity is observed in the thermal affected regions, in comparison to the sample subjected to 4 cycles. However, evidence of martensite stabilization is clearly observed in the base material closer to the heat affected zone. This change is attributed to the introduction of dislocations during the loading path, preventing full recovery by superelasticity. These results are in line with a recent publication of Sedmak *et al*. [14] and are of great importance, when envisaging the use of NiTi in structural or functional applications based on the superelastic effect.

Although martensite stabilization, either thermal or stress-induced, has been studied separately, to the authors' knowledge, this is the first study which evidences both types of martensite stabilization occurring within the same material during mechanical solicitation of a specific industrial application, that is, laser welding.

Acknowledgements

JPO and FBF acknowledge funding of CENIMAT/I3N by FEDER funds through the COMPETE 2020 Program and National Funds through FCT - Portuguese Foundation for Science and Technology under the project number POCI-01-0145-FEDER-007688, Reference UID/CTM/50025. RMM acknowledges funding of UNIDEMI by FEDER funds through the COMPETE 2020 Program and National Funds through FCT - Portuguese Foundation for Science and Technology under the project UID/EMS/00667/2013. The authors acknowledge DESY and HZG for beamtime and travel reimbursement under proposal I-20120563 EC FP7/2007-2013 grant agreement n° 312284. JPO acknowledges FCT/MCTES for funding PhD grant SFRH/BD/85047/2012.

References

[1] A. Tuissi, S. Besseghini, T. Ranucci, F. Squatrito, M. Pozzi, Effect of Nd-YAG laser welding on the functional properties of the Ni–49.6at.%Ti, Mater. Sci. Eng. A. 273-275 (1999) 813–817. doi:10.1016/S0921-5093(99)00422-0.

[2] H. Gugel, A. Schuermann, W. Theisen, Laser welding of NiTi wires, Mater. Sci. Eng. A. 481-482 (2008) 668–671. doi:10.1016/j.msea.2006.11.179.

[3] Y.G. Song, W.S. Li, L. Li, Y.F. Zheng, The influence of laser welding parameters on the microstructure and mechanical property of the as-jointed NiTi alloy wires, Mater. Lett. 62

(2008) 2325–2328. doi:10.1016/j.matlet.2007.11.082.

[4] L.A. Vieira, F.M.B. Fernandes, R.M. Miranda, R.J.C. Silva, L. Quintino, A. Cuesta, et al., Mechanical behaviour of Nd:YAG laser welded superelastic NiTi, Mater. Sci. Eng. A. 528 (2011) 5560–5565. doi:10.1016/j.msea.2011.03.089.

[5] J.P. Oliveira, F.M.B. Fernandes, N. Schell, R.M. Miranda, Shape memory effect of laser welded NiTi plates, Funct. Mater. Lett. 08 (2015) 1550069. doi:10.1142/S1793604715500691.

[6] G. Tan, Y. Liu, Comparative study of deformation-induced martensite stabilisation via martensite reorientation and stress-induced martensitic transformation in NiTi, Intermetallics. 12 (2004) 373–381. doi:10.1016/j.intermet.2003.11.008.

[7] C. Maletta, E. Sgambitterra, F. Furgiuele, R. Casati, A. Tuissi, Fatigue properties of a pseudoelastic NiTi alloy: Strain ratcheting and hysteresis under cyclic tensile loading, Int. J. Fatigue. 66 (2014) 78–85. doi:10.1016/j.ijfatigue.2014.03.011.

[8] Y. Liu, D. Favier, Stabilisation of martensite due to shear deformation via variant reorientation in polycrystalline NiTi, Acta Mater. 48 (2000) 3489–3499. doi:10.1016/S1359-6454(00)00129-4.

[9] Y. Liu, G.S. Tan, Effect of deformation by stress-induced martensitic transformation on the transformation behaviour of NiTi, Intermetallics. 8 (2000) 67–75. doi:10.1016/S0966-9795(99)00079-5.

[10] J.P. Oliveira, R.M. Miranda, N. Schell, F.M. Braz Fernandes, High strain and long duration cycling behavior of laser welded NiTi sheets, Int. J. Fatigue. 83 (2016) 195–200. doi:10.1016/j.ijfatigue.2015.10.013.

[11] A.R. Pelton, Nitinol fatigue: A review of microstructures and mechanisms, J. Mater. Eng. Perform. 20 (2011) 613–617. doi:10.1007/s11665-011-9864-9.

[12] R. Delville, B. Malard, J. Pilch, P. Sittner, D. Schryvers, Transmission electron microscopy investigation of dislocation slip during superelastic cycling of Ni-Ti wires, Int. J. Plast. 27 (2011) 282–297. doi:10.1016/j.ijplas.2010.05.005.

[13] A. Yawny, M. Sade, G. Eggeler, Pseudoelastic cycling of ultra-fine-grained NiTi shape-memory wires, Zeitschrift Fur Met. 96 (2005) 608–618. doi:10.3139/146.101078.

[14] P. Sedmák, P. Šittner, J. Pilch, C. Curfs, Instability of cyclic superelastic deformation of NiTi investigated by synchrotron X-ray diffraction, Acta Mater. 94 (2015) 257–270. doi:10.1016/j.actamat.2015.04.039.

[15] A.P. Hammersley, S.O. Svensson, M. Hanfland, A.N. Fitch, D. Hausermann, Two-

dimensional detector software: From real detector to idealised image or two-theta scan, High Press. Res. 14 (1996) 235–248. doi:10.1080/08957959608201408.

[16] J.P. Oliveira, F.M. Braz Fernandes, R.M. Miranda, N. Schell, On the mechanisms for Martensite formation in YAG Laser Welded Austenitic NiTi, Shape Mem. Superelasticity. 2016 (accepted for publication).

[17] X.J. Yan, D.Z. Yang, Corrosion resistance of a laser spot-welded joint of NiTi wire in simulated human body fluids, J. Biomed. Mater. Res. - Part A. 77 (2006) 97–102. doi:10.1002/jbm.a.30378.

[18] K. Otsuka, X. Ren, Physical metallurgy of Ti-Ni-based shape memory alloys, Prog. Mater. Sci. 50 (2005) 511–678. doi:10.1016/j.pmatsci.2004.10.001.

[19] C.P. Frick, A.M. Ortega, J. Tyber, A.E.M. Maksound, H.J. Maier, Y. Liu, et al., Thermal processing of polycrystalline NiTi shape memory alloys, Mater. Sci. Eng. A. 405 (2005) 34–49. doi:10.1016/j.msea.2005.05.102.

[20] S. Wang, K. Tsuchiya, L. Wang, M. Umemoto, Deformation Mechanism and Stabilization of Martensite in TiNi Shape Memory Alloy, J. Mater. Sci. Technol. 26 (2010) 936–940. doi:10.1016/S1005-0302(10)60151-X.

[21] R. Delville, B. Malard, J. Pilch, P. Sittner, D. Schryvers, Microstructure changes during non-conventional heat treatment of thin Ni-Ti wires by pulsed electric current studied by transmission electron microscopy, Acta Mater. 58 (2010) 4503–4515. doi:10.1016/j.actamat.2010.04.046.

Highlights

- Thermal and stress-induced martensite stabilization in laser welded NiTi plates is evidenced using synchrotron X-ray diffraction.
- Thermal stabilization of martensite occurs in the heat affected and fusion zones due to the welding procedure.
- Martensite stabilization during superelastic cycling occurs more significantly in the early stages of the cyclic deformation.
- After 600 mechanical cycles at 10% strain the base material, which was originally fully austenitic, starts to present martensite.

Graphical Abstract