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Q1 Residual stress analysis in laser welded NiTi sheets using synchrotron X-ray diffraction

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ABSTRACT

Synchrotron radiation was used for fine probing the different regions of a laser welded NiTi joint. Measurements were taken at 0.2 mm intervals, starting in the non-thermal affected material, through the heat affected zone, the fusion zone and again across the heat affected zone finishing in the base material. Along the longitudinal direction the residual stresses were found to be always tensile in the heat affected and fusion zones; along the transversal direction, an inversion of the stress states was found. The magnitude of the residual stresses was found to be more significant in the transversal direction due to constraints imposed during welding. The heat introduced during welding has a stress relief effect on the cold-rolled base material, although it does not promote any solid-state transformation. The effect of the heat input on the residual stress pattern and magnitude was identified and it was seen that high heat input leads to a higher magnitude of the residual stresses in the weld and the stress relief effect is observed over a larger extension from the weld centerline. This paper presents the first experimental study on the determination of residual stresses on laser welded NiTi shape memory alloys.

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1. Introduction

NiTi shape memory alloys are stimulus-responsive materials which present both superelasticity and shape memory effect [1,2]. Laser welding of NiTi shape memory alloys has widespread rapidly over the last years. Several works report the effect of laser welding on the mechanical and functional properties of the welded joints [3–8]. It is a well-accepted fact that, after laser welding, the material presents a decrease in the mechanical properties when compared to the base material [4,9]. The presence of precipitates in the fusion zone, such as Ti₂Ni, is pointed out as a reason for the decreased ductility of the welded material [9,10].

In fusion based welding processes, such as laser, the gradient of peak temperature followed by a variation in heating and cooling rates along a weld induces residual stresses in the weld region and its neighborhood [11,12]. The welding residual stress field depends on a variety of factors such as: material properties, welding process parameters, pre-heating temperature (if any) and dimensions of the structure to be welded as well as imposed external restrain conditions [13]. Residual stresses may give origin to distortions in the welded material, and these may cause premature failure during service [14,15].

Residual stresses can occur due to a structural mismatch or by an uneven distribution of nonelastic strains. Thermal stresses occur in welding due to a localization of the heating source which originates

non uniform temperature distributions in the weld itself and surrounding medium [16]. Additionally, the molten zone shrinks during solidification and this shrinkage is constrained by the surrounding cooler material [17]. If any phase transformations, with associated volume change, occur during cooling, it is possible to compensate the tensile contraction stresses that occur in the fusion zone, provided that the phase transformation is associated with a volume expansion and not contraction.

Considering a stress-free base material it is expected that, after welding, the heat affected zone presents compressive stresses, while in the fusion zone tensile stresses should be found. This can be explained based on the "Three-bar Arrangement Theory" [18]: consider three similar metal bars, all at room temperature, connected by two rigid blocks. The bar located in the middle is heated up. However, its thermal expansion is restrained by the other two side bars. As such, compressive stresses are developed in the middle bar (while the side bars are in tension) and increase with increasing temperature. When heating stops and the middle bar is allowed to cool down, its thermal contraction is restrained again by the side bars. Then, the stress state is reversed: the middle bar is under tensile stress and the side bars under compressive stresses. Comparing to the different regions of the welded material, the middle bar acts as the fusion zone and the side bars as the heat affected zone. Fig. 1 depicts the expected residual stresses in these regions of the weld. In the non-thermally affected base material the development of residual stresses is not expected.

It must be noticed that residual stresses are always present after welding. In case of large structures post-weld heat treatments are

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Fig. 1. Expected residual stresses in the heat affected and fusion zones after welding.

Fig. 2. DSC measurement of base material.

performed so that the yield stress decreases and the residual stresses left on the structure are of the order of magnitude of the yield strength of the material at the temperature of the heat treatment, decreasing their magnitude and increasing the joints mechanical performance [19,20].

Measuring residual stresses by X-ray diffraction methods is a precision reliable technique. These measurements can be performed either with laboratorial sources (low energy X-rays) or in dedicated facilities, known as storage rings, with high energy X-rays. The former are used to determine the residual stresses in a restrict thickness of the welded samples due to its low energy and, hence, its low material penetration. In opposition, in synchrotron facilities the same analysis can be performed throughout the material, as its energy is considerably higher (up to 300 keV). The use of these hard X-ray sources allows for a greater penetration in the analyzed samples, thus it is possible to work in transmission mode even when using thick samples (up to 10 mm, for example). Other advantages of the use of synchrotron radiation include the very fast acquisition time (below the millisecond range if necessary) and the ability to decrease the analyzed spot size while keeping an intense beam (high photon flux). The capabilities of using synchrotron radiation, for fine probing of materials joining was, recently, observed by Cavaleiro et al. [21] in self propagated diffusion joining of NiTi.

Although it is usual to analyze the effect of the welding process on the residual stresses of the welded joints [22–24], no such work was yet performed in laser welded NiTi shape memory alloys. As such, there is a gap in research that needs to be filled for this specific class of functional materials. Due to the intrinsic characteristics of laser welding (high energy density with reduced width of the thermally affected zones) analysis of the different regions by X-ray diffraction requires precise control of the beam dimension and its positioning in the weld. Synchrotron X-ray diffraction was carried out for such analysis, as the radiation beam is very small, has a high photon flux, and can be precisely directed to the different regions of the weld.

In this work, X-ray synchrotron radiation was used to probe laser welded NiTi samples from the base material to the fusion zone using a beam with a cross section of $200 \times 200 \mu\text{m}$. The calculation of the residual stresses was performed using Rietveld analysis with MAUD.

2. Experimental procedure

Plates 1 mm thick of 50.8Ni-Ti (at.%) shape memory alloy, supplied by Memry in the flat annealed condition, were used. To characterize the structural transformation temperature of the base material, Differential Scanning Calorimetry (DSC) was used. The temperature range was set between -160 and 70°C . Both cooling and heating rates were set at 10 K/min . From these measurements it was confirmed that the base material was fully austenitic at room temperature (Fig. 2).

A Nd:YAG laser, operating in continuous wave mode, from Rofin Sinar was used for laser butt joining the plates. Prior to welding, the samples

were cut using a precision cutting machine into $30 \times 30 \text{ sq mm}$ and cleaned with alcohol and acetone. Two welding conditions were selected and are presented in Table 1. These parameters were chosen in order to obtain full penetration joints, without defects, but with significantly different heat inputs. Argon and helium were used as shielding gases on the face and on the root, respectively, to prevent oxidation.

Microstructure of the welded joints (Fig. 3) revealed that heat affected zone was constituted by coarser equiaxed grains when compared to the base material. From the favorable oriented grains in the heat affected zone, columnar epitaxial grains grew in the fusion zone.

X-ray diffraction was performed at beamline P07 High Energy Materials Science (HEMS) of Petra III/DESY, using a wavelength of $0.1426 \text{ \AA}$ (87 keV) and a 2D detector Mar345 placed at 1.35 m from the sample. The pixel size of the detector was of $200 \times 200 \mu\text{m}$ and the corresponding accuracy of the measured 2θ angle was of 0.0084° . A probing line, perpendicular to the weld bead, started and ended in the base material, 3 mm away from the weld centerline, passing through the heat affected zone and the fusion zone.

The experimental setup used during the X-ray diffraction experiments is depicted in Fig. 4. Based on the sample orientation in terms of laboratory referential (Fig. 4), the azimuthal angle, φ , is defined as equal to 0° in the longitudinal direction of the weld bead and equal to 90° for the transversal direction (perpendicular to the weld bead).

Throughout the experiments, performed at room temperature, complete Debye-Scherrer diffraction rings were obtained (Fig. 5).

For residual stress analysis, Rietveld code implemented in MAUD [25] was used following a similar procedure as described in [26]. During Rietveld refinement, parameters such as background, intensity, profile shape, unit cell, texture and stress model were refined. For the Rietveld analysis, four austenite peaks were considered: (100), (110), (111) and (200). When both austenite and martensite were present, five additional martensite peaks were considered: (101), (110), ($1\bar{1}1$), (020) and (211). These peaks used for Rietveld analysis are shown in Fig. 6. During the Rietveld analysis performed in MAUD, integration in 5° steps along the azimuthal angle was performed. The mechanical properties of both phases used in the stress model are described in [27,28].

Table 1
Laser welding parameters.

Sample	Power [W]	Welding speed [mm/s]	Heat input [J/cm]
A	990	25	396
B	1485	20	743

Fig. 3. Microstructure of the welded joints: a) sample A; b) sample B.

Following other authors [29] that have made similar assumptions when performing residual stress analysis using X-ray diffraction, a plane-stress condition was assumed owing to the reduced thickness of the material (1 mm).

According to the configuration used during X-ray diffraction experiments (Fig. 4), the principal directions of the system are:

- x component, along the longitudinal direction to the weld bead;
- y component, perpendicular to the weld bead;
- z component, normal to the material surface.

As a plane-stress condition is assumed, the principal stress along the principal direction z, σ_z , will be null, while the remaining

principal stresses, σ_x and σ_y , can be calculated through Rietveld refinement.

3. Results and discussion

Fit2D [30] was used to analyze the diffraction images in the 2θ range from 3.4 to 4.2° . For obtaining the X-ray diffraction patterns correspondent to each analyzed spot, the raw diffraction images were integrated along the full azimuthal angle ϕ . As such, it was possible to perform a microstructural characterization of the existing phases in the different regions of the welds. Fig. 7 depicts the superimposition of the diffractograms obtained for samples A and B. It is observed, that, in the former, the base material was fully austenitic, but the heat affected and the fusion zones showed austenite and martensite, the latter being present at room temperature due to the welding thermal cycle. Sample B had also austenite and martensite in the thermally affected zones, but the extension of each region was slightly larger due to the higher heat input introduced in this weld.

The variation of the 2θ peak position of (110) austenite peak, which is the most intense for the NiTi system, in welds A and B, in three distinct regions of the weld (base material, heat affected and fusion zones) is depicted in Fig. 8. Fig. 8a and d, correspond to the base material and show that the 2θ position of the (110) austenite peak does not vary significantly along the azimuthal angle. However, in the heat affected zone (Fig. 8b and e) the variation is more notorious and is impaired in the fusion zone (Fig. 8c and f). It must be noticed that in the thermally affected zones a coarse grain structure is observed due to grain growth at high temperature. These coarse grains cause high intensity peaks.

After Rietveld refinement, good agreement in position, width and intensity between the calculated and experimental patterns was observed (Fig. 9), indicating a good fitting.

The calculated principal residual stresses, σ_x and σ_y , for the welded samples are depicted in Fig. 10.

Considering both samples, the σ_x residual stress in the heat affected zone range from roughly 10 to 105 MPa, while in the fusion zone the residual stresses reach up to 135 MPa for the sample welded with the highest heat input. It can be observed an inversion of the stress state when approaching the fusion zone from the heat affected zone: the σ_y residual stresses are always compressive in the heat affected zone and range between 20 and 60 MPa in the fusion zone.

Fig. 4. Experimental setup for the X-ray diffraction experiments (not to scale): 3D schematic view.

Fig. 5. Typical Debye-Scherrer rings for laser welded NiTi: a) base material, b) heat affected zone, c) fusion zone.

The inversion of the residual stress conditions in the principal directions, measured in the heat affected and fusion zones was also reported for other materials, such as steels [31]. The base material presented also residual stresses originated during its manufacturing process. It can also be observed that the distribution of the residual stresses in the material is symmetric relative to the weld centerline axis, which is characteristic of laser welding with a top hat or Gaussian beam profiles.

Comparing the residual stresses along the principal directions for the same welded sample, it can be observed that the absolute value of the residual stresses is higher along the y axis than along the x axis. This is due to the fact that, during welding, a constraint was applied along this direction in order to guarantee a good fit-up throughout the full weld length. As such, the material was not able to move freely, generating residual thermal stresses along this direction.

Of particular interest is the stress relief phenomenon observed for the principal stress σ_y in the base material, marked in Fig. 10. Similarly,

along the other principal direction (x axis), the same feature is observed. The reason for this behavior is related to the fact that, along the transversal direction (y axis), there is a decrease in the tensile stresses in the work-hardened base material. Since no distortions were observed after welding, this means that an overall equilibrium was achieved, justifying the inversion of the stress state in the base material along the longitudinal direction. In the base material, the occurrence of microstructural modifications was not expected as the maximum temperatures reached are below those needed to induce any precipitation and/or recrystallization phenomena. This stress relief of the work-hardened base material occurs in the regions that have been heated up to temperatures below 350 °C. As a consequence, the residual stresses magnitude in this region decreases due to the fusion welding process, reverting the effect of cold working without changing the grain structure of the material [32]. In the regions heated above 350 °C, Ni-rich precipitates, such as Ni_4Ti_3 , may be formed for time

Fig. 6. Austenite (A) and martensite (M) peaks considered for Rietveld refinement: a) typical diffractogram from the base material; b) typical diffractogram from the heat affected and fusion zones.

Fig. 7. 3D stacking of the diffractograms performed on laser welds; a) sample A; b) sample B.

lapses in the order of a few seconds [33], increasing the transformation temperatures, thus leading to the presence of martensite at room temperature.

In order to understand the feasibility of the welding procedure to promote the aforementioned stress relief phenomenon, the peak temperatures in the base material were computed based on the 2D solution of the Rosenthal equation:

$$T - T_0 = \frac{Q}{2\pi k g} e^{(v x/2\alpha)} K_0 \left[\frac{v R}{2\alpha} \right]$$

where:

T , is the peak temperature at a distance x in the (x, y) plane (K);

T_0 , is the room temperature (K);

Q , is the heat input (J/m).

k , is the thermal conductivity ($\text{W m}^{-1} \text{K}^{-1}$);

g , is the plate thickness (m);

v , is the welding speed (m s^{-1});

α , is the thermal diffusivity ($\text{m}^2 \text{s}^{-1}$);

K_0 , is a modified Bessel function of the second kind and zero order; R , is the radial distance ($R = \sqrt{x^2 + y^2}$) (m).

Both x and y directions were defined as in Fig. 4. Fig. 11, depicts the peak temperature variation as a function of the distance to the weld centerline along the y direction for welds A and B. It can be observed, that the distance from the weld centerline at which the temperature range is between 200 and 350 °C (for promoting the stress relief phenomenon), is similar to those obtained from the X-ray diffraction measurements, thus validating this assumption.

The effect of the higher heat input in sample B, gives rise to a higher magnitude of the residual stresses in the different regions of the weld (heat affected zone and fusion zone), in either the principal directions, when compared to sample A. A similar effect of the heat input on the magnitude of the residual stresses was observed by other authors [34]. This can be explained using the “Three-bar Arrangement Theory” described in the introduction.

Another interesting consequence of the higher heat input introduced in sample B is that the stress relieving phenomena in the base material occurs at a longer distance from the weld centerline, when

Fig. 8. Variation of the 2θ peak position of the (110) austenite peak along the azimuthal angle, φ , sample A (top) and B (bottom). a) and d) correspond to the base material; b) and e) to the heat affected zone and c) and f) to the fusion zone.

Fig. 9. Calculated and measured diffraction patterns: a) in the base material where only austenite (A) exists; b) in the fusion zone where both austenite and martensite (M) are present.

Fig. 11. Peak temperatures, from the weld centerline along the y axis, at a fixed distance of $x = 1$ mm from the weld centerline. The black box represents the temperature range where stress relief phenomenon may occur.

the remaining regions of the welded material the von Mises stresses are below this range.

The residual stresses were also measured for the martensite in both the heat affected and fusion zones, where this phase was observed. However, the residual stresses calculated for this phase ranged between –6 and 8 MPa, which can be negligible considering the magnitude of the residual stresses found for the austenitic phase. The reason for this to occur is related to the mechanical properties of austenitic and martensitic NiTi. The former has a higher Young's modulus than the latter. As such, the load will be predominantly transferred to the "harder" material, which is austenite in this case. Additionally, martensitic NiTi has a lower constant stress plateau than austenitic NiTi, which enables stress relaxation of the neighboring austenite. Similar behavior was also reported to occur for Cu-based shape memory alloys [37].

No distortions were observed after butt welding the NiTi plates. This is related to the fact that the global residual stresses along the material are in equilibrium.

It is possible to alter the residual stresses within a welded joint by applying proper post-weld heat treatments. The effect of different post-weld heat treatments on the distribution of the residual stresses in laser welded NiTi joints is currently under study.

4. Conclusions

The first experimental study for the determination of residual stress in laser welded NiTi shape memory alloys is presented. For this,

Fig. 10. Calculated residual stress for welds A and B: a) σ_x , b) σ_y .

Fig. 12. von Mises stresses along the welded materials.

synchrotron radiation based X-ray diffraction and this data was exploited with Rietveld analysis. The following can be concluded:

- The principal stresses along the x axis were found to fall within the range of 10 to 105 MPa in the heat affected zone, while in the fusion zone they increase up to 135 MPa. The σ_y residual stresses were found to be always compressive in the heat affected zone, while in the fusion zone there is a tensile stress state with a magnitude ranging from 20 to 60 MPa.
- The inversion of the stress states in the different regions of the weld (heat affected and fusion zones) is in good agreement with general welding thermo-mechanical analysis.
- The heat input introduced in the welds promoted stress relief phenomena on the work-hardened base material. As such, the residual stresses in this region decreased.
- Stress relieving phenomena on the base material occur at temperatures below 350 °C.
- Higher heat inputs originate higher magnitude of the residual stresses, in absolute modulus, in the heat affected and fusion zones.
- No distortions were observed after welding and this results from an overall near equilibrium stress state.

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