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An in-situ synchrotron study of the B2→B19' phase transformation in a Ni-Ti alloy subjected to uniaxial monotonic tension

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Abstract

A cold-drawn and annealed 56Ni-44Ti wt.% alloy was subjected to in-situ uniaxial monotonic tension in a synchrotron. Spatially resolved diffraction data was acquired along the gauge length by pausing the loading at five select macroscopic strains within the stress plateau region. This enables tracking localised transformation phenomena by sub-dividing the gauge length into transformation band, untransformed and apparently transformed regions. Within the macroscopic stress plateau region: (i) the highly strained B2 phase within the propagating transformation band and apparently transformed regions produces a relaxation of the B2 phase within the untransformed region. (ii) The newly formed B19' grain families exhibit a transition in *relative* lattice strain values from the transformation band through to the apparently transformed region. (iii) The $\langle 111 \rangle_{B2}$ fibre texture transforms to the $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$ and $[010]_{B19'}$ such that the latter fibres continue to record increases in maximum intensity up to maximum load. Within the slowly rising macroscopic stress region and beyond a critical stress value of ~426 MPa: (i) the *relative* lattice strains of the $(1\bar{2}0)_{B19'}$ and $(020)_{B19'}$ grain families deviate from linearity along the axial and transverse directions, respectively and, (ii) the anisotropy in crystallite size and micro-strains in all B19' grain families reduces markedly.

Keywords: NiTi; Shape memory alloy; Tension; Texture; Rietveld refinement; Synchrotron x-ray diffraction

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1. Introduction

In NiTi shape memory alloys, uniaxial monotonic tension at low initial strain rates (between $\sim 10^{-3}$ and $\sim 10^{-4}$ s $^{-1}$) typically results in a macroscopic stress-strain curve comprising an elastic region, a stress plateau region (between ~ 0.01 - 0.12 engineering strains) followed by a slowly rising stress region up to a maximum stress value [1-7]. Superelasticity of NiTi alloys refers to the ability to recover a large macroscopic strain and corresponding deformation under repeated loading-unloading cycles within the macroscopic stress plateau region. Such recoverable strains result from the reversible transformation between the primitive cubic B2 (nominally referred to as austenite) and the monoclinic B19' (or martensite) phases and the values are significantly higher than those of the classic metals or alloys [1, 8]. Consequently, the study of the B2 $\rightarrow$ B19' phase transformation within the macroscopic stress plateau region is best captured by employing a variety of in-situ techniques.

In-situ digital image correlation (DIC) studies of NiTi alloys subjected to uniaxial monotonic tension have revealed that upon yielding, the localised development of strain concentration occurs at one end of the parallel gauge length; marking the initiation of phase transformation prior to the onset of the macroscopic stress plateau region [1, 9]. Higher macroscopic tensile strains result in further strain field localisation that cause the Lüders/transformation band, typically inclined at $\sim 60^\circ$ with respect to the axial/loading direction [10-12], to propagate along the gauge length [1, 2, 4, 9, 10]. Within the macroscopic stress plateau region, the gauge length is sub-divided into three general regions. They comprise: (i) a narrow transformation band within which the B2 $\rightarrow$ B19' phase transformation is current, (ii) a region ahead of the transformation band comprising as-yet untransformed B2, and (iii) a region behind the transformation band where the B2 $\rightarrow$ B19' phase transformation is apparently complete. In the following paragraphs, the literature and the data recorded within the macroscopic stress plateau region are discussed based on these three regions.

In-situ diffraction studies involving laboratory X-ray, neutron, and synchrotron X-ray sources are routinely coupled with thermal and/or mechanical testing [3, 13-19]. In this regard, synchrotron sources have the following attributes: (i) the high beam energy facilitates lattice strain and phase fraction measurements over reasonably large gauge volumes. (ii) The high X-ray flux ensures reduced data collection times; thereby overcoming the macroscopic stress relaxation effect when loading is paused. (iii) 2D detector technology images the Debye-Scherrer rings and enables quantitative texture analysis via Rietveld refinement [20-23]. Here, texture analysis accounts for the variation in intensity along a Debye-Scherrer ring for a given hkl reflection as well as the intensity variations between different reflections [24-26].

Several in-situ synchrotron diffraction studies have investigated the B2 $\rightarrow$ B19' phase transformation during the uniaxial monotonic tension of NiTi. Raghunathan *et al.* [27] observed stress relaxation of the (110)_{B2} grain family upon transformation in a rolled 57.93Ni-42.07Ti (in wt.% from hereon) sheet. Schmahl *et al.* [28] and Polatidis *et al.* [16] studied cold-rolled 55.75Ni-44.25Ti and thermally cycled 55.36Ni-44.64Ti alloys and reported that within the transformation band, the

remnant¹ (110)_{B2} grain family is under larger strain compared to the untransformed region. Young *et al.* [14] studied the internal strains of the B2 and B19' phases of a drawn 55.95Ni-44.05Ti wire along the gauge length for a particular macroscopic strain within the stress plateau region. They found that the (001)_{B19'} grain family within the transformation band was under compression along the axial direction and under tension along the transverse direction.

During the loading stage of cyclic loading-unloading tensile tests, Cai *et al.* [29] noted opposite lattice strain evolution for the (110)_{B2} and (211)_{B2} grain families within the transformation band. Since both these grain families have an elastic anisotropy factor of 0.25, the variation in lattice strain was attributed to the B2↔B19' orientation relationship and the difference in elastic moduli between the corresponding B2 and B19' grain families. It was suggested that the highly strained B2 phase in the immediate vicinity of the mobile front of the transformation band facilitates further B2→B19' phase transformation by reducing the local critical stress for transformation [11, 14, 29]. It is pointed out that our knowledge on the strain evolution of the B2 and B19' phases during loading/unloading, the load partitioning between them and the correlation of these phenomena to the overall transformation mechanism is limited. For instance, in Ref. [14], the data points are limited and transformation to the R-phase also occurs; which further complicates the strain analysis of the B2 and B19' phases.

While the texture evolution of the B19' phase induced by deformation accommodation mechanisms such as slip, deformation twinning and de-twinning has been investigated [30-33], data on texture evolution during the B2→B19' phase transformation is limited. For example, Hasan *et al.* [15] reported on texture evolution during the uniaxial tension of a cold-rolled 55.8Ni-44.2Ti alloy with an initial B2 phase. With the rolling direction parallel to the axial/loading direction, (110)_{B2} transformed to (020)_{B19'} and the other B19' grain families rotated away from the axial direction. In Ref. [34], a cold-drawn 55.7Ni-44.3Ti bar with an initial (111)_{B2} B2 phase was reported but texture evolution during tensile loading was not studied. Cai *et al.* [35] performed Rietveld-based texture analysis of the Debye-Scherrer rings of a cold-drawn 56.1Ni-43.9Ti wire subjected to uniaxial monotonic tension and found that the initial [334]_{B2} fibre transforms to $\bar{1}30$ _{B19'}.

Most importantly, DIC analyses of NiTi alloys subjected to uniaxial monotonic tension routinely suggest that the heterogeneous nucleation of the B19' phase is local strain specific and is favoured by stress enhancement due to sample geometry. Consequently, phase transformation tends to initiate at the end of the parallel gauge length [1, 9, 10, 36-39]. However, the majority of in-situ neutron/synchrotron diffraction experiments to-date are based on measurements obtained solely from the centre of the gauge length. The combination of: (i) the localised nature of phase transformation, and (ii) the diffraction data obtained from the centre of the gauge length, does not reflect the overall micro-mechanical behaviour of the bulk sample. Thus, such diffraction data does not capture the onset of phase transformation or track the propagation of the transformation band [16]. Consequently, phase

¹ Throughout the text, the term “remnant” refers to the residual B2 phase in the transformation band and apparently transformed regions.

transformation analysis is only possible when the transformation band reaches the centre of the gauge length at relatively higher macroscopic strains.

With the above outlook in mind, this in-situ synchrotron study provides detail on the B2→B19' phase transformation in a cold-drawn and annealed 56Ni-44Ti alloy subjected to uniaxial monotonic tension. Spatially resolved diffraction data was acquired along the gauge length at five select macroscopic strains within the stress plateau region. By doing so, the onset, propagation, and completion of transformation was tracked in terms of: (i) the lattice strains of individual grain families for both phases, (ii) the phase fractions within the transformation band and in the untransformed and apparently transformed regions ahead of and behind the transformation band, respectively and (iii) the texture of the B19' phase.

2. Experimental and analytical procedures

A $\varnothing 10$ mm diameter 56Ni-44Ti polycrystalline rod sourced from Nitinol Devices and Components Inc. was cut into smaller $\varnothing 10 \times 42$ mm² rods that 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 10 (gauge length) $\times$ 5 (width) $\times$ 0.7 (thickness) mm³ tensile dog-bone samples with their gauge lengths parallel to the rod axial direction (Fig. 1a).

In-situ diffraction measurements were performed in transmission geometry on the Powder Diffraction beamline at the Australian Synchrotron. For a detailed description of in-situ loading experiments performed on the beamline, the reader is referred to Refs. [40, 41]. Diffraction data was acquired using 21 keV beam energy (wavelength 0.59041 Å) and a beam size of 1.0×0.5 mm² along the axial/loading (vertical) and transverse (horizontal) directions, respectively. The sample-to-detector distance was 415.261 mm and the exposure time for each measurement was 240 s. A Mar345 image plate detector with a 345 mm diameter and pixel size of 100 μm was used to record the Debye-Scherrer ring patterns. Prior to the monotonic tensile test, calibration was performed using a LaB₆ standard (NIST Standard Reference Material SRM-660). The diffraction geometry and the obtained image of the Debye-Scherrer rings are schematically shown in Fig. 1a for the end of the slowly rising macroscopic stress region (macroscopic strain (5) in Fig. 1b). On the image (detector) plane, azimuth angles (η) of 90° and 270° refer to the axial/loading direction while 0° and 180° refer to the transverse direction. The lattice strain sensitivity is estimated to be ± 150 $\mu\epsilon$ for these measurements.

(a)

(b)

Figure 1. (a) Schematic illustration of the tensile sample dimensions in mm and the synchrotron diffraction measurements in transmission mode. (b) The macroscopic stress-strain curve with characteristic transitions (numbers and letters) during uniaxial monotonic tension. Squares denote the positions at which diffraction measurements were performed. White - the elastic region, grey - the macroscopic stress plateau region, black - the slowly rising macroscopic stress region.

Uniaxial monotonic tensile testing was undertaken at room temperature on a Deben 5 kN tensile stage with the sample grips modified to suit synchrotron diffraction measurements in transmission mode. The macroscopic engineering strain was determined by correcting the extension data for compliance errors via application of the macroscopic elastic stress-strain data from prior digital image correlation measurements undertaken on an Instron universal testing machine [9, 42]. All loading was performed at a cross-head speed of $0.02 \text{ mm} \cdot \text{min}^{-1}$. By pausing at macroscopic strains of interest, diffraction data from the centre of the gauge length (0 mm, Fig. 1a) was collected throughout the experiment. In addition, spatially resolved diffraction data was also collected at nine locations along the gauge length (i.e. - the centre of the gauge length and four locations above and below it), for five select

macroscopic strains within the stress plateau region. This was acquired by pausing the test and translating the stage from -4 mm to +4 mm in steps of ± 1 mm as shown in Fig. 1a. In Fig. 1b, the intermediate macroscopic strains $(3a) = 0.0125$, $(3b) = 0.0202$, $(3c) = 0.0280$, $(3d) = 0.0384$ and $(3e) = 0.0481$ are the macroscopic strains at which diffraction data was collected along the gauge length. During data acquisition, the sample symmetry and the experimental setup result in a macroscopic stress relaxation within 5 MPa that is covered by the squares indicating the measurement points (Fig. 1b).

Fit-2D [43] was used for radial integration of the 2D diffraction ring patterns. The beam centre, sample-to-detector distance, and tilt of the detector (roll, pitch, and yaw) were calibrated using the recorded LaB_6 standard ring pattern. The acquired ring patterns were segmented into 5° slices. With each slice, an X-Y plot of 2θ versus total X-ray intensity was obtained. Thus, each integrated diffraction spectrum is indicative of the microscopic deformation behaviour within $\pm 2.5^\circ$ to the direction of interest (Fig. 1a).

For single peak fitting, diffraction spectra corresponding to the axial ($\eta = 90^\circ$) and transverse ($\eta = 180^\circ$) directions were fitted using a TOPAS refinement code [44]. The observed reflections were indexed for the B2 and B19' ($a < c < b$, $\gamma \neq 90^\circ$) phases (Fig. 1a). In the case of the B2 phase, only the $(110)_{\text{B2}}$ peak was fitted as the $(200)_{\text{B2}}$ reflection was too weak and consequently, excluded from the fitting. In doing so, the lattice strain, peak width (full width at half maximum, FWHM) of individual grain families, and phase volume fraction was extracted. For evaluating lattice strains (ε_{hkl}), the following equation is used:

$$\varepsilon_{hkl} = \frac{d^{hkl} - d_0^{hkl}}{d_0^{hkl}} \quad (1)$$

where, d^{hkl} is the lattice spacing at a given macroscopic strain. For the B2 phase, d_0^{hkl} is the reference lattice spacing at zero load before monotonic loading. As described in Ref. [17], the reference lattice spacings of the B19' grain families were selected when they first appeared along the gauge length at low macroscopic strains (in the present case, at macroscopic strains $(3a)$ or $(3b)$ as illustrated in Fig. 1b). It follows that for the B19' phase, *relative* lattice strains were calculated using Eq. (1). The integrated intensities from both axial and transverse directions were used to evaluate phase volume fractions (V) based on the following equations [45]:

$$\frac{\sum(I_{\text{B2}}^{hkl}/R_{\text{B2}}^{hkl})}{\sum(I_{\text{B19}'}^{hkl}/R_{\text{B19}'}^{hkl})} = \frac{V_{\text{B2}}}{V_{\text{B19}'}} \quad (2)$$

$$R = \left(\frac{1}{v^2}\right) \left[|F|^2 p \left(\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \right) \right] (e^{-2M}) \quad (3)$$

$$V_{\text{B2}} + V_{\text{B19}'} = 1 \quad (4)$$

where, I is the integrated intensity. The calculation of the R -value for each reflection involves v = unit cell volume, F = structure factor for a given hkl reflection, p = multiplicity factor, e^{-2M} = temperature

factor. Summing the integrated intensities of multiple B19' reflections reduces the possible effect of texture on the calculation of the phase fraction [45].

Rietveld analysis of the diffraction ring patterns was carried out using the Material Analysis Using Diffraction (MAUD) software suite. For texture analysis, it is known that the variation of intensity around the Debye-Scherrer ring is proportional to the pole intensity of the orientation sphere imposed on the sample. When viewed along the axial/loading direction, each Debye-Scherrer ring is projected as a pair of curved lines separated by a distance of 2θ on the $\{hkl\}$ pole figure [22]. From the literature [11, 34] and earlier electron back-scattering diffraction work [9, 46], we know that the material possesses an initial $\langle 111 \rangle_{B2}$ fibre texture along the axial/loading direction. Moreover, uniaxial tensile loading leads to the development of an axisymmetric fibre texture. Consequently, fibre symmetry was imposed to improve on the effective pole figure coverage. The tomography-based Extended Williams-Imhof-Matthies-Vinel (E-WIMV) approach [22, 24, 47], which refines orientation distributions via an iterative scheme, was applied to account for the incomplete coverage of the orientation distribution space.

For the analysis of crystallite size and micro-strain broadening, the Popa model [48, 49] was applied within MAUD. In this model, the anisotropic broadening of the diffracted peaks is derived according to Laue groups. For the current analysis, a harmonic order of 4 is sufficient to determine refinable parameters describing the structures of both phases; such that further refinement did not result in any significant improvement.

Stress analysis based on the azimuth angle (η) dependence of diffraction peak shifts was performed using the moment pole stress with the "BulkPathGEO" micro-mechanical model that takes crystallographic texture into account [24, 50, 51]. The elastic tensors for both phases were acquired from Refs. [52, 53]. The axial stress component (σ_{33}) (along the z-direction in Fig. 1a) is refined and is positive when the material is under tension. The applied stress model was used to fit the sinusoidal oscillations in d -spacing with respect to η (Fig. 2a). In this model, the symmetric stress tensor contains hydrostatic and deviatoric stress components. The hydrostatic stress component is generally large and is accounted for by refining the lattice parameters. On the other hand, the deviatoric stress component is small and is considered by assuming a linear stress-strain relationship for small strains [24]. Consequently, in the applied stress model, the deviatoric stress component is refined in order to account for the sinusoidal oscillations in d -spacings; effectively eliminating errors in fitting the Bragg peak and intensity maxima for all diffraction data of interest. Fig. 2a shows the representative experimental (bottom) and fitted (top) stack of diffraction spectra indicating reasonably good fits. Fig. 2b depicts representative diffraction spectra along the axial ($\eta = 90^\circ$) and transverse ($\eta = 180^\circ$) directions at macroscopic strain (3b) for the -4 mm gauge length position.

Figure 2. (a) A representative stack of the experimental (bottom) and fitted (top) diffraction spectra at (3b) and -4 mm gauge length position in Fig. 1, with ~5 vol.% of remnant B2 phase. Reference d -spacings are indicated by dashed lines. (b) Diffraction spectra along the axial ($\eta = 90^\circ$) and transverse ($\eta = 180^\circ$) directions for the same loading condition in (a).

3. Results

3.1 Localised B2→B19' phase transformation

Fig. 1b presents the typical macroscopic engineering stress (σ) versus strain (ϵ) response during in-situ uniaxial monotonic tensile testing, as commonly reported in the literature [1, 4, 5, 28, 32, 36, 54]. In the following paragraphs, the macroscopic axial engineering strain (ϵ) is referred to as the macroscopic strain. The numbers (1) to (5) mark the characteristic transitions along the macroscopic stress-strain curve. In-between these transitions, the combination of number and alphabet index denotes intermediate macroscopic strains of interest. In this regard, macroscopic strains (1) to (2) span from zero load, through the elastic region, up to ~410 MPa. The present 56Ni-44Ti alloy returned an apparent elastic modulus of 73.1 GPa for the B2 phase, which is within the 40-90 GPa range reported in the literature [14]. Macroscopic strains (2) to (3a) span from the end of the elastic region, through the stress drop, and up to the onset of the stress plateau region at ~360 MPa. Macroscopic strains (3a) to (4a) span the stress plateau region extending up to ~370 MPa. Finally, macroscopic strains (4a) to (5) account for the slowly rising stress region.

This section characterises the micro-mechanical behaviour of the B2 and B19' phases. Fig. 3 shows the lattice strain evolution of the $(110)_{B2}$ grain family as a function of the macroscopic stress and strain along the axial and transverse directions at the centre of the gauge length. Within the stress plateau region, the phase fraction, axial FWHM and lattice strains of the $(110)_{B2}$ grain family are studied by gathering spatially resolved diffraction data along the gauge length and graphing the values using contour plots. These parameters are discussed in terms of detected location within the transformation band, and in the untransformed and apparently transformed regions ahead of and behind the transformation band, respectively (Fig. 4).

Similarly, the *relative* lattice strain data for various B19' grain families obtained from the centre of the gauge length is shown in Fig. 5. This is supplemented by contour plots that illustrating the axial and transverse *relative* lattice strains for the $(020)_{B19'}$ and $(111)_{B19'}$ grain families within the macroscopic stress plateau region (Fig. 6).

By sampling in the above manner: (i) data from the centre of the gauge length is representative of the sample bulk for the elastic and slowly rising macroscopic stress regions, and (ii) the contour plots detail localised transformation phenomena and track the propagation of the transformation band along the gauge length within the macroscopic stress plateau region.

In Figs. 3a and 3b, the data recorded throughout the elastic region between macroscopic strains (1) and (2) was used to estimate the mechanical properties of the B2 phase. The slopes of macroscopic stress versus lattice strain along the axial (Ax) and transverse (Tr) directions returned Young's moduli of $E_{Ax} = 82$ GPa and $E_{Tr} = 140$ GPa and a Poisson's ratio $\nu = 0.59$ for the $(110)_{B2}$ grain family [14]. Plotting the lattice strain against macroscopic strain (Fig. 3b) showed that following the stress drop between macroscopic strains (2) and (3a), the onset of the stress plateau region coincides with relaxation. The latter is denoted by decreases in the axial and transverse lattice strains from $\sim 4700 \mu\epsilon$ to $\sim 4100 \mu\epsilon$ and from $\sim -2600 \mu\epsilon$ to $\sim -2200 \mu\epsilon$, respectively and marks the start of B2 $\rightarrow$ B19' phase transformation.

Figure 3. (a) The macroscopic stress versus lattice strain and (b) lattice strain versus macroscopic strain for the $(110)_{B2}$ grain family at the centre of the gauge length throughout the experiment and along the axial (Ax) and transverse (Tr) directions. White - the elastic region, grey - the macroscopic stress plateau region, black - the slowly rising macroscopic stress region.

The relaxation in the lattice strain between macroscopic strains (3a) and (3d) at the centre of the gauge length corresponds to the B2 phase in the untransformed region. It follows that the overall macroscopic stress plateau region is better understood with the aid of the spatially resolved in-situ measurements along the gauge length. As shown in Fig. 4a, the B19' phase appears at the bottom of the

gauge length by $\sim$ macroscopic strain (3a), steadily propagates to the centre by $\sim$ macroscopic strain (3e), and apparently consumes the entire gauge length by macroscopic strain (4a).

Between macroscopic strains (3a) and (3d), the $(110)_{B2}$ grain family within the transformation band records increases in FWHM from $\sim 0.06^\circ$ to $\sim 0.10^\circ$ (Fig. 4b) and lattice strain from $\sim 3900 \mu\epsilon$ to $\sim 5300 \mu\epsilon$ (Fig. 4c). In the apparently transformed region, these parameters are higher but approximately stable throughout for the ~ 5 vol.% of remnant B2 phase. It follows that during the propagation of the transformation band, the highly strained B2 phase within the transformation band and apparently transformed regions produces a relaxation of the B2 phase within the untransformed region. Consequently, the lattice strains at the centre of the gauge length between macroscopic strains (3b) and (3d) tend to cluster together and/or remain at approximately the same value (Fig. 3).

Once the transformation band reaches the centre of the gauge length by macroscopic strain (3e) and propagates past it by macroscopic strain (4a), the axial lattice strain of the remnant B2 phase records incremental increases up to $5200 \mu\epsilon$ (Fig. 3b). Beyond macroscopic strain (4b), the transverse diffraction signal is lost.

Figure 4. Contour plots showing (a) phase fraction, (b) axial FWHM and (c) lattice strain of the $(110)_{B2}$ grain family along the gauge length within the macroscopic stress plateau region.

Since *relative* lattice strains are computed for the $B19'$ phase (see Section 2), their evolution with respect to transformation is studied in Fig. 5 as a function of uniaxial, monotonic loading from macroscopic strains (3e) through (5).

Between macroscopic strains (3e) and (4a), the $(1\bar{2}0)_{B19'}$ and $(1\bar{1}1)_{B19'}$ grain families appear along the axial direction only whereas the $(002)_{B19'}$ grain family only appears along the transverse direction. The $(1\bar{2}0)_{B19'}$ grain family exhibits axial *relative* lattice strains that increase from 2200 to 4100 $\mu\epsilon$ whereas the $(002)_{B19'}$ grain family shows a decrease in transverse *relative* lattice strain from -1900 to -4300 $\mu\epsilon$. For the $(1\bar{1}1)_{B19'}$ grain family, the axial *relative* lattice strain is between $\sim$ -1100 and $\sim$ -900 $\mu\epsilon$. The $(020)_{B19'}$ and $(111)_{B19'}$ grain families appear along both axial and transverse directions. In this regard, the *relative* lattice strains of the $(020)_{B19'}$ grain family change from 550 to 1700 $\mu\epsilon$ along the axial direction and from 6200 to 5700 $\mu\epsilon$ along the transverse direction. Alternatively, the $(111)_{B19'}$

grain family records *relative* lattice strains of -1500 to -1200 $\mu\epsilon$ along the axial direction and 250 to -1200 $\mu\epsilon$ along the transverse direction.

Figure 5. The *relative* lattice strain versus the macroscopic strain evolution for the B19' grain families from the centre of the gauge length throughout the experiment. Axial direction (Ax) - solid lines, transverse direction (Tr) - dashed lines. Grey - the macroscopic stress plateau region, black - the slowly rising macroscopic stress region. The error bars are smaller than the symbol size and are in the same order.

The contour plots in Fig. 6 depict the evolution of the axial and transverse *relative* lattice strains for the $(020)_{B19'}$ and $(111)_{B19'}$ grain families along the gauge length within the macroscopic stress plateau region. Comparing these plots with Fig. 5 at macroscopic strains (3e) and (4a), it is seen that the newly formed B19' grain families exhibit a transition in *relative* lattice strain values from the transformation band through to the apparently transformed region.

Figure 6. Contour plots showing the (a, c) axial and (b, d) transverse *relative* lattice strains for the (a, b) $(020)_{B19'}$ and (c, d) $(111)_{B19'}$ grain families along the gauge length within the macroscopic stress plateau region.

In Fig. 5 the data from the centre of the gauge length for macroscopic strains (4b) through (5) is obtained within the slowly rising macroscopic stress region and corresponds to deformation accommodation by the $B19'$ phase. From $\sim$ macroscopic strain (4c) corresponding to a critical stress of ~ 426 MPa, deviations from linearity are noted in the *relative* lattice strains of the $(1\bar{2}0)_{B19'}$ grain family along the axial direction and the $(020)_{B19'}$ grain family along the transverse direction. On the other hand, the axial *relative* lattice strains of the $(020)_{B19'}$, $(111)_{B19'}$ and $(1\bar{1}1)_{B19'}$ grain families and the transverse *relative* lattice strains of the $(111)_{B19'}$ and $(002)_{B19'}$ grain families continue recording approximately linear trends up to macroscopic strain (5).

3.2 Rietveld analysis

3.2(a) Crystallite size, micro-strain and phase stress

Anisotropic peak broadening due to plastic strain can be interpreted in terms of the evolution of the anisotropic crystallite size and non-uniform micro-strain (root mean square or *r.m.s* strain, $\langle \epsilon^2 \rangle^{1/2}$) [48, 49]. Here crystallite size refers to the size of coherently diffracting domains within a crystal or grain [44] such that the crystallites show a degree of orientation-based similarity with each other. Micro-strain results from the local deviation of d -spacings from their average value and is caused by the generation of local defects. Instead of peak shifts, the mismatch between crystallites and the variation of d -spacings results in differences in diffraction angle.

The data shown in Fig. 7 is representative of the centre of the gauge length. Fig. 7a presents the evolution of crystallite size and micro-strain with respect to the macroscopic strain for the $(110)_{B2}$ grain family. Figs. 7b and 7c show the same for the various B19' grain families.

In Fig. 7a, loading within the elastic region between macroscopic strains (1) and (2) returned relatively large crystallite sizes ($\sim 1020 \pm 20$ nm) and low non-uniform micro-strains ($\sim 35 \pm 10$ $\mu\epsilon$). Within the macroscopic stress plateau region between macroscopic strains (3a) and (3d), the crystallite size of the B2 phase is approximately constant while the gradual increase in micro-strain values is indicative of the centre of the gauge length being affected by the approaching transformation band. By macroscopic strain (3e), the transformation band reaches the centre of the gauge length such that the $(110)_{B2}$ peak broadens and the crystallite size reduces to $\sim 100 \pm 14$ nm and the micro-strain increases markedly to $\sim 200 \pm 15$ $\mu\epsilon$. Within the slowly rising macroscopic stress region, both parameters are approximately constant and correspond to the large plastic strain accommodated by the ~ 5 vol.% of remnant $(110)_{B2}$ grains. All of the above are reflected in similar changes to the FWHM values in Fig. 4b: (i) low values ($\sim 0.05^\circ$) within the untransformed region, (ii) a transition from $\sim 0.06^\circ$ to $\sim 0.10^\circ$ within the transformation band, and (iii) higher values ($\sim 0.12^\circ$) in the apparently transformed region.

For the B19' grain families, the first data points are recorded within the macroscopic stress plateau region at macroscopic strain (3e) (Figs. 7b and 7c). Within the slowly rising macroscopic stress region, all B19' grain families exhibit similar general trends comprising a decrease in crystallite size and a concomitant increase in micro-strains. It is noted that the various grain families exhibit an anisotropy in crystallite sizes and micro-strains at relatively low macroscopic strains (for example - between macroscopic strains (3e) and (4b)). The anisotropy reduces markedly from macroscopic strain (4c) or critical stress ~ 426 MPa onwards.

(a)

(b)

(c)

Figure 7. Evolution of crystallite size and non-uniform micro-strain versus the macroscopic strain for the (a) $(110)_{B2}$ and (b, c) $B19'$ grain families from the centre of the gauge length. White - the elastic region, grey - the macroscopic stress plateau region, black - the slowly rising macroscopic stress region.

In Fig. 8, the estimated σ_{33} phase stresses borne by the B2 and $B19'$ phases within the macroscopic stress plateau region are shown. Fig. 8 is analogous to the B2 phase volume fraction (Fig. 4a) such that the B2 phase bears the majority of the uniaxial tensile load until its transformation to $B19'$.

Figure 8. Contour plots showing phase stresses for the (a) B2 and (b) B19' phases along the gauge length within the macroscopic stress plateau region.

3.2(b) Texture evolution of the B19' phases

The inverse pole figures (IPFs) in Figs. 9 and 10 depict the texture evolution of the B2 and B19' phases within the macroscopic stress plateau and slowly rising macroscopic stress regions, respectively. Fig. 9a is representative of the initial $\langle 111 \rangle_{B2}$ fibre texture with a maximum intensity $f(g) = 3.8$, comprising the $(110)_{B2}$ grain family whose plane normals are parallel to the axial/loading direction at macroscopic strain (2) in the centre of the gauge length. The obtained texture is consistent with previous reports on drawn and shape-set NiTi alloys [3, 48, 55]. Figs. 9b-9f correspond to IPFs of the remnant B2 phase within the transformation band between macroscopic strains (3a) and (3e) in Fig. 4a. Uniaxial tension results in an overall weakening of the $\langle 111 \rangle_{B2}$ fibre texture such that at macroscopic strain (3e), the ~ 16 vol.% of remnant B2 phase returned a maximum intensity $f(g) = 2.2$.

Figure 9. Inverse pole figures along the axial/loading direction of the B2 phase obtained from: (a) the untransformed region ((2), 0 mm in Fig. 1) and the propagating transformation band in Fig. 4a: (b) ((3a), -4 mm), (c) ((3b), -3 mm), (d) ((3c), -2 mm), (e) ((3d), -1 mm), (f) ((3e), 0 mm).

Fig. 10 provides representative IPFs of the B19' phase between macroscopic strains (4a) and (5) at the centre of the gauge length. They depict stronger $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ and weaker $[010]_{B19'}$ texture components. For the macroscopic strains depicted here, the maximum intensities of the $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ components increases from $f(g) \sim 15$ to ~ 17 and the $[010]_{B19'}$ increases from $f(g) \sim 8$ to ~ 14 .

Figure 10. Inverse pole figures along the axial/loading direction of the B19' phase obtained from the centre of the gauge length (0 mm in Fig. 1a) at macroscopic strains: (a) - (4a), (b) - (4b), (c) - (4c), (d) - (4d), (e) - (5).

4. Discussion

4.1 Macroscopic stress versus strain response during uniaxial monotonic tension

In previous digital image correlation work undertaken in Ref. [1], two broad factors were found to affect phase transformation during room temperature uniaxial tension: (i) grain orientation, and (ii) test conditions such as the sample thickness-to-length ratio and the loading speed. In polycrystalline NiTi alloys, the B2→B19' phase transformation preferentially initiates in $\langle 111 \rangle_{B2}$ orientations that are parallel to the macroscopic tensile loading direction [1, 3, 11]. Higher stresses are needed to transform unfavourably oriented B2 grains; which results in stress fluctuations within the macroscopic stress plateau region. In terms of test conditions, the propagation of the transformation band is intrinsically linked to the latent heat release within the transformation band. Insufficient heat release due to large thickness-to-length ratios and fast loading speeds results in deformation accommodation behaviour changing from the propagation of a single inclined band to multiple but similarly inclined bands. The latter phenomenon also results in a more homogenous strain distribution along the entire gauge length [4].

In the present case, the predominantly $\langle 111 \rangle_{B2}$ oriented grains (Fig. 9a) preferentially transformed to B19' such that a single transformation band propagates easily along the entire gauge length. This causes inhomogeneity in strain distribution along the gauge length between the B2 and B19' phases (Fig. 4c). A 1:14 sample thickness-to-length ratio and a crosshead speed of $0.02 \text{ mm} \cdot \text{min}^{-1}$ (corresponding to an initial strain rate of $1 \times 10^{-5} \text{ s}^{-1}$) suggests sufficient heat release within the transformation band and extensive B2→B19' phase transformation. The above is exemplified by the ~5 vol.% of remnant B2 grains at the centre of the gauge length by macroscopic strain (4a). These factors also result in a relatively small stress fluctuation within the macroscopic stress plateau region and a relatively large nominal transformation strain of 0.0640^2 [3].

4.2 The transformation band

Both previous digital image correlation work [1, 9, 36] and the present study show that phase transformation occurs within the propagating transformation band. Consequently, the load bearing ability is shared between the B2 and B19' phases in the untransformed and apparently transformed regions, ahead of and behind the transformation band, respectively.

In this section, the micro-mechanical behaviours of both phases are discussed along the gauge length in terms of the transformation band and the untransformed and apparently transformed regions (Figs. 3, 4, 6 and 8). As shown in Figs. 3 and 4c, the axial lattice strain of the $(110)_{B2}$ grains in the untransformed region undergoes relaxation whereas it increases by $\sim 1500 \text{ } \mu\epsilon$ within the

² This value is calculated by subtracting the macroscopic strain at the end of the elastic region from the macroscopic strain at the end of the macroscopic stress plateau region (i.e. - the macroscopic strains (2) and (4a) in Fig. 1b).

transformation band. In the apparently transformed region between macroscopic strains (4a) and (5), the axial lattice strain of the ~5 vol.% of remnant $(110)_{B2}$ grains records only small increases as most of the load is borne by the $B19'$ grains. Generally speaking, the present results are in agreement with Ref. [29] where small increments in lattice strain values were noted for the $B2$ grain families in the untransformed region when loading into the macroscopic stress plateau region of a loading-unloading tensile test. In this regard, the stress relaxation of the $(110)_{B2}$ grain family reported in Ref. [27] may refer to the measurements obtained from the untransformed centre of the gauge length and result from the strained $B2$ phase within the transformation band and apparently transformed regions elsewhere along the gauge length. The present results are in agreement with Polatidis *et al.* [16] and Schmahl *et al.* [28] who concluded that compared to the untransformed region, highly strained $B2$ grains are accumulated in the immediate vicinity of the mobile front of the transformation band. Such highly strained $(110)_{B2}$ grains further facilitate the $B2 \rightarrow B19'$ phase transformation by reducing the critical stress for transformation in its immediate vicinity at the mobile band front. According to Polatidis *et al.* [16], this results in the continuous transformation of neighbouring grains and propagation of a single transformation band instead of the nucleation of new and parallel transformation bands elsewhere along the gauge length.

Compared to the untransformed region, the remnant $B2$ phase within the transformation band displays higher FWHM values and corresponding increase in micro-strain and decrease in crystallite size (Figs. 4b and 7a); indicating dislocation mediated events in the $(110)_{B2}$ grains. Consequently, it is suggested that strain accommodation is realised by a combination of slip system activity in the $B2$ phase, $B2 \rightarrow B19'$ phase transformation and deformation accommodation by the $B19'$ phase. Here slip activity in the $B2$ phase is sustained by the increased distortion stress field at/near the $B2$ - $B19'$ interfaces that in turn, is associated with lattice shear as $B19'$ variants grow [29, 37, 56].

The formation of $B19'$ grain families is studied by considering the lattice correspondence between the $B2$ and $B19'$ phases [3, 54]. In this regard, lattice correspondence was reported for the following planes: $(011)_{B2}-(020)_{B19'}$, $(111)_{B2}-(\bar{1}\bar{2}0)_{B19'}$, and $(001)_{B2}-(100)_{B19'}$ [17, 54, 57]. It is noted that the $B2 \rightarrow B19'$ phase transformation does not result in a one-to-one correspondence due to the differences in structure factors and the multiplicity of lattice planes between the phases. Consequently, several $B19'$ grain families are associated with a single family of $B2$ grains. For example, in Ref. [15], the $(110)_{B2}$ grain family transformed into four $B19'$ grain families: $(020)_{B19'}$, $(\bar{1}\bar{1}1)_{B19'}$, $(002)_{B19'}$ and $(111)_{B19'}$. This also rationalises the observation of several $B19'$ grain families in the present diffraction patterns (Fig. 1a). In this regard, the $B19'$ phase comprises a mix of grain families that are either elastically: (i) harder (for example, $(020)_{B19'} = 102.0$ GPa, $(\bar{1}\bar{1}1)_{B19'} = 84.0$ GPa) or, (ii) softer ($(\bar{1}\bar{2}0)_{B19'} = 54.4$ GPa, $(\bar{1}\bar{2}1)_{B19'} = 65.3$ GPa), than the $(110)_{B2}$ grains (82 GPa). It follows that the increased axial lattice strains of the remnant $(110)_{B2}$ grains within the transformation band is the result of the transformation strain and continued load bearing due to their interaction with elastically softer $B19'$ grains in their immediate vicinity. We compared the general trends in the *relative* lattice strains of $(020)_{B19'}$ (Fig. 6) and $(001)_{B19'}$

from Ref. [14]. In this study, the lattice parameter sequence $a < c < b$, $\gamma \neq 90^\circ$ whereas in Ref. [14] $a < b < c$, $\beta \neq 90^\circ$ was used. Consequently, the $(010)_{B19'}$ of this study and the $(001)_{B19'}$ from Ref. [14] correspond to the largest d -spacing of the unit cell and refer to the same lattice plane such that their trends are analogous. Similar to this study, rather large variations in lattice strain values for different B19' grain families were also noted by Schmahl *et al.* [28]. They attributed the observation to the stress field build-up across the transformation band as the material tries to maintain strain-continuity.

4.3 Deformation accommodation within the B19' phase

At macroscopic strain (4c) corresponding to a critical stress of ~ 426 MPa, the *relative* lattice strains of the $(1\bar{2}0)_{B19'}$ and $(020)_{B19'}$ grain families deviate from linearity along the axial and transverse directions, respectively (Fig. 5). Additionally, anisotropy in crystallite size and micro-strains is observed in all B19' grain families up to macroscopic strain (4c) (Figs. 7b and 7c).

Similar tendencies in lattice strain evolution for the B19' phase were noted in Refs. [14, 29]. In those studies, the various B19' grain families that formed in the macroscopic stress plateau region recorded *relative* lattice strains that first increased linearly within the slowly rising macroscopic stress region and then deviated from linearity above a critical stress. In Ref. [14], data prior to deviation from such linearity was used to evaluate the elastic moduli of the B19' reflections. Several researchers [1, 14, 29] have associated the deviation from linearity to the activation of deformation accommodation mechanisms such as elasto-plastic deformation, variant re-orientation, twinning and de-twinning of the B19' phase.

4.4 Texture evolution during uniaxial monotonic tension

The pole intensity in the IPF is related to the volume fraction of a given orientation g following $V_{f(g)} = \Delta V_{(g)} / V_{total} = \int f_{(g)} dg$ [58]. The present study shows that within the macroscopic stress plateau region, the initial $\langle 111 \rangle_{B2}$ fibre texture transforms to the predominant $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ and weaker $[010]_{B19'}$ components (Figs. 9 and 10). Weakening of the $\langle 111 \rangle_{B2}$ fibre texture (Fig. 9) results from its transformation to B19' variants and the subsequent redistribution of the local elastic stress field in adjacent grains [59, 60]. In Ref. [60] wherein a 54.1 Ni-45.9Ti rolled sheet was studied during the loading stage of a uniaxial cyclic loading-unloading test, the original rolling texture component of the B2 phase was reported to weaken upon transformation to six different B19' variants. In the slowly rising macroscopic stress region and up to macroscopic strain (5), the $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$ and $[010]_{B19'}$ components record increases in maximum intensity. Similar trends were reported in Ref. [35], in which an initial $[334]_{B2}$ fibre of a 56.1Ni-43.9Ti cold-drawn wire, which is $\sim 8^\circ$ deviated from the ideal $[111]_{B2}$ fibre, transformed to the $[\bar{1}30]_{B19'}$ fibre. Such $[334]_{B2} \rightarrow [\bar{1}30]_{B19'}$ fibre transformation is further rationalised by considering lattice shear and rigid body rotation such that the large d -spacing of $(\bar{1}30)_{B19'}$ planes makes them the most favourable amongst all potential variants transformed from the $(334)_{B2}$ grain family.

Sittner *et al.* [3] undertook a theoretical calculation of the transformation strain required for potential B19' variants with respect to the starting B2 orientation and found that $(110)_{B2} \rightarrow (020)_{B19'}$ and $(111)_{B2} \rightarrow (1\bar{2}0)_{B19'}$ transformations result in maximum macroscopic strains of ~ 0.077 and ~ 0.10 , respectively during tension. In agreement with this, a $(110)_{B2}$ oriented cold-rolled 55.8Ni-44.2Ti sheet subjected to uniaxial tension [15] transformed to $(020)_{B19'}$; as the latter grain family is preferentially aligned along the macroscopic tensile direction and accommodates the largest transformation strain. Ye *et al.* [61] calculated the transformation strain in the B19' coordinate frame and showed that the $[\bar{1}50]_{B19'}$ fibre accommodated transformation strains as large as 0.10 along the axial/loading direction.

Within the macroscopic stress plateau region of this study, the $\langle 111 \rangle_{B2}$ fibre transforms to the $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ and $[010]_{B19'}$ fibres. The latter fibres record increases in maximum intensity ($f(g)$) at higher macroscopic strains in the slowly rising macroscopic stress region. It follows that once the transformation band propagates through the entire gauge length, re-orientation and de-twinning may be active within the B19' phase up to macroscopic strain (5).

The rationale supporting this hypothesis is gleaned from Refs. [30-33] who showed that the macroscopic stress plateau region results from variant re-orientation and de-twinning (mainly of $[011]_{B19'}$ type-II twins). In turn, this led to the preferential increase in maximum intensity ($f(g)$) of the $[010]_{B19'}$ and $[\bar{1}50]_{B19'}$ fibres up to the end of the macroscopic stress plateau region.

Within the macroscopic stress plateau region, Liu and Xie [62] reported that the de-twinning of $\langle 011 \rangle$ type-II twins is realised via atomic shear along the $[011]$ shear direction on $(11\bar{1})$ planes. This led to the cooperative advancement of $(11\bar{1})$ ledges along their normal directions. Consequently, the irrational $(0.7205 \pm 1\bar{1})$ twin plane migrates and results in the growth of one B19' variant at the expense of the other. Other reports on texture evolution within the macroscopic stress plateau region state that the initial $[\bar{1}20]_{B19'}$, $[120]_{B19'}$ and $[102]_{B19'}$ fibres rotate to the $[\bar{1}20]_{B19'}$ fibre which subsequently rotates to $[\bar{1}30]_{B19'}$ on account of B19' de-twinning [35].

Based on the literature, further loading into the slowly rising stress region (and well beyond macroscopic strain (5) of the present study) may result in the following: (i) The $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ components rotate to $[110]_{B19'}$ when new twinning modes such as $(20\bar{1})$ deformation twinning occur [35]. (ii) The $[010]_{B19'}$ fibre rotates to $[230]_{B19'}$ and/or $[110]_{B19'}$ due to a combination of slip, (001) compound twinning or $(20\bar{1})$ and (113) deformation twinning [30, 35].

5. Conclusions

In-situ synchrotron was used to study the B2 $\rightarrow$ B19' phase transformation in a cold-drawn and annealed 56Ni-44Ti wt.% alloy during uniaxial monotonic tension. Spatially resolved diffraction data acquired along the gauge length at five select macroscopic strains within the stress plateau region tracked localised transformation phenomena. The results can be summarised as follows:

1. The initial $\langle 111 \rangle_{B2}$ grains, a small sample thickness-to-length ratio and a slow crosshead speed favours the B2 $\rightarrow$ B19' phase transformation and contributes to a single transformation band

propagating through the gauge length. This also causes a relatively small stress fluctuation of ± 10 MPa within the macroscopic stress plateau region and a relatively large nominal transformation strain of 0.0640.

2. Within the macroscopic stress plateau region, the B2 phase is highly strained within the propagating transformation band and apparently transformed regions, which produces a relaxation of the B2 phase within the untransformed region. The *relative* lattice strains of the B19' grain families exhibit a transition from the transformation band through to the apparently transformed region. The $\langle 111 \rangle_{B2}$ fibre texture transforms to $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$ and $[010]_{B19'}$; the latter components continue to record increases in maximum intensity up to maximum load.

3. Within the slowly rising macroscopic stress region and beyond a critical stress value of ~ 426 MPa, the $(\bar{1}20)_{B19'}$ and $(020)_{B19'}$ grain families show deviations from linearity in the *relative* lattice strains along the axial and transverse directions, respectively. The anisotropy in crystallite size and micro-strains in all B19' grain families reduces markedly.

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Data Availability statement

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

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