Quantitative Microstructural Characterization of Precipitates and Oxide Inclusions in Inconel 625 Superalloy Additively Manufactured by L-PBF Method



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In this study, we perform quantitative characterization of precipitates and oxide inclusions in Inconel 625 additively manufactured by the laser powder-bed fusion (L-PBF) process. The application of different microscopy techniques allowed us to characterize the microstructure at micro- and nano-scale in the as-built and stress-relieved condition and correlate the features of grains and cellular substructure with parameters of particles along the planes parallel and perpendicular to the build direction. The optimized imaging conditions and image analysis procedure allowed easily distinguishing precipitates and oxide inclusions and performing their quantitative analysis. The results showed that intercellular areas are the preferential sites of precipitation of the Laves phase and NbC carbides with diameters in the range of 10 to 440 nm. Moreover, aluminum oxide inclusions with diameters in the range of 30 to 300 nm are randomly distributed. Regardless of the processing conditions of the examined samples, the influence of the stress-relief annealing on the secondary phases was not observed. In both the as-built and stress-relieved samples, the size of precipitates is in submicrometer scale. The analysis provided detailed information about the parameters of particles depending on the orientation versus the build direction. It was demonstrated that despite the tendency for columnar grain morphology and the anisotropy of the cellular substructure, the particle distribution is almost uniform throughout the volume of the additively manufactured L-PBF Inconel 625.

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I. INTRODUCTION

INCONEL 625 is a nickel-based superalloy solid solution strengthened by the content of chromium, molybdenum and niobium, which exhibits high strength and corrosion resistance in a variety of environments and at high and low temperature.^[1] Due to its properties, Inconel 625 has found applications in energy, aviation, automotive and chemical industries.^[2,3] Unfortunately, the disadvantage of the alloy is its poor machinability, which makes it difficult to manufacture parts with complex shapes. This limitation can be overcome by using additive manufacturing (AM) processes, which allow the fabrication of finished parts without additional operations, e.g., cutting or drilling, and reducing the number of components needed to assemble in larger-sized structures.^[4–6] For these reasons, AM processes are increasingly used in various industries to manufacture components made of Inconel 625.^[6,7]

The most widely applied AM process used for the fabrication of Inconel 625 components is Laser-assisted Powder Bed Fusion (L-PBF), in which a laser beam fuses successively applied powder layers along a predefined path. The process is complex and demanding and requires selection of optimal process parameters that ensure avoiding porosity, the so-called operating window.^[8-10] On the basis of extensive research, it has been established that the most important parameters of the L-PBF process are laser power, scanning speed, hatch spacing, layer thickness, scanning strategy, protective atmosphere and powder materials properties.^[11] Publications on Inconel 625 L-PBF manufacturing present limited data on process parameters and focus mainly on laser power, scanning speed and hatch spacing.^[12-14] The optimized conditions are determined for a specific machine and specific powder and cannot be generalized to the one set of parameters because they need to be adapted depending on the built geometry, material properties requirements and the process productivity.

Some general guidelines were proposed by Oliveira *et al.*^[10] who provided a summary of how the different parameters of the L-PBF process can be set to overcome microstructure and materials-related problems.

Studies of the microstructure and properties of the Inconel 625 additively manufactured by L-PBF are the subject of many reports, *e.g.*,.^[13–19] Regardless of the type of machine and powder characteristics, the published data on L-PBF Inconel 625 indicate that microstructure is primarily controlled by solidification characteristics.^[10] Building parts from layers of rapidly melting and solidifying powder result in the formation of a complex microstructure with typical features including melt pools, fine columnar and equiaxed grains, and the cellular or dendritic substructure within grains. Routinely as-built Inconel 625 parts are subjected to stress-relief annealing at a temperature of 870 °C for 1 hour.^[20] Alternatively, annealing at 980 °C for 1 hour, which is the standard treatment of conventionally manufactured Inconel 625, is also applied.^[14,21]

The L-PBF Inconel 625 is characterized by a strong morphological and crystallographic texture. The results described in the literature show that in the longitudinal plane, the large columnar grains elongated in the built direction (BD) predominate, while in the perpendicular plane, the grains are mostly equiaxed in shape.^[14,22,23] Such a grain morphology is associated with a crystallographic texture with a predominant {011} (100) and comparatively much weaker {001} (100) components.^[22]

Many studies focused on the cellular substructure have revealed that Nb and Mo segregate into intercellular areas, while cells are richer in Ni and Cr.^[24–27] The microsegregation of alloying elements leads to the precipitation of carbides and intermetallic phases in the intercellular areas. A similar phenomenon has been described previously in the Inconel 625 welds, which also exhibit a cellular-dendritic substructure but on a different size scale. It was revealed that the dendrite core regions are enriched in Ni and Fe and depleted in Nb, Si and Mo, which segregate to the interdendritic areas.^[28–30] Furthermore, it was observed that after solidification, the dendritic structure contains various combinations of eutectic components $\gamma/MC(NbC)$, $\gamma/$ Laves and $\gamma/M_6C^{[30]}$ as well as TiN or TiNbC precipitates.^[31,32]

Characterization of precipitates in as-built and stress-relieved L-PBF Inconel 625 recently attracted growing interest of researchers. It was shown that Nb-rich precipitates are MC carbides (cubic, Fm-3m),^[26] while Nb- and Mo-rich particles with plate-like morphology can be the γ'' phase (tetragonal, I4/mmm)^[19] or the δ phase precipitates (orthorhombic, Pmmm).^[20] The presence of the Laves phase (hexagonal, P63mmc), M₆C carbides (cubic, Fd3m), M₂₃C₆ carbides (cubic, Fm-3m) and P phase (orthorhombic, Pnma) is also possible.^[21,33]

Although a lot of research has been done on the phase analysis, so far there are only a few reports dedicated to the quantitative analysis of particles in L-PBF Inconel 625. Marchese *et al.*^[26] observed isolated precipitates as well as forming groups with a length in the range of 200 nm to 2 μ m in the intercellular areas of as-built

specimens. In turn, Li *et al.*^[24] reported the presence of Nb-rich carbides with diameters from 50 to 80 nm. Meanwhile, Inaekyan *et al.*^[12] observed larger carbides with elliptical morphology, size < 25 μ m and particle number density equal to 0.38 μ m⁻².

Furthermore, besides the precipitates, oxide inclusions with diameters $< 1 \ \mu$ m were observed in L-PBF Inconel 625.^[24,34] Non-metallic inclusions were also observed in various steels produced by L-PBF.^[35–38] They appeared in the form of silicon, manganese, iron and titanium oxide inclusions. Diameters of the oxide particles ranged from several nanometers up to 100 μ m, and a bimodal size distribution of oxides was often observed. Remarkably, the size of the small oxide particles in all these studies was between 10 nm and 80 nm, despite very different processing routes.^[38] It was determined that coarse oxides with diameters in the range of 10 to 100 μ m are created when the AM steel is processed using low laser power and slow scanning speed.^[38]

The origin of the oxide inclusions may be related to the oxidation of the feedstock powder and the phenomena occurring during the L-PBF process. The first possible stage at which oxides may form is the oxidation of the metal powder during its production by gas atomization.^[4,39,40] Oxygen may also come from an environment caused by moisture during powder storage and transport.^[37] Another possibility is the oxidizing of the metal powder due to the traces of oxygen in the shielding gas used in the L-PBF process.^[37] Literature reports indicate that regardless of the source of origin, oxide dispersoids are probably formed in most of the metal alloys produced by L-PBF and contribute to their dispersion strengthening.^[36,37]

The phase composition, size distribution and number density of precipitates and oxide inclusions in the L-PBF Inconel 625 are important microstructural characteristics in the as-received condition because they affect the further evolution of the microstructure and thus the properties at service conditions. However, there is still a lack of reports on a quantitative microstructural analysis of the L-PBF Inconel 625. Moreover, although it is well known that the highly textured columnar grains are typical structural features, it is not well documented yet whether it also results in the anisotropy in the cellular substructure and also the distribution of precipitates.

Therefore, in this study, we have performed the quantitative microstructural analysis of precipitates and oxide inclusions in as-built and stress-relief annealed Inconel 625 additively manufactured by the L-PBF process and correlated the results with the analysis of grains and cellular substructure along the planes parallel and perpendicular to the BD.

II. EXPERIMENTAL

A. Materials

The samples of Inconel 625 were fabricated by using two laser powder bed fusion 3D printers, Sisma MySint 100 (Sisma S.p.A., Italy) and EOS M290 (EOS GmbH, Germany), in optimal process parameter windows. The cube-shaped samples with the side length of 15 mm were produced by application of 67 deg built strategy. The samples fabricated by means of the Sisma MySint 100 were examined in as-built condition, while those achieved by the EOS M290 printer were additionally annealed at a temperature of 980 °C for 1 hour, followed by slow cooling in an argon atmosphere. The aim of heat treatment was stress relief and partial solution annealing.

B. Experimental Procedure

The microstructure of L-PBF Inconel 625 was analyzed using the specimens prepared from two orthogonal cube planes of AM sample, namely, the vertical plane parallel to the BD, marked as *xz*, and the horizontal plane, perpendicular to the BD, marked as *xy*. Quantitative microstructural analysis was performed in three different zones in the *xz* plane along the BD, namely, the bottom, middle and top of the samples.

The LM analyses were carried out using metallographic microscope MM100 (OPTA-TECH, Poland). Scanning electron microscopy (SEM) analysis was performed using an Inspect S50 microscope (FEI, USA) operated at 10 to 15 kV voltage. Metallographic specimens were ground with sandpaper and polished with the Al_2O_3 and diamond suspensions. To reveal the microstructure, the specimens were electrochemically etched with 60 pct nitric acid at 1.1 V for about 7 seconds and analyzed using LM and SEM by secondary electron (SE) imaging. In turn, to examine the oxide inclusions, non-etched samples were investigated using SEM backscattered electron (BSE) imaging. Microanalvsis of the chemical composition was performed by means of energy-dispersive X-ray spectroscopy (EDS) in SEM using the Octane Elect Plus spectrometer (EDAX, USA). EBSD scans were performed using a Field Emission Gun Nova NanoSEM 450 microscope equipped with an EBSD camera operating at 15 kV acceleration voltage. The step size was 1.5 μ m. The TSL-OIMTM analysis software was used for the analysis.

The microstructure and chemical composition were also examined by transmission (TEM) and scanning-transmission electron microscopy (STEM) using thin foils prepared by jet electropolishing. The TEM and STEM analysis was performed using a JEM-1010 ARP microscope (Jeol, Japan) and Tecnai TF 20 X-TWIN (FEI, USA) equipped with a STEM high-angle annular dark-field (HAADF) detector and an EDS r-TEM spectrometer of EDAX.

C. Image Analysis

For image analysis, ImageJ/Fiji software was used (NIH and LOCI, University of Wisconsin, USA). The image analysis of SEM and TEM images aimed at quantitative characterization of grains, cellular substructure, precipitates and non-metallic inclusions. To examine the size and morphology of grains, the EBSD images were used. The analysis of the cellular substructure was performed using SEM SE images. The ratio of the major axis (E_{2a}) to the minor axis (E_{2b}) of the ellipse described on the grain/cell was chosen as a measure of the shape grains and cells (grain aspect ratio, *GAR*, and cell aspect ratio, *CAR*). A scheme of determining the major and minor axis of an ellipse fitted to the particle/cell is given in supplementary Figure S-1 with the detailed description of the measurement procedure (refer to electronic supplementary material). The area fraction of intercellular areas was also determined.

As the parameters describing the particles, the area fraction A_A and the equivalent circle diameter (D) were used. The number density of the particles in unit area N_A was calculated according to Eq. [1]:

$$N_{\rm A} = \frac{N}{A},\tag{1}$$

where N is the number of particles and A is the total analyzed area.

To investigate particles that could not be seen in SEM images because of limited spatial resolution, TEM analysis of thin foils was performed. The equivalent diameters D and the number density of particles per unit volume N_V values were determined. The N_V was calculated by Eq. [2]:

$$N_{\rm V} = \frac{N_{\rm A}}{t + \overline{D}},\tag{2}$$

where N_A is the number density in unit area, t is the thickness of the thin foil, and \overline{D} is the arithmetical mean equivalent diameter of the particles.

The particle sphericity parameter C was calculated by Eq. [3]:

$$C = 4\pi \sum \frac{A_i}{L_i^2},$$
 [3]

where A_i and L_i are the area and perimeter of particular particles.

III. RESULTS AND DISCUSSION

LM images in Figures 1(a), (b) and (e), (f) show the microstructure of the as-built and stress relieved L-PBF Inconel 625 with the visible arrangement of the melt paths according to the scanning strategy. For both conditions in the xz plane, the profiles of the melt pools along the BD are U-shaped, and the regular fish scale pattern is observed (Figures 1(a), (e)). In the xy plane, the melt path direction varies by a rotation angle of 67 deg between successive layers following the scan strategy (Figures 1(b), (f)). Figures 1(c), (d) and (g), (h) shows the SEM SE images in which both in the as-built and stress-relieved condition the melt paths containing fine grains with cellular substructure can be seen.



Fig. 1—Microstructure of the as-built and stress-relieved L-PBF Inconel 625 observed in xz and xy planes: (a, b and e, f) LM images showing melt pool contours and melt paths as well as (c, d and g, h) SEM images of the cellular substructure.

Table I.Parameters Describing the Length and Width of Grains (E_{2a} and E_{2b}) As Well As Grain Aspect Ratio GAR in the
Bottom, Middle and Top of the Samples at the xz Plane

		Bottom	Middle	Тор
	As-Built			1
Grain Length E _{2a} [µm]	Min	5.27	4.81	5.79
	Max Maan Standard Deviation SD	207.48	151.56	185.91
Grain Width Eat [um]	Mean \pm Standard Deviation, SD Min	33.03 ± 31.00 2 24	28.34 ± 21.21 2.03	33.04 ± 27.04 3.17
	Max	60.56	42.25	45.78
	Mean \pm SD	10.08 ± 7.11	9.04 ± 5.80	10.24 ± 6.49
Grain Aspect Ratio GAR [-]	Min	1.04	1.08	1.07
	Max Mean + SD	12.62 3 55 + 2 02	11.6/ 3 33 + 1 88	15.16 3.42 ± 2.08
		5.55 ± 2.02	5.55 ± 1.00	J.42 ± 2.00
	Stress-Relief Annea	led		
Grain Length E _{2a} [µm]	Min	6.17	7.88	8.82
	Max	219.10	170.26	187.63
Croin Width E [um]	Mean \pm SD	32.71 ± 22.88	30.05 ± 18.83	32.12 ± 20.64
Grain width E_{2b} [μ in]	Mini Max	5.92 55.78	5.54	5.70
	Mean \pm SD	11.81 ± 6.48	11.59 ± 6.81	11.89 ± 7.69
Grain Aspect Ratio GAR [-]	Min	1.07	1.01	1.08
	Max	9.54	10.70	10.20
	Mean \pm SD	2.88 ± 1.36	2.82 ± 1.46	2.95 ± 1.50

A. Grain Structure

The grain structure examined by EBSD shows the grain morphology and orientation along the BD at the bottom, middle and top of the as-built sample (Figure 2(a)) and subjected to the stress-relief annealing (Figure 2(b)).

In the as-built sample in all the examined locations, most of the grains exhibit the columnar morphology with a distinct tendency to elongate along the BD or at a slight angle with respect to the BD. Such a morphology of grains in the vertical plane of as-built L-PBF Inconel 625 was also previously reported in the litera-ture.^[14,22,23,41] A high temperature gradient between the built platform and the upper layers heated by the laser along the BD results in the columnar grain growth. The deviation of the long axis of the columns from the BD proves that the actual grain growth direction in the melt pool during the L-PBF process is the balance between the preferred crystallographic orientation and the maximum heat flow direction.^[42] The results of the measurements of parameters describing the length and width of grains as well as the shape factor GAR are given in Table I, and the histogram of the GAR distribution is shown in Figure 2(c). In the bottom, middle and top areas the differences in the values of grain parameters are not distinct. The length of the grains varies from 4.81 to 207.48 μ m, and the longest grains occur at the bottom because of the intensive cooling by the building platform. The width of grains is much smaller, in the range from 2.03 to 60.56 μ m. The elongation parameter GAR does not significantly differ along the BD, and the predominant frequency of grains (17 pct) is characterized by GAR close to 2, while for some grains the GAR value even reaches 15 (Figure 2(c)).

In the stress-relieved sample the shape of the grains is varied for equiaxed, irregular and columnar, which are small and narrow grains and also bigger and wide. The length of grains varies from 6.17 to 219.10 μ m, while the range of grain width is much narrower, from 3.54 to 67.22 μ m. The elongation parameter GAR varies from 1.01 to 10.70. Figure 2(c) shows that the highest frequency of grains (22 pct) exhibits a GAR value close to 2, indicating a tendency to formation of elongated grains. Some of them even reach a GAR value around 10.

The formation of equiaxed grains in L-PBF Inconel 625 stress-relief annealed at 980 °C was also reported by Li et al.;^[24] however, similarly as in our study, the cellular structure was preserved. The appearance of, in addition to columnar grains, also equiaxed and irregular ones in the sample stress-relief annealed at 980 °C may indicate the nucleation of new grains due to the recrystallization. The literature reports that after annealing in the temperature range from 930 °C to 1100 °C,^[41,43] the microstructure with elongated grains initially aligned with the BD is transformed into equiaxed randomly oriented grains. Recently, also the dynamic recrystallization during L-PBF was observed by Sabzi et al.^[44] using ex-situ EBSD and by Schmeiser *et al.*^[45] by in situ diffraction experiments using synchrotron radiation. It was shown that the dislocation substructure created in



(a) As-built



(b) Stress-relief annealed



Fig. 2—EBSD maps of the grain orientations in the bottom, middle and top of the (a) as-built sample, (b) stress-relief annealed sample at the xz plane (color-coded triangles are displayed in the bottom left corner) and (c) grain aspect ratio distribution in the examined samples (Color figure online).

the as-built condition plays a crucial role in the recovery and recrystallization phenomena. Moreover, De Terris *et al.*^[43] revealed that L-PBF Inconel 625 manufactured with high volumetric energy conditions recrystallizes earlier because of a larger density of geometrically necessary dislocations. Based on the above literature results, it can be suspected that the creation of the equiaxed grains after stress-relief annealing is related to recrystallization. However, the shape of grains observed on images is a two-dimensional representation of a three-dimensional bulk, and some of them may be cross-sections of elongated grains. Support for this



(a) As-built



(b) Stress-relief annealed

Fig. 3-(111) and (200) pole figures for the examined sections: (a) as-built sample and (b) stress-relief annealed sample; in the (111) pole figures an ideal position of the main texture components of FCC materials are marked.

possibility is the observation of the preserved cell structure.

Further analysis was focused on grain orientation and texture analysis by EBSD.

Figure 3 presents the (111) and (200) pole figures for three analyzed regions (bottom, middle and top) in the as-built and stress-relief annealed sample. The as-built sample is characterized by slightly stronger texture than the stress-relief annealed. In the as-built samples the texture maximum (equal to 6) was observed for the top region. The texture intensity increases with growing distance from built platform. However, all regions in the stress-relieved sample are characterized by similar maximum of texture intensity.

For identification of the texture components, on each (111) pole figure (Figure 3) the ideal pole locations of Cube $(\{001\}\langle 100\rangle),$ Goss $(\{110\}\langle 001\rangle),$ Brass $(\{110\}\langle 112\rangle)$ and Copper $(\{112\}\langle 111\rangle)$ texture components are marked. For as-built sample, the locations of observed texture maxima were close to the ideal position of Cube components; this is especially clearly visible in the (111) pole figures for top and middle regions. In the bottom region, the texture maxima are located between ideal position Brass and Cube components and also Goss and Copper components. The observations indicate that the predominant texture component is a Cube type for middle and top regions. However, for bottom region, texture components close to the Cube, Brass, Goss and Copper types can be distinguished. The main Cube $(\{001\}\langle 100\rangle)$ texture components are observed also in the L-PBF manufactured Inconel 625^[46] and in the L-DED fabricated Inconel 718,^[47] but in other papers^[48] the Goss ($\{110\}\langle 001\rangle$) texture component was observed as predominant in the additive manufactured nickel-base superalloy. However, depending on the additive manufacturing parameters, the predominant texture components can be different, as pointed out by Ghorbanpour *et al.*^[49] in the functionally graded L-PBF Inconel 718. In this research the crystallographic texture of the sample performed with 250 W laser power showed a weak Goss texture component, but samples produced with higher laser power had strong cube textures. In our investigations, the participation of the particular texture components also depended on location in the sample (in the bottom, middle or top).

For comparison, in the stress-relief annealed sample the predominant texture component is not identified, which suggests that the sample could be partially recrystallized. The texture maxima are placed between to the ideal positions of Cube, Brass, Goss and Copper texture components. However, clear differences in texture may result not only from the heat treatment conditions, but also many process parameters. The issues of recrystallization and its role in the evolution of the Inconel 625 L-PBF microstructure are the subject of further research.

B. Cellular Substructure

The cellular substructure, typical for L-PBF alloys, was observed in the as-built sample, as was preserved in the stress-relieved condition (Figure 4). SEM analysis in the xz plane showed no distinct differences in the cellular substructure at the bottom, middle and top of the samples. Exemplary SEM images are given in supplementary Figure S-2. Therefore, quantitative analysis of cells in the vertical and horizontal planes was performed using specimens prepared from the region in the middle of the sample height, as a representative.

Figures $\hat{4}(a)$, (b) and (g), (h) shows the SEM images of intercellular areas in the xz plane and the xy planes of

as-built and stress-relieved samples, and their binary images after the segmentation procedure are given in Figures 4(d), (e) and (j), (k).

In vertical and horizontal planes of both sample variants, the cells inside grains exhibited differences in size and morphology according to the non-equilibrium solidification conditions in the L-PBF process (Figures 4(a), (b) and (g), (h)). Depending on the cooling rate and the thermal gradient, the equiaxed, elongated and/or columnar cells as well dendrites with short secondary arms were formed.^[50] The epitaxial growth of cells over melt pool boundaries was also observed. The results of the measurements of cells and intercellular areas are given in Table II.

Both the maximum and mean cell lengths are larger in the xz than the xy plane. Hence, the elongation factor CAR shows a similar dependence. In as-built Inconel 625, the mean CAR in xz plane is equal to 2.01, while the maximum reaches 12.00, which indicates a significant elongation of cellular structures. Meanwhile, in the xy plane, the cells are less elongated, as evidenced by the mean CAR equal to 1.74, and the maximum value was equal to 6.76 μ m.

On the other hand, in the stress-relief annealed sample the mean value of CAR in the xz plane is equal to 3.45, whereas the maximum reaches 8.55. The direction of the elongated cells shows the tendency of orientation towards the BD. In turn, in the xy plane the mean and maximum CAR values are equal to 1.81 and 4.47, respectively. Similar results were reported by Javidrad *et al.*^[51]. As in the case of the grain growth, the direction of the elongation of cells is affected by the heat flux direction during L-PBF manufacturing and the preferred crystallographic direction of crystal growth. Our results indicate that the elongated grains contain cell structures with columnar shape with a large length-towidth ratio.

The measurements of the area fraction of the intercellular areas A_A in the as-built sample revealed that it is almost equal in both planes and amounts to 32.74 pct in the xz plane and 30.51 pct in the xy plane. Meanwhile, the analysis of stress-relief annealed sample showed that in the xz plane A_A is equal to 30.6 pct, while in the xy plane it is higher and amounts to 42.5 pct. It can be related to the columnar shape of cells, which exhibit the tendency for elongation in the built direction, or occasionally change the direction according to the local thermal gradient. Depending on the intersection plane, cells can show the shape of small equiaxed ones; hence, we observe differences in the volume fraction of intercellular areas.

C. Precipitates

EDS microanalysis revealed that the intercellular areas are enriched in Nb and Mo (Figures 4(c), (f) and (i), (l)), which is in agreement with literature data.^[24–27] Figures 5(a) through (d) shows that intercellular areas are the preferential sites of precipitation of secondary phases in both as-built and stress-relieved Inconel 625. The precipitates are seen as bright particles. The particle size distribution is shown in Figures 5(e) and (f).



Fig. 4—Cellular substructure of the as-built and stress relieved L-PBF Inconel 625 observed in xz and xy planes: (a, b) and (g, h) SEM images, (d, e) and (j, k) binary images after segmentation as well as (c, f) and (i, l) EDS spectra acquired in intercellular and cellular areas at points marked respectively as 1 and 2 in (a) and (h).

Summary of the results of quantitative microstructural analysis of precipitates performed using SEM SE images is given in Table III.

In the as-built sample, the mean diameter \overline{D} is similar in both planes, equal to 110 nm for the xz plane and 100 nm for the xy plane. The highest frequency of particles has a diameter in the range of 100 to 150 nm. The number density N_A of the precipitates is slightly higher for xy plane, while the area fraction A_A is very similar for both planes.

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		xz Plane	xy Plane
	As-Built		,
Parameters of Cells			
Cell Length E _{2b} [µm]	Min	0.08	0.08
	Max	3.86	3.73
	Mean \pm SD	0.76 ± 0.52	0.60 ± 0.37
	Variation Coefficient [pct]	68.44	61.52
Cell Aspect Ratio CAR [–]	Min	1.00	1.00
	Max	12.00	6.76
	Mean \pm SD	2.01 ± 0.93	1.74 ± 0.62
A mar Transform of the Internet II from America A. [mat]	Variation Coefficient [pct]	46.51	35.79
Area Fraction of the Intercellular Areas $A_{\rm A}$ [pct]		32.74	30.51
Stre	ess-Relief Annealed		
Parameters of Cells			
Cell Length E _{2b} [µm]	Min	0.02	0.01
	Max	3.77	2.43
	Mean \pm SD	1.37 ± 0.66	0.71 ± 0.42
~ /	Variation Coefficient [pct]	48.50	59.11
Cell Aspect Ratio CAR [–]	Min	1.00	1.05
	Max	8.55	4.47
	Mean \pm SD	3.45 ± 1.20	1.81 ± 0.69
Area Erection of the Intercellular Areas 4 [not]	variation Coefficient [pct]	34.80	37.96 42.5
Area Fraction of the intercentular Areas $A_{\rm A}$ [pct]		30.0	42.3

In turn, in the stress-relief annealed sample, the size distributions tend to be flatter and more spread out than in the as-built one (Figures 5(e), (f)). The highest frequency of particles has a diameter in the range of 150 to 250 nm, and the mean diameter is equal to 210 and 200 nm, respectively, for xz and xy planes. The results show that the particle size parameters have higher values than in the previous sample. However, because the samples were printed using different machines, it cannot be clearly stated that these differences are solely due to annealing.

The number density reaches similar values for both planes, whereas the area fraction for the xz plane is higher than for the xy plane. Compared with the as-built sample, in the stress-relieved one, the number density is smaller, while the area fraction of the precipitates on the similar level.

Our results show that regardless of the processing conditions, in both the as-built and stress-relieved samples the size of precipitates is in submicrometer scale, and their number density is similar in the vertical and horizontal planes. This means that despite the morphological and crystallographic texture of grains, and thus the anisotropy of the cellular substructure, the particle distribution is uniform throughout the volume of the additively manufactured L-PBF Inconel 625. As presented above, our analysis shows that the growth nature of the grains governs the morphology of the cellular substructure and the arrangement of the intercellular boundaries, which are uniformly decorated with precipitates. The literature data show that the precipitation and growth of precipitates are dependent not only on the annealing heat treatment. Even if the samples are printed in similar L-PBF processing conditions and heat treated at the same conditions as in the present study,^[23] pronounced differences in the cellular substructure and secondary phase precipitates can occur. Therefore, based on the above results, it can be suspected that in the examined stress-relieved sample heat treatment has no effect on the dissolution or growth of the precipitates.

More detailed studies of particles performed with TEM and STEM allowed to observe also smaller particles that could not be seen in SEM. TEM images of as-built and stress-relief annealed samples shown in Figures 6(a) and (b) present the fine precipitates with irregular or globular shapes and size up to 100 nm located at the cell boundaries, as well as randomly distributed spherical or hexagonal nanoparticles. Particles composed of several precipitates are also present. Apart from particles, dislocations inside cells and forming splits at cell boundaries are present, as seen in STEM-HAADF image in Figure 6(c). Two variants of the particles were distinguished using STEM imaging in HAADF mode and EDS linescans (Figures 6(d) and (e)). Particles characterized by brighter contrast are niobium and molybdenum rich precipitates. Meanwhile, the dark contrast of particles indicates that they contain

Table III. Results of Quantitative M	licrostructural Analysis	of Precipitates in 1	L-PBF Inconel 625
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Parameter	xz Plane	xy Plane
As-Built		
Total analyzed Area A $[\mu m^2]$	1240.29	847.30
Mean Area $\overline{A_i} \left[\mu m^2 \right]$	0.01	0.01
Area Fraction A _A [pct]	4.47	4.33
Number Density $N_A \left[\mu m^{-2}\right]$	3.93	4.47
Diameter D Range [nm]	36–434	36–270
Mean Diameter $\overline{D} \pm SD$ [nm]	110 ± 40	100 ± 20
Median Diameter D50 [nm]	100	100
Variation Coefficient [pct]	36.04	22.49
Stress-Relief Annealed		
Total Analyzed Area A $[\mu m^2]$	1223.85	824.69
Mean Area $\overline{A_i} \left[\mu m^2 \right]$	0.04	0.03
Area Fraction A _A [pct]	4.28	3.60
Number Density $N_A [\mu m^{-2}]$	1.18	1.06
Diameter D Range [nm]	10-440	30–390
Mean Diameter $\overline{D} \pm SD$ [nm]	210 ± 60	200 ± 50
Median Diameter D50 [nm]	200	200
Variation Coefficient [pct]	29.72	52.71

light elements. EDS microanalysis confirmed that they are enriched in aluminum and oxygen and thus are oxide inclusions. This result is in line with the findings of Hyer *et al.*,^[52] who also observed similar precipitates in L-PBF Inconel 625.

Phase identification performed by means of selected area electron diffraction combined with EDS microanalysis revealed that the particles located at cell boundaries are the Laves phase, enriched up to 15 pct Mo and 12 pct Nb, and the MC precipitates containing up to 40 pct Nb (Figures 6(f) through (h)). The EDS spectra of the exemplary particles and the γ matrix are given in supplementary Figure S-3. The Laves phase had an irregular shape and frequently appeared in the form of several merged particles with the size from 30 to 250 nm. Despite the observed differences in the Nb content between the adjacent Laves phase particles (Figure 6(e)), no precipitates consisting of two phases were detected. In TEM diffraction contrast, the thin platelets inside Laves particles are visible, and the diffraction pattern contains smeared spots perpendicular to the platelets (Figure 6(f)). This is the characteristic feature of the Laves phase related to the presence of planar defects, which are visible in the HRTEM image in Figure 6(g). The MC carbides exhibited globular morphology and size from 20 to 80 nm (Figure 6(h)). The result of the diffraction analysis is in agreement with literature data, which states that in superalloys Nb segregates strongly during solidification to the liquid phase and that the Nb-rich MC carbides and the Laves phase precipitates can be formed in eutectic reactions.^[53-55] Niobium-rich carbide precipitates were also observed in the intercellular regions of L-PBF Inconel 625 by Marchese et al.^[26]

These results differ from those reported for as-solidified welds in Inconel 625, in which besides the Nb-rich MC carbides and the Laves phase, Ti-rich particles, such as TiN or TiNbC, are also present.^[31,32] The results of quantitative analysis of particles performed using TEM images are presented in Figures 7(a), (f) and Table IV. In both the as-built and stress-relief annealed samples, the mean diameter values are slightly larger for the xy than xz plane. Meanwhile, the particle number density is larger for the xz plane. This may be related to the crystallographic relationships between the precipitates and the γ matrix, leading to the preferential growth with the orientation close to the preferred orientation of grains in the vertical plane. Further studies are necessary to confirm this assumption.

The great advantage of manufacturing Inconel 625 by L-PBF is that precipitates of secondary phases are on a submicrometer scale, thus being much smaller compared to similar fusion processes such as welding.^[29,30]

The sphericity parameter of particles observed in TEM images was also determined. Figures 7(b), (c) and (g), (h) shows the graphs illustrating the values of sphericity parameter versus equivalent diameter and the area of particles. In addition, for a better visualization of the image segmentation, TEM images (Figures 7(d), (e) and (i), (j)) are included along with their equals of binary images of the analyzed particles. It was revealed that the values in the range of 0.8 to 1.0 characterizing almost spherical particles were exhibited by most of the precipitates with diameters < 40 nm in both the as-built and stress-relief annealed samples. Microstructural analysis showed that mostly MC carbides and oxide inclusions are of this size.

Furthermore, histograms combining the precipitate size distributions obtained based on SEM SE and TEM images are presented in Figures 8(a) through (d). It was clearly revealed that the particles in as-built and stress-relieved microstructure exhibited bimodal size distribution. The largest number of precipitates observed on TEM images in both conditions has



Fig. 5—SEM images of the cellular substructure with exemplary precipitates marked: (a, b) as-built and (c, d) stress-relief annealed sample, as well as (e, f) size distributions of precipitates observed in SEM SE images in both xz and xy planes.



Fig. 6—Precipitates and oxide inclusions in the L-PBF Inconel 625: (*a*, *b*) TEM images of as-built and stress-relief annealed specimens, (*c*) STEM-HAADF image of the dislocation substructure, (*d*, *e*) STEM-HAADF image and the corresponding EDS linescan across the marked line, (*f*, *g*) TEM, SAED and HRTEM image of the Laves phase and (*h*) TEM image and SAED pattern of the NbC carbide.

diameters in the range 40 to 60 nm for the *xz* plane and 60 to 80 nm for the *xy* plane.

The larger particles were identified as the Laves phase. Literature reports that the occurrence of the Laves phase particles in Inconel 625 is detrimental, when they are coarse, *e.g.*, while formed during solidification of welds.^[28-30] Such large particles are potential fracture initiation sites and can therefore deteriorate the mechanical properties. In additively manufactured nickel-based superalloys Inconel 625^[56,57] and Inconel 718,^[58] Laves phase is considered to be harmful when the particles are of submicrometer scale. However, it has been ascertained that the small and granular Laves phase particles have positive effect on the high-temperature mechanical properties of Inconel 718.^[59] Nevertheless, the size and distribution of the precipitates in L-PBF manufactured of Inconel 625 are significantly dependent on the process parameters. Although detailed printing conditions are not always given, the published data show that the use of different L-PBF machines and printing parameters, and thus different volumetric energy density, can lead to both the absence^[14] and occurrence of the small Laves phase precipitates.^[52]

In addition to the parameters related to the machine, the chemical composition of the powder may have a significant impact on the occurrence of the Laves phase. According to the fundamental study of Floreen *et al.*,^[60] precipitation of the Laves phase in Inconel 625 depends on the Fe content. A low concentration of Fe reduces the temperature range of solidification and thus minimizes the segregation and hinders the precipitation of the Laves phase.

D. Oxide Inclusions

SEM BSE images of non-etched polished sections exhibit compositional contrast; thus, microstructural features containing heavier elements are brighter, while the areas with lighter elements are darker. Using this kind of imaging allowed to observe fine oxide inclusions with dark contrast and spherical or hemispherical shape, which are evenly distributed in the L-PBF Inconel 625 in both as-built and stress-relieved condition (Figures 9(a) through (d)). It seems that they are exogenous inclusions possibly present already in the powder used for additive manufacturing. There are many reports in the literature about the presence of oxide inclusions enriched with Si and Mn in steels produced by AM methods.^[34,36–38,61] Oxide inclusions were present throughout the material, inside the grains, along the grain boundaries as well as the cell boundaries.^[37] Our results confirm that oxide inclusions are also present in L-PBF Inconel 625. Using SEM BSE images, measurements of oxide particles were performed. The results are given in Table V. The size distributions are shown in Figures 9(e) and (f).

Inconel 625 shows the same values for the oxides in both planes in the as-built conditions. The mean diameter \overline{D} of the oxides was 90 nm for both planes, and the area fraction A_A of the oxides is < 0.2 pct.

Under stress-relief annealing conditions for both xzand xy planes, the mean diameter \overline{D} of oxide inclusions is the same and equal to 100 nm. However, the area fraction A_A and number density N_A of inclusions in the xz plane are larger than in the xy plane and equal to 0.84 and 0.52 pct, respectively. The differences in the values of parameters describing oxide inclusions between the horizontal and vertical sections are small, which proves the homogeneity of the particle distribution in the whole volume of the additively manufactured sample.

The recent literature provides several reports on the presence of non-metallic inclusions in additively manufactured metal alloys. However, the references are fragmentary and relate mainly to stainless steel. Lou et al.^[36] reported that the sizes of the non-metallic inclusions in stainless steel were in the range of 50 nm to 1 μ m and that the mean size of the inclusions was 300 nm. Hemispherical and spherical particles rich in Si, Mn and Mo were observed.^[36] Bajaj *et al.*^[38] examined very small spherical impurities with diameters < 1 nm containing N and H as well as oxide inclusions based on silicon, magnesium, iron and titanium with diameters ranging from a few nanometers to even 100 μ m.^[38] It was determined that coarse oxides are created when the AM steel is processed using a low laser power and slow scanning speed. Deng et al.[37] showed that the oxides formed on the surface of the powder have little effect on the formation of nanoscale oxide inclusions in additive processes, while the formation of fine oxide inclusions is due to the high cooling rate of melt pools. Additionally, the smaller size of the inclusions indicated that most of the nano-scale oxide inclusions were formed by dissolving and re-precipitation of the oxides in the manufacturing process of the 3D printed parts. Although manufacturers try to purify the inert gas during the L-PBF process, it cannot be completely eliminated from the working environment. In the literature, the problem of oxygen absorption in the L-PBF process,^[62,63] as well as the inclusion of foreign oxygen into the elements in the L-PBF process^[37,64–66] by the formation of oxides on the surface of the molten pool and on the particles of spatters, has been discussed. Iams et al.^[67] have found that high levels of oxygen in the super-duplex stainless steel powder promote the formation of oxide inclusions in the AM process. Small inclusions with an average equivalent circular diameter equal to 476 nm, rich in Mn, Si and O, were observed in the microstructure. It has been reported in the literature that high levels of oxygen in the supplied stainless steel powder favor the formation of oxide inclusions in AM materials. Moreover, at slower cooling rates corresponding to AM processes, stable oxide spinels, metastable phases and amorphous inclusions were obtained.^[67] Gasper et al.^[63]



Fig. 7—(a, f) Precipitates size distributions for both xz and xy planes and the distributions of the sphericity parameter of particles: (b, g) xz plane, (c, h) xy plane, (d, e) and (i, j) TEM images and the corresponding binary images of analyzed particles.

in their study revealed that the oxides in Inconel 718 produced by L-PBF come from the oxidation of the molten pool surface.^[63] Additionally, contamination of the metal powder with moisture resulting from the storage and transport of the powder causes an increase in the number of oxide inclusions.^[37] There are several theories explaining the origin of oxygen in 3D printed parts. These are the contamination of the powder with oxygen during the powder production, the oxidation of the powder during storage and transport, and the occurrence of oxygen in the L-PBF process as the process is performed in an argon atmosphere, not a vacuum. The excess of oxygen in the steel can react with

chemical elements exhibiting a high affinity for oxygen and form oxide inclusions.^[40,68] The presence of oxide inclusions reduces the strength of the material, as oxides are the sites of initiation and microcracking.^[36] Given that oxygen cannot be eliminated from L-PBF production, the examination of oxide inclusions is a topical research issue. Our research has revealed that the oxide inclusions in L-PBF Inconel 625 can be easily detected using SEM BSE imaging. The results of the quantitative analysis of oxides show that due to their homogenous distribution, nanometric size and volume fraction < 1 pct, they can act as dispersion strengthening particles.

Table IV.	Results of TEM Microstructural	Analysis of Particles	of L-PBF Manufactured Inconel 625
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Parameter Value	xz Plane	xy Plane
As-Built		
Total Analyzed Area A $[\mu m^2]$	51.05	54.93
Mean Area $\overline{A_i} [\mu m^2]$	$4 \cdot 10^{-3}$	$5 \cdot 10^{-3}$
Area Fraction A_A [pct]	2.58	2.25
Number Density $N_V [\mu m^{-3}]$	63.42	41.54
Diameter D Range [nm]	15–232	16–211
Mean Diameter $\overline{D} \pm SD$ [nm]	61 ± 31	71 ± 32
Median Diameter D50 [nm]	56	67
Variation Coefficient [pct]	50.11	44.46
Stress-Relief Annealed		
Total Analyzed Area A $[\mu m^2]$	44.70	30.22
Mean Area $\overline{A_i} [\mu m^2]$	$2 \cdot 10^{-3}$	$5 \cdot 10^{-3}$
Area Fraction A_A [pct]	2.70	4.02
Number Density $N_V [\mu m^{-3}]$	104.25	90.66
Diameter D Range [nm]	10–137	16–231
Mean Diameter $\overline{D} \pm SD$ [nm]	50 ± 22	70 ± 37
Median Diameter D50 [nm]	47	62
Variation Coefficient [pct]	43.45	52.71



Fig. 8—Precipitates size distribution in the as-built and stress-relieved Inconel 625 manufactured by the L-PBF method obtained for SEM SE and TEM images for both (a, c) xz plane and (b, d) xy planes.



Fig. 9—Microstructure of L-PBF manufactured Inconel 625 observed on SEM BSE images of non-etched polished sections in: (a, b) as-built and (c, d) stress-relief annealed. The fine spherical inclusions with dark contrast are visible; (e, f) size distributions of oxide inclusions observed in SEM BSE images for both xz plane and xy plane; (e) as-built and (f) stress-relief annealed.

Table V.	Results of SEM BSE Microstructural	Analysis of Oxi	ide Inclusions in L-PB	F Manufactured Inconel 625

Parameter Value	xz Plane	xy Plane
As-Built		
Total Analyzed Area A $[\mu m^2]$	1091.15	1087.20
Mean Area $\overline{A_i} \left[\mu m^2 \right]$	0.01	0.01
Area Fraction A _A [pct]	0.15	0.18
Number Density $N_A \ [\mu m^{-2}]$	0.25	0.29
Diameter D Range [nm]	50-160	50-170
Mean Diameter $\overline{D} \pm SD$ [nm]	90 ± 20	90 ± 20
Median Diameter D50 [nm]	80	80
Variation Coefficient [pct]	19.36	18.69
Stress-Relief Annealed		
Total Analyzed Area A $[\mu m^2]$	1100.33	1103.07
Mean Area $\overline{A_i} [\mu \text{m}^2]$	0.01	0.01
Area Fraction A _A [pct]	0.84	0.52
Number Density $N_A [\mu \text{m}^{-2}]$	0.95	0.53
Diameter D Range [nm]	30–300	40–220
Mean Diameter $\overline{D} \pm SD$ [nm]	100 ± 30	100 ± 30
Median Diameter D50 [nm]	90	110
Variation Coefficient [pct]	29.86	28.83

IV. SUMMARY AND CONCLUSIONS

In this paper we presented and discussed the results of the microstructural characterization of Inconel 625 superalloy additively manufactured by the L-PBF process focused on the quantitative analysis of precipitates and oxide inclusions. The data reported in the literature show the growing importance of the quantitative characterization of additively manufactured superalloys for the future development of this process.

Therefore, our study is focused on the quantification of precipitates and oxide inclusions in as-built and stress-relief annealed samples in the planes parallel and perpendicular to the build direction and correlation of the results with multiscale characterization of grain structure and cellular substructure.

The results and discussion of the quantitative microstructural analysis provided herein complete the missing information on the distribution of particles and could be used to elaborate the geometrical model of its microstructure and to understand the process-structure-property relationship in L-PBF Inconel 625.

The following conclusions may be drawn from this study:

1. The microstructure of the as-built sample of L-PBF Inconel 625 is characterized by the columnar grains exhibiting a distinct tendency to elongating along the BD or at a slight angle with respect to the BD. A Cube ($\{001\}\langle 100\rangle$) texture component is predominant, but its contribution depends on location in the sample (the bottom, middle or top regions). The texture components close to Brass, Goss and Copper types are also visible. The texture intensity increases with growing distance from the build platform.

In the sample subjected to stress-relief annealing, the shape of the grains is varied as equiaxed, irregular and columnar. The maximum texture intensity is weaker and the distinct predominant texture components are not distinguished in all examined locations along the BD.

- 2. Accordingly, the grain morphology is accompanied by the morphology of cellular substructure. The intersections of elongated columns in the horizontal plane show small equiaxial cells and thus the greater fraction of intercellular areas. TEM and STEM analysis revealed that the intercellular areas are the preferential sites of precipitation of the Laves phase and NbC carbides as a result of the solute redistribution on the front of the solid-liquid interface and thus the microsegregation.
- 3. Regardless of the processing conditions and heat treatment (stress relief annealing) of L-PBF Inconel 625, the size of precipitates is in submicrometer scale, and their number density is similar in the vertical and horizontal planes. Despite the morphological and crystallographic texture of grains, and thus the anisotropy of the cellular substructure, the particle distribution is uniform throughout the volume of the additively manufactured L-PBF Inconel 625.
- 4. TEM, STEM-HAADF and EDS analysis allowed to identify the oxide inclusions as aluminum oxides. It is demonstrated that SEM-BSE imaging of non-etched polished section is appropriate for unambiguous analysis of oxide inclusions in L-PBF Inconel 625. Quantitative analysis revealed that oxide inclusions with the mean diameter around 100 nm and the area fraction < 1 pct are randomly distributed in the additively manufactured Inconel 625 superalloy.

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CONFLICT OF INTEREST

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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SUPPLEMENTARY INFORMATION

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