



Article

Influence of Laser Spot Size on the Microstructure, Transformation Temperatures, and Ni Content of a Nickel-Rich LPBF Ti-Ni Alloy

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Abstract

This study investigates the influence of laser spot size ($\varnothing 100\ \mu\text{m}$ vs. $\varnothing 50\ \mu\text{m}$) on the microstructure, transformation behavior, and hardness of Ti-51.17 at.%Ni shape memory alloy samples fabricated by laser powder bed fusion. Samples were produced using identical processing parameters covering laser powers of 50–150 W, scanning speeds of 500–750 mm/s, and matched volumetric energy densities (28–83 J/mm³), with spot size as the only variable. Although both laser spot sizes produced comparable melt pool geometries and predominantly B2 austenite matrices with minor fractions of B19' martensite, the $\varnothing 50\ \mu\text{m}$ samples exhibited finer microstructures, higher residual stresses, and lower martensitic transformation temperatures in the as-built state. Heat treatment at 800 °C reduced residual stresses and partially homogenized the microstructure; however, differences in their transformation behavior remained. These differences were attributed to the spot-size-dependent Ni evaporation. Although transformation temperatures increased with increasing energy density for both conditions, the $\varnothing 50\ \mu\text{m}$ samples consistently showed lower values. In the 55–83 J/mm³ volumetric energy density range, the estimated Ni content ranged from 51.15 to 50.42 at.%, and the $\varnothing 50\ \mu\text{m}$ samples showed approximately 0.1–0.2 at.% greater Ni loss than their $\varnothing 100\ \mu\text{m}$ counterparts. These findings demonstrate that Ni evaporation is a laser-spot-size-dependent phenomenon that must be considered when processing near-equiatomic Ti-Ni alloys.

Keywords: Ti-Ni shape memory alloy; additive manufacturing; laser powder bed fusion; Ni evaporation; Ni loss; laser spot size effect; compositional change; transformation temperature; lattice parameter



Academic Editor: David L. Sales

Received: 14 July 2026

Revised: 30 July 2026

Accepted: 4 August 2026

Published: 6 August 2026

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1. Introduction

Ti-Ni shape memory alloys (SMAs) are widely used in biomedical devices, including orthodontic archwires, guidewires, and vascular stents, as well as in numerous engineering applications. Despite being intermetallic compounds, near-equiatomic Ti-Ni alloys exhibit remarkable ductility, often exceeding 50% elongation [1], which places them among the most practical and reliable SMAs. Their functional behavior arises from a reversible, diffusionless transformation between B2-austenite and B19' martensite. Under mechanical load, B2 transforms into stress-induced B19' martensite. Upon unloading or heating, this martensite reverts to austenite, enabling full shape recovery through superelasticity or the shape-memory effect.

The transformation is characterized by four temperatures: the austenite start (A_s) and finish (A_f), and the martensite start (M_s) and finish (M_f). These temperatures determine whether the alloy exhibits superelasticity or shape memory effect at a given operating temperature. Perfect superelasticity occurs when $A_f < T < T_s$, where T_s is the critical temperature above which martensitic transformation is suppressed and plastic slip dominates [2]. In the $A_s < T < A_f$ range, both superelasticity and shape memory effect contribute. When $M_f < T < M_s$, austenite and martensite coexist, and the shape memory effect dominates. Importantly, the transformation temperatures are extremely composition-sensitive: a change in only 0.1 at.%Ni can shift A_s , A_f , M_s and M_f by about 10 °C. For example, solution-treated Ti-50.6 at.%Ni shows superelasticity around −52 °C [3], while its Ti-50.0 at.%Ni counterpart behaves similarly near 90 °C [4].

Processing history also plays a major role. Plastic deformation introduces dislocations and internal stresses that stabilize martensite and shift transformation temperatures [5]. Heat treatments above A_f partially relieve these effects. For example, in a Ti-50.0 at.%Ni alloy subjected to 20% deformation, increasing the annealing temperature from 350 to 500 °C reduces M_s from 62 to 53 °C and A_f from 104 to 103 °C [6]. After 40% deformation and annealing at 500 °C, M_s and A_f decrease further and become, respectively, 45 and 90 °C. As a result, depending on processing history, near-equiatomic alloys may exhibit at room or human-body temperature, either superelasticity or shape memory effect, or their combination [7]. Grain size also influences transformation behavior: nanocrystalline structure (~60 nm) can suppress martensitic transformation entirely [8], whereas coarse-grained materials (millimeter scale) show negligible grain-boundary effects.

Additive manufacturing, especially laser powder bed fusion (LPBF), introduces additional complexity. First, Ti-Ni powders with tightly controlled Ni content (± 0.1 at.%) are not widely available. Second, rapid melting and solidification during the process can cause Ni evaporation, form non-equilibrium microstructures and generate residual stresses [9,10], all of which influence the transformation behavior. Even slight Ni loss during processing can significantly alter functional properties. Strategies such as using Ni-rich powders [11] or adjusting LPBF parameters [12] help mitigate such composition shifts. In general, increasing VED promotes greater Ni evaporation, leading to lower Ni contents, higher martensitic transformation temperatures, and reduced superelasticity at room temperature. For example, a Ti-50.6 at.% Ni powder processed at volumetric energy densities (VED) of 111 and 126 J/mm³ yielded final compositions of 50.4 and 50.0 at.% Ni, respectively [13]. Similarly, processing Ti-50.8 at.% Ni powder at VED of 42 and 52 J/mm³ produced alloys containing 50.5 and 50.2 at.% Ni, respectively [14]. However, excessive energy input may reverse this trend due to changes in melt pool dynamics and thermal history [15,16]. Post-processing treatments also influence the transformation behavior: solution annealing typically decreases transformation temperatures, whereas subsequent aging increases them, with longer aging times producing a more pronounced rise [17].

Although using optimized alloy compositions and LPBF process conditions can ensure that targeted transformation temperatures and functional properties are achieved [18], differences between LPBF systems, such as laser spot size, can also affect Ni evaporation. In general, smaller laser spots generate higher local heat intensities, promoting melt pool instability, spatter formation, and Ni loss, whereas larger spots distribute the energy more evenly, reducing evaporation and improving melt pool stability [19]. This study investigates how two different LPBF systems operating at similar energy densities and build rates, but employing different laser spot sizes, influence the microstructure, phase composition, transformation temperatures, and Ni loss in near-equiatomic Ti-Ni alloys.

2. Materials and Methods

The starting powder was a Ti-Ni feedstock supplied by Avimetal Powder Metallurgy Technology Co., Ltd., Beijing, China, with a nominal composition of Ti-51.17at.%Ni according to the manufacturer's specification. The particle size distribution measured using an LS 13 320 XR apparatus (Beckman Coulter, Indianapolis, IN, USA) ranged from 15 to 53 μm .

Two independent LPBF systems were used in this study. The first system was an EOSINT M280 (EOS. GmbH., Krailling, Germany) equipped with a 400 W continuous-wave Ytterbium fiber laser operating at a wavelength of 1075 nm and a laser spot size of $\varnothing 100 \mu\text{m}$. The second system, a TruPrint 1000 (Trumpf, Ditzingen, Germany), also employed a 200 W continuous-wave Ytterbium fiber laser operating at 1070 nm but with a smaller laser spot size of $\varnothing 50 \mu\text{m}$. All builds were performed under argon with an oxygen concentration below 100 ppm.

Five (5) identical printing parameter sets (Table 1) were applied on both machines covering a laser power range from 50 to 150 W and a scanning speed range from 500 to 1000 mm/s. On both systems, the layer thickness (t) was set to 30 μm , and the hatch spacing (h) to 80 μm . A scanning strategy of hatch angle rotations of 90° and 67° between successive layers was used for the EOSINT M280 and TruPrint 1000 systems, respectively.

Table 1. Processing parameters.

Samples	Laser Spot Size Independent Process Parameters					Laser Spot Size Specific Process Parameters			
	P (W)	v (mm/s)	LED (J/mm)	VED (J/mm ³)	BR (cm ³ /h)	$\varnothing 100 (\mu\text{m})$		$\varnothing 50 (\mu\text{m})$	
						Ei (J/mm ³)	SPE (J)	Ei (J/mm ³)	SPE (J)
#1	50	750	0.07	28	6.5	8.5	0.007	34.0	0.003
#2	100	750	0.13	56	6.5	17.0	0.013	67.9	0.007
#3	150	750	0.2	83	6.5	25.5	0.020	101.9	0.010
#4	100	500	0.2	83	4.3	25.5	0.020	101.9	0.010
#5	66.5	500	0.13	55	4.3	16.9	0.013	67.8	0.007

Five aggregated process metrics were used in this study: linear energy density (LED), volumetric energy density (VED), build rate (BR), energy intensity (Ei), and specific point energy (SPE). The linear energy density ($\text{LED} = P/v$) quantifies the energy delivered per unit length of the scan track during LPBF single-track fabrication. The volumetric energy density ($\text{VED} = P/(v \cdot h \cdot t)$) quantifies the energy input per unit volume for bulk samples, while the build rate ($\text{BR} = v \cdot h \cdot t$) characterizes the deposition productivity. The selected VED-BR combinations (Table 1, Figure 1) correspond to the processing window known to produce Ti-Ni components with >99.9% density [20]. Samples #1, #2 and #3 were fabricated at a constant scanning speed of 750 mm/s and BR of 6.5 cm³/h, while varying VED from 28 to 83 J/mm³ by increasing laser power from 50 to 150 W. Samples #4 and #5 were printed at lower constant scanning speeds of 500 mm/s and BR of 4.3 cm³/h, but with laser powers of 100 and 66.5 W and VED of 83 and 55 J/mm³, respectively.

Also, with the objective of assessing the effect of laser spot size (d), two additional aggregated process metrics were introduced: (i) the energy intensity ($\text{Ei} = 4P/(v \cdot \pi \cdot d^2)$) [21] and (ii) the specific point energy ($\text{SPE} = P \cdot d/v$) [22]. Both metrics depend directly on the laser spot diameter and could influence the local melt pool temperature and vaporization recoil pressure [23]. For the $\varnothing 100 \mu\text{m}$ laser spot, Ei ranged from 8.5 to 25.5 J/mm³ and SPE from 0.007 to 0.013 J. For the $\varnothing 50 \mu\text{m}$ laser spot, Ei ranged from 34 to 67.8 J/mm³ and SPE from 0.003 to 0.007 J (Table 1).

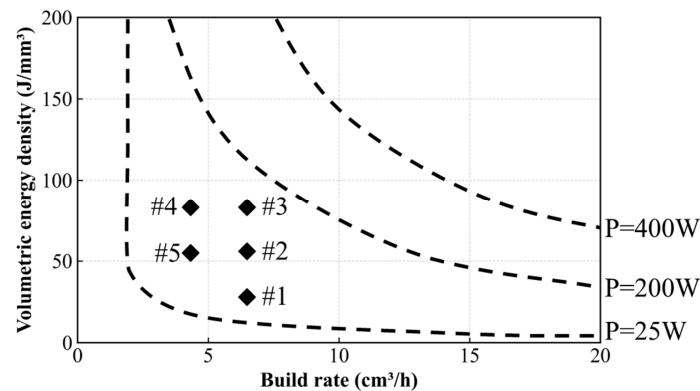


Figure 1. LPBF process parameters in the VED-BR space.

Samples of different types were manufactured: (i) single-track deposits on a solid Ti-Ni substrate covered with a 30 μm powder layer (Figure 2a), and (ii) cylindrical bulk samples ($\varnothing 7 \text{ mm} \times 15 \text{ mm}$) for thermal analysis, density, hardness and microstructural characterization (Figure 2b).

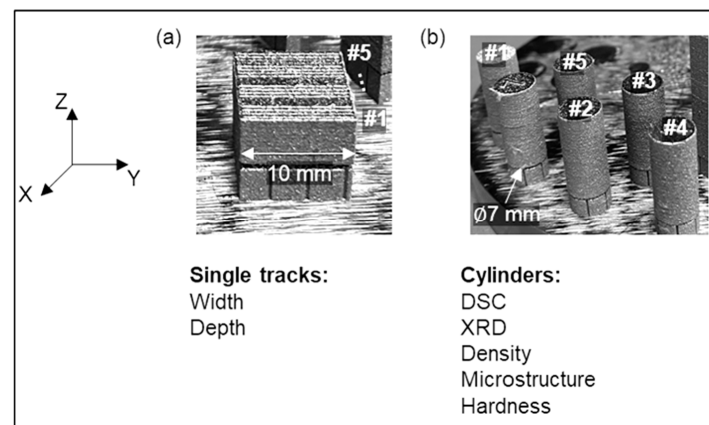


Figure 2. Printed samples: (a) single track deposits; (b) cylindrical bulk samples for thermal analysis, density, hardness, and microstructural characterization.

After fabrication, part of the samples was retained in the as-built state, and the remainder were subjected to solution annealing at 800 $^{\circ}\text{C}$ for 30 min in a Borel KN 1050-320 tilting furnace (Borel Swiss, Delémont, Switzerland). The heat treatment of 800 $^{\circ}\text{C}$ for 30 min was selected to relieve residual stresses, homogenize the microstructure, and avoid the formation of Ni-rich or oxygen-containing Ti-rich brittle phases [24]. Samples were water-quenched immediately after annealing to preserve the solid-solution structure and prevent the formation of precipitates.

All samples were mechanically polished using SiC papers (p320, p600 and p800), followed by polishing with 9 μm diamond suspension and colloidal silica. Final etching in a HF, HNO₃ and H₂O (1:3:10) solution was performed to remove polishing-induced deformation and reveal the microstructure.

Differential scanning calorimetry (DSC) testing was performed using a DSC2500 system (TA Instruments, New Castle, DE, USA). Measurements were conducted between -85°C and $+100^{\circ}\text{C}$ with heating and cooling rates of 10 K/min. Transformation temperatures M_s , M_f , A_s , A_f were extracted using the tangent method (ASTM F2004-2017 [25]), and the peak temperatures (M_p and A_p) were also recorded. Repeated heating/cooling cycles produced highly consistent transformation peaks, confirming measurement repeatability.

X-ray diffraction (XRD) analysis was carried out at 20 $^{\circ}\text{C}$, 75 and 100 $^{\circ}\text{C}$ using an Anton Paar XRDynamic 500 diffractometer (Anton Paar GmbH, Graz, Austria) with Cu K α

radiation (40 kV, 49 mA). Scans were conducted over $2\theta = 35\text{--}80^\circ$, with a step size of 0.02° , for a total acquisition time of 1 h 35 min per sample. Phase identification of the austenitic (B2) and martensitic (B19') structures was performed using 04-015-0487 and 04-007-4052 ICDD cards. The B2 lattice parameter was calculated using (110), (200) and (211) diffraction peaks and the Nelson–Riley extrapolation method [26].

Local analytical techniques such as EDX (Hitachi SU-8230 SEM, Tokyo, Japan), spark emission spectroscopy (SPECTROMAXx Metal Analyzer from AMETEK, Berwyn, PA, USA) and electron probe microanalysis (EPMA, Cameca SX100 FiveFE system (Ametek, Gennevilliers, France)) were used to estimate matrix composition; their typical scatter ($\sim 0.5\text{--}1$ at.%) is too large for the present study, because compositional variations as small as 0.1 at.% can already shift transformation temperatures in near-equiatomic Ti-Ni alloys by ~ 10 K [27–33]. ICP measurements could indeed provide a higher bulk composition accuracy. However, these measurements may be affected by the dissolution of LPBF-induced surface oxides or oxygen-rich surface films during sample preparation, leading to values that do not necessarily represent the effective composition of a B2 matrix.

Therefore, an alternative approach was adopted in this study. Instead of relying on direct compositional measurements, the Ni content was estimated by combining two experimentally established correlations: (i) the dependence of the martensite start temperature (M_s) on Ni content, and (ii) the dependence of the B2 austenite lattice parameter on Ni content.

The optical microscopy analysis of the melt pools and microstructures was conducted using a Keyence VHX-7000 microscope (Keyence Corp., Osaka, Japan). The melt pool widths (top view, X-Y plane) were obtained from eight measurements along each track, and the results are presented as means $\pm$ standard deviations. Cross-sectional melt pool dimensions were measured after repeated polishing and etching. The microstructure features were measured using the line-intercept method in accordance with ASTM E112. For each condition, several representative micrographs were selected, and a set of test lines was superimposed at different orientations (0° , 45° , 90°) across the image. At least 100 grain-boundary intercepts were collected per condition, which is sufficient to obtain statistically meaningful mean values and standard deviations.

Microtomography measurements were performed using a Nikon XTH 225 system (Nikon, Tokyo, Japan) for parts produced using the $\varnothing 100$ μm system and an Yxlon FF-35 (Comet Yxlon GmbH, Hamburg, Germany) for the $\varnothing 50$ μm system samples. Both acquisitions voxel sizes were set to 10 μm . The relative density was measured using Dragonfly software (Comet Yxlon GmbH, Hamburg, Germany) by comparing the volume fraction of pores with the volume fraction of melted material using manual thresholding. Micro-CT provides a full-volume measurement of porosity; therefore, each scan can be considered statistically representative of the entire sample.

Vickers microhardness tests were carried out on the X-Y plane (perpendicular to the build direction) of the specimens following the ASTM E92/E384 standard and using a Struers Duramin-40 M1 tester (Struers, Ballerup, Denmark) with a diamond indenter. Nine indentations per sample were performed at 500 gf with a 15 s dwell time, maintaining a spacing of at least 2.5 times the indent diagonal. Results are presented as means $\pm$ standard deviations.

3. Results

3.1. Single-Track Morphology and Melt Pool Characteristics

The formation of dense parts in LPBF relies on a controlled overlap of successive melt tracks, making track morphology and melt pool geometry critical indicators of process stability and fusion quality. Figure 3 presents representative top-view and cross-sectional

optical micrographs of single tracks produced using two laser spot diameters: $\varnothing 100\ \mu\text{m}$ and $\varnothing 50\ \mu\text{m}$. From a track morphology standpoint, the top-view images reveal comparable track continuities between both spot sizes across similar processing conditions. A clear transition in melting regimes is observed when increasing LED from 0.07 to 0.2 J/mm, which corresponds to 28 and 83 J/mm³ for bulk VED. At low energy inputs, the process is dominated by insufficient melting, leading to discontinuous and unstable tracks characterized by balling (Figure 3a,f). As the LED increases to intermediate values (0.13 J/mm or 56 J/mm³ for VED), the process shifts toward a stable regime, where continuous tracks are formed due to improved wetting and material redistribution (Figure 3b,e,g,j). At the highest LED (0.2 J/mm or 83 J/mm³ for VED), further spreading of the molten material results in wider tracks, indicating increased thermal input and fluid flow (Figure 3c,d,h,i).

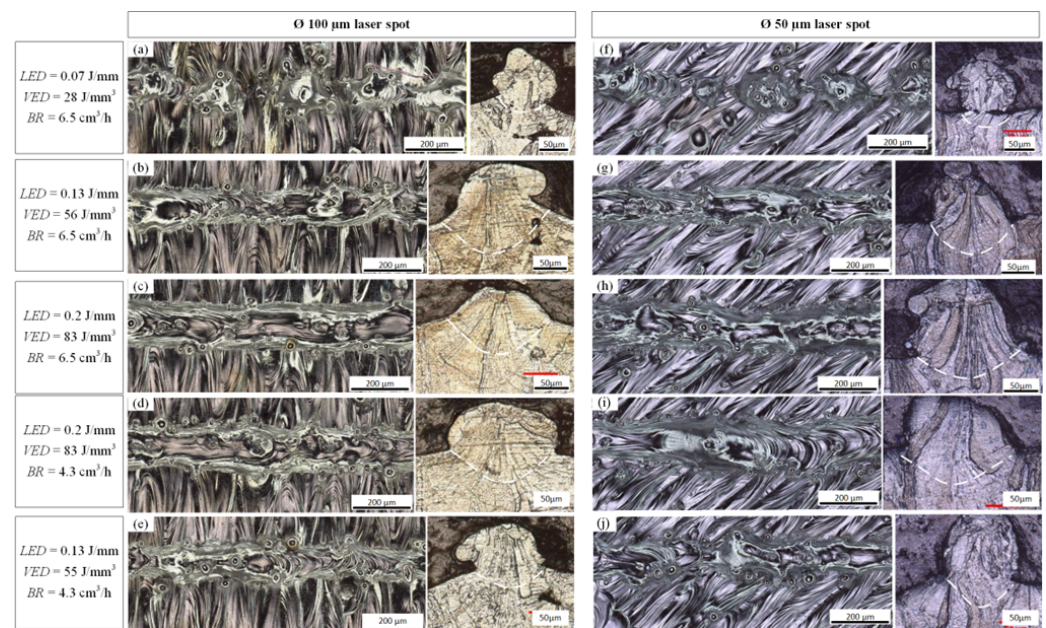


Figure 3. Top-views and cross-sections of single tracks processed using 100 μm (a–e) and 50 μm (f–j) spot sizes where (a,f) correspond to sample #1, (b,g) #2, (c,h) #3, (d,i) #4, and (e,j) #5.

These transitions are reflected in the melt pool geometry. Low linear energy densities ($\text{LED} = 0.07\ \text{J/mm}$ or $\text{VED} = 28\ \text{J/mm}^3$) produce shallow melt pools with limited penetration, while higher linear energy inputs ($0.13\text{--}0.2\ \text{J/mm}$ or $55\text{--}83\ \text{J/mm}^3$) lead to deeper and more elongated geometries. The absence of excessive penetration depth or instability suggests that the process remains within a conduction-mode melting regime across the investigated range.

While LED governs the overall melting behavior, the influence of the LPBF system becomes evident when comparing melt pool morphology in cross-section micrographs. A smaller laser spot diameter is expected to generate more localized energy input, resulting in higher peak temperatures and steeper thermal gradients. This should promote stronger melt pool convection and recoil pressure, leading to material redistribution and the formation of elevated tracks in cross-section. Indeed, tracks produced with a $\varnothing 50\ \mu\text{m}$ laser spot exhibit a pronounced increase in track height in cross-section compared to those produced with a $\varnothing 100\ \mu\text{m}$ laser spot. Material accumulation toward the trailing edge of the melt pool inevitably leads to increased surface roughness, with the solidified tracks occasionally exceeding the nominal layer thickness (30 μm).

The effect of linear energy density (LED) on the single-track width and depth is shown in Figure 4. A clear dependence of the track width on LED is observed for tracks #1–#5 in Figure 4a. The width increases from track #1 to reach a maximum for tracks #3 and

#4, followed by a reduction for track #5. Both the $\varnothing 50$ and $100\ \mu\text{m}$ tracks show similar trends, although slight systematic differences exist between them. This confirms that increasing energy input promotes melt pool/track broadening due to enhanced lateral heat diffusion. Tracks produced with different spot sizes but with similar LED values, such as #2 and #5 ($0.13\ \text{J/mm}$) or #3 and #4 ($0.2\ \text{J/mm}$), show slight differences in melt pool width, highlighting distinct contributions from the laser power and scanning speed embedded within LED. Note, however, that no evident or systematic trends were observed across the tested conditions for the laser spot size-dependent aggregated process metrics, such as the energy intensity (E_i) and the specific point energy (SPE). This indicates these metrics capture only partial aspects of the laser–matter interaction, and, therefore, cannot be correlated with variations in the single-track width.

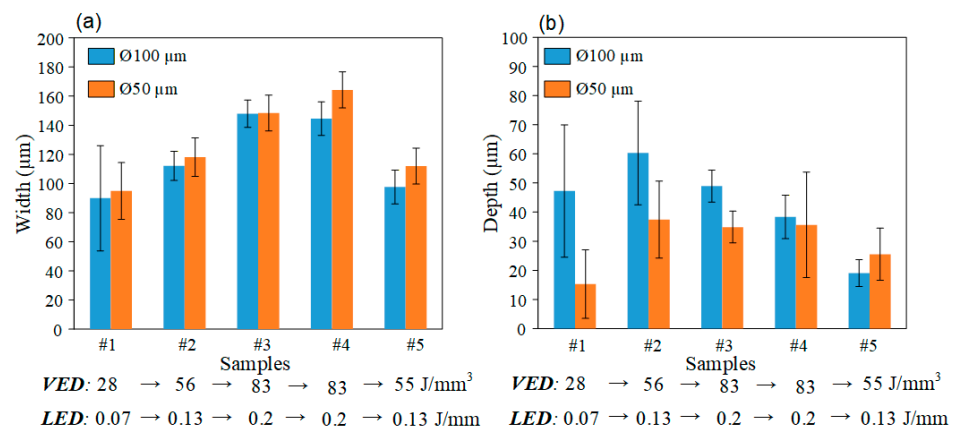


Figure 4. Width (a) and depth (b) of single tracks.

In contrast to the melt pool width, the melt pool depth does not exhibit a strong or monotonic increase with processing parameters. Part of the scatter arises from a relatively low contrast of the melt pool boundaries in Ti-Ni, which introduces uncertainty in depth measurements. Overall, the depth increases from track #1 to track #2 but then progressively decreases toward track #5. For the $\varnothing 100\ \mu\text{m}$ condition, track #1 shows a relatively shallow melt pool with high variability, consistent with unstable melting at low energy inputs.

3.2. Density of Bulk Samples

Figure 5 shows micro-CT images of bulk samples produced using both laser spots and Table 2 contains their respective density values. Note that while CT scans effectively detect large pores, the measurements are limited by voxel resolution and may overlook sub-micron defects, leading to a slight overestimation of printed density.

Samples #2–#5 produced with the $\varnothing 100\ \mu\text{m}$ spot size using VED of 55–83 J/mm^3 achieved nearly full densification, with CT-measured density of approximately 99.9–100% (Figure 5a, Table 2). In contrast, samples fabricated with the $\varnothing 50\ \mu\text{m}$ spot size exhibited a slightly lower relative density of 99.5–99.9% (Figure 5b and Table 2). In addition to the printing density values, the corresponding melt pool width-to-hatching space (W/h) and melt pool depth-to-layer thickness (D/t) ratios are also added to Table 2. It can be observed that the W/h values vary between 1.1 and 2.1 for both spot sizes, meaning that adjacent tracks always overlap, ensuring adequate lateral bonding between successive laser tracks [9]. The slightly reduced densification observed for the $\varnothing 50\ \mu\text{m}$ spot size is consistent with its shallower melt pool depth, which limits vertical overlap between successive layers.

At the lower energy density of 28 J/mm^3 , the melt pool is simultaneously shallower and narrower. This results in incomplete fusion of adjacent tracks and layers, producing lack-of-fusion porosity, preventing strong interlayer bonding for both spot sizes (Figure 5,

sample #1). Notably, the $\varnothing 50\ \mu\text{m}$ sample #1 exhibits, once again, a lower relative density than its $\varnothing 100\ \mu\text{m}$ counterpart.

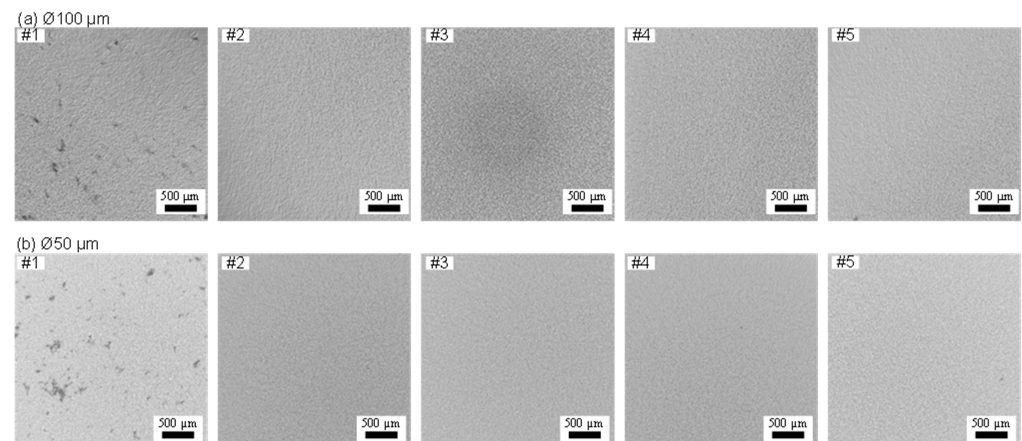


Figure 5. Micro-CT images of samples produced with $\varnothing 100\ \mu\text{m}$ (a) and $\varnothing 50\ \mu\text{m}$ (b) laser spot sizes.

Table 2. Melt pool width-to-hatching ratios and relative densities of bulk samples.

Samples	$\varnothing 100\ \mu\text{m}$ Laser Spot			$\varnothing 50\ \mu\text{m}$ Laser Spot		
	Density	W/h	D/t	Density	W/h	D/t
#1	98.81	1.1	1.6	96.90	1.2	0.5
#2	99.99	1.4	2.0	99.53	1.5	1.2
#3	99.98	1.8	1.6	99.99	1.9	1.2
#4	100.00	1.8	1.3	99.99	2.1	1.2
#5	100.00	1.2	0.6	99.84	1.4	0.9

These findings confirm that dense Ti-Ni parts (>99% relative density) can be reliably fabricated on both EOSINT M280 and TruPrint 1000 systems using a hatching space of $80\ \mu\text{m}$ and a layer thickness of $30\ \mu\text{m}$ as far as the volumetric energy density is maintained between 55 and $83\ \text{J}/\text{mm}^3$. These results indicate that maintaining the VED within a stable range is a key condition for achieving reproducible LPBF processing of Ti-Ni alloys across both systems of this study.

3.3. Thermal Behavior

Figure 6 shows the cooling and heating DSC curves for the powder, as-built, and heat-treated samples. All samples exhibit the characteristic exothermic peak during cooling ($B2 \rightarrow B19'$) and the endothermic peak during heating ($B19' \rightarrow B2$). The peak temperatures M_p and A_p correspond to the maximum transformation rate, while the peak areas represent the transformation enthalpy and are proportional to the fraction of transformed material. The positions of M_p and A_p temperatures are marked in Figure 6, and the numerical values can be found in Table A1.

The powder exhibits relatively broad peaks (Figure 6a), consistent with the heterogeneous powder microstructure and the high dislocation density, lattice defects, and internal stresses. The XRD peaks of the as-built samples are also broadened, although to a lesser extent than those of the powder, reflecting an improved material homogeneity (Figure 6b,c). Note that none of the as-built LPBF samples show R-phase shoulders, indicating a direct $B2 \rightarrow B19'$ transformation. As-built sample #1 ($28\ \text{J}/\text{mm}^3$) exhibits M_p and A_p temperatures that are overall lower than those of the initial powder (see Figure 7). Increasing VED from 28 to $83\ \text{J}/\text{mm}^3$ (samples #1 to #3) shifts both M_p and A_p to higher temperatures for both spot sizes. For example, M_p increases by $\sim 42\ ^\circ\text{C}$ between samples #1 and #3, while A_p

increases by $\sim 58^\circ\text{C}$ for $\varnothing 100\ \mu\text{m}$ and $\sim 34^\circ\text{C}$ for $\varnothing 50\ \mu\text{m}$. Samples #3 and #4 exhibit the highest M_p and A_p temperatures among all samples, with values that are close to or even higher than those of the initial powder (Figure 7).

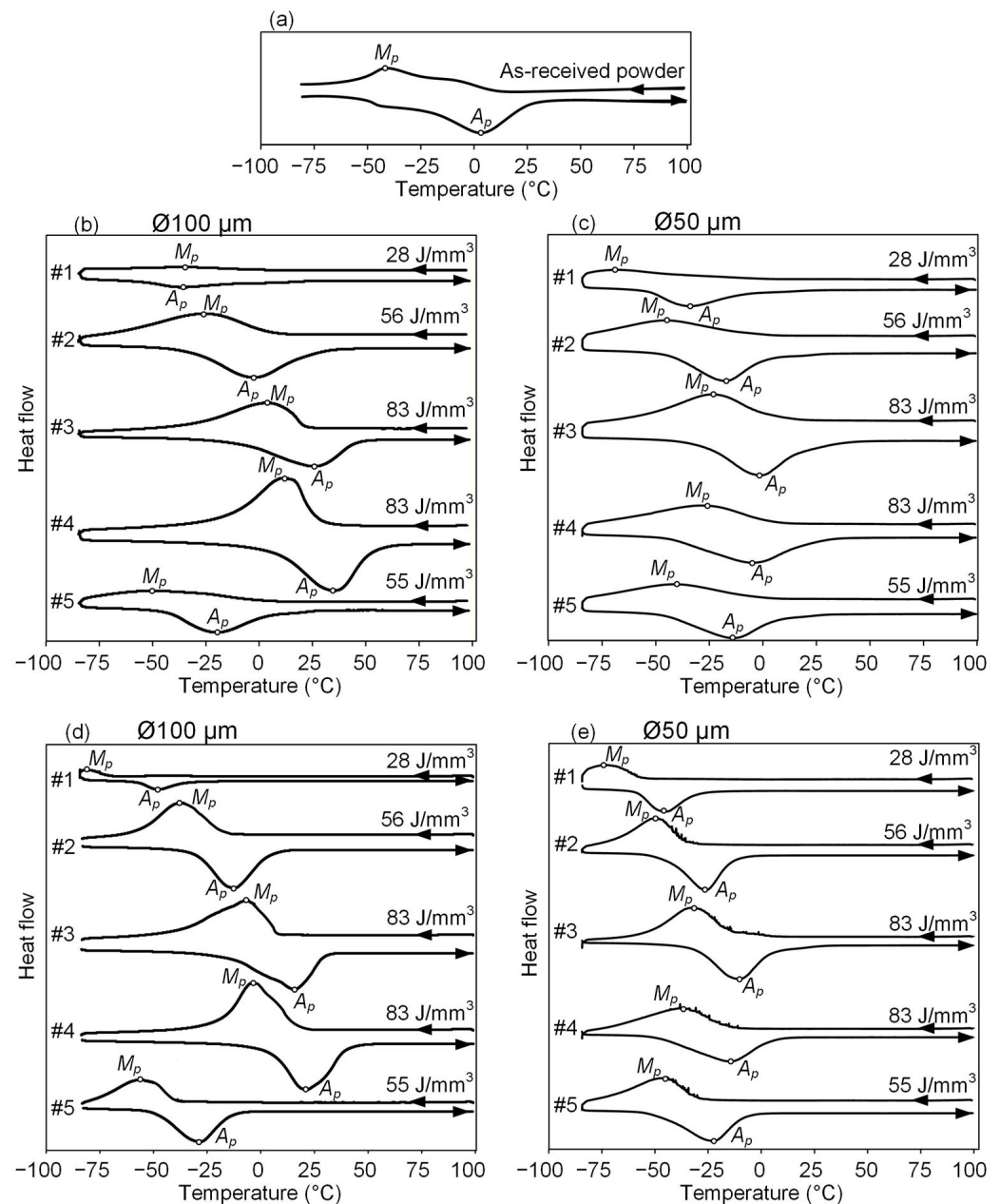


Figure 6. DSC results for powder (a), as-built (b,c) and heat-treated (d,e) samples produced using $\varnothing 100\ \mu\text{m}$ (b,d) and $\varnothing 50\ \mu\text{m}$ (c,e) spot sizes.

The characteristic transformation temperatures (M_s , M_f , A_s , A_f) describe the onset and completion of the direct and reverse martensitic transformations and their values are plotted in Figure 8 for the as-built and heat-treated samples, respectively. The samples exhibit variable differences in their transformation temperatures relative to those of the initial powder, reflecting the influence of the LPBF process and its parameters on the resulting transformation behavior. For both spot sizes, the lowest VED results in lower transformation temperatures than the powder, whereas increasing VED shifts all characteristic temperatures upward. The $\varnothing 100\ \mu\text{m}$ samples #3 and #4 show M_s , M_f , A_s , and A_f values that exceed those of the powder, suggesting that microstructural state, residual stresses, and Ni depletion induced by increased VED collectively govern the transformation behav-

ior. Furthermore, A_f exceeds room temperature for $\varnothing 100 \mu\text{m}$ samples #2–#5 and $\varnothing 50 \mu\text{m}$ samples #3 and #4, indicating the coexistence of B2 and B19' phases at this temperature. M_f increases with VED but remains below 0°C for all samples. The M_s – M_f interval shows a clear dependence on VED. Generally, in Ti–Ni alloys, such an increase in the temperature difference (M_s – M_f) is typically associated with internal stresses [34]. Note that, for sample #1, the M_f cannot be reliably determined because it lies near or below the DSC measurement range. For samples #2 and #3, increasing VED narrows the M_s – M_f interval for both spot sizes, indicating more homogeneous transformation behavior at higher energy inputs. In contrast, despite similar VED (55 J/mm^3), sample #5 shows a wider M_s – M_f interval than sample #2, revealing greater levels of residual stresses and local heterogeneity.

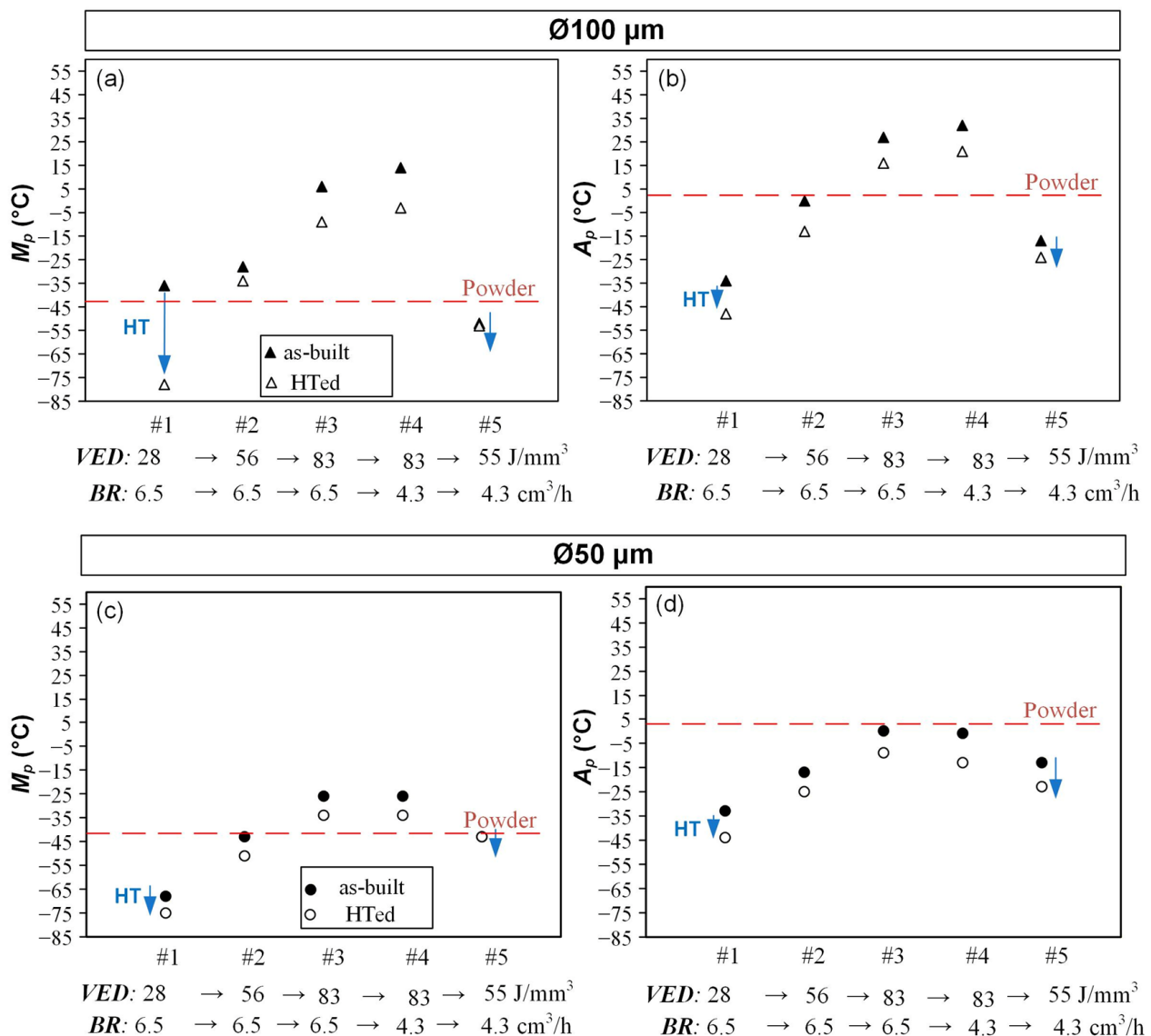


Figure 7. M_p (a,c) and A_p (b,d) temperatures of the as-built and heat-treated samples produced with $\varnothing 100 \mu\text{m}$ (a,b) and $\varnothing 50 \mu\text{m}$ (c,d) laser spots.

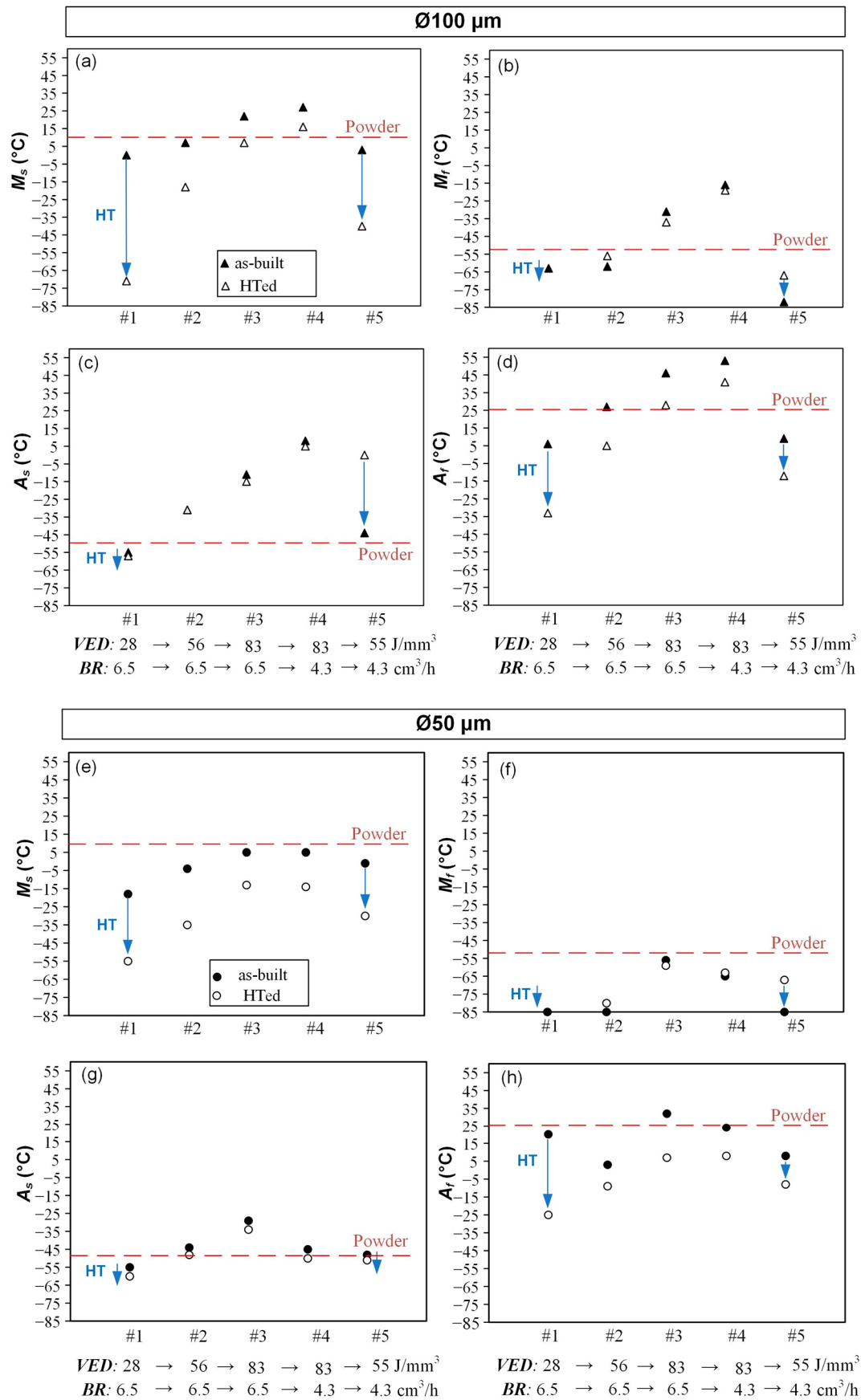


Figure 8. M_s (a,e), M_f (b,f), A_s (c,g) and A_f (d,h) temperatures of the as-built and heat-treated samples produced with Ø100 µm (a–d) and Ø50 µm (e–h) laser spots.

The effect of BR can be assessed by comparing samples fabricated at similar VED (samples #2 vs. #5 at $\sim 55 \text{ J/mm}^3$ and samples #3 vs. #4, at 83 J/mm^3), as shown in Figure 8. At 55 J/mm^3 , a lower BR ($4.3 \text{ vs. } 6.5 \text{ cm}^3/\text{h}$) decreases all transformation temperatures (M_p , A_p , M_s , M_f , A_s , A_f) of the $\varnothing 100 \text{ }\mu\text{m}$ samples by more than 10°C . In contrast, $\varnothing 50 \text{ }\mu\text{m}$ samples (#2 and #5) show nearly identical transformation temperatures, falling between those of the corresponding $\varnothing 100 \text{ }\mu\text{m}$ samples. At 83 J/mm^3 , the trend reverses for the $\varnothing 100 \text{ }\mu\text{m}$ spot size: sample #4 (lower BR) exhibits M_p , A_p , M_s , M_f , A_s and A_f values that are more than 5°C higher than those of sample #3. The $\varnothing 50 \text{ }\mu\text{m}$ samples again show limited BR sensitivity for M_p and A_p , although sample #3 (higher BR) exhibits slightly higher M_f , A_s , and A_f values. Overall, these results indicate that BR has a stronger and more complex influence on transformation temperatures for the $\varnothing 100 \text{ }\mu\text{m}$ spot size, whereas the $\varnothing 50 \text{ }\mu\text{m}$ spot size exhibits comparatively weak BR dependence.

When comparing samples processed at the same VED and BR but different spot sizes, M_p and A_p are almost always lower for the smaller laser spot ($\varnothing 50 \text{ }\mu\text{m}$). Depending on the VED and BR values, a decrease in the M_p temperature ranges from ~ 10 to 40°C and that in the A_p temperature from ~ 1 to 30°C , when the spot size decreases from $\varnothing 100$ to $\varnothing 50 \text{ }\mu\text{m}$. A similar trend is observed for all transformation temperatures: M_s and A_f differ by $\sim 1\text{--}30^\circ\text{C}$, and M_f and A_s differ by $\sim 3\text{--}50^\circ\text{C}$, with the $\varnothing 50 \text{ }\mu\text{m}$ samples consistently exhibiting lower values. The reduced $M_s\text{--}M_f$ intervals observed for the $\varnothing 100 \text{ }\mu\text{m}$ samples indicate lower internal stress gradients and a more uniform microstructural state.

After heat treatment, sample #1 shows a slightly sharper exothermic peak, although the peak height remains low, indicating that only a limited fraction of martensite forms upon cooling of this sample to -85°C (Figure 6d,e). For the other samples, the exothermic and endothermic peaks become more pronounced, reflecting a larger transformed fraction and a more uniform transformation in the -85 to $+100^\circ\text{C}$ temperature range. Heat treatment shifts all transformation temperatures to lower values (Figure 8), with sample #1 exhibiting the largest decrease in the M_p , A_p , M_s , and A_f temperatures. This shift is consistent with the reduction in dislocation density, relaxation of internal stresses, and microstructure homogenization, as also reported in [35,36]. However, the magnitude of the temperature shift decreases with increasing VED, with A_s and M_f being less sensitive to heat treatment than their A_f and M_s counterparts. Despite these changes, the overall trends persist: higher VED results in higher transformation temperatures, and the influence of BR depends on both the laser spot size and the applied VED. At intermediate VED (55 J/mm^3), the larger spot size ($\varnothing 100 \text{ }\mu\text{m}$) shows a stronger BR effect, whereas at high VED (83 J/mm^3), the BR sensitivity diminishes or reverses, depending on the thermal history associated with each spot size. The remaining differences in transformation temperatures between the samples and the initial powder are likely governed by compositional variations (e.g., Ni/Ti ratio) and microstructural state than by the processing-induced heterogeneity alone.

In Ti-Ni, M_s and A_s temperatures are particularly important because they determine the activation of the shape memory and superelasticity effects, and they are particularly sensitive to composition and processing conditions. As a result, shifts in M_s and A_s provide a direct indication of changes in functional behavior. In our case, A_s shows less pronounced changes after heat treatment as compared to M_s , which increases from sample #1 to sample #4 and then slightly decreases for sample #5. For samples processed with similar VED (#2 vs. #5 at 55 J/mm^3 and #3 vs. #4 at 83 J/mm^3), $\varnothing 100 \text{ }\mu\text{m}$ samples show comparable M_s values, whereas $\varnothing 50 \text{ }\mu\text{m}$ samples exhibit larger variations, indicating a more pronounced influence of BR. Finally, in the 28 to 83 J/mm^3 VED range, the M_s temperatures of samples produced with two spot sizes differ by $\sim 10\text{--}20^\circ\text{C}$, with the M_s temperature being almost always lower for $\varnothing 50 \text{ }\mu\text{m}$ samples than for their $\varnothing 100 \text{ }\mu\text{m}$ counterparts.

3.4. Room Temperature Microstructure Evolution

Figure 9 represents the optical micrographs of Ti-Ni samples observed on planes (XY) perpendicular to the build direction, showing how the microstructure varies with processing conditions and spot sizes. Overall, samples #1, #2, and #5, produced at low and intermediate energy densities (28 and 56 J/mm³), exhibit broadly similar features between the Ø100 µm and Ø50 µm samples. In contrast, samples #3 and #4 reveal more pronounced spot-size-dependent differences.

At 28 J/mm³ (#1), both spot sizes produce microstructures containing visible porosity characteristic of lack-of-fusion defects, thus confirming the micro-CT measurements. These defects interrupt an otherwise near-equiaxed grain structure (50 ± 18 µm for Ø100 µm sample and 45 ± 14 µm for Ø50 µm sample). Increasing the energy density to 56 J/mm³ (sample #2) results in a dense and continuous microstructure. In the Ø100 µm sample, alternating light and dark regions reflect the 90° layer-rotation scanning strategy applied between consecutive layers (Figure 8a). The lighter regions correspond most likely to square- or rectangular-shaped B2 austenite grains (61 ± 17 µm on average) formed during primary melt-track solidification and epitaxial growth across melt pool boundaries. The darker regions likely represent melt pool overlap zones and remelting interfaces, containing either B19' martensite or fine B2 grains with different crystallographic orientations, which produce optical contrast. However, optical microscopy does not allow distinguishing B2 and B19' phases; phase identification is therefore based on the XRD data.

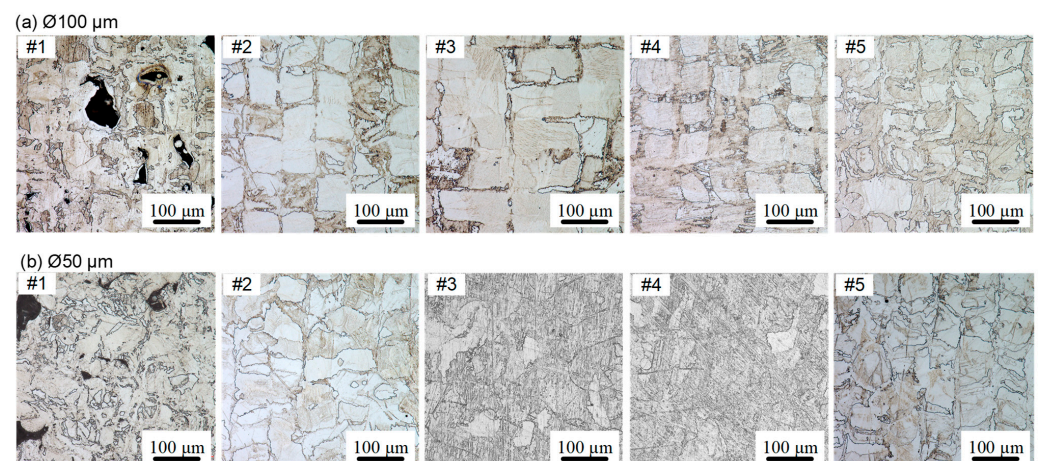


Figure 9. Microstructure of as-built samples produced with Ø100 µm (a) and Ø50 µm (b) spot sizes.

These overlap regions experience repeated thermal cycling, leading to microstructure refinement and local variations in phase composition, texture, and elemental distribution. Repetitive remelting can enhance Ni evaporation, causing local Ni depletion. Because reduced Ni content can increase the martensite start temperature (M_s), these regions may contain a higher fraction of B19' martensite at room temperature. The Ø50 µm sample #2 shows similar features (Figure 9b). Differences in the B2 grain morphology and size of structural elements between Ø50 and Ø100 µm samples are due to the difference in laser spot size and scanning strategy, which modifies thermal gradients and solidification rates.

For the Ø100 µm spot size, increasing the energy density from 55 to 83 J/mm³ (samples #2 and #3) enlarges the square- or rectangular-shaped grains (from 61 ± 17 µm to 91 ± 33 µm), indicating stronger texture development. The Ø100 µm sample #3 shows the most pronounced block-like morphology among all the samples (Figure 9a). In contrast, Ø50 µm sample #3 exhibits isolated light blocky islands (62 ± 21 µm) surrounded by darker regions, which likely contain finer grains or a higher martensite fraction, again indicating localized variations in composition and solidification behavior.

Sample #4, produced at the same VED as sample #3 but at a lower BR, shows finer blocky regions in both spot sizes ($77 \pm 22 \mu\text{m}$ for $\varnothing 100 \mu\text{m}$ samples and $59 \pm 18 \mu\text{m}$ for $\varnothing 50 \mu\text{m}$ samples). The reduced BR increases melt pool overlap, enhancing remelting and Ni evaporation. This likely promotes grain refinement, modifies crystallographic texture, and increases the fraction of martensite formed, particularly in $\varnothing 50 \mu\text{m}$ samples where the concentrated laser spot may produce steeper thermal gradients.

Despite its lower VED, sample #5 again exhibits a blocky morphology. This can be attributed to the combined influence of processing parameters that enhance remelting and Ni evaporation. However, the blocky structure is less pronounced than in sample #2, which was produced at a comparable VED. Overall, both energy density and build rate influence the melt pool overlap, grain size, and resulting distribution of B2 and B19' phases in the LPBF-processed Ti-Ni alloy.

Figure 10 shows the microstructure of samples after their heat treatment. The heat-treated $\varnothing 100 \mu\text{m}$ samples retain a microstructure very similar to their as-built condition (Figures 9a and 10a), indicating that, for these processing conditions, heat treatment at 800°C has only a limited effect on the recrystallization behavior. Samples #2–#5 exhibit comparable square- or rectangular-shaped grains with an average size of approximately $80 \mu\text{m}$. Thus, the heat treatment does not significantly modify the grain morphology of $\varnothing 100 \mu\text{m}$ samples. In contrast, the heat-treated $\varnothing 50 \mu\text{m}$ samples show melt pool boundaries that are faded or partially erased compared to the as-built state (Figures 9b and 10b), consistent with partial chemical homogenization and reduction in residual stresses. Samples #3 and #4 exhibit clearer grain boundary definition (Figure 10b). Although some regions appear more equiaxed, the original LPBF melt pool architecture remains partially visible, confirming that the 800°C treatment promotes microstructural recovery but does not fully eliminate the as-built LPBF microstructure.

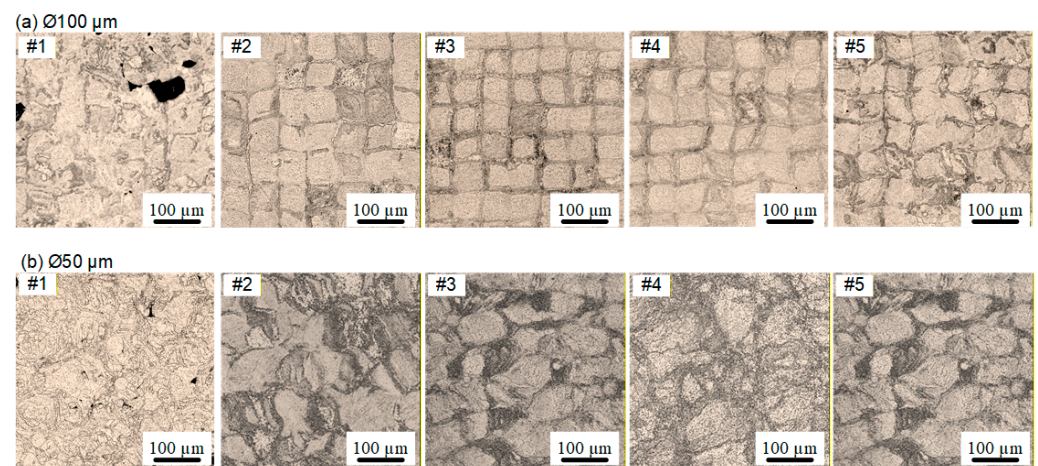


Figure 10. Microstructure of heat-treated samples produced with $\varnothing 100 \mu\text{m}$ (a) and $\varnothing 50 \mu\text{m}$ (b) spot sizes.

3.5. Phase Composition at Room Temperature and Temperature Dependence of B2 Lattice Parameter

Figure 11 shows the room-temperature X-ray diffraction (XRD) patterns of Ti-Ni samples produced using the two spot sizes. The results reveal a laser-spot-size-dependent response of the alloy to the applied VED. Even under nominally identical VED-BR conditions, $\varnothing 100 \mu\text{m}$ and $\varnothing 50 \mu\text{m}$ spot sizes produce different diffraction signatures, reflecting differences in thermal history, melt pool geometry, and solidification behavior. Across all samples, the ordered cubic B2 austenite phase dominates the diffraction patterns, with characteristic (110), (200) and (211) reflections. Only weak monoclinic B19' martensite peaks are detected, indicating that the martensite fraction remains small in all as-built samples.

However, the relative intensities, peak widths, and sharpness of B2 reflections vary with both energy density and spot size.

At low VED (28 J/mm^3 , sample #1), the $(110)_{\text{B2}}$ reflection is strongly enhanced relative to the pattern of the initial powder. This indicates the development of crystallographic texture during LPBF processing. The $\varnothing 50 \text{ }\mu\text{m}$ -sample peaks appear slightly broader, suggesting more heterogeneous microstructures and increased levels of microstrains.

At intermediate VED ($\sim 55\text{--}56 \text{ J/mm}^3$, samples #2 and #5), the large spot-size-dependent differences are observed. $\varnothing 100 \text{ }\mu\text{m}$ samples retain relatively sharp B2 peaks with lower broadening, while $\varnothing 50 \text{ }\mu\text{m}$ samples show significant broadening of the B2 reflections and reduced peak symmetry. This broadening suggests higher lattice strains, defect accumulations, or compositional heterogeneity in the $\varnothing 50 \text{ }\mu\text{m}$ builds. Both spot sizes exhibit a clear evolution toward a $\langle 100 \rangle$ texture, evidenced by an increasing intensity of the $(200)_{\text{B2}}$ peak relative to the $(110)_{\text{B2}}$ peak. As VED increases from samples #1 to #2, the $I_{\{110\}}/I_{\{200\}}$ ratio decreases, confirming the strengthening of the $\langle 100 \rangle$ texture along the build direction. Sample #5, produced at the same VED but with a lower BR, shows a lower $I_{\{110\}}/I_{\{200\}}$ ratio. This indicates a weaker $\langle 100 \rangle$ texture, likely due to a more concurrent grain growth mechanism under faster solidification conditions.

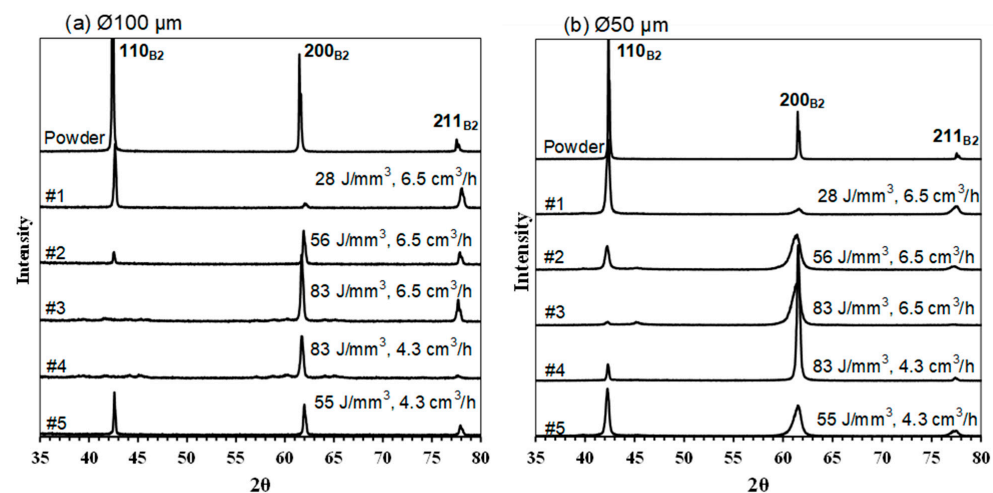


Figure 11. XRD for as-built samples produced with $\varnothing 100 \text{ }\mu\text{m}$ (a) and $\varnothing 50 \text{ }\mu\text{m}$ (b) laser spots.

At high VED ($\sim 83 \text{ J/mm}^3$, samples #3 and #4), the $(200)_{\text{B2}}$ peak becomes strongly dominant, reflecting the development of an even stronger $\langle 100 \rangle$ texture. Sample #4, built at the same VED as sample #3 but at a lower BR, exhibits a further increase in the $(200)_{\text{B2}}$ peak intensity, consistent with a longer laser-matter interaction time, slower cooling, and deeper melt pools that promote epitaxial grain growth along the $\langle 100 \rangle$ direction. Despite this, the $\varnothing 50 \text{ }\mu\text{m}$ samples consistently retain broader peaks than their $\varnothing 100 \text{ }\mu\text{m}$ counterparts. Overall, the $\varnothing 100 \text{ }\mu\text{m}$ laser spot shows a stronger texture sensitivity to VED, whereas the $\varnothing 50 \text{ }\mu\text{m}$ spot exhibits a greater level of microstrains and broadened diffraction response.

Figure 12 shows the room-temperature XRD patterns of the heat-treated Ti-Ni samples produced using both spot sizes. The $800 \text{ }^\circ\text{C}$ heat treatment is intended to drive the alloy from a highly non-equilibrium LPBF state toward more thermodynamically stable and chemically homogeneous conditions. In principle, such treatment should promote strain recovery, dissolve precipitates back into the B2 matrix, reduce local Ni depletion, and modify martensitic transformation kinetics. However, the XRD results show that the spot-size- and energy-density-dependent differences persist even after this heat treatment, indicating that full homogenization is not achieved under these conditions and/or that intrinsic compositional differences remain.

Across all samples, the heat treatment leads to a decrease in B2 peak intensity accompanied by an increase in B19' martensite reflections. The martensite reflections are highlighted in Figure 12b,d, which zoom in on the region around the $(110)_{B2}$ reflection ($37\text{--}47^\circ 2\theta$). This shift reflects changes in phase fraction at room temperature. Despite high-temperature (800°C) treatment, the XRD peaks remain relatively broad, demonstrating that the LPBF microstructure, characterized by high defect density, microstrains, and residual stresses, is only partially relieved (Figures 12 and 13). The persistence of peak broadening suggests that complete recrystallization has not occurred under the present heat-treatment conditions.

The $\varnothing 100\ \mu\text{m}$ samples processed at 56 and $83\ \text{J}/\text{mm}^3$ (#2 and #3) exhibit a strengthening of the $(110)_{B2}$ reflection after heat treatment, indicating partial recrystallization. As shown in Figure 13a, the $\varnothing 100\ \mu\text{m}$ heat-treated samples #3 and #4 ($83\ \text{J}/\text{mm}^3$) exhibit a reduction in peak broadening relative to their counterparts, while heat-treated sample #4 is expected to retain $\langle 100 \rangle$ texture (Figure 12a). The sharper B2 peaks indicate stress relaxation and reduced lattice strains. Notably, $\varnothing 100\ \mu\text{m}$ samples #2 and #5 (both $\sim 55\text{--}56\ \text{J}/\text{mm}^3$) converge to near identical diffraction signatures after heat treatment, despite their different as-built states, suggesting that the 800°C treatment partially equalizes microstructural differences resulting from the application of different VED conditions. The improved peak symmetry further indicates reduced compositional variations.

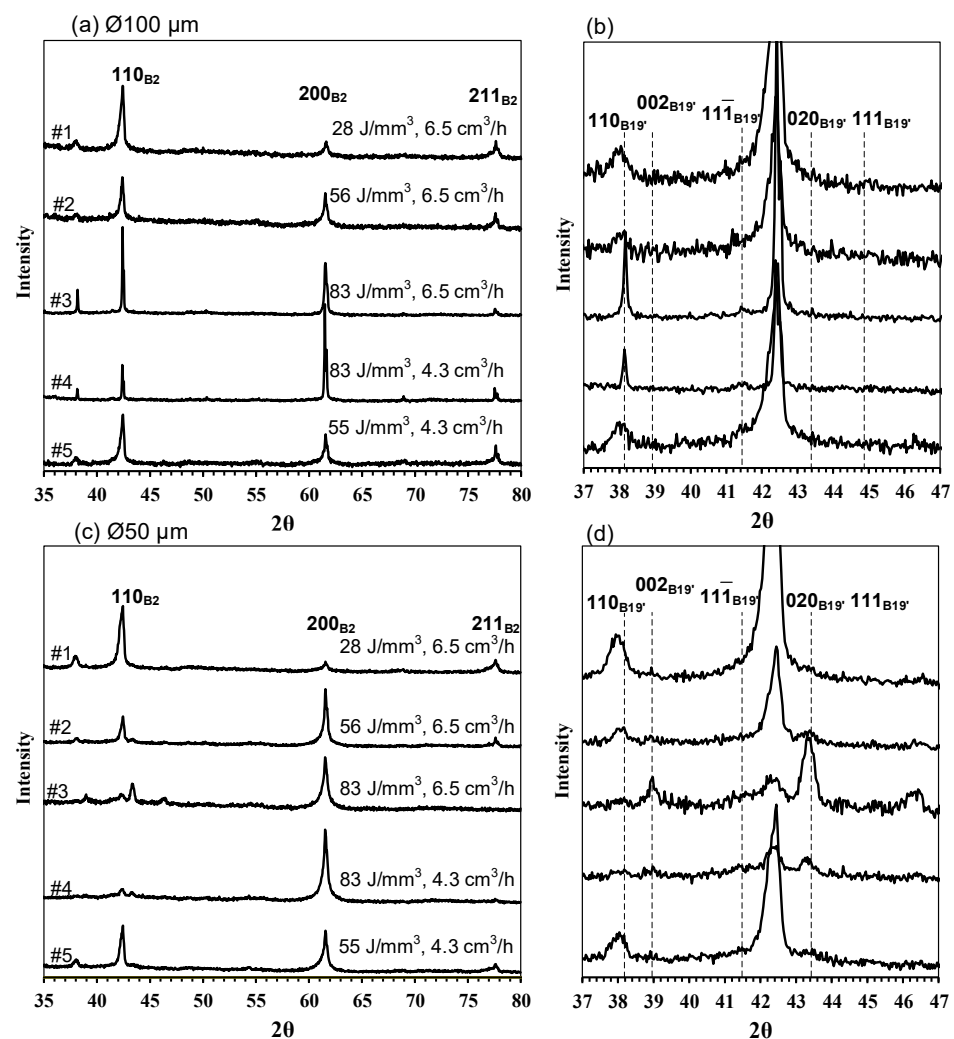


Figure 12. XRDs for heat-treated samples produced with $\varnothing 100\ \mu\text{m}$ (a,b) and $\varnothing 50\ \mu\text{m}$ (c,d) laser spots with a zoom near the $(110)_{B2}$ reflection (b,d).

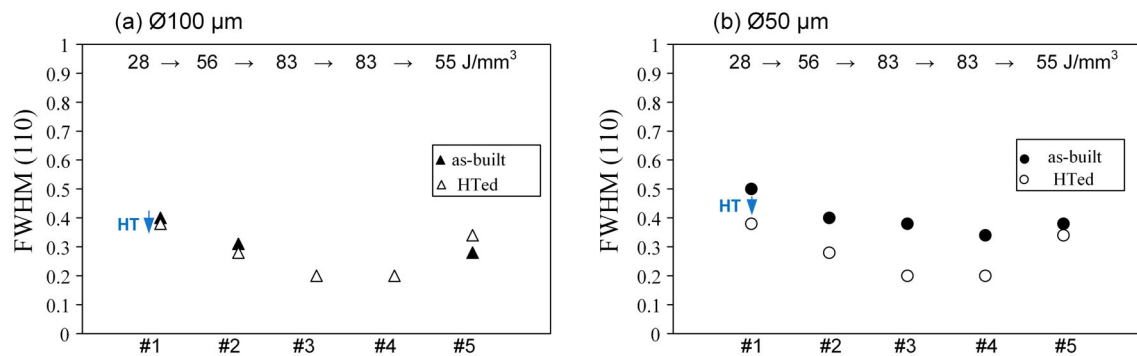


Figure 13. Full width at half maximum (FWHM) of the $(110)_{B2}$ reflection for as-built and heat-treated samples: (a) $\varnothing 100\ \mu\text{m}$ and (b) $\varnothing 50\ \mu\text{m}$.

In contrast, $\varnothing 50\ \mu\text{m}$ samples (#2–#5) retain pronounced texture, broad B2 peaks, and clear signs of microstructural heterogeneity even after heat treatment, thus revealing higher residual stresses, greater microstrain, or more stable defects than their $\varnothing 100\ \mu\text{m}$ counterparts (Figures 12c and 13a).

The B2 lattice parameter of heat-treated samples at room temperature is plotted in Figure 14. Using multiple reflections improves the precision of the extrapolated lattice parameter. For the as-built samples, however, strong crystallographic texture limited the number of usable B2 peaks; therefore, lattice parameters from the as-built condition were not included in the quantitative comparison.

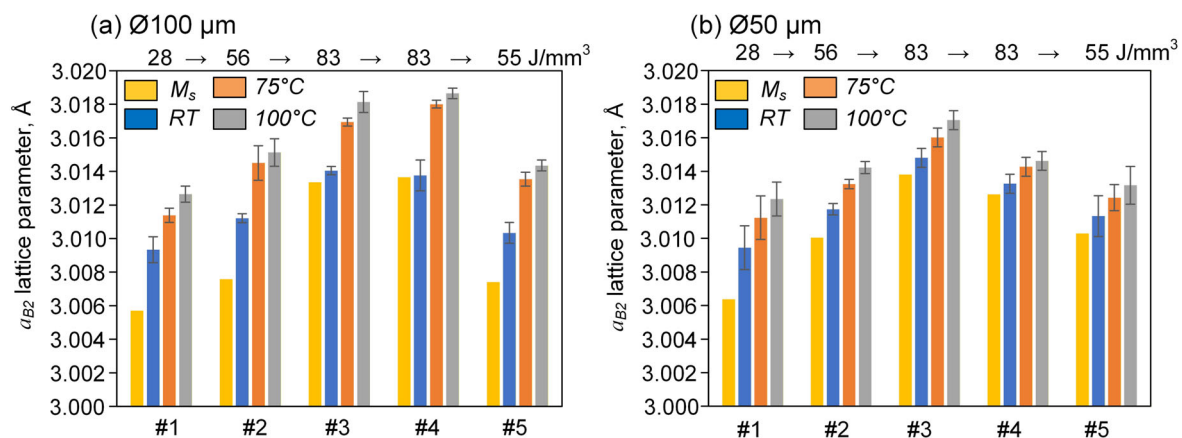


Figure 14. B2 lattice parameter at different temperatures for samples produced using $\varnothing 100\ \mu\text{m}$ (a) and $\varnothing 50\ \mu\text{m}$ (b) laser spots.

The lattice parameters of the heat-treated samples show a clear dependency on both the energy density and spot size. Sample #1, processed at $28\ \text{J/mm}^3$, exhibits the smallest a_{B2} value for both spot sizes. As VED increases from 28 to $83\ \text{J/mm}^3$, the B2 lattice parameter increases from 3.0093 to $3.0140\ \text{\AA}$ for $\varnothing 100\ \mu\text{m}$ samples and from 3.0095 to $3.0148\ \text{\AA}$ for $\varnothing 50\ \mu\text{m}$ samples. Samples produced at identical VED conditions (either ~ 55 or $83\ \text{J/mm}^3$) show very similar B2 lattice parameter values within each system. For example, it corresponds to $3.0140\ \text{\AA}$ and $3.0138\ \text{\AA}$ for $\varnothing 100\ \mu\text{m}$ samples #3 and #4, and $3.0148\ \text{\AA}$ and $3.0133\ \text{\AA}$ for $\varnothing 50\ \mu\text{m}$ samples #3 and #4. The $\varnothing 100\ \mu\text{m}$ samples, however, exhibit closer values, indicating a more uniform response to energy density.

Because differences between the martensite start temperature (M_s) and room temperature are not identical for all samples, the B2 lattice parameter measured at room temperature does not correspond to the same thermodynamic states across the dataset. Samples with M_s far below room temperature contain austenite during XRD, whereas those with M_s

closer to room temperature retain residual martensite or transformation-induced strain. To approximate the lattice parameter at M_s for each sample, additional XRD measurements were performed at 75 °C and 100 °C. It should be noted that even at 100 °C, approximately 5% of martensite was retained. This means that the measured B2 lattice parameter at these temperatures still reflects small contributions from transformation-induced strains. However, because the retained martensite fraction is small and decreases with temperature, the B2 lattice parameter extracted at 75 °C and 100 °C still provides reliable points along the thermal-expansion curve of the B2 phase.

The temperature-dependent lattice parameters for both laser spots (Figure 14) show an approximately linear increase with temperature. The average thermal-expansion coefficients were $1.2 \times 10^{-5} \text{ K}^{-1}$ for Ø100 µm samples and $1.0 \times 10^{-5} \text{ K}^{-1}$ for Ø50 µm samples, which is consistent with normal thermal expansion of the B2 phase. For each sample, the lattice parameters at RT, 75 °C, and 100 °C were fitted with a linear least-square regression and extrapolated to the corresponding M_s temperatures, obtained from the DSC measurements described in the previous section. This approach compensates simultaneously for the different amounts of residual martensite at RT, different thermal-expansion coefficients, and different degrees of transformation-induced strain. Thus, extrapolating to a_{B2} at M_s provides the most accurate and comparable estimation of the intrinsic B2 lattice parameter for each sample.

The extrapolated lattice parameters at M_s follow the same overall trends observed at room temperature: samples produced at similar VED values (55–56 and 83 J/mm³) exhibit nearly identical lattice parameters, particularly for the Ø100 µm laser spot. This confirms that the VED- and laser spot-dependent differences in the lattice parameter values originate from compositional variations caused by LPBF processing.

3.6. Hardness

Table 3 summarizes the hardness test results obtained for samples processed using the Ø100 µm and Ø50 µm spot sizes. It should be noted that the reported values correspond to so-called “apparent” hardness. In shape memory alloys, indentation can activate stress-induced martensite transformation in addition to conventional plastic deformation mechanisms; therefore, the measured hardness values may be different from conventional hardness obtained in other materials. As-built sample #1 exhibits relatively high hardness despite its low relative density (98.81% for Ø100 µm and 96.90% for Ø50 µm samples), which is lower than that of samples #2–#5 processed at 55–83 J/mm³, where the average relative density exceeds 99.50%. Among as-built samples #2 to #5, sample #5 (55 J/mm³) shows the highest hardness for both spot sizes.

Table 3. Vickers hardness test results.

Samples	E (J/mm ³)	Ø100 µm Laser Spot		Ø50 µm Laser Spot	
		As-Built	Heat-Treated	As-Built	Heat-Treated
#1	28	309 ± 6	349 ± 15	303 ± 11	333 ± 13
#2	56	252 ± 9	295 ± 8	275 ± 7	320 ± 14
#3	83	226 ± 6	268 ± 10	254 ± 12	288 ± 16
#4	83	251 ± 9	257 ± 11	298 ± 8	311 ± 6
#5	55	292 ± 9	323 ± 9	302 ± 12	297 ± 10

A meaningful correlation appears to exist between hardness and transformation temperatures. Specifically, lower transformation temperature (M_s) corresponds to higher hardness (Figure 15 and Table 3). When M_s is well below room temperature, B2 austenite is more stable, and higher critical stresses are required to induce B2 ↔ B19' transforma-

tion [37,38]. Consequently, samples #1 and #5, which exhibit the lowest M_s values, show the highest hardness, whereas samples #3 and #4 with the highest M_s exhibit the lowest hardness. A similar trend is observed after heat treatment: M_s decreases and hardness increases compared to the as-built condition. After heat treatment, sample #1 again shows the lowest M_s and the highest hardness, while samples #3 and #4 show the lowest hardness, with M_s values that are higher and close to room temperature. When M_s is near the test temperature, the B2 and B19' coexist, and hardness reflects contributions from both phases, where the presence of martensite additionally reduces the hardness value [39]. Overall, the evolution of hardness aligns with the corresponding changes in M_s , demonstrating that LPBF processing parameters significantly influence the transformation behavior and mechanical performance of the Ti-Ni alloy.

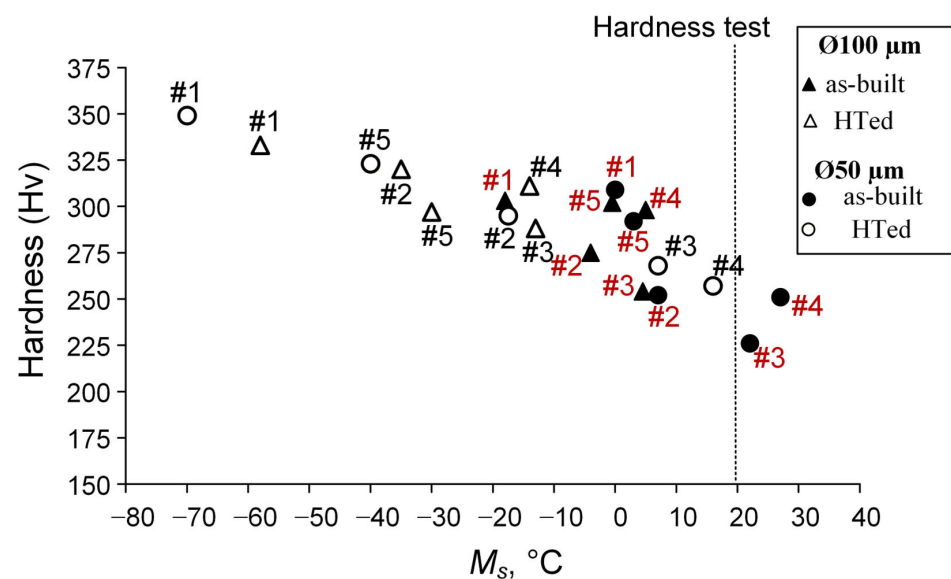


Figure 15. Hardness evolution as function of M_s for samples produced with different spot sizes.

4. Discussion

4.1. Spot-Size-Dependent Thermal Regimes

Although both LPBF systems were operated at nominally identical volumetric energy densities (VED), their distinct laser spot diameters (EOS: Ø100 µm; TruPrint: Ø50 µm) resulted in distinct thermal conditions. In contrast to the VED-BR coupled representation, the aggregated (spot size and laser power) metrics such as energy input (E_i) and specific point energy (SPE) indicated in Table 1 did not reveal clear or systematic trends. This is because E_i and SPE represent only partial aspects of the energy–material interaction and do not fully capture the coupled effect of heat distribution, melt pool dynamics, and material deposition conditions. As a result, neither parameter scaled consistently with melt pool width, depth, or stability.

Previous studies have shown that smaller spots increase thermal gradients and melt pool instability, and create a greater risk of keyhole porosity, whereas larger spots provide a more uniform energy distribution and promote greater melt pool continuity [40–43]. Furthermore, melt pool depth is reported to be more sensitive to spot size than melt pool width, especially near the transition from conduction to keyhole melting mode [44].

The present results confirm and extend these findings for Ti-Ni alloys. The melt pool track widths were comparable for both spot sizes, which is consistent with the matched VED values. However, the penetration depth was strongly affected by spot size, with Ø50 µm spot producing shallower melt pools. At low VED, the Ø50 µm condition also

resulted in partial melt pool instability, whereas the Ø100 µm condition produced more stable melt pools.

Laser spot size also influences cooling conditions during LPBF and therefore affects grain size, microstrains, and residual stresses. Prior studies on IN625, 316L, Ti-6Al-4V and AlSi10Mg [44–47] have shown that smaller laser spots produce smaller melt pool zones, thus generating higher cooling rates and finer microstructures. Consistent with these findings, the Ø50 µm samples in the present work consistently exhibited a finer microstructure. Higher cooling rates are also expected to create higher thermal gradients, and, therefore, produce higher residual stresses after solidification. In the present work, this effect is inferred from the broader B2 peaks observed for the Ø50 µm samples than for their Ø100 µm counterparts. Overall, the results demonstrate that laser spot size modifies the thermal history during LPBF and consequently influences the microstructure and phase transformation behavior of Ti-Ni alloys.

4.2. Transformation Temperatures in the As-Built and Heat-Treated Samples

In the as-built condition, both laser spots exhibit a clear increase in M_s , M_f , A_s and A_f temperatures with increasing energy density, with Ø50 µm samples consistently showing lower transformation temperatures than their Ø100 µm counterparts. This difference can be attributed to variations in effective Ni content, microstrain levels, grain size, and chemical and structural homogeneity. For example, higher residual stresses and increased microstructural heterogeneity in LPBF Ti-Ni are known to introduce local variations in transformation behavior, thereby broadening the DSC peaks and decreasing their intensity.

Heat treatment at 800 °C partially reduces crystallographic texture, relieves residual stresses, and homogenizes local chemical composition. At this temperature, Ni_4Ti_3 precipitates are expected to dissolve, so the treatment acts primarily as solution annealing, with partial recrystallization possible. Under these conditions, the martensitic transformation is expected to proceed directly (no R-phase), and the measured M_f , M_s , A_s and A_f temperatures are expected to more closely reflect the bulk Ni composition.

For all the samples subjected to this heat treatment, transformation peaks sharpen, indicating reduced microstructure heterogeneity, stress relief, and improved chemical uniformity. Moreover, after annealing, all transformation temperatures decrease significantly. Although their absolute values cannot be directly compared with those reported in the literature because of differences in alloy composition, powder chemistry, and processing parameters, the observed trends are consistent with previous data [48]. The various transformation temperatures (A_s , A_f , M_s and M_f) do not respond equally to processing and heat treatment: M_s remains the most sensitive to changes in composition and microstructural conditions, whereas A_s shows less sensitivity, in agreement with observations for conventionally manufactured Ti-Ni alloys [49].

4.3. Estimated Ni Contents in the Heat-Treated Samples

It is well established that the martensite start temperature (M_s) of binary Ti-Ni alloys decreases with increasing Ni content. Therefore, after heat treatment, when residual stresses are reduced and the microstructure is homogenized, the measured M_s values can be used as indirect indicators of the effective Ni composition of Ti-Ni alloys. This approach is based on the experimentally obtained M_s -Ni correlations [27–33].

Figure 16a,b compares the measured M_s values with these reference relationships. For the Ø100 µm samples, the estimated Ni content ranges from approximately 50.42 to 51.15 at.% Ni, depending on the processing parameters (Figures 16a and 17a). For the Ø50 µm samples produced over the identical process parameter window, the estimated Ni

content lies between 50.62 and 51.05 at.%Ni (Figures 16b and 17a). These values suggest that, at lower energy densities, the smaller spot samples exhibit greater Ni depletion, consistent with higher local power density and more unstable melt pool dynamics that promote Ni evaporation. In contrast, at higher energy densities, the Ø100 µm spot shows greater Ni depletion, likely due to larger and deeper melt pools and longer exposure times at elevated temperatures. For samples produced under identical nominal processing parameters, the difference in estimated Ni content corresponds to 0.1–0.2 at.% Ni.

Although the M_s -based estimation provides a reliable first approximation of the bulk Ni content, it remains sensitive to microstructure features such as grain size. The 800 °C (30 min) heat treatment performed in the present work did not allow full recrystallization, which generally requires higher temperatures (up to 1000 °C) and longer durations (up to 24 h under vacuum) [49]. Therefore, an additional independent indicator of composition was used in this study, namely the B2 lattice parameter.

The B2 lattice parameter is directly related to the cubic B2 austenitic structure and is therefore primarily governed by the average composition of the Ti-Ni matrix. This relationship is valid for quasi-recrystallized Ti-Ni alloys without Ni_4Ti_3 precipitates, which correspond to the conditions of the heat-treated alloy investigated in this study. Under these conditions, an increase in Ni content leads to a decrease in the B2 lattice parameter [49]. However, room temperature measurements of the B2 lattice parameters can be affected by two factors: (1) the coexistence of B2 and B19' phases, and (2) the temperature difference between M_s and room temperature. To minimize these effects, the lattice parameters were linearly extrapolated to $T = M_s$ using measurements acquired at 20, 75 and 100 °C.

It may be observed that extrapolated a_{B2} values increase with increasing energy density and decreasing build rate, which is consistent with enhanced Ni evaporation under higher thermal input conditions. At the ~28–55 J/mm³ range, the lattice-based method indicates greater Ni depletion with the smaller beam size, with differences of 0.05–0.4 at.%Ni (Figures 16c,d and 17b). At 83 J/mm³, the difference in Ni depletion estimation between the Ø100 µm and Ø50 µm samples becomes smaller, ranging from 0.05 to 0.14 at.%Ni depending on the build rate, with lower build rates amplifying this difference. These observations are consistent with the numerical simulations performed in [21], which showed that increasing the beam diameter from 15 to 25 Å reduced Ni vaporization in the lower energy density regime. In contrast, under higher energy input conditions, the larger spot size produces a larger melt pool volume, resulting in a comparatively smaller influence on the final Ni content.

At the lowest energy density (28 J/mm³), the lattice-based estimation suggests a Ni content higher than that of the initial powder. Two factors may explain this: (1) oxygen-induced Ti depletion and (2) uncertainty in the lattice-based estimation. At low energy density, lack-of-fusion defects and Ti-rich oxides can form more readily, depleting Ti from the matrix and producing a locally Ni-rich B2 phase. This reduces the B2 lattice parameter and artificially depresses M_s [49,50]. At higher energy density, Ti-rich oxides present in raw powder can be reduced due to the extremely high melting temperatures, and both estimation methods converge. Regarding the second factor, only a limited number of studies provide reliable B2 lattice-Ni data, particularly for Ni-rich Ti-Ni alloys. Small systematic differences between datasets may therefore introduce minor variations in the estimated composition. However, despite these uncertainties, both independent approaches consistently indicate the same overall trend in Ni loss as a function of different energy density, build rate, and spot size.

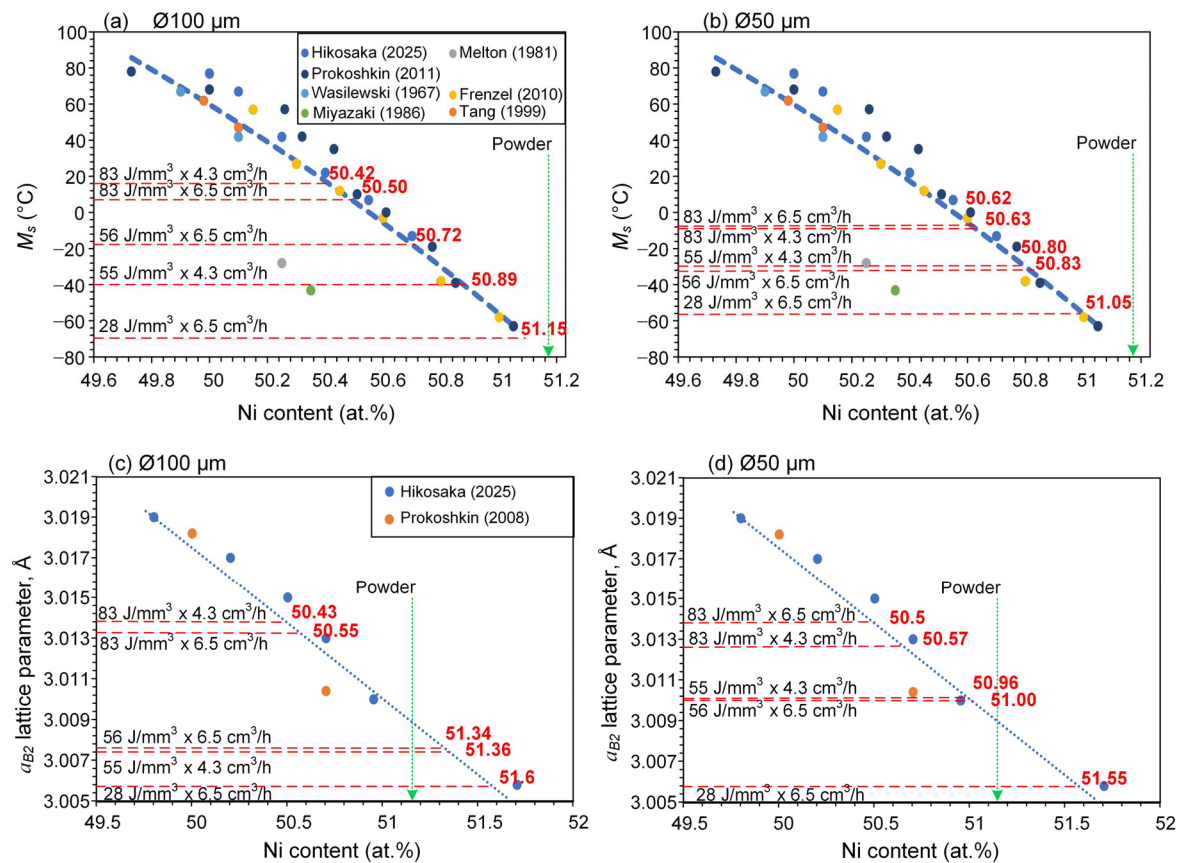


Figure 16. M_s of each sample and Ni content determined using the data reported in the previous studies [27–33,51]: (a,c) Ø100 µm and (b,d) Ø50 µm samples.

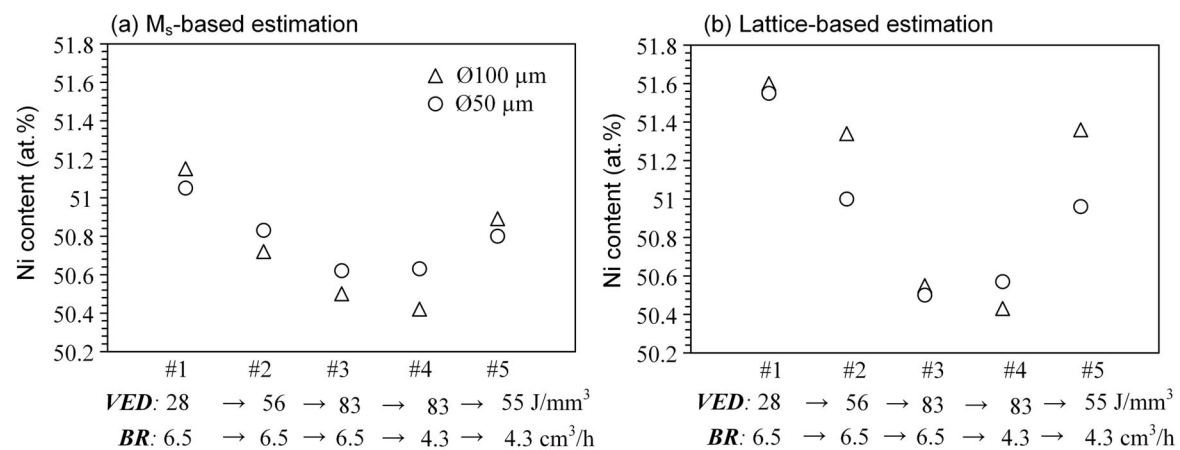


Figure 17. Estimated Ni content in samples produced with different spot sizes using M_s -based (a) and lattice-based (b) estimations.

4.4. Correlations Between Process

Conditions–Density–Composition–Microstructure–Microhardness, and the Laser Spot Size

The transformation temperatures, hardness, and estimated Ni content all exhibit a non-monotonic dependence on energy density and build rate, while the trends differ between the two systems. The samples produced at low VED (28 J/mm³), which contain a larger number of pores and therefore exhibit a lower relative density (96.9% and 98.81%), show the highest hardness values (303 ± 11 and 309 ± 6). As the VED increases to 83 J/mm³, the density rises to 99.98–99.99%, while the hardness decreases to 226 ± 6 or 254 ± 12. This inverse relationship between porosity and hardness is consistent with previous LPBF

Ti-Ni studies and reflects the combined influence of microstructure and Ni content on the transformation temperatures, which, in turn, govern the functional behavior at room temperature [52]. Nevertheless, for the high-density samples produced at 55–83 J/mm³, the Ø50 µm specimens consistently exhibit higher hardness than the Ø100 µm samples. This difference is likely associated with the finer microstructure obtained with the smaller spot size.

Higher energy density generally increases the transformation temperatures, indicating that the superelastic response will occur above room temperature. In contrast, samples processed at low energy density are expected to retain a composition closer to that of the initial powder; for Ti-51.17 at.% Ni, this favors superelasticity at room temperature. Nevertheless, the spot size introduces systematic differences: the average M_s , M_f , A_s , and A_f values differ by approximately 17–25 °C between the Ø100 µm and Ø50 µm conditions, corresponding to estimated compositional variations of roughly 0.1–0.2 at.% Ni.

These observations indicate that Ni evaporation is one of the key effects occurring in Ti-Ni alloys associated with changes in laser spot size during LPBF processing, in addition to the resulting microstructural differences. The parameter sets developed for one LPBF system cannot be directly transferred to another system with a different spot size without comprehensive process and material requalification. Industrial LPBF of Ti-Ni should rely on function-driven process qualification, where VED, BR and spot size jointly define composition, microstructure, and residual stresses, all of which govern the mechanical and functional performance of the alloy. Although heat treatment can homogenize the microstructure and reduce residual stresses, the Ni loss introduced during processing remains and continues to influence the transformation behavior.

5. Conclusions

This work systematically compares Ti-Ni samples fabricated using two LPBF systems with different laser spot sizes under matched volumetric energy densities and build rates. The study evaluates how processing conditions influence microstructural evolution, phase constitution, transformation behavior, and hardness. The main conclusions drawn from this investigation are summarized below.

1. Volumetric energy density (VED) is the dominant processing parameter controlling microstructure and functional behavior of LPBF Ti-Ni alloys. Increasing volumetric energy density enlarges melt pool dimensions, enhances remelting, promotes microstructure coarsening, and drives the formation of a $\langle 100 \rangle$ B2 texture parallel to the build direction at a high energy density (83 J/mm³). These changes directly influence phase stability and transformation temperatures.
2. Laser spot size (in Ø50–Ø100 µm range) modifies melt pool geometry, microstructure, and Ni evaporation. The Ø50 µm spot produces melt pools of comparable width but reduced depth, which slightly decreases final material density. It also generates finer microstructures and higher microstrains. Importantly, Ni losses depend on a combined effect of spot size and energy density. At low VED, the smaller spot promotes greater Ni losses. However, high VED produces deeper melt pools and longer thermal exposure; therefore, the overall amount of Ni evaporated at high VED is still greater than at low VED, regardless of spot size.
3. Transformation temperatures and hardness exhibit systematic, non-monotonic dependences on both VED and spot size. In general, increasing VED and using a larger spot size increase M_s , while reducing room temperature hardness.
4. Heat treatment at 800 °C partially homogenizes the alloy and reveals the true composition-driven behavior. This treatment relieves residual stresses, sharpens DSC peaks, lowers M_s for all samples, and increases hardness at room temperature.

After heat treatment, transformation temperatures more accurately reflect the bulk Ni content rather than the microstructure heterogeneity induced during LPBF.

5.
- The combined M_s -based and lattice-parameter-based Ni estimations demonstrate that laser spot size is a critical, yet often overlooked, factor in LPBF of Ti-Ni alloys. Small variations in Ni loss (0.1–0.2 at.% Ni) associated with changing the spot diameter from Ø50 to Ø100 µm produce substantial shifts in transformation temperatures. On average, these shifts approach ~20 °C, thus highlighting the sensitivity of Ti-Ni alloys to minor compositional changes and indicating significant changes in their thermomechanical behavior.

Author Contributions: Conceptualization, A.K., D.C. and V.B.; methodology, E.B. and D.C.; validation, A.K. and D.C.; formal analysis, A.K. and D.C.; investigation, D.C.; data curation, D.C. and E.B.; writing—original draft preparation, A.K.; writing—review and editing, V.B.; visualization, A.K. and D.C.; supervision, A.K. and V.B.; project administration, A.K.; funding acquisition, A.K. and V.B. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Natural Sciences and Engineering Research Council of Canada (NSERC) under Discovery Grants RGPIN-2025-04142 (A. Kreitchberg) and GPIN-2020-05800 (V. Brailovski).

Data Availability Statement: The datasets generated during the current study are available from the corresponding author upon reasonable request.

Acknowledgments: The authors acknowledge the financial support provided by NSERC (Natural Sciences and Engineering Research Council of Canada) through the Discovery Grant program. During the preparation of this article, the authors used ChatGPT (GPT-4.0) for language refinement. All generated content was reviewed, edited, and validated by the authors, who take full responsibility for the final version of this publication.

Conflicts of Interest: The authors declare no conflicts of interest.

Appendix A

Table A1. Characteristic transformation temperatures (M_p , A_p , M_s , M_f , A_s , A_f) for the as-built and heat-treated samples.

Ø100 µm Laser Spot							Ø50 µm Laser Spot					
Transformation Temperatures, °C												
Samples	M _p	A _p	M _s	M _f	A _s	A _f	M _p	A _p	M _s	M _f	A _s	A _f
As-built state												
#1	−36	−34	0	−63	−55	6	−68	−33	−18	<−85	−55	20
#2	−28	0	7	−62	−31	27	−43	−17	−4	−85	−44	3
#3	6	27	22	−31	−11	46	−26	0	5	−56	−29	32
#4	14	32	27	−16	8	53	−26	−1	5	−65	−45	24
#5	−52	−17	3	−82	−44	9	−43	−13	−1	−85	−48	8
Heat-treated state												
#1	−78	−48	−71	n/a	−57	−33	−75	−44	−55	n/a	−60	−25
#2	−34	−13	−18	−56	−31	5	−51	−25	−35	−80	−48	−9
#3	−9	16	7	−37	−15	28	−34	−9	−13	−59	−34	7
#4	−3	21	16	−19	5	41	−34	−13	−14	−63	−50	8
#5	−53	−24	−40	−67	−49	−12	−43	−23	−30	−67	−51	−8

References

- Otsuka, K.; Ren, X. Physical metallurgy of Ti–Ni-based shape memory alloys. *Prog. Mater. Sci.* **2005**, *50*, 511–678. [\[CrossRef\]](#)
- Yoneyama, T.; Miyazaki, S. *Shape Memory Alloys for Biomedical Applications*; CRC Press: Boca Raton, FL, USA, 2008.
- Miyazaki, S.; Otsuka, K.; Suzuki, Y. Transformation pseudoelasticity and deformation behavior in a Ti–50.6at%Ni alloy. *Scr. Metall.* **1981**, *15*, 287–292. [\[CrossRef\]](#)
- Tang, W. Thermodynamic study of the low-temperature phase B19' and the martensitic transformation in near-equiatomic Ti–Ni shape memory alloys. *Metall. Mater. Trans. A* **1997**, *28*, 537–544. [\[CrossRef\]](#)
- Lin, H.C.; Wu, S.K.; Chou, T.S.; Kao, H.P. The effects of cold rolling on the martensitic transformation of an equiatomic TiNi alloy. *Acta Metall. Mater.* **1991**, *39*, 2069–2080. [\[CrossRef\]](#)
- Bian, C.; Wen, L.; Wu, X.; Cheng, C.; Wan, Z.; Li, P.; Liu, Y.; Shi, X. Effect of cold-drawn deformation and annealing temperature on reverse transformation of pre-deformed nanocrystalline Ni₅₀Ti₅₀ alloys. *Mater. Charact.* **2026**, *232*, 115999. [\[CrossRef\]](#)
- Miyazaki, S.; Ohmi, Y.; Otsuka, K.; Suzuki, Y. Characteristics of deformation and transformation pseudoelasticity in NiTi alloys. *J. Phys. Colloq.* **1982**, *43*, C4-255–C4-260. [\[CrossRef\]](#)
- Waitz, T.; Kazykhanov, V.; Karnthaler, H.P. Martensitic phase transformations in nanocrystalline NiTi studied by TEM. *Acta Mater.* **2004**, *52*, 137–147. [\[CrossRef\]](#)
- Wang, X.; Yu, J.; Liu, J.; Chen, L.; Yang, Q.; Wei, H.; Sun, J.; Wang, Z.; Zhang, Z.; Zhao, G. Effect of process parameters on the phase transformation behavior and tensile properties of NiTi shape memory alloys fabricated by selective laser melting. *Addit. Manuf.* **2020**, *36*, 101545. [\[CrossRef\]](#)
- Jamshidi, P.; Panwisawas, C.; Langi, E.; Cox, S.C.; Feng, J.; Zhao, L.; Attallah, M.M. Development, characterisation, and modelling of processability of nitinol stents using laser powder bed fusion. *J. Alloys Compd.* **2022**, *909*, 164681. [\[CrossRef\]](#)
- Xue, L.; Atli, K.; Zhang, C.; Hite, N.; Srivastava, A.; Leff, A.; Wilson, A.; Sharar, D.; Elwany, A.; Arroyave, R. Laser powder bed fusion of defect-free NiTi shape memory alloy parts with superior tensile superelasticity. *Acta Mater.* **2022**, *229*, 117781. [\[CrossRef\]](#)
- Zhan, J.; Wu, J.; Ma, R.; Li, K.; Huang, T.; Lin, J.; Murr, L.E. Effect of microstructure on the superelasticity of high-relative-density Ni-rich NiTi alloys fabricated by laser powder bed fusion. *J. Mater. Process. Technol.* **2023**, *317*, 117988. [\[CrossRef\]](#)
- Speirs, M.; Wang, X.; Van Baelen, S.; Ahadi, A.; Dadbakhsh, S.; Kruth, J.P.; Van Humbeeck, J. On the Transformation Behavior of NiTi Shape-Memory Alloy Produced by SLM. *Shape Mem. Superelast.* **2016**, *2*, 310–316. [\[CrossRef\]](#)
- Li, M.; Contreras-Almengor, O.; Ordoño, J.; Aguilar Vega, C.; Zapata Martínez, R.; Echeverry-Rendón, M.; Díaz-Lantada, A.; Molina-Aldareguia, J. The microstructure and mechanical properties of nitinol manufactured by LPBF: Differences between Ni-rich and Ti-rich compositions. *Virtual Phys. Prototyp.* **2025**, *20*, e2476006. [\[CrossRef\]](#)
- Nematollahi, M.; Safaei, K.; Bayati, P.; Elahinia, M. Functionally graded NiTi shape memory alloy: Selective laser melting fabrication and multi-scale characterization. *Mater. Lett.* **2021**, *292*, 129648. [\[CrossRef\]](#)
- Yang, Y.; Zhan, J.B.; Sui, J.B.; Li, C.Q.; Yang, K.; Castany, P.; Gloriant, T. Functionally graded NiTi alloy with exceptional strain-hardening effect fabricated by SLM method. *Scr. Mater.* **2020**, *188*, 130–134. [\[CrossRef\]](#)
- Saedi, S.; Turabi, A.S.; Taheri Andani, M.; Haberland, C.; Karaca, H.; Elahinia, M. The influence of heat treatment on the thermomechanical response of Ni-rich NiTi alloys manufactured by selective laser melting. *J. Alloys Compd.* **2016**, *677*, 204–210. [\[CrossRef\]](#)
- Saedi, S.; Moghaddam, N.S.; Amerinatanzi, A.; Elahinia, M.; Karaca, H.E. On the effects of selective laser melting process parameters on microstructure and thermomechanical response of Ni-rich NiTi. *Acta Mater.* **2018**, *144*, 552–560. [\[CrossRef\]](#)
- Sow, M.C.; De Terris, T.; Castelnau, O.; Hamouche, Z.; Coste, F.; Fabbro, R.; Peyre, P. Influence of beam diameter on Laser Powder Bed Fusion (L-PBF) process. *Addit. Manuf.* **2020**, *36*, 101532. [\[CrossRef\]](#)
- Campion, D.; Kreitzberg, A.; Brailovski, V. Simulation-based training of multi-material machine learning models for the prediction of melt pool dimensions and as-printed densities in laser powder bed fusion parts. *Int. J. Adv. Manuf. Technol.* **2026**, *143*, 4023–4044. [\[CrossRef\]](#)
- Li, G.; Yu, T.; Wu, P.; Chen, M. Molecular Dynamics Simulation of NiTi Shape Memory Alloys Produced by Laser Powder Bed Fusion: Laser Parameters on Phase Transformation Behavior. *Materials* **2023**, *16*, 409. [\[CrossRef\]](#) [\[PubMed\]](#)
- Suder, W.J.; Williams, S.W. Investigation of the effects of basic laser material interaction parameters in laser welding. *J. Laser Appl.* **2012**, *24*, 032009. [\[CrossRef\]](#)
- Xi, C.; Hai-Xing, W. A calculation model for the evaporation recoil pressure in laser material processing. *J. Phys. D. Appl. Phys.* **2001**, *34*, 2637. [\[CrossRef\]](#)
- Zhang, H.; Wu, B.; Yi, J.; Rao, Z.; Wang, P.; Wang, S. Elucidating the mechanism for high-temperature heat treatment induced embrittlement of laser-powder-based fusion manufactured NiTi alloy. *J. Mater. Sci. Technol.* **2025**, *216*, 52–65. [\[CrossRef\]](#)
- ASTM F2004; Standard Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis. ASTM International: West Conshohocken, PA, USA, 2017.

26. Nelson, J.B.; Riley, D.P. An experimental investigation of extrapolation methods in the derivation of accurate unit-cell dimensions of crystals. *Proc. Phys. Soc.* **1945**, *57*, 160. [\[CrossRef\]](#)
27. Hikosaka, G.; Shinohara, Y.; Matsumura, R.; Nishida, M.; Inamura, T. Lattice Parameters of B2 and B19' Phases in Ti–Ni Alloys Near Transformation Temperatures: Effects of Ni Concentration and Temperature. *Shape Mem. Superelast.* **2025**, *11*, 579–592. [\[CrossRef\]](#)
28. Prokoshkin, S.D.; Korotitskiy, A.V.; Brailovski, V.; Inaekyan, K.E.; Dubinskiy, S.M. Crystal lattice of martensite and the reserve of recoverable strain of thermally and thermomechanically treated Ti–Ni shape-memory alloys. *Phys. Met. Metallogr.* **2011**, *112*, 170–187. [\[CrossRef\]](#)
29. Frenzel, J.; George, E.P.; Dlouhy, A.; Somsen, C.; Wagner, M.F.X.; Eggeler, G. Influence of Ni on martensitic phase transformations in NiTi shape memory alloys. *Acta Mater.* **2010**, *58*, 3444–3458. [\[CrossRef\]](#)
30. Wasilewski, R.J.; Butler, S.R.; Hanlon, J.E. On the Martensitic Transformation in TiNi. *Met. Sci. J.* **1967**, *1*, 104–110. [\[CrossRef\]](#)
31. Tang, W.; Sundman, B.; Sandström, R.; Qiu, C. New modelling of the B2 phase and its associated martensitic transformation in the Ti–Ni system. *Acta Mater.* **1999**, *47*, 3457–3468. [\[CrossRef\]](#)
32. Melton, K.N.; Mercier, O. The mechanical properties of NiTi-based shape memory alloys. *Acta Metall.* **1981**, *29*, 393–398. [\[CrossRef\]](#)
33. Miyazaki, S.; Otsuka, K. Deformation and transition behavior associated with the R-phase in Ti–Ni alloys. *Metall. Trans. A* **1986**, *17*, 53–63. [\[CrossRef\]](#)
34. Miyazaki, S.; Igo, Y.; Otsuka, K. Effect of thermal cycling on the transformation temperatures of Ti–Ni alloys. *Acta Metall.* **1986**, *34*, 2045–2051. [\[CrossRef\]](#)
35. Bandyopadhyay, A.; Krishna, B.V.; Xue, W.; Bose, S. Application of laser engineered net shaping (LENS) to manufacture porous and functionally graded structures for load bearing implants. *J. Mater. Sci. Mater. Med.* **2009**, *20*, S29–S34. [\[CrossRef\]](#) [\[PubMed\]](#)
36. Balla, V.; Bose, S.; Bandyopadhyay, A. Laser Processing of Net-Shape NiTi Shape Memory Alloy. *Metall. Mater. Trans. A* **2007**, *38*, 1096–1103. [\[CrossRef\]](#)
37. Farhat, Z.N.; Zhang, C.A.N. The Role of Reversible Martensitic Transformation in the Wear Process of TiNi Shape Memory Alloy. *Tribol. Trans.* **2010**, *53*, 917–926. [\[CrossRef\]](#)
38. Generali, L.; Malovo, A.; Bolelli, G.; Borghi, A.; La Rosa, G.R.M.; Puddu, P.; Lusvardi, L.; Rota, A.; Consolo, U.; Pedullà, E. Mechanical Properties and Metallurgical Features of New Green NiTi Reciprocating Instruments. *Materials* **2020**, *13*, 3736. [\[CrossRef\]](#) [\[PubMed\]](#)
39. Wei, S.; Zhang, J.; Zhang, L.; Zhang, Y.; Song, B.; Wang, X.; Fan, J.; Liu, Q.; Shi, Y. Laser powder bed fusion additive manufacturing of NiTi shape memory alloys: A review. *Int. J. Extrem. Manuf.* **2023**, *5*, 032001. [\[CrossRef\]](#)
40. Mumtaz, K.; Hopkinson, N. Top surface and side roughness of Inconel 625 parts processed using selective laser melting. *Rapid Prototyp. J.* **2009**, *15*, 96–103. [\[CrossRef\]](#)
41. Khairallah, S.A.; Anderson, A. Mesoscopic simulation model of selective laser melting of stainless steel powder. *J. Mater. Process. Technol.* **2014**, *214*, 2627–2636. [\[CrossRef\]](#)
42. Liu, Y.J.; Li, S.J.; Wang, H.L.; Hou, W.T.; Hao, Y.L.; Yang, R.; Sercombe, T.B.; Zhang, L.C. Microstructure, defects and mechanical behavior of beta-type titanium porous structures manufactured by electron beam melting and selective laser melting. *Acta Mater.* **2016**, *113*, 56–67. [\[CrossRef\]](#)
43. Roehling, T.T.; Wu, S.S.; Khairallah, S.A.; Roehling, J.D.; Soezeri, S.S.; Crumb, M.F.; Matthews, M.J. Modulating laser intensity profile ellipticity for microstructural control during metal additive manufacturing. *Acta Mater.* **2017**, *128*, 197–206. [\[CrossRef\]](#)
44. Weaver, J.S.; Heigel, J.C.; Lane, B.M. Laser spot size and scaling laws for laser beam additive manufacturing. *J. Manuf. Process.* **2022**, *73*, 26–39. [\[CrossRef\]](#) [\[PubMed\]](#)
45. Yildiz, R.A.; Popa, A.-A.; Malekan, M. On the effect of small laser spot size on the mechanical behaviour of 316L stainless steel fabricated by L-PBF additive manufacturing. *Mater. Today Commun.* **2024**, *38*, 108168. [\[CrossRef\]](#)
46. Power, J.J.; Robinson, J.; Dowling, D.P. Application of laser spot size to control the microstructure and printability of Ti-6Al-4V alloy parts. *J. Manuf. Process.* **2026**, *168*, 524–538. [\[CrossRef\]](#)
47. Hung, C.-H.; Chu, C.-W.; Chang, T.-L.; Lin, T.-C.; Chen, Y.-P. Effect of focal spot size on AlSi10Mg alloy parts fabricated by laser powder bed fusion: Process window, mechanical properties, and microstructure. *J. Alloys Compd.* **2024**, *977*, 173338. [\[CrossRef\]](#)
48. Ratajski, J.; Bałasz, B.; Peta, A.; Krupski, P.; Bochenek, K.; Major, Ł. Tailoring phase transformation behavior and functional properties of LPBF-fabricated NiTi alloys via post-processing heat treatment. *Arch. Civ. Mech. Eng.* **2026**, *26*, 134. [\[CrossRef\]](#)
49. Frenzel, J.; Zhang, Z.; Somsen, C.; Neuking, K.; Eggeler, G. Influence of carbon on martensitic phase transformations in NiTi shape memory alloys. *Acta Mater.* **2007**, *55*, 1331–1341. [\[CrossRef\]](#)
50. Honma, T.; Funakubo, H. *Shape Memory Alloys*; Gordon and Breach Science Publishers: New York, NY, USA, 1987; pp. 61–115.

51. Prokoshkin, S.D.; Korotitskiy, A.V.; Gundyrev, V.M.; Zeldovich, V.I. Low-temperature X-ray diffraction study of martensite lattice parameters in binary Ti–Ni alloys. *Mater. Sci. Eng. A* **2008**, *481–482*, 489–493. [[CrossRef](#)]
52. Xiang, Z.; Yang, Q.; Zhang, S.; Zhang, T.; Xia, Z.; Huang, M.; Chen, J.; Huang, S. Influence of LPBF Parameters and Post-Annealing Temperature on Martensitic Transformation and Superelasticity of Ni-Rich Ni_{51.9}Ti_{48.1} Alloy. *Metals* **2025**, *15*, 1180. [[CrossRef](#)]

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