Materials Science & Engineering A 726 (2018) 102–112

Contents lists available at ScienceDirect

Materials Science & Engineering A

journal homepage: www.elsevier.com/locate/msea

A digital image correlation study of a NiTi alloy subjected to monotonic uniaxial and cyclic loading-unloading in tension

Xiaohui Bian[a,b,*], Ahmed A. Saleh[a], Elena V. Pereloma[a,b], Christopher H.J. Davies[c], Azdiar A. Gazder[b]

[a] School of Mechanical, Materials, Mechatronic and Biomedical Engineering, University of Wollongong, New South Wales 2522, Australia
[b] Electron Microscopy Centre, University of Wollongong, New South Wales 2500, Australia
[c] Department of Mechanical and Aerospace Engineering, Monash University, Victoria 3800, Australia

ARTICLE INFO

Keywords:
NiTi
Shape memory alloys
Digital image correlation
Tension
Martensite
Electron back-scattering diffraction

ABSTRACT

This digital image correlation study details the mechanical behaviour and pattern evolution of the transformation band of a 56Ni-44Ti wt% shape memory alloy subjected to monotonic uniaxial and loading-unloading cycles in tension. The broadened single inclined band front and multiple criss-crossing patterns are found to relieve the in-plane moment caused by local shear strains and straighten the sample edges during testing. The magnitude of the maximum local strain rate suggests its feasibility to understand the direction and extent of the localised transformation. Specifically, the changes to the maximum local strain rate during monotonic uniaxial tension are generally analogous to the stages in the macroscopic stress-strain curve. The microstructure before and after mechanical testing was characterised via electron back-scattering diffraction. Estimates of the kernel average misorientation show that residual strains upon unloading are linked to high intragranular misorientation within the original B2 grains and the remnant B19′ variants.

1. Introduction

NiTi shape memory alloys subjected to monotonic uniaxial tension at low initial strain rates (between ~10^{-3} and ~10^{-4} s^{-1}) typically present with macroscopic stress-strain curves comprising an initial elastic region, a stress plateau region (between ~0.01–0.12 engineering strains) followed by a rising stress region up to a maximum stress value [1–6]. Superelasticity is the ability of NiTi alloys to sustain repeated loading and unloading cycles within the stress plateau region; on account of the initial primitive cubic B2 (or austenite) phase undergoing a *fully* reversible, stress-induced transformation to monoclinic B19′ (or martensite). Consequently, the study of martensitic transformation within the macroscopic stress plateau region necessitates employing in-situ techniques.

One of the effective techniques typically combined with mechanical testing is digital image correlation (DIC), which is an optical correlation technique that records and measures the displacement of random speckle patterns painted along the gauge length, in order to compute the evolution of surface strain during loading [7,8]. Daly et al. [1] undertook the first in-situ DIC study of rolled, thin sheets of fully austenitic 52Ni-48Ti wt% alloy to quantify the evolution of localised strain fields up to the end of the macroscopic stress plateau region for one loading-unloading cycle in tension.

Since then, researchers have routinely applied DIC to characterise strain fields during the mechanical testing of thermo-mechanically processed NiTi alloys [3,9–14]. For example, DIC investigations revealed the development of a region of strain concentration upon deviation from linearity towards the end of the elastic region; suggesting the initiation of phase transformation just prior to the onset of the macroscopic stress plateau [1]. Thereafter, further localisation in the strain fields results in Lüders/transformation band(s) that propagate along the gauge length with higher macroscopic tensile strains [1,3,11,12,14–16]. The bands are typically inclined at ~60° with respect to the loading direction [12,17–21] and divide the gauge length into domains with varying local axial strain extrema comprising fully martensitic and as-yet untransformed B2 regions [11,12,14,15]. The axial strain values within the mobile band fronts do not saturate but record a continuous increase with higher macroscopic tensile strains [1,14].

While a single band propagates along the gauge length during monotonic uniaxial tension, multiple criss-crossing bands develop during both cyclic loading-unloading in tension and fatigue tests [1,9,11,13,15,19]. However, the mechanism behind the forward propagation of the mobile front of single bands along the gauge length and

* Corresponding author at: School of Mechanical, Materials, Mechatronic and Biomedical Engineering, University of Wollongong, New South Wales 2522, Australia.
E-mail address: xb124@uowmail.edu.au (X. Bian).

https://doi.org/10.1016/j.msea.2018.04.081
Received 15 March 2018; Received in revised form 18 April 2018; Accepted 19 April 2018
Available online 21 April 2018
0921-5093/

the switching from single to multiple criss-crossing bands is unclear. To-date, a couple of studies have suggested that shear strains lead to an in-plane moment, which in turn may influence the properties of the mobile band front [16,19].

Fatigue testing of NiTi alloys typically involves repeated cycles of loading up to the end of the macroscopic stress plateau region and then unloading back to zero load. Such investigations reported increases in the irreversible strain component (through highly accumulated dislocation densities and the formation of stable martensite variants with successive cycles) contributing to an increase in the residual strain upon unloading and a progressive degradation in superelasticity involving the retention of an increasing phase fraction of B19′ [14,15,22–29].

However, the typical fatigue test as described above is markedly different from loading-unloading cycles, involving small increments to the macroscopic strain per cycle, within and up to the end of the macroscopic stress plateau region. Although the latter type of mechanical tests are less frequently reported, it is necessary to enable: (i) an understanding of localised deformation accumulation through axial/transverse/shear strain pattern evolution via incremental increases to the macroscopic strain and, (ii) studying the degradation in superelasticity as a function of the overall stress versus strain response with successive cycles.

While the changes in the DIC-based axial strains during mechanical testing have been catalogued, the local strain rate, which affects the direction and extent of phase transformation within the macroscopic stress plateau region, has only been reported for a select macroscopic tensile strain [3]. It was found that the local strain rate is positive only within the narrow propagating band front whereas it is close to zero elsewhere along the gauge length [3]; indicating that phase transformation is confined within the band front area. In addition, the maximum local strain rate for the select time step is approximately an order of magnitude higher than the macroscopic strain rate, which suggests its feasibility as a descriptive indicator of the rate of localised transformation occurring at each time step [3].

With the above outlook in mind, the present DIC study aims to detail the mechanical behaviour of a NiTi shape memory alloy subjected to monotonic uniaxial and loading-unloading cycles in tension. For the latter test type, loading-unloading cycles are undertaken with small increments to the macroscopic tensile strain per cycle, within and up to the end of the macroscopic stress plateau region. In particular, a systematic description of the mechanisms of single transformation band front broadening and criss-crossing patterns is provided. Furthermore, the evolution of maximum local strain rate with macroscopic strain is explored. Microstructure comparisons before and after mechanical testing were undertaken via electron back-scattering diffraction (EBSD).

2. Experimental procedure

An as-received 56Ni-44Ti wt% rod produced by Nitinol Devices and Components Inc. was cut into smaller $\varnothing 10 \times 70$ mm^2 rods by electro-discharge machining. These small rods were encapsulated in a quartz tube under an Ar atmosphere, annealed at 700 °C for 120 s in a muffle furnace and immediately water quenched. The resultant microstructure comprised a single B2 phase with an average grain size of $\sim 10 \pm 5.9$ µm.

The annealed rods were electro-discharge machined into 18 (gauge length) $\times$ 5 (width) $\times$ 1 (thickness) mm^3 flat dog-bone samples with their gauge lengths parallel to the rod axial direction. For DIC measurements, a random speckle pattern was created by spraying a black background and white spots along the entire gauge length prior to tension.

Two types of mechanical tests involving monotonic uniaxial tension and cyclic loading-unloading were performed at room temperature using a computer-controlled servo-hydraulic 100 kN Instron 1341 universal testing machine operating at a crosshead speed of 0.48 mm•min^{-1}; which corresponds to an initial strain rate of 1×10^{-4} s^{-1}.

During uniaxial tension, the sample was monotonically loaded beyond the macroscopic stress plateau region, well into the slowly rising macroscopic stress region and then unloaded; in order to cover all characteristic deformation stages. Cyclic loading-unloading in tension was undertaken in 0.005 engineering strain increments per cycle, for a total of 20 cycles. The loading-unloading speeds were the same.

The force data from the load cell was transmitted as an analogue signal to the Instron and DIC computers; both of which were synchronised to record concurrently time-stamped stress readings and image frames. DIC data contained recording images of the random speckles painted along the gauge length on a Dantec Dynamics Q-400 system comprising two close circuit digital cameras operating at 5 frames per second for the duration of the mechanical tests.

Thereafter, an in-house developed Matlab script was used to compute the macroscopic engineering strain. The script identifies the grid locations of all speckles centroids along the gauge length span for the first image frame (used as the reference image) and then calculates their displacements in successive frames. For each successive frame, the average axial displacement was used to compute the macroscopic average engineering strain ($\bar{\varepsilon}$). The evolution in axial and shear strains along the entire gauge length is shown using contour maps computed using the Instra-4D software suite.

Microstructure characterisation was undertaken by polishing the flat dog-bone sample face up to the 1 µm diamond stage and then electro-polishing at room temperature using a Struers Lectropol-5 operating at 20 V (~1.8 A) for 180 s with an electrolyte mixture comprising 73% ethanol + 10% butoxyethanol + 9% water + 8% perchloric acid by volume.

EBSD was performed on a JEOL JSM-7001F field emission gun-scanning electron microscope operating at 15 kV, ~6.5 nA probe current 12 mm working distance and fitted with an Oxford Instruments Nordlys-II(S) camera interfacing with the AZtec acquisition software suite. A magnification of 150× and a step size of 0.5 µm was employed for the annealed sample whereas a magnification of 2000× and a step size of 0.03 µm was used for the two samples after mechanical testing.

The EBSD maps were post-processed using the Oxford Instruments Channel-5 software suite. The maps were cleaned by removing the wild spikes, cyclic extrapolation of zero solutions to five neighbours and thresholding the band contrast to delineate unindexed regions. In this study, phases are superimposed on the band contrast map such that the red, teal and white denote the B2 and B19′ phases and unindexed regions, respectively. For all calculations, misorientations between 2 and 15° and greater than 15° are classified as low (LAGBs, in silver) and high (HAGBs, in black) -angle boundaries, respectively. The kernel average misorientation (KAM) maps were computed using a 3×3 square filter and a critical subgrain angle of 2°.

3. Results

In this study, the macroscopic engineering stress versus strain responses are plotted and analysed, as they are commonly reported in the literature [1,3,13,30]. In the following paragraphs, all strains are axial unless explicitly stated otherwise. The macroscopic average axial engineering strain ($\bar{\varepsilon}$) is referred to as the macroscopic strain whereas the local axial engineering strains and strain rates along the gauge length are referred to as local strains and strain rates, respectively.

3.1. Monotonic uniaxial tension

Fig. 1a depicts the macroscopic engineering stress (σ) versus average strain ($\bar{\varepsilon}$) curve during monotonic uniaxial tension. For the points highlighted by red circles, the corresponding sequence of full-field DIC images with varying scale bars are given in Fig. 2. The numbers indicate characteristic stages at various macroscopic strain values whereas letters denote intermediate points between stages.

The present 56Ni-44Ti wt% alloy returned an apparent elastic