High-resolution micro-CT as a tool for 3D surface roughness measurement of 3D additive manufactured porous structures

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Abstract
Since commercially available profiling systems fail when determining the surface roughness of 3D additive manufactured porous structures, a novel protocol for surface roughness quantification of these porous structures has been developed based on high-resolution micro-CT images. The influence of the noise in the images and the spatial image resolution on the accuracy of the micro-CT-based roughness measurements has been investigated by comparing the roughness parameters of 2D flat substrates determined both with commercially available (optical and contact) profiling systems and the micro-CT-based roughness measurement protocol. This comparison showed that micro-CT can be applied accurately and in a robust manner for surface roughness quantification of 3D additive manufactured porous materials, but that depending on the dimensions of the roughness, the micro-CT acquisition parameters need to be fine-tuned.

Keywords: micro-CT, roughness measurements, 3D additive manufactured porous structures

1 Introduction and aim of the study
The surface topography and roughness of a material can have a significant influence on its functional properties, such as the fluid dynamics [1,2], optical properties [3], frictional behavior [4], heat transfer [5], mechanical properties [6], etc. To understand the relationship between the surface topology and the functional properties, thorough quantitative surface topology analysis is essential. For flat (2D) surfaces, standard techniques such as optical or contact profilometry have been accepted as standard technique for roughness measurements. These line-of-sight techniques however fail when determining the internal surface roughness of 3D porous structures since they can only measure 3D surfaces with a small curvature that are within the line of sight. Additionally, they are not able to non-destructively determine the surface roughness inside a 3D porous structure.

For these reasons, in this study a novel, non-destructive protocol for quantitative 3D analysis of the surface topology of additive manufactured (AM) porous structures has been developed based on high-resolution microfocus X-ray computed tomography (micro-CT) images. It allows to non-destructively assess the roughness of these porous materials at the outer surface as well as inside the structure. To show the potential and limitations of the novel micro-CT based protocol for surface roughness measurements, the roughness parameters of two flat substrates with a high (micro-scale) and low (submicron-scale) roughness were determined both using standard profile measuring systems, namely optical and contact profilometry, and the micro-CT-based surface roughness measurement protocol, and the results were compared. The influence of the noise level in the micro-CT images as well as the spatial image resolution on the roughness measurements was assessed to indicate the limits of the use of high-resolution micro-CT for quantification of the roughness parameters. To establish the novel surface roughness measurement protocol for 3D AM porous structures with a micro-scale roughness,
the surface roughness of selective laser melted (SLM) porous Ti6Al4V structures was assessed as case study prior to and after surface roughness modification.

2 Materials and methods

2.1 Materials

2.1.1 Ti6Al4V flat substrates
As reference materials to validate the micro-CT-based roughness measurement protocol, two Ti6Al4V plates with a width and thickness of 3 mm were produced using SLM, an additive manufacturing technique. Both plates showed a significant roughness (fig. 1B) due to (i) non–melted powder grains, with a size distribution around 25-30 µm, attached to the surface and (ii) the waviness of the surface inherently related to the production technique. One of both plates was then grinded with a rough grinding paper (P80) in order to both reduce the surface waviness and remove the non-melted powder grains, resulting in a 2D plate with a relatively low, i.e. nanometres scale, roughness (fig. 1A).

Figure 1. A typical 3D micro-CT representation of the (A) grinded Ti6Al4V plate with low roughness and (B) as-produced Ti6Al4V plate with high roughness caused by non–melted powder grains attached to the surface and a significant surface waviness inherently related to the production technique. Scale bars = 1 mm.

2.1.2 3D porous Ti6Al4V structures

To establish the micro-CT-based surface roughness measurement protocol for 3D AM porous materials, porous Ti6Al4V structures produced on an in-house developed SLM machine [7] were selected. The design was based on a parametric unit cell (fig. 2A), which consists entirely of identical beams with constant circular cross-section (0.1 mm). Figure 2B visualizes a produced open porous Ti6Al4V structure and figure 2C shows the non–melted powder grains attached to the surface similar
to the flat substrates. Due to the production (the Z-direction in fig. 2C is the building direction), a higher surface roughness was noticed at the bottom surface of the struts compared to the top surface.

In order to homogenize the roughness throughout the full porous structure and to reduce the surface roughness of the as-produced scaffolds, a combination of chemical etching (CH) and electrochemical polishing (EP) was applied, as described in detail in Ref. [8]. Briefly, during CH the samples were immersed for 10 minutes in a chemical solution based on hydrofluoric acid. As a second step, EP was performed. The micro-CT-based surface roughness measurement protocol, described in detail below, was used to determine after each surface roughness modification step the surface roughness parameters of the porous Ti6Al4V structures.

2.2 High-resolution micro-CT-based surface roughness measurement protocol

Figure 3. (A) A typical high resolution (voxel size = 1.5 µm) 2D micro-CT cross-sectional image of a single strut of a porous Ti6Al4V structure and (B) a binarized section of (A) with the corresponding profile lines. Scale bars = 200 µm.

In the micro-CT-based surface roughness measurement protocol, the roughness was determined based on the profile lines of the strut surfaces of the porous structures in the binarized 2D cross-sectional micro-CT images (fig. 3). These profile lines, extracted using an in-house developed MatLab tool, were then used to calculate the following surface roughness parameters (in correspondence with ISO 4287-1997, but not taking into account the proposed measuring length since in 3D porous materials, the proposed measuring length is usually not available):

- arithmetic mean absolute deviation of the roughness profile:
  \[ P_a = \frac{1}{n} \sum_{i=1}^{n} |y_i| \]  

- root mean square deviation of the roughness profile from the mean line:
  \[ P_q = \sqrt{\frac{1}{n} \sum_{i=1}^{n} y_i^2} \]  

- total height of the roughness profile:
  \[ P_T = P_P - P_V \]  

where \( n \) = number of data points in X-direction, \( y \) = the surface height relative to the mean plane, \( P_P \) = the maximum profile peak height and \( P_V \) = the maximum profile valley depth.

2.3 Validation of the micro-CT-based approach versus standard roughness measurement techniques

2.3.1 Optical profilometer

Optical profilometry is a fast measuring technique that can be applied for large scanning areas and for both submicron- and micro-scale roughness measurements of 2D or 3D surfaces with a small curvature. For surfaces with a large roughness, however, thus when the slopes of the peaks are too large, the optical interferometer can no longer measure the reflection correctly, and possibly an erroneous interpolation is made between the points that can be measured. In this study, a WYKO
NT3300 Profiling System [Veeco Instruments Inc., USA] was used. The measuring length, as for all the other measuring techniques, was 1.2 mm.

2.3.2 Contact profilometer

With contact profilometry, a stylus is traversed across the surface. The movement of the stylus is registered by a laser interferometer. A straightness datum is incorporated in the system allowing measurements without a reference to an external straight line datum. However, this implies that 3D surfaces with a significant curvature, such as strut of 3D porous structures, cannot be measured. In this study, a Taylor-Hobson Form Talysurf – 120L [Taylor-Hobson, England] was used with a diamond stylus tip (radius 1.5 – 2.5 µm), resulting in a resolution of 10 nm. The measuring length was 1.2 mm.

2.3.3 Micro-CT imaging

The two flat substrates were scanned using a nano-CT device, namely the NanoTom S [General Electric, Germany]. To assess the limits of the micro-CT based roughness measurement protocol, different micro-CT acquisition settings were applied, i.e. a low (HIGH NOISE) and high (LOW NOISE) frame averaging to determine the effect of the noise level in the images and different voxel sizes ranging from 1.5 µm to 6 µm to assess the influence of the spatial image resolution. The tube voltage and current were kept constant for all scans, namely at 110 kV and 50 µA respectively, and a copper filter of 1 mm was put between X-ray source and sample.

2.3.4 Statistical analysis

To determine the significance level (95 %) between the roughness parameters from the standard profile measuring techniques and the micro-CT-based roughness measuring protocol for all different micro-CT acquisition settings and the two measuring lengths, a 1-way ANOVA test was performed with the results from the standard profile measuring techniques (optical and contact) taken as controls.

3 Results and discussion

3.1 Validation of the micro-CT-based approach versus standard roughness measurement techniques

For the flat substrate with high (micro-scale – 10-20 times larger than the spatial image resolution) roughness, there were no significant differences in $P_a$ values compared to the standard measuring techniques for all micro-CT acquisition settings (Fig. 4A). This implies that for roughnesses with a $P_a$ value in the range of 5 – 30 µm, the noise levels and spatial image resolutions applied in this study were sufficient and not limiting the measurement of the surface roughness. Figure 4C shows a typical 3D micro-CT representation of the high roughness flat substrate, imaged with a high frame averaging (LOW NOISE) and a voxel size of 1.5 µm. Increasing the noise level (fig. 4D) or decreasing the spatial image resolution to 6 µm (fig. 4E) did not show differences in the global roughness. The results for the optical profilometer were higher than for the other techniques because of the sharp, steep slopes on the high-roughness surface ($P_a$ value more than 15 µm). They are believed to introduce diffraction effects at the sharp edges caused by height steps of the order of the wavelength of light and hence some erroneous interpolations between the points that can be measured.

For the flat substrate with low (submicron-scale) roughness, it can be seen that, for both measuring lengths, micro-CT overestimated the $P_a$ value (Fig. 4B). The noise level in the images played a significant role, i.e. a higher noise level resulted in a higher $P_a$ value. This is also clear in figure 4G, where the noise level is higher compared to figure 4F. The spatial image resolution also influenced the results significantly. The largest voxel size, i.e. 6 µm (fig. 4H), could not capture the submicron-scale roughness. Even the smallest voxel size, i.e. 1.5 µm (fig. 4F), was not sufficient for this roughness.
Hence, roughnesses with a $P_a$ value below 1 µm could not accurately be quantified with the acquisition setting used in this study.

![Figure 4](image1)

**Figure 4.** $P_a$ values, measured using micro-CT, and optical and contact profilometry for the flat substrate with (A) high roughness and (B) low roughness for a measuring length of 1.2 mm [*p < 0.05 = significant difference*]. Typical 3D micro-CT representations for the flat substrate with (C-E) high roughness and (F-H) low roughness. For micro-CT, both a low frame averaging (HIGH NOISE; D, G) and a high frame averaging (LOW NOISE; C, E, F, H) were applied, and scans were made with voxel sizes of (C, D, F, G) 1.5 µm and (E, H) 6 µm. Scale bars = 500 µm.

### 3.2 Application of the micro-CT-based roughness measurement protocol on 3D additive manufactured porous structures

Figure 5A shows the surface roughness parameters, averaged for the top and bottom of the struts, of the porous Ti6Al4V structures prior to surface roughness modification (fig. 5B) and after each surface roughness modification step (fig. 5C and 5D). In figure 5C it can be seen that the CH effectively removed all the non-melted attached powder grains from the scaffolds surface, as was previously shown by Pyka *et al.* [7]. This was reflected in a significant difference in $P_t$ (fig. 5A) after CH. The EP made the surfaces smoother, which introduced a significant difference in surface roughness compared to the as-produced (raw) samples.

As indicated in figures 6A and 6B, the surface roughness, averaged for the top and bottom of the struts, was measured respectively in the centre and at the edges of the porous structure after each surface modification step. There were no significant differences in surface roughness between the centre and the edges of the porous Ti6Al4V structures after production (fig. 6C) and after CH (fig. 6D), indicating globally a homogeneous distribution of the roughness throughout the structures after production and a robust and homogeneous reduction of the roughness by CH. EP however introduced significant differences in surface roughness throughout the scaffold (fig. 6E), indicating that the EP settings still...
need to be optimized. This analysis shows that the proposed micro-CT-based surface roughness measurement protocol can be applied to quantify the roughness not only at the edges of a porous AM material, but also inside and throughout the entire sample in a non-destructive manner.

Figure 5. (A) The surface roughness parameters, averaged for the top and bottom of the struts, for the porous Ti6Al4V structures prior to surface roughness modification and after each surface roughness modification step (chemical etching (CH) and electrochemical polishing (EP)) and typical high-resolution (voxel size = 1.5 µm) 2D micro-CT images of a single strut of a porous Ti6Al4V structure (B) prior to surface roughness modification, (C) after CH and (D) after EP [* p < 0.05 = significant difference]. Scale bars = 100 µm.

Figure 6. Typical axial, sagital and coronal micro-CT sections of a porous Ti6Al4V structure prior to surface roughness modification indicating the location of the roughness measurements, i.e. (A) in the centre and (B) at the edges of the porous structure. The roughness parameters, averaged for the top and bottom of the struts, determined at the centre and the edges of the porous Ti6Al4V structures (C) prior to surface roughness modification, (D) after CH and (E) after EP.
3 Conclusion

It is essential to quantify the surface roughness of a porous material to be able to correlate the functional behaviour to the surface properties. In this way, the most optimal surface properties for future designs and production of porous structures can be looked for. In this study, the validity and potential of micro-CT for the quantification of the roughness of AM 3D porous surfaces was shown. For high roughness (micro-scale) surfaces the micro-CT based protocol can be used with comparable or even better accuracy than standard profile measuring systems. For low roughness (submicron-scale), the spatial image resolution and the noise level in the images can limit the use of the micro-CT protocol. Thus, depending on the dimensions of the roughness, the micro-CT acquisition parameters need to be fine-tuned. Also, it was shown that the non-destructive character of the micro-CT-based protocol allows a thorough analysis of the surface topology throughout the entire porous structure.

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