LETTER

Microconical surface structuring of aluminium tubes by femtosecond laser processing

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Keywords: femtosecond laser, aluminium, tubes, nanostructuring, surfaces

Abstract

Femtosecond laser microstructuring is a convenient technology for the targeted surface functionalization of various materials. Commonly, the structuring process is performed on planar surfaces. Here, we investigated femtosecond laser structuring of aluminium tubes. Process parameters, i.e. the number of pulses per spot on the surface and the line distance, have been transformed from a line-by-line process on planar samples towards a helical process. The process is based on laser treating the rotating tube while the laser beam is moved along the axis of the tube. A significant difference of the surface structure obtained on a cylinder in comparison to the planar geometry is revealed. With exactly matching process parameters, a strong increase of the dimensions of the surface structures on aluminium tubes has been observed. With a typical parameter set to achieve microconically structured aluminium, the cone height increases from 5 to 24 μm and the cone-to-cone distance from 13 to 59 μm. The structure sizes were found to be unaffected from the diameter of the tube within a range from 12 to 40 mm. A possible explanation for the increased structure size is given by altered particle redeposition. Two different parameter sets have been transformed from a planar geometry to the cylindrical geometry. Deep black aluminium tubes providing hydrophobicity with a water contact angle up to 148° and a thermal emissivity up to 87% are demonstrated.

1. Introduction

Femtosecond (fs) laser structuring is one of the most versatile methods to adjust surface properties such as absorptivity (color), emissivity, wettability, adhesion and many more by a precisely targeted nanostructure. Recent reviews cover the choice of materials, structures and resulting surface properties [1–4]. The columnar structural motif, often termed microcones, spikes, whiskers or bumps, is the most common surface structure obtained by femtosecond laser micromachining (see figure 1) [2]. The resulting cones can act as light traps and provide lotus-like wettability. Aluminium as a common construction material with a high thermal conductivity is comparably difficult to nanostructure due to its low melting point of 660 °C [5]. Nevertheless, well-defined aluminium surface structures obtained by femtosecond laser irradiation are reported in literature and allow to achieve superhydrophobicity, strong light absorption and other properties [6–11].

Usually, plane samples are structured during femtosecond laser processing by scanning a pulsed laser beam on a surface in parallel lines at a constant distance to the focusing lens. Structuring of other geometries requires more complex laser setups to maintain the focus spot size (controlled by the distance to the focusing lens) and the scanning speed of the laser spot on the surface constant for the complete object. By self-focusing femtosecond laser filamentation [12], a curved surface in form of a section of an aluminium sphere has been homogeneously laser structured by moving the sample in x-y direction underneath a laser beam with a constant diameter to achieve a black surface [13]. In the area of biomedical applications, laser processing of tubes is a
A well-established method for the production of stents [14]. From a thin-walled tube, a coarse mesh based on thin rods remains after laser microcutting with femto- or nanosecond lasers [15–17]. These thin cylindrical meshes made from stainless steel and nitinol have subsequently been treated by femtosecond laser pulses while being rotated around their central axis to achieve a nanostructured surface coating to enhance biocompatibility or as drug delivery system [18–20]. Large steel cylinders used for embossing have been engraved according to digital templates by treatment with highly parallel picosecond laser beams while the cylinder spins around the central axis [21].

Here, we investigated the transformation of the laser parameters for femtosecond laser treatment of an aluminium plate towards an aluminium tube. To the best of our knowledge, we report on the first direct comparison of the microconical surface formed by self-organizing processes of femtosecond laser irradiation on the planar surface in comparison to the structure achieved by corresponding parameters on the spinning tube.

2. Materials and methods

Our general setup for femtosecond laser processing is based on the usual construction with a fs-laser source, a laser scanner to adjust spot size and position of the spot on a surface and has been reported previously [22, 23]. To allow for very slow movements of the laser spot in the direction of the sample tube, the laser scanner head (intelliSCAN 20, SCANLAB) has been mounted on a spindle axis (IAT-80-1036, Jenaer Antriebstechnik) powered by a linear actuator screw drive (23S31-0650-805L7-55(30), Jenaer Antriebstechnik) controlled by ECO Studio 3.2.0. Cylindrical samples have been mounted on the axis of a stepper motor (PD3-140-42-SE-485, Trinamic) controlled by the TMCL-IDE 3.0. Samples have been fixed on the central axis by wedged sliding blocks.

An AMPHOS 400 high power Yb:YAG laser system (maximal optical output power 400 W, limited to 200 W here) emitting linearly polarized light at a center wavelength of $\lambda = 1030 \text{ nm}$ and a pulse length of $\tau = 750 \text{ fs}$ has been used as laser source. The direction of polarization correlated with the scanning direction. The spot diameter $S_{\text{spot}}$ has been adjusted by the height of the 420 mm focal length $F$-theta lens of the laser scanner head (z-axis) and is given at the threshold width of $1/e^2$ of the gaussian beam profile. To minimize air breakdown, the focus point was always below the sample plane. A pulse repetition rate of $f_{\text{PRF}} = 1 \text{ MHz}$ has been applied. The pulse energy $E_{\text{pulse}}$, the spot size $S_{\text{spot}}$ and the resulting laser fluence $F$ have been set according to two in-house parameter sets for aluminium and are given in table 1 [23]. The scanning speed of the laser spot on the surface $v_{\text{scan}}$, the resulting number of laser pulses per spot on the surface $N$ and the line to line distance $L_{\text{line}}$ of multiple parallel lines processed on the surface has been controlled by the laser scanner for plane samples. For rotating tubes, the corresponding parameters are given by the rotational speed $\omega_{\text{cyc}}$ controlled by the stepper motor and the slow movement of the laser scanner head $v_{\text{scan,tube}}$ controlled by the linear actuator. Samples have been processed at air with an airflow of approx. $12 \text{ m s}^{-1}$ (rel. humidity $\text{RH} = 40\%$ at $20^\circ \text{C}$) through the processing chamber.

Owed to the varying machinability of the various types of aluminium, EN AW 1050A has been used for Al plates with 1 mm thickness (surface roughness $R_a = 0.3 \mu\text{m}$, rapa GmbH), EN AW 6060 for Al tubes ($D = 12 \text{ mm outer diameter}, 1 \text{ mm thickness}, R_a = 0.3 \mu\text{m}$, Gust. Alberts GmbH & Co. KG), and EN AW 2007 for a tiered aluminium rod ($R_a = 0.2 \mu\text{m}$). The tiered rod has been fabricated on a lathe at the machine shop of...
is transformed to a helical process by rotating a tube with the diameter of the planar surface is scanned with a laser spot of the size \( S_{\text{spot}} \). The basic concept of transferring laser parameters from a plane surface to a cylinder is presented in section 3. Results and discussion.

All samples have been cleaned by ultrasonification with acetone and deionized water before laser processing.

Surfaces have been analyzed by scanning electron microscopy (SEM) with an EVO MA10 (Zeiss) microscope operated at 10 kV and an integrated energy dispersive x-ray spectroscopy (EDX) unit (Bruker XFlash 6/30). Surface roughness and depth information has been measured by laser scanning microscopy (LSM) with a VK-1300 (Keyence) microscope operated at 408 nm. High resolution optical microscope images have been taken with a VHX-7000 4K digital microscope (Keyence). Thermal emissivity measurements have been performed at 50 °C and recorded by MIR imaging with a T885 (Testo) MIR camera. Contact angles have been taken from photographic images of water droplets with a volume of 10 \( \mu l \) on the samples.

All contact angles have been measured 30 s after application of the drop on the sample and 2 weeks after laser processing. Samples have been stored at ambient conditions in the dark.

### 3. Results and discussion

The basic concept of transferring laser parameters from a plane surface to a cylinder is presented in figure 2. The planar surface is scanned with a laser spot of the size \( S_{\text{spot}} \) in parallel lines with a scanning speed \( v_{\text{scan}} \) at a line distance \( L_{\text{line}} \). Assuming the curvature of a tube to be negligible for small spot sizes \( S_{\text{spot}} \), the scanning speed \( v_{\text{scan}} \) is transformed to a helical process by rotating a tube with the diameter \( D \) at a rotational speed \( \omega_{\text{cyc}} \) underneath the laser spot. The line distance \( L_{\text{line}} \) is resembled by the pitch of the helix, which is achieved by slowly moving the laser spot along the axis of the tube at the speed \( v_{\text{scan, tube}} \). The parameters are transformed according to equations (1) and (2).

\[
\omega_{\text{cyc}} = \frac{v_{\text{scan}}}{\pi \cdot D}
\]

\[
v_{\text{scan, tube}} = L_{\text{line}} \cdot \omega_{\text{cyc}}
\]

\[
\text{helix angle} = \arctan \left( \frac{v_{\text{scan, tube}}}{v_{\text{scan}}} \right)
\]

As a matter of fact, the laser processed line of a helix on a tube is slightly tilted from the parallel lines achieved on a plate. This deviation is quantified by the helix angle, which is given at 0° on the plate in comparison to the angle calculated by equation (3) on a tube. Since the orientation of the laser polarization affects the structure formation process [24], laser polarization was kept constant in scanning direction or in direction of rotation, respectively (see figure 2).

Laser parameters for the structuring of aluminium have been evaluated in detail in literature [5–11, 25]. Due to the low melting point of aluminium, comparably low laser fluences close to the ablation threshold \( F_{\text{tho}} \) of aluminium (\( F_{\text{tho}} = 0.11 \text{ J cm}^{-2} [25] \)–0.4 J cm \(^{-2} \) [5]) have been applied here, which is in accordance with literature to prevent smearing of the structures [25]. A microconical surface coverage providing black surfaces and superhydrophobicity was the desired target structure here. Consequently, and in accordance with literature, a high number of laser pulses \( N_{\text{pulses}} > 100 \) repeatedly hitting every spot on the surface has been applied to

### Table 1. Laser parameters applied on aluminium plates and tubes.

| parameter                  | lp | 1t | 2p | 2t |
|----------------------------|----|----|----|----|
| \( E_{\text{pulse}} \) (mJ) | 0.043 | 0.043 | 0.021 | 0.021 |
| \( f_{\text{rep}} \) (MHz)  | 1 | 1 | 1 | 1 |
| \( S_{\text{spot}} \) (mm)  | 204 | 204 | 97 | 97 |
| \( F \) (J cm\(^{-2}\))   | 0.13 | 0.13 | 0.28 | 0.28 |
| \( v_{\text{scan}} \) (m s\(^{-1}\)) | 0.5 | — | 0.05 | — |
| \( L_{\text{line}} \) (\( \mu m \)) | 8 | — | 13 | — |
| \( \omega_{\text{cyc}} \) (m s\(^{-1}\)) | — | 159.2D\(^{-1}\) | — | 15.92D\(^{-1}\) |
| for \( t_{12} \) (s\(^{-1}\)) | 13.3 | 1.274D\(^{-1}\) | — | 0.207D\(^{-1}\) |
| \( v_{\text{scan, tube}} \) (m s\(^{-1}\)) | — | 0.106 | — | 0.017 |
| for \( t_{12} \) (mm s\(^{-1}\)) | — | 0.106 | — | 0.017 |
| helix angle (°) (mm\(^2\)) | 0 | arctan(0.0025D\(^{-1}\)) | 0 | arctan(0.0041D\(^{-1}\)) |
| for \( t_{12} \) (°) | 0.012 | 0.012 | 0.019 | 0.019 |
| \( N_{\text{pulses}} \) | 408 | 408 | 1940 | 1940 |

* for a tube with an outer diameter of \( D = 12 \text{ mm} \).
achieve the columnar motif [26]. On laser setups with high repetition rates \( \sim 1 \text{ MHz} \), an increasing number of pulses is required to achieve the microconical surface [27]. A transfer of laser parameters for aluminium from a Ti:Sapphire laser setup to the high power high repetition rate system used here has been discussed in a previous publication [23]. The transformation of laser parameters from a plate towards a tube has been investigated with two different parameter sets. The first set (1) is based on a fluence of \( F = 0.13 \text{ J cm}^{-2} \) and a number of pulses per spot of \( N_{\text{pulses}} = 408 \) (see table 1). In accordance with literature [25], a homogeneous microconical surface coverage has been achieved, which is visualized by the scanning electron microscopy (SEM) images (figure 3(b)) of the deep black aluminium sample. Since visual appearance and microscopic structure matched with the expectation, laser parameters have not been optimized further. The cones act as light traps [9, 10], which explains the dark black appearance of the aluminium surface after laser treatment. The geometry of the microcones has been analyzed.
Further by laser scanning microscopy (LSM). An average cone distance of 13 μm and an average cone height of 4.5 μm has been determined (see table 2). The settings of the parameter set 1p have been transferred to an aluminium tube with a diameter of D = 12 mm as described above. A rotational speed of ω_0 = 13.3 s⁻¹ is necessary to match with the scanning speed of v_scan = 0.5 m s⁻¹ on the plate. Although 13 rotations per second are rather fast, it can be handled without vibrations by mounting the aluminium tube on a steel axis guided by a ball bearing (see figure 4c). The pitch of the helix is given by the small line distance L_line = 8 μm of the parameter set 1p and leads to a corresponding movement of the laser spot in direction of the tube with v_scan,tube = 0.106 mm s⁻¹. This speed is too low to be handled by the internal mirror system of the laser scanner head precisely. Consequently, the complete scanner was moved by a linear actuator screw drive. As discussed above, a small tilt of the laser processed line on the tube in comparison to the lines on a plate is caused by the helix angle. With the given parameters, a negligible helix angle of 0.012° above, a small tilt of the laser processed line on the tube in comparison to the lines on a plate is caused by the helix further by laser scanning microscopy. Parameter set 1t12, the aluminium tube presented a dark black appearance as expected for the transfer of laser parameters for black aluminium from a plate towards a tube. The SEM images present a homogeneously microconically structured surface (figure 3a). Looking at the surface structure more closely, reveals a significantly increased structure size in comparison to the laser processed plate (see figures 3b and c). The individual cones show a well-defined, spiky appearance.

Analyzing the dimensions of the cones by LSM quantifies the enlargement of the surface structures (table 2). The cones are arranged at an average distance of 59 μm and reveal an average height of 24 μm, which is approx. five times larger than the cones achieved on the plate. To further investigate the influence of the tube diameter on the structure formation process, a tiered aluminium rod has been used as target for laser structuring (figure 4a). The rod consists of 6 stages with a diameter from D = 15 to 40 mm. The laser parameters are recalculated for each diameter according to the generalized parameters given in table 1 to match with the parameter set 1p. The surface structures on each stage of the rod have been investigated by laser scanning microscopy and the area surface roughness parameter S_a has been determined as most precise measure for the dimensions of the surface structures. After laser processing, every segment 1t12–1t40 of the rod provided a deep black appearance visibly not differing from each other (figure 4b). The surface area roughness S_a = 7.2 μm of the segment with D = 15 mm matches well with the roughness S_a = 7.2 μm obtained for the aluminium tube with D = 12 mm discussed above. Interestingly, the rod diameter did not affect the structure sizes formed during laser irradiation with for each diameter adapted laser parameters. As example, figure 4c visualizes the height profile for the segment with D = 25 mm which is in good agreement with the cone height and distance obtained for the tube 1t12 as discussed above. The arithmetic mean of the surface area roughness S_a for all rod diameters is calculated to S_a = 6.94 ± 0.83 μm. Most of the values for S_a obtained for each segment are within the range of one standard deviation of the mean (figure 4d). In contrast, the surface area roughness of the plate determined to S_a = 2.3 μm differs significantly since structure sizes are greatly reduced as discussed above.

The centrifugal acceleration a_c caused by the rotational motion has been calculated for each diameter from the angular velocity ω_{rad} according to equation (4) and is plotted in figure 4d.

a_c = \frac{\omega_{rad}^2 \cdot D}{2} \tag{4}

The resulting g-force increases from 1 g to 3 g for the parameter set 1t with decreasing rod diameter from D = 40 to 15 mm. Consequently, the forces are rather low, yet may be sufficient to accelerate particles formed on the surface of the cylinder or to inhibit particle deposition. Since the cone size did not reveal a dependency on the rod diameter, we assume a more general influence of the structuring process on a spinning tube in contrast to the plane surface to cause the observed discrepancy. Particle deposition on the surface during femtosecond laser structuring has been described in literature to strongly influence the structure formation process. Two different
experiments have been discussed in literature concerning the influence of particle deposition on the laser structuring process on aluminium. In the first case, structure formation was investigated in dependency of the total pressure at air \([28]\). At a reduced pressure, particles formed during laser irradiation fly away from the surface while at higher pressures they are confined and redeposited close to the laser spot. As a consequence, the ripples formed on the aluminium surface revealed an increase in periodicity and width at a reduced pressure \([28]\). This observation matches with the increased cone width and distance observed on the rotating aluminium tube investigated here. On the other hand, the depth of the ripples has shown to increase with increasing pressure, which has been explained by particle redeposition on the surface and increased absorption mechanism of the particle coated area \([28]\). It has to be kept in mind that the oxygen content of the atmosphere is reduced with decreasing pressure, too, which minimizes chemical processes of the reactive gas atmosphere with the aluminium surface \([23, 28]\). In a second example, femtosecond laser structuring of aluminium has been performed in the additional presence of an argon plasma \([29]\). In this case, a single line written by femtosecond laser pulse did not show a difference of the ablated volume of aluminium in presence or in absence of an argon plasma. In contrast, laser structuring of an area treated with lines at a line distance of \(L_{\text{line}} = 70\ \mu\text{m}\) was significantly enhanced in the presence of the argon plasma. The width, height and distance of the microcones formed on the aluminium surface was found to be strongly increased in the presence of the plasma (figures 5(a), (b)). In fact, the surfaces obtained in presence of the argon plasma seem quite comparable to the surface obtained on the rotating aluminium tube here. The main reason for the improved structuring process has been given by efficient particle removal by the plasma \([29]\). It is noteworthy to point out, that the main effect is only observed in the case of structured areas of several adjacent laser processed lines, i.e. the second laser process conducted on the timescale of some seconds after the first line. Here, it is imaginable that the rotating surface minimizes particle redeposition, too, and consequently the dimensions of the surface structures are increased (figure 5(c)). Obviously, it takes one full turn of the tube until the adjacent line is processed, which gives enough time for particle removal.

To further investigate the effect of particle deposition, laser processing of aluminium tubes was repeated with a second parameter set. The parameter set 2 (see table 1) is based on an increased laser fluence of \(F = 0.28\ \text{J cm}^{-2}\) and a slower scanning speed of \(v_{\text{scan}} = 0.05\ \text{m s}^{-1}\). The lower scanning speed results in an increased number of laser pulses \(N_{\text{pulses}}\) per spot on the surface (see table 1). A comparable set of laser parameters providing a porous surface structure has been published previously \([23]\). Here, the laser fluence was increased to
force oxidation processes. For comparison, these parameters have been processed on an aluminium plate first. The formation of fine aluminium oxide powder is observed. The laser treated surface is covered with broad cones, which reveal a blurred appearance caused by a fluffy but stable layer visualized in the SEM image (figure 3(d)). Energy-dispersive spectroscopy (EDX) confirms the strongly increased oxygen content of 2p in comparison to 1p. The surface layer consist basically of aluminium oxide (1p: 38.8 atom-% O; 2p: 55.7 atom-% O; calcld. (Al2O3): 60 atom-% O). In accordance with the increased number of pulses \(N_{\text{pulses}}\), the resulting microcones are broader and larger in case of 2p (cone height: 17 \(\mu\)m) compared to 1p (cone height: 4.5 \(\mu\)m) [26].

Transferring the parameters to an aluminium tube with a diameter of \(D = 12\) mm according to the parameter set 2t12 given in table 1 leads to a homogeneous conical surface structure (see figure 3(a)). Comparing the surface obtained on the plate to the tube, the removal of the particle layer is evident (figure 3(e)). The SEM images reveal some particle residues on the conical structure, possibly due to a rather low rotational speed of \(\omega_{\text{cyc}} = 1.33\) s\(^{-1}\) derived from \(v_{\text{scan}} = 0.05\) m s\(^{-1}\). According to the LSM analysis, the height of the cones is strongly increased from 17 \(\mu\)m (2p) to 40 \(\mu\)m (2t12) by performing the laser process on the spinning tube and nearly reaches into a naked-eye-visible range. Consequently, the surface structure has been investigated by high-resolution optical microscopy. The surface of the aluminium tube 2t12 reveals a grayish appearance (figure 6(a)) in contrast to the deep black surfaces obtained with the parameter set 1t (figure 4(b)). Looking at the surface more in detail (figure 6(b)) presents the coarse conical structures formed on the surface contaminated with some white spots of...
residual particles. Looking at a single cone in detail by SEM reveals the porous oxidic substructure which is formed as top layer of every microcone (figure 3(f)).

Overall, we conclude that particle redeposition is hindered on the spinning tube or particle removal is favored during each turn of the tube. Both parameter sets 1t and 2t revealed a significant increase in the height of the microcones formed during laser processing on the spinning tube by a factor of 2.5–5.

Finally, the surface properties in terms of wettability and thermal emissivity of the plates and tubes laser structured with corresponding parameter sets have been compared. Water contact angles of laser treated surfaces have to be discussed with caution, since contact angles change in the course of time. Usually, freshly prepared surfaces are superhydrophilic, but slowly change to superhydrophobic due to condensation reactions of terminal M-OH groups and a coverage with non-polar organic residues within some days [30–33]. Here, contact angles have been taken two weeks after laser processing. Interestingly, the contact angles of the plates and the tubes are identical for the corresponding parameter sets (see table 2 and figures 6(c), (d)). The water contact angle of 1p/1t12 measured to Θ = 128° reveals a reduced hydrophobicity compared to the surface structure of 2p/2t12 with Θ = 148°. Consequently, the dimensions of the microstructure do not dominate the wetting behaviour in this case. Instead, the increased hydrophobicity of 2p/2t12 may be explained by the porous oxidic substructure favouring a higher density of organic residues becoming immobilized onto the surface during time [30].

Laser structured surfaces have shown to provide an increased emissivity [1]. Since aluminium oxide itself provides a high thermal emissivity of approx. ε = 80% at 50 °C [34], porous and oxide-rich surfaces obtained on aluminium plates by laser processing with parameter set 2p reach a thermal emissivity of ε = 85% (see table 2). Here, we processed aluminium tubes with a diameter of D = 12 mm and a length of 5 cm according to parameter set 2t12. The tubes have been plugged with sealant and filled with hot water. After a thermal equilibrium with a water temperature at 50 °C has been reached, the thermal emissivity has been measured by medium infrared (MIR) imaging (8000–14 000 nm). The tube showed a homogeneous MIR emittance at ε = 87% (figure 6(e)). The laser processed tubes did not reveal differences to the structured plates (see table 2) although the height of the microcones is significantly enlarged in case of 2t in comparison to 2p. Comparing the emissivity of the tube 2t12 with a blank aluminium tube (figure 6(e)) demonstrates the strong increase in thermal emissivity. Pure aluminium has a thermal emissivity of approx. ε = 9%, which is slightly increased on the tube to ε = 18% and visibly inhomogeneous due to surface impurities and reflections (figure 6(e)). The strong increase to ε = 87% by laser processing with parameter set 2t demonstrates the applicability of laser structured aluminium tubes for the thermal management of heat pipes and tubular reactors.

4. Conclusions

Usually, only planar surfaces are treated by femtosecond laser pulses to change their surface properties since complex geometries require an exact repositioning of the laser spot. Here, we investigated the adaption of the line-by-line processing scheme of planar samples to a helical process on aluminium tubes. By spinning the tube at a rotational speed ωrot underneath a laser beam, the number of pulses on each spot of the surface matches with the original parameter derived from vscan on the surface. The line distance Lline is resembled by the pitch of the helix and controlled by the slow movement of the laser beam along the axis of the tube during the structuring process. With exactly matching parameters, we observed a strong increase in the dimensions of the conical surface structure on the tube in comparison to the plate. For a typical parameter set to achieve microconical ‘black aluminium’, we found the height of the cones to increase from 5 to 24 μm and the cone-to-cone distance from 13 to 59 μm. An explanation for the enlargement of the surface structures may be given by hindered particle redeposition. If one would be interested to replicate the structure from a plate on a tube, we assume a line-by-line process with the laser scanning direction along the axis of the tube to be appropriate.

Depending on the chosen parameter set, well-defined properties such as deep black aluminium tubes, nearly superhydrophobic tubes or tubes with a high thermal emissivity of ε = 87 have been achieved here. Overall, we conclude femtosecond laser structuring by the helical process to proceed well and to allow for the routine surface functionalization of cylindrical samples.

Acknowledgments

We gratefully acknowledge financial funding from the Bavarian Ministry of Economic Affairs, Regional Development and Energy. We sincerely thank Torben Lüddecke for fabrication of the metal parts used for this work at the machine shop of the Clausthal University of Technology and Mohamed Arabi (Keyence Deutschland GmbH) for 4 K microscopic images. We sincerely acknowledge financial support by the Open Access Publishing Fund of the Clausthal University of Technology.
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