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Effect of laser welding parameters on the austenite and martensite phase fractions of NiTi

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article info abstract

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Although laser welding is probably the most used joining technique for NiTi shape memory alloys there is still a lack of understanding about the effects of laser welding parameters on the microstructural induced changes: in both the heat affected and fusion zones martensite may be present, while the base material is fully austenitic. Synchrotron X-ray diffraction was used for fine probing laser welded NiTi joints. Through Rietveld refinement the martensite and austenite phase fractions were determined and it was observed that the martensite content increases towards the weld centreline. This is related to a change of the local transformation temperatures on these regions, which occurs due to compositional variation in those regions. The martensite phase fraction in the thermally affected regions may have significant implications on functional properties on these joints.

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1. Introduction

In order to obtain complex shaped structures using NiTi shape memory alloys joining methods are required owing to the poor machinability of these materials [\[1\].](#page--1-0) Among the different joining techniques available, fusion-based ones, such as tungsten inert gas (TIG) [\[2\]](#page--1-0) and laser welding [\[3](#page--1-0)–6], have presented the best results in terms of the mechanical properties of the joints. Laser welding is by far the most used process for welding NiTi due to the reduced extension of the thermally affected regions, which is especially important for NiTi due to the deterioration of its functional properties (shape memory effect and superelasticity) at high temperatures.

It was observed that laser welding is responsible for a change of the local chemical composition, thus changing the transformation temperatures of those thermally affected regions. Namely, an originally fully austenitic base material can present either martensite or a mixture of both martensite and austenite in those regions after welding.

Conventional analytical techniques for determining the phase transformation characteristics of NiTi shape memory alloys are differential scanning calorimetry (DSC) [\[7,8\]](#page--1-0) and X-ray diffraction [\[9,10\]](#page--1-0) using conventional lab sources. However, these techniques are not suited to provide the spatial resolution required for an in-depth understanding of the microstructural changes occurring along both the heat affected and fusion zones due to the laser welding procedure. While for DSC it is not

Corresponding author. E-mail address: jp.oliveira@campus.fct.unl.pt (J.P. Oliveira). possible to ensure that only the heat affected or fusion zone are being analysed [\[6\],](#page--1-0) these regions tend also to present a microstructural gradient thus preventing a reliable analysis of the phase transformation characteristics. Additionally, for X-ray diffraction analysis using conventional lab sources, it is known that in the Bragg-Brentano geometry, often used for such analysis, does not ensure the required spatial resolution for detailed analysis of the thermally affected regions.

A possible way to surpass this setback is with the use of high energy synchrotron-based X-ray diffraction operating in transmission mode. Owing to the high photon flux of these sources, the beam spot size can be very narrow, while still providing good statistics [\[11\].](#page--1-0) Additionally, it is possible to obtain precise control of the beam position, ensuring that the analysed region corresponds entirely to a given region. If necessary, it is still possible to probe the material within these thermally affected regions, in order to determine the existence of any microstructural gradient.

In this work, synchrotron X-ray diffraction was used for fine probing the different regions of laser welded NiTi joints. This is the first investigation addressing the effect of laser welding parameters on the phase content in the thermally affected regions of NiTi welds.

2. Experimental Procedure

Ni-rich NiTi shape memory alloys, 50.8 at% Ni, superelastic at room temperature, purchased from Memry in the flat annealed condition, were used in this investigation in the form of 1 mm thick plates. Differential scanning calorimetry of the base material was

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performed in a temperature range between -160 °C and 70 °C, at a heating/cooling rate of 10 °C/min, to determine its transformation temperatures. It was confirmed that, at room temperature, the material was fully austenitic.

Laser butt welding of 30×30 mm NiTi plates was performed using a Nd:YAG laser from Rofin Sinar, operating in continuous wave mode. The base material was cleaned using alcohol and acetone, prior to welding, to remove impurities. Two distinct welding conditions (varying power, P, and welding speed, v) were selected in order to ensure that full penetration was achieved, with no microstructural defects, such as porosity or lack of penetration. The selected welding parameters were: a) $P = 990$ W and v = 25 mm/s, for sample A; b) $P = 1485$ W and $v = 20$ mm/s, for sample B. So, it was ensured that significantly different heat inputs (HI), which is defined as the power (P) to welding speed (v) ratio (P/v), allowed obtaining defect-free joints. Shielding gases were used to prevent oxidation: argon on the face and helium on the root. The flow rate of the protection gases was set at 40 l/min for Ar and 50 l/ min for He.

Microstructural characterization by means of X-ray diffraction analysis using synchrotron radiation was performed at beamline P07 High Energy Materials Science of Petra III/DESY. A high energy beam of 87 keV (corresponding to a wavelength of 0.1426 Å) was used and the sample to the 2D detector Mar345 was set at 1.35 m. The beam spot was a 200×200 µm. The samples were analysed perpendicularly to the weld bead, starting in the base material, going through the heat affected and fusion zones and finishing on the other side on the base material. A length of nearly 6 mm was probed with a distance of 200 μm between two adjacent analysed spots. All X-ray diffraction analysis was performed at room temperature.

The raw 2D images were treated using Fit2D [\[12\]](#page--1-0) as described in [\[13\]](#page--1-0). Determination of the phase fraction of the existing phases at each analysed spot was performed using Rietveld code available in Material Analysis Using Diffraction (MAUD) program [\[14\].](#page--1-0)

3. Results and Discussion

The X-ray diffractograms of samples A and B at room temperature exhibit similar information: while the base material is fully austenitic, the thermally affected regions present a mixture of both austenite and martensite (Fig. 1). The formation of this thermally stable martensite occurs by two distinct reasons:

- (i) in the heat affected zone, $Ni₄Ti₃$ precipitation increases the transformation temperatures allowing for martensite to be stable at room temperature [\[15,16\]](#page--1-0),
- (ii) in the fusion zone, preferential Ni volatilization has the same effect of increasing the transformation temperature in this region [\[15,17\]](#page--1-0).

Fig. 1. X-ray diffraction patterns of samples A (a) and B (b).

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