Challenges During Microstructural Analysis and Mechanical Testing of Small-Scale Pseudoelastic NiTi Structures

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Published online: 23 March 2016
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Abstract Most investigations on NiTi-based shape memory alloys involve large-scale bulk material; knowledge about the martensitic transformation in small-scale NiTi structures is still limited. In this paper, we study the microstructures of thin NiTi layers and their mechanical properties, and we discuss typical challenges that arise when experiments are performed on small samples. A physical vapor deposition (PVD) process was used to deposit thin NiTi wires with a cross section of $15 \times 15 \mu\text{m}^2$ and dogbone-shaped samples $5 \times 500 \mu\text{m}^2$. Microstructural properties were characterized by X-ray diffraction, electron backscatter diffraction, and scanning electron microscopy. Moreover, tensile tests were performed using optical strain measurements in order to observe martensite band formation during cyclic loading. The surfaces of the crystalline wires reflect the columnar growth of NiTi during deposition. The wires exhibit pseudoelastic material behavior during tensile testing. Fracture typically occurs along the columns because the column growth direction is perpendicular to the straining direction. Electropolishing removes these local stress raisers and hence increases fracture strains. Our results demonstrate that the pseudoelastic properties of the PVD-processed materials agree well with those of conventional NiTi, and that they provide new opportunities to study the fundamentals of martensitic transformation in small-scale model systems.

Keywords Small-scale testing · NiTi · Phase transformation · Nucleation and propagation of martensite bands

Introduction

Recent research on shape memory alloys (SMAs) has focused on new developments, such as high temperature SMAs, ternary or quaternary systems, novel Ti-based or Ta-based alloy systems. Yet, after decades of research, simple, binary NiTi still is the most commonly used SMA today. A number of groundbreaking studies have been performed on NiTi, and much of our current understanding of the fundamentals of SMA behavior has been obtained studying this alloy system. In the last 10–20 years, there has been a push for miniaturization in many applications, leading to an increasing interest in micro- or nano-electromechanical systems (MEMS or NEMS, respectively). SMAs offer many possibilities to include new functions, e.g., as sensor or actuator materials, in MEMS. This has renewed the motivation to study shape memory and transformation behavior of SMAs on small-length scales. Binary NiTi, because it is quite well understood as bulk material, certainly is well-suited as model system to study and compare SMA behavior on smaller length scales.

Scaling down the size of samples is in itself related to scaling effects: when sample dimensions are decreased to the order of characteristic microstructural length scales, one can expect new effects (like changes in mechanical strength, and also potentially changes in transformation behavior) to occur [1, 2]. Another important aspect is the effect of novel processing techniques on the resulting microstructures in small samples: conventional bulk NiTi is typically produced by melting, casting, followed by

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rolling, swaging, or wire drawing [3]. The resulting microstructures are typically characterized by considerably deformed grains, high dislocation densities (after cold work), and pronounced textures [4]. Physical vapor deposition (PVD) methods have emerged as an interesting alternative to these conventional processing routes; these PVD methods offer certain advantages, such as high purity (because samples are deposited in high vacuum), and because (pre-alloyed) targets can be carefully fine-tuned to the desired alloy composition [5–7]. Film morphologies, crystal growth, and textures can in principle be controlled using different substrate materials and subsequent heat treating conditions. Moreover, surface features and morphologies (like roughness and waviness) of the substrates are directly reflected by the thin films deposited on top. Consequently, these novel methods, which typically lead to small sample dimensions that are the topic of this paper, may well be associated with different microstructures. It is therefore important to characterize transformation behavior and thermo-mechanical properties of small-scale samples produced by PVD processes, and to compare these to their large-scale counterparts.

Research on small samples is also characterized by additional practical challenges: sample preparation and handling considerably smaller samples in the lab; adapting experimental equipment; higher demands on resolution of force, displacement, thermal, or optical measurement techniques; increased effects of surface features and scatter on different properties even in the same batch of material, to name just a few. Consequently, previous research on thin films and/or small-scale shape memory alloys has often been focused on processing parameters and their effect on the resulting material microstructures, while topics like careful mechanical testing have hardly been addressed. In this paper, we study pseudoelastic binary NiTi produced by PVD, and we consider several aspects related to small-scale testing: we document typical microstructural features associated with PVD processing. Using similar initial microstructures, we compare two different types of samples during tensile testing, and we discuss how different surface morphologies, clamping conditions, and strain measurement techniques affect the thermo-mechanical response. We also compare the material's tendency for localized deformation (i.e., formation and growth of fully transformed martensite in distinct bands) in small-scale samples to the well-known bulk behavior of NiTi [8, 9].

Materials and Experiments

The materials used in this study were obtained from Acquandas, Kiel, Germany. They were produced by a PVD magnetron direct current sputter process and deposited on

planar Si substrates under ambient conditions. The thin films were then heat-treated to promote crystallization, resulting in homogeneous NiTi films with a well-defined chemical composition of about 50.6 at.% Ni. Further details on the deposition process and heat treatments have been described elsewhere [10]. Lithography techniques [11] were then used to produce two types of samples for tensile testing: the first sample type consists of straight wires with a square cross section ($15 \times 15 \mu\text{m}^2$), while the second sample type is designed as a dogbone specimen ($5 \times 500 \mu\text{m}^2$), Fig. 1a. The gage length was 5 mm for both sample geometries. To study the effect of surface morphology, some of the wire and dogbone samples were subsequently electropolished, while others were kept in the initial surface state. To determine the phase transformation temperatures, differential scanning calorimetry (DSC) measurements were performed in a Mettler Toledo DSC1 (FRS 5 Sensor) apparatus with a cooling/heating rate of 10 K min^{-1} and sample masses of about 1 mg.

The microstructures of the small samples were analyzed by X-ray diffraction (XRD) in Bragg–Brentano geometry. A Bruker AXS D8 Discover XRD set-up with Cu-K α radiation was used to determine phases and textures. Electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM) was further used to investigate a possible texture, grain size, and grain size distributions. For the EBSD measurements, the sample surfaces were polished in a VibroMet2 vibratory polisher (polishing time 24 h, in a MasterMet 2 solution by Buehler). Sample surfaces were also studied in detail in a Zeiss NEON40EsB SEM to document surface morphologies. Chemical composition was confirmed by EDX measurements to be about 50.6 at.% Ni. The SEM was further used to analyze fracture initiation and fracture surfaces after tensile testing.

Uniaxial tensile testing was performed with both types of specimens in a Zwick/Roell tensile testing apparatus equipped with a 10 N high precession Xforce load cell with an accuracy grade 1 (0.02 % of maximum force). Both types of samples were clamped in planar ceramic grips, Fig. 1a. Strain measurements were performed in two ways: for the tests on both wire and dogbone samples, we determined (engineering) strains by considering cross-head displacement values. In addition, more detailed strain measurements were performed on the dogbone samples, using the optical method known as digital image correlation (DIC). To produce local strain maps, we used macro optics (Mitutoyo) and the DIC analysis software package ARAMIS by GOM [12]. The wires were deformed up to about 6 % strain, and then unloaded. This cycle was repeated twice. The samples were then finally loaded until fracture. The nominal strain rate in these experiments was set to $2.5 \times 10^{-2} \text{ s}^{-1}$. The dogbone samples were loaded and unloaded three times up to 8 % (as determined from

Fig. 1 Samples and experimental set-up for small-scale testing of NiTi structures. **a** Image of the two different sample geometries used in this study. The dogbone sample (*top*) has a cross section of $5 \times 500 \mu\text{m}^2$; the wire samples with cross sections of $15 \times 15 \mu\text{m}^2$

are shown below. **b** Clamping set-up for tensile testing using planar ceramic grips. A dogbone sample is indicated by the *arrow*. Strain measurements on the dogbone samples were performed using the optical strain measurement set-up (not shown here)

the cross-head displacement) with two different nominal strain rates of $(2.5 \times 10^{-2} \text{ s}^{-1}$ and $2.5 \times 10^{-4} \text{ s}^{-1})$. Finally, the optical strain measurement method was used in order to further analyze strain localization (i.e., formation of martensite bands) during pseudoelastic loading.

Microstructural Observations

Working with small samples does lead to a couple of practical challenges that are worth mentioning in this paper. We first discuss examples related to microstructural analysis methods. Microstructural characterization of small sample volumes typically enforces additional requirements for the measurement set-up. In most cases, the intensities of the physical signals are lower by orders of magnitude in comparison to measurements performed on conventional, larger samples. Due to the lower signal-to-noise ratio that is measured from small samples, it is typically necessary to use high-resolution techniques.

For instance, Fig. 2 shows the DSC curves obtained from wires and dogbones in a 20- μl cup. We note that, for reliable DSC measurements, sufficiently large sample masses (about 1 mg in our DSC) and contact areas between sample material and cup are needed even when using a high-precision calorimeter. We found it convenient to use material adjacent to the individual samples produced by lithography, since this part of the thin films is microstructurally similar and provides larger, fully connected flat pieces. The DSC data in Fig. 2 exhibit the typical features known from measurements on conventional bulk NiTi; they indicate that the material is austenitic at room temperature and that it shows a multi-step transformation (involving the formation of the intermediate R-phase) during cooling and heating. While further work is

Fig. 2 DSC curves measured in one cooling/heating cycle with an unpolished wire sample. The material exhibits a multiple-step transformation involving the R-phase both during cooling and heating. The austenite finish temperature is near room temperature

required to fully analyze the nature of the two superimposed peaks just below room temperature during cooling, the DSC data show that in both wire and dogbone samples, the martensite start temperatures (M_s) are about -50°C . Moreover, in both cases, the austenite finish temperatures (A_f) are near room temperature. Therefore, one can in principle expect pseudoelastic behavior during tensile testing.

XRD measurements on small volumes are challenging also in the sense that individual peaks are more difficult to recognize with respect to low level noise. On the other hand, while XRD measurements with Cu- K_α radiation typically only penetrate the surface regions of large bulk samples (in NiTi: penetration depth of about $15 \mu\text{m}$), the XRD data in our much smaller samples actually represent

their entire volume. In Fig. 3a, we compare the XRD data for wire and dogbone samples. In addition, we present XRD data from a conventional pseudoelastic NiTi wire (produced by wire drawing, but with a comparably small diameter of 20 μm). The red line marks the intensities for different crystal planes of the B2 phase, determined with a lattice parameter of 3.015 $\text{\AA}$. All characteristic peaks of the wire, as well as those of the dogbone sample, belong to the B2 phase and no additional peaks of Ti- or Ni-rich phases are observed. Due to the higher volume of the dogbone sample, the intensities for the characteristic crystal planes ((100), (110), (111)) are higher in comparison to the wire data (with smaller sample size). The XRD data from the drawn wire clearly indicate that there is a strong texture: only the (110) peak is pronounced, whereas the other peaks can hardly be observed. In contrast, the data for the PVD-processed samples do not indicate any specific texture.

More detailed information on microstructural features and texture can be obtained from EBSD measurements. Figure 3b shows one typical example from the surface of a dogbone sample; similar observations were also made for the wire samples but are not shown here. The color-coded orientation map (based on the inverse pole figure, see inset of Fig. 3b) indicates which crystallographic planes are parallel to the sample surface in each grain. It is well known that PVD-processed materials can exhibit strong textures. However, such textures typically have strong components in growth direction, which is directly related to the deposition process. Moreover, in our case, where deposition was performed at low temperatures, one does not expect any pronounced texture to occur. The data in

Fig. 3b do not provide full statistical information on grain orientations in three dimensions, but it clearly confirms that the PVD-processed material studied here does not exhibit any texture. Furthermore, from the EBSD analysis we observe polygonal grains and a monomodal grain size distribution. From the grain size (area) distribution data sets, the average grain size was determined as about 5 μm . These microstructural results show that the PVD process allows producing high-purity binary NiTi with globular grains and without texture—an ideal model system for detailed thermo-mechanical testing. With respect to tensile testing, we note that, despite the relatively small grain size, only about 10 grains are located in each cross section of the wire sample. Moreover, we expect that the dogbone samples typically consist of a single layer of (randomly oriented) grains in each cross section. One of the objectives of our tensile tests (see next section) therefore was to study whether the transformation behavior of such small-scale samples was notably affected by the behavior of individual grains.

Finally, given the small scale of the samples, surface features are likely to also have a pronounced effect on the mechanical behavior. In order to evaluate the surface conditions of our samples, we analyzed secondary electron micrographs. Figure 4a shows the surface of a wire sample prior to tensile testing. One can clearly observe a certain roughness related to columnar growth during the PVD process. These features are only observed on the surface, whereas our EBSD data clearly demonstrate that grains in the interior of the sample are globular. Electropolishing can be an effective method to remove these surface features;

Fig. 3 XRD and EBSD data of wires and dogbone samples: **a** phase analyses of investigated dogbone (blue), wire (red), and conventional drawn wire (green) with a diameter of 20 μm is shown in the X-ray diffractogram. All three samples show peaks that are related to B2 austenite (cubic phase, $a = 3.015 \text{ \AA}$ marked by the black dashed line). The drawn NiTi wire shows a significant (110) peak due to its processing history and texture, while the dogbone and wire data also

show significant (100), (110), and (111) peaks. **b** The inverse pole figure map of a dogbone sample obtained by EBSD (viewing direction perpendicular to the loading direction; colors indicate grain orientation normal to the viewing plane) shows randomly oriented polycrystalline grains. The average size of the globular grains is about 5 μm

Fig. 4 SEM micrographs of wires with a cross section of $15 \times 15 \mu\text{m}^2$ (loading direction *left to right*; growth direction *bottom to top*). **a** Typical surface condition of an unpolished wire with growth artifacts related to the deposition process. **b** The surface of the polished wire

Fig. 4b shows an electropolished wire sample that has a closed, smooth, and homogeneous surface with no sputter artifacts (such as pores or columnar crystal growth that ends in domes on the surface). However, the electropolishing procedure typically results in a slight waviness of the surface that can also be observed in Fig. 4b and that needs to be considered when evaluating the mechanical behavior.

Tensile Testing

We now discuss how the mechanical behavior of our small pseudoelastic samples can be determined in well-defined tensile tests. Because of the small dimensions of the samples, a couple of typical issues arise and need to be tackled for precise measurements. One important point simply is careful handling of the samples in order to avoid deformation prior to testing. Moreover, careful alignment in the testing machine and measurement of the really small values for the applied load need to be performed with special care. Typically, force values are in the range of 10–500 mN. Therefore, it is particularly necessary to avoid mechanical vibrations in the vicinity of the testing set-up in order to reduce the noise level in the load cell and in optical strain measurement set-ups.

Figure 5 shows the stress–strain curves of two wire samples (unpolished surface: Fig. 5a; polished surface: Fig. 5b) that were cycled three times in the pseudoelastic range and then loaded until fracture (with a nominal strain rate of $2.5 \times 10^{-2} \text{ s}^{-1}$). In both cases, the stress–strain curves exhibit the well-known transformation characteristics: after elastic loading, the transformation stress is reached and first martensite is formed. With increasing strain, the volume fraction of martensite increases and this process is clearly indicated by the constant stress plateau. It

exhibits a lower roughness compared to the unpolished sample. The growing direction of the thin film cannot be observed on the surface, but some additional waviness occurs

is worth noting that at similar nominal strain rates, larger bulk samples typically tend to self-heating, which is associated with a finite slope of the loading plateau. For the small samples tested in the present study, the surface-to-bulk ratio allows for fast heat transfer to the environment and therefore the effects of thermo-mechanical coupling on the mechanical behavior are less pronounced even at relatively high (but still quasi-static) nominal strain rates.

In the first three cycles, we unloaded the samples before the end of the plateau (at strains of about 8 %) was reached. During unloading, the reverse transformation from stress-induced martensite to austenite is also associated with a distinct plateau (at a lower stress level than during loading). Both samples also exhibit what is commonly referred to as functional fatigue [13]: the plateau stresses (in particular during loading) decrease with increasing number of cycles; this process typically reaches a saturation after a relatively small number of cycles and therefore, the differences between cycles 3 and 4 are already almost negligible. Moreover, we hardly observe any residual strains after unloading. Note that, in the fourth cycle, when the samples are deformed to strains exceeding the maximum strain values of the previous cycles, stresses increase again to the initial plateau stress values. This effect is also well known from bulk sample testing and can be explained by the fact that further grains, which have not been subjected to the stress-induced martensitic transformation in the first cycles, are now forced to transform for the first time. Beyond the pseudoelastic plateau, the stress–strain curves clearly show elastic loading (and potentially additional deformation mechanisms, such as detwinning or reorientation) of martensite, until fracture occurs at about 14 % (for the unpolished sample) and 18 % (for the polished sample). In summary, despite the much smaller dimensions and the very different processing history of our samples, the PVD-processed materials exhibit excellent

Fig. 5 Stress–strain curves during pseudoelastic cycling (*top*), SEM micrographs of the fractured samples (*center*), and fracture surfaces (*bottom*) of unpolished (*left*) and polished samples (*right*). **a, b** Stress–strain curves of unpolished/polished wire samples, respectively. Three loading/unloading cycles were performed, the fourth cycle was loaded until fracture. The material response shows typical features of functional fatigue, as indicated by decreasing plateau stresses. **c** SEM micrograph of the unpolished wire after fracture. The *white circle* marks cracks on the sample surface that can be directly related to the thin film growing direction. **d** SEM micrograph of the polished wire

after fracture. Because of the smoother surface condition after polishing, the fracture is more ductile. On the polished surface, the (plastic) deformation of individual grains, as well as traces from distinct martensite plates can be observed. **e** SEM micrograph of the fracture surface (*top view*) of the unpolished wire sample. **f** SEM micrograph of the fracture surface of the polished sample (tilted by about 90°). Locally, the sample has been subjected to a considerable amount of necking by ductile deformation, and the fracture surface shows strong evidence for ductile fracture

pseudoelastic behavior that compares very well with observations on conventional, bulk NiTi. This is particularly noteworthy since a much smaller number of grains is

located in each cross section. The material also seems to be quite stable with respect to functional fatigue and it is likely that it can in principle be used for a large number of

pseudoelastic cycles, as has for example been described by Quandt et al. [14].

In principle the unpolished and the polished samples show nearly the same pseudoelastic behavior; however, considering plateau stresses, there are small differences. The unpolished material shows a slightly higher plateau stress compared to the polished material. This effect can be explained by the waviness of the polished sample, where locally the outer dimensions of the cross section may well vary a little (but considerably less than one micron). Given the small total area of $15\text{ }\mu\text{m}$ by $15\text{ }\mu\text{m}$ of the nominal cross section (which is used to calculate engineering stresses), even these small local deviations result in distinct changes in terms of the calculated plateau stress values. Using nominal cross section values to calculate engineering stresses during small-scale testing clearly can introduce experimental errors that need to be taken into account when evaluating stress strain data qualitatively and quantitatively. However, we highlight that, in order to determine effective cross section values for stress calculations, the SEM needs to be used; this procedure again requires considerable care and more effort than handling larger conventional tensile samples, particularly when one considers that waviness leads to locally varying cross sections. Furthermore, the differences in terms of fracture strains can be related to the different surface conditions: polishing prevents early crack nucleation and therefore leads to larger fracture strains. In Fig. 5c, d, we present SEM micrographs from the samples after fracture. The unpolished sample exhibits a large number of parallel cracks on the surface, and it is reasonable to assume that these cracks (which result from the growth-related surface features) promote early fracture compared to the polished sample, where no such features are observed on the surface. Considering potential applications, electropolishing clearly is beneficial in eliminating critical sites for crack nucleation. Likewise, compared to the fracture surface of the unpolished sample (Fig. 5e), the fracture surface of the polished sample (Fig. 5f) seems to indicate a more ductile fracture that is also in line with the somewhat higher fracture strains.

Compared to the wire samples, the dogbone samples are characterized by a considerably different aspect ratio (1:100), and typically consist of a single layer of grains. Due to the larger surface area, in addition to using cross-head displacements to determine nominal strains, we could also perform optical strain measurements. Moreover, because of their larger cross sections, higher forces are needed to deform the dogbone samples, and this increases the likelihood of slip events in the mechanical grips. Whenever a clamped sample slips in the grips, its nominal deformation as determined from cross-head displacement data differs from the true strain state. This issue can be avoided by using optical measurements where full-field

strain maps are determined directly on the sample surface. Figure 6 shows a corresponding stress–strain curve for a dogbone sample that was mechanically cycled three times. Stresses were recorded from the force data from the load cell, while strains were determined from the relative displacements of two points on the sample surface (similar to the workings of a conventional clip-on extensometer). Interestingly, and even though only a single layer of grains is subjected to mechanical loading, the mechanical behavior of the dogbone sample is quite similar to the behavior of the wire samples (Fig. 5) recorded at the same nominal strain rate. A circle in Fig. 6 highlights a discontinuity in the stress–strain curve of the first cycle that can be directly related to a slip event: while the cross-head is being displaced continuously at a constant velocity, slipping in the grips leads to temporary (elastic) unloading of the sample, and this in turn leads to locally decreasing strains in the DIC-defined “gage length” between the two observed marker points. Once slipping stops, the sample is again deformed to larger strains, and stresses increase again. The relative amount of slip can also be determined by comparing the maximum strains in the first and subsequent cycles. While the testing set-up was programmed to perform 3 cycles with constant strain amplitude (with strains in the control loop determined from cross-head displacement), the sample was actually only deformed to a somewhat smaller maximum strain in the first cycle (see second, dashed circle in Fig. 6).

Our experiments on the dogbone samples also revealed that, because of the larger cross sections, and because of the higher forces required for straining, more deformation occurred in the grips. Considering the critical strains for the onset of the martensitic transformation, we found that

Fig. 6 Pseudoelastic stress–strain curve of a dogbone sample subjected to cyclic loading (strains determined by DIC). The *full circle* marks a slip event in the grips. The *blue dashed line circle* indicates the resulting difference between strains determined from cross-head displacement and true axial strains

strain values determined directly from the cross-head displacements can be off by a factor of 3–4; only by using the DIC method, reliable strain data could be obtained. One additional advantage of the DIC measurements, particularly useful in studying NiTi-based shape memory alloys, is that they allow the direct and spatially resolved observation of localization phenomena. The formation of distinct martensite bands is often observed in bulk NiTi subjected to uniaxial tension, and it is often related to texture effects, e.g. [15]. In our untextured, small-scale dogbone samples, however, very similar effects could be observed as well.

Fig. 7 Strain maps obtained by DIC during tensile loading and unloading (dogbone samples: $5 \mu\text{m} \times 500 \mu\text{m}$ cross section, 5 mm gage length, average grain size $5 \mu\text{m}$). The color-coded plots represent uniaxial tensile strain. **a** Pseudoelastic cycle with a nominal strain rate of $2.5 \times 10^{-4} \text{ s}^{-1}$. Observation of one martensite band moving through the sample gage length during: loading (left) up to homogeneous strains beyond the pseudoelastic plateau (second from the left); during subsequent unloading (second from the right) is associated with austenite nucleation in the gage length; final elastic unloading (right). **b** Pseudoelastic cycle with a higher nominal strain rate of $2.5 \times 10^{-2} \text{ s}^{-1}$. Due to the higher strain rate, more than one martensite band is formed during loading

Figure 7 shows strain maps recorded during the first cycles of two experiments on dogbone samples at nominal strain rates of $2.5 \times 10^{-4} \text{ s}^{-1}$ and $2.5 \times 10^{-2} \text{ s}^{-1}$, respectively. We note that, because of localized transformation, local strain rates may differ by an order of magnitude from the nominal ones [16].

At the lower nominal strain rate (Fig. 7a), the strain maps clearly show propagation of a single martensite band along the free gage length. The first strain map (from left to right) was recorded during loading along the pseudoelastic plateau, and two distinct regions, corresponding to elastically strained austenite, and fully transformed martensite, can be distinguished. At this relatively slow strain rate, one single band was nucleated in the clamping region and stable propagation along the gage length was observed. This is an interesting observation because despite the much smaller number of grains per cross section, the localization behavior corresponds very closely to well-known observations in macro-scale samples, see e.g. [16]. The second strain map was recorded at stresses beyond the pseudoelastic plateau, where the material is fully martensitic, as indicated by the correspondingly high and homogeneously distributed strains. During subsequent unloading (third strain map), several austenitic regions were formed simultaneously; the reverse transformation proceeded by growth of these regions along the gage length and once it was completed, the sample was fully austenitic again (fourth strain map). Formation of several austenite bands also is a typical observation in macro-scale samples that have been strained beyond the end of the pseudoelastic plateau: during unloading, favorable conditions for austenite nucleation obviously exist throughout the gage length. This observation also demonstrates that the micro-scale samples studied here consist of a homogeneous microstructure.

At the higher nominal strain rate (Fig. 7b), we observed the formation of several martensite bands along the gage length during loading (first strain map); this effect is related to local self-heating, which increases the critical stress required for the formation of martensite and hence slows down propagation of a martensite band, in turn leading to the nucleation of additional martensite bands in other locations. Once the bands have coalesced, the sample is again fully martensitic (second strain map), and unloading also involves the nucleation and growth of several austenite bands (maps three and four). The local strains near the interfaces between austenitic and martensitic regions (both in Fig. 7a, b) also indicate that, similar to macro-scale samples, the interface might in principle be characterized by an angle of inclination with respect to the tensile axis. We emphasize that such an angle is not a well-defined parameter because strains vary continuously across the interface region and the exact location and orientation of a

soft interface cannot be determined unambiguously. Moreover, interface inclination can also fluctuate (or switch between positive and negative angles of inclination, see also [16]; similar effects were observed in our experiments particularly when individual bands (such as then austenite bands in Fig. 7b) coalesced. This study is one of the first to report on localized deformation in micron-sized samples. While further work clearly is necessary to fully document interface propagation in our small samples, our first results presented here highlight an important point: even on a length scale where the mechanical response of individual grains can be expected to strongly affect the mechanical behavior, transformation by stable propagation of phase interfaces across the entire cross section seem to be a favored mode of deformation in NiTi.

Conclusions

We have considered microstructural and mechanical properties of small-scale NiTi samples produced by PVD processes. Special emphasis was placed on typical challenges while handling and analyzing these small-scale samples by DSC measurements, XRD analysis, electron microscopy, and tensile testing. The main conclusions of our paper are summarized below:

- Our study demonstrates the special significance of high-resolution equipment because small-scale testing is typically characterized by low signal-to-noise ratios.
- Mechanical testing of small samples is associated with certain challenges; particularly, slip events in mechanical grips need to be taken into account.
- Surface features related to the deposition process affect crack initiation and fracture behavior. Electropolishing can fully remove those artifacts, but it can lead to waviness, making it difficult to accurately determine cross section dimensions.
- Optical methods, like DIC measurements, can be used to document local mechanical behavior. Our first results using this technique reveal that, while smaller samples may well be affected by surface effects or the effects of individual grains, the PVD-processed samples studied here actually exhibit very similar behavior in terms of stress–strain hysteresis and localized deformation compared to conventional bulk samples.

Acknowledgments The authors would like to thank Acquandas, Germany, for providing the PVD materials and Rodrigo Lima de Miranda for many fruitful discussions.

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