Laser-based powder bed fusion of niobium with different build-up rates

Tjorben Griemsmann¹ · Arvid Abel¹ · Christian Hoff¹ · Jörg Hermsdorf¹ · Markus Weinmann² · Stefan Kaierle¹

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Abstract

Niobium is an important material for high temperature applications, in space, in superconductors or in chemical process constructions. Laser-based powder bed fusion of niobium (PBF-LB/M/Nb) offers new opportunities in design, though it is still an expensive technique. The build-up rate is an important factor for economical manufacturing using PBF-LB/M/Nb. It is largely influenced by variation of process parameters, affecting the heat flow during the manufacturing process. In this work, an empirical model for PBF-LB/M/Nb is developed. Based on this model, manufacturing parameter sets using different volume build-up rates are predicted and confirmed. They enable the manufacture of parts with homogeneous and crack-free microstructure with more than 99.9% relative density. Tensile and hardness tests of specimens, which were manufactured using different parameter sets, are performed to determine the effects of the build-up rate—and thus the heat flow during manufacturing—on different mechanical properties. The ultimate tensile strength and yield strength of as-manufactured specimens reach values up to 525 MPa and 324 MPa, respectively, while the elongation at break ranges between approximately 8 and 16%. The Vickers hardness of all specimens was in the range of 149 ± 8 HV0.1. In addition, the microstructure of the manufactured samples is investigated by means of light as well as scanning electron microscopy.

Keywords Laser-based powder bed fusion · Niobium · Mechanical properties · Design of experiments

Abbreviations

BEC	Backscattered electron composition
b_r	Build-up rate
EBSD	Electron backscatter diffraction
h	Hatch distance
PBF-LB/M/Nb	Laser-based powder bed fusion of metals/niobium
P	Laser power
t	Layer thickness
pc	Parameter combination
SEI	Secondary electron imaging
SEM	Scanning electron microscope
v	Scanning speed
UTS	Ultimate tensile strength

E_v	Volume energy
YS	Yield strength

1 Introduction

Niobium belongs to the group of refractory metals, which are characterized by a high resistance to electrochemical corrosion, high temperature strength and good electrical and heat conducting properties [1]. The major consumer of niobium is the steel industry, where it is used as an alloying element to improve the welding behaviour, the heat resistance and the yield and tensile strength [2]. Applications for pure niobium are also in the electronic industry, i.e. capacitors [3–5], in superconductors due to the high superconducting transition temperature [6–8], in the chemical process industry due to the high corrosion resistance [9] and the low thermal neutron capture cross-section [10] makes it suitable to control nuclear reactions. Other upcoming usages are as a bio inert material for medical devices and as absorber for gases in vacuum technology [11]. Niobium is highly ductile and accordingly easy to process by means of machining, forging, drawing etc. A relatively

✉ Tjorben Griemsmann
t.griemsmann@lzh.de

¹ Laser Zentrum Hannover e.V., Hollerithallee 8,
30419 Hannover, Germany

² TANIOWIS GmbH, Im Schleeke 78-91,
38642 Goslar, Germany

new approach to manufacturing niobium and niobium-based alloys is additive manufacturing, also referred to as 3D printing. Laser-based powder bed fusion, also known as Selective Laser Melting, is a powder bed-based additive manufacturing technique for metals. For the manufacturing process, the CAD model is sliced virtually with a specific thickness. To manufacture one layer, powder with the layer thickness is coated above the previous layer and selectively melted by a laser beam. The process repeats until the part is finished. PBF-LB/M/Nb offers many advantages compared to conventional manufacturing technologies. The major advantages are the freedom in designing complex parts like lightweight structures, contoured cooling channels and the economic benefit in the manufacture of individual components [12]. Moreover, metals or metal alloys with very high melting points such as niobium, tantalum or tungsten, which cannot be cast, could be processed into complex parts without constraints. There are several research groups working on the manufacturing process of niobium alloys with PBF-LB/M [13–18], but only one group is processing pure niobium. They produced a porous coating from niobium with PBF-LB/M/Nb. It is reported about difficulties to melt the powder completely, due to the high melting point of 2468 °C, and therefore to create a dense layer [19]. In addition, there is a research group around Terrazas et al. working on fabricating pure niobium through Electron Beam Melting. Relative densities up to 99.7% could be achieved. The ultimate tensile strength (UTS) and yield strength (YS) are with 225 MPa and 140 MPa in mean slightly higher and the elongation at break (34%) is slightly lower than that of conventionally wrought niobium (UTS = 205 MPa, YS = 135 MPa and elongation at break = 45%) [20, 21]. However, the manufacturing costs with additive technologies are still relatively high. A large proportion of the costs arises from the expensive plant technology. Therefore, the economics could be improved by manufacturing parameters which enable faster throughput thereby increasing the build-up rate. Higher build-up rates will lead to shorter laser powder interaction times and a change in heat flow and cooling rate of the specimen, due to a faster moving heat source, respectively laser beam. Because the general cooling rate during the PBF-LB/M/Nb process has a big impact on the microstructure and consequently on the mechanical properties [22–26], it is assumed that different manufacturing parameters could result in different mechanical properties, as shown in [27] for stainless steel and laser powers of 400 W and 1000 W. The aim of this paper is to investigate the impact of the heat flow and thermal gradients on the microstructure and the mechanical properties of niobium through different build-up rates. For this purpose, several PBF-LB/M parameter sets are explored to manufacture pure niobium parts with densities above 99.9%. Using design

of experiments (DoE) techniques, an empirical model for the achieved relative density is developed and validated to derive parameters which are resulting in high densities and different build-up rates. The hypothesis of changing properties by a variation of the heat flow is verified by tensile tests, hardness tests and a discussion of the microstructure.

2 Materials and methods

The powder used for the investigations was a gas atomized pure niobium powder (AMtrinsic® Spherical Niobium, Taniobis GmbH) of the sieve fraction 10 to 63 µm (D(0.10): 22.37 µm, D(0.50): 39.42 µm, D(0.90): 65.97 µm, measured by laser light scattering) which has been obtained using EIGA techniques (EIGA = Electrode Induction-melting Gas Atomization). The oxygen level was 556 ppm, tap density was 5.56 g/cm³ and Hall Flow (0,1') was 12 s. Full analytical details are provided in the product data sheet which is available online [28]. To investigate the morphology of the powder, scanning electron microscope (SEM) images, using a Quanta 400 FEG (FEI Thermo Fisher Scientific, USA), were recorded of the powder at different magnifications as shown in Fig. 1. Powder bed SEM images and polished cross-sections show that most particles exhibit a perfectly spherical shape. Only a negligible number of particles has irregular, i.e. non-spherical surfaces. In few cases, satellite particles are visible which adhere to larger spheres. The polished cross-section of embedded powders demonstrates a more or less perfect density and homogeneity of the powder particles. Voids, cracks or segregations are hardly visible in secondary electron imaging (SEI), Fig. 1(c) or backscattered electron composition (BEC) imaging. The BEC image in Fig. 1(d) not only confirms the findings of the SEI image but it also visualizes crystal sizes and orientations within the spherical particles. The PBF-LB/M/Nb processes were carried out using an additive manufacturing system TruPrint 1000 (Trumpf GmbH & Co. KG; Germany). The additive manufacturing system was equipped with an ytterbium fiber laser used in continuous wave mode with a maximum power of 170 W and a wavelength of 1070 nm. The optics included a galvanometric scanner and an f theta lens, which created a focus diameter of 30 µm on the working level. The maximum build space was 100 mm in diameter and 80 mm in height. The oxygen content of the building chamber was reduced to 200 ppm by the use of argon as an inert gas.

2.1 Experimental methods

In a first step, an empirical model for the relative density as a function of the PBF-LB/M parameters laser power P ,

Fig. 1 SEM images of the used AMtrinsic® Spherical Niobium powder at $\times 250$ **a** and $\times 500$ **b** magnification. SEI **c** and BEC **d** images of a polished cross-section of resin-embedded powder at $\times 100$ and $\times 1500$ magnification, respectively

scanning speed v and hatch spacing h was constructed to determine different process parameter combinations (pc), which led to high relative densities in the manufactured part. For this purpose, cubes with an edge length of 5 mm were manufactured on 2-mm support structures. All specimens were built with a layer thickness of $20\text{ }\mu\text{m}$, a rotation of 15° around the z -axis and chess pattern with a squared maximum field size of 4 mm and a $25\text{-}\mu\text{m}$ distance between the fields. The fields were filled with an unconnected zigzag pattern as illustrated in Fig. 2. Each layer was rotated with an increment angle of 90° and shifted by 1 mm in y and x direction. Accordingly every fourth layer had the same hatching strategy. The powder feed rate was kept constant at 220% and the oxygen content below 200 ppm. No contour scans were done to reduce the factors, which have an influence on the relative density, and the amount of required trials. The values for the input variables were chosen through a DoE approach to reduce the necessary trials. In this case, a central composite design was used. In this type of design, the variable matrix is set in the shape of a ‘cube’ and a ‘star’. As demonstrated in Fig. 3, the manufactured parameter sets were the value combinations in the corners of the cube and in the additional star points with the centre point. The values are given in Table 1. It was important that every combination led to a successfully built specimen with a measurable relative density. For

this reason, the choice of the parameter limits was based on pre trials. With each parameter set resulting from the ‘cube’ matrix, two specimens were manufactured. The centre point of the ‘star’ matrix was used for manufacturing specimens eight times and the outer points two times. In sum, 36 specimen were manufactured in one PBF-LB/M/Nb process. The whole process is carried out twice to involve reproducibility. The predicted empirical model is a mathematical approximation. The model was confirmed by building additional test samples, which should theoretically lead to high relative densities. To evaluate the influence of the built-up rate on the mechanical properties, confirmed parameter combinations were chosen to manufacture test specimen for tensile tests according to DIN 50125. For each combination, three specimens were manufactured orientated in building direction.

2.2 Methods for analysis

The ‘cube’ specimens were embedded in epoxy and cross-sections of two parallel levels created to evaluate the relative density. Images of the cross-sections were taken using a light microscope (Olympus BX60) with a magnification of five and a microscope camera (Zeiss AG AxioCam MRc). The images were processed with a computer vision application using OpenCV. The application detects the

Fig. 2 Schematic illustration of the scanning paths in each consecutive layer

contours of the specimen and the pores based on colour differences as shown in Fig. 4. The relative density was calculated by setting the sum of the areas determined as

pores (red lines in Fig. 4) in relation to the area determined as the specimen (green line in Fig. 4), according to Eq. 1.

$$Rel.Density = \left(1 - \frac{\sum Area_{pores}}{Area_{CrossSection}} \right) \cdot 100\% \quad (1)$$

Based on the calculated values, a mathematical model using the statistics software JMP (SAS Institute) was fitted to describe the relative density as a function of laser power, scanning speed, hatch spacing and second orders of the variables or interactions between them. Only high significant (p value < 0.0001) influences were used for the regression. The mathematical model was used to

Fig. 3 Schematic illustration of a central composite design. Each dot marks a parameter combination

Table 1 Values for the input variables in the central composite design

	Star 0	Cube 0	Centre	Cube 1	Star 1
Laser power P in W	90	110	130	150	170
Scanning speed v in mm/s	500	750	1000	1250	1500
Hatch spacing h in μm	20	40	60	80	100

calculate 84 parameter combinations, which should lead to relative densities above 99.9% in theory. The parameter combinations were used to build cubic specimens two times and the mathematical model was updated with the measured relative densities to achieve a better approximation. The build-up rate B_r and volume energy E_V of the parameter combinations were calculated according to Eqs. 2 and 3 [29] where t is the layer thickness. For the build-up rate, the time needed for recoating, jumps, acceleration and deceleration of the scanner was not considered. The volume energy is related to the hatch spacing and the thickness of one layer.

$$B_r = v \cdot h \cdot t \quad (2)$$

$$E_V = \frac{P}{v \cdot h \cdot t} \quad (3)$$

A set of specimens manufactured with parameter combinations with different build-up rates, which led to high relative densities, were prepared for hardness testing. For the evaluation, a Nexus 4000 (Innovatest, Netherlands) was used and the HV0.1 Vickers hardness determined according to DIN EN ISO 6507-1:2018 applying an indentation force of 0.981 N. The hardness was correlated with the energy input and the built-up rates of the specimens to detect trends or significances. The tensile specimens were tested with the tensile testing machine Landmark 370.10 (MTS, USA). The machine elongates the specimen until it fractures. The applied force was recorded together with the elongation to compare the different built-up rates. To determine the fracture mechanism, SEM images of the fracture surface were taken and interpreted. To generate an understanding, how the build-up rate or volume energy influences

the microstructure cross-sections were etched with a mixture of nitric acid, hydrochloric acid and hydrofluoric acid. The grains were investigated via the light microscope and for a more detailed insight through electron backscatter diffraction (EBSD).

3 Results and discussion

3.1 Empirical model

All cubic specimens were successfully built without obvious defects on the surface. The relative densities of the specimens had the lowest value with $81.55 \pm 0.37\%$ for the parameter combination of $P = 110$ W, $v = 1250$ mm/s and $h = 80$ μm . The highest density value was determined with $99.74 \pm 0.05\%$ for $P = 150$ W, $v = 750$ mm/s and $h = 80$ μm . The cross-section of the best specimen is shown in Fig. 5. The relative densities of all 72 (2×36) specimens of the central composite design were used for the mathematical approximation to determine the relative density as a function of the laser power, scanning speed and hatch spacing. Only highly significant values (p value < 0.0001) were used for the regression given in Eq. 4. The regression equation is only valid in the range of the central composite design given in Table 1. Based on Eq. 4, greymaps with fixed hatch spacing in 5- μm steps from 30 to 95 μm were created. An example ($h = 65$ μm) is given in Fig. 6. Based on these greymaps parameter combinations in the area of relative densities $> 99.9\%$ were chosen in steps of 10 W for the laser power and 250 mm/s for the scanning speed to confirm the

Fig. 4 Example of a cross-section; contour of the specimen (green line) and contours of the pores (red lines)

Fig. 5 Cross-sections of the specimen with the highest density ($P = 150$ W, $v = 750$ mm/s, and $h = 80$ μm)

Fig. 6 Greymap of Eq. 4. The plot represents the regression based on the 72 specimens of the central composite design. The hatch spacing is fixed to 65 μm

model. To extend the range of the empirical model, also extrapolated parameter combinations with a scanning speed of 250 mm/s were considered. This resulted in 84 parameter combinations for confirmation trials.

$$\begin{aligned}
 & \text{Rel.D.}(P, v, h) \\
 &= -3.6 \cdot 10^{-3} \frac{\%}{W^2} \cdot P^2 - 8.1 \cdot 10^{-6} \frac{\% \cdot s^2}{mm^2} \cdot v^2 \\
 & - 2.3 \cdot 10^{-3} \frac{\%}{\mu m^2} \cdot h^2 + 369 \cdot 10^{-6} \frac{\% \cdot s}{W \cdot mm} \cdot P \cdot v \\
 & - 150 \cdot 10^{-6} \frac{\% \cdot s}{mm \cdot \mu m} \cdot v \cdot h + 760.5 \cdot 10^{-3} \frac{\%}{W} \cdot P \\
 & - 31.6 \cdot 10^{-3} \frac{\% \cdot s}{mm} \cdot v + 449 \cdot 10^{-3} \frac{\%}{\mu m} \cdot h + 41.1\% \quad (4)
 \end{aligned}$$

The lowest density value out of the 84 confirmation specimens was 97% for the parameters $P=170\text{ W}$, $v=1500\text{ mm/s}$ and $h=35\text{ }\mu\text{m}$. Eighteen out of the 84 predicted and tested parameter combinations exhibited a relative density $>99.9\%$; they are listed in Table 2. The build-up rates ranged from $0.33\text{ mm}^3/\text{s}$ (pc 1) to $1.63\text{ mm}^3/\text{s}$ (pc 4) which is about five times this value. The volume energy was calculated for pc 1 to 400 J/mm^3 , which is about four times higher than that of pc 4 which accounted to 104.6 J/mm^3 . The regression from Eq. 4 was updated using the results of the 84 confirmation specimen and is therefore even more precise in predicting parameter combinations with high relative densities than before. The new regression is given in Eq. 5 and is illustrated as a greymap in Fig. 7. As shown in Eq. 5, the variables laser power P , scanning speed v and hatch spacing h have a negative quadratic effect on the relative density. There is a maximum in relative density for each variable, but it shifts if another variable is changed. That is because the variables interact with each other as shown in the terms with $P \cdot v$ and $v \cdot h$. Due to the high number of different pc resulting in high relative densities, the DoE

approach with approximation of a mathematical model has been considered suitable for this process development.

$$\begin{aligned}
 & \text{Rel.D.}(P, v, h) \\
 &= -3.4 \cdot 10^{-3} \frac{\%}{W^2} \cdot P^2 - 8.9 \cdot 10^{-6} \frac{\% \cdot s^2}{mm^2} \cdot v^2 \\
 & - 2.1 \cdot 10^{-3} \frac{\%}{\mu m^2} \cdot h^2 + 344 \cdot 10^{-6} \frac{\% \cdot s}{W \cdot mm} \cdot P \cdot v \\
 & - 90 \cdot 10^{-6} \frac{\% \cdot s}{mm \cdot \mu m} \cdot v \cdot h + 732.2 \cdot 10^{-3} \frac{\%}{W} \cdot P \\
 & - 31.1 \cdot 10^{-3} \frac{\% \cdot s}{mm} \cdot v + 359 \cdot 10^{-3} \frac{\%}{\mu m} \cdot h + 46.1\% \quad (5)
 \end{aligned}$$

3.2 Microstructure

Light microscope images of etched cross-sections of manufactured specimens were prepared to obtain information on grain growth and orientation as a function of process parameters and scanning paths. The examples given in Fig. 8, i.e. pc 17 (a) and pc 14 (b) revealed two types of grain growth, depending on the sectioned scanning paths. Figure 8(a) shows a cross-section of a specimen which is obtained by utilization of zigzag scan vectors in each consecutive layer. The left side of the cross-section, which is graphically separated by a red line from the right to one-third of the image, indicates a vertical columnar grain orientation. Here, the laser beam moved nearly perpendicular to the image plane, as indicated by the x and $\bullet$ icons in the upper figure part. In contrast, in the section on the right side of Fig. 8(a), a wave-type triangular crystal orientation is observed. The distance between a wave maximum and the minimum was $310\text{ }\mu\text{m}$, which is approximately the distance between the middle of two neighbored melt tracks, taking the rotation of 15° around the z -axis and a hatch spacing of $85\text{ }\mu\text{m}$ in mind (compare with Fig. 2). The grains are orientated in direction of the main heat conduction, which points in a direction between the negative build direction and the negative scanning direction [23]. So, it can be assumed that the zigzag pattern resulted in a negative or positive slope of the wave-type grains, depending on the direction of the scanning track. A schematic illustration of the shape origin is given in Fig. 9, where the heat conductions of two neighbored scanning tracks are modelled as bars and cut in the angle of the cross-section. Figure 8(b) corresponds to a specimen which was obtained by application of zigzag scan vectors with alternating xy -orientation, like the most of the investigated specimens. The scanning paths rotated at least every second layer by 90° . Therefore, the grain growth appeared less ordered compared with that shown in subfigure (a). A difference in the microstructure was not observed between the compared pc, only the alignment of the scanning paths had an influence in this investigation.

Table 2 Build-up rates and volume energy for pc with relative densities above 99%

Parameter combination pc	Laser power P in W	Scanning speed v in mm/s	Hatch spacing h in μm	Relative density in %	Build-up rate B_r in mm^3/s	Volume energy E_v in J/mm^3
1	130	250	65	99.90	0.33	400.0
2	130	500	65	99.92	0.65	200.0
3	140	500	65	99.94	0.65	215.4
4*	170	1250	65	99.94	1.63	104.6
5	130	500	70	99.94	0.70	185.7
6	150	750	70	99.92	1.05	142.9
7*	110	250	75	99.95	0.38	293.3
8	120	250	75	99.94	0.38	320.0
9	130	250	75	99.94	0.38	346.7
10*	130	500	75	99.97	0.75	173.3
11	150	750	75	99.92	1.13	133.3
12	110	250	80	99.94	0.40	275.0
13*	120	250	80	99.96	0.40	300.0
14	140	500	80	99.92	0.80	175.0
15	150	750	80	99.93	1.20	125.0
16*	110	250	85	99.94	0.43	258.8
17	120	250	85	99.91	0.43	282.4
18	130	250	85	99.91	0.43	305.9

*Parameter combinations chosen for tensile tests

The EBSD image displayed in Fig. 10 represents a clip which typically appears in areas with triangular wave-type grain growth mentioned above with three different orientations. Axis 2 (A2) corresponds to the orientation with respect to the build direction and a mixture of 001 alignment on the connection of the triangular wave-type grains and a strong 111 alignment between the connection lines. The 001 alignment was also observed in the transverse (axis 1) and normal (axis 3) direction, but less intense. The texture between the 001 alignment in A1 and A3 was mainly orientated in 101.

Fig. 7 Greymap of Eq. 5. The plot represents the regression based on the 72 specimens used to generate the empirical model and the 84 confirmation specimens and is therefore more precise. The hatch spacing is fixed to 65 μm

3.3 Hardness tests

The hardness of each printed specimen was determined using the Vickers hardness test according to EN ISO 6507-1:2018. Like the microstructure discussed above, the Vickers hardness (HV0.1) was not obviously influenced by different energy inputs or build-up rates. In contrast to the microstructure, the hardness has neither been influenced by the scanning pattern. The 18 tested specimens, manufactured with the pc which led to relative densities above 99.9% (c.f. Table 2), showed hardness values of $149 \pm 8 \text{ HV0.1}$. These values are high compared with cold rolled niobium sheets which typically possess hardness values of approximately 50 HV0.1 [30]. An explanation for these high hardness values could be due to the high cooling rates which are typical in the PBF-LB/M process and the fine columnar microstructures achieved thereby [15, 31–33].

3.4 Tensile tests

The tensile test specimens were built with pc 4, 7, 10, 13 and 16 (c.f. Table 2). Accordingly, three sets of specimens, which were manufactured using relatively low build-up rates and thus high volume energies of approximately 260–300 J/mm^3 (pc 7, 13, 16), one set with medium build-up rate and medium volume energy (173.3 J/mm^3 , pc 10) and one set with a high build-up rate (pc 4) and low volume

Fig. 8 Light microscope images of etched cross-sections, scanning paths are shown on top (rotated about 15° around build direction). The sectioned level of **a** (pc17) has columnar (left of red line) and triangular growth (right of red line). Sectioned level of **b** (pc14) showed an inhomogeneous grain growth

Fig. 9 Schematic illustration of wave-type grain orientation; **a** top view with scanning direction of two neighbouring scanning tracks; **b** bars which indicate the direction of heat conduction and grain orientation, as described in [23], with cut at the cross-sectioned plane of Fig. 8

Fig. 10 EBSD images of pc 16 in the area of triangular grain growth (compare with Fig. 8a right side of image). Build direction (BD, A2) points from left to right, and the orientation of the axis is given on the right side

Table 3 Results of tensile tests

Parameter combination pc	Build-up rate B_r in mm^3/s	Volume energy E_v in J/mm^3	Ultimate tensile strength in MPa	Yield strength in MPa	Elongation at break in %
7	0.38	293.3	489 ± 13	310 ± 10	11.1 ± 1.6
13	0.40	300.0	525 ± 19	316 ± 25	12.6 ± 0.4
16	0.43	258.8	500 ± 16	309 ± 4	11.1 ± 2.0
10	0.75	173.3	475 ± 11	309 ± 11	16.6 ± 0.8
4*	1.63	104.6	444	324	8.3

*Only one value

energy of approximately $105 \text{ J}/\text{mm}^3$ were considered for tensile testing. Three specimens of each pc were tested. Results for ultimate tensile strength (UTS), yield strength (YS) and elongation at break are given in Table 3. The bar charts provided in Fig. 11 indicate only minor differences in the determined values. Only a slight tendency of the UTS for lower values with increasing build-up rate or decreasing volume energy was observed. The highest value was $525 \pm 19 \text{ MPa}$ measured for pc 13 ($B_r = 0.4 \text{ mm}^3/\text{s}$), while the lowest value corresponded to the highest build-up rate ($B_r = 1.63 \text{ mm}^3/\text{s}$) with pc 4 and 444 MPa . For pc 4, only one value was available, because of an error

of the coating mechanism during the building process, which led to an early fracture in the tensile test. For the yield strength, no significant differences were observed. All values ranged slightly above 300 MPa . The values for UTS and YS were almost twice as high as for the reported electron beam-melted and wrought samples from Terrazas et al. [20]. It is known that the mechanical properties of niobium are affected by its oxygen content. For example, a niobium sheet containing 800 ppm of oxygen has been reported to possess similar values for the UTS and YS as the specimens investigated in this study [34]. This could explain the high strength of PBF-LB/M processed niobium,

Fig. 11 Bar charts of ultimate tensile strength, yield strength and elongation at break in the order of increasing build-up rate. Bottom: Stress-strain diagrams of all tested specimen

Fig. 12 SEM images of fractured surfaces (pc 13). The overview **b** shows areas with ductile **a** and transgranular **c** fractured surfaces

taking into account that the starting powder had an oxygen content of approximately 556 ppm, which may further be increased during the PBF-LB/M process itself [35]. Another additional explanation is the long and fine grain shaped microstructure, typical for PBF-LB/M processed parts, which is caused by high cooling rates and thermal gradients, increasing the mechanical properties [36]. Grain shape and orientation are a possible reason for the relatively low elongation at break, which was approximately 8 to 16%. Conventionally manufactured niobium, even with high oxygen content, has an elongation of about 40 to 50% [34], while niobium with long fine grains, manufactured by electron beam melting, showed similar values < 15% [37] compared with PBF-LB/M processed niobium. The SEM images of the fracture surfaces appeared more or less identical, suggesting a similar fracture behaviour of all five sets built using different pc. As an example, SEM images of pc 13 are illustrated in Fig. 12. The fractured

specimen is significantly necked as evident from Fig. 12(b). SEM images 12 a and 12 c, which were recorded at a higher magnification, point to the presence of both ductile and transgranular brittle fracture behaviour, as there are areas with lamellar surface structures but also dimples. The surfaces suggesting ductile fracture have a higher proportion than those which appear transgranular. The presence of transgranular fracture could be an explanation for the low measured elongation at break as well.

3.5 Demonstrator part

Niobium and its alloys are potential materials for application in space or high temperature environment. To provide an example, the investigated parameter combination 7 and 10 were used to manufacture a design specimen of a navigation nozzle for satellites. The nozzle design was optimized for the additive manufacturing process and was manufactured in two parts. Those were assembled subsequently, as shown in Fig. 13. The design contained a 0.5-mm curved wall and a tube with 1-mm inner diameter. Both pc led to successfully manufactured parts without obvious defects.

Fig. 13 Construction of a navigation nozzle for satellites. Top: CAD model; bottom: PBF-LB/M/Nb manufactured part

4 Conclusion

The present study describes an approach for the development of process parameter combinations (pc) for the additive manufacturing of spherical niobium powders by means of PBF-LB/M. The developed model allows a prediction of the process results considering a variation of process parameters such as laser power, scanning speed and hatch spacing. The described approach shall be principally adaptable to other materials than niobium discussed here. For PBF-LB/M processing of niobium, the following conclusions can be drawn:

- In a first step, a process field with 13 different pc has been investigated. Based on the results for the relative

densities of the built specimens, an empirical model was fitted. Using this empirical model, 84 pc have been chosen, which should theoretically provide parts with relative densities > 99.9%. The second step, the validation run of the 84 pc, resulted in specimens with at least 97% relative density. Eighteen pc actually led to parts with relative densities > 99.9%. It can thus be assumed that the DoE approach is highly applicable to predict process results in this case.

- The build-up rates for the 18 pc with relative densities > 99.9% varied from 0.33 to 1.63 mm³/s. This provides a high potential for cost savings, especially in tightly packed build cycles with long laser scanning times in each layer.
- The microstructures of the manufactured parts depended on the direction of the scanning paths. Scanning paths nearly orthogonal to the cross-section resulted in vertically oriented columnar grains, while scanning paths nearly parallel to the section plane resulted in a triangular grain growth, caused by the direction of heat conduction.
- All tested specimens exhibited high Vickers hardness of approximately 150 HV0.1. This is most probably attributed to high cooling rates occurring in PBF-LB/M and the associated formation of special fine and long grained microstructures.
- There were no significant differences detectable in the mechanical properties of niobium manufactured using different pc. Regarding UTS, there was only a slight trend observable: UTS was decreasing with higher build up rates or with lower volume energy input. However, all pc led to high values for both UTS and YS between 444 and 525 MPa as well as 309 MPa and 324 MPa, respectively. It can therefore be assumed that an increase of oxygen content during the process or powder atomization increased these values. The elongation at break was lower compared with conventionally processed niobium; the determined values are in the range of approximately 8 to 16%. SEM investigations revealed the presence of both transgranular and ductile fracture. The simultaneous appearance of ductile and transgranular (brittle) fracture was most probably caused by the special morphology, i.e. relatively long and fine-shaped grains.
- The applicability of the investigated parameters has been shown through a design demonstrator of a navigation nozzle for satellites. No obvious defects were observed.

All in all, the hypothesis of a significant dependency of the mechanical properties on the built-up rate could not be confirmed within the scope of this trial. However, since the available laser power of the manufacturing equipment was

limited to 170 W, further investigations should be addressed using higher laser power and increased scanning speeds as well as powder bed thickness, improving the throughput, i.e. build-up rate and thus overall efficiency of the process. A variation of the hatching strategy might also influence the microstructure and properties of the manufactured part as it directly influences the direction of the heat conduction and thus the grain orientation.

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Author contribution

Tjorben Griemsmann: conception; analysis; interpretation; visualization; writing—original draft preparation
 Arvid Abel: conception; execute trials; analysis; interpretation; visualization
 Christian Hoff: writing—review and editing; supervision
 Jörg Hermsdorf: writing—review and editing; supervision
 Markus Weinmann: interpretation; writing—original draft preparation / review and editing
 Stefan Kaierle: writing—review and editing; supervision

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Data availability Data will be available upon request.

Declarations

Conflict of interest The authors declare no competing interests.

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