Micro-CT based characterisation of the effect of surface modification on the morphology and roughness of selective laser melted Ti6Al4V open porous structures

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Introduction and aim
Additive manufacturing (AM) represents the most advanced method to build porous structures with a controlled and robust internal and external geometry. However, it does not allow a high control of the surface properties at the micro-scale¹⁻³. For that reason, an appropriate surface modification is needed as a post-production treatment. Controlled surface modification of three-dimensional (3D) open porous structures is complicated since it requires surface treatment both on the outer and inner surfaces of the structure. Chemical and/or electrochemical treatment can provide a solution for this problem since acid-based solutions can penetrate porous structures through the interconnected pores⁴⁻⁶. It is obvious that by modifying the surface properties, the morphological characteristics of the porous structure are changed. Therefore, a thorough analysis of the surface morphology of porous structures is crucial. In this study, Ti6Al4V open porous structures, produced by selective laser melting (SLM) were treated with chemical etching and electrochemical polishing to homogenise the surface roughness throughout the entire structure. To quantitatively assess the quality of these treatments, microfocus X-ray computed tomography (micro-CT) was used for the morphological characterisation of the Ti6Al4V open porous structures prior to and after each surface treatment step. The specific surface after chemical etching was used as input to optimise the electrochemical polishing. The aim of this study was to assess the effect of the surface treatments on the morphology of the porous structures and the strut surface roughness and hence on the effectiveness of the surface treatments.

Materials and methods
SLM was used in this study to produce open porous structures starting from bio-inert Ti6Al4V powder. Cylindrical porous structures were designed using Magics software [Materialise NV, Haasrode, Belgium] with an open porous unit cell. Three different architectures with designed strut thickness of 100, 140 and 180 µm (Strut 100, Strut 140 and Strut 180 respectively) and a pore size of 1 mm were tested. The designed diameter and height of the porous structures were respectively 6 mm and 12 mm. More information about the porous structure design and production can be found in Ref. [1].

Two consecutive surface roughness modification procedures, suggested by Pyka et al.⁷, were applied: (i) 10 minutes of chemical etching (CHE) to remove the entire strut surface, including loosely sintered SLM powder remnants and (ii) 8 minutes of electrochemical
polishing (ECP) to remove metal ions from the surface and obtain a smoother and more homogenous surface. The dissolution rate depends on the current density, which is governed by the surface topology. In order to ensure homogeneous ECP reduction rates, the current density was kept at 2 mA/mm² for all tested designs.

To determine the current to be applied, the surface area of the porous structure was calculated by means of micro-CT. Additionally, the complete porous structure morphology, prior and after each surface modification step, was evaluated by micro-CT-based image analysis using the Philips HOMX 161 microfocus X-ray system with AEA Tomohawk CT software. The pixel size of the images was 12.6 µm. Manual, but consistent global segmentation was carried out to allow quantification of the surface area, as well as the porosity, average pore and strut thickness and their distributions using CTAn [Skyscan NV, Kontich, Belgium].

The roughness was determined based on 2D cross-sectional micro-CT images using the surface profile line for the calculations. The pixel size of the images was 1.75 µm. As the 2D images were taken with a high-resolution SkyScan 1172 micro-CT system, which required no special sample preparation, the measurements could be performed in a non-destructive way. Compared to commercially available profile measuring systems, the novel protocol for surface roughness measurement offers the possibility to quantitatively analyse the strut surface morphology of complex 3D porous structure as part of the manufacturing process. The applied acquisition parameters for both the morphological characterisation as the roughness measurements are presented in Table 1.

**Table 1.** Micro-CT acquisition parameters used for both the morphological characterisation as the roughness measurements of the as-produced and surface treated Ti6Al4V SLM structures.

<table>
<thead>
<tr>
<th>CT system</th>
<th>Voltage</th>
<th>Current</th>
<th>Filter material</th>
<th>Voxel size</th>
</tr>
</thead>
<tbody>
<tr>
<td>Philips HOMX 161</td>
<td>90 kV</td>
<td>390 µA</td>
<td>1 mm aluminium</td>
<td>12.6 µm</td>
</tr>
<tr>
<td>SkyScan 1172</td>
<td>100 kV</td>
<td>100 µA</td>
<td>0.5 mm Cu</td>
<td>1.75 µm</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.5 mm Al</td>
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</tbody>
</table>

**Results and discussion**

**Surface roughness measurements of the Ti6Al4V open porous structures**

The untreated SLM Ti6Al4V porous structures (i.e. as-produced structures) for the different strut thicknesses showed a non-uniform roughness (Fig. 1a, b and c) primarily caused by spatial differences in attached, non-melted powder grains. Also a higher designed strut thickness resulted in a larger amount of non-melted grains on the strut surface.

**Figure 1:** Representative scanning electron microscope (SEM) images of a single strut of the as-produced Ti6Al4V porous structures with designed strut thickness: a) 100 µm, b) 140 µm and c) 180 µm (the y-direction is the building direction, T-strut top, B-strut bottom).
The results of the overall strut roughness measurements of the top and bottom of the struts, presented in Figure 2a, did not show significant differences between designs Strut 100 and Strut 140 as well as between designs Strut 140 and Strut 180 for the as-produced porous structures and after ECP. However, taking into account the visible spatial differences, roughness analysis performed separately for the strut top and bottom revealed significant differences between top and bottom. Moreover, that difference increased for the porous structures produced with thicker struts, which confirmed visual inspection of the samples (Fig. 1), where more powder grains attached to the bottom of the strut were noticed for design Strut 140 and 180 compared to design Strut 100. This spatial difference confirmed the need of an appropriate post-production surface treatment that can homogenize the strut surface roughness throughout the complete porous structure for the different designs.

In Figure 2a, it can be seen that the total roughness of the structures significantly decreased for all tested designs after each applied surface treatment step. Comparison of the roughness for the top and bottom of the struts (Fig 2b) showed that the roughness reduction of the strut bottom was higher compared to the top after CHE, which reduced the top-bottom dissimilarities. Only for design Strut 100, the difference between the strut top and bottom was insignificant after CHE and combined CHE-ECP. Figure 2c shows that the effectiveness of CHE depended on the porous structures design and was the highest for the porous structures Strut 100, which contained the least amount of non-melted powder grains after SLM production.

- **Morphological characterization of the Ti6AlV open porous structures**
Morphological analysis of the as-produced and surface treated porous structures revealed that the average strut thickness reduction of the porous structures with design Strut 140 and
180 were comparable, but a higher reduction of the absolute average strut thickness was found for design Strut 100. This implied that the CHE effectiveness depends on the initial amount of non-melted powder grains attached to the bottom surface rather than the initial strut thickness. For porous structures with design Strut 100, 10 minutes of CHE was sufficient to remove all grains, but not enough for designs Strut 140 and 180. In order to increase the efficiency of the CHE, the treatment time should be optimized depending on the initial strut thickness. Based on the surface area of the samples after CHE determined by micro-CT (i.e. 9, 10, 11 cm² for Strut 100, 140 and 180 respectively), the following current values were applied during ECP: 1.2, 1.6 and 1.9 mA for designs Strut 100, 140 and 180 respectively. However, after ECP a higher reduction of the struts thickness was observed for structures with thicker struts (Fig. 2d). Visual inspection of the cross-sectional micro-CT images of the as-produced structures, (fig. 3) obtained with an isotropic voxel size of (12.6 µm)³, revealed that this limited spatial image resolution makes it difficult to discriminate the non-melted grains attached to the strut surface from the surface itself (average grain size is about 30 µm). Because of the partial volume effect, using a global threshold for segmentation, the strut thickness might be increased by including the non-melted powder grains as a coherent part of the structure. Because of this, the surface area would be determined incorrectly, having an influence on the current density calculations. This effect would even more be expressed for designs Strut 140 and 180, where a larger amount of non-melted surface-attached grains were observed. Because of the potential error in the measurement of the surface area, the applied current density might not be equal for all designs, and hence might have led to a higher reduction rate for design Strut 140 and 180, explaining the differences in ECP effectiveness. In future experiments, higher resolution micro-CT images will be used to determine the surface area of the porous structures after CHE as input for ECP.

Figure 3: A representative cross-sectional micro-CT image of an as-produced Ti6Al4V porous structure with designed strut size 180 µm. The isotropic voxel size was (12.6 µm)³.

Conclusion
Inhomogeneous roughness of open porous metal structures hampers it use for different applications, also for the SLM Ti6Al4V open porous structures, assessed in this study, which contained a high and inhomogeneous strut surface roughness. This problem was addressed by the introduction of an appropriate and robust surface modification method and combined assessment of the as-produced porous structures to bring their microscale morphological and surface topological properties to a controllable level. The assessment could be done by a thorough characterisation using micro-CT, enabling the quantification of the changes in surface roughness and the morphological properties due to surface modification. This
quantitative characterisation of the effect of surface modification on meso- and micro-scale morphological properties of the Ti6Al4V porous structures is a powerful tool to optimise the surface modification protocol according to desired morphological properties and feedback could be provided for optimisation or fine-tuning of the production technique.

It was shown that roughness inhomogenity can be reduced by the combination of CHE and ECP. However the effectiveness of the surface treatment depends both on the design, as well as on the applied spatial image resolution of the micro-CT images. A higher initial design-dependent roughness after production resulted in an inadequate removal of the non-melted powder grains by CHE, and hence makes CHE optimisation design-dependent. Also incorrect calculation of the strut surface area caused by the partial volume effect and segmentation errors might have influenced the ECP effectiveness and could be solved by using high-resolution micro-CT.

References: