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High Strain and Long Duration Cycling Behavior of Laser Welded NiTi Sheets

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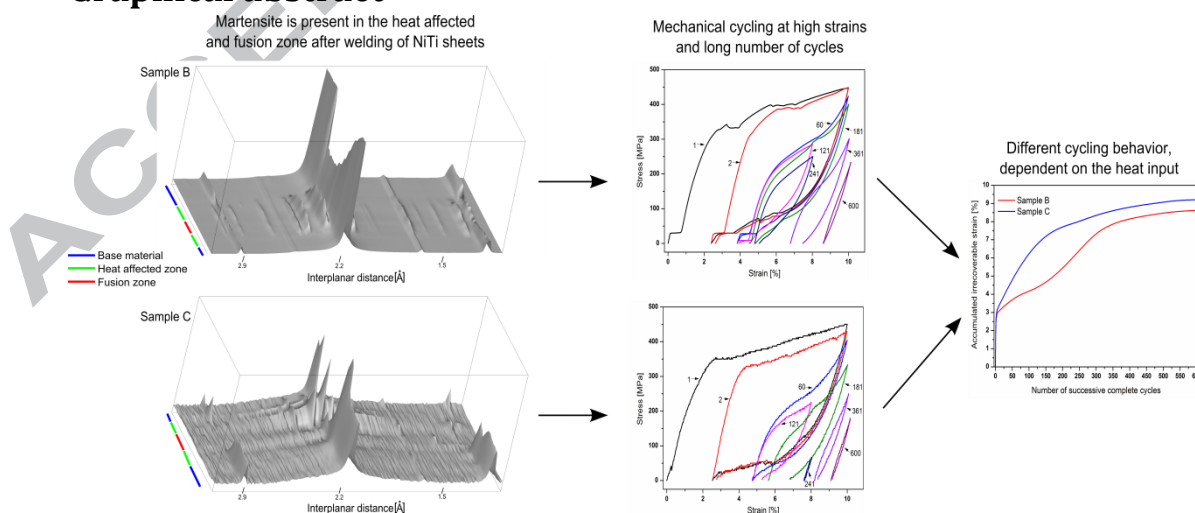
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Abstract

The use of NiTi in complex shaped components for structural applications is limited by the material cost and machinability and adequate joining techniques have been investigated to minimize the thermal cycle effect on the superelastic and shape memory effects exhibited by NiTi. Laser welding is the most used joining process for this material. However, existing studies mainly address the functional properties of laser welded NiTi wires, and the superelastic cycling tests are limited to either a low number of cycles (maximum 100) or to low strains (below 6%). This paper discusses the results of the cycling behavior exhibited by laser butt welded 1 mm thick NiTi plates, when tested to high strains (up to 10%) and for a large number of cycles (600). The superelastic effect was observed and the microstructural changes induced by the laser welding procedure, namely the extension of the thermal affected regions, were seen to influence the evolution of the accumulated irrecoverable strain. Thus, it is possible, by controlling the heat input introduced during welding, to tune the maximum superelastic recovery presented by NiTi laser welds.

Keywords: NiTi; Shape memory alloys; Laser welding; High strain cycling; Superelasticity.

Graphical abstract



Highlights

- Cycling at high strains and for a high number of cycles was performed on laser welded NiTi sheets. This is the first study of this kind.
- Process parameters influence the superelastic recovery. Lower heat input implies lower irrecoverable strain.
- Welded samples were successfully load/unloaded 600 times, 110 to 130 MPa below their ultimate tensile strength.
- Martensite in the thermal affected regions is responsible to lower the superelastic plateau of the welds.

1. Introduction

In the past years we have seen an increasing use of NiTi shape memory alloys in applications, such as: biomedical [1], automotive [2] and aerospace [3]. Some of these applications require the use of complex shapes, which are not easy to obtain due to the difficult machinability of NiTi. For this, intensive research has been devoted to joint NiTi aiming to minimize thermal effects of welding on the material functional properties, that is: shape memory effect and superelasticity.

Amongst the welding processes studied, laser welding is the most used since the laser beam can be focused into a small spot diameter, reducing the extension of the fusion and the heat affected zones. As upon sequence, the welded NiTi joints exhibit mechanical properties below those of the as-received material, namely lower ultimate tensile strength and elongation to fracture.

Understanding the influence of laser welding on the functional fatigue (as defined by Eggeler *et al.* [4]) in terms of the shape memory effect or superelasticity, is of major importance. The effect of laser welding on the functional properties of NiTi joints was studied by several authors [5 – 10] and it is, nowadays, well established that the welding thermal cycle, originates a microstructural gradient from the fusion zone towards the base material with coarse grains in the molten and solidified region.

The shape memory effect was seen to be preserved in the fusion and heat affected zones after welding [10], even if there is a change in the transformation temperatures of these regions when compared to the base material.

Superelasticity of the welded NiTi joints has also been studied, but for a low number of cycles and at low strains [9], [11, 12]. Up to now the maximum cyclic deformation imposed to a laser welded NiTi joint was of 8% for a total of 10 cycles and with a low strain-rate[12] which is known to affect, for example, the superelastic recovery upon unloading of NiTi [13, 14]. Chan *et al.* studied the fatigue behavior of laser welded NiTi wires in small strain cyclic bending tests [15]. Previously, Chan *et al.* [16] analyzed the effect of post-weld heat treatments on the cyclic behavior of laser welded NiTi wires by testing samples up to a maximum of 100 cycles at a maximum imposed strain of 4%.

Additionally, most of the existing studies focus on the analysis of the effect of laser welding on NiTi thin sheets or wires (below 0.5 mm in thickness/diameter) [12], [15 – 19]. Studies on the

superelastic effect presented after laser welding of NiTi is scarce for sheets with more than 0.5 mm thickness [9], [20]. Vieira *et al.* [9] studied the superelastic behavior of laser welded NiTi sheets by analyzing the mechanical behavior of the welds at a 6% strain during 30 cycles. Hsu *et al.* [20] applied a total of 50 cycles up to 3% strain to 2 mm thick NiTi plates welded by laser.

While thin wires are mostly used for biomedical and microelectromechanical systems, sheets or large diameter wires are often required for structural applications. Thus, the analysis of the cyclic behavior in more severe conditions (high applied strains and/or high number of cycles) is required to understand the feasibility of the use of laser welded NiTi in such structures. That is, concerning the superelasticity of laser welded NiTi, there is an area that was not yet addressed: the study of the behavior of welded joints when cycled at high strains and for a high number of cycles.

The authors have previously studied the effect of laser welding parameters on one of the functional characteristics: the shape memory effect of laser welded NiTi plates [10]. The present paper presents a study performed to assess the functional fatigue in terms of the superelastic behavior of laser butt joints in NiTi plates at high strains (up to 10%) and for 600 cycles. A structural analysis by X-ray diffraction supports the discussion of the superelastic behavior observed in the welds. This will open up the possibility of designing and manufacturing complex shaped structural devices using fusion welding by laser.

2. Experimental Procedure

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. Specimens were cut with a diamond wheel on a precision cut-off machine ATM Brilliant from as-received plates to 30x30 mm samples for butt welding. Surfaces to join were inspected and hand finished in order to ensure the best surface contact along all the joint length. Surface oxides were removed by chemical etching in a HF:HNO₃:H₂O solution with a dilution of 1:5:10.

A DY033 Nd:YAG laser power source from Rofin-Sinar, operating in a continuous wave mode, was used with a gas protection of Argon injected in a special chamber built *in situ*. Table 1 summarizes welding speed and laser power selected to verify the influence of the welding parameters on the mechanical properties. The beam was focused to 0.45 mm diameter and the focal point position (FPP) was set on the upper surface. Butt welding was performed with zero gap between opposite plates.

Table 1 - Welding parameters for butt joining 1 mm thick plates.

Power [W]	Welding Speed [mm/s]	Heat Input [J/cm]	Sample Reference
990	25	396	A
990	20	495	B
990	15	660	C

A precision cutting machine was used to prepare the tensile specimens from the butt-welded NiTi plates with the following dimensions: length 60 mm, thickness 1 mm, width 3 mm (Figure

1). All the mechanical tests were conducted at room temperature with an Autograph Shimadzu AG50kNG machine, using a load cell type SFL-50 kN AG. The cross-head displacement speed of 2 mm/min and an average gauge length of 30 mm were selected for the mechanical tests. Uniaxial tensile tests of similar laser welded butt joints in NiTi were performed with single loading until rupture. From the tensile tests, the ultimate tensile strength (UTS) and deformation to rupture were registered and those results were taken into account to fix the conditions for the cyclic loading tests using the same cross-head displacement speed (2 mm/min) and gauge length (30 mm) as before. Cycling tests were performed in the following sequence: stage 1: 60 cycles at 10% of strain; stage 2: 60 cycles at 8%; stage 3: 60 cycles at 10%; stage 4: 60 cycles at 8%; stage 5: 360 cycles at 10%.

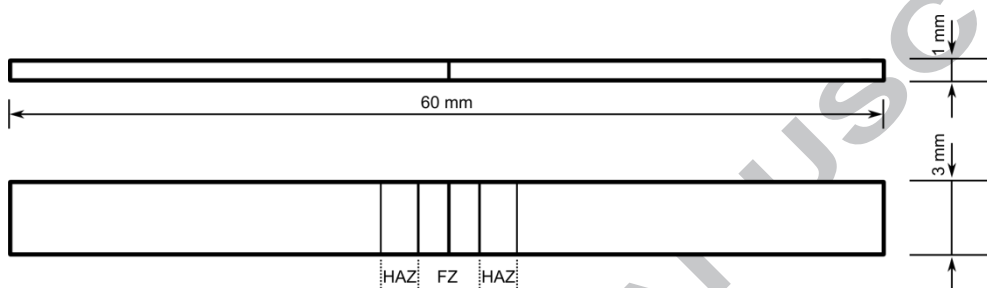


Figure 1 – Dimensions of the tensile specimens. The heat affected (HAZ) and fusion zones (FZ) are not to scale as they depend on the laser welding parameters.

Structural analysis was performed on all samples in line P07 of Petra III/DESY using a wavelength of 0.1426 Å (87 keV). A length of about 6 mm was analyzed, starting in the base material, through the heat affected zone and the weld bead and finishing in the base material again. The distance between shots was of 200 μm and the beam dimensions were 200 x 100 μm.

The data acquired was treated using the program Fit2D [21] and was possible to obtain the diffractograms in the different regions of the welds: base material, heat affected and fusion zones.

3. Results and Discussion

Table 2 presents the strength and ductility parameters for the welded samples studied (Figure 2). The NiTi base material had an UTS of 1530 MPa with an elongation to fracture of 20.1 %. The superelastic plateau of the base material was at 350 MPa. All uniaxial-tensile tests until rupture of the laser welds fractured in the fusion zone.

Table 2 - Strength and ductility parameters of the samples analyzed, obtained from tensile tests to rupture.

Sample Reference	Ultimate tensile strength [MPa]	Elongation to fracture [%]
A	537	12.1
B	511	10.6
C	530	11.1

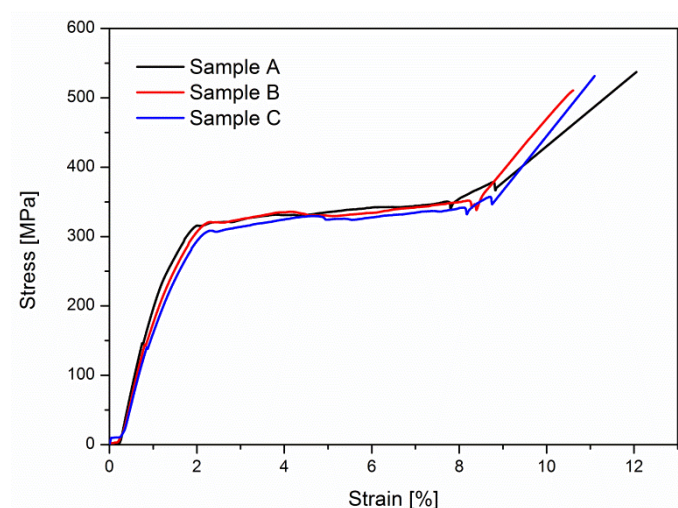


Figure 2 – Tensile tests of the analyzed laser welded NiTi samples.

The mechanical cycling tests for the welded samples A, B and C, are depicted in Figure 3 a), b) and c), respectively. For all samples a selected set of load/unload cycles are shown. Sample A fractured at cycle number 303 in the base material due to a pre-existing defect in this region. So, the welding procedure did not have an influence on the fracture of this sample.

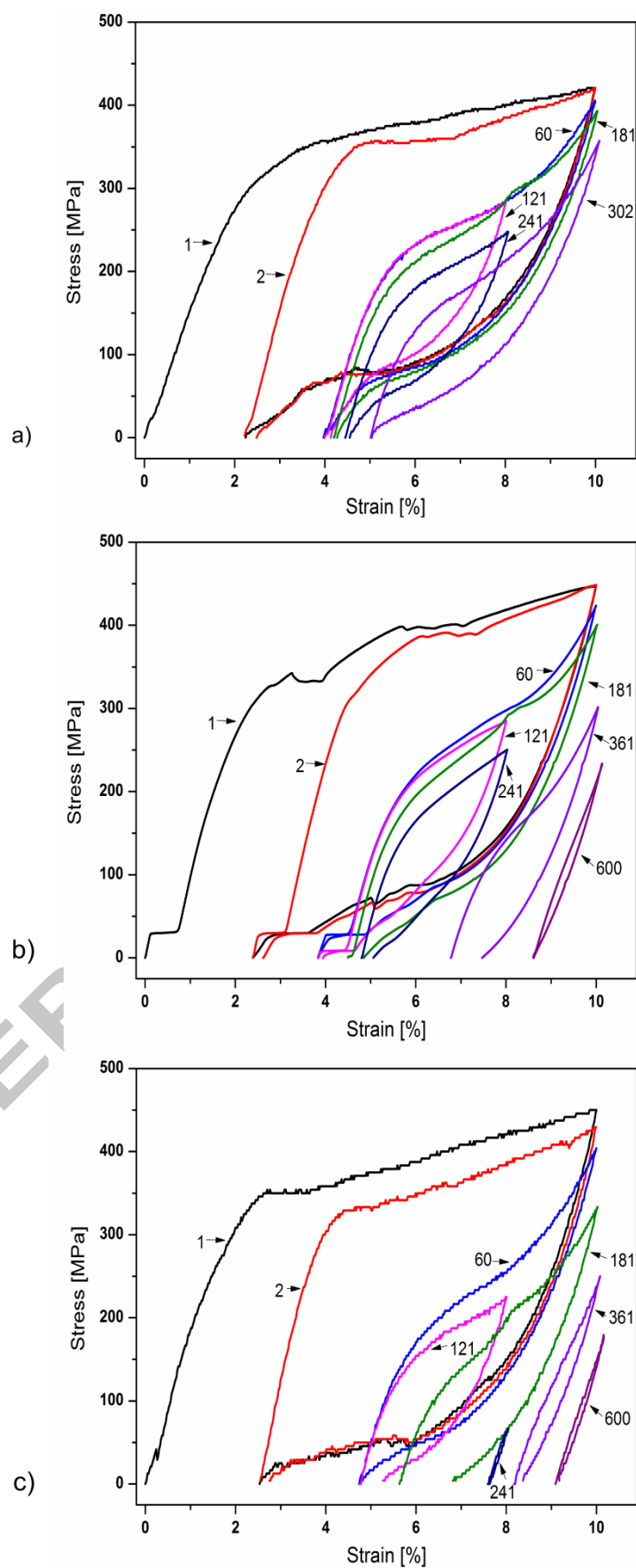


Figure 3 – Cycling tests for: a) sample A; b) sample B; c) sample C.

It must be noticed the great resistance of the samples when subjected to load/unload cycling with very large alternating strains (8% / 10%). In particular, samples B and C were seen to stand the entire programmed cycling test without breaking.

In all samples, the superelastic plateau on the first cycle occurs at about 350 MPa, similar to those of the base material. Also, samples B and C were cycled up to 600 times, at a stress level 110 to 130 MPa below their UTS, without rupture. In sample B a very small plateau at low stress was detected in the initial stage of the cycling protocol which is attributed to the presence of residual R-phase. However, with the prosecution of the cyclic solicitation, the R-phase transformation is suppressed.

In an ideal superelastic behavior, a complete strain recovery could be obtained in NiTi SMAs for strains up to 10% [22]. However, there are tests, as well as material parameters, that give raise to a non-complete ideal superelastic behavior. For example, the temperature at which the tests are performed, the strain rate imposed [13, 23], the maximum strain [24], the effect of thermo-mechanical treatments [25] and the grain size [26] are all known to affect the superelastic behavior of NiTi.

Thus, a comprehensive structural characterization of the welds was performed by X-ray diffraction using synchrotron radiation in order to get a fine spatial resolution. This study revealed a mixed microstructure in the heat affected and in the fusion zones, with both martensite and austenite, present at room temperature. Figure 4 shows three diffractograms from spots located at three different regions of the welded material in sample B (as an example). The base material is fully austenitic at room temperature. On the other hand, the heat affected zone and the fusion zone, have both martensite and austenite at room temperature; the extension of these regions may then be associated to the existence of the martensite peaks.

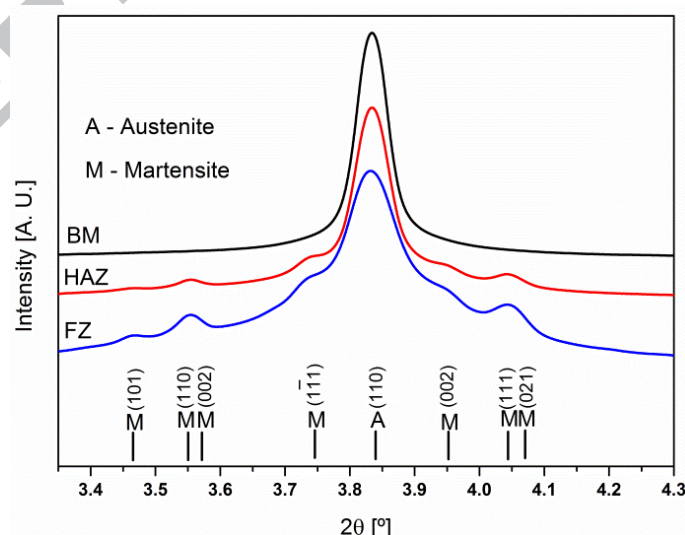


Figure 4 – Diffractograms of the base material (BM), heat affected zone (HAZ) and fusion zone (FZ) of sample B. For each identified phase, austenite (A) and martensite (M), the (hkl) family plane is evidenced.

Figure 5 depicts the 3D stacking of the diffractograms for the analyzed samples, showing the extent of each region. As expected, the extension of the heat affected and the fusion zones is

related to the welding parameters: higher values of heat input (power to welding speed ratio) originate larger thermal affected regions.

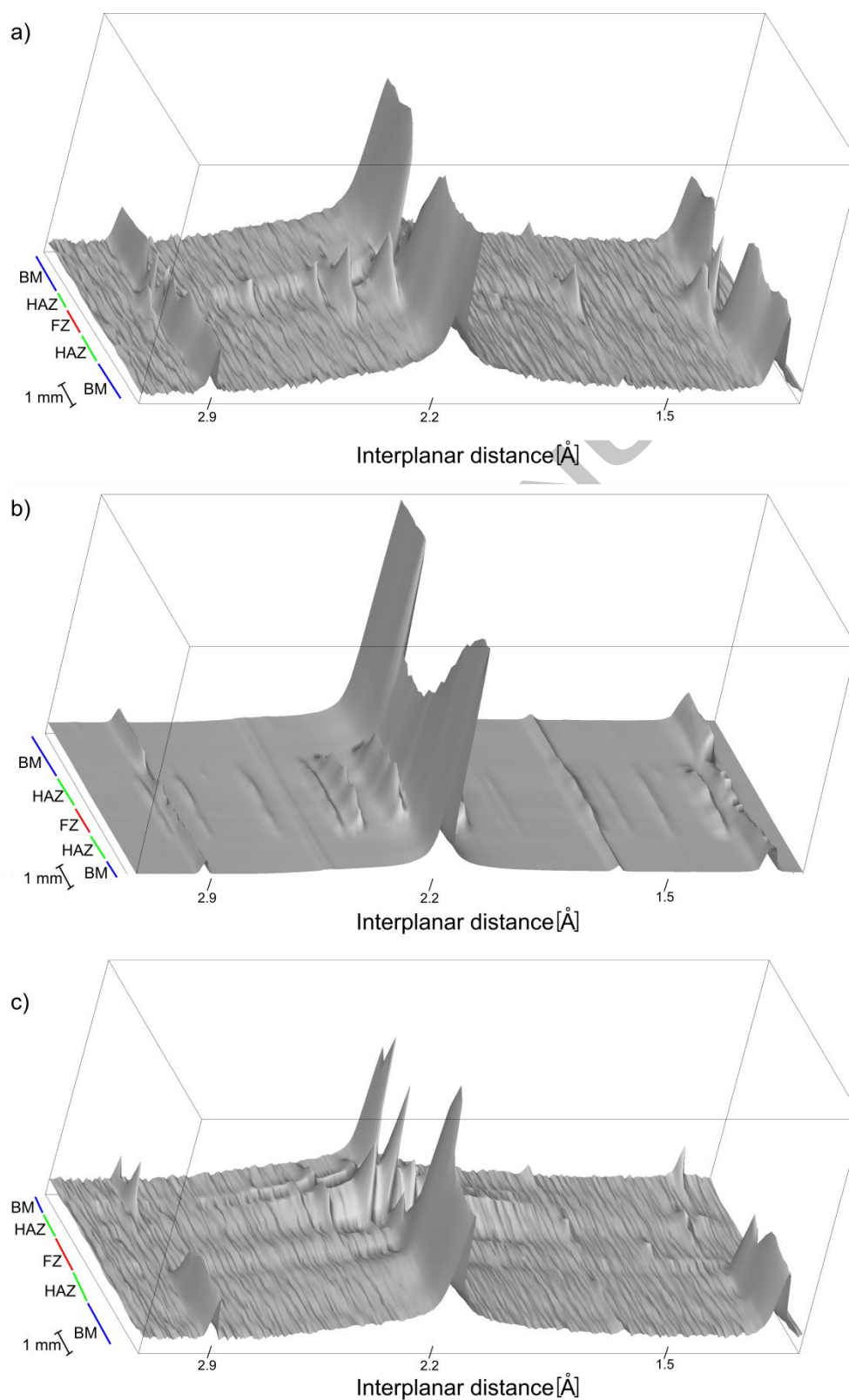


Figure 5 – Diffractograms of welded samples: a) sample A; b) sample B; c) sample C. BM – Base material; HAZ – Heat affected zone; FZ – Fusion zone.

The very large variations of the diffraction peaks intensities observed in some diffractograms is clearly a result of coarse grain size in the heat affected zone and fusion zone.

As the superelastic effect is dependent on the amount of austenite available to undergo the stress induced transformation, the presence of martensite at room temperature will have an implication on the functional behavior of the welded joints. It is expected that the superelastic recovery decreases with the increase in martensite fraction in the heat affected zone and in the fusion zone.

The presence of martensite in the heat affected zone and fusion zone of the welds brings particular features to the cyclic response of the tested materials. As the individual presence of each phase gives different contributes regarding the strain recovery upon unloading during cyclic solicitation, the presence of both phases in the welded material must be considered when analyzing the mechanical cycling tests.

It is well known that in the martensitic phase the cyclic behavior is heavily dependent on the applied strain [27, 28]. In this phase, as soon as the plateau for the detwinning martensite is reached, an irrecoverable strain is expected to occur which should correspond to the difference between the initial elastic deformation of martensite and the applied strain. However, as long as the applied stress is not enough to promote dislocations slip, a significant, if not total, recovery of the deformation can be achieved by heating the material above A_f , triggering the shape memory effect.

Tuissi *et al.* [5] suggested that the heat affected zone had a softening effect, lowering the stress level of the plateau in NiTi welds. Their tests were performed above the A_f temperature of the base material, but DSC analysis of the fusion zone revealed that, at testing temperatures, austenite was not yet completely transformed, having some martensite in this region. For this reason, the strain recovery of the welds was lower than that of the corresponding base material. However, the softening effect caused by the presence of martensite in the thermal affected region is evidenced in our laser welds by a significant decrease of the onset for the superelastic plateau, as the number of cycles increases.

In the present study, since there was a high strain applied to the welds, the martensite in the heat affected zone and in the fusion zone are detwinned and upon unloading a significant irrecoverable strain is observed.

The evolution of the accumulated irrecoverable strain with the number of cycles for each sample is shown in Figure 6. It can be seen that a significant amount of accumulated irrecoverable strain occurs at the end of the first cycle of the load/unload protocol. This can be explained due to the presence of martensite in the heat affected zone and in the fusion zone of the laser welds. When compared to the reported literature on laser welding of NiTi wires, it is noticed that the accumulated irrecoverable strain is lower than in the results recorded in our plates. The main reason for this lies on the high strain applied which is considerably higher than the values reported by others [16, 18, 20] (ranging from 2.5 to 4% strain) and, thus, a higher irrecoverable strain is expected.

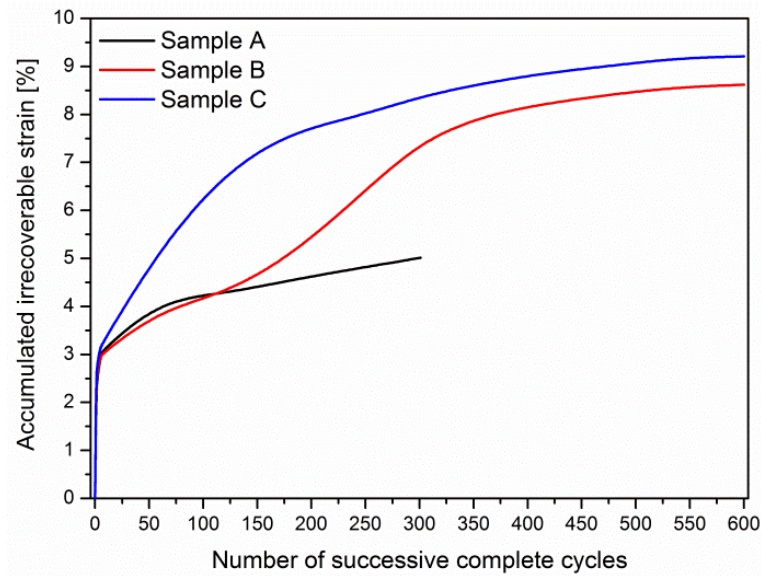


Figure 6 – Evolution of the accumulated irrecoverable strain with the number of cycles for each sample.

Load/unload tensile tests performed by Wada *et al.* [28] on martensitic NiTi show that, for tests at 8% strain, the recoverable strain, upon unloading to zero stress, is of about 1%, corresponding to the elastic deformation of martensite. Further deformation would result in dislocation slip. In our laser welds with the presence of martensite, aside from austenite, in both heat affected zone and fusion zone, the evolution of the accumulated irrecoverable strain for each sample can be explained as follows: for the sample with higher heat input, a higher amount of martensite is present, as it can be observed in the diffractograms of sample C in Figure 5 c). Thus, a higher irrecoverable strain will occur for this sample associated to the detwinning of the existing martensite, as well as some dislocation slip due to the high strain imposed. For the sample with lower heat input the irrecoverable strain tends to be, correspondingly, lower.

Miyazaki *et al.* [29] showed that, under superelastic cycling of NiTi, dislocations and other lattice defects are introduced during the stress induced transformation and tend to pile-up with increasing number of cycles, being responsible for the increase of the accumulated irrecoverable strain after each cycle. In our case, for the existing austenite in the welded material, it is reasonable to conclude that some of the increase in the accumulated irrecoverable strain is due to the introduction of dislocations during the stress induced transformation. These defects do not allow for the stress induced martensite to revert to austenite upon unloading, leading to a stabilization effect of martensite. This stabilized martensite does not participate in the subsequent superelastic transformation cycles as the number of cycles increases.

It is also observed that in stage 2 of the cycling tests performed (corresponding to the first set of 60 cycles at 8% strain) the superelastic plateau abruptly changed its slope as a result of the strain-hardening and the corresponding increased dislocation defects that resulted from the first set of 60 cycles at 10% strain. As the number of cycles continues, the accumulated irrecoverable strain increases up to a point where the stabilization plateau is reached, as depicted in Figure 6 for samples B and C. The convergence to a stabilized value of the accumulated irrecoverable strain occurs almost at the same time for these two samples.

Another interesting feature that can be inferred from the mechanical tests of the welds is that all samples were mechanically tested for a significant number of cycles at stress values (110 to 130 MPa below) close to the ultimate tensile strength of the welds tested until rupture.

Grain size effects cannot also be neglected when looking at these results. In the fusion zone a coarse grain structure is found, as it can be seen in the macrographs of samples A and C, Figure 7 a) and b), respectively. For NiTi shape memory alloys it is known that the larger the grain size, the lower the superelastic recovery is [26]. That is, since the fusion zone and the heat affected zone have a coarser grain structure than the base material, they also contribute for the accumulated irrecoverable strain presented by the welded joint.

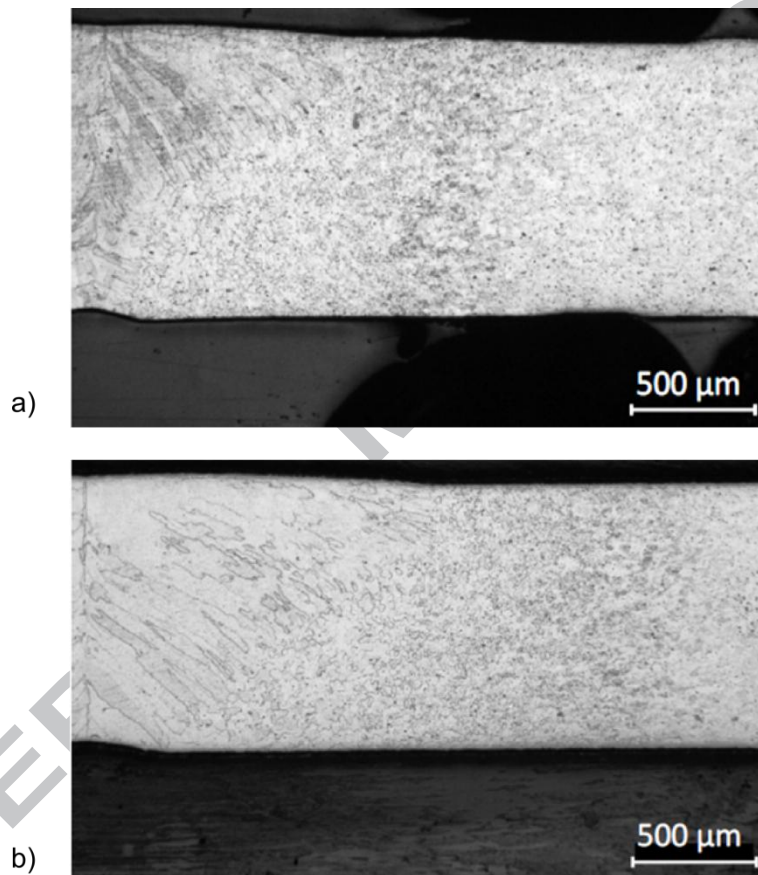


Figure 7 – Macrographs of: a) sample A; and b) sample C.

4. Conclusions

Mechanical behavior of laser welded superelastic NiTi plates was investigated by cyclic loading/unloading tests up to strains of 10% and 600 cycles.

This is the first study addressing mechanical cycling of welded plates at high strains and for a high number of cycles. The mechanical cycling of the laser joints was performed 110 to 130 MPa below the UTS of the welded joints.

It was observed that:

- The welding procedure affects the mechanical behavior of the welded plates; the higher the heat input, the more martensite is observed in the heat affected and fusion zone, and the higher the extension of these thermal affected regions.
- The extent of the heat affected zone and the fusion zone and, as a consequence, the existing martensite in these regions, affects the accumulated irrecoverable strain. The higher the extension of these regions (due to higher values of heat input), the higher the accumulated irrecoverable strain of the welds observed. Thus, a higher amount of martensite also corresponds to a higher amount of accumulated irrecoverable strain.
- Despite the presence of martensite in the heat affected zone and in the fusion zone, overall superelastic behavior of the welds was observed in all samples.
- Control of the process parameters (amount of heat input) needs to be taken into account in order to get the most adequate superelastic properties after welding. Lower heat input, thus with a lower extension of the heat affected zone and fusion zone, allows for a greater superelastic recovery, which is of significant importance for structural applications that require such functional property.

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