Femtosecond laser texturing of DLC-based coatings by DLW method with sub-micrometre precision

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Abstract
Laser texturing of surfaces may provide specific functionality, such as altering hydrophobicity, changing optical properties, or reducing friction and wear. For the latter, surfaces of parts are often coated with protective and/or solid lubricant coating, and texturing may further improve the tribological properties in both dry or lubricated sliding. New laser technologies, such as the direct laser writing (DLW) method using a femtosecond laser, allow the production of extremely precise textures directly into the coating. Here, we describe a method of preparing ultra-precise textures into diamond-like carbon (DLC) coatings on a large area. The textured topography was assessed by 3D laser scanning microscope, which confirmed the repeatability of fs laser processing. Raman spectroscopy mapping, SEM, and XPS were combined to investigate the effect of laser processing on DLC coating in terms of oxidation or structural changes. Traditional process (i.e., coating textured surface) often results in coating adhesion/cohesion failure due to deposition of sharp edges produced by texturing, whereas our approach eliminates this issue. Even complex textures inside the coating are fabricated with a fast speed of 10 s per mm² and a high precision in texture depth (tens of nanometers), unlocking many application fields in tribology or microfluidics.

Keywords Femtosecond laser · Laser texturing · Miniaturization · DLC coating · Precise ablation · Graphitization

1 Introduction
Many industrial areas face various pressure such as cost-effectiveness, environmental aspects, substitutability of different technologies, and its consequent flexibility of change. Surface treatments, namely laser surface texturing (LST) by ultra-short pulsed (USP) lasers, have been already used in many industrial fields: in the cutting tool industry [1] to improve the cutting process. Study by Hao et al. [2] solving a laser texturing of polycristaline diamon (PCD) tools by ns pulses in combination of fluorination process showed a reduction of cutting forces. Dependence of LST on behavior of cemented carbide tools is also shown in study [3], where state of minimal quantity of lubrication (MQL) increased the cutting performance by micro-/nano-textures. Other industrial fields solving the enhancement of fuctional properties by LST can be found in piston rings [4] and die preparations for metal forming [5], in bearings [6] for friction reduction and in other fields such as biomedical equipment [7] or storage devices [8].

Many scientific papers [9–15] dealt with LST of DLC coatings, which consisted of dimple shapes limited by
applied laser spots from $d = 10 \, \mu m$ [14] up to $30 \, \mu m$ [10] in rectangular/square patterns. The performed experiments showed the coefficient of friction (CoF) reduction when using ultra-short pulsed laser processing [10, 13, 15], instead of short-pulsed laser processing [16], where the heat-affected zone (HAZ) seems to be limiting improvement in tribological properties. Other studies [17–20] aimed to fabricate different textures, e.g., squares, lines, or circles in many possible patterns.

These studies were mainly focused on the impact of various densities of geometrical entities, but not on the achieved depth of textures, which did not allow a proper insertion of laser texturing into various places of the technological production chain. In the study of Dumitru et al. [12], laser texturing was performed in two variants: into the substrate before deposition of DLC coating and after the deposition. However, the depths of the texture were not provided. A review by Mao et al. [21] shows many studies on LST topics, but no one of them did not solved a fabrication of precise textures in term of accurate depth by fs lasers.

This study focuses on the fabrication of textures with defined geometrical entities and conditions by ultra-short pulsed (USP) laser system tuned to a pulsed length of 240 fs. Our main goal is to compare the feasibility of two texturing processes. In the first case, designed textures were micromachined directly into the substrate (tool steel 1.2379) and then, the samples were deposited by DLC coating (hereinafter textured-coated). In the second case, the laser fabrication of textures was made into a DLC coating (hereinafter coated-textured). A set of 5 different textures was designed for each combination of technological chain regarding to the miniaturization possibilities by direct laser writing (DLW) method, used laser system and its limits. We focus mainly on the precision of texture formation; the parameters were selected to produce the depth 500 nm in both cases.

Especially, texturing of the coating instead of the substrate provides significant technological advantages. Thanks to the high depth accuracy of USP laser texturing, we can fabricate channels for microfluidics. For multi-layered coatings, we can connect various coatings layers to build small lab-on-chip devices (e.g., multilayer with alternating conductive and dielectric layers). In tribology, fabrication of texture into coating instead of a substrate and subsequent deposition of a coating is extremely beneficial.

Moreover, the deposition of the coatings on textured surfaces such as steel is challenging. These processes are typically separated, and textured steel is prone to oxidation/corrosion, which may hinder the coating adhesion. As a consequence, the textured surface must be protected (typically by oils), but standard cleaning processes are not very effective for a textured surface. Therefore, the cleaning process is more complex and can lead to contaminated surface, which again compromises coating adhesion. Our process eliminates these drawbacks.

### 2 Experimental setup

#### 2.1 Material characterization

For the purpose of designed experiment, a DLC-based coating was chosen due to a large application range, e.g., in biological [22, 23], automotive [24], and other fields [25]. Specifically, tungsten-doped diamond-like coating, DLC-W, was deposited as a functional film. To improve adhesion between the steel substrate and DLC-W coating, a CrN interlayer was used. The thickness of the DLC-W coating measured by the ball-cratering technique was 2.2 µm, whereas the thickness of the CrN interlayer was 2.25 µm giving the total thickness of coating 4.45 µm. A polished tool steel 1.2379 (X153CrMoV12) was used as a substrate ($\varnothing = 20.8 \, \text{mm}$, thickness $= 5 \, \text{mm}$). Thanks to the high hardness, 60–62 HRC, and thus, high resistance to abrasive and adhesive wear, the substrate material finds many applications in bending and extrusion tools, thread rolling dies, cutting and punching tools, etc. The chemical composition of both materials is shown in Table 1.

The area for laser texturing was designed in the form of a square ($171.6 \, \text{mm}^2$) placed in the center of the sample. Then, a simple line was produced below the covered area as a marker for sample orientation.

#### 2.2 Laser system and setup

All textures were fabricated by a laser system equipped with a tunable ultra-short pulsed laser source, where pulse length $\tau_p$ in the range of 10 ps up to 240 fs can be set. A laser source (Light Conversion, Lithuania) generates an average output power of $P_m = 3 \, \text{W}$ at $\lambda = 515 \, \text{nm}$ wavelength. A repetition rate of used laser source works at $f_p = 200 \, \text{kHz}$. A laser beam with quality of $M^2 \leq 1.3$ is guided through all optical and opto-mechanical components (Fig. 1) to the galvo scanner with digital encoders (intelliSCAN by Scanlab).

Since miniaturization of textures was among the main goals of this experiment, it had to be taken into account two optical configurations after galvo scanner. Hence,
F-theta telecentric lens with a focal length of \( f = 100 \) mm (Fig. 1a) and fixed micro-objective with a numerical aperture \( NA = 0.26 \) (Fig. 1b) were installed to improve the resolution of fabricated textures. These optical setups with help of a beam expander (Fig. 1 – BE) are able to vary a laser beam to spot sizes in a range of \( d \leq 30; 11 > \mu m \), respectively \( d \leq 7; 4.5 > \mu m \). The overview of laser system parameters is presented in Table 2.

Due to the limited working field of micro-objective in plane XY, the final area of each sample has been stitched by stages movement. The working field of F-theta telecentric lens was sufficiently large for designed texture; therefore, no stitching of textured area was needed.

In all cases of laser texturing by galvo scanners, a definition of used scanning strategies must be defined. This scanning strategy is characterized in Fig. 2 by pulse overlap \( S_p \), which can be applied by a range of \( S_p < 0; 100 \% \) and hatching step \( H < 0; 100 \% \).

The pulse overlap \( S_p \) is defined by applying scanning speed \( v_f \), repetition rate \( f_p \) and laser spot \( d \) according to Eq. 1. To calculate hatching step \( H \) (Eq. 2), a parameter \( p_y \) is defined as a pitch between two consecutive hatches.

\[
S_p = \left(1 - \frac{v_f}{d \cdot f_p}\right) \cdot 100\% \tag{1}
\]

\[
H = \left(1 - \frac{p_y}{d}\right) \cdot 100\% \tag{2}
\]

### 2.3 Designed types of textures

As mentioned above, two basic types of textures were designed. The first type is represented by octagons (Fig. 3a), which were fabricated as pillars (Fig. 3a with line hatches only) and pockets (the same figure but with dashed hatches). Both octagonal textures were designed in dimension ratio of constraints \( t \) and \( s \) according to Eq. 3:

\[
\frac{s}{t} \approx 6 \tag{3}
\]

### Table 2 Laser system specification used for experiments

| Wavelength | 515 nm |
|------------|--------|
| Pulse duration | 240 fs |
| \( M^2 \) (TEM\(_{00}\)) | \( \leq 1.3 \) |
| Repetition rate | 200 kHz |
| Maximum energy (Ep) | 15 \( \mu J \) |
| Polarization | circular/linear |
| Entrance beam size for F-theta (\( f = 100 \) mm) | \( \sim 5.32 \) mm (if \( BE = 3x \)) |
| Entrance beam size for microscopic obj. (NA 0.26) | \( \sim 2.66 \) mm (if \( BE = 1.5x \)) |
| Laser spot (F-theta \( f = 100 \) mm) | 11 \( \mu m \) |
| Laser spot (microscopic obj. NA 0.26) | 4.97 \( \mu m \) |
Another texture consists of rectangles (Fig. 3b) reminiscent to anti-slide texture. There is an assumption of model simplification due to miniaturization, where instead of model rectangles, only lines for manufacturing are used. The rectangle shapes are created themselves according to used laser spot without sharp corners.

2.4 Parametrical programming of designed textures

The parametrical algorithmizing was chosen for better downsizing of the fabricated textures. By this type of programming, the smallest possible size with the current optical setup can be easily set. To ensure the scalability of designed textures according to Fig. 3, several dimensions had to be locked in certain ratios. Octagonal textures were fabricated in dimension ratio according to Eq. 3, whereas anti-slide texture (Fig. 3b) has the following fixed dimensions:

\[ b = c \]  \tag{4}  
\[ m_x = m_y \]  \tag{5}  

Equality operators in Eqs. 4 and 5 state that anti-slide texture is created by 3 rectangles, which are inscribed in a square. Another parameter for anti-slide texture is \( w \), which defines the size of separation according to Eq. 6:

\[ w = m_x - c = m_y - b \]  \tag{6}  

This separation parameter \( w \) is used to vary texture density; three various anti-slide textures were thus produced by changing the parameter \( w \) solely.
2.5 Devices for experiment evaluation

During the texture fabrication, a 3D laser scanning microscope OSL5000 by Olympus was used for the basic assessment of texture topography like dimensions, depths, and quality of created textures. Detailed morphology of the produced textures was obtained by Mira 3 XMU (Tescan, Czech Republic) scanning electron microscope (SEM). Elemental surface composition was measured with the energy-dispersive X-ray spectroscopy (EDS) (Oxford Instruments, United Kingdom). Furthermore, the DLC-W coating structure was measured with Raman spectroscopy (Horiba Scientific, France) ($\lambda_{\text{laser}} = 532$ nm).

The possible effect of thermal load on the coatings that were ablated after the deposition was assessed by Raman spectroscopy mapping (on the surface) and by X-ray photoelectron spectroscopy (XPS) depth profiling. X-ray photoelectron spectroscopy (XPS) analyses were performed on the ablated and non-ablated area using a Kratos XPS spectrometer (Shimadzu) equipped with a monochromated Al Kα X-ray source ($h_\nu = 1486.6$ eV). During the measurements, the base pressure inside the XPS chamber was kept constant at around $5 \times 10^{-9}$ Torr. High-resolution XPS spectra were recorded with a 0.1-eV step. The surface etching was performed using Minibeam 6 Ar Gas Cluster Ion Source (GCIS, Kratos Analytical) operated at a cluster mode (cluster size of Ar500+, impact energy of 10 keV, equating to partition energy of 20 eV per atom). For the ion beam, a raster size of $2 \times 2$ mm was used for etching experiments. Etching time was 5 min per step; 20 etch steps were conducted in total. The deconvolution of the high-resolution C 1 s spectra was carried out using Tougaard background and with the assumption that C 1 s spectrum of such type of coating is represented by the following components: C-W, C=C (sp2), C–C (sp3), C-O, and C=O [28–32].

| No of texture | w, µm | Density, entities, mm$^{-2}$ |
|---------------|-------|-----------------------------|
| Anti-slide 1  | 2.2   | 10 800                      |
| Anti-slide 2  | 5     | 7 500                       |
| Anti-slide 3  | 10    | 4 800                       |

3 Results

3.1 Optical setup for miniaturization and fabrication debugging

As defined in Table 3, in two types of focusing, optics were used for the experiment. In the first case, the F-theta telecentric lens was applied (Fig. 1a). To generate the smallest spot size (11 µm), the beam expander was set on the highest value of its magnification ($3 \times$).

3.1.1 Octagonal texture with pillars

The first texture of octagonal pillars was tested in a small area with a spot size of $d = 11$ µm. Basic dimensions of octagons according to Eq. 2 were set after a few step testing. Final dimensions according to Fig. 3a are as follows: $t = 8.97$ µm and $s = 48.05$ µm.

After defining basic dimensions $t$ and $s$, other details had to be taken into account. When fabricating a rectangular pattern by whole closed octagons (Fig. 4a), the result had many defects in the form of deeper places where vertical and horizontal lines of octagons were overlapped (doubled). It means that part of the texture was fabricated twice, penetrating the whole thickness of the DLC-W coating. These
areas are clearly visible in yellow circles in Fig. 4a with cut-section A-A evaluation, where the depth was 0.984 µm. Blue lines in the topographical image show the undercuts of upper layer.

From these reasons, the octagon model was slightly modified to avoid these defects, as shown in the fabrication scheme in Fig. 4b. Modification consists of opened and interrupted contour, which in the case of a rectangular pattern prevents lines from doubling. The principle is characterized in schematic part in Fig. 4b. The first open contour fabricated is presented by a solid line, second one by a dashed line, third one by a dashed line with one dot, and lastly a dashed line with two dots. The evaluation of cut-section B-B showed a depth value 0.510 µm.

3.1.2 Octagon texture with pockets

Another texture with octagons has an inverse shape against the first one — the pockets instead of pillars were fabricated. After the determination of \( t \) and \( s \) values (Fig. 3a), two types of optics were tested for shape comparison and details. This case is compared in Fig. 5, where F-theta lens with spot size \( d = 11 \) µm (Fig. 5a) shows faster texturing technique with worse shape quality of pocket edges. Installation of microscopic objective (NA 0.26) with spot size \( d = 5 \) µm presents slower texturing technique with better edge quality and more accurate shape result (Fig. 5b).

Each shape of octagonal pocket was fabricated first by contour and then hatched in a horizontal direction in Fig. 6. Schematic overviews of area 0.2 × 0.18 mm where parametrical algorithm uses different separation parameter \( w \): a) \( w = 2.2 \) µm, b) \( w = 5 \) µm, c) \( w = 10 \) µm. Each overview has parameter \( b \) set as constant value. d) Fabricated anti-slide texture with separation value of \( w = 2.2 \) µm where texture stitching is visible.
3.1.3 Anti-slide textures and their modifications

For anti-slide texture fabrication, the optical setup according to Fig. 1b was used thanks to microscopic objective NA 0.26 and its possibility of better miniaturization.

In the case of the last designed template of texture, it was essential to set parameter b, which represents the length of the basic rectangle (depicted in Fig. 3b). After short testing, the value of b parameter was set to $b = 14.8 \, \mu m$. Then, the separation parameter $w$ was determined in certain dimensions, which influenced the anti-slide texture density. The 3 types of anti-slide textures were designed by varying separation parameter $w$ in a range of $<2.2; 10>$ $\mu m$. When the smallest separation parameter was set ($w = 2.2 \, \mu m$), the density of used geometrical entities (one entity = one rectangle) reached the value of 10,800 per 1 mm$^2$. In the case of the largest separation parameter ($w = 10 \, \mu m$), the density of geometrical entities reached the value of 4800 per mm$^2$. All separation parameters $w$ and densities of anti-slide textures are summarized in Table 3.

Figure 6 shows an overview of these textures with a dedicated $w$ parameter setup in the parametrical model for area $0.2 \times 0.2$ mm.

As mentioned before, the textured area on the sample ($13.1 \times 13.1$ mm$^2$) was stitched due to the limited working view of the microscopic objective with NA 0.26. The scanning field was reduced to $0.4 \times 0.4$ mm per stitched area to provide constant fabrication conditions. For the whole area, $33 \times 33$ fields were stitched.

Figure 6d shows the fabrication of anti-slide texture 1 with $w = 2.2 \, \mu m$, where details of stitched areas ($0.4 \times 0.4$ mm) are clearly visible.

3.2 Investigation of sub-micrometer ablation

After determining optimal optical setup for all textures, texture-specific approaches were required to reach expected depth. Here, we present in detail sub-micrometer ablation for coated-textured type of production chain; the
fabrication of textured-coated samples follow the same methodology. All depth measurements were performed by 3D laser scanning microscope OSL5000 from Olympus; the depth is an average of 9 measurements.

### 3.2.1 Octagonal texture with pillars

To set the required depth, two different strategies are needed. The first one uses constant laser fluence $F$ and a spot overlap $S_p$; the depth depends on the number of repetitions (i.e., passes over the same place) (Fig. 7a). With spot size of $d = 11 \, \mu m$, the laser fluence is $F = 1.05 \, J/cm^2$. Scanning strategy, particularly a spot overlap $S_p$, was also set as a constant value ($S_p = 90\%$). Figure 7a shows a linear dependence of the depth as a function of repetitions; the ablation rate for coating $z_{abl}$ is constant and closer to 0.2 $\mu m$. As the number of repetitions is an integer, more subtle parameter set is required to reach a precise thickness. For example, to reach a depth of 0.5 $\mu m$, we can use two repetitions to remove the $0.38 \pm 0.079 \, \mu m$ and then to use our second strategy.

A spot overlap, $S_p$, may by adjusted to further increase the depth. As shown in Fig. 7b, we can vary $S_p$ in a range of $S_p < 90.9; 92.3 > \%$ representing the scanning speed $v_f$ in range of $<200; 170 >$ mm/s. Using the curve shown in Fig. 7b, we can estimate $S_p$ to reach the set depth 0.5 $\mu m$.

### 3.2.2 Octagon texture with pockets

This type of texture consists of two operations: pockets and dimples fabrications. This texture fabrication was split due to different technological methods. Pockets were ablated by scanning strategy with defined hatching, and dimples were created by single pulses only. For both geometrical entities, a spot size $d = 5 \, \mu m$ was used.

Accurate depth for pockets was obtained in two-step iteration. For both iterations, laser fluence $F$ was set as constant value $F = 2.55 \, J/cm^2$, spot overlap $S_p$ was also set constant at 85% (that corresponds to scanning speed of $v_f = 150$ mm/s). All constant parameters and dependencies are shown in Fig. 8a.

An optimum hatching step $H$ to reach set depth was established as two steps. In the first step, hatching step $H$ was set to 70, 80, and 90%, which allowed to estimate approximate $H$ to reach the required depth (0.5 $\mu m$). Then, in the second step, a smaller range of $H (74–78%)$ was used. For $H = 74\%$, the depth was $0.52 \pm 0.013 \, \mu m$, i.e., only 20 nm from the required depth. Figure 8b shows the depth as a function of laser fluence; only one pulse $N = 1$ was applied.

### Table 4 Comparison of the 2D measurement of samples

|       | $t$, $\mu m$ | $s$, $\mu m$ | $b$, $\mu m$ | $w$, $\mu m$ |
|-------|--------------|--------------|--------------|--------------|
| Octagons — pillars | 8.97         | 48.05        | -            | -            |
| Octagons — pockets  | 10.65        | 55.82        | -            | -            |
| Anti-slide 1        | -            | -            | 14.82        | 2.28         |
| Anti-slide 2        | -            | -            | 14.85        | 4.95         |
| Anti-slide 3        | -            | -            | 14.84        | 9.46         |

Therefore, any depth can be achieved by a combination of repetition and spot overlap strategies. According to the methodology above, two depths (0.2 and 0.8 $\mu m$) were tested. The 0.2 $\mu m$ depth was reached at $1 \times$ repetition rate and overlap $S_p$ 91.6% with a standard deviation of $\pm 0.065 \, \mu m$. For a depth of $0.8 \pm 0.053 \, \mu m$, a setting of $4 \times$ repetitions with an overlap $S_p$ of 91.0% was required.
in testing. Again, we can quickly select the fluence to reach the required depth.

### 3.2.3 Anti-slide textures

The last texture with a different density setup required only one round of testing. Due to the very small sizes of designed geometrical entities (parameter $b = 14.8$ µm), a slow scanning speed of $v_f = 40$ mm/s was chosen, corresponding to 96% of spot overlap $S_p$. Then, the laser fluence was varied in a range of $F < 1.02; 1.53 > J/cm^2$ (Fig. 9).

### 3.3 Evaluation of fabricated textures

#### 3.3.1 Assessment in XY plane

All 2D measurements were performed by 3D laser scanning microscope OSL5000 (Olympus). Applied magnification for both octagonal textures was $2336 \times$. In the case of anti-slide textures, a magnification of $3504 \times$ was used. Dimensions are taken from randomly chosen places on samples and are summarized in Table 4. Measured dimensions to designed textures correspond to parameters according to Fig. 3.

#### 3.3.2 SEM and EDS analysis

From the Fig. 10, textures (type: textured-coated) remained well visible after the depositions, i.e., coating did not overfill the pattern. Moreover, from Fig. 10, it follows that coating deposited on the edges of the structures is smooth, i.e., the height difference did not cause cracking of the coating.

Next, Fig. 11 shows the SEM micrographs from the samples which were first coated, then ablated.

From Fig. 11a, it can be seen that edges of octagonal pillars are rough, as a consequence of the laser ablation. On the contrary, edges of the pockets (Fig. 11b) are smooth and the transition to the unablated surface is gradual. Anti-slide patterns (Fig. 11c–e) look regular and well-made, in terms of both orientation and the shape. These findings are probably related with applied laser spots. Only octagonal pillars (Fig. 11a) have been fabricated by $d = 11$ µm. The rest of textures have been created by $d = 5$ µm.

EDS measurements were performed together with the SEM analysis; they are summarized in Table 5. As expected, EDS results for textured coated samples are identical for all the produced structures and identical to coating deposited on flat substrate (C: 93 at. %, W: 2 at. %, and O: 5 at. %). For pillars, the composition varied in selected areas, so we present the range of composition measured for individual elements. Tungsten content is always higher, but it is expected due to a gradient nature of the coatings with decreasing of W content towards the surface. The oxidating content varies as well, but we can conclude here that EDS did not indicate any significant compositional change of the ablated coating. To investigate possible structural change, Raman spectroscopy and XPS were employed.

#### 3.3.3 Raman and XPS analysis

Raman spectra were recorded from both batches of the samples. For the textured-coated samples, spectra were recorded in several positions on different samples and

| Table 5 | EDS composition of the samples from the coated-textured batch |
|---------|-------------------------------------------------------------|
| Structure | Place of measurement | C (at. %) | W (at. %) | O (at. %) |
| All textured-coated surface | Any position | 93 | 2 | 5 |
| Octagonal pillars | Ablated (i.e., area next to the pillar) | 83–92 | 6–9 | 2–8 |
| | Non-ablated (top of the pillar) | 86–96 | 6–7 | 1–7 |
| Octagonal pockets | Ablated (i.e., bottom of the pocket) | 85 | 7 | 8 |
| Anti-slide patterns | Non-ablated (i.e., next to the pocket) | 91 | 6 | 3 |
no major differences were found. Figure 12a shows representative Raman spectra from octagonal pillars. The main characteristics of Raman spectra (Fig. 12a) are weak D peak ($\approx 1337$ cm$^{-1}$, $I_D = 0.35$), strong G peak ($\approx 1538$ cm$^{-1}$, $I_G = 0.86$), and no evidence of W oxide species in the coating. $I_D/I_G$ ratio was calculated as 0.41.

The same texture (octagonal pillars) from the batch of coated-textured samples was also characterized by Raman spectroscopy (Fig. 12b). Spectra were taken from the areas where the coating was ablated and an additional spectrum was taken from a non-ablated area as a reference. As expected, this reference is identical to spectra from the textured-coated batch. Figure 12b shows that the ablation caused identical changes to the carbon structure in the DLC coating for both octagonal pillars and octagonal pockets. As a consequence of ablation, carbon structure at the surface ($D \approx 1336$ cm$^{-1}$, $I_D = 0.42$, $G \approx 1587$ cm$^{-1}$, $I_D = 0.44$, $I_D/I_G = 0.95$) partially graphitized.

We employed Raman mapping to show the distribution of graphitization area across the coated-textured surface. In this way, we can identify the local distribution of thermally induced structural transformation of DLC coating. Mapping was performed on the pockets and pillars textures at the unablated areas.

Figures 13 and 14 show Raman maps on the octagonal pillars in perpendicular and parallel directions to the sample orientation. The graphitization of the DLC coating on top of the pillar is mostly symmetrical; more importantly, graphitization is localized to the very edges of the structure. Hence, the laser ablation did not cause major changes to the unablated DLC coating. Identical analysis was performed for the octagonal pockets and the results are shown in Figs. 15 and 16.

On octagonal pockets, changes of the surrounding DLC structure are asymmetrical (Figs. 15 and 16) due to few reasons. Namely, where the structures are closest to each other (horizontally oriented Raman map, Fig. 15), the graphitization of the deposited DLC coating is symmetrical on both sides. On the other hand, Fig. 16 shows that the vertically oriented Raman map shows changes between the two-neighboring structure, which are asymmetrical.

Finally, XPS analysis of the spectrum taken from the surface of as-deposited coatings (Fig. 17a) reveals a broader shape compared to one after etching (Fig. 17b). Broadening towards higher binding energies is attributed to surface contamination with carbon oxides (single and double bonded) [29–34]; more prominent right (low binding energy) shoulder can be caused by a presence of tungsten carbides [32, 35]. The 5-min etching led to partial removal of the contaminations, while after 100-min etching, the shape and intensity of the spectra were already representative for bulk coating. Figure 17a shows the C 1 s spectra taken from the as-deposited coating; a deconvolution of the spectrum reveals components centered at 283.3 (C-W), 284.2 (C=C), 285.1 (C–C), 286.5 (C-O), and 287.8 eV (C=O) [29, 32–38]. The slight shift of the C-W peak towards higher binding energy is typical for the coatings with the excess of carbon [30, 39]. The same trend was observed in the case of laser treated coating (Fig. 17b). The peaks centered at analogous positions 283.3 (C-W), 284.2 (C=C), 285.1 (C–C), 286.4 (C-O), and 287.5 eV (C=O) and very similar peak intensity ratios were observed.
We have demonstrated that the laser can be used to fabricate complex shape textures inside DLC coating with precise shape and depth. We provide a detailed strategy on how to set laser parameters to produce required topographic features (pillars, octagons, or anti-slide tiles) quickly and reliably. Moreover, we have suggested further improvements, e.g., negligible undercuts in octagonal textures with pillars (Fig. 4b) could be further reduced by investigating galvo-scanner delays or by shortening of certain lines in a modified octagonal model.

We compared two technological approaches; coating applied on the textured steel sample (Fig. 10) and texture produced onto the coated surface (Fig. 11). As expected, the coating applied on a textured surface mimicked the texture and showed identical chemical composition and structure to that on a flat substrate. On the other hand, the EDS analysis indicated that texturing of the coated sample did not have the same effect on the octagonal pillars and pockets textures.

**4 Discussion**

![Fig. 13 Raman mapping of octagonal pillar — horizontal direction](image)

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![Fig. 14 Raman mapping of octagonal pillar — vertical direction](image)
Evaporation of material during laser ablation did not alter the chemical composition significantly as shown in Table 5.

The Raman mapping indicated an expected local graphitization of ablated fields in coated-textured types of samples. For octagonal pillars, the graphitization was localized only nearby the edges, whereas, for octagonal pockets, the dependence of hatching strategy setup on graphitization was observed. The laser processing setup (horizontal direction of hatching shown in Fig. 5b) resulted in symmetric graphitization (see Raman map in Fig. 15) due to bi-directional mode, where the beginnings and ends of every line were continuously swapped. Therefore, accelerating and decelerating of galvo mirrors had no major role. Asymmetricity of graphitization in pockets for the vertically oriented map is caused by two factors. The first is hatching strategy (from bottom to top), resulting in heat accumulation phenomena [40, 41].

The second factor is the fact that hatching lines were uncentered on the pocket contour, which created different gaps between the bottom part of the contour and first hatching line (up to 0.0025 mm) and the top part of the contour and last hatching line (up to 0.0015 mm). Note that defined gap between sloped/vertical parts of contour and beginnings/ends of hatching lines is 0.0013 mm.

Since the measurement area of XPS is significantly larger than that of Raman spectroscopy, the signal from a very limited grafitized zone was mixed with the one from the unaffected zone. As a result, XPS analysis of as-deposited and laser-treated coating surfaces did not indicate any significant changes in chemical composition or bonding.

From view of duration of texture fabrication, there might be used many approaches for getting some time reduction: from tuning up the laser process: optimization of process

**Fig. 15** Raman mapping of octagonal pocket — horizontal direction

**Fig. 16** Raman mapping of octagonal pocket — vertical direction
parameters (laser fluence $F$, spot overlap $S_p$, hatching step $H$ and others), bigger stitching field, higher scanning speeds of galvo mirrors (incl. galvo delay setup) up to different optical setups. After initial testing of new textures by DLW method, other possibilities promising bigger productivity of scanning devices can be applied (e.g., interference patterning DLIP [42, 43], diffractive optics elements, e.g., spatial light modulators SLM [44–46]). DLW method is not suitable for serial production of textures regarding to the durations, but it is essentially desirable for standard prototype texturing.

The duration of each fabricated texture differs in correlation of used optical setup and the level of miniaturization. All durations are involved in Fig. 18.

If the type of used optical setup is considered, the F-theta telecentric lens generally has the shortest durations against microscopic objective with NA 0.26. For that reason, the duration per unit of area was 7.9 s/mm² for the textures of octagonal pillars. Contrary to this option, the F-theta lens does not provide such an advanced level of miniaturization. If the textures fabricated by microscopic objective are compared, then, textures with octagonal pockets have the worst duration per unit of area (24.5 s/mm²). The biggest geometrical complexity of octagonal pockets (fabrication of contour, hatching processing, and dimples creation) took the longest duration per area unit in comparison to other textures. Anti-slide textures show the decreasing dependence of duration per area unit on increasing separation parameter $w$. The duration for the highest density of anti-slide texture ($w = 2.2$ µm) is 17.5 s/mm². The lowest density of anti-slide texture ($w = 10$ µm) was reduced by about 42% (9.1 s/mm²) in comparison with the highest density.

Fig. 17 High resolution carbon 1 s spectra of a as deposited coating, and b laser treated coating.

Fig. 18 Overview of fabrication durations per 1 mm².
5 Conclusion

This study demonstrated the fabrication feasibility of user-defined textures and their scalability in combination with the achievement of sub-micrometer depths regardless of the type of chosen technological chain. In summary, 10 textures (5 textures for each technological chain) have been defined and fabricated by femtosecond pulsed laser system. After initial debugging of texture shapes from the view of miniaturization, the textures have been created in lower tens of micrometers. Texture dimensions have been slightly different due to various placement of laser texturing in technological chains. Hence, the deposition process had a direct impact on final texture dimensions. The evaluated depths for both types of technological chains are ranging in 0.484 up to 0.544 µm. All investigated attributes have been kept, well-defined, and fabricated.

The Raman spectroscopy showed that graphitization of the DLC-based coating caused by laser texturing is a very local phenomenon observed only on the very edges of the pattern elements (octagonal pillars and pockets). XPS spectroscopy revealed no significant changes in surface chemistry when large areas of laser-treated and pristine areas of the DLC-based coating were compared. SEM analysis of the laser textured surface showed a homogenous and featureless structure of the coating without cracking on the pattern edges.

The influence and potential benefits of differently designed technological chains, where the laser texturing according to this study was applied, will be the object of further experiments and investigations.

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Declarations

Ethics approval Not applicable.

Consent to participate Not applicable.

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