



# Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF

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**Abstract** NiTi dominates the market of shape memory materials due to its optimal combination of mechanical, functional, and biocompatibility properties, which enabled its use for several applications, in particular for the biomedical and the aerospace sectors. However, due to its poor machinability, NiTi is a challenging material from the manufacturing standpoint. Therefore, in the last years, researchers have focused on the production of NiTi components by additive manufacturing processes, which also enable the manufacturing of complex shape parts that cannot be produced with conventional methods. The aim of this study is to provide insights on the optimization of the functional performances of NiTi produced by Laser Powder Bed Fusion, leveraging on the building orientation and post-processing heat treatments. Uniaxial mechanical tests have been performed in tension and compression, and the influence of heat treatments and building orientation on the mechanical behavior of pseudoelastic NiTi has been evaluated. Different heat treatment schedules have been evaluated, leading to transformation strains up to 2.7% in tension and 4.6% in compression. This study confirms that Laser Powder Bed Fusion is a promising additive manufacturing technology for the production of net-shape and near defect-free NiTi components, exhibiting remarkable functional properties.

**Keywords** NiTi alloy · L-PBF process · Mechanical behavior · Transformation strain · Pseudoelasticity

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## Introduction

Shape memory alloys (SMAs) exhibit unique functional properties such as the shape memory, the pseudoelasticity (or superelasticity) [1], and the elasto-caloric effect [2, 3]. These properties make the SMAs suitable for applications in the biomedical, aerospace, civil, and automotive sectors [4–6]. Among the different SMAs, NiTi is the most widely studied, due to its superior transformation strain, mechanical properties, and biocompatibility [7, 8]. The capability of NiTi to recover its original shape is determined by a reversible phase transformation between the cubic B2 austenitic phase and the monoclinic B19' martensitic phase [9, 10]. In spite of the excellent functional properties, one of the main drawbacks of NiTi is the poor machinability that limits to some extent its fields of application [11, 12]. Given this context, the possibility to realize NiTi components by additive manufacturing (AM) gathered an increasing interest [13–18], since it allows producing, layer-by-layer, net-shape 3D objects, avoiding the typical constraints of the conventional melting and forming methods [19].

The AM functional materials that are able to change their shape providing an external stimulus (e.g., mechanical, thermal, magnetic or electric) is commonly referred to as “4D printing” [20]. One of the most widespread AM techniques for metals is the Laser Powder Bed Fusion (LPBF), which uses a high-power laser beam to melt locally a layer-wise deposited metallic powder. The recent literature is rich of research works focused on the 4D printing of NiTi, in particular using LPBF [21–33], showing the interest of the scientific community on this topic, as also evidenced by a substantial number of reviews [13, 14, 34].

The production of NiTi components by LPBF brings up new challenges such as the precise control of the chemical composition to obtain the desired functional properties in final parts [21]. Small variations in the Ni/Ti ratio cause relevant changes in the characteristic transformation temperatures of the material, and therefore in its mechanical and functional performances [35]. In particular, nickel is more volatile than titanium and tends to evaporate under the laser radiation [36, 37]. Consequently, a proper optimization of the process parameters is of paramount importance to achieve fully dense parts with sound functional properties [38]. Biffi et al. [33] reported that with an energy density within the interval  $63 \div 160 \text{ J/mm}^3$ , it was possible to obtain high values of relative density for NiTi specimens produced by LPBF. Saedi et al. [21] fabricated NiTi samples with good pseudoelastic performance with an energy density of  $222.2 \text{ J/mm}^3$ . Moghaddam et al. [26] demonstrated the potential of LPBF to tune the functional behavior of NiTi by a proper calibration of the process parameters. For a sample manufactured with an energy density of  $83.33 \text{ J/mm}^3$ , they reported a strain recovery of 5.62% without any post-process heat treatment.

Even though acceptable functional behavior has been reported also for the as-built NiTi specimens [26], heat treatments provide a further possibility to tailor and enhance the NiTi functional properties [22, 31, 33, 39]. In this regard, Haberland et al. [39] reported the formation of a high amount of finely dispersed  $\text{Ni}_4\text{Ti}_3$  precipitates after solution annealing followed by aging at  $350 \text{ }^\circ\text{C}$ . These precipitates are considered to play a fundamental role in the functional behavior of the material, since they can act as nucleation sites for the martensite. However, as a drawback, the precipitates are reported to embrittle the metal matrix and prolonged aging can lead to their coarsening and, consequently, reduced pseudoelastic performances. Saedi et al. [22] studied the effect of aging treatments on the functional properties of  $\text{Ni}_{50.8}\text{Ti}_{49.2}$  (at.%) alloy produced by LPBF. The authors reported that, after solution annealing at  $950 \text{ }^\circ\text{C}$  followed by aging at  $350 \text{ }^\circ\text{C}$ , the material exhibited good pseudoelastic behavior, i.e., 5.5% strain recovery with 0.3% unrecoverable strain. They also observed a stabilized strain recovery of 4.2% following ten loading cycles. In another study, Saedi et al. [31] obtained a strain recovery of 5.5% after aging at  $600 \text{ }^\circ\text{C}$ , without solution annealing. Biffi et al. [33] obtained a full recoverable strain of 6% after heat-treating additively manufactured NiTi at  $500 \text{ }^\circ\text{C}$ .

It should be noted that the former cited studies focused on the NiTi testing under pure compression loads. This is due to the fact that NiTi has scarce LPBF processability and parts without a significant amount of porosity and defects, which are detrimental for the tensile properties, can be hardly produced [14]. Only recently, research

studies showing tensile properties of NiTi produced by LPBF have been published [40–42]. Nematollahi et al. [40] reported a strain recovery of 5.31% in tension for the as-built dog-bone NiTi specimen manufactured by LPBF. Lu et al. [41] obtained stable tensile recovery strain up to 3.74% during cyclic tests of aged NiTi specimens. They attributed this to the saturation of the dislocations formed during the cycles, thanks to the dislocation pile-up around the  $\text{Ni}_4\text{Ti}_3$  nanoprecipitates. More recently, Xue et al. [42] obtained tensile pseudoelasticity values up to 6% in the as-built state by tailoring process parameters and by a strict control of the oxygen content within the building chamber. We also note that other additive manufacturing technologies applied to NiTi have shown promising tensile properties, such as Electron Beam Freeform [43] and Wire Arc Manufacturing [44].

It becomes evident that, for a widespread industrial application, more efforts are required to guarantee excellent tensile properties of NiTi produced by LPBF. For this reason, this study aims to further investigate the tensile behavior of NiTi produced by LPBF focusing on two loading directions—namely vertical and horizontal—with respect to the building orientation. In this regard, one of the most important characteristics of the AM process is the mechanical anisotropy, which arises from the formation of columnar grains, crystallographic texture, and “fish-scale” structure typical of the as-printed material [45]. This is of particular importance considering that, for NiTi alloys, the transformation strain associated with the austenitic-martensitic transformation is strongly affected by the crystallographic orientation of grains [24, 40, 46–48]. Nematollahi et al. [40] investigated for the first time the effects of the building orientation on the microstructure and the functional properties of 3D printed NiTi. The samples with a prevalent  $\langle 001 \rangle$  orientation along the loading direction showed a tensile strain recovery of 2.87%, while a prevalent  $\langle 110 \rangle$  orientation resulted in a strain recovery of 5.31%.

The microstructure of NiTi might also determine a strong heterogeneous martensitic transformation which does not occur with the same extent across the whole volume of the specimen, while it can be locally favored for grains having a more suitable orientation. Therefore, in the last years, it has become common practice the use of Digital Image Correlation (DIC) to collect full field strain maps and to locally quantify the degree of phase transformation and recoverable strains [40, 49]. In the present study, DIC strain measurements were carried out to study the macro- and micro-transformation strains providing fundamental insights into the transformation behavior of the present LPBF NiTi alloy.

The literature review reported in the previous paragraphs shows that more efforts are necessary for a

complete understanding of the combined effect of building orientation and heat treatments on the functional behavior of additively manufactured NiTi and for its full exploitation in real applications. Moreover, few attempts to investigate the tensile behavior of additively manufactured NiTi have been performed so far. In this regard, we carried out an experimental campaign which involved the design of different heat treatment routes and the tensile/compression testing of NiTi samples produced by LPBF along different orientations. Uniaxial mechanical tests have been performed both in tension and compression and DIC has been used to investigate the transformation strain heterogeneities exhibited by the manufactured specimens.

## Materials and Methods

### Powder and Specimen Manufacturing

NiTi gas-atomized powder with a particle size distribution between 15 and 45  $\mu\text{m}$  was provided by SAES Getters. The nominal composition of the powder was 50.8 at.% Ni and 49.2 at.% Ti. All the specimens were produced by means of a Renishaw AM250 industrial LPBF system, equipped with a 200 W single mode fiber laser (R4 from SPI, Southampton, UK) and an optical chain which provides a 75  $\mu\text{m}$  beam diameter in the focal position. A Reduced Built Volume (RBV) platform was used for this work, so that the build chamber was limited to  $78 \times 78 \times 50 \text{ mm}^3$ . The specimens were obtained using a meander scanning strategy. To minimize oxygen pickup, the process was carried out under a controlled argon flow.

The selection of processing condition was performed by varying the process parameters to obtain a proper energy density and specimens with high relative density. Suitable process parameters were found to be a power of 150 W, an exposure time of 60  $\mu\text{s}$ , a point distance of 45  $\mu\text{m}$ , and a hatch distance of 45  $\mu\text{m}$ , leading to a relative density of 99.3% and crack-free specimens. Tensile tests were performed using dog-bone specimens, with external dimensions of  $23 \text{ mm} \times 6 \text{ mm} \times 1.5 \text{ mm}$  (Fig. 1a) and cross section of  $3 \times 1.5 \text{ mm}^2$ , whereas compressive tests were carried out on solid cuboidal specimens, with dimensions of  $8 \text{ mm} \times 8 \text{ mm} \times 16 \text{ mm}$  (Fig. 1b). Two batches of tensile specimens were printed, with two different loading axis orientations, namely *vertical* (i.e., loading direction parallel to the building direction) and *horizontal* (i.e., loading direction orthogonal to the building direction). The compression specimens, instead, were manufactured only in the vertical direction. Figure 1 shows the sizes of the specimens, along with their orientation on the build platform.

### Specimen Preparation and Experimental Setup

Samples for metallographic analysis were grinded with sandpapers down to 2500 grit and polished with diamond pastes with particle size in the range of 6 to 1  $\mu\text{m}$ . The samples for Electron Backscattered Diffraction (EBSD) analyses were finally polished by colloidal silica. The tensile specimens were polished on both sides to reach a thickness of 1 mm. Moreover, the lateral sides of the specimens were machined to improve surface quality.

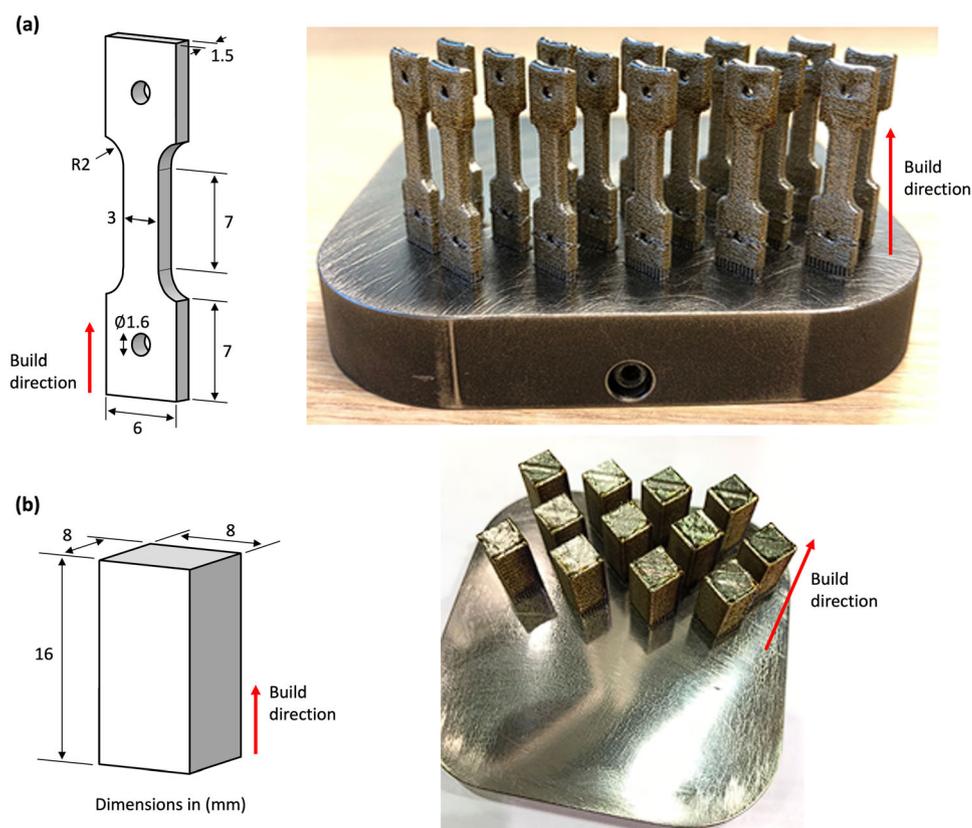
The phase composition of the materials was studied by means of X-ray diffraction (XRD) analyses using a Rigaku SmartLab SE X-ray diffractometer with Cu  $K_\alpha$  radiation ( $\lambda = 0.154 \text{ nm}$ ). XRD patterns were collected at 40 kV and 40 mA with Bragg–Brentano geometry in a  $2\theta$  range of  $10 \div 120^\circ$  with a step size of  $0.02^\circ$  and a speed of  $1^\circ/\text{min}$ . Pattern indexing and phase identification were performed with the aid of the software SmartLab Studio II.

Microstructural analyses were carried out by a Nikon Eclipse LV150NL optical microscope (OM) and a Zeiss Sigma 500 field emission scanning electron microscope (FE-SEM) equipped with an EBSD detector. To perform OM and FE-SEM analyses, samples were etched with a solution composed by 5 mL of hydrofluoric acid (HF), 20 mL of nitric acid ( $\text{HNO}_3$ ), and 25 mL of water, according to what reported in [50]. Water was used instead of acetic acid ( $\text{CH}_3\text{COOH}$ ) to avoid pitting and overetching. The software used to perform EBSD analyses and to elaborate the acquired data were AZtec and Tango, respectively, developed by Oxford Instruments. For all the analyses, tilt angle and accelerating voltage were set at  $70^\circ$  and 20 kV, respectively, while step size was set differently depending on the magnification (i.e., 1.3  $\mu\text{m}$  for  $250 \times$  and 0.04  $\mu\text{m}$  for  $10\,000 \times$ ).

Tensile tests were performed on dog-bone specimens by means of a DEBEN Microtest dual screw testing stage, which can sustain a maximum load of 5 kN. Compressive tests on solid cuboidal specimens were performed using an MTS Alliance RF/150 electromechanical testing machine. Both the tensile and compressive tests were carried out at room temperature in displacement control (i.e., 0.8 mm/min in compression and 0.3 mm/min in tension).

For all the mechanical tests, DIC was used to measure local strain fields during specimen deformation. A thin layer of white and black paints was sprayed on the specimens by means of an Iwata airbrush which enabled to produce an appropriate speckle pattern for DIC measurements. The field of view for the real-time DIC measurements was approximately  $6 \text{ mm} \times 12 \text{ mm}$  for the compressive specimens and  $3 \text{ mm} \times 9 \text{ mm}$  for the tensile specimens. The images were continuously acquired during loading by a high-resolution Allied Vision Manta CCD camera, while the specimen's surface was illuminated by

**Fig. 1** Geometry and the as-built appearance of **a** the tensile specimens and **b** the compressive specimens. All dimensions are in mm



two LED lights. Successively, all the acquired data were analyzed with the VIC-2D DIC software.

The measured DIC strain fields were used to produce two types of strain measurements. At the macro-scale, an averaged axial strain (labeled as *global strain*) represents the bulk mechanical behavior of the specimen. This strain measurement is similar to the strain that can be obtained by an extensometer. On the other side, local strains were also pinpointed in the regions of the specimens' surfaces that displayed the largest deformations (labeled as *local strains*). This type of measurement enabled the characterization of the maximum transformation strain for all the conditions investigated in the present study (i.e., heat treatments, loading orientation, and compression/tension).

## Heat Treatments

The influence of heat treatments on the mechanical properties of the material was evaluated by implementing different heat treatment routes, including solution annealing and aging. Aging was performed starting from samples both in the as-built condition and after solution annealing (SA) at 850 °C for 30 min, followed by water quenching (WQ). Aging temperatures were set at 400 °C and 450 °C for 15 min and 500 °C for one hour, respectively. The details are reported in Table 1. Heat treatments were

carried out in electrical resistance furnaces Carbolite HRF 7/22D for aging and Carbolite 3216 GPC 12/36 for solution annealing.

The effect of the heat treatments on the transformation temperatures of NiTi was evaluated by means of DSC analyses using a DSC-Q2000 by TA instruments. After evaluating reproducibility on sacrificial test pieces, measurements were performed on 20 ÷ 100 mg samples, cycling from 150 to – 150 °C and back, with a thermal rate of 10 °C/min. The analyses were performed on powder and bulk samples. The transformation temperatures were evaluated by means of the tangent method, measuring the onset and the offset of the transformation peaks.

**Table 1** Heat treatment schedules adopted for the experiments

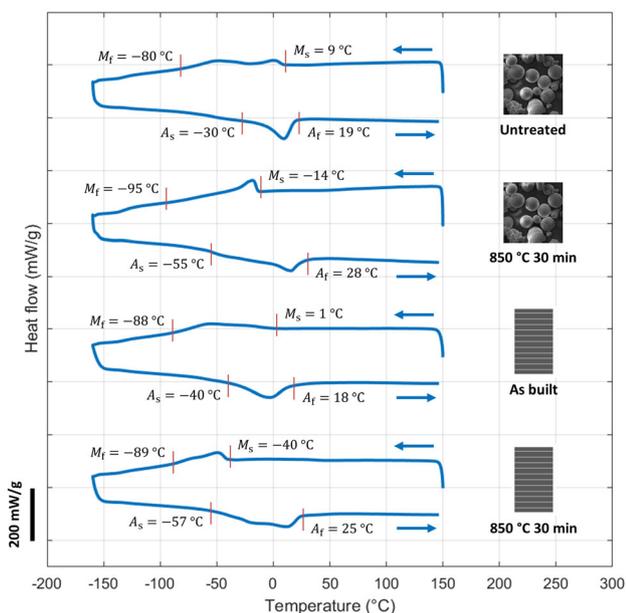
Heat treatment	Temperature	Time
Aging	400 °C	15 min
Aging	450 °C	15 min
Aging	500 °C	1 h
SA + WQ	850 °C	30 min
SA + WQ + aging	850 °C + 400 °C	30 min + 15 min
SA + WQ + aging	850 °C + 450 °C	30 min + 15 min

## Results and Discussion

### Effect of the LPBF Process on the transformation temperatures

The results of the DSC analyses performed on the powder and on the printed specimens are reported in Fig. 2. The transformation temperatures are highlighted by the red vertical lines. The untreated powder exhibits a double transformation peak on cooling, which can be likely justified in two ways. Firstly, the presence of residual stresses due to the rapid cooling rate at which the powder was subjected during the atomization process can induce the formation of the R-phase as an intermediate step in the transformation of austenite into martensite [28]. Secondly, local chemical inhomogeneity (micro-segregations) within the microstructure can induce some regions to transform before others, making the transformation to occur in different steps [28, 51]. A confirmation of these two explanations could be the fact that the solution-treated powder exhibits only one transformation peak on cooling, a smaller hysteresis and a slight shift of transformation to higher temperatures, since solution annealing allows to alleviate the residual stresses and to provide chemical homogenization.

For what concerns the bulk samples, the DSC curve of the as-built condition shows a trend that is similar to that of the untreated powder. Solution annealing has an important effect on the transformation temperatures also for the printed specimen. The  $A_f$  temperature slightly increases

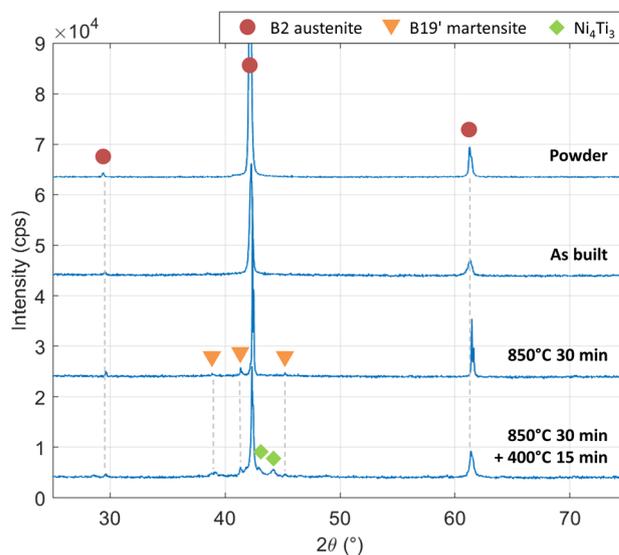


**Fig. 2** DSC curves of the NiTi powder and printed samples in different conditions

from 18 to 25 °C after solution annealing. A more pronounced effect can be seen on the forward transformation in terms of shift in the  $M_s$  temperature, which is reduced from 1 to -40 °C after the heat treatment. It is assumed that the macroscopic residual stresses induced by LPBF are released upon solution treatment, stabilizing the parent phase. Furthermore, the repeated thermal cycles during LPBF processing can have an effect similar to that of aging, causing precipitation of  $Ni_4Ti_3$  precursors and inducing a double stage transformation that involves the R-phase transformation [52]. In fact, the as-built specimen exhibits a broader peak related to the martensitic transformation, while after solution annealing, the peak appears to be sharper. This can be an indication of the capability of solution annealing to reduce chemical and microstructural inhomogeneities, dislocation density, and residual stresses [53]. The decreasing in  $M_s$  temperature after solution annealing can be also justified considering that, during the heat treatment, Ni-rich second phases are dissolved in the austenitic matrix, thus increasing the nickel content [54]. The increase of nickel content in the matrix is well known to reduce the  $M_s$  temperature [35].

### Phase Identification

XRD analysis was performed both on powder and the as-built samples. The effect of solution annealing and aging was investigated as well. The diffractograms are shown in Fig. 3. The results indicate that both the untreated powder and the as-built sample are mainly composed by fully B2 austenitic structure. Regarding the solution-treated sample,



**Fig. 3** From the top to the bottom: XRD diffractograms of the untreated  $Ni_{50.8}Ti$  powder and the as-built material, after solution annealing at 850 °C and after solution annealing at 850 °C followed by aging at 400 °C

small peaks belonging to the B19' martensitic phase also arise, suggesting a minor amount of martensite. In addition, the pattern of the solution-treated and aged sample shows peaks of the  $\text{Ni}_4\text{Ti}_3$  precipitates formed during the heat treatment at 400 °C.

### Microstructure

A SEM micrograph of the gas-atomized powder used for this work is reported in Fig. 4a. The powder particles have a size between 15 and 45  $\mu\text{m}$ . An optical micrograph performed on the as-built material is reported in Fig. 4b. The microstructure is characterized by large columnar grains that grew epitaxially through the layers. The key feature behind this kind of growth is the partial re-melting of the previous solidified layer, which induces the grains to grow with a crystallographic orientation similar to that of the grains of the already solidified layer [40]. The grains which are preferentially oriented along the direction of the temperature gradient tend to grow at a higher rate, at the expenses of the less favorably oriented neighboring grains.

The melt pools are not visible in the optical micrographs, but they can be clearly seen in the SEM micrograph in Fig. 4c. The columnar grains that grew through the different layers are also noticeable. At higher magnification, fine solidification cells are revealed, as shown in Fig. 4d. The formation of this fine microstructure can be ascribed to the high cooling rate and severe thermal gradients, which occur in LPBF.

The EBSD maps in Fig. 5 show the grain structure of the central area of the as-built specimen. The grains grew

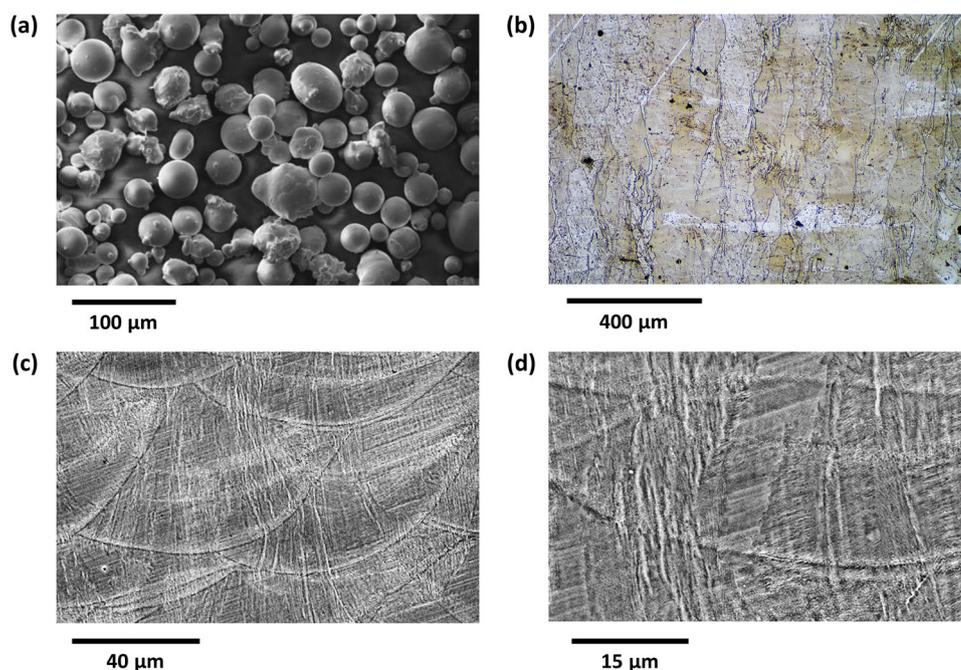
for hundreds of micrometers along the building direction (in this case identified by the  $y$  axis). The phase map reported in Fig. 5 suggests that the material is in a completely austenitic state. However, at higher magnification (Fig. 6), martensite is visible at grain boundaries. The formation of martensite can be ascribed to heterogeneities in the composition or to the presence of residual stresses between adjacent grains.

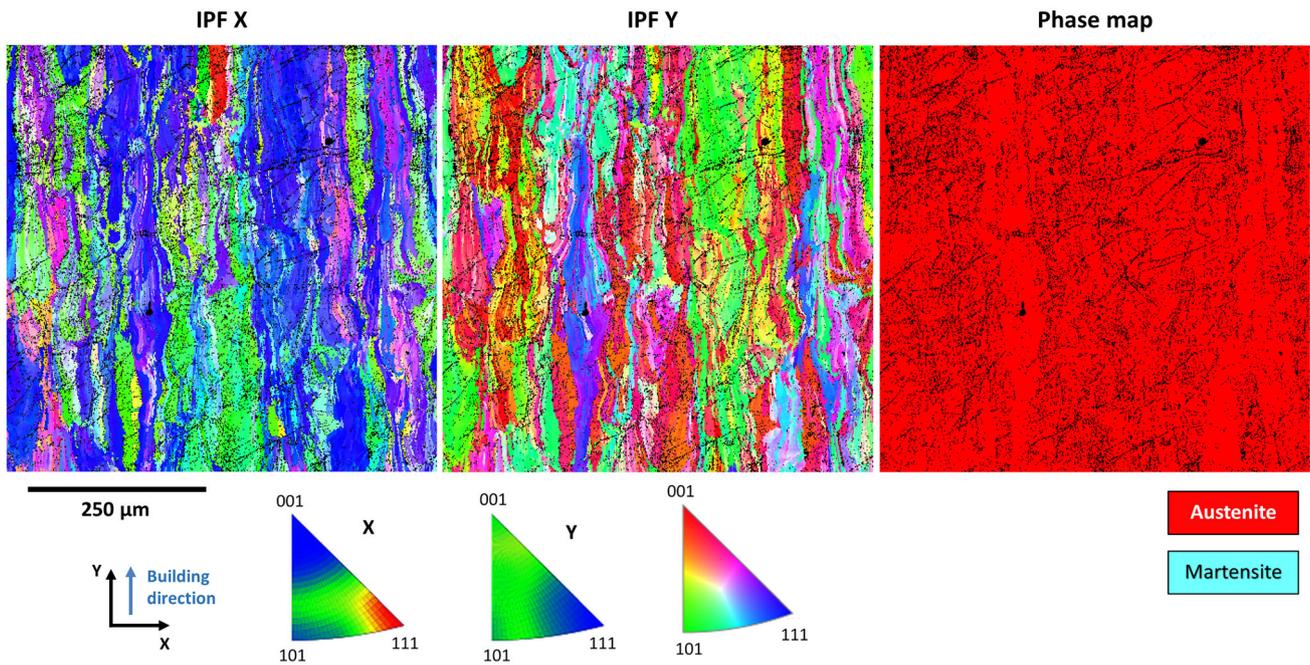
The kernel average misorientation (KAM) map of the austenitic grains is also reported in Fig. 6, showing high misorientation level next to the martensite areas. Such local strains could be responsible for the formation of stress-induced martensite (SIM).

### Compression Tests

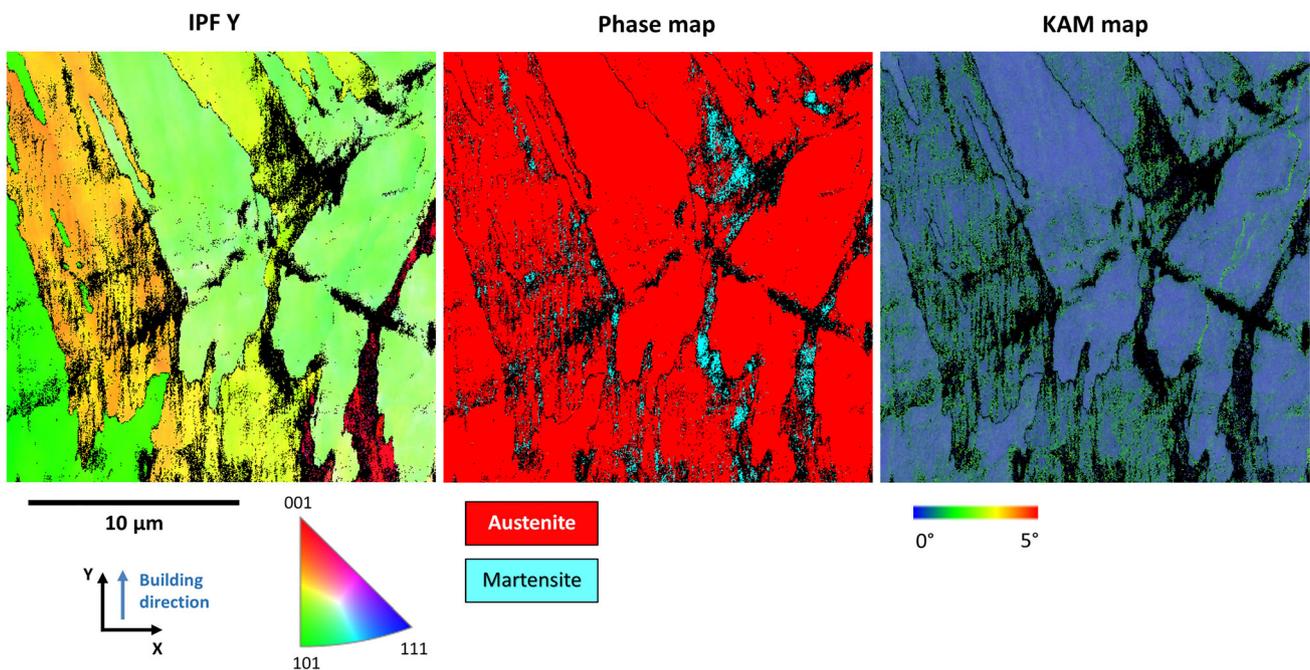
Compression specimens were tested with the loading direction parallel to the building direction. The corresponding curves are shown in Fig. 7a, where the labels indicate the heat treatment performed on the specimen at which each curve corresponds. For all the tests, the critical stress to induce the martensitic transformation ( $\sigma_{\text{PE}}$ ) and the extent of pseudoelastic recoverable strain ( $\varepsilon_{\text{PE}}$ ) were evaluated according to the scheme in Fig. 7b. The critical stress  $\sigma_{\text{PE}}$  was evaluated as the onset of the stress–strain curve corresponding to a deviation from the initial linear elastic behavior of 0.2%, while the recoverable strain  $\varepsilon_{\text{PE}}$  was calculated by subtracting the maximum strain in correspondence of a fully linear elastic unloading and the residual strain upon unloading at zero stress. The values of  $\sigma_{\text{PE}}$  and  $\varepsilon_{\text{PE}}$  are summarized in Fig. 7c.

**Fig. 4** **a** SEM micrograph of  $\text{Ni}_{50.8}\text{Ti}$  powder. **b** Optical micrograph of the as-built LPBF produced NiTi showing the grains grown epitaxially toward the building direction. **c** and **d** SEM micrographs of the as-built LPBF produced NiTi highlighting the appearance of the melt pools and the cellular structure of the grains which is a consequence of the rapid solidification rate





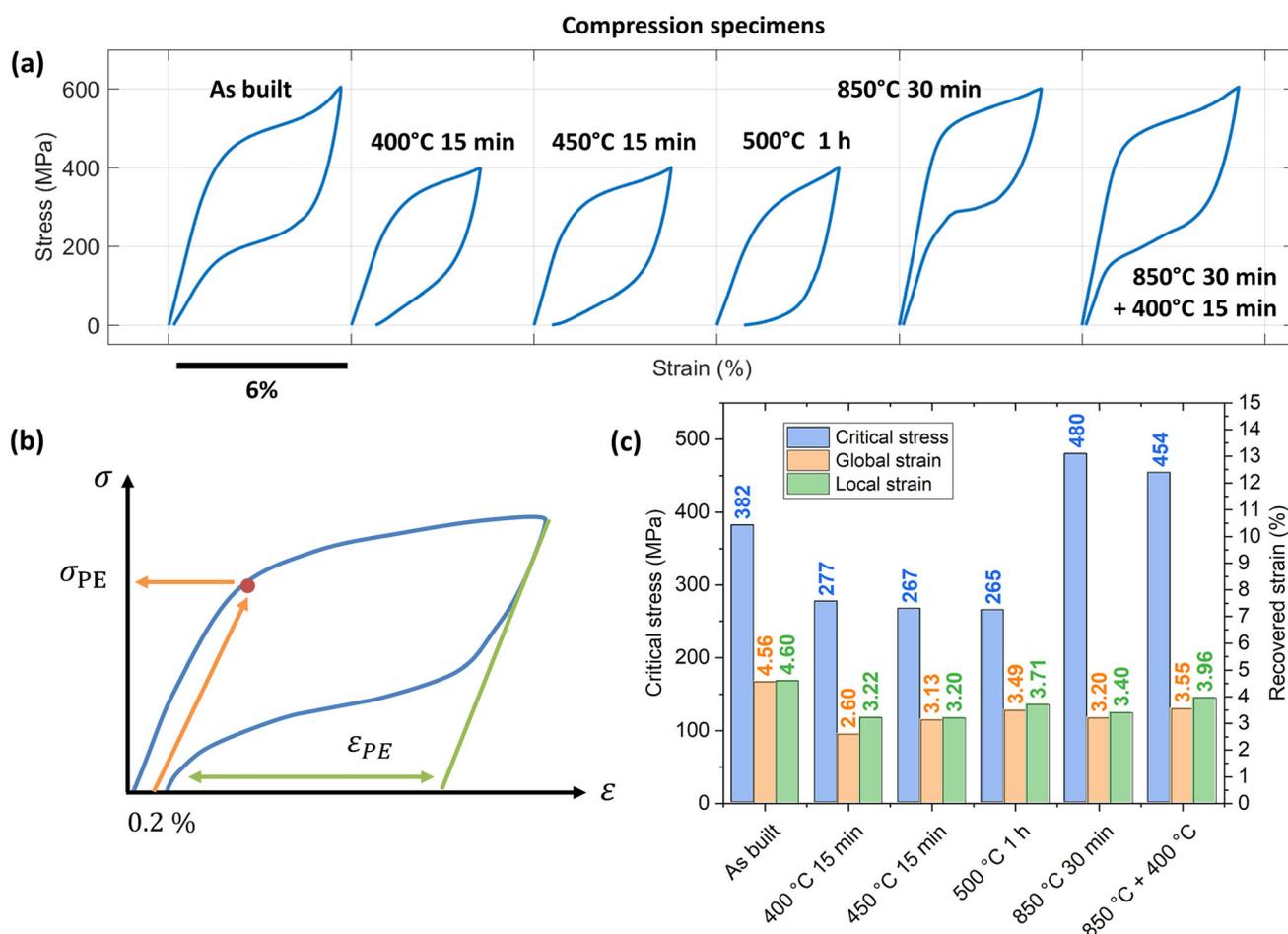
**Fig. 5** The IPF map of the as-built NiTi in the x direction (perpendicular to the building direction), the IPF map in the y direction (parallel to the building direction), and the phase map



**Fig. 6** The IPF map of the as-built NiTi in the y direction (corresponding to the building direction), the phase map, and the kernel average misorientation (KAM) map

The material exhibits pseudoelastic behavior at room temperature in all the testing conditions. The aged specimens show lower  $\sigma_{PE}$  with respect to the solution-treated specimen. This difference can be related to the increase in the transformation temperatures after the aging treatment

[22]. The XRD analysis has shown that the aging treatment promotes the formation of  $Ni_4Ti_3$  precipitates. It is widely recognized that the formation of such Ni-rich precipitates depletes Ni from the matrix, thus altering the Ni/Ti ratio and, therefore, modifying the transformation temperatures.



**Fig. 7** **a** Stress–strain curves of the specimens tested in compression; the labels indicate the heat treatment conditions of each specimen. **b** A schematic illustrating the approximation used for the evaluation of the critical stress to induce the martensitic transformation  $\sigma_{PE}$  and

the extent of recoverable strain due to the pseudoelastic effect ( $\epsilon_{PE}$ ). **c** Graphical summary of the main results obtained by compression testing of NiTi, in terms of critical stress and the extent of recovered strain both globally and locally

Moreover, the Ni-rich nano-sized coherent precipitates likely favor local stress intensification, leading the austenite to transform into martensite at lower values of nominal stress [55].

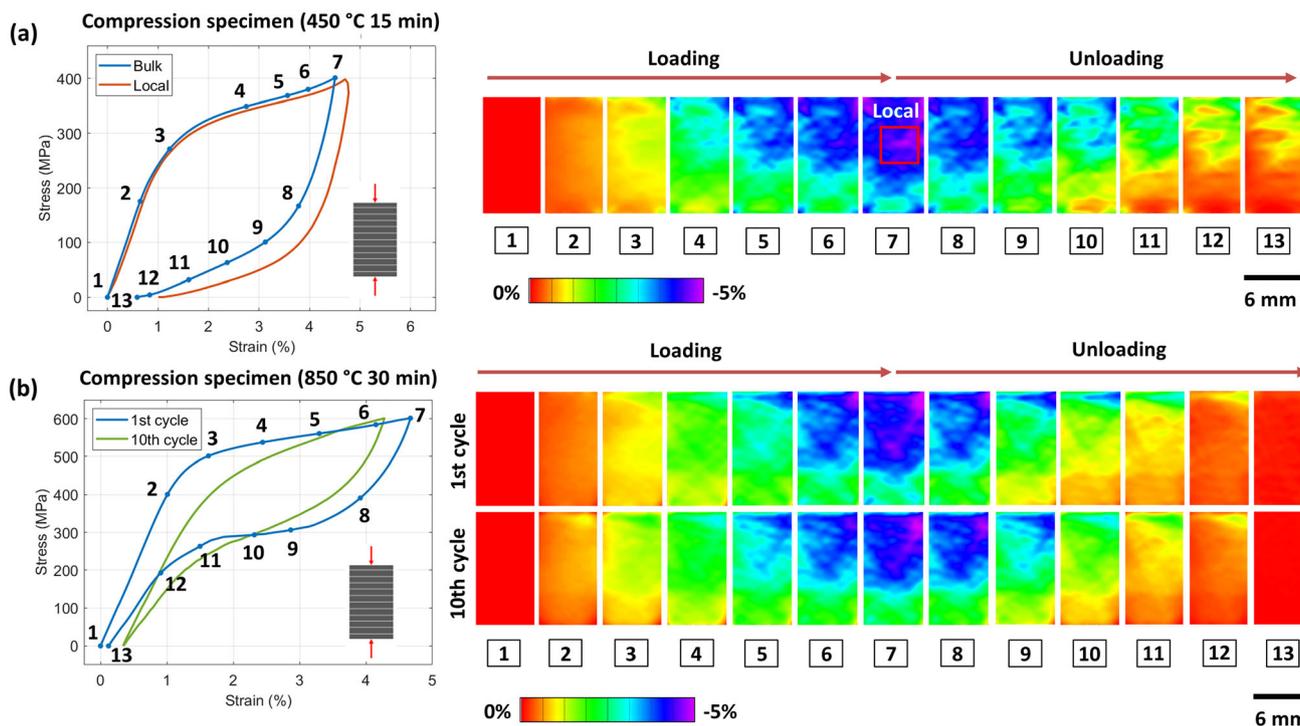
In Fig. 8, more details regarding the specimen aged at 450 °C and the specimen annealed at 850 °C are reported in terms of stress–strain curves and DIC strain maps. DIC contours allow to observe that the martensitic transformation does not occur with the same extent across the whole specimen. This supports the strong influence of the coarse-grained microstructure observed in the EBSD maps (Fig. 5).

The effect of cyclic loading was also investigated for the solution-treated specimen. The specimen was loaded up to 600 MPa and unloaded for 10 cycles. The stress–strain diagrams and DIC contours are reported in Fig. 8b. It is observed that, after cycling, the curve stabilizes and the unrecovered strain settles on 0.36%, which is a low value with respect to other results reported in the literature, since

it is generally found that the curve of the 10th cycle in compression settles on a strain value higher than 2% [21, 22, 26, 28]. Moreover, the curve related to the 10th cycle is characterized by a reduced hysteresis and a lower value of recoverable strain with respect to the curve related to the 1st cycle (i.e., 2.46% with respect to 3.20%). This indicates that the pseudoelastic performances tend to degrade while increasing the number of loading cycles, as reported also in other studies on LPBF produced NiTi [21, 22, 26, 28]. This is consistent with the fact that the material is not trained [56], even though other works report a faster stabilization of the loading curve of LPBF produced NiTi with respect to the conventionally produced NiTi [13].

### Tensile Behavior

The experimental results of the tensile tests are reported in Fig. 9a for the vertical specimens and in Fig. 9b for the

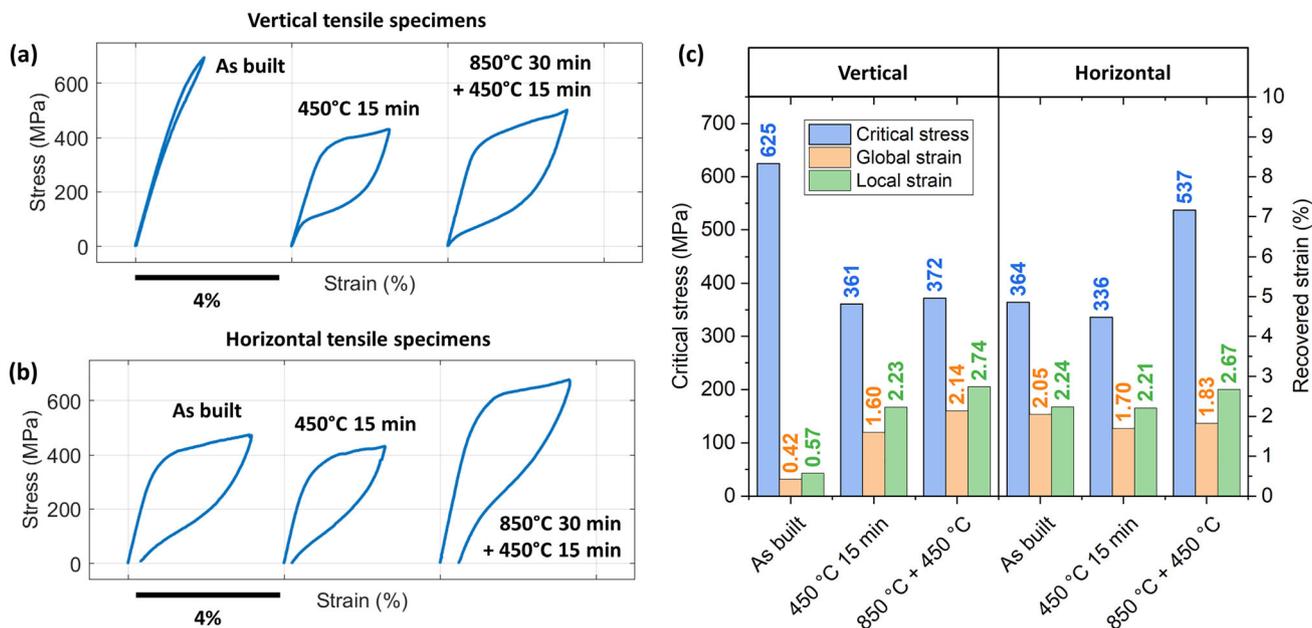


**Fig. 8** Stress–strain curves and DIC contours for two compressive specimens: **a** the specimen aged at 450 °C and **b** the specimen annealed at 850 °C

horizontal specimens. The specimens were tested in different conditions: in the as-built state, after aging at 450 °C and after solution annealing at 850 °C followed by aging at 450 °C. The evaluated values of  $\sigma_{PE}$  and global and local

$\epsilon_{PE}$  for all the testing conditions are reported in Fig. 9c for both vertical and horizontal specimens.

The stress–strain diagram of the as-built vertical specimen shows a limited hysteresis and a very high value of



**Fig. 9** Stress–strain curves for **a** the three vertical specimens and **b** the three horizontal specimens tested in tension; the labels indicate the testing conditions of each specimen, namely the heat treatments

performed. **c** Graphical summary of the main results obtained by tensile testing of NiTi, in terms of critical stress and the extent of recovered strain both globally and locally

critical stress (i.e., 625 °C). On the other hand, the aged specimens show a larger hysteresis and a lower value of critical stress (i.e., 361 MPa and 372 MPa). In all cases, a complete reverse phase transformation is observed, without irreversible strains. In Fig. 10a, more details about the behavior of the specimen aged at 450 °C are reported. The local stress–strain curve shows higher transformation strain values with respect to the global curve. In addition, DIC contours in Fig. 10a show that in the unloaded state, the specimen was able to recover almost all the strain across the whole surface.

For what concerns the horizontal specimens, the  $\sigma_{PE}$  was found to be 363 MPa in the as-built condition, in contrast to the value of 625 MPa found for the vertical specimen. Moreover, the horizontal specimen exhibits residual strains upon unloading, likely indicating the presence of a significant amount of martensite in the material. Such differences may be related also to the differences in the thermal histories. During the production of the horizontal specimens, larger areas are scanned in a given layer. Heat accumulation is more likely to occur during the scanning of horizontal specimens due to a prolonged laser interaction

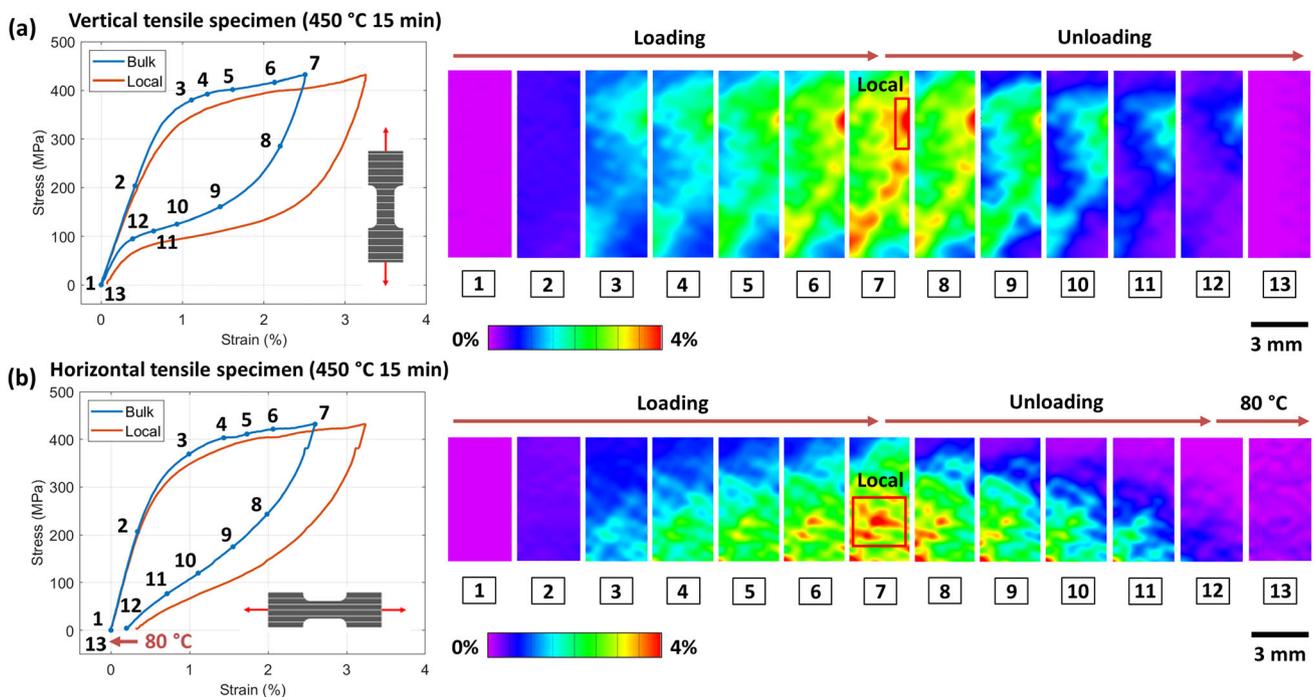
A similar behavior is shown by the aged specimen. In this case, a residual deformation of 0.2% is reported. In this case, the specimen was heated up to 80 °C after unloading to induce the residual martensite to reversely transform into austenite. Then, the specimen was further analyzed with DIC, and the results are shown in Fig. 10b. It was found that the residual strains have almost totally recovered,

indicating that the transformation temperatures are slightly higher than that of testing. Finally, the solution-treated and aged specimen was deformed up to 3% and showed a residual deformation of 0.5%.

As a final remark, it is worth noting that the values of  $\epsilon_{PE}$  reported in Fig. 9c allow to point out that the material in the heat-treated state shows higher local values of strain with respect to the global values, while in the as-built state the local and global behavior are almost the same. Moreover, Fig. 9c shows that the values of critical stress are not comparable between vertical and horizontal specimens, thus indicating a possible asymmetry in the mechanical behavior of the material depending on loading direction. The highest  $\sigma_{PE}$  was reported in the as-built condition (i.e., 625 MPa) for the vertical specimens and in the solution-treated and aged condition (i.e., 537 MPa) for the horizontal specimens. For both vertical and horizontal samples, the lowest  $\sigma_{PE}$  was shown after aging at 450 °C (i.e., 361 MPa and 336 MPa for the vertical and the horizontal specimens, respectively).

## Conclusions

This work shows that LPBF is a process suitable to manufacture NiTi components with sound functional properties and provides valuable data that can be of interest for the exploitation of 3D printed NiTi pseudoelastic structures. The main findings can be summarized as follows:



**Fig. 10** Stress–strain curves and DIC contours for two tensile specimens aged at 450 °C: **a** the vertical and **b** the horizontal specimen

- The tested specimens exhibited pseudoelastic behavior at room temperature both in compression and in tension, in all the testing conditions.
- Post-process heat treatments were found to highly influence the pseudoelastic features of the material. Solution annealing at 850 °C increases the critical stress for the martensitic transformation, while aging between 400 and 500 °C decreases it.
- Regarding the mechanical behavior in compression, the highest values of recoverable strain were reported for the as-built condition (i.e., 4.56% globally and 4.60% locally). The highest value of critical stress (i.e., 480 MPa) was achieved by the specimen solution treated at 850 °C, while the lowest values (i.e.,  $277 \div 265$  MPa) were shown by the specimens aged between 400 and 500 °C.
- The tensile specimens showed the highest strain recoverability after solution annealing at 850 °C followed by aging at 450 °C. The reported strain values were 2.14% globally and 2.74% locally for the vertical specimen, and 1.83% globally and 2.67% locally for the horizontal sample. Nevertheless, significant differences in the behavior of vertical and horizontal specimens can be pointed out, likely attributed to the different building orientations. The vertical specimen exhibited a lower value of critical stress (i.e., 372 MPa) with respect to the horizontal specimen (i.e., 537 MPa). Moreover, the horizontal specimen showed a residual strain of 0.5%, while the vertical specimen was able to recover completely.

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