IN SITU STRUCTURAL CHARACTERIZATION OF FUNCTIONALLY GRADED Ni-Ti SHAPE MEMORY ALLOY DURING TENSILE LOADING

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ABSTRACT

A functionally graded NiTi shape memory alloy wire was investigated by in-situ synchrotron radiation-based x-ray diffraction during cyclic tensile deformation. The transformation temperatures were determined by DSC and the thermomechanical behaviour was analysed by three-point bending. The present study focussed on the localized heat treatment (Joule heat effect, reaching 300°C, 350 and 400°C pulses for 10 minutes) of NiTi wires, using an equipment that allows a large variety of localized heat treatments. Structural, mechanical and thermomechanical characterization is presented in order to get a perspective of the optimization parameters for the adequate graded functionality. Experimental results put in evidence the structural gradient resulting from the localized heat treatments. The analysis of the in-situ X-ray diffraction (XRD) provide detailed information about the phase transformations in different regions of the wire, at different steps of the cyclic load/unload, giving a better understanding of the overall mechanical behaviour of a functionally graded material.

KEYWORDS: Shape Memory Alloy; X-ray diffraction; Synchrotron; Functionally graded material.

1. INTRODUCTION

Shape memory alloys (SMA) present interesting functional characteristics (shape memory effect and superelasticity) that make them quite attractive for a wide range of applications. These functional characteristics are a consequence of phase transformations that take place within well-defined temperature ranges or stress ranges, depending on being thermal or stress-induced. These temperature/stress ranges are a function of chemical composition and heat treatment of the material. For applications requiring a wider controllable range, a wider temperature/stress range than that associated to a specific composition/heat treatment may be required. In such a situation, the possible solution will be to use a functionally graded material. Functional gradient may be introduced by: (i) a geometrical gradient (variable cross-section along a specific direction [Shariat 2013]), (ii) a chemical composition gradient (either along the longitudinal direction [Shariat 2012], or along the thickness [Martins 2009]), (iii) a graded heat treatment [Meng 2016], or (iv)

may indirectly arise from a processing technique, such as laser welding [Oliveira 2016].

The aim of the present study is:

- to perform a parallel study of the mechanical behaviour under tensile test,
- detailed study of the local phase transformations during deformation (tensile and torsion) and correlate them to the global mechanical behaviour of the functionally graded wire,
- to analyse the effect of controlled localized heat treatment for applications where a functional gradient will be very attractive, such as orthodontic archwires, differentiating the actuation force between the front (lighter force) and the back (higher force) or as reinforcement actuators for surface morphing.

2. EXPERIMENTAL METHODS

The material used in this study was NiTi wire 0.4 mm diameter (supplied by Euroflex), with localized heat treatment at 300, 350 and 400°C. The localized heat treatment (Joule heat effect) of NiTi wires, used an equipment developed by Telmo Santos at UNIDEMI [Rodrigues 2017], which allows a well-controlled large variety of graded treatments by adjusting with a great flexibility several parameters:

(i) current intensity (programmable as a function of time and position),

(ii) velocity of the electrodes and their position along time.

The in situ study of the mechanical behaviour used a tensile test rig (20 kN load cell), mounted on a heavy load hexapod. XRD data was acquired (2D Perkin-Elmer fast detector XRD 1622) in transmission mode using a beam spot of 0.5 x 0.5 mm² with energy 100 keV and a.

Before running the tensile tests (Fig. 1-a), a scan of each wire along its gauge length was performed to identify the graded structure (Fig. 1-b). Two types of tensile tests were carried on:

(i) a series of superelastic cycles (continuous load/unload at 0.01mm/s up to 8% maximum strain), with the X-ray beam always hitting at the centre of the wire gauge length (Fig. 1-c and contour plots 1 to 8),

(ii) one superelastic cycle, stopping at predetermined values of the strain, where the full gauge length was scanned.

Figure 1: Localized heat treatment at 350°C during 10 min: (a) one full superelastic cycle (load/unload); (b) 3D plot of the scan along the gauge length before tensile test; (c) 3D plot of the structural evolution during one full superelastic cycle for the point at the centre of the gauge length; (d) irrecoverable strain and specific energy absorbed along 40 successive superelastic cycles. At the 2nd and 3rd rows: contour plots for 8 different points along the superelastic cycle ($3^\circ < 2\theta < 5^\circ$; azimuthal angle between 0° and 360°), 2θ scans for azimuths 0° and 90° (top of each contour plot) and maximum intensity for B2 (110) and B2 (200) versus azimuthal angle (at right of each contour plot).

3. RESULTS

The Debye-Scherrer rings were analysed for their intensity distribution: azimuthally, radially (to identify the peak shift). This information was combined with that from the precipitation phenomena (mostly Ni_4Ti_3) that took place in each region during the graded heat treatment and the corresponding transformation temperatures (determined by DSC) of the different segments of the wires. The irrecoverable strain and the specific energy absorbed at each load/unload cycle was evaluated (Fig. 1-d).

4. CONCLUSION

The in situ XRD measurements provide information about the nucleation of martensite during tensile deformation and its correlation with the specific graded treatment. The role of localized heat treatments will be highlighted in order to get the best combination of overall mechanical behaviour and graded functionality.

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