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Influence of feature size and shape on corrosion of 316L lattice structures fabricated by laser powder bed fusion

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ABSTRACT

Laser powder bed fusion (LPBF) has become an established method for manufacturing end-use metal components. Exploiting the geometric freedom of additive manufacturing (AM) offers broad possibilities for part optimization and enables performance enhancements across industry sectors. However, part shape and feature size have been found to locally affect residual stresses, melt pool cooling rates, microstructure, and thus the mechanical properties of components. Even though the mesoscale structure can locally induce microstructural changes, there are no prior studies on how it influences corrosion. Using AM-produced, optimized parts in critical applications necessitates a better understanding of their long-term performance. In this study, lattice structures were used to probe the influence of feature size and shape on corrosion susceptibility and its spatial localization.

The susceptibility of submillimeter LPBF-fabricated 316L stainless steel lattice structures to corrosion was investigated by conducting a 21-day immersion corrosion test in an aqueous 3.5 wt% NaCl solution. Schoen gyroid and Schwarz diamond triply periodic minimal surface lattices were manufactured with three unit cell sizes and wall thicknesses (0.867, 0.515, and 0.323 mm). The nominal surface and cross-sectional areas were the same for the two geometries. X-ray microcomputed tomography (microCT) scans before and after the corrosion test were compared for volumetric losses. In addition, the mechanical properties and microstructure of the samples were evaluated. As part of the study, a workflow to register, index, and analyze volumetric changes of consecutive microCT image stacks was developed. The reported method is applicable to any time-lapse studies with microCT.

Three out of five of the 0.323 mm wall thickness lattices displayed visually aggressive pitting. Based on the microcomputed tomography data, the mass losses were localized either in the entrapped powder particles or partially melted surface globules. Corrosion did not occur in the dense base material. The total mass losses ranged from 8 to 19 mg. Despite visual indications to support a higher corrosion susceptibility for the smallest lattice sizes, the mass loss values did not confirm this conclusion. The tensile test results did not provide any clear indications of latent corrosion effects on mechanical properties.

1. Introduction

In the production of metal components using additive manufacturing (AM), laser powder bed fusion (LPBF) has become one of the most established methods. Owing to its outstanding mechanical properties, ductility, and high corrosion resistance, 316L stainless steel is a widely used alloy. In many studies, the corrosion resistance of the LPBF-fabricated 316L variant is found to be better than that of wrought materials [1]. The design freedom of AM enables product designers to explore the uncharted territories of complex part geometries and unlock designs that can outperform their preceding conventional solutions. The use of a lattice structure is one of the more structured design tools that benefit from the AM geometric complexity [2,3]. Lattices are 3D repeating size-scalable units [2] categorized as strut-based skeletal lattices or surface lattices, depending on the type of their connecting elements. Complete local control over unit geometry, wall thickness, volume fraction, and surface area makes them useful, for example, in part weight reduction and stiffness control. As part of the surface lattice category, triply periodic minimal surfaces (TMPS) are a case of zero-mean curvature mathematical isosurfaces created via

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trigonometric functions [4–6]. Owing to their continuous surfaces, TPMS lattices have a higher surface area to volume ratio. This attribute can be useful for surface area–dependent applications such as heat transfer [7]. Hence, some possibilities for increasing the part performance are dependent on the finesse of the features. In a few studies on 316L, the reduction in part wall thickness has been found to alter the part microstructure and hence the part mechanical properties [8–11]. It appears that a specific feature size limit exists for LPBF-produced 316L below which standard mechanical properties can no longer be expected.

In a typical LPBF system, the laser spot diameter is approximately 100 µm and the layer thickness ranges between 20 and 100 µm. The manufacturing process is characterized by fast local melting and cooling of material. Consecutive layers contribute to the remelting and solidification of previous melt tracks, rendering the thermal history of a volumetric material unit complicated. The temperature range within and near the vicinity of the laser focal spot can vary from 400 to 2500 K, where metal can exist as a solid, liquid, or gas [12]. In addition to evaporative and radiative cooling, laser heat is conducted away from the melt pool via the part itself, support structures, build plate, and to a lesser extent, via the loose powder surrounding the part. Consequently, the thermal history is geometry dependent and can locally affect the part microstructure, composition, mechanical properties, and residual stresses. This geometry dependence becomes more distinct as the part feature size is reduced to the submillimeter scale. Smaller geometries limit the volume of the heat-conducting part around the moving melt pool. With finer features, laser scanning also transitions from hatch-dominant scanning to contour-dominant scanning.

This study explores the indirect influences of feature size and shape on the corrosion susceptibility of LPBF-manufactured 316L parts. The microstructural changes in the small parts and overall rough surface finish of LPBF-fabricated parts raise several research questions: 1) How would fine features that approach the lower resolution limit respond to corrosive environments? 2) Does the feature size or shape affect the corrosion susceptibility of 316L, a material generally known for its high corrosion resistance? 3) Can optimized parts with submillimeter features be safely used in industrial applications and demanding environments? To the best of the authors’ knowledge, none of these questions have been explored in the literature. A broader understanding of the corrosion of complex AM-fabricated structures would benefit applications such as medical implants, nuclear components, and fuel cells [1]. Understanding the long-term performance of a part is imperative for its use, especially in safety-critical applications.

Owing to its high chromium (17–19 wt%) and nickel (13–15 wt%) contents and the added molybdenum (2.25–3 wt%), 316L is a low-carbon austenitic stainless steel that is highly resistant to general and localized corrosion. In the presence of oxygen, stainless steel develops a passive chromium oxide layer that protects the metal surface [13]. The electrochemical process of localized corrosion (pitting and crevice corrosion) is influenced by the formation of an aggressive anolyte between the chloride ions and metal cations. Pitting corrosion can be safely used in industrial applications and demanding environments. Moreover, some possibilities for increasing the part perforation cover is no longer required to sustain the reaction [14]. When the perforation cover develops into a self-sustaining stable growth pit, the cover is no longer required to sustain the reaction [14].

The corrosion performance of a manufactured part primarily depends on the microstructural features across the entire part volume, particularly its surfaces [16]. Process-related microstructural phenomena, defects, and anisotropic properties can influence the part corrosion resistance [1, 13, 17, 18]. The anisotropic microstructure [19], grain size [20], dislocation structure [17], track and layer boundaries [21, 22], residual stresses [23], porosity [13], inclusion size [1], and surface roughness [24] have all been studied in the context of AM-fabricated 316L corrosion. However, comparative studies between LPBF-manufactured 316L and conventional 316L are sometimes conflicting. The defects caused by the LPBF process, such as cracks, porosity, and secondary phases, can explain some discrepancies. When the process defects are eliminated, the corrosion performance of AM-fabricated 316L is found to be better than that of wrought materials [25]. The annihilation of MnS inclusions due to rapid solidification in LPBF-produced 316L has been proposed to improve the corrosion performance [26]. Instead, oxide inclusions may influence the AM-fabricated 316L pit initiation [25]. Porosity and high surface roughness are common features of LPBF-fabricated parts, which can influence corrosion. Porosity can lead to local acidification and increase the pitting susceptibility of the material matrix. Both the pore size and morphology have an influential role [22].

When the AM complexity is exploited to the limits, the design feature size becomes finer and approaches the melt pool diameter. Consequently, the material properties begin to differ. As mentioned, the size dependence of the mechanical properties, microstructure, and texture of LPBF-produced parts has been discussed in only a few studies to date [8–11]. Britt et al. [8] studied a singular lattice node point and struts with a planar X-shaped sample geometry. As features shrink, the local material cooling rate decreases, and the cooling gradient affects the microstructure and size of sub-grain cells. Lower hardness due to larger cell diameters and lower dislocation density were reported for a strut thickness of 0.5 mm compared to 1 mm and 1.5 mm. Wang et al. [10] identified a distinct increase in spacing for the sub-grain cell structure in fine 0.5 mm and 0.25 mm thick struts. In these feature sizes, a few laser passes already cover the manufacturable features. The surface area to volume ratio increases, and the surface roughness becomes dominant compared to the dense base material [9]. With smaller feature sizes, the deviations in strut shape and diameter can become considerable and reduce the load-bearing cross-sectional area, as revealed by scanning electron microscopy [27] and microcomputed tomography (microCT) [28]. In fact, microCT has been found to be an especially well-suited evaluation method for complex parts produced using AM [29–32]. In addition to routine porosity and defect inspection, this method has been employed in assessing part geometric accuracy, surface characterization [33, 34], and corrosion evaluation [35].

In summary, the implications of complex AM designs have not been studied in the context of corrosion. It is currently poorly understood how a part mesoscale geometry can induce local microstructural changes. Fine, submillimeter-sized features, overall rough surfaces, and global or local changes in the microstructure could influence the corrosion susceptibility of LPBF-fabricated parts. It is important to study how the size and shape of features affect the LPBF process inducing local changes in part microstructure, material composition, and mechanical properties. In addition, these changes need to be studied experimentally on realistic components to understand their implications on component long-term performance. This study considers the latter aspect. The experiment involves an immersion corrosion test of three submillimeter-sized TPMS lattice structures. Lattice structures offer an ideal probe to study the influence of the mesoscale geometry on the microscale. In this study, the influence of feature size and geometry shape are studied on two levels. Weight loss measurements reveal the overall trends in corrosion susceptibility between the geometries. A microCT workflow was developed to validate results and assess the exact location of volumetric changes concerning the lattice unit cell shape.

2. Materials and methods

As an overview, 24 TPMS lattice structure tensile samples were manufactured using 316L produced by LPBF on an EOS M290 system. Half of the samples were subjected to a 21-day immersion corrosion test in a 3.5 wt% NaCl aqueous solution. The samples were CT scanned before and after the corrosion test. The data from the two scans were registered and compared for volumetric differences. The mechanical
properties of all samples were obtained via tensile testing, and the part microstructures were evaluated via metallographic examination. All relevant data for research reproducibility are made available through the Zenodo open data service [36].

2.1. Lattice design

This study intentionally explored the lower feature size range for 316L parts manufactured using the LPBF process (100 μm laser spot diameter) to determine the criticality of feature size and part shape on part corrosion susceptibility. The repeating lattice cells offer a convenient and controlled way of studying the assumed corrosion influences of feature size and shape. Each unit cell embodies multiple features of interest, including thin walls, downskin surfaces, and bridging. Every unit has a similar spatial surface roughness variation and cooling gradients during processing (excluding those near the build platform, where the cooling rate is faster). Hence, corrosion effects induced by the geometric shape repeat in the structure. From the shape perspective, each unit cell can be considered an individual sample. The diamond and gyroid TPMS structures were selected as they have been found to perform satisfactorily against other unit cell types (even with decreasing relative densities) [37] and can be printed without support structures.

Three submillimeter wall thicknesses that gradually approach the feature size limits of the current LPBF process were selected. The geometries were created using the implicit 3D modeling software nTopology (nTopology, New York, USA). An example of an open-source research-oriented option for lattice design is the ‘Flat Pack’ by Maskery et al. [38]. The test set consisted of three sizes (denoted hereinafter as S, M, and L) and two unit cell types, namely, the Schoen gyroid and Schwarz diamond. The surface and cross-sectional areas were adjusted to correspond as close as possible to the two cell types in each size category. The unit cell sizes of the gyroid samples were fixed. The central, non-graded wall thickness was adjusted for a 50% volume fraction. The cell sizes of the diamond lattices were then adjusted to match the surface area of the baseline gyroid structure. The relevant lattice parameters and metrics are listed in Table 1, and all lattice sections of the samples are shown in Fig. 1.

The graded lattice structure was embedded into a gauge section of a custom tensile test specimen. A relatively thick cross section (10 × 10 mm) was selected to fit multiples of lattice unit cells in all directions and lattice sizes. The main sample dimensions are presented in Fig. 1a. The central lattice wall thickness was kept constant within the length of 10 mm. A symmetrical grading on the length of 4 mm at both ends was added to avoid stress concentrations at the transition from the length of 10 mm. A symmetrical grading on the length of 18 mm, where most of the plastic deformation occurred, was used to calculate the engineering strain. The sample surfaces were prepared using standard mechanical polishing procedures for optical microscopy and microstructure evaluation. The microstructure reference sample was electrolytically polished in A2 electrolyte and externally etched in a 60% HNO₃ solution. The remaining samples were wet etched using a Beraha II color etchant. The grain size parameters, size dispersion, and grain size distribution were calculated with a MATLAB tool by Lehto et al. [40,41].

2.2. Additive manufacturing

The print job preparation was performed using the EOSPRINT software (version 2.6, EOS GmbH, Krailling, Germany) based on the contours exported from the nTopology software. The test samples were manufactured using an EOS M290 system installed with a Yb-fiber laser having a spot diameter of 100 μm and maximum power of 400 W. The 316L stainless steel powder was supplied by EOS Finland (Turku, Finland). The commercial, locked parameter set 316LSurfaceM291 1.10 and a layer thickness of 20 μm were utilized to simulate the industrial production of end-use parts. Argon was used as the inert process gas. For both platforms, two 20 × 20 × 10 mm reference parts were included for metallographic evaluation. Four samples per lattice and size category, two on each platform, were printed in a planar orientation and arranged randomly on the two platforms, as indicated in Fig. 2. The corresponding sample identifiers are provided in Table 2. One gyroid sample (Gyroid S 01 – 1) was machined into a cylindrical shape. A smaller sample diameter allowed a smaller voxel size in the CT scanning with the nanofocus X-ray tube. Excess powder was manually removed. Both platforms were subjected to a standard solution annealing heat treatment in a vacuum furnace. The temperature was increased 5–6 degrees per minute, held at 1030 °C for one hour, and parts were quenched with pressurized nitrogen. Finally, the samples were (wire) electric discharge machined (EDM) off the platform.

2.3. Mechanical properties and microstructure

Tensile tests were performed at room temperature using an MTS 810 50 kN material testing system (Eden Prairie, Minnesota) at a 1 mm/min test speed. The engineering stresses for all samples were calculated based on a cross-sectional average area of 50 mm². A sample graded length of 18 mm, where most of the plastic deformation occurred, was used to calculate the engineering strain. The sample surfaces were prepared using standard mechanical polishing procedures for optical microscopy and microstructure evaluation. The microstructure reference sample was electrotyically polished in A2 electrolyte and externally etched in a 60% HNO₃ solution. The remaining samples were wet etched using a Beraha II color etchant. The grain size parameters, size dispersion, and grain size distribution were calculated with a MATLAB tool by Lehto et al. [40,41].

2.4. Immersion corrosion testing

The test setup is based on an ASTM G31–72 standard “Laboratory Immersion Corrosion Testing of Metals”. The manuscript adheres to the standard recommendations for data reporting. The test is normally designed to study uniform corrosion instead of localized corrosion (ASTM G48 defines the pitting corrosion test in a more aggressive ferric chloride solution). However, the test solution (3.5 wt% NaCl) was selected based on the hypothesis that small, embedded lattices with enclosed features, overall poor surface roughness, large sample surface area, and microstructural changes due to thin walls in LPBF-fabricated 316L can promote pitting even in a milder NaCl environment.

A 3.5 wt% NaCl solution was prepared using chemically pure sodium chloride (Sigma-Aldrich) and distilled water. The initial mass of each sample was recorded as the average of three measurements on a precision scale. The ASTM G31 standard recommends a minimum of 0.40 mL of solution per square millimeter of the sample surface area, resulting in an average of 5 L per sample. Two 40 L corrosion tanks were built. There were six samples in corrosion tank 1 (Gyroid L 02 – 3, Gyroid M 02 – 2,

| Cell size xyx  | Central wall thickness | End wall thickness | Volume fraction | Central lattice, surface area | Average section area |
|----------------|------------------------|--------------------|-----------------|-------------------------------|----------------------|
| (mm)           | (mm)                   | (mm)               | (%)             | (mm²)                         | (mm²)                |
| Gyroid S       | 1.25                   | 0.323              | 0.431           | 0.505                         | 4626.86              | 50.81                |
| Diamond S      | 1.53                   | 0.323              | 0.431           | 0.491                         | 4626.19              | 49.48                |
| Gyroid M       | 2.09                   | 0.515              | 0.687           | 0.504                         | 2951.71              | 50.33                |
| Diamond M      | 2.47                   | 0.515              | 0.687           | 0.484                         | 2951.60              | 49.35                |
| Gyroid L       | 3.33                   | 0.867              | 1.153           | 0.508                         | 1882.22              | 50.47                |
| Diamond L      | 4.13                   | 0.867              | 1.153           | 0.496                         | 1881.86              | 49.31                |
Gyroid S 02 – 1, Diamond L 01 – 12, Diamond M 02 – 5, and Diamond S 02 – 4) and seven samples in tank 2 (Gyroid L 02 – 9, Gyroid M 02 – 8, Gyroid S 02 – 7, machined Gyroid S 01 – 1, Diamond L 02 – 6, Diamond M 01 – 11, and Diamond S 01 – 10). The samples were suspended in a corrosive medium with fishing lines. The containers were kept inside a room at a constant temperature of 20 ±2 °C and allowed to ventilate. Both tanks were occasionally stirred and aerated using a blender. The test duration was 21 days. After the corrosion test, all the samples were rinsed in water, ultrasonically cleaned in acetone, and air-dried. The mass of each sample was recorded again, and the ultrasonic cleaning cycle was repeated until a negligible mass difference was obtained between measurements. The corrosion rates were calculated in millimeters per year according to the ASTM G31–72 (Eq. 1):

\[
\text{Corrosion rate} = \frac{K \times W}{A \times T \times D}
\]  

where \(K\) is a constant 8.76 \(\times 10^4\), \(W\) is the mass loss in grams, \(A\) is the area in cm\(^2\), \(T\) is the time of exposure in hours, and \(D\) is material density.
2.5. MicroCT scanning

The microCT scans were obtained using a GE Phoenix v-tome|x s 240 (Wunstorf, Germany) industrial CT scanner. The samples were scanned twice (before and after immersion corrosion testing) with identical settings, as listed in Table 3. Three samples were stacked for each scan with a 240 kV microfocus X-ray tube and fastened directly into the machine chuck. The 180 kV nanofocus scan sample was machined cylindrically from the Gyroid S 01 – 1 geometry (Fig. 9a). The sample was fastened to the CT device chuck and centered using a goniometer.

During reconstruction, the images were corrected for ring artifact reduction and beam hardening (correction coefficient 6/10). The reconstruction output was 32-bit float to avoid automatic grayscale scaling, but the images were converted to 16-bit unsigned for ease of analysis. The resulting original image stacks are referred to as RAW. The stacks were binarized (BIN) by the 2-phase watershed segmentation recipe in ThermoFisher PerGeos 2020.2, using consistent parameters across different samples and scans.

2.6. Geometrical accuracy assessment

A point cloud and a mesh of the original implicit geometry were exported to the open-source software CloudCompare (Daniel Girardeau-Montaut, Grenoble, France) to compare the nominal 3D geometry with those of the as-printed samples. The CT data were processed using the open-source software ImageJ developed by the National Institute of Health (Bethesda, Maryland, USA) and the University of Wisconsin (Madison, Wisconsin, USA). The BIN data were exported as a surface point cloud from ImageJ to CloudCompare. A 3-point alignment of the nominal mesh and CT point cloud was followed by fine registration with scale correction. Once aligned, the geometries were compared with cloud-to-mesh signed distances.

2.7. CT data registration and analysis

A workflow to register and analyze the microCT stacks before and after corrosion was developed. For each comparison, the full RAW CT stacks (approximately 1000 slices) were manually clipped and roughly aligned in the Z direction. The ImageJ plugin Fijiyma (Romain Fernandez & Cedric Moisy, France) was used for fine registration on a reduced stack (approximately 50 slices) considering XYZ translations, rotations, and isotropic scaling [42]. The full RAW and BIN stacks were aligned with the resulting transformation matrix. The pixel value differences of each voxel location were calculated with ImageJ macros. A gray value comparison before and after the immersion corrosion test made it possible to localize volumetric changes and study their 3D morphology. All volume changes larger than 10 voxels and exceeding a gray value threshold of 15000 (16-bit unsigned values) were indexed using the ImageJ 3D Particle Analysis tool [43]. The 3D data including the indexed volumetric changes was visualized with the Drishti open-source software [44]. The influence of shape on corrosion susceptibility was studied by first sectioning all individual lattice unit cells from the microCT data. A 3D unit cell heatmap was constructed as a sum of changes (gray value threshold of 15000) across all unit cells to reveal any location-specific, statistical differences. All ImageJ macros are provided as part of the data package.

3. Results and discussion

3.1. Lattice structure characterisation

Poor surface finish and incomplete depowdering of some samples was expected. Fig. 3 displays optical micrographs of the manufactured structures. The laser hatch pattern is visible for the largest as-designed wall thickness sample (0.865 mm), whereas the smallest geometries (0.323 mm) are plotted using contour lines only. As shown in Fig. 3, the proportion of powder residue relative to the feature size increases as the unit cell size is reduced. Thinner structures inhibit efficient cooling. The solid and powder materials around the laser spot, including a few previous layers, are affected by heat. This resulted in a very rough surface with partially molten individual powder particles and larger globules.

Fig. 4 presents a representative CT slice for each sample size category. Most of the samples contain partially molten or entrapped loose powders within the lattice. Only two CT scanned samples, namely, Gyroid L 02 – 3 and Diamond L 02 – 6, are powder free. The percentages of entrapped powder in the L, M, and S lattices are estimated at 0–20%, 20–50%, and 70–90%, respectively. The fractured samples in the tensile test allowed some powder to be discharged from within the lattice. As depicted in Fig. 3, only a very thin powder layer is sintered on the sample surface. The rough surface topography and continuous funnel shape of TPMS structures can entirely obstruct the movement of loose powder in a relatively large cavity compared to the powder particle size.

The CT scans with the nanofocus tube offer a more detailed view with a voxel size of 3.5 μm. At this voxel size, the size and shape of the individual powder particles, detailed surface topography, and voids larger than approximately 10 μm can be distinguished. Fig. 5 displays a higher-resolution cropped slice of the Gyroid S 01 – 1 sample. The surface roughness is high and the surrounding loose powder is partially sintered and trapped within the structure. Deep cavities and cracks stretch up to hundreds of micrometers into the solid material, sometimes covering half of the wall thickness. The material structure is poor in terms of structural integrity and localized corrosion. Surface features and cracks act as stress concentration sites, while cavities enveloped by partially molten powder provide prospective sites for localized corrosion.

The local energy density, beam shape, and laser scanning modes can be further optimized to manufacture higher-quality thin-walled geometries. Pulsed laser scanning, instead of the continuous wave mode, is an option for customizing the energy input in some LPBF systems. It offers better control in manufacturing fine features [45–47]. Single-track experiments on 316L pulsed versus continuous wave lasers are provided in [46]. Customizing the process to achieve the finest surface roughness will ease part depowdering. Naturally, a smaller particle size and higher sphericity of the feedstock powder are beneficial.

Comparing the nominal with the as-printed geometries reveals information on their global 3D accuracy. Fig. 6 illustrates the color-coded signed distances in mm (top row) and distance histograms (bottom row). Red denotes positive, oversized geometries, whereas blue represents points with a negative value below the nominal surface. The main
6

contributor to the undersized features is the EDM wire-cut surface. The Gaussian mean in all histograms is positively shifted, indicating a tendency for oversized surfaces in the samples. The Gaussian mean values for the largest samples (Gyroid L 02 – 3 and Diamond L 02 – 6) are 0.021 and 0.026 mm, respectively. The shift is slightly increased by the rougher surface and entrapped powder in the Gyroid M 02 – 2 and Diamond M 02 – 5 samples (0.038 and 0.041 mm, respectively). Reliable 3D registration of the nominal and microCT scanned data of the S lattices was no longer possible because of the higher amount of entrapped powder inside the structure, hindering a proper mesh-to-point cloud 3D registration in the CloudCompare software.

3.2. Immersion corrosion tests in 3.5 wt% NaCl

One week after the 21-day immersion corrosion test, three out of five S lattices displayed visually discernible corrosion products pushed out and lingering at the bottom of the test tank (Diamond S 01–10 and

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Fig. 3. Surface topography and partially melted powder as seen on the L (top row) and S diamond lattices (bottom row). Pictures from left to right: top surface, side surface, and EDM wire cut surface. Scale bars: 500 µm.

Fig. 4. Representative CT slices displaying the amount of entrapped powder within the structures. a) Diamond L 02 – 6, b) Gyroid M 02 – 2, and c) Gyroid S 02 – 1. Scale bar for all images: 1 mm.

Fig. 5. An example slice of the Gyroid S 01 – 1 sample as seen in the nanofocus CT scan (3.5 µm voxel size). Scale bar: 500 µm.
machined Gyroid S 01 – 1 from tank 2 and Diamond S 02 – 4 from tank 1). The remaining two weeks of testing did not produce notable visual changes, suggesting that the existing corrosion sites were passivated and did not develop into stable growth pits. Notable surface staining caused by the corrosion deposits on the Diamond S 02 – 4 lattice can be seen in Fig. 7 (bottom rightmost) after the 21-day test. Optical microscopy images of the top, side, and bottom surfaces of Diamond S 02–4 in Fig. 8 exhibit a more detailed post-corrosion view. The machined Gyroid S 01 – 1 sample, shown in Fig. 9a, displays a similar discharge. However, duplicates with the same geometry are visually devoid of corrosion. The striking difference between the EDM cut surface (Fig. 7b) and the as-built top surface (Fig. 7a) is noteworthy.

The bottom EDM surface in Fig. 7b has a uniform brownish tint, suggesting surface corrosion. The EDM wire-cutting process is known to create a 5–80 µm white layer with microstructural and chemical changes compared to the base material [49]. The thin surface layer contains microcracks, craters, and chemical heterogeneities, rendering it less resistant to localized corrosion. Heat treatment of parts after EDM alleviates this problem. The cylindrical sample was machined, thus eliminating the influence of EDM. However, the sample was among the most (visually) corroded.

The initial hypothesis was that the effect of semi-closed and continuously curved internal surfaces together with entrapped powder and corrosion particles could create a more aggressive and dynamic corrosive environment deeper inside the lattice structure. The few aggressively corroded S lattices partly supported this hypothesis. Fig. 9a shows an overview and Fig. 9b a side surface close-up of the machined Gyroid S 01 – 1 sample after corrosion testing. However, as exhibited in Fig. 9c, the corrosion deposits are limited to a very shallow depth and do not indicate localized corrosion within the lattice.

The mass losses and corrosion rates of the individual samples after corrosion testing and ultrasonic cleaning are listed in Table 4. The mass losses range from 8 to 19 mg. The sample geometry (gyroid or diamond) and unit cell size do not influence the results. Before the ultrasonic wash cycles, the mass differences were within 5 mg. The smallest lattices had a slight trend of mass increase before the wash cycles, possibly because of corrosion deposits. After cleaning, the mass losses of the S lattices were lower than those of the M and L lattices. Either the corroded mass in these samples was negligible, or most of the corrosion deposits were not removed during sample cleaning. The measurements alone did not reveal whether the mass loss was caused by localized corrosion or by detached powder particles during ultrasonic cleaning. For the same
reason, the calculated corrosion rates and mass losses in Table 4 are only comparable for the Gyroid L 02 – 3 and Diamond L 02 – 6 samples, which were verified to be powder free in the CT scans. The daily mass loss per full sample surface area ranges from 0.0042 to 0.0114 mg/cm² per day.

A few immersion corrosion tests for polished samples of the LPBF-fabricated 316L in a more aggressive 5–6 wt% ferric chloride (FeCl₃) solution are provided for reference [23,50,51]. Sander et al. [23] measured mass losses ranging between 0.011 and 0.022 mg/cm², depending on the build orientation after 48 h of immersion in 6 wt% FeCl₃. Cruz et al. [51] measured mass losses ranging from 3.5 to 4.5 mg/cm² per day in a similar solution (up to 72 h) for as-built and three different heat treatment conditions. The accumulated mass loss of 316L reference samples immersed in 5.6 wt% FeCl₃ for 10 days in a study by Valente et al. [50] plateaued at approximately 1–1.5 mg/cm². These studies, including the current work, highlight the challenges of the corrosion mass-loss method when used alone. The stochastic nature of corrosion, accuracy of the precision scale, and employed rust removal procedure can have significant influences on the obtained results. Furthermore, complex structures such as TPMS lattices will introduce more variables that can complicate mass measurements: first, the entrapped powder and its possible detachment, and second, the appropriate cleaning of corrosion deposits within the complex structure.
Analyzing volumetric changes by CT scanning is an attempt to alleviate these uncertainties.

### 3.3. MicroCT data analysis

The aligned microCT scans before and after the corrosion test were compared for volumetric differences. The possible effects of corrosion can be visualized in the images as either mass losses (corroded or eroded material) or mass accumulation (iron oxide deposits or shifted powder). Hence, the volumes indexed using the 3D Particle Analysis tool are denoted “material losses” because their primary cause is not necessarily corrosion. Video 1 presents all the results as rotational videos with partial opacity through the BIN lattice surfaces. Fig. 10 depicts a few descriptive examples, where the indexed volume losses are highlighted in red and overlaid with the full 16-bit data. The losses throughout the data are localized either on the partially melted surface globules and poor surface roughness (Fig. 10c) or within the entrapped powder (Fig. 10d). In the second case, the material losses are generally observed near the surfaces of the entrapped powder volumes. Nevertheless, no clear instances of corrosion penetrating the dense 316L base material were found. The full dataset is available for download.

Table 5 lists the material loss volumes (3D particles) for each CT scan indicating their total count, average mass, total mass, and average bounding box dimensions. Most occurrences of material losses lie within 1 mm of the sample surface. The particles were small, with masses of approximately a few hundred micrograms. The bounding box width is the CT scan X direction, height is the Y direction, and depth denotes the Z direction. The bounding box dimensions do not reveal dominant shape preferences. Only the particle depth is slightly pronounced.

The variations in both the particle count and lost mass across the samples are considerable. The total mass losses do not agree with the measurements and are, at worst, an order of magnitude off. The entrapped powder is considered the largest contributor to the poor fit. Changes in the location of loose powder and accumulated corrosion deposits inside the lattices can distort the results. Samples free of entrapped powder (Diamond L 02 – 6 and Gyroid L 02 – 3) provided the correct magnitude.

The mass loss particle count of the S lattices is low, even though a few of the samples are among the most visually corroded. The small cell-sized S lattices having a rough surface topography can completely block the movement of the entrapped powder. In contrast, the M lattices allow some volumes of the entrapped powder to detach and move. The difference is evident in the scatter plots of each analyzed geometry in Fig. 11, where the point size is scaled based on the mass loss particle volume. The M lattices, and the only L lattice with entrapped powder (Diamond L 1 – 2) display a higher particle count and volume variation (Fig. 11c-f). Constellations of particles that seem to be aligned on the same plane, as shown in Fig. 11c, d, and f, can indicate minor alignment inconsistencies or scanning artifacts between the two microCT scans. An accurate mass loss analysis based on microCT would also consider the adhered iron oxide on sample surfaces. However, the size of individual particles is extremely small at 20–30 nm [52]. Distinguishing even larger aggregates requires the CT resolution to be in the nanometer range.

The influence of lattice unit cell shape on corrosion was studied statistically by sectioning the microCT data into individual unit cells. Volumetric losses of all cells were summed to generate a 3D heatmap. Fig. 12 presents the heatmap of the Diamond M 02 – 5 specimen as a summation of 63 unit cells. A higher intensity of losses (magenta) would indicate preferential sites for corrosion. A descriptive lattice surface is visualized in white. The losses have a slight tendency to locate near downskin surfaces where partially sintered and entrapped powder is more likely to occur. Due to the limitations of the experimental setup, the results are rather inconclusive. For the heatmap to provide useful information on the influence of shape on corrosion, the medium should be corrosive enough to penetrate the solid material.

The corrosion was limited to a shallow depth on the outer surfaces of a few S lattices. No corrosion sites were found deeper within the lattice. This raises the question of whether there are inconsistencies in the solution chloride concentration or whether the surface of the S lattices can promote a locally higher chloride ion concentration before an equilibrium is reached. Nevertheless, all corrosion sites were passivated during the 21-day test, indicating that the 3.5 wt% sodium chloride solution was not aggressive enough to sustain localized corrosion in the solution-annealed 316L manufactured using LPBF. As a reference, Laleh et al. [35] studied the 3D localized corrosion of solution-annealed 316L samples fabricated using LPBF in a 6 wt% ferric chloride solution. The fine 2.95 μm voxel size in microCT allowed a detailed characterization of the corrosion propagation. The lack-of-fusion pores in the polished samples were found to be susceptible to localized corrosion in ferric chloride, and the corrosion propagated via irregularly shaped, occluded pores extending inside the material.

### 3.4. Mechanical properties and microstructure

The tensile test results were collected from the engineering stress-strain curves in Fig. 13. All samples display an elasto-plastic strain hardening behavior. The results are clustered in terms of the yield and ultimate tensile strengths based on the unit cell size and wall thickness, despite the unified nominal volume fraction (approximately 50%) and cross-sectional area (approximately 50 mm²) between all sample sizes. The yield point for the S lattice geometries is approximately 300 MPa. The yield points for the M and L lattices are 280 and 240 MPa, respectively. Similarly, the ultimate tensile strength sequence is 500 MPa for S lattices, 460 MPa for M lattices, and 420 MPa for L lattices. Common to all sizes is the instability in the plastic region, as indicated by the constant fluctuation of the signal. In addition, the sample stress deviation seems to increase for samples with thinner walls. As exhibited in the detailed CT scan of the Gyroid S 01 – 1 sample, voids and cracks in the structure affect a large proportion of the load-bearing cross-sectional area. The samples lose their structural integrity at a fairly clear location, followed by up to 1 mm (5%) of necking before the final fracture. The gyroid elongation at break follows a sequential order where the elongation is approximately 6 mm (33%) for the L lattices, 5 mm (27%) for the M lattices, and 4 mm (22%) for the S lattices. The elongation-at-break sequence for the diamond samples is similar, except for the L lattice size. On average, the diamond geometries display slightly more pronounced necking compared to the gyroid geometries.

The main goal of the tensile test was to verify no latent corrosion effects compromise the structural integrity of the geometries. Based on

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**Video 1.** Rotational video of each microCT scanned and analyzed sample. The spatial locations of the indexed mass losses are highlighted through a selectively transparent lattice surface. A video clip is available online. Supplementary material related to this article can be found online at doi:10.1016/j.addma.2022.103288.
the overall results, no significant changes were observed between non-corroded and corroded samples. The slightly lower tensile strength of corroded S and M lattices may hint a minor size-dependent effect. However, the low sample count and the influence of entrapped powder prevent a statistically significant conclusion.

Yan et al. [53] identified a similar clustering of mechanical properties based on unit cell size in compressive tests of various skeletal 316L lattices. The difference was then explained by the gradually increasing porosity of the thicker struts. Later, Maskery et al. [54] noticed the same tendency when varying the cell size of constant relative density skeletal BCC lattices manufactured from Ti6Al4V. CT examination refuted the variation in material porosity as the cause. Instead, entrapped powder, loosely bound surface particles, and grain sizes approaching the strut diameter were provided as possible causes.

In this study, one cause for the clustered results can be the differences in lattice connectivity between lattice sizes. Based on the microCT data, the average sectional areas of all lattices are close to the as-designed value of 50 mm². However, the effective load-bearing area, particularly for the L lattices, is smaller than the as-designed value. The non-connected overhanging features at the peripheries do not contribute to...
the tensile strength (for example, those shown at the rightmost vertical edges in Fig. 10a and b). The M and S lattices offer better connectivity and a larger effective load-bearing section. Replotted graphs with selected sectional areas of 40 mm² for L lattices, 45 mm² for M lattices, and 50 mm² for S lattices unify the stress–strain curves. The entrapped powder within the structure could, to a degree, explain the higher tensile strength of the S and M lattices. The S lattices were found to contain up to 80–90% powder, which could have been partially sintered in the solution annealing at 1030°C. The material strength, modulus, and elongation typically decrease with decreasing strut size [9,10]. The possible strengthening effect of the entrapped powder could compensate for the otherwise inferior strength of the uneven and cavity-ridden surface of the S lattices.

Figs. 14a and 14b displays the microstructure of a LPBF-manufactured 316L reference sample perpendicular and parallel to the build direction. The distribution of grain sizes is visualized in Figs. 14c and 14d with the Hall-Petch grain size parameter $d^{-0.5}$. Examining the variation of the Hall-Petch grain size parameter is helpful because the inverse square root of average grain size is shown to correlate with material mechanical properties [55]. The volume-weighted average grain size proposed by Lehto et al. [40] considers the grain size variation in heterogeneous materials and provides a better estimate of the Hall-Petch relationship.

The perpendicular direction displays a multimodal grain distribution with small equiaxial, medium-sized elongated, and larger, more equiaxial grains. The network of denser grain bands corresponds to the...
centerlines of melt pools. Based on 3356 identified grains in the perpendicular direction, the volume-weighted average grain size is 14.45 \( \mu m \), and relative grain size dispersion is 5.41. The line intercept method yields an average grain size of 6.49 \( \mu m \). In the parallel direction, the grains extend in the build direction. Grain shape is dominantly columnar, both in smaller and larger grains. Grain size dispersion in the parallel to build direction is slightly less pronounced. The grain size values in the parallel direction are 18.01 \( \mu m \), 4.95, and 8.87 \( \mu m \) respectively (1558 identified grains).

The non-uniform grain distribution implies that the 1030 \(^\circ\) C solution annealing has not recrystallized the microstructure. For conventional stainless steel a solution annealing at 1100–1200 \(^\circ\) C with water quenching is often recommended to maximize corrosion resistance. A fast cooling through the sensitization temperature range from 500 to 800 \(^\circ\) C avoids carbide precipitation at the grain boundaries, which can hinder the alloy resistance to intergranular corrosion and stress corrosion cracking [56]. However, the LPBF-fabricated 316L microstructure, phase composition, and inclusions differ from wrought 316L. Consequently, its behavior in heat treatment is different [57]. The superior corrosion resistance of as-built 316L has been linked with the complete elimination of MnS inclusions, higher alloying at subgrain boundaries, and passive film composition [58]. Kong et al. [59] found a solution annealing at 1200 \(^\circ\) C (compared with 1050 \(^\circ\) C) increased the grain size more rapidly, eliminated most dislocations, and achieved complete recrystallization. However, the results indicate a lower pitting resistance for higher heat treatment temperatures and longer holding times. The changes in pitting resistance were attributed to decreases in the passive film thickness. Laleh et al. [60] attributed the reduced pitting performance after high-temperature annealing to the formation of MnS inclusions at 1100 \(^\circ\) C and 1200 \(^\circ\) C. MnS precipitation was also identified by Wang et al. [57] after a long holding time of 8 h at 1100 \(^\circ\) C. The abovementioned studies corroborate the reduced pitting resistance in higher temperature solution annealing and would support a slightly lower heat treatment temperature below 1000 \(^\circ\) C.

Figs. 15a and 15b display a Beraha II tint-etched microstructure of a solution annealed reference sample parallel and perpendicular to the build direction. The color etching of the smallest S lattice (Gyroid S 01 – 1) proved difficult. The lattice pattern seems to disturb the Beraha sulfide film formation due to surface tension effects near the edges. A clear transition is seen when moving from the solid material to the lattice section in Fig. 15c. Interestingly, the etching contrast is only lost in the lattice region perpendicular to the build direction (Fig. 15c and 15e). In addition, 50–100 \( \mu m \) thick ‘rings’ surround the edges and obstruct the grain boundaries. The direction parallel to the build direction in Fig. 15d and Fig. 15f is etched with good contrast. Both samples were placed in the same mount.

Compared to the parallel reference (Fig. 15b), the grain direction in the parallel lattice sample (Fig. 15d and 15f) is less uni-directional. Grain direction and slenderness are influenced by the surrounding geometry. Changes in grain aspect ratio [8] and preferred grain growth direction [10] with thin struts have been previously associated with the heat flow direction. The partially sintered powder at the outer lattice edges mainly consists of finer equiaxial grains. Where present, the powder microstructure blends with the columnar grains up to 50 \( \mu m \) from the lattice surface. Such bimodal distribution could have implications for the strengthening effect and explain the clustering of tensile testing results presented earlier. Fig. 16 visualizes the distribution of the Hall-Petch grain size parameter of the parallel-sectioned lattice (Fig. 15d). Based on 2300 identified grains, the volume-weighted average grain size is 28.77 \( \mu m \), and relative grain size dispersion is
The line intercept method yields an average grain size of 14.13 µm. Compared with the solid reference sample, the average grain size is larger and grain dispersion comparable.

3.5. Limitations and suggestions for future studies

To the authors’ knowledge, the corrosion of lattice structures has not been previously studied, and no established methods exist to address the topic. The novelty is a challenge and there is room for improvement in the presented experimental design. The mass losses extracted from the microCT data were inconsistent with the as-measured data. The entrapped powder within samples, coarse voxel size in microCT, and the limited corrosiveness of the media were identified as the main contributors to the poor accuracy. Combining the global microCT analysis with a more detailed metallographic evaluation would be beneficial.

The geometry and the process parameters can be optimized to manufacture powder-free structures. Miniaturized tensile specimens with a thickness of only a few unit cells may be justified for a few reasons. First, the depowdering of the structure is facilitated. Second, the smaller size allows a finer voxel resolution in microCT (inversely proportional to the sample size). Third, an optical line of sight is beneficial in examining individual corrosion pits. After precise sectioning, the microstructure and composition of the selected pitting locations can be further studied, for example, with electron backscatter diffraction (EBSD) and energy-dispersive X-ray spectroscopy (EDS). In this study, the standard industrial process parameters were used across all samples. However, the laser parameters, beam shape, and laser scanning modes in LPBF could be further optimized to manufacture higher-quality thin-walled geometries without entrapped powder. Instead of the continuous wave mode, pulsed laser scanning can offer better control in manufacturing fine features [45–47].

Tensile strength and percent elongation are valuable metrics to study material embrittlement due to corrosion (ASTM G31-72). The lack of a tensile testing standard for lattice structures is unfortunate. The ISO 6892 recommendation on specimen proportionality between gauge length and cross-sectional area can be partially adapted. Otherwise, compressive testing, according to ISO 13314, is an option.

A finer voxel size and thorough rust removal can improve the
accuracy of the microCT analysis. The densities of 316L steel (> 7.97 g/cm³) and iron oxide (5.24 g/cm³) should be differentiable in microCT. However, the minuscule size of individual iron oxide particles (20–30 nm [52]) renders them undetectable, even with high-end nanoCT devices. The diameter of passivated corrosion pits ranges from 100 nm to 10 µm [14]. Near or sub-micrometer voxel sizes are recommended to localize sustained corrosion sites and some larger passivated pits. The ASTM G1 provides general guidelines for rust removal.

The authors expected the poor surface roughness, surface defects, and microstructural differences of fine LPBF-fabricated 316L lattices to promote more severe and sustained localized corrosion in the selected 3.5 wt% NaCl solution. However, the corrosion was mainly limited to a very shallow depth on the outer surfaces of the S lattices. This raises the question of whether there were inconsistencies in the solution chloride concentration and whether only the S lattice surface topography promoted pitting before an equilibrium concentration was reached. In future corrosion studies on LPBF-manufactured 316L lattices, a more aggressive 6 wt% FeCl₃ is suggested as a corrosive medium.

The corrosion (and fatigue) performance of AM-fabricated parts can be improved in a few ways. Heat treatment and hot isostatic pressing (HIP) can alleviate process-induced microstructural variation and minimize void defects. A polished surface does not provide as many prospective sites for pit initiation. However, complex structures with fine internal channels or lattices are largely out of reach for sanding media-based polishing techniques. Consistent abrasive polishing is challenging as the near-surface locations and corners tend to wear more. Chemical polishing [61], electropolishing [62], and magnetic abrasive finishing [63] have been successfully utilized for surface polishing of internal features. The results of chemical polishing appear to be the least affected by geometry [61]. However, the evaluated geometries to date have been simple. A uniform polish of internal, arbitrary geometries to a submicron surface roughness remains a challenge.

The CT data reconstruction and segmentation are vulnerable to artifacts and noise [29,30]. Geometrically simple reference parts can be used as a baseline for thresholding. In this regard, non-subjective workflows, such as those introduced for AM part density measurement by du Plessis et al. [57], can be helpful. Nevertheless, the possibilities of microCT as a research tool in AM and corrosion are extensive. In-situ corrosion testing and time-lapse microCT scans [31] would allow a three-dimensional, dynamic view of corrosion and its propagation. The method developed in this study is not only applicable to corrosion evaluation. Other potential use cases include for example tensile or compressive testing, fatigue testing, evaluation of surface roughness prior and after polishing, and localization and removal of entrapped powder. Further study directions on the corrosion of complex geometries could encompass microfluidic effects and capillary motion. Together the mesoscale geometry, rough surfaces, trapped material powder, and corrosion deposits can form nano- and micrometer-sized channel systems, which can influence the local corrosion dynamics.

4. Conclusions

This study investigated how LPBF-fabricated 316L lattice feature size and geometry influence its susceptibility to localized corrosion. Only the S lattice category samples (nominal wall thickness of 0.323 mm) were found to initiate noticeable localized corrosion when immersed in aqueous 3.5 wt% NaCl. All pits were passivated during the first week of the 21-day test, indicating that the medium was not aggressive enough to sustain corrosion.

(1) Only the finest 0.323 mm wall thickness S lattices displayed noticeable localized corrosion (three out of five samples). Based on the immersion tests, the S lattice cavity-ridden surface covered with a partially molten powder influences pit initiation. The less efficient cooling of thin features and contour-dominant scanning parameters can influence the microstructure.
(2) The sample mass losses after the 21-day test ranged from 8 to 19 mg. Despite the visually aggressive pitting of the S lattices, the measured mass losses were not found to vary with the unit cell size or geometry type (gyroid or diamond lattice).
(3) The mass losses calculated from the microCT data did not agree with the measurements. Entrapped powder, coarse microCT voxel size, and limited corrosiveness of the media complicated the workflow. Suggestions to improve the experimental setup are provided in Section 3.5. Nevertheless, the presented microCT workflow is applicable to any metallic material and corrosive medium. The workflow and digital tools to register and analyze before-and-after microCT scans are presented and fully disclosed in this study and the accompanying data package.
(4) Based on the microCT scan analysis, the mass loss was related to the detachment, shift, or corrosion of partially melted or entrapped powder. No corrosion sites penetrated the dense base materials. The centroids of most of the material losses were less than 1 mm from the sample surface. In addition, the sliced Gyroid S 01 – 1 sample displayed corrosion deposits mainly on its outer
Fig. 16. Variation of the Hall-Petch grain size parameter of the lattice micrograph in Fig. 15d.

surfaces. Hence, the internal structures did not promote localized corrosion.

CRediT authorship contribution statement

Tuomas Puttonen: Writing – review & editing, Writing – original draft, Software, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. Sergei Chekurov: Writing – review & editing, Writing – original draft, Validation. Jukka Kuva: Writing – review & editing, Methodology, Data curation. Roy Bjorkstrand: Writing – review & editing, Validation, Resources. Jouni Partanen: Supervision, Funding acquisition. Mika Salmi: Writing – review & editing, Validation, Project administration, Funding acquisition, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

To allow full validation of the results and research reproducibility, all 3D models, original and analyzed microCT data, tensile testing csv-files, microscopy images, and code resources are published as a data package in the Zenodo open-research database maintained by CERN. The files are accessible via the link: https://doi.org/10.5281/zenodo.7260218, [36]. Kindly cite this article and the associated Zenodo dataset when utilizing or modifying the data. In addition, full RAW CT files, microscopy images, and code resources are published as a data package in the Zenodo open-research database maintained by CERN. The files are accessible via the link: https://doi.org/10.5281/zenodo.7260218, [36]. Kindly cite this article and the associated Zenodo dataset when utilizing or modifying the data. In addition, full RAW CT

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