Deformation mechanisms in a superelastic NiTi alloy: An in-situ high resolution digital image correlation study

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HIGHLIGHTS
• High-resolution digital image correlation is used to unveil the deformation mechanisms that occur in superelastic NiTi.
• Deformation mechanisms: those resulting in non-recoverable strain and martensite formation resulting in recoverable strain.
• Good agreement with Schmid law for the martensite transformation and with the theoretical transformation strain is seen.

GRAPHICAL ABSTRACT

ABSTRACT
An in-situ high resolution digital image correlation investigation during uniaxial tensile deformation reveals the recoverable and the non-recoverable strain mechanisms in a Ni51Ti49 alloy with a mean grain size of 35 μm. Recoverable strain is due to the martensitic transformation, for which more than one variant per grain can be activated. The majority of the activated variants exhibit high Schmid factor. The variant selection can be influenced by shear transmission across grain boundaries, when the geometrical compatibility between the neighboring habit plane variants is favourable; in these cases variants that do not have the highest Schmid factor, with respect to the macroscopically applied load, are activated. The experimentally determined transformation strains agree well with theoretical calculations for single crystals. The non-recoverable strain is due to deformation slip in austenite, twinning in martensite and residual martensite. The results are discussed in view of possible twinning modes that can occur in austenite resulting in significant non-recoverable strain.

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1. Introduction
NiTi alloys in near-equiatomic compositions are well known for their superelasticity which results from the reversible austenite-martensite transformation. These alloys can be found in various applications including biomedical [1], actuators and robotics [2]. The deformation behaviour of NiTi strongly depends on the crystallographic orientation...
with respect to the loading direction in single crystals [3,4], and the
crystallographic texture in polycrystalline alloys [5]. The martensitic
variants have been determined by the characterization of the shear
bands and their inclination with respect to the loading direction in com-
bination with the Schmid factor. Such experiments have been carried
out under tension, compression [6,7] and nanoin dentation [8] using
SEM/EBSD. During loading, martensite variants with the highest Schmid
factor are first activated [5], but usually more than one martensite vari-
ant may form in each austenite grain [7]. During reverse transforma-
tion, both favourable and less-favourable variants contribute to the
recovered strain [5].

TEM results have shown that dislocations slip, twinning and residual
martensite are responsible for non-recoverable strains and loss of
superelasticity [9–16], though, how these deformation mechanisms co-
operate at the mesoscopic level, where grain-to-grain interactions can
affect the activation of these mechanisms, is not clear. HR-DIC in SEM
has recently become a powerful tool with sub-micron spatial resolution
to study the activation of slip in various steels [17–22], Ti-alloy [23], Mg
alloys [24] and the fracture mechanics in oxide layers [25], Conventional
DIC [26–28] and HR-DIC [29,30] have also been applied to investigate
the martensitic transformation in NiTi. However, the low density of
the DIC speckle pattern did not allow achieving the necessary resolution
for characterizing the martensite variants and the reversible transforma-
tion strain of individual variants. Recently HR-DIC and EBSD have
been combined to study the martensitic transformation during multiax-
ial loading and strain path changes in a superelastic NiTi alloy having a
microstructure composed of nanocrystalline substructures [31]. By cor-
relating HR-DIC strain maps to the EBSD data, it was found that bands of
nanocrystalline austenite grains transform collectively into martensite.
HR-DIC can quantify the strain magnitude of the austenite to mar-
tensite transformation and the recoverable strain upon unloading. Com-
paring recoverable and non-recoverable strain allows the identification
of the coexisting deformation mechanisms in addition to the martens-
itic transformation, such as slip [12] or deformation twinning
[13–16,32,33]. This information is essential for developing and
informing existing phase-field models that incorporate mechanisms of
multi-variant phase transformations and poly twinning [34–37].

The objective of this study is to perform HR-DIC on a coarse-grained
superalastic NiTi alloy to reveal the deformation mechanisms resulting
in recoverable and non-recoverable strain. Using a dense silica speckle
pattern, HR-DIC allows the characterization of the activated habit
plane variants, the slip planes and the deformation twinning. Moreover,
it allows the direct measurement of the transformation strain originat-
ing from the activated variants. Such information is essential for im-
proving the analytical tools for predicting the mechanical behaviour of
NiTi that up to now rely on simplified assumptions for the
micromechanical response of the material.

2. Materials and methods

2.1. Experimental procedures

A rectangular ingot (20 × 76 × 90 mm3) with nominal composition
Ni51Ti49 (in at.%) was produced by vacuum induction melting using
nickel (Ni purity > 99.98 wt%) and titanium (Ti purity > 99.995 wt%) at
the Ruhr-University Bochum (Germany). Melting was performed in
a graphite crucible using a VSG 010 furnace from PVA TePla AG in a
high-purity Ar Atmosphere (99.998 vol%) under a pressure of
500 mbar. The melt was then poured into a pre-heated mould
(500 °C) that had been coated with an yttria slurry. The ingot was solu-
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2.1. Experimental procedures
2. Materials and methods

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sample might contain thickness variations, which cause local stress concentrations and locally high strains possibly beyond the superelastic limit of the material. As the material is seen to exhibit significant non-recoverable strain, the latter probably occurs, and it is further discussed in Section 4.2. The images for the HR-DIC analysis were acquired with a Zeiss ULTRA 55 FEG-SEM using an in-lens secondary electron detector at 6.9 mm working distance with 3 kV acceleration voltage and an aperture size of 30 μm. A series of SEM images with a resolution of 3072 × 2304 pixels were taken at ×2000 magnification and with 25 μsec dwell time. The Ncorr MATLAB code [51] was used for the HR-DIC analysis using a subset size of 15 pixels and subset spacing of 1 pixel. Previous HR-DIC measurements [21] have shown that the error due to drift and lens distortion is insignificant, compared to the magnitude of strains of slip and transformation bands, and therefore no corrections have been applied. The identification of the grain boundaries on the HRDIC map is challenging due to the high deformation and rotation of the grains. This is done using the position of the grain boundaries in the undeformed sample obtained from EBSD and the usage of the open source python code, DefDap [52].

The surface of the unloaded dogbone was re-polished using diamond paste (with particle size of 1 μm and 0.25 μm) followed by a final vibration polishing for 24 h in order to smoothen the deformation-induced surface relief and to achieve better surface quality for post-mortem EBSD investigations. Although some material was removed from the surface, the same grains as seen with HR-DIC could be observed the post-mortem in EBSD investigation.

Transmission electron microscopy (TEM) characterization was undertaken using a JEOL-2010 microscope operated at 200 kV. TEM sample was prepared by extracting a rectangular lamella from the surface plane in grain 1 as shown in Fig. 1f, and milling it using a focused ion beam (FIB) on a Zeiss NVision-SEM/FIB. The FIB-milling was undertaken using a 30 kV and 3 nA FIB. After the lift-out, the lamella was thinned by FIB using 30 kV and successively 700 pA, 300 pA and 80 pA current as the thickness was reducing. The final surface cleaning was done using a 5 kV and 100 pA FIB.

2.2. Habit plane traces analysis

Upon loading, the austenite (cubic B2 phase, ordered BCC) transforms into twinned martensite (monoclinic B19° phase) bands where the interface between the austenite and the twinned martensite is an invariant plane also called the habit plane. In the present work, we consider only type II twinning, since the latter has been found to be a dominant twinning mode in SE NiTi alloys under deformation [11]. This type of twinning has also been seen to agree well with the habit-plane variants (HPVs) in polycrystalline materials under deformation [4,6–8]. The HPVs, also called correspondent variant pair (CVP), of type II twinning \([0.8684 0.2688 0.4138] - [0.4580 0.7706 0.4432]\) are listed in Table 1 using the experimentally determined systems in [53]. Note the that indices of the habit plane and those of the twinning shear direction were normalized and that they are all expressed in the coordinate system of the B2 austenite. It is also noted that although the HPVs in Table 1 were identified in single crystals for thermally-induced martensite [54], they agree well with the observed stress-induced HPVs in [6–8].

The inclination of the habit plane trace with respect to the loading direction can be calculated as follows [7]:

The habit plane and the transformation shear direction \(b\) of the shape strain is given in the form:

\[
(h k l)_{\alpha, \text{a}} |u v w|_{\alpha, \text{a}}\]

where \(\alpha = 0, 1, 2, \ldots, 24\), for the 24 possible habit plane variants. The orientation of the parent B2 phase is given in the form: \([HKL]_{\text{b}} |U V W|_{\text{b}}\), for the \(\beta\)-th grain of a polycrystal, where \([H K L]\) and \([U V W]\) are the normal direction of the grain and the tensile direction of the grain, respectively. A list of the normal and the loading directions for all the investigated grains is given in Table S1 of the Supplementary material. The habit plane trace direction of the \(\alpha\)-th variant in the \(\beta\)-th grain is then given by the cross product [7,55]:

\[
\left[ R^{\beta} S^{\alpha}_{\text{HKL}} T^{\alpha}_{\text{b}} \right] = [hikl]_{\beta} \times [HKL]_{\beta}
\]
where $\mathbf{R}$, $\mathbf{S}$, $\mathbf{T}$ are the indices of the habit plane trace direction. The angle $\phi_{ij}^n$ between the habit plane trace of the $\alpha$-th variant and the loading direction in the $\beta$-th grain is given by [7]:

$$
\phi_{ij}^n = \cos^{-1}\left(\frac{\mathbf{R}_{ij}^n \mathbf{U}_j + \mathbf{S}_{ij}^n \mathbf{V}_j + \mathbf{T}_{ij}^n \mathbf{W}_j}{\sqrt{\left(\mathbf{R}_{ij}^n \mathbf{U}_j\right)^2 + \left(\mathbf{S}_{ij}^n \mathbf{V}_j\right)^2 + \left(\mathbf{T}_{ij}^n \mathbf{W}_j\right)^2}}\right)
$$

The same approach can be used to calculate the trace inclination of a slip system where instead of the habit plane the slip plane is used in the optimum superelastic microstructure consists of sub-grain structures.

### Table 1

<table>
<thead>
<tr>
<th>Variant, notation from ([54])</th>
<th>Habit plane</th>
<th>Invariant shear direction</th>
</tr>
</thead>
<tbody>
<tr>
<td>1(+), -1(+)</td>
<td>(0.8684 0.2688 -0.4138)</td>
<td>[-0.458 0.7706 -0.4432]</td>
</tr>
<tr>
<td>1(-), -1(-)</td>
<td>(0.8684 0.4138 -0.2688)</td>
<td>[-0.458 0.4432 -0.7706]</td>
</tr>
<tr>
<td>1(-), -1(-)</td>
<td>(0.8684 0.2688 -0.4138)</td>
<td>[0.458 0.7706 -0.4432]</td>
</tr>
<tr>
<td>2(+), -1(+)</td>
<td>(0.8684 0.4138 0.2688)</td>
<td>[-0.458 0.7706 -0.4432]</td>
</tr>
<tr>
<td>2(-), -1(-)</td>
<td>(0.8684 0.4138 0.2688)</td>
<td>[-0.458 0.4432 -0.7706]</td>
</tr>
<tr>
<td>2(-), -1(-)</td>
<td>(0.8684 0.2688 0.4138)</td>
<td>[0.458 0.7706 -0.4432]</td>
</tr>
<tr>
<td>3(+), -1(+)</td>
<td>(0.8684 0.2688 0.4138)</td>
<td>[-0.4432 -0.458 0.7706]</td>
</tr>
<tr>
<td>3(-), -1(-)</td>
<td>(0.2688 -0.8684 -0.4138)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
<tr>
<td>3(-), -1(-)</td>
<td>(0.2688 0.8684 -0.4138)</td>
<td>[0.7706 -0.458 -0.4432]</td>
</tr>
<tr>
<td>4(+), -1(+)</td>
<td>(0.4138 0.8684 0.2688)</td>
<td>[0.458 -0.458 0.7706]</td>
</tr>
<tr>
<td>4(-), -1(-)</td>
<td>(0.4138 0.8684 0.2688)</td>
<td>[0.458 -0.458 0.7706]</td>
</tr>
<tr>
<td>5(+), -1(+)</td>
<td>(0.2688 -0.8684 0.4138)</td>
<td>[0.7706 -0.458 0.4432]</td>
</tr>
<tr>
<td>5(-), -1(-)</td>
<td>(0.4138 -0.8684 0.2688)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
<tr>
<td>5(-), -1(-)</td>
<td>(0.2688 0.4138 0.8684)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
<tr>
<td>6(+), -1(+)</td>
<td>(0.2688 0.4138 0.8684)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
<tr>
<td>6(-), -1(-)</td>
<td>(0.4138 0.2688 -0.8684)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
<tr>
<td>6(-), -1(-)</td>
<td>(0.2688 0.4138 -0.8684)</td>
<td>[0.7706 0.458 -0.4432]</td>
</tr>
</tbody>
</table>

where $\varepsilon_{ij}^n$ is the crystallographic direction of the loading axis in the $\beta$-th grain [57]. Eq. (4) has been applied to each grain, $\beta$, to calculate the transformation strain that can be recovered for each habit plane variant $\alpha$. It is also worth noting that, as a first order approximation, neglecting the volume change associated with the austenite to martensite transformation, the transformation strain is directly proportional to the SF [58], i.e., $\varepsilon = SF \cdot |b|$ where $|b|$ is the magnitude of the transformation shear which is equal to 0.13 for type II twinning.

### 3. EBSD and HR-DIC results

The initial microstructure with equiaxed grains with an average grain size of ~35 μm is shown in Fig. 1a–d. The grain orientation maps (Fig. 1a and b) reveal a relatively strong (111)-out of plane texture, as also reported in [38]. Additionally, there is a relatively strong alignment of the (101) direction parallel to the loading direction, see Fig. 1b. Fig. 1d shows the HR-DIC map of the deformed material at 10% strain averaged over the field of view. The grain boundaries are delineated with white lines for clarity.

As shown in Fig. 1d, the strain is accommodated heterogeneously in traces of high strain. The localized strain traces resemble the deformation bands indexed as martensite by EBSD [6,7]. The regions with low homogeneous strain are possibly retained austenite as also observed by EBSD in [6,7]. Some grains (e.g., G3, G11 and G14) exhibit high and more homogeneous strain where possibly the transformation is relatively homogenous and covers the larger area of the grains. Fig. 1e shows that significant strain is recovered upon unloading implying that the martensitic transformation is reversible, however, residual strain is also significant as seen in Fig. 1f implying that non-recoverable deformation mechanisms are operational. The observed strain traces in each labelled grain is illustrated in Fig. 1c with red lines. Besides strain traces in the grains, some amount of strain is accommodated close to the grain boundaries, see e.g., the boundary between grains G4, G8, G10 and G11, as shown in Fig. 1d.

### 4. Discussion of the deformation mechanisms

Recoverable strain is expected due to the reversible austenite-martensite phase transformation, which results in the superelasticity of the material. The presence of significant non-recoverable strain is rather unexpected considering the relatively good superelasticity shown in Fig. S1 of the Supplementary material. This can be possibly caused by small variations in thickness or width of the HR-DIC sample (Fig. S2) causing stress concentrations and local plastic deformation. Since the material locally exceeds its superelastic capability, besides the martensite formation, plastic deformation mechanisms occur. The recoverable strain traces (Fig. 1e) and non-recoverable strain traces (Fig. 1f) are discussed in detail in the following paragraphs. It should be noted that the microstructure consists of large equiaxed grains, of the order of tens of micrometres, which cannot exhibit the excellent superelastic cyclic behaviour of optimized NiTi microstructures. An optimum superelastic microstructure consists of sub-grain structures of the order of nanometres in size [12,31,59].

#### 4.1. Recoverable strain mechanism

##### 4.1.1. Reversible martensitic transformation

Table 2 lists the observed trace inclinations and the theoretical trace inclinations for the potentially activated habit plane variants (HPVs). It is seen that the variant selection is dependent on the orientation of the austenite grain. The theoretical transformation strains are also given in Table 2 and will be discussed in the following paragraphs. The experimental transformation strain is determined by subtracting the average strain within the strain traces (averaged using image) in the unloaded state (shown in Fig. 1f) from the strain within the traces at maximum load (shown in Fig. 1d). As an example, grain G3 exhibits traces with an inclination of 153 ± 3° with respect to the loading direction. The HPV 4(+) with the highest positive SF (SF ≈ 0.41) would result in a trace with an inclination of 152° with respect to the loading direction and theoretical transformation strain of 5.2%. The experimentally determined transformation strain is 5.6%. Note that the variant with the highest positive SF should form under tension, whereas the
variant with the most negative SF should form during compression [60]. It can be thus concluded with confidence that some of the traces observed in grain G3 may be due to the activation of the HPV 4(+) . There are two reasons for that: (i) the theoretical trace inclination of HPV 4(+) agrees well with the observed one (by HR-DIC) and more importantly (ii) its theoretical transformation strain agrees very well with that determined by HR-DIC, see Table 2. If the specific trace would be ascribed to e.g. slip only, criterion (ii) would not be fulfilled. Since in the majority of the grains, a strain recovery could be detected completely vanish after unloading (shown with red arrows) which can only be ascribed to the presence of reversible martensite. Moreover, Mao et al. identified similar plates appearing in a superelastic NiTi alloy with large grains as martensite using EBSD [7]. As shown in Table 2, the locally recovered strains determined by HR-DIC are in good agreement with the theoretical transformation strains calculated for the formation of HPV's in single crystals. The transformation strain including detwinning of the initially twinned HPV under tension (the so-called transformation into lattice correspondent variants - LCVs), is about 10% for the crystallographic orientations of the investigated grains, which is higher than the measured recoverable strain. Therefore, the measured transformation strain agrees better with the predicted transformation strain originating from the formation of HPV's rather than LCVs. It should be noted that in the present study the grain size of 35 μm is large compared to the thickness of the sample (80 μm) and therefore constraints normally occurring in a polycrystal might be relaxed to some extent [50]. Hence, it is not surprising that the behaviour of each individual grain approaches the behaviour of a single crystal. In some grains (e.g. G8 and G16), the measured recoverable strain is lower than the theoretical transformation strain possibly due to partial reversibility of the stress-induced martensite.

In grains G1, G2, G3, G4, G5, G10 and G15, only the variant with the highest SF is found to be activated. In contrast, other grains (G7, G9, G11, G12, G13, G14, G16) exhibit two or more variants and in most cases include the highest-Schmid-factor HPVs. The activation of multiple HPVs in the same grain is in contrast with previous analytical studies [5,61] where the existence of only one martensite variant (with the highest SF) was considered. This led to an overestimation of the calculated transformation strain, since variants with highest SF exhibit the highest transformation strain, as discussed in Section 2.3. In summary, the recoverable strain is due to the reversible martensitic transformation. Despite grain interactions that cause multiaxial stress states at the intragranular lengthscale, the two other principal stress components are negligible compared to the applied stress component [49]. Therefore, the activation of the HPVs can be well predicted by application of the Schmid law assuming uniaxial tension, which is in good agreement with [6,7].

### 4.1.2. Non Schmid behaviour and variant transfer across grain boundaries

Grains G6, G9, G12 and G16 exhibit a deviation from the Schmid law in some grains (e.g. G8 and G16), the measured recoverable strain is lower than the theoretical transformation strain possibly due to partial reversibility of the stress-induced martensite.

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meanwhile maintaining coherency at the grain boundaries during homogeneous deformation of polycrystalline materials [62]. This approach has until now only been used to explain the transfer of twinning across grain boundaries in Mg alloys [63,64] or Ti alloys [65]. This factor, designated as $m'$, can be calculated from the knowledge of two angles: the angle, $\theta$, between the shear directions in grains I and II, and the angle, $\psi$, between the normal to the habit planes.

$$m' = \cos \theta \cos \psi$$

Table 3 lists the active variants of the neighboring grains, and the compatibility factor between the activated habit plane variants for the grains that show variants deviating from the Schmid law. For comparison, the compatibility factor between the highest SF variants and the active variants of the neighboring grains are given. It is observed that in two cases (grains G6 and G9) the variant transmitted through the boundary from the neighboring grain results in perfect compatibility, i.e., $m' = 1$. The activated “non Schmid” variant and the variant of the neighboring grain in G12 and G16 exhibit higher compatibility than the non-activated variants with the highest SF. In all cases however the SF values of the activated variants is relatively high (3rd or 5th highest out of the 24), as the resolved shear stress on the habit plane needs to be high enough.

The transmitted variants in G8-G16 are examples of variants with relatively low geometrical compatibility. The HPV pair \(4'(+)\) and \(3(-)\) in grains G8-G16 exhibits lower geometrical compatibility ($m' = 0.16$) than the HPV pair with the highest SF, i.e. \(4'(+)\) and \(3(-)\) ($m' = 0.4$). As mentioned already before, the behaviour of the grains agrees with the Schmid law assuming a pure uniaxial stress state in the grains, however, the small deviations on this rule can be explained by local stress concentrations or appearance of multiaxial stress state in the grain level.

### 4.2. Non-recoverable strain mechanisms

#### 4.2.1. Slip in austenite

The significant non-recoverable strain after unloading indicates the loss of superelasticity which is detrimental for the cycling stability of this material. Slip is one of the possible deformation mechanisms contributing to the non-recoverable strain after complete unloading.

Table 4 lists for each austenite grain the observed non-recovered trace inclinations (shown in Fig. 1f) with respect to the loading direction and the expected trace inclinations for the (110)(100) B2 slip system with the highest SF [66].

In Fig. 1f, grain G1 exhibits residual strain traces inclined $151 \pm 2^\circ$ with respect to the loading direction. The slip system with the highest SF ($SF \approx 0.40$) would give a trace of $148^\circ$ with respect to the loading direction. The second highest SF slip system ($SF \approx 0.38$) has a trace of $123^\circ$ with respect to the loading direction. Therefore, the non-recoverable traces observed in G1 with an inclination of $151 \pm 2^\circ$ can partially correspond to dislocation slip of the system with the highest SF.

In grain G5, the traces are hardly recovered (see Fig. 1d–f) and the trace inclination agrees relatively well with (110)[001] dislocation slip (see Table 4). Grain G5 has a significantly different crystallographic orientation compared to the rest of the grains. Its orientation, along the loading direction is close to (001), which is not favourable for the formation of stress induced martensite [60], thus slip seems to be the dominant mechanism despite that some recovered strain can also be attributed to the activation of one HPV variant (with the highest SF).

In grains G1, G2, G3, G4, G5, G8 and G13 the observed trace inclinations can be correlated with those belonging to the potential slip systems with one of the highest SFs (ranked first to third) with a tolerance of $\pm 5^\circ$, but no correlation can be found for the traces seen in grains G6, G7, G9, G10, G11, G12; the irreversible strain in these grains can be due to other mechanisms that are discussed in the following sections. In summary, deformation by (110)[100] B2 slip contributes to the irreversible strain shown in Fig. 1f.

#### 4.2.2. Deformation twinning

Looking closer to the strain traces in the HR-DIC maps, Fig. 3a and b show an example of the evolution of the trace morphology in G1 upon straining between 8% and 10% average strain. The straight bands at 8% average strain, develop to a “zig-zag” structure when the strain in the FOV increases to 10%, as shown in Fig. 3a. Similar “zig-zag” structures were also observed in several other grains such as in G2, G7, G9. The appearance of “zig-zag” structures and the significant increase of the strain, within the initially low strain trace due to martensite formation, suggests that an additional deformation mechanism succeeds the activation of martensite. After unloading...
TEM studies support that {114} austenite twinning appears after deformation [70,71]. Ezaz et al. [68] show the angle between two {114} bands is ~17°. As for {114} B2T, the theoretical angle between two {114} planes is ~33.6°, which suggests that the angle between the two {114} planes should be ~17°. As for {114} martensite twinning, the theoretical angle between two {114} orientations is ~21°. As seen in Fig. 4c, the misorientation between two regions forming a zig-zag pattern is ~21°.

Table 4

<table>
<thead>
<tr>
<th>Grain</th>
<th>Angle of trace with respect to loading direction from HR-DIC (degrees)</th>
<th>Highest SF slip system, SF value</th>
<th>Theoretical trace angle of highest SF (degrees)</th>
<th>Possible activated variant rank of SF</th>
</tr>
</thead>
<tbody>
<tr>
<td>G1</td>
<td>151 ± 2</td>
<td>(0–11)[100], SF = 0.399</td>
<td>148</td>
<td>1st</td>
</tr>
<tr>
<td>G2</td>
<td>143 ± 2</td>
<td>(011)[100], SF = 0.377</td>
<td>135</td>
<td>2nd</td>
</tr>
<tr>
<td>G3</td>
<td>153 ± 3</td>
<td>(0–11)[100], SF = 0.336</td>
<td>154</td>
<td>1st</td>
</tr>
<tr>
<td>G4</td>
<td>37 ± 1</td>
<td>(0–11)[100], SF = 0.399</td>
<td>54</td>
<td>2nd</td>
</tr>
<tr>
<td>G5</td>
<td>155 ± 2</td>
<td>(0–10)[00–1], SF = 0.388</td>
<td>39</td>
<td></td>
</tr>
<tr>
<td>G6</td>
<td>50 ± 1</td>
<td>(011)[100], SF = 0.438</td>
<td>101</td>
<td>No correlation</td>
</tr>
<tr>
<td>G7</td>
<td>141 ± 3</td>
<td>(0–11)[100], SF = 0.334</td>
<td>156</td>
<td>3rd</td>
</tr>
<tr>
<td>G8</td>
<td>29 ± 4</td>
<td>(0–11)[100], SF = 0.354</td>
<td>27</td>
<td>1st</td>
</tr>
<tr>
<td>G9</td>
<td>42 ± 1</td>
<td>(011)[100], SF = 0.437</td>
<td>36</td>
<td>3rd</td>
</tr>
<tr>
<td>G10</td>
<td>160 ± 3</td>
<td>(010)[001], SF = 0.416</td>
<td>66</td>
<td></td>
</tr>
<tr>
<td>G11</td>
<td>33 ± 4</td>
<td>(0–11)[100], SF = 0.248</td>
<td>156</td>
<td>No correlation</td>
</tr>
<tr>
<td>G12</td>
<td>127 ± 1</td>
<td>(0–11)[100], SF = 0.372</td>
<td>23</td>
<td>No correlation for the second trace</td>
</tr>
<tr>
<td>G13</td>
<td>34 ± 2</td>
<td>(011)[100], SF = 0.417</td>
<td>34</td>
<td>1st</td>
</tr>
<tr>
<td>G14</td>
<td>32 ± 3</td>
<td>(0–11)[100], SF = 0.271</td>
<td>133</td>
<td>4th</td>
</tr>
<tr>
<td>G15</td>
<td>147 ± 4</td>
<td>(0–11)[100], SF = 0.382</td>
<td>137</td>
<td></td>
</tr>
<tr>
<td>G16</td>
<td>33 ± 1</td>
<td>(011)[100], SF = 0.408</td>
<td>35</td>
<td>2nd</td>
</tr>
<tr>
<td>G17</td>
<td>27 ± 1</td>
<td>(011)[100], SF = 0.376</td>
<td>124</td>
<td>2nd</td>
</tr>
<tr>
<td>G18</td>
<td>148 ± 3</td>
<td>(0–10)[00–1], SF = 0.369</td>
<td>143</td>
<td></td>
</tr>
</tbody>
</table>

in G1, two regions within the “zig-zag” structure are indexed as austenitic phase with high confidence using EBSD. In addition to the matrix, two distinctly new austenite orientations appear, i.e. three austenite orientations exist in the grain. These regions within the “zig-zag” morphology have crystallographic orientations different from the initial parent grain, as shown in Fig. 3c, implying that a twinning mechanism occurs in austenite upon unloading. The noise seen as spots with different colours in Fig. 3c can be attributed to unindexed patterns. Due to overlapping interfaces of the two different austenite orientations, local high deformation at the interface of these bands and/or possible presence of small fractions of martensite, the indexing of the Kikuchi patterns is impossible.

The formation of twinned austenite, hereafter denoted as B2T via the sequence B2–B19′–B2′, has been a topic of recent interest [13–16,67–69]. Similar microstructure reported in the literature is a result of either [112] austenite twins inherited from [113] martensite twins [69] or [114] austenite twins [13–16,70] inherited from [201] martensite twins [70,71]. Ezaz et al. [68] showed that the [201] martensite twinning induced at early stages of plastic deformation of martensite, while [113] martensite twinning occurs at later stages of deformation or severe plastic deformation [69]. A recent series of comprehensive TEM studies supports that [114] austenite twinning appears after deformation beyond the yield point of martensite [13–16]. Another investigation claims that the appearance of [112] austenite twin is a misinterpretation of [114] twinning [72] while Goo et al. evoke the possibility of formation of disordered [112] twinning in B2, the so-called [112] pseudo twins [73].

Assuming [112] B2′, the theoretical angle between two [112] planes bounding the “zig-zag”, i.e. (112) and (121), should be ~33.6° and the projection of this angle on the sample surface should be ~17°. As for [114] B2′, the theoretical angle between two [114] planes bounding the “zig-zag”, i.e. (114) and (141), should be ~27.3° and the projection of this angle on sample surface should be ~23°, as shown in Fig. 3. The inclination between the traces observed on the EBSD map shown in Fig. 3c is in relatively good agreements (within ±3°) with both [112] B2′ and [114] B2′. The misorientation measured along the line drawn across the “zig-zag” bands in Fig. 3c correspond to 35° and 15° misorientation with respect to the parent grain, i.e. a misorientation of ~20° between the two regions forming the “zig-zag”.

In Fig. 4a shows a collection of bright field TEM images of the twin-related austenite microstructure (zig-zag features) within the previously transformed zone (HPV martensite). The red dashed lines indicate the interface of the austenite matrix and the twinned region, which is recognized as the HPV traces of either 4′(+) or 4′(−) (see Table 2). In the vicinity of the austenite/martensite boundary, significant diffraction contrast is due to dislocations formed during austenite slip as well as the martensitic transformation. Selected area diffraction patterns (SADP) taken outside and inside the “zig-zag” area (Fig. 4b and d–e) show three different austenitic orientations. Fig. 4b is a SADP of the parent grain in a [113]-type zone axis. While moving on the “zig-zag” bands without tilting, the SADP is no longer in a [113]-type zone axis, suggesting a misorientation with respect to the parent grain. Fig. 4d–e show the SADPs of the two observed new orientations (the two “zig-zag”), which are ~21° misoriented to each other, confirming the existence of twinned austenite.

The material in the present study was not severely deformed and the non-recoverable strain possibly occurs from stress concentrations due to variations in the sample cross-section area. It appears hence that the most possible deformation mechanism is [114] B2′ inherited from [201] martensite twins which only appear at early stages of martensitic deformation. The exact type and the mechanism of the B2′ formation...
Fig. 3. Local strain maps obtained in grain G1 showing the evolution of the same bands at average strains of (a) 8% and (b) 10%. The dark red lines correspond to strain values more between 0.3 and 0.4 strain. The transformation of habit plane variants into “zig-zag” morphologies is seen by comparing (b) to (a). The white dashed line in (b) shows the approximate location of EBSD, after some gentle polishing. (c) EBSD map of the same bands in the same grain G1 (out of plane projection) after unloading showing that the “zig-zag” structure comprises of two austenite regions with different orientations (“pink” - (327) and “blue” - (545)) with respect to the parent grain orientation (“purple” - (658)). A line scan (rainbow line) from the parent grain along the “zig-zag” structure shows 35° and 15° misorientation with respect to the orientation of the parent phase. The traces of the (112) or (141) and (121) or (141) planes are shown in (c) and align well with the “zig-zag” bands. The loading direction corresponds to the y-axis.

Fig. 4. (a) Montage of bright field TEM images showing the “zig-zag” structure which formed by deformation twinning inside a martensite variant (either 4′(+) or 4′(-)). (b) SADP of the parent grain, taken outside the “zig-zag” structure shown in (a). (c) Details of the area indicated with red square in (a) showing the “zig-zag” structure where two austenite orientations can be recognized. The corresponding diffraction patterns of these two austenite orientations are shown in (d) and (e), respectively, where a 20° misorientation can be measured. (f) Dark field image showing retained martensite as bright structures. The red circle in (f) indicates the position from where the SADP is taken. The spot used for the dark field imaging is highlighted with a green circle in (g). The loading direction corresponds to the y-axis.
requires further investigations which are currently not the target of this study.

Finally, TEM reveals the presence of small fractions of retained martensite as shown in Fig. 4f-g. Fig. 4f is a dark-field TEM image using the spot indexed as (011) martensite in Fig. 4g. Martensite appears to be locked in the microstructure at the intercepts of the “zig-zag” bands in good agreement with [13,16], however it is interestingly low in fraction. The small fraction of residual martensite is below the EBSD detection limit and therefore the “zig-zag” bands where indexed as fully austenitic. Residual martensite is another mechanism for accumulating residual strain.

In summary, part of the non-recoverable strain is due to residual martensite and due to the occurrence of austenite twinning upon reverse phase transformation, which is in good agreement with [13]. The fact that the “zig-zag” traces appear in bands already correlated to HPVs suggests that the austenite twinning is inherited from twinning in martensite, i.e. following the deformation sequence: B2 → B19 → B2.

5. Conclusions

HRDIC in combination with EBSD is capable of capturing the recoverable and non-recoverable strain. In combination with TEM, the operation of multiple deformation mechanisms in a superelastic coarse-grained NiTi is revealed. Analysis based on the recoverable strain and trace inclination shows the activation of habit plane variants of martensite are detected, contributing to the non-recoverable strain. The coexistence of stress-induced martensite, slip and twinning in martensite/austenite results in deformation bands with high axial strain.

CRediT authorship contribution statement

E. Polatidis: Investigation, Data curation, Formal analysis, Writing - original draft. M. Smid: Investigation, Data curation, Formal analysis, Writing - review & editing. I. Kubena: Investigation, Data curation, Formal analysis, Writing - review & editing. W.-N. Hsu: Investigation, Data curation, Formal analysis, Writing, review & editing. G. Laplanche: Resources, Writing - review & editing. H. Van Swygenhoven: Supervision, Funding acquisition, Writing, review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

EP, MS, IK, W-NH and HVS thank the European Research Council for the ERC-advanced Grant MULTIAAX (339245). IK acknowledges the Ministry of Education, Youth and Sports of the Czech Republic for the project m-IPMinfra (CZ.02.1.01/0.0/0.0/16_013/0001823). The infrastructure of IPMinfra were used for the TEM investigations. GL acknowledges funding from the Alexander von Humboldt Foundation. The authors acknowledge Dr. Rolf Brönnimann (EMPA, Dübendorf) for the assistance in the sample preparation with the picosecond laser. 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.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.matdes.2020.108622.

References
