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3D RECONSTRUCTION AND CHARACTERIZATION OF THE POROUS MICROSTRUCTURE OF AL$_2$O$_3$-COATINGS BASED ON SURFACE DATA

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ABSTRACT

The functionalities and properties of components strongly correlate to the used material's structure and the microstructure of the surface. Therefore, by characterizing the structure of a material or surface information is indirectly gained on its properties. In this work porous aluminum oxide surface coatings for the usage of tribological pairings are investigated. The tribological behavior of a component is influenced by the surface structure. Further, in porous material the fluid flow strongly dependents on morphological and topological parameters like the pore size and shape and the pore connectivity. To gain meaningful information on the coatings properties, we present a new approach to obtain 3D measurement data of porous structures and reconstructing the porous material. This is implemented in an alternating process of milling the surface and measuring the surface data. Based on the surface height data and a registration process the three dimensional porous structure of the material is reconstructed.

Index Terms - 3D-Reconstruction, CLSM, 3D Measurement Data, Porous Material

1. INTRODUCTION

The surfaces of components hold many different functionalities. These are influenced by the surface characteristics and their microstructure. Well-known applications of the correlation of the surface structure and their functionalities are for example optical applications like the Fresnel lens, hydrodynamic applications as known with commercial golf balls or as in our approach a mechanical application: the influence of the surface characteristics on the friction and wear behavior in tribological pairings [1]. The objective of this work is to improve the friction and wear behavior of endoprostheses, artificial hip joints or knee prostheses. A critical factor of endoprostheses is the quality of the sliding surface. As wear particles can cause foreign body reactions, which in turn can lead to a loosening and thus the failure of the endoprosthesis, the endurance of the implants depends largely on the wear resistance and lubrication of the sliding surface. Therefore, in this research project we seek to develop functionally integrated, porous implant coatings, which possess a high wear resistance due to the coating material itself and further ensure an active friction reduction due to a reservoir of lubricant, see figure 1. As in porous media the fluid flow strongly depends on morphological and topological parameters like the pore size and shape and the pore interconnectivity. Further to the surface structure we seek to investigate the material structure to additionally draw correlations between the structure and the functionality.

Therefore, 3D measurement data of the surface coating is needed to be investigated. We present a new approach to gain 3D measurement data of the porous structure and the top

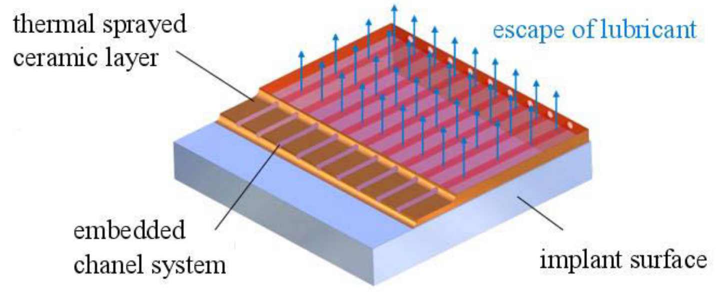


Figure 1: Functional integrated surface.

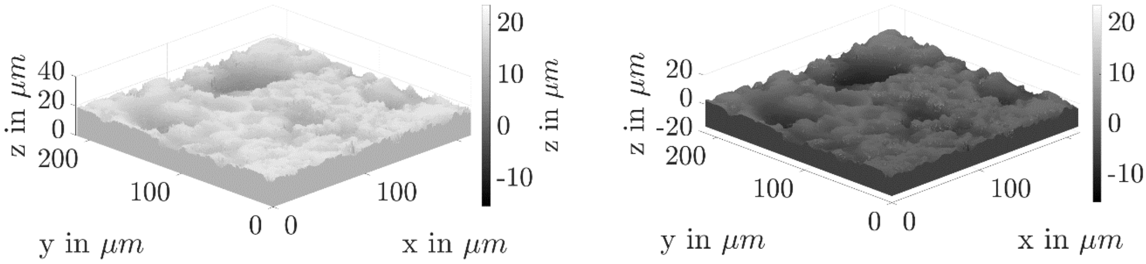
surface. The three dimensional data of the porous material is reconstructed based on surface height data gained in an alternating process of milling the surface, measuring it stepwise and merging it afterwards. Based on the reconstruction data the porous material and the surface structure's functionality can be characterized indirectly by the characterization of the porous microstructure. This serves a better understanding of the material properties and the manufacturing process.

2. STATE OF THE ART

Although, characterizing porous material can be realized in many different ways, varying from porosimetry over stochastically modeling to a direct 3D reconstruction of the microstructure, the characterization of disordered pore networks is still a great challenge. Both modelling and direct 3D measuring are categorized in digital reconstruction approaches of porous media. Modeling is accomplished either by stochastic methods or process-based methods. In the field of modeling a porous structure based on stochastic methods the porous media is reconstructed on statistical information of one or several thin 2D slices. Whereas with process-based methods, the structure is reconstructed by imitating the physical properties [2]. Modeling approaches have the advantage of not requiring costly specialized 3D scanners. However, with direct 3D measuring approaches most information about the actual material and its structure is gained and is, therefore, favored in the characterization processes. Direct 3D reconstruction of porous material can also be realized in different ways, e.g., with the help of computer tomography or by milling the surface via the focused ion beam (FIB) technique and measuring it by scanning electron microscopy (SEM).

Direct 3D reconstruction approaches have been used by Drach et al., Balach et al. and Joos et al. to reconstruct porous material. The studies of Drach et al. focus on the characterization and statistical modeling of irregular porosity and are based on X-ray micro tomography data. The micro tomography data, with a voxel resolution of 14.7 µm, is binarized with a predefined threshold to distinguish the pores' boundaries. They characterize the pore geometry and orientation distribution in chemical vapor infiltrated carbon/carbon composites [3]. A different approach is used by Balach et al. and Joos et al. Both use the combination of FIB and SEM for the 3D reconstruction of porous media [4, 5]. In the work of Balach et al. mesoporous carbon with tailored pore size is reconstructed and characterized whereas Joos et al. study the porous network of electrodes. As within the work of Drach et al. the pore structure is segmented by thresholding: meaning the porosity is highly dependent on the chosen threshold. Both Balach et al. and Joos et al. address the difficulty of finding the correct threshold to segment the pore structure from the solid material in their work.

In the presented approach the drawback concerning the determination of a proper threshold to segment the pore space from the material is not an issue. This is overcome due to measuring



(a) Surface height data *(b)Preprocessed surface height data*

Figure 2: Example surface height data of the porous Al_2O_3-layer.

the surface with a confocal laser scanning microscope (CLSM) capturing the exact structure boundaries and, therefore, no threshold determination is needed.

3. METHODS AND MATERIALS

The investigated surface coatings are aluminum oxide (Al_2O_3) layers manufactured by a thermal spraying process. As the three dimensional data is sustained by an alternating process of milling by fly cutting and measuring the surface data with a CLSM, the VK-X-210 by Keyence, a precise registration process is necessary to align the consecutively obtained measurement data. Based on the aligned data the pores are reconstructed by isolines. Isolines belonging to one pore are mapped by a nearest neighbor approach. Last, morphological and topological features of the porous structure are characterized.

3.1 Measurement data of Al_2O_3-coatings

The Al_2O_3-coatings are manufactured by a thermal spraying process and are subsequently polished. The porosity is affected by the parameters of the manufacturing process and the powder composition. Beneath the surface the layers hold a channel system which is realized with the help of a sacrificial material, which is removed after the spraying process. The channel system is to work as a reservoir for the lubricant. Due to the porosity of the ceramic the lubricant is supposed to pass through the material and escape on the top layer.

The three dimensional data of the layers is gained by a similar process like the combination of FIB and SEM. The coatings are milled with a fixed decrement and each subsurface is measured with the CLSM. This way surface height data is recorded at each milled level. Data shown in this work was acquired either with a 10 x magnification lens with a horizontal resolution of 1.402 µm or with a 50 x magnification lens with a horizontal resolution of 0.277 µm. The milling step is about 2 µm. Before registration or reconstruction, each data set is preprocessed by a noise filter based on nonlinear anisotropic diffusion and tilt correction implemented by a robust plane fit based on a total least squares approach. Example surface height data of a porous Al_2O_3-coating is shown in figure 2(a), the preprocessed data after noise suppression and tilt correction is shown in figure 2(b).

The noticeable porous microstructure in the surface height data sets of the milled subsurfaces is highly discontinuous. Pores not as deep as the milling depth will vanish in the subsequent, further milled, data sets and pores, which were embedded in the coatings may be opened and arise in the lower measurement data. Further, the boundaries of pores observable in successive data sets may change due to undercuts. Measuring with a CLSM, a topview of the surface is acquired, thus, angles steeper than approximately 88 degrees cannot be measured. Overall, the surface structure varies the most in the region of the milling depth of subsequent data as the lower data set shows higher porosities for the same depth most likely due to the before mentioned small pores and undercuts. To gain the actual 3D microstructure, these dissimilarities are most challenging.

3.2 Registration

Due to inaccuracies caused by the measurement process a precise registration is needed to align the subsurface height before the reconstruction. The registration is implemented in a two-step approach, a coarse alignment by 3D phase correlation and an ensuing fine registration by an iterative closest point (ICP) algorithm. It is implemented in an iteratively pairwise alignment of the successive height data.

The main misalignment is translational. The horizontal shift is caused by placing the sample in turns under the CLSM and in the milling machine. The vertical shift occurs due to the milling itself. Therefore, the coarse alignment is limited to a translational transformation. The misalignment is detected by the 3D phase correlation. Phase correlation is a frequency approach and based on the frequency-domain representation of the, to be aligned, data. Because a 3D transformation is striven, the surface height data needs to be transformed into a volume representation, denoted as $V_{1_{x,y,z}}$ and $V_{2_{x,y,z}}$, and the volume data is transformed in the frequency domain by the Fast Fourier Transformation, denoted as $F_{1_{x,y,z}}$ and $F_{2_{x,y,z}}$. The shift between two subsequent volumes is determined by the detection of the maximum peak of the Fourier inverse of the normalized cross power spectrum of the two Fourier transformed volumes. Where $F_{3_{x,y,z}}$ denotes the normalized cross power spectrum defined by:

$$F_{3_{x,y,z}} = \frac{F_{1_{x,y,z}} \odot F^{*}_{2_{x,y,z}}}{|F_{1_{x,y,z}} \odot F^{*}_{2_{x,y,z}}|},$$

with * denoting the complex conjugate, $\odot$ the element wise multiplication and |x| the magnitude function. Further, the shift between the two volumes $(\Delta x, \Delta y, \Delta z)$ denotes the location of the maximum peak detected in the Fourier inverse $V_{3_{x,y,z}}$ of $F_{3_{x,y,z}}$:

$$(\Delta x, \Delta y, \Delta z) = -[\arg \max_{(x,y,z)} V_{3_{x,y,z}}].$$

The phase correlation shows high robustness to the discontinuities of the pore structure and gives a good approximation for the coarse alignment.

The fine alignment is implemented in an ICP approach, which was first introduced by Chen and Medioni and Besl and McKay [6, 7]. The fine registration performs a rigid transformation. The ICP works well on roughly preregistered point clouds. One point cloud serves as the model points $M = \{m_i\}_1^{N_m}$ to which the second data points of the second point cloud $P = \{p_i\}_1^{N_p}$ are matched. Therefore, the second point cloud is transformed interactively to minimize an error function specified by the mean-square distance between the two point sets:

$$m_{cl}(i, R, t) = \arg \min_{m \in M} \left|\left|m - p_i(R, t)\right|\right|,$$
$$m_i(R, t) = \left|\left|m_{cl}(i, R, t) - p_i(R, t)\right|\right|.$$

Because of the discontinuity and the not to be aligned plane data points (that are milled in-between the measurements) a modified version of the ICP is used rather than the original approach. The trimmed ICP by Chetverikov et al. takes not all points into account but rather works with a specified overlap rate ξ [8]. The rigid transformation is estimated on the least trimmed squares. The overlap rate is estimated by the coarse alignment. Data point above the

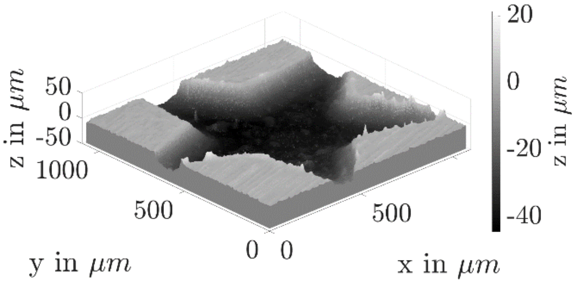


Figure 3: Surface height data of sunk-in star structure of the back of a 1 cent euro coin.

vertical shift and outside the boundaries of the horizontal shift are left outside in the matching process. For a detailed description on the registration process see [9].

3.3 Reconstruction

Individual pores are reconstructed based on isolines derived from the registered data. Isolines are lines of a constant height and in this application represent the boundaries of the pore structure. Therefore, the surface height data is segmented by a height threshold and binarized. The boundaries of the biniarized data are the pore contour lines at a specific height level. The pore structure is then synthesized starting at the lowest acquired height of all subsurfaces' height data. According to the vertical shift between successive milled surfaces, the isolines are provided by the height data of different data sets of the subsurfaces. With a milling decrement of about 2 µm the vertical resolution of the reconstructed pores is rather poor. To acquire more information of the pore structure the step size between derived isolines is adjusted to the horizontal resolution of the data set. Meaning isolines are determined at a step size equal to the horizontal resolution. However, pores smaller than the milling decrement are still undetectable.

The matching of the contour lines associated to one pore is implemented in a nearest neighbor approach. The merging process is started at the lowest height of the isolines. For each boundary the best match in the upper set of boundaries is determined by the nearest neighbor. If there is no good match found, the set of points associated with one pore is enclosed. Further, pore branching and pore merging need to be considered. E.g., when contours of an upper height level are matched to contour it is check whether the added point are already an element of another point set of a pore, if yes the two pore branches are merged.

4. EXPERIMENTAL RESULTS

The results on the reconstruction approach of the porous structure based on milled surfaces are shown separately for a simple “pore” structure as the sunk-in star structure on the back of a 1 Euro Cent and on highly disordered pore structures as occurring on the thermal sprayed Al_2O_3-surface layers.

4.1 Reconstructing sunk-in star of a 1 cent euro coin

The reconstruction of the sunk-in star of the back of a 1 cent euro coin can be seen as the reconstruction of one single pore, without undercuts. The sunk-in star structure was measured with an objective lens with a 10 x magnification. Exemplary height data of one milled subsurface of the structure is shown in figure 3. To measure a single pore without undercuts the here presented reconstruction approach is unnecessary. However, this example is shown since the single steps are well comprehensible and illustrate the approach well.

In total the measurement data set contains data of 18. different heights. Therefore, the first step after acquiring and preprocessing the data is the registration. In figure 4 the result of the registration process of four subsequent subsurfaces is illustrated by a profile view, cut horizontally through the surface height data at y = 210 µm of all measured subsurfaces. Following to the registration each subsurface is segmented and the boundaries of the pore structure are derived, see figure 5(a). In this example one isoline is used per subsurface. The reconstructed structure based on the isolines is seen in figure 5(b). However, as mentioned due to no undercuts or enclosed pores that are newly exposed due to the milling, the reconstruction of the star structure does not provide any additional information.

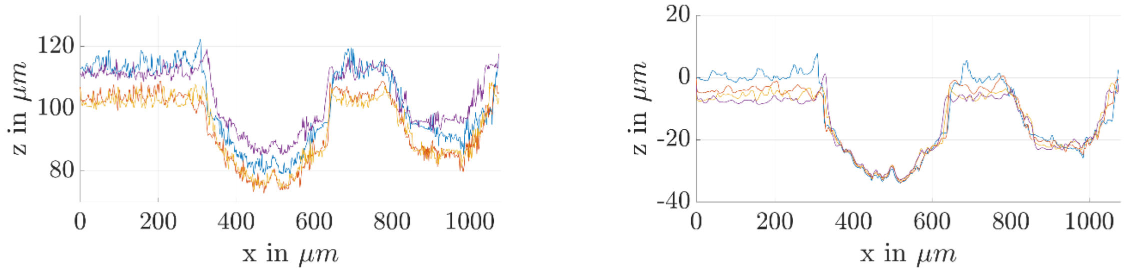
4.2 Reconstructing porous Al_2O_3-coatings

The Al_2O_3-coatings are measured with a 50 x magnification. The porous structure of the coatings is reconstructed very similarly to the 1 cent euro coin. The difference to the reconstruction of the cent is the number of isolines used per each subsurface. Instead of using just one isoline per subsurface the isolines are determined at a step size of the horizontal resolution of 0.277 µm.

Figure 6(a) illustrates the contour lines of the porous material. No merging of the contour lines is performed. Figure 6(b) shows isolines associated to one pore after the merging.

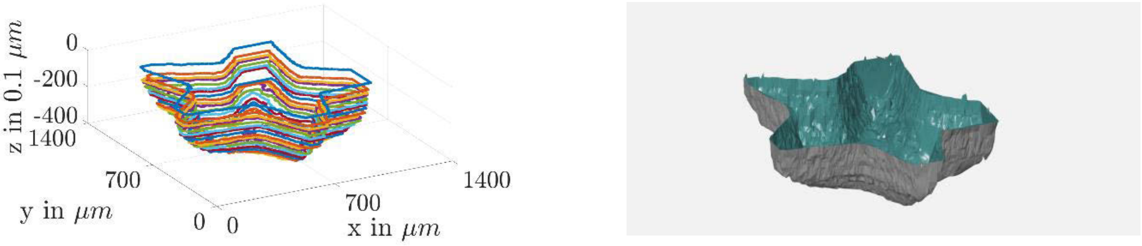
5. CONCLUSION AND OUTLOOK

An approach to reconstruct porous material was presented based on measuring a stepwise milled surface by fly cutting with a CLSM. It was shown, that based on isolines, the contours of the pore structure at stepwise increased heights, the pore structure can be reconstructed.
Future work addresses the actual characterization of the pore structure, like the connectivity



(a) Profile view at y = 210 µm of unregistered raw data. *(b) Profile view at y = 210 µm of preprocessed registered data.*

Figure 4: Result of registration process of four subsequent measured subsurfaces.



(a) Constructed isolines one per milled subsurface. *(b) Reconstructed structure based on the isolines.*

Figure 5: Reconstructed sunk-in star structure of a 1 cent euro coin.

of the pores and the single pore shape and size. Further, we would like to correlate these characteristics to the process parameters of the thermal spraying process and the tribological behavior of the surfaces.

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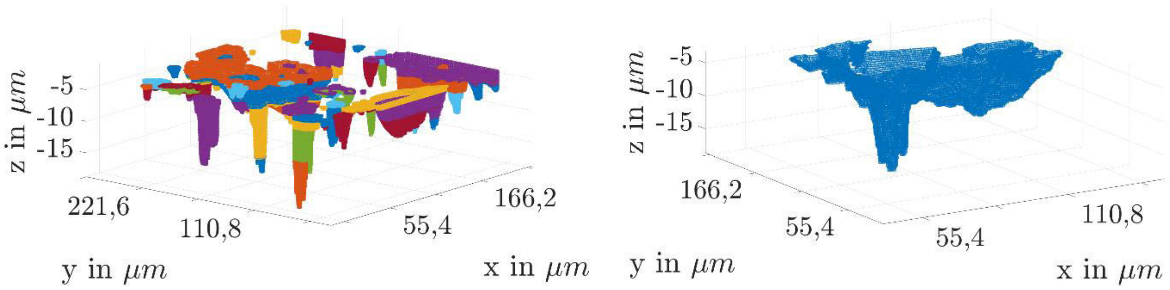

(a) Constructed isolines at a height difference of the horizontal resolution.

(b) Set of contour lines associated to one pore.

Figure 6: Pore structure of the Al_2O_3-coating.

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