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An in situ synchrotron study of the localized B2↔B19' phase transformation in an Ni-Ti alloy subjected to uniaxial cyclic loading-unloading with incremental strains

Abstract

© 2020. High-resolution in situ synchrotron X-ray diffraction was applied to study a cold-drawn and solution-treated 56Ni-44Tiwt% alloy subjected to uniaxial cyclic loading-unloading with incremental strains. The micro-mechanical behaviour associated with the partial and repeated B2↔B19' phase transformation at the centre of the sample gauge length was studied with respect to the macroscopic stress-strain response. The lattice strains of the (110)B2 and different B19' grain families are affected by (i) the transformation strain, the load-bearing capacity of both phases and the strain continuity maintained at/near the B2-B19' interfaces at the centre of the gauge length, and (ii) the extent of transformation along the gauge length. With cycling and incremental strains (i) the elastic lattice strain and plastic strain in the remnant (110)B2 grain family gradually saturate at early cycles, whereas the plastic strain in the B19' phase continues to increase. This contributes to accumulation of residual strains (degradation in superelasticity), greater non-linearity and change in the shape of the macroscopic stress-strain curve from plateau type to curvilinear elastic. (ii) The initial 111B2 fibre texture transforms to [120]B19', [130]B19', [150]B19' and [010]B19' orientations. Further increase in the applied strain with cycling results in the development of [130]B19', [102]B19', [102]B19', [100]B19' and [100]B19' orientations.

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An *in situ* synchrotron study of the localized B2↔B19' phase transformation in an Ni–Ti alloy subjected to uniaxial cyclic loading–unloading with incremental strains

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High-resolution *in situ* synchrotron X-ray diffraction was applied to study a cold-drawn and solution-treated 56Ni–44Ti wt% alloy subjected to uniaxial cyclic loading–unloading with incremental strains. The micro-mechanical behaviour associated with the partial and repeated B2↔B19' phase transformation at the centre of the sample gauge length was studied with respect to the macroscopic stress–strain response. The lattice strains of the (110)_{B2} and different B19' grain families are affected by (i) the transformation strain, the load-bearing capacity of both phases and the strain continuity maintained at/near the B2–B19' interfaces at the centre of the gauge length, and (ii) the extent of transformation along the gauge length. With cycling and incremental strains (i) the elastic lattice strain and plastic strain in the remnant (110)_{B2} grain family gradually saturate at early cycles, whereas the plastic strain in the B19' phase continues to increase. This contributes to accumulation of residual strains (degradation in superelasticity), greater non-linearity and change in the shape of the macroscopic stress–strain curve from plateau type to curvilinear elastic. (ii) The initial ⟨111⟩_{B2} fibre texture transforms to $[\bar{1}20]_{B19'}$, $[130]_{B19'}$, $[\bar{1}50]_{B19'}$ and $[010]_{B19'}$ orientations. Further increase in the applied strain with cycling results in the development of $[130]_{B19'}$, $[\bar{1}02]_{B19'}$, $[102]_{B19'}$, $[\bar{1}00]_{B19'}$ and $[100]_{B19'}$ orientations.

1. Introduction

Under uniaxial monotonic tension, NiTi shape memory alloys are characterized by a stress plateau region followed by a slowly rising stress region in their macroscopic stress–strain curves. The maximum stress plateau region can extend up to ~0.12 engineering strains at low initial strain rates (between ~10^{−3} and ~10^{−4} s^{−1})¹ (Bian, Saleh *et al.*, 2019; Stebner *et al.*, 2015; Zhang *et al.*, 2010; Daly *et al.*, 2007; Šittner *et al.*, 2005; Huang & Liu, 2001). Superelasticity of NiTi alloys allows for large reversible strains under loading–unloading cycles within the maximum stress plateau region, which is associated with the reversible transformation between the primitive cubic B2 (austenite) and the monoclinic B19' (martensite) phases (Daly *et al.*, 2007; Šittner *et al.*, 2005; Huang & Liu, 2001; Shaobin & Yongjun, 2018; Schaefer & Wagner, 2009; Yawny *et al.*, 2005). In several engineering applications, NiTi alloys are subjected

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¹ The maximum macroscopic stress plateau region and the slowly rising macroscopic stress region are referred to in the case of uniaxial monotonic tension from here on.

to repetitive cyclic loading–unloading within or up to the maximum macroscopic stress plateau region. In this regard, the study of superelasticity generally includes fatigue testing and cyclic loading–unloading and they are best tracked by employing *in situ* characterization techniques.

For typical fatigue testing that involves repeated loading–unloading cycles up to the maximum macroscopic stress plateau region, the number of cycles to failure and the associated energy dissipation were studied (Shaobin & Yongjun, 2018; Zotov *et al.*, 2017; Xie *et al.*, 2016; Maletta *et al.*, 2014; Kim & Daly, 2011). *In situ* digital image correlation revealed that Lüders-like transformation band(s) of higher macroscopic tensile strains (than the untransformed region) propagate and shrink/remain throughout the gauge length. They develop as a single band inclined at an angle η ($\sim 60^\circ$)/ $-\eta$ ($\sim 120^\circ$) to the sample axial direction, or two bands criss-crossing each other at angles η and $-\eta$ (Daly *et al.*, 2007; Zotov *et al.*, 2017; Xie *et al.*, 2016; Maletta *et al.*, 2014; Kim & Daly, 2011; Shaw & Kyriakides, 1997; Xiao *et al.*, 2017). *In situ* synchrotron/neutron diffraction² was used to investigate the micro-mechanical behaviours such as lattice strain and peak width of individual grain families and phase volume fraction (Xie *et al.*, 2016; Sedmák *et al.*, 2015; Hasan *et al.*, 2008; Polatidis *et al.*, 2015). It was shown that the change in the shape of the macroscopic stress–strain curve can be attributed to several microstructural mechanisms, such as the formation of stable B19' variants and their re-orientation and de-twinning, slip system activities and the generation of lattice defects mainly in the form of geometrically necessary dislocations (Zotov *et al.*, 2017; Sedmák *et al.*, 2015; Cai *et al.*, 2015; Young *et al.*, 2010).

To characterize all aspects of the mechanisms of stress-induced B2→B19' transformation and its reversibility, successive cycles of tensile loading–unloading with incremental strains are needed. Our previous digital image correlation studies (Bian *et al.*, 2018; Bian, Gazder *et al.*, 2019) showed that single inclined band(s) develop at one end of the gauge length, expanding and shrinking during early cycles. Thereafter, multiple criss-crossing patterns occur at the pre-existing strained band and propagate through to the untransformed region. Such pattern evolution assists in straightening the sample edges by releasing an in-plane asymmetric moment caused by the local shear strains of individual bands (Jiang, Kyriakides & Landis, 2017; Jiang, Kyriakides, Landis & Kazinakis, 2017; Waitz *et al.*, 2008). Upon unloading, the most recently formed bands tend to transform back more than the bands that formed earlier, with the latter experiencing a greater number of cycles and accumulating higher residual strains. Upon subsequent loading, these retained bands first accommodate the incremental strains until saturation such that new criss-crossing bands develop through

to the virgin untransformed regions. Although there are studies following the transformation start stress, the recovered strain, the transformation modulus and the accumulated residual strain (Huang & Liu, 2001; Yawny *et al.*, 2005; Maletta *et al.*, 2014; Yinong *et al.*, 1999), the micro-mechanical behaviour of the partial and repeated B2↔B19' phase transformation is not easily captured due to the forward/reverse propagation of the localized bands (Sedmák *et al.*, 2015; Cai *et al.*, 2015). However, systematic study of such phase transformation is essential since it enables (i) better understanding of the localized strain accumulation with incremental macroscopic strain, (ii) study of the degradation in superelasticity and the change of transformation mode with successive cycles and increasing applied strains (Koster *et al.*, 2015), and (iii) assessment of the practical lifetime (*i.e.* accumulated residual strain) of the material when subjected to cyclic loading–unloading involving variation of applied strains, for example in the construction and biomedical fields (Shaobin & Yongjun, 2018; Xie *et al.*, 2016).

Compared with laboratory X-ray and neutron diffraction, synchrotron X-ray diffraction offers the following benefits: (i) accurate elastic lattice strain and phase fraction measurements due to the extremely intense and highly collimated beam, (ii) reduced data collection times and negligible stress relaxation as a result of the high X-ray flux, and (iii) the ability to undertake quantitative texture analysis combined with Rietveld refinement of the Debye–Scherrer rings (Lowell *et al.*, 2006; Lonardelli *et al.*, 2005; Ischia *et al.*, 2005; Wenk & Grigull, 2003; Wenk *et al.*, 2014; Lutterotti *et al.*, 2014; Grässlin *et al.*, 2013). Schmahl *et al.* (2004) studied a cold-rolled 55.75Ni–44.25Ti (in wt% from here on) alloy and Polatidis, Zotov & Mittemeijer (2015) studied a thermally cycled 55.36Ni–44.64Ti alloy during uniaxial tension with synchrotron diffraction. They observed highly strained remnant (110)_{B2} grains within the transformation band compared with the untransformed region. Cai *et al.* (2015) performed *in situ* synchrotron-based measurements on a cold-drawn 56.06Ni–43.94Ti wire under loading–unloading cycles for three selected incremental macroscopic strains covering the maximum stress plateau region and the slowly rising stress region. They observed opposite evolution of lattice strains for the (110)_{B2} and (211)_{B2} grain families within the mobile transformation band at the centre of the gauge length. Since both grain families have an elastic anisotropy factor of 0.25, the difference in lattice strains was rationalized by considering the B2↔B19' orientation relationship and the variations in elastic moduli between the corresponding B2 and B19' grain families. However, the effect of loading–unloading cycles, incremental strains and overall phase transformation along the gauge length on the diffraction data was not evaluated. Moreover, the evolution of texture in the stress-induced B19' phase was not reported in that study. In addition, information on the strain state and load bearing of the B2 (remnant) and B19' phases and their relation to the transformation mechanism is still limited.

The texture evolution during uniaxial monotonic tension associated with B19' deformation via slip, twinning and

² For *in situ* diffraction measurements, data from a local region (typically the centre of the sample gauge length) are acquired when the sample consists of a single B2 or B19' phase, *i.e.* at points such as (i) the start of the elastic region and (ii) the end of the maximum macroscopic stress plateau region of each cycle, or (iii) strains of interest in the case of sampling from the entire gauge length. Consequently, the obtained data are reflective of the sample bulk.

de-twinning has been extensively investigated via *in situ* synchrotron/neutron diffraction (Cai *et al.*, 2013; Stebner *et al.*, 2013; Benafan *et al.*, 2012, 2013; Qiu *et al.*, 2011; Nicholson *et al.*, 2018). In contrast, transformation texture studies of the stress-induced B19' phase are limited and there are even fewer data on cyclic loading–unloading, due to the localized nature of the phase transformation process and the general diffraction measurement configuration (*i.e.* sampling from the centre of the gauge length). Rietveld-based texture analysis (Cai *et al.*, 2013) of the Debye–Scherrer rings of a cold-drawn 56.1Ni–43.9Ti wire subjected to uniaxial monotonic tension revealed that the initial $[334]_{B2}$ fibre transforms to $[\bar{1}30]_{B19'}$ via lattice deformation and rigid-body rotation. Stebner *et al.* (2015) carried out *in situ* neutron diffraction measurements on a cold-drawn 55.87Ni–44.13Ti alloy under uniaxial monotonic tension, and studied the texture evolution in the B2 and product B19' phases. It was reported that the initial $[111]_{B2}$ fibre transforms to $[\bar{1}21]_{B19'}$, $[\bar{2}10]_{B19'}$ and $[102]_{B19'}$ orientations, as a result of a competition between elastic energy minimization and the geometric restrictions of the B2–B19' interfaces upon transformation.

The aim of the present *in situ* synchrotron study is to provide further insight into the B2 $\leftrightarrow$ B19' phase transformation in a cold-drawn and solution-treated 56Ni–44Ti alloy subjected to uniaxial cyclic loading–unloading with incremental strains. The phase fraction, lattice strain and peak width of individual grain families and the texture of both phases were captured and studied with respect to the macroscopic stress–strain curves, with the aid of strain distribution along the gauge length derived from prior digital image correlation studies.

2. Experimental and analytical procedures

The source 10 mm-diameter 56Ni–44Ti polycrystalline rod from Nitinol Devices and Components Inc. was cut into smaller 10 mm (diameter) $\times$ 42 mm (total length) rods. Sample preparation for mechanical testing involved (i) encapsulation of the small rods in quartz tubes under an Ar atmosphere, (ii) annealing at 973 K for 120 s in a muffle furnace followed by water quenching to room temperature and (iii) electro-discharge machining into 10 mm (gauge length) $\times$ 5 mm (width) $\times$ 0.7 mm (thickness) dog-bone samples with their gauge lengths parallel to the rod axial direction (Fig. 1). The heat treatment resulted in an initial single B2 phase with an average grain size of $\sim 10 \pm 5.9 \mu\text{m}$ in the solution-treated samples.

In situ synchrotron diffraction measurements in transmission mode were carried out on the Powder Diffraction beamline at the Australian Synchrotron, using a 21 keV X-ray source (wavelength 0.59041 Å) with a beam size of $1.0 \times 0.5 \text{ mm}^2$ along the axial/loading (vertical) and transverse (horizontal) directions, respectively. A detailed description of the *in situ* experiments undertaken on the beamline is available in the work of Bettles *et al.* (2011) and Wang, Lynch *et al.* (2018). A sample-to-detector distance of 415.261 mm and exposure time for each measurement of 240 s were used. The

Debye–Scherrer rings were collected by a Mar345 image-plate detector (345 mm diameter, pixel size of $100 \times 100 \mu\text{m}$). Prior to mechanical testing, calibration data from an LaB₆ standard (NIST Standard Reference Material SRM-660) were recorded. The lattice strain sensitivity is estimated to be $\pm 150 \mu\epsilon$. Fig. 1 shows the diffraction geometry and the Debye–Scherrer rings of the NiTi sample for the maximum applied strain of the first loading–unloading cycle. On the image (detector) plate, azimuth angles (η) of $90^\circ/270^\circ$ and $0^\circ/180^\circ$ correspond to the axial/loading (Ax) and transverse (Tr) directions, respectively.

The uniaxial cyclic loading–unloading test was performed at room temperature on a Deben Instruments 5 kN tensile stage integrated into the beamline at a loading–unloading speed of 0.02 mm min^{-1} . The macroscopic engineering stress was converted from the force data recorded by the load cell and the macroscopic engineering strain was determined by correlating the displacement data to the macroscopic elastic stress–strain data from prior digital image correlation measurements conducted on an Instron universal testing machine (Bian *et al.*, 2018; Kada *et al.*, 2016). The first loading–unloading cycle involved 0.05 maximum engineering strain, which was determined from prior *in situ* synchrotron measurement under uniaxial monotonic tension such that the localized B2 $\rightarrow$ B19' phase transformation was captured at the centre of the gauge length (Bian, Saleh *et al.*, 2019). The subsequent loading–unloading involved ~ 0.015 engineering strain increments per cycle and a total of seven cycles were performed. In this way, the obtained macroscopic stress–strain curves cover the maximum stress plateau region and the slowly rising stress region, and the time for loading–unloading and data acquisition was acceptable. Diffraction data from the centre of the gauge length were collected by pausing the test at strain points of interest. These include (i) points at the start and middle of the elastic region, (ii) the end of the elastic region or the onset of the stress plateau region, (iii) the maximum applied strain of the macroscopic stress plateau region, (iv) points at the middle and two-thirds through the unloading region, and (v) the point in the unloaded state. During data acquisition, the sample symmetry and the experimental setup led to a macroscopic stress relaxation within 5 MPa, which is negligible compared with the macroscopic stress–strain curve (Brinson, 2004; Brinson *et al.*, 2002).

Figure 1
A schematic of the sample dimensions (in mm) and the synchrotron diffraction setup in transmission mode.

Post-processing of the Debye–Scherrer rings involved single peak fitting and Rietveld refinement. Single peak fitting yields information on the lattice strain, peak width (full width at half-maximum, FWHM) of individual grain families and phase volume fractions. To start with, the recorded LaB₆ standard ring pattern was used to calibrate the beam centre, sample-to-detector distance and tilt of the detector (roll, pitch and yaw) using *Fit2D* (Hammersley *et al.*, 1996; Hammersley, 2016). Thereafter, the acquired ring patterns were segmented into 5° slices, which were radially integrated into a diffraction spectrum that corresponds to an *X–Y* plot of 2θ versus total X-ray intensity and thus is reflective of the micro-mechanical behaviour within ±2.5° of the direction of interest (Fig. 1). In this regard, diffraction spectra corresponding to the axial/loading and transverse directions were fitted using a *TOPAS* refinement code (Bruker, 2005; Coelho, 2019). The observed reflections were indexed for the B2 and B19' ($a < c < b$, $\gamma \neq 90^\circ$) phases. In the case of the B2 phase, only the (110)_{B2} reflection was fitted as the (200)_{B2} reflection was too weak and consequently excluded from the fitting. The lattice strain (ε_{hkl}) of individual grain families was evaluated using the following equation:

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

where d^{hkl} is the instantaneous lattice spacing upon mechanical testing and d_0^{hkl} is the reference lattice spacing that was obtained before mechanical testing. For the B19' phase and according to Sedmák *et al.* (2015), the reference lattice spacings were taken from an analogous synchrotron-based uniaxial monotonic tensile test using the same experimental setup as the current study (Bian, Saleh *et al.*, 2019). In the work of Bian, Saleh *et al.* (2019), diffraction data were collected along the gauge length at macroscopic strains following the onset of the stress plateau region. Using equation (1), relative lattice strains were obtained with respect to the B19' grain families that first appeared along the gauge length at relatively low macroscopic strains. Phase volume fractions (*V*) were then assessed from the integrated intensities in both the axial/loading and transverse directions using equations (2)–(4) (Cullity & Stock, 2001):

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

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

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

where *I* = integrated intensity. For each reflection, the calculation of the *R* value involves *v* = unit-cell volume, *F* = structure factor for a given *hkl* reflection, *p* = multiplicity factor and $\exp(-2M)$ = temperature factor. Combining the integrated intensities of multiple B19' reflections effectively reduces the possible texturing in calculating the phase volume fraction (Cullity & Stock, 2001).

Rietveld refinement of the Debye–Scherrer rings was carried out using the *Material Analysis Using Diffraction (MAUD)* software. An azimuth (η) range from 110 to 250° was used in order to exclude the shadowed region due to the experimental setup. It is known that the material under study possesses an initial (111)_{B2} fibre texture along the axial/loading direction and develops an axisymmetric fibre texture upon uniaxial tensile loading (Bian *et al.*, 2018; Bian, Gazder *et al.*, 2019; Bian, Saleh *et al.*, 2019; Šittner *et al.*, 2005; Mao *et al.*, 2010; Gall *et al.*, 2005). Texture is sufficiently strong such that it was refined at an early stage in order to provide reasonable initial estimates of intensities for refining the microstructure and phase stresses. The intensity variation along a Debye–Scherrer ring is proportional to the pole intensity of the orientation sphere imposed on the sample. When viewed along the axial/loading direction and without rotating the sample, each Debye–Scherrer ring is limited to a pair of curved lines with a 2θ distance to each other on the pole figure (Lonardelli *et al.*, 2005; Wenk *et al.*, 2014). Fibre symmetry was imposed in such a way as to improve on the effective pole figure coverage. The tomography-based extended Williams–Imhof–Matthies–Vinel (E-WIMV) approach was used (Lonardelli *et al.*, 2005; Wenk *et al.*, 2014; Lutterotti *et al.*, 2007; Matthies & Vinel, 2006). It refines orientation distributions via an iterative routine and accounts for irregular coverage of the orientation distribution space.

Plastic strain-induced anisotropic peak broadening was evaluated in terms of crystallite size and microstrain (root-mean-square or r.m.s. strain, $\langle \varepsilon^2 \rangle^{1/2}$) of the B2 and B19' phases. The Delft line broadening model combined with Popa anisotropic broadening rules was used. The anisotropic size and microstrain models depend on Laue groups. The microstrain is developed into series with two and nine coefficients for the B2 (cubic) and B19' (monoclinic) phases, respectively. The crystallite size is developed into a convergent series of symmetrized spherical harmonics with refinable coefficients (Popa & Balzar, 2008; Popa, 1998). In the current analysis, a harmonic order of 4 was enough to describe the microstructure since higher orders did not result in any significant improvement.

The azimuthal dependence of diffraction peak shifts along the Debye–Scherrer rings was evaluated using the Moment pole stress incorporating the 'BulkPathGEO' micro-mechanical model which takes into account crystallographic texture (Wenk *et al.*, 2014; Nielsen *et al.*, 2015; Sumin *et al.*, 2012). In this model, the elastic tensors for both phases are required for a quantitative interpretation of their macro-stress state (Thomasová *et al.*, 2017; Bihlmayer *et al.*, 1994). The symmetric stress tensor consists of hydrostatic and deviatoric stress components. The hydrostatic stress component is generally large and is refined in terms of the lattice parameters, whereas the deviatoric stress component is small and is considered by assuming a linear stress–strain relationship for small strains (Wenk *et al.*, 2014). In this regard, the axial deviatoric stress component (σ_{33} , along the *z* direction in Fig. 1) was refined in order to account for the sinusoidal oscillations in *d* spacing with respect to the azimuth angle (η), which effectively eliminates errors in fitting the positions and

Figure 2
(a) A representative stack of the experimental (bottom) and fitted (top) diffraction spectra at the maximum applied strain of cycle C6, with ~ 64 vol.% of B19' phase. Reference d spacings are indicated by dashed lines. (b) Rietveld refinement output for the diffraction spectra along the axial (Ax) and transverse (Tr) directions for the same loading condition in (a). The black points are the experimental data and the red line is the Rietveld fit. The tick marks indicate reflections from the B2 and B19' phases. The difference curve between refinement and measurement is shown at the bottom.

intensity maxima of all peaks of interest. Fig. 2 gives a representative Rietveld refinement for the data at the maximum applied strain of loading–unloading cycle C6, which indicates reasonably good fits. Fig. 2(a) shows the stack of fitted (top) and experimental (bottom) diffraction spectra; Fig. 2(b) depicts the quality of the refinement for the same data along the axial and transverse directions in Fig. 2(a).

3. Results

3.1. Macroscopic stress–strain response and the localized B2 $\leftrightarrow$ B19' phase transformation

Fig. 3 presents the macroscopic engineering stress–strain response. The colour scheme in Fig. 3(a) (from here on) specifies each loading–unloading cycle, with the squares denoting the applied strains for diffraction measurements

Figure 3
(a) The macroscopic stress–strain curve during uniaxial cyclic loading–unloading in tension with strain increments for each cycle. The colour scheme outlines plots of cycles C1–C7. Squares denote the positions at which diffraction measurements were performed upon loading (solid markers) and unloading (open markers). (b) Evolution of the transformation start stress (σ_s) and superelastic strain (ϵ_{SE}) versus the number of cycles. In the inset, definitions of the corresponding parameters are represented on the plot of cycle C2.

during loading (solid markers) and unloading (open markers). In the following paragraphs, the macroscopic strain and local strain refer to the macroscopic axial engineering strain and local axial engineering strain, respectively. To correlate the macroscopic stress–strain response with single peak fitting analysis at the centre of the gauge length, we have provided a schematic illustration of the local strain evolution along the gauge length for characteristic deformation stages at cycles C1–C3 and C6 (Fig. 4): (1) the middle of the elastic region, (2) the end of the elastic region or the onset of the stress plateau region, (3) the maximum applied strain (*i.e.* the end of the stress plateau region), (4) the unloaded state of individual cycles.

Fig. 4 is based on prior digital image correlation studies and spatially resolved *in situ* synchrotron measurements along the gauge length (Bian *et al.*, 2018; Bian, Gazder *et al.*, 2019; Bian, Saleh *et al.*, 2019; Daly *et al.*, 2007; Polatidis, Zotov, Bischoff *et al.*, 2015; Jiang, Kyriakides & Landis, 2017; Jiang, Kyriakides, Landis & Kazinakis, 2017). The level of local strain is proportional to the extent of phase transformation therein, *i.e.* the B19' phase fraction (Fig. 5). As detailed in the following,

Figure 4

A schematic derived from digital image correlation measurements which illustrates the axial strain evolution (phase transformation) along the gauge length, for characteristic deformation stages (1) the middle of the elastic region, (2) the end of the elastic region or the onset of the stress plateau region, (3) the maximum applied strain (*i.e.* the end of the stress plateau region) and (4) the unloaded state at cycles C1–C3 and C6. The rectangle 1.0×0.5 mm signifies the beam size.

single peak fitting and Rietveld refinement of data obtained from the centre of the gauge length are interpreted accounting for the accompanying transformation along the gauge length and incremental strain with cycle number.

In Fig. 3(a), the elastic region of cycle C1 returned an apparent elastic modulus of 73.1 GPa for the B2 phase (calculated for 0–300 MPa and 0–0.0037 macroscopic strain), comparable to the 40–90 GPa range reported in the literature (Young *et al.*, 2010). This is followed by a stress relaxation from ~430 to ~380 MPa, which represents the onset of the stress plateau region. Upon unloading, the macroscopic stress–strain curve recorded (i) a deviation from linearity due to the difference in the elastic moduli of the B2 and B19' phases, (ii) a stress plateau region and a stress increment derived from the reverse transformation of B19'→B2, and (iii) a near-linear reduction in stress pertaining to the unloading of the B2 and retained B19' phases.

On the basis of Figs. 3(a), 4 and 5, the elastic region is characterized by the uniform local strain along the gauge length [C1 (1)]. At the end of the elastic region, the localized development of strain concentration results in the initiation of phase transformation at one end of the parallel gauge length [C1 (2)]. The drop in stress is associated with the full development of a new transformation band across the sample width, whereas the stress plateau region corresponds to the expansion of such a new band [C1 (2)–(3)]. At the end of the stress

plateau of C1, the transformation band reaches the centre of the gauge length such that ~87 vol.% of B19' phase is detected [see C1 (3) and Fig. 5]. Upon unloading, the reverse transformation of B19'→B2 leads to ~5 vol.% of remnant B19' phase at the centre of the gauge length [C1 (4) and Fig. 5].

From cycle C2 onwards, loading towards the middle and the end of the apparently elastic region results in phase transformation favoured in the remnant/pre-existing band(s), which leads to nonlinearity [Fig. 3(a)] and progressive increase in the B19' fraction in Fig. 5 [*e.g.* C1 (4)–C2 (1)–C2 (2), C2 (4)–C3 (1)–C3 (2)]. As strain accommodation saturates within these

Figure 5

Evolution of the B19' phase volume fraction measured at the centre of the gauge length versus total engineering strain. Solid marker = loading, open marker = unloading.

Figure 6

Evolution of (a) axial lattice strain and (b) FWHM versus total engineering strain for the (110)_{B2} grain family. Solid marker = loading, open marker = unloading.

pre-existing band(s) [C2 (2), C3 (2)], the drop in stress and the subsequent transitional stress plateau region signify the occurrence of new transformation band(s) nucleating at (i) the pre-existing strained transformation band front to constitute criss-crossing patterns [C2 (3), C3 (3)] and (ii) the end of the gauge length, favoured by intrinsic lattice defects (*e.g.* dislocations and grain boundaries) of the B2 phase and the stress enhancement caused by the sample geometry [C2 (3), C3 (2)–(3)] (Waitz *et al.*, 2008; Cohen, 1992). It follows that such non-consecutive development of new transformation bands leads to variations in the B19' phase fraction detected at the centre of the gauge length (Fig. 5). At cycle C6, the remnant transformation bands of increasing residual strains are distributed through the gauge length such that the macroscopic stress fluctuations are caused by the transformation of the remaining B2 regions between these bands [C6 (2)–(3)]. Only limited reverse transformation occurs upon unloading as a result of the accumulated residual strain [C6 (4)]. The increasing stabilized B19' phase reaches up to ~35 vol.% after unloading from cycle C7, which lowers the transformation start stress in its vicinity and the residual stress fields promote plastic deformation. Consequently, the macroscopic stress–strain curves exhibit greater nonlinearity: the stress relaxation and subsequent transitional stress plateau region (C2 to C4) give way to a slowly rising stress region (C5 to C7); and their shape changes from plateau type to curvilinear elastic. In Fig. 4, following the forward/reverse phase transformation, the centre of the gauge length comprises a mixture of (i) low-

strain B2 phase, (ii) strained B2 phase, (iii) the newly formed B19' phase and (iv) highly strained B19' phase.

To better understand the macroscopic response, we defined parameters that are represented on the macroscopic stress–strain curve of cycle C2 and studied their evolution with respect to the number of loading–unloading cycles [Fig. 3(b)]. In Fig. 3(b) (inset), the transformation start stress (σ_s) is defined as the macroscopic stress corresponding to the deviation from linearity at the end of the elastic region (Montecinos *et al.*, 2008). The applied superelastic strain is defined as $\varepsilon_{SE}^i = \varepsilon_t^i - \varepsilon_e^i - \varepsilon_{UL}^i$, where ε_t^i , ε_e^i and ε_{UL}^i refer to the applied strain, the elastic strain upon unloading and the residual strain, respectively, for the *i*th cycle (Sutou *et al.*, 2005). The ratio of superelastic strain to applied strain is shown instead of the absolute superelastic strain to account for the slight difference in the maximum applied strain per cycle. This shows that the transformation start stress decreases monotonically; the superelastic strain increases from cycle C1 to C2 before decreasing progressively.

Figs. 6 and 7 illustrate the general evolution of the axial lattice strain and FWHM of the $(110)_{B2}$, $(020)_{B19'}$ and $(1\bar{1}1)_{B19'}$ grain families, respectively, as a function of the total engineering strain and the number of cycles. Within the elastic region of cycle C1, the lattice strain of the $(110)_{B2}$ grain family increases linearly up to ~5100 $\mu\epsilon$ [Fig. 6(a)] and the FWHM values cluster at ~0.05° [Fig. 6(b)]. The subsequent decrease in the lattice strain to ~4700 $\mu\epsilon$ and the concomitant stability of the FWHM values (~0.05°) signify the onset of the

macroscopic stress plateau region and stress relaxation of the B2 phase away from the transformation band. At the maximum applied strain, the transformation band reaches the centre of the gauge length such that the remnant B2 phase therein is characterized by increases in lattice strain to ~5500 $\mu\epsilon$ and FWHM to ~0.12° (the last solid square). Upon unloading, the lattice strain decreases to ~700 $\mu\epsilon$ and the FWHM continues to increase progressively up to ~0.14°. From cycle C2 onwards, the lattice strain (i) increases through the apparently elastic region, (ii) varies from ~6000 $\mu\epsilon$ (C2) to ~7800 $\mu\epsilon$ (C7) within the macroscopic stress plateau region, as a result of transformation along the gauge length, and (iii) decreases to ~1800 $\mu\epsilon$ upon unloading (C3–C7). Also from cycle C2 onwards, the FWHM decreases within the apparently elastic region, stabilizes within the macroscopic stress plateau region and gradually increases to ~0.13–0.15° upon unloading.

In Fig. 7, the first data points are obtained at the maximum applied strain of cycle C1 (which corresponds to the

Figure 7
Evolution of (a), (c) axial relative lattice strain and (b), (d) FWHM versus total engineering strain for the (a), (b) $(020)_{B19'}$ and (c), (d) $(1\bar{1}1)_{B19'}$ grain families. Solid marker = loading, open marker = unloading.

first detection of the B19' phase in the present experiment). The $(020)_{B19'}$ and $(\bar{1}21)_{B19'}$ grain families evolve similarly with regard to the axial relative lattice strains to the $(110)_{B2}$ grain family. Their axial FWHM values, for the middle of the elastic region of each cycle, (i) remain approximately the same (for instance compare the last open square of a given cycle with the first solid square of a subsequent cycle) for $(020)_{B19'}$ (C1 to C5) and $(\bar{1}21)_{B19'}$ (C1 to C3) and (ii) increase for $(020)_{B19'}$ (C6–C7) and $(\bar{1}21)_{B19'}$ (C5 and C7). The evolution of FWHM with cycling indicates an increase in the plastic strain for the $(020)_{B19'}$ grain family [Fig. 7(b)] and an early saturation of plastic strain for the $(\bar{1}21)_{B19'}$ grain family [Fig. 7(d)].

Fig. 8 examines the characteristic deformation stages (1)–(4) between loading–unloading cycles, in terms of the integrated intensities, axial lattice strains and FWHM of the lattice correspondence planes $(110)_{B2}$ and $(020)_{B19'}$, given that phase transformation is concurrent along the gauge length. The main characteristics are summarized as follows.

The two grain families show opposite evolution of the integrated intensities. In particular, the $(020)_{B19'}$ relative intensity [Fig. 8(b)] varies for the maximum applied strain and gradually increases for the other three stages. The lattice strain for the $(110)_{B2}$ grain family [Fig. 8(c)] gradually approaches saturation for each stage. In Fig. 8(e), the initial FWHM value ($\sim 0.05^\circ$) of cycle C1 is a reflection of the recrystallized state of the B2 phase. Thereafter, the FWHM remains almost constant at higher values from cycle C2 onwards.

For the $(020)_{B19'}$ grain family, the axial relative lattice strain [Fig. 8(d)] for the maximum applied strain remains roughly constant at $\sim -4600 \mu\epsilon$ from cycles C1 to C4 and then increases rapidly to $\sim -2500 \mu\epsilon$ (C7). In contrast, this value fluctuates between loading–unloading cycles at $\sim -4500 \mu\epsilon$ for the middle and the end of the elastic region and $\sim -6500 \mu\epsilon$ for the unloaded state. In Fig. 8(f), the FWHM shows a clear increase from C1 to C7 for each stage.

3.2. Rietveld analysis of the uniaxial cyclic loading–unloading results

3.2.1. Evolution of crystallite size, microstrain and phase stress. The initial crystallite size [$\sim 1200 \pm 100$ nm, Fig. 9(a)] and microstrain [$\sim 120 \pm 10 \mu\epsilon$, Fig. 9(b)] values of the B2 phase remain approximately constant up to the maximum applied strain. Thereafter, the transformation band reaches the centre of the gauge length and the remnant B2 peaks broaden. Correspondingly, the crystallite size reduces markedly to $\sim 90 \pm 10$ nm and the microstrain increases to $\sim 250 \pm 15 \mu\epsilon$. During unloading, the crystallite size remains at $\sim 90 \pm 10$ nm whereas the microstrain continues to increase to $\sim 340 \pm 15 \mu\epsilon$. From cycle C2 onwards, the crystallite size increases to $\sim 600 \pm 10$ nm within the macroscopic stress plateau region and decreases progressively to $\sim 100 \pm 10$ nm upon unloading. The microstrains continuously increase up to $\sim 550 \pm 60 \mu\epsilon$ in the unloaded state. For the start of the

Figure 8 Evolution of (a), (b) relative intensity, (c), (d) (relative) lattice strain and (e), (f) FWHM versus the number of cycles for the (a), (c), (e) $(110)_{B2}$ and (b), (d), (f) $(020)_{B19'}$ grain families, represented for characteristic deformation stages (1) the middle of the elastic region, (2) the end of the elastic region or the onset of the stress plateau region, (3) the maximum applied macroscopic strain (*i.e.* the end of the stress plateau region) and (4) the unloaded state for each cycle.

Figure 9

Evolution of (a) crystallite size and (b) non-uniform microstrain versus total engineering strain for the B2 phase. Solid marker = loading, open marker = unloading.

next loading–unloading cycle, they decrease sharply to $\sim 230 \pm 20 \mu\epsilon$ and increase thereafter through the cycle.

Fig. 10 shows the evolution of the crystallite size and microstrain as a function of the total engineering strain for the $(020)_{B19'}$ grain family.³ The two grain families exhibit similar general trends. The crystallite size decreases upon unloading in cycle C1. From cycle C2 onwards, the crystallite size decreases upon loading and starts to increase upon unloading. However, an overall decreasing trend is observed with increasing number of cycles. For the microstrain, a rapid increase for cycles C2–C3 and a slight increase from cycle C4 is noted during loading to the middle of the elastic region. This is followed by a rapid increase upon further loading up to the maximum applied strain and a sharp decrease upon unloading.

Fig. 11 illustrates the estimated σ_{33} phase stresses borne by the B2 and B19' phases as a function of the total engineering strain. The positive values of σ_{33} indicate tensile stresses for the remnant B2 and product B19' phases. Upon loading of cycle C1, the B2 phase bears the majority of the uniaxial tensile load until its transformation to B19'. Thereafter, both the B2 and B19' phase stresses recorded repeated cycles of increase upon loading within the apparently elastic region, a

Figure 10

Evolution of (a) crystallite size and (b) non-uniform microstrain versus total engineering strain for the $(020)_{B19'}$ grain family. Solid marker = loading, open marker = unloading.

Figure 11

Evolution of phase stress σ_{33} versus total engineering strain for the B2 and B19' phases. Solid marker = loading, open marker = unloading.

small rise (up to C5), a rapid rise (for C6–C7) through the end of the elastic region to the macroscopic stress plateau region and rapid decrease upon unloading.

3.2.2. Texture evolution in the B2 and B19' phases. Figs. 12 and 13 show the inverse pole figures along the axial/loading direction of the B2 and B19' phases, respectively, for characteristic deformation stages (1)–(4) at cycles C1–C3 and C6. In Fig. 12, cycle C1 (1)–(2) represent an initial $(111)_{B2}$ fibre

³ Data for crystallite size and microstrain for the $(1\bar{1}21)_{B19'}$ grain family are given as Fig. S1 in the supporting information, as the trends are similar to those for the $(020)_{B19'}$ grain family.

Figure 12

Inverse pole figures along the axial/loading direction of the B2 phase obtained for characteristic deformation stages (1) the middle of the elastic region, (2) the end of the elastic region or the onset of the stress plateau region, (3) the maximum applied macroscopic strain (*i.e.* the end of the stress plateau region), and (4) the unloaded state at cycles C1–C3 and C6.

texture with a maximum intensity $f(g) = 6.4$, which is consistent with previous studies on drawn and shape-set NiTi alloys (Šittner *et al.*, 2005; Mao *et al.*, 2010; Gall *et al.*, 2005; Luo *et al.*, 2017). The $\langle 111 \rangle_{B2}$ fibre texture corresponds to the $(110)_{B2}$ grain family whose plane normals are parallel to the axial/loading direction. The texture after cyclic loading–unloading (C2–C6) is similar to that of the initial state, with a slight decrease in the maximum intensity around the $\langle 111 \rangle_{B2}$ fibre [$f(g) \sim 5.0$].

In Fig. 13, cycle C1 (3) indicates that the initial $\langle 111 \rangle_{B2}$ fibre texture transforms to relatively stronger $[\bar{1}20]_{B19'}$ and $[130]_{B19'}$ and weaker $[\bar{1}50]_{B19'}$ and $[010]_{B19'}$ texture components upon

uniaxial tensile loading into the macroscopic stress plateau region. Upon unloading [C1 (4)], all these B19' texture components weaken with concomitant formation of even weaker $[130]_{B19'}$ and $[100]_{B19'}$ components for the remnant ~ 5 vol.% of the B19' phase. Upon cycle C2 (1), the same fraction of remnant B19' phase yields orientations scattered around $[\bar{1}20]_{B19'}/[\bar{1}30]_{B19'}$, $[010]_{B19'}$, $[130]_{B19'}$, and newly formed $[\bar{1}02]_{B19'}$ and $[102]_{B19'}$. These orientations exhibit variations in the maximum intensities with respect to the B19' phase fraction at stages C2 (2)–(4).

For cycles C3 and C6, $[\bar{1}20]_{B19'}/[\bar{1}30]_{B19'}$, $[130]_{B19'}$, $[\bar{1}00]_{B19'}$ and $[100]_{B19'}$ vary in the maximum intensities at different stages. In particular, $[130]_{B19'}$ continues to strengthen upon loading [C3 (1)–(3)] and weakens upon unloading [C3 (4)]. $[\bar{1}02]_{B19'}$ and $[102]_{B19'}$ develop through C6 (1)–(4).

4. Discussion

4.1. Deformation behaviour during uniaxial cyclic loading–unloading

In the present study, the smooth macroscopic stress plateau region (stress fluctuation of ± 4 MPa) upon loading of cycle C1 suggests the ease of the B2 $\rightarrow$ B19' phase transformation propagating progressively along the gauge length. This is favoured by the predominantly $\langle 111 \rangle_{B2}$ -oriented grains [C1 (1)–(2), Fig. 12], a small 1:14 sample thickness to length ratio and a slow crosshead speed of 0.02 mm min^{-1} (corresponding to an initial strain rate of $1 \times 10^{-5} \text{ s}^{-1}$) (Zhang *et al.*, 2010; Daly *et al.*, 2007; Šittner *et al.*, 2005; Šittner, Lukáš *et al.*, 2004; Huang & Liu, 2001; Mao *et al.*, 2010). Upon unloading in cycle C1, the early reverse transformation of preferentially oriented monoclinic B19' variants back to B2 takes place within a stress plateau region, causing anisotropic changes to the local stress fields that hinder the reverse transformation of other variants (Xie *et al.*, 2016; Zheng *et al.*, 2017; Ammar *et al.*, 2017). The latter variants require an increase in stress [see in Fig. 3(a) the end of the stress plateau on unloading] to overcome the residual strain surrounding them and reverse transform to B2 (Huang & Liu, 2001).

From a microscopic point of view, the B2 $\rightarrow$ B19' phase transformation is a shear displacement process that occurs on specific invariant habit planes along specific shear directions (Zotov *et al.*, 2017). Upon cyclic loading–unloading, several microstructural mechanisms are also involved, *e.g.* re-orientation, twinning and de-twinning of the B19' phase, slip system activity, and generation of lattice defects mainly in the form of dislocations and remnant thermodynamically stable B19' variants

Figure 13

Inverse pole figures along the axial/loading direction of the B19' phase obtained for characteristic deformation stages (1) the middle of the elastic region, (2) the end of the elastic region or the onset of the stress plateau region, (3) the maximum applied macroscopic strain (*i.e.* the end of the stress plateau region), and (4) the unloaded state at cycles C1–C3 and C6.

(Yawny *et al.*, 2005; Xie *et al.*, 2016; Maletta *et al.*, 2014; Cai *et al.*, 2015). In particular, geometrically necessary dislocations are introduced in order to accommodate the shape strain and the distortion stress fields associated with the lattice shear as B19' variants grow. Sedmák *et al.* (2015) proposed a hybrid slip/transformation process such that, during forward loading, each time the mobile phase transformation band passes through a given crystal volume, dislocations form alongside the mobile band and leave the particular crystal volume slightly elongated. In addition, our previous electron back-scattering diffraction maps (Bian *et al.*, 2018; Bian, Gazder *et al.*, 2019) showed that upon unloading after cyclic loading–unloading, the intra-granular heterogeneity of plastic strain originates from (i) the B2 phase that has experienced reversible transformation, (ii) the interfaces between the B2 and B19' phases and (iii) the remnant B19' variants. These dislocation activities, in turn, inhibit the B19' variants from recovering their original configuration by dissipating the elastic strain energy accumulated during forward transformation (Maletta *et al.*, 2014).

Correspondingly, the deformation mode changes from being localized to homogeneous along the gauge length, and macroscopic residual (irreversible) strain (degradation in superelasticity) accumulates. Upon unloading, the lack of elastic strain energy results in limited reverse transformation such that the stress plateau region and the increase in stress are replaced by a nonlinear stress–strain response from cycle C4 onwards.

4.2. The microstructural evolution during the localized B2↔B19' phase transformation upon uniaxial cyclic loading–unloading

The diffraction data are indicative of the local region and contain information on the internal strain fields (load bearing) and the density of defects of grain families therein with respect to the extent of phase transformation (phase fractions) and the external load exerted on the bulk sample.

The changes in the FWHM values of the (110)_{B2} grain family [Figs. 6(b) and 8(e)] are analysed together with the crystallite size and microstrain of the B2 phase in Fig. 9. At the sub-micrometre scale and upon loading of cycle C1, forward transformation occurs such that B19' variants form within the original recrystallized B2 grains (Shaobin & Yongjun, 2018). Here, we defined 'active B2 region' (*i.e.* the region that transformed to B19') in contrast to the remnant B2 matrix. As mentioned in Section 4.1, the transformation process induces dislocation activities. Consequently, the crystallite size decreases to a lower limit and the microstrain increases markedly. This results in an increase in FWHM of the remnant (110)_{B2} grain family. Upon unloading, the B19' variants partially transform back to B2 (*i.e.* within the prior 'active B2 region'). Larger plastic strain is accumulated in the 'active B2 region' than in the remnant B2 matrix, as the former experienced forward/reverse transformation upon tensile loading–unloading. This contributes to the continuous rise in the microstrain. As a result, the FWHM of the (110)_{B2} grain family

increases continuously. From cycle C2 onwards, the generally decreasing FWHM upon loading up to the middle of the elastic region results from the ongoing transformation of the 'active B2 region' to B19', which leads to an overall increase in the crystallite size and a decrease in the microstrain for the remnant (110)_{B2} grain family. Alternatively, the approximately constant FWHM values within the macroscopic stress plateau region suggest saturation of plastic strain in the remnant B2 matrix. It is seen that, at this stage, the approximately stable crystallite size dominates the FWHM evolution, albeit with an increasing microstrain. Upon unloading, the reverse-transformed 'active B2 region' contributes to an overall decrease in crystallite size and an increase in microstrain, and thus increased FWHM for the (110)_{B2} grain family.

For the middle of the elastic region, the diffraction data were acquired at macroscopic stresses 238 ± 17 MPa for cycles C1 to C7 [Fig. 3(a)]. In Fig. 8(c), by examining the lattice strain of the (110)_{B2} grain family and considering its axial diffraction elastic modulus [$D_{(110)_{B2}} = 82$ GPa] (Bian, Saleh *et al.*, 2019; Šittner *et al.*, 2014), the internal stress [$\sigma_{\text{internal}} = D_{(110)_{B2}} \mu\epsilon$] in these grains can be estimated as 246 MPa for cycle C1, 295 MPa for cycle C2 and 390 MPa from cycle C3 onwards, approaching the value of the forward transformation start stress. It follows that, upon loading to this stage, the highly strained B2 phase favours transformation, which leads to the increasing nonlinearity in the macroscopic stress–strain curves and the decreasing transformation start stress in Fig. 3.

The formation of the B19' grain families is studied by considering the B2–B19' orientation relationship and lattice correspondence planes, namely (011)_{B2}–(020)_{B19'}, (111)_{B2}–(1 $\bar{2}$ 0)_{B19'} and (001)_{B2}–(100)_{B19'} (Šittner *et al.*, 2005; Šittner, Lukáš *et al.*, 2004; Sedmák *et al.*, 2015). Several B19' grain families can form from a single family of B2 grains due to the differences in structure factors and the multiplicity of lattice planes between the phases (Hasan *et al.*, 2008; Šittner, Lukáš *et al.*, 2004). Variations in the lattice strains of different B19' grain families were attributed to (i) the transformation strain, (ii) the continued load bearing due to their interaction with the corresponding B2 grains and the different elastic moduli of B2 and B19' grain families (Cai *et al.*, 2015), and (iii) the strain-continuity maintained at/near the B2–B19' interfaces (Schmahl *et al.*, 2004). The axial relative lattice strains of the B19' grain families at the centre of the gauge length [Figs. 7(a), 7(c) and 8(d)] are comparable to those obtained within the transformation band upon loading within the macroscopic stress plateau region in the work of Bian, Saleh *et al.* (2019), given that the B19' peaks shift to lower d spacing upon tensile loading–unloading (Sedmák *et al.*, 2015).

The changes in the FWHM and the B19' phase fraction are analysed with respect to the defined characteristic deformation stages. From the middle to the end of the apparently elastic region in the macroscopic stress–strain curve, the increase in the B19' fraction (Fig. 5) and FWHM (Fig. 7) indicate that this loading stage is actually associated with the occurrence of B2→B19' phase transformation and the development of plastic strain in the B19' phase at the centre of the gauge length. Within the macroscopic stress plateau

region, the constant values of FWHM and the fluctuations in the B19' fraction could be related to the transformation along the gauge length. Upon unloading, the constant FWHM values indicate limited plastic strain accumulation within one cycle.

Comparing Figs. 8(c)–8(d) and Figs. 8(e)–8(f) suggests that the load-bearing capacity and plastic strain of the (110)_{B2} grain family saturate after early cycles. For the (020)_{B19'} grain family, the load borne increases noticeably at the maximum applied strain (cycles C4–C6), whereas the plastic strain accumulates at all stages with cycling. In the work of Sedmák *et al.* (2015), *in situ* synchrotron diffraction was carried out on a 55.9Ni–44.1Ti wire subjected to ten tensile loading–unloading cycles to the end of the macroscopic stress plateau region, and it was reported that, for the unloaded state, the load gradually transfers from the B2 phase to the residual B19' phase. Under the present loading path, the non-uniform strain accommodation along the gauge length results in the above B19' stress states and remnant B19' fractions being different from those described by Sedmák *et al.* (2015).

4.3. Texture development during uniaxial cyclic loading–unloading

Upon the stress-induced B2→B19' phase transformation, the transition of the initial $\langle 111 \rangle_{B2}$ fibre texture to the $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$, $[\bar{1}50]_{B19'}$ and $[010]_{B19'}$ components [C1 (1)–(3), Fig. 13] is in agreement with those reported in the literature. Upon cycling, the B2↔B19' phase transformation and the resultant redistribution of the local elastic stress fields in the adjacent B2 grains lead to the weakening of the $\langle 111 \rangle_{B2}$ fibre texture (Stebner *et al.*, 2015; Wang, Ma *et al.*, 2018; Barney *et al.*, 2011). The effect of external stress on the B19' texture can be approximated by assuming that the B19' lattice correspondent variants mainly form by providing maximum strain in the loading direction of individual crystallites (Šittner *et al.*, 2005; Šittner, Neov *et al.*, 2004; Novák *et al.*, 2009). Šittner, Neov *et al.* (2004) calculated the transformation strains of potential B19' variants appearing in three basic orientations of B2 grains and concluded that the transformations of $(111)_{B2} \rightarrow (\bar{1}20)_{B19'}$ and $(110)_{B2} \rightarrow (010)_{B19'}/(020)_{B19'}$ produce maximum tensile strains of ~ 0.10 and ~ 0.077 , respectively. Ye *et al.* (2007) predicted the transformation strain in various directions in the B19' coordinate frame using the 12 lattice correspondent variants and lattice parameters of both phases. It was shown that the B19' variants possess the highest $(150)_{B19'}$ pole intensity along the loading direction at a transformation strain of 0.10.

From the experimental aspect, a $(110)_{B2}$ -oriented cold-rolled 55.8Ni–44.2Ti sheet transformed to $(020)_{B19'}$ (Hasan *et al.*, 2008); a cold-drawn 56Ni–44Ti rod with an initial $\langle 111 \rangle_{B2}$ fibre transformed to $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$ and $[010]_{B19'}$ (Bian, Saleh *et al.*, 2019b). In these studies, the product B19' grain families are preferentially aligned along the macroscopic tensile direction and confirm the relatively large transformation strains. In the work of Cai *et al.* (2013), an initial $[334]_{B2}$ fibre (deviating by $\sim 8^\circ$ from $[111]_{B2}$) of a cold-drawn 56.1Ni–43.9Ti wire transformed to a $[\bar{1}30]_{B19'}$ fibre. It was reported

that the large d spacing of the $(\bar{1}30)$ planes makes them the most favourable amongst all potential variants transformed from the $(334)_{B2}$ grain family via lattice deformation and rigid-body rotation.

With cycling and increasing applied strains in this study, $[\bar{1}20]_{B19'}/[\bar{1}30]_{B19'}$ and $[130]_{B19'}$ orientations intensify upon loading and weaken upon unloading (cycle C2). $[\bar{1}20]_{B19'}/[\bar{1}30]_{B19'}$ gives way to orientations such as $[130]_{B19'}$, $[102]_{B19'}$ and $[\bar{1}02]_{B19'}$ upon cycles C3–C6. In particular, $[130]_{B19'}$, $[\bar{1}00]_{B19'}$, $[100]_{B19'}$, $[102]_{B19'}$ and $[\bar{1}02]_{B19'}$ orientations continue to strengthen for the unloaded state. These orientations can be induced by re-orientation and de-twinning within the B19' phase upon repeated B2↔B19' phase transformation and the concomitant increasing plastic strain.

Similar trends were reported for uniaxial monotonic tensile loading into the slowly rising macroscopic stress region. Cai *et al.* (2013) reported that $[\bar{1}30]_{B19'}$ yielded a slight decrease in the maximum intensity at a macroscopic strain of 0.17, which indicates B19' re-orientation. Stebner *et al.* (2015) studied a cold-drawn 55.9Ni–44.1Ti bar with initial $(111)_{B2}$ fibre texture and pointed out that the formation of the B19' variants is a result of the competition between elastic energy minimization and the geometric restrictions of the B2–B19' interfaces. The former indicates that most open-packed planes ($[\bar{1}50]_{B19'}$) align with the loading direction, whereas the latter restricts the direct transformation of the material to the elastically preferred orientations, but results in rather non-optimal orientations such as $[\bar{1}21]_{B19'}$, $[\bar{2}10]_{B19'}$ and $[102]_{B19'}$. In addition, the texture evolution in the present study can be further rationalized by the strain accommodation mechanisms in the B19' phase. According to the literature, further loading into the slowly rising stress region results in (i) rotation from the $[\bar{1}20]_{B19'}$ and $[\bar{1}30]_{B19'}$ components to $[110]_{B19'}$ and $[230]_{B19'}$ due to the formation of $(20\bar{1})$ and (100) deformation twins and slip system activity, and (ii) further nucleation and growth of $(20\bar{1})$ and (100) and possible formation of new (113) twins and dislocations (Cai *et al.*, 2013; Benafan *et al.*, 2012; Liu & Xie, 2003; Tan & Liu, 2004; Ezaz *et al.*, 2011; Liu *et al.*, 1998).

5. Conclusions

A cold-drawn and solution-treated 56Ni–44Ti wt% alloy was subjected to *in situ* uniaxial cyclic loading–unloading combined with synchrotron diffraction. The partial and repeated B2↔B19' phase transformations at the centre of the sample gauge length were studied with respect to the loading–unloading cycles and increasing applied strains on the bulk sample. The results can be summarized as follows:

(i) The initial $\langle 111 \rangle_{B2}$ texture, a small sample thickness to length ratio and a slow crosshead speed facilitate the B2→B19' phase transformation and contribute to a smooth macroscopic stress plateau region. Upon unloading, the stress plateau region is associated with the reverse transformation of preferentially oriented B19' variants back to B2; the resultant anisotropic changes to the local stress fields hinder the reverse transformation of other variants and an increase in stress is required to overcome the residual strain surrounding them.

(ii) The lattice strain of the $(110)_{B2}$ grain family and the variations in the relative lattice strains of different $B19'$ grain families are affected by (a) the transformation along the gauge length, (b) the transformation strain, (c) the continued load bearing of corresponding $B2$ and $B19'$ grains possessing various elastic moduli, and (d) the strain-continuity maintained at/near the $B2$ – $B19'$ interfaces at the centre of the gauge length.

(iii) Load bearing and plastic strain in the remnant $(110)_{B2}$ grain family gradually saturate at early cycles, whereas plastic strain in the $B19'$ phase is enhanced in a cycle-to-cycle manner with increasing applied strains instead of within individual cycles. This results in accumulated residual strains (degradation in superelasticity), greater nonlinearity and changes to the shape from plateau type to curvilinear elastic in the macroscopic stress–strain curves.

(iv) Upon loading of the first cycle, the initial $\langle 111 \rangle_{B2}$ fibre texture transforms to $[\bar{1}20]_{B19'}$, $[\bar{1}30]_{B19'}$, $[\bar{1}50]_{B19'}$ and $[010]_{B19'}$ texture components. Upon cycling and increasing applied strains, $[\bar{1}20]_{B19'}/[\bar{1}30]_{B19'}$ weakens whereas orientations such as $[130]_{B19'}$, $[102]_{B19'}$ and $[\bar{1}02]_{B19'}$ develop. In particular, $[130]_{B19'}$, $[\bar{1}00]_{B19'}$, $[100]_{B19'}$, $[102]_{B19'}$ and $[\bar{1}02]_{B19'}$ orientations continue to strengthen for the unloaded state.

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References

- Ammar, O., Haddar, N. & Dieng, L. (2017). *Intermetallics*, **81**, 52–61.
- Barney, M. M., Xu, D., Robertson, S. W., Schroeder, V., Ritchie, R. O., Pelton, A. R. & Mehta, A. (2011). *J. Mech. Behav. Biomed. Mater.* **4**, 1431–1439.
- Bettles, C. J., Lynch, P. A., Stevenson, A. W., Tomus, D., Gibson, M. A., Wallwork, K. & Kimpton, J. (2011). *Metall. Mater. Trans. A*, **42**, 100–110.
- Benafan, O., Padula, S. A., Noebe, R. D., Brown, D. W., Clausen, B. & Vaidyanathan, R. (2013). *Acta Mater.* **61**, 3585–3599.
- Benafan, O., Padula, S. A., Noebe, R. D., Sisneros, T. A. & Vaidyanathan, R. (2012). *J. Appl. Phys.* **112**, 093510.
- Bian, X., Gazder, A. A., Saleh, A. A. & Pereloma, E. V. (2019). *J. Alloys Compd.* **777**, 723–735.
- Bian, X., Saleh, A. A., Lynch, P. A., Davies, C. H. J., Pereloma, E. V. & Gazder, A. A. (2019). *Mater. Sci. Eng. A*, **743**, 327–338.
- Bian, X., Saleh, A. A., Pereloma, E. V., Davies, C. H. J. & Gazder, A. A. (2018). *Mater. Sci. Eng. A*, **726**, 102–112.
- Bihlmayer, G., Eibler, R. & Neckel, A. (1994). *Phys. Rev. B*, **50**, 113.
- Brinson, L. C. (2004). *J. Mech. Phys. Solids*, **52**, 1549–1571.
- Brinson, L. C., Schmidt, I. & Lammering, R. (2002). *J. Intell. Mater. Syst. Struct.* **13**, 761–772.
- Bruker (2005). *TOPAS Total Pattern Analysis Solutions*. Bruker AXS, Madison, WI, USA.
- Cai, S., Schaffer, J. E., Ren, Y. & Yu, C. (2013). *Appl. Phys. Lett.* **103**, 241909.
- Cai, S., Schaffer, J. E., Yu, C., Daymond, M. R. & Ren, Y. (2015). *Metall. Mater. Trans. A*, **46**, 2476–2490.
- Coelho, A. A. (2018). *J. Appl. Cryst.* **51**, 210–218.
- Cohen, M. (1992). *Mater. Trans. JIM*, **33**, 178–183.
- Cullity, B. D. & Stock, S. R. (2001). *Elements of X-ray Diffraction*, 3rd ed. Harlow: Pearson.
- Daly, S., Ravichandran, G. & Bhattacharya, K. (2007). *Acta Mater.* **55**, 3593–3600.
- Ezaz, T., Sehitoglu, H. & Maier, H. J. (2011). *Acta Mater.* **59**, 5893–5904.
- Gall, K., Tyber, J., Brice, V., Frick, C. P., Maier, H. J. & Morgan, N. (2005). *J. Biomed. Mater. Res. A*, **75**, 810–823.
- Grässlin, J., McCusker, L. B., Baerlocher, C., Gozzo, F., Schmitt, B. & Lutterotti, L. (2013). *J. Appl. Cryst.* **46**, 173–180.
- Hammersley, A. P. (2016). *J. Appl. Cryst.* **49**, 646–652.
- Hammersley, A. P., Svensson, S. O., Hanfland, M., Fitch, A. N. & Häusermann, D. (1996). *High Pressure Res.* **14**, 235–248.
- Hasan, M., Schmahl, W. W., Hackl, K., Heinen, R., Frenzel, J., Gollerthan, S., Eggeler, G., Wagner, M., Khalil-Allafi, J. & Baruj, A. (2008). *Mater. Sci. Eng. A*, **481–482**, 414–419.
- Huang, X. & Liu, Y. (2001). *Scr. Mater.* **45**, 153–160.
- Ischia, G., Wenk, H.-R., Lutterotti, L. & Berberich, F. (2005). *J. Appl. Cryst.* **38**, 377–380.
- Jiang, D., Kyriakides, S. & Landis, C. M. (2017). *Extreme Mech. Lett.* **15**, 113–121.
- Jiang, D., Kyriakides, S., Landis, C. M. & Kazinakis, K. (2017). *Eur. J. Mech. A Solids*, **64**, 131–142.
- Kada, S. R., Lynch, P. A., Kimpton, J. A. & Barnett, M. R. (2016). *Acta Mater.* **119**, 145–156.
- Kim, K. & Daly, S. (2011). *Exp. Mech.* **51**, 641–652.
- Koster, M., Lee, W. J., Schwarzenberger, M. & Leinenbach, C. (2015). *Mater. Sci. Eng. A*, **637**, 29–39.
- Liu, Y., Liu, Y. & Humbeeck, J. (1998). *Scr. Mater.* **39**, 1047–1055.
- Liu, Y. & Xie, Z. L. (2003). *Acta Mater.* **51**, 5529–5543.
- Lonardelli, I., Wenk, H.-R., Lutterotti, L. & Goodwin, M. (2005). *J. Synchrotron Rad.* **12**, 354–360.
- Lowell, M., Sébastien, M., Takehiko, Y., Nagayoshi, S., Yasuo, O. & Hans-Rudolf, W. (2006). *J. Phys. Condens. Matter*, **18**, S995.
- Luo, J., Bobanga, J. O. & Lewandowski, J. J. (2017). *J. Alloys Compd.* **712**, 494–509.
- Lutterotti, L., Bortolotti, M., Ischia, G., Lonardelli, I. & Wenk, H. R. (2007). *Z. Kristallogr. Suppl.* **2007**, 125–130.
- Lutterotti, L., Vasin, R. & Wenk, H.-R. (2014). *Powder Diffr.* **29**, 76–84.
- Maletta, C., Sgambitterra, E., Furguele, F., Casati, R. & Tuissi, A. (2014). *Int. J. Fatigue*, **66**, 78–85.
- Mao, S. C., Luo, J. F., Zhang, Z., Wu, M. H., Liu, Y. & Han, X. D. (2010). *Acta Mater.* **58**, 3357–3366.
- Matthies, S. & Vinel, G. W. (2006). *Phys. Status Solidi B*, **112**, K111–K114.
- Montecinos, S., Cuniberti, A. & Sepúlveda, A. (2008). *Mater. Charact.* **59**, 117–123.
- Nicholson, D. E., Benafan, O., Padula, S. A., Clausen, B. & Vaidyanathan, R. (2018). *Shape Memory Superelasticity*, **4**, 143–157.
- Nielsen, M. B., Parisiades, P., Madsen, S. R. & Bremholm, M. (2015). *Dalton Trans.* **44**, 14077–14084.
- Novák, V., Šittner, P., Pilch, J. & Delville, R. (2009). *European Symposium on Martensitic Transformations*, article No. 03009. Les Ulis: EDP Sciences.

- Polatidis, E., Zotov, N., Bischoff, E. & Mittemeijer, E. J. (2015). *Scr. Mater.* **100**, 59–62.
- Polatidis, E., Zotov, N. & Mittemeijer, E. J. (2015). *Powder Diff.* **30**, S76–S82.
- Popa, N. C. (1998). *J. Appl. Cryst.* **31**, 176–180.
- Popa, N. C. & Balzar, D. (2008). *J. Appl. Cryst.* **41**, 615–627.
- Qiu, S., Clausen, B., Padula, S. A. II, Noebe, R. D. & Vaidyanathan, R. (2011). *Acta Mater.* **59**, 5055–5066.
- Schaefer, A. & Wagner, M. F.-X. (2009). *European Symposium on Martensitic Transformations*, article No. 06031. Les Ulis: EDP Sciences.
- Schmahl, W. W., Khalil-Allafi, J., Hasse, B., Wagner, M., Heckmann, A. & Somsen, C. (2004). *Mater. Sci. Eng. A*, **378**, 81–85.
- Sedmák, P., Šittner, P., Pilch, J. & Curfs, C. (2015). *Acta Mater.* **94**, 257–270.
- Shaobin, Z. & Yongjun, H. (2018). *Structures*, **135**, 233–244.
- Shaw, A. & Kyriakides, S. (1997). *Int. J. Plast.* **13**, 837–871.
- Šittner, P., Heller, L., Pilch, J., Curfs, C., Alonso, T. & Favier, D. (2014). *J. Mater. Eng. Perform.* **23**, 2303–2314.
- Šittner, P., Liu, Y. & Novak, V. (2005). *J. Mech. Phys. Solids*, **53**, 1719–1746.
- Šittner, P., Lukáš, P., Novák, V., Daymond, M. R. & Swallowe, G. M. (2004). *Mater. Sci. Eng. A*, **378**, 97–104.
- Šittner, P., Neov, D., Lukas, P. & Toebbens, D. M. (2004). *J. Neutron Res.* **12**, 15–20.
- Stebner, A., Paranjape, H., Clausen, B., Brinson, L. & Pelton, A. (2015). *Superelasticity*, **1**, 252.
- Stebner, A. P., Vogel, S. C., Noebe, R. D., Sisneros, T. A., Clausen, B., Brown, D. W., Garg, A. & Brinson, L. C. (2013). *J. Mech. Phys. Solids*, **61**, 2302–2330.
- Sumin, V. V., Papushkin, I. V., Vasin, R. N., Venter, A. M. & Balagurov, A. M. (2012). *J. Nucl. Mater.* **421**, 64–72.
- Sutou, Y., Omori, T., Yamauchi, K., Ono, N., Kainuma, R. & Ishida, K. (2005). *Acta Mater.* **53**, 4121–4133.
- Tan, G. & Liu, Y. (2004). *Intermetallics*, **12**, 373–381.
- Thomasová, M., Seiner, H., Sedlák, P., Frost, M., Ševčík, M., Szurman, I., Kocich, R., Drahokoupil, J., Šittner, P. & Landa, M. (2017). *Acta Mater.* **123**, 146–156.
- Waitz, T., Antretter, T., Fischer, F. D. & Karnthaler, H. P. (2008). *Mater. Sci. Technol.* **24**, 934–940.
- Wang, L., Ma, L., Liu, C., Zhong, Z. Y. & Luo, S. N. (2018). *Mater. Sci. Eng. A*, **718**, 96–103.
- Wang, Z., Lynch, P. A., Kada, S. R., Armstrong, N., Motlagh, E. B., Kimpton, J. A. & Barnett, M. R. (2018). *J. Appl. Cryst.* **51**, 1082–1093.
- Wenk, H.-R. & Grigull, S. (2003). *J. Appl. Cryst.* **36**, 1040–1049.
- Wenk, H.-R., Lutterotti, L., Kaercher, P., Kanitpanyacharoen, W., Miyagi, L. & Vasin, R. (2014). *Powder Diff.* **29**, 220–232.
- Xiao, Y., Zeng, P., Lei, L. & Zhang, Y. (2017). *Mater. Des.* **134**, 111–120.
- Xie, X., Kan, Q., Kang, G., Lu, F. & Chen, K. (2016). *Mater. Sci. Eng. A*, **671**, 32–47.
- Yawny, A., Sade, M. & Eggeler, G. (2005). *Z. Metallkd.* **96**, 608–618.
- Ye, B., Majumdar, B. S. & Dutta, I. (2007). *Appl. Phys. Lett.* **91**, 061918.
- Yinong, L., Houver, I., Xiang, H., Bataillard, L. & Miyazaki, S. (1999). *Metall. Mater. Trans. A*, **30**, 1275–1282.
- Young, M. L., Wagner, M. F. X., Frenzel, J., Schmahl, W. W. & Eggeler, G. (2010). *Acta Mater.* **58**, 2344–2354.
- Zhang, X., Feng, P., He, Y., Yu, T. & Sun, Q. (2010). *Int. J. Mech. Sci.* **52**, 1660–1670.
- Zheng, L., He, Y. & Moumni, Z. (2017). *Int. J. Plast.* **90**, 116–145.
- Zotov, N., Pfund, M., Polatidis, E., Mark, A. F. & Mittemeijer, E. J. (2017). *Mater. Sci. Eng. A*, **682**, 178–191.