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In situ structural characterization of laser welded NiTi shape memory alloys

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Abstract. Preserving the shape memory effect (SME) and superelasticity (SE) in shape memory alloys (SMAs) after a joining process is of great importance. In this work Ni-rich NiTi SMAs were laser welded using a Nd:YAG laser in continuous mode. A detailed structural characterization was carried by means of X-ray diffraction (XRD). It was possible to observe the existence of a microstructural gradient from the base material (BM), which contained only austenite, through the heat affected zone (HAZ) and the fusion zone (FZ), where both zones contained austenite and martensite. The mechanical behavior under cyclic load/unload of the welded samples may be explained taking in consideration the structural information retrieved from the XRD analysis.

Introduction

The demand of emerging joining techniques for shape memory alloys (SMA) has become of great importance, as their functional properties i.e. shape memory effect (SME) and superelasticity (SE) present unique solutions for state-of-the-art applications. Literature shows that significant efforts have been spent on laser-welding of these alloys, although only very limited results concerning mechanical properties have been repeatedly achieved.

The interest of welding Ni-Ti shape memory alloys (SMA) preserving their original functional properties comes for their many applications on engineering fields like biomedics, civil construction, metalworking, aerospace and automotive [1-3].

Mechanical aspects of Nd:YAG laser welded NiTi shape memory alloys, were studied by Schlossmacher *et al* [4], who evaluated the mechanical behaviour of a laser welded Ni-rich SMA, performing tensile tests at high strain rates that showed the formation of stress-induced martensite (SIM); also, the ultimate tensile strength (UTS) reached up to 80% of that of the base material. Tuissi *et al* [5] evaluated the effect on functional properties using a Ni-rich SMA at two different initial conditions – fully recrystalized. and partially annealed – in order to optimize pseudoelasticity, concluding that the SIM and strain recovery, by superelasticity (SE) were weakly modified when the base material was fully recrystalized. Falvo *et al* [6] found a reduction on UTS and in shape memory effect (SME), with reduced recoverable strain for strains up to 4 %. Falvo *et al* [7] also studied the functional behaviour of Ti-rich laser welded plates in terms of SME; a growing trend to accumulate inelastic strain over up to 7 training cycles was observed, when strain recovery was done by heating up to parent phase and then cooling down to room temperature (RT). Concerning welding, recent advances have been reported where Nd:YAG lasers were successfully applied in order to minimize the heat input effect. The effect of postweld heat treatment on the fatigue behaviour of laser welded NiTi wires has been studied by Yan *et al* [8], showing that the material annealed at 400 °C for 1 h has the best fatigue strength, while the fatigue strength of the material annealed at 500 °C for 1 h is lower than that of the as-received material. The difference of fatigue life in the heat-treated materials is attributed to the different Ti_3Ni_4 precipitate size: the smaller precipitates are coherent with the matrix and improved the fatigue strength of the NiTi alloy. Regarding the welding parameters and their effect on mechanical properties, Khan *et al* [9] conducted a study on a Ni-rich NiTi alloy, where cyclic loads were applied 50 times up to 6% strain, and the permanent residual strain was used to assess the welding parameters. Gugel *et al* [10] reported, for laser welded Ni-rich wires, a tensile strength up to 75% of the base material, as well as a pronounced SE up to 10 cycles. Maletta *et al* [11] evaluated the fracture behaviour of laser welded NiTi and reported very interesting results regarding tensile strength of welded joints. As a continuation of a previous study [9], Khan *et al* [12] analyzed the effects of laser processing on the transformation temperatures of Nitinol; their DSC results revealed additional high temperature transformation peaks that can be attributed to the local phase conversion induced by laser processing; these results were further corroborated with RT XRD analysis, showing only austenite in the base metal and added martensite peaks in the melted metal. Chan *et al* [13] have found a small amount of Ni_3Ti in the welds; their DSC results show that the onset of transformation temperatures (A_s and M_s) of the NiTi welds shifted to lower temperatures as compared to the as-received NiTi alloy; they assumed that Ni_3Ti precipitation was responsible for the transformation temperatures decrease found by DSC; this argument is not consistent with the fact that the Ni_3Ti precipitation leads to Ti enrichment of the matrix and thus should instead give rise to higher transformation temperatures; UTS of the NiTi welds was comparable to the as received NiTi alloy, but a little reduction in the strain recovery by SE was noted. Vieira *et al.* [14] observed that the rolling direction plays an important role, as far as joint functionality is concerned, as specimens welded across the rolling direction exhibited approximately 2.5 % longer superelastic plateaus and higher UTS; the superelastic behaviour of the welds was observed for applied stresses close to about 100 to 150 MPa below the UTS of the welded joints which is a remarkable result, as far as industrial applications involving welding are concerned. Tam *et al* [15] suggested that preferential vaporization of nickel played a role in the change in transformation temperature at the fusion zone (FZ).

All the above mentioned results on laser welding of shape memory alloys reported up to now by other authors face the problem that the structural characterization as well as the phase transformation history were limited by the spatial resolution of the techniques used; DSC samples of the welds were always incorporating remnant parts of the heat-affected zone (HAZ) and/or the base material; also, the use of X-ray lab sources for phases identification was limited by the spatial resolution of the beam spots, even when fine slits are used. The results here presented are the first reported in the literature to probe the microstructural characteristics of laser welding using a finer spatial resolution allowed by the high brilliance of synchrotron radiation.

Experimental Methods

Material. Commercially available 50.8 at% Ni - Ti cold rolled plates, 1 mm thick, from *Memory-Metalle GmbH* were used. Specimens were cut from as-received plates to 30x30 mm^2 samples for butt welding. Surfaces to join were inspected and hand finished in order to ensure best surface contact along all the joint length. Surface oxides were removed by chemical etching in a $HF:HNO_3:H_2O$ solution with a dilution of 1:5:10.

Laser Welding. A DY033 Nd:YAG laser power source from *Rofin-Star*, operating in a continuous wave mode, was used with a gas protection of Argon on top and Helium in the root with 40 and 50 l/min, respectively, injected in a special chamber built *in situ*. Bead on plate welds were performed on plates, varying welding speed and laser power. The beam was focused to 0.45 mm and the focal point position (FPP) was set on the upper surface. Butt welding was performed with zero gap between opposite plates using the welding parameters summarized in Table 1.

Table 1 – Laser welding parameters for butt joints

Sample reference	Power	Welding speed	Heat input
	W	mm/s	J/cm
A-A	990	25	396
C-C	990	15	660

X-Ray Diffraction Analysis. XRD analysis was performed at the beamline P07 of the PETRA III (DESY, Hamburg), using a 2D detector MAR-3450. Radiation was tuned to 87 keV (0.142 Å). Data acquisition was performed on spots along a line perpendicular to the weld bead (beam spots of 0.1x0.1 mm, spaced by 0.2 mm). The experimental setup used for the XRD is schematically presented in Fig. 1. Tests were run at ambient temperature of 21 ºC.

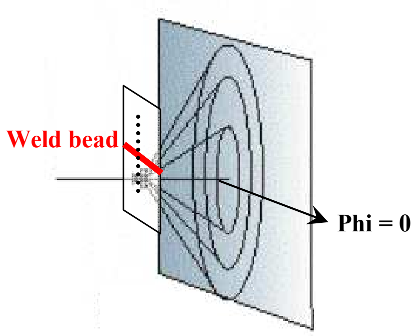


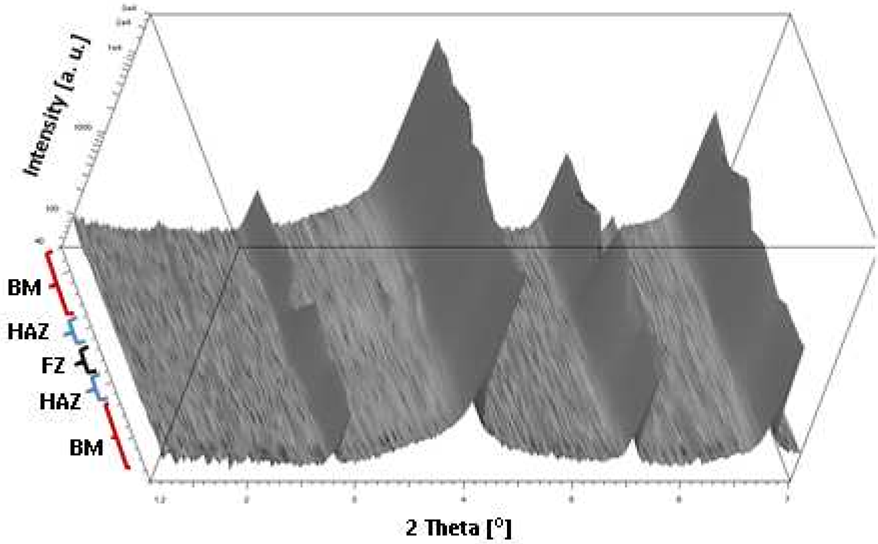
Figure 1 – Schematics of the experimental setup for the laser weld structural analysis.

Results and Discussion

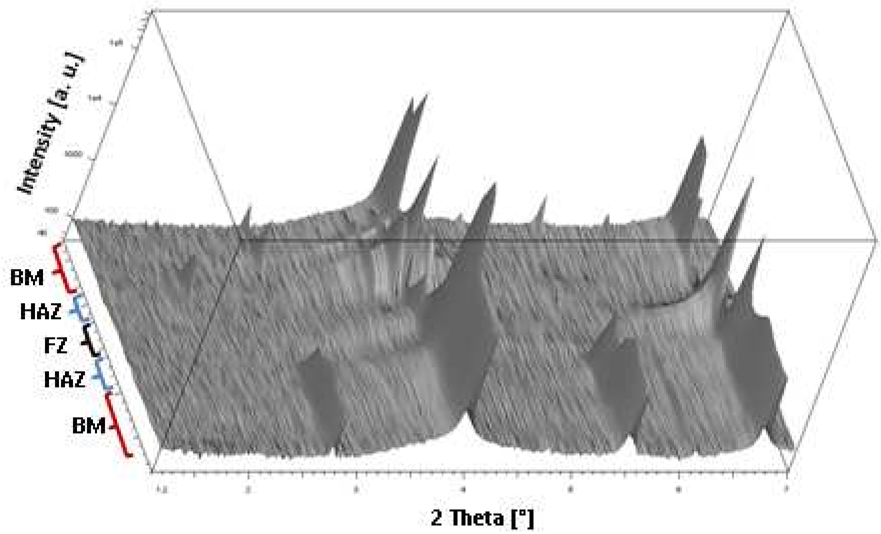
A preliminary study [16] was conducted on NiTi cold rolled plates 1 mm thick with 50.8 at% Ni, a commercial SMA from *Memory-Metalle GmbH* that is specified as superelastic at RT.

The microstructural changes in the weld bead and its neighborhood were analyzed by 2D XRD (I-20100250 EC at beamline P07B, 30/06/2011 to 03/07/2011) using a beam spot of 0.2x0.1 mm along a line crossing the weld bead, from the base material, through the HAZ to the FZ and finally to the base material (Fig. 1). It was noticed that, at RT, while the base material is austenitic, martensite is present in the HAZ and FZ.

Fig. 2 shows the 3D plot of the XRD patterns sequence along a line across the laser weld bead, where the evolution from austenite in base material to prevailing martensite in FZ is clearly perceptible. It is possible to notice the existence of different phases in the base material, HAZ and the FZ as a consequence of the welding process. In particular this is due to heat input in each sample. In the base material, as expected, there is only austenite in all samples. However the relative amounts of martensite and austenite in the FZ and in the HAZ differ from sample to sample. Sample A-A presents both martensite and austenite in the FZ, but martensite prevails. Sample C-C shows a wider region that is thermally affected (HAZ plus FZ), with an even more prevailing amount of martensite compared to sample A-A. The modification of the existing phases relatively to the base material is a consequence of chemical variations that occur in the HAZ and in the FZ. This chemical variation may be due to oxidation or volatilization phenomena or, most likely, due to solubilization of precipitates (in FZ) or precipitation (in HAZ). These chemical alterations lead to changes in the transformation temperature, which in this case justifies the existence of martensite at RT in these thermally affected regions.



A-A



C-C

Figure 2:- X-ray diffraction patterns sequence along line scans across the laser weld bead.

Samples A-A and C-C (lowest and highest heat input, respectively) have been tested for 300 cycles of loading/unloading, strained up to 10%. In both samples, the amount of residual martensite is sufficient to give a considerable accumulated irrecoverable strain in the early cycles. In the long term, the accumulated irrecoverable strain increased much steeper for sample C-C due to the existence of more martensite along a wider band comparing to sample A-A. This increasing irrecoverable strain may be explained by the fact that only the stress induced martensite will recover the strain upon unloading (superelasticity). The martensite that is thermally stable at ambient temperature will not recover upon loading, thus contributing to the accumulated irrecoverable strain with increasing number of cycles.

Conclusions

The texture in the base material and in the HAZ does not vary significantly for the samples analyzed. However, when we approach the FZ, we obtain “spikes” with very high intensity as a result of the existence of a coarse grained microstructure. In the FZ the texture of the base material is completely lost as a consequence of fusion.

The existence of martensite at RT, in the HAZ and, especially in the FZ, lead to an alteration on the mechanical behavior of the samples subjected to cyclic stress, when compared only with the base material. This change of the mechanical behavior is specially noticed, as far as the irrecoverable accumulated strain of the samples is concerned.

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