



## Structure and friction properties of laser-patterned amorphous carbon films



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### ABSTRACT

In the paper we report on laser surface modification of super hard micrometer-thick tetrahedral amorphous carbon (ta-C) films in the regime of single-shot irradiation with KrF laser pulses (wavelength 248 nm, pulse duration 20 ns), aimed at investigations of the laser-induced changes of the structure and surface properties of the ta-C films during graphitization and developing ablation processes. Based on the analysis of surface relief changes in the laser-irradiated spots, characteristics of the single-shot graphitization and ablation of the 2-μm-thick ta-C film are determined. Using Raman spectroscopy, it is found that during the graphitization regime the structure transformation and growth of graphitic clusters occur according to the relationship  $I(D)/I(G) \propto L_a^2$ , but after reaching the ablation threshold the Tuinstra-Koenig relationship  $I(D)/I(G) \propto 1/L_a$  describes further growth of the graphitic cluster size ( $L_a$ ) during developing ablation of the ta-C film with nanosecond pulses. The maximal size of graphitized clusters is estimated as  $L_a = 4\text{--}5$  nm. The studies of nanomechanical properties of laser-patterned ta-C films using the lateral force microscopy and force modulation microscopy have evidenced lower friction forces (between diamond-coated tips and film surface) and lower stiffness in the laser-graphitized areas. The laser-produced graphitic layer acts as a solid lubricant during sliding of the diamond-coated tips on the ta-C film surface in ambient air (~50% RH); the lubricating role of adsorbed water layers is suggested to be significant at low loads on the tips. The results of this work demonstrate that the UV laser surface texturing in the regime of graphitization is a promising technique to control the friction and surface elasticity of super hard amorphous carbon films on the micro and nanoscale.

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### 1. Introduction

Owing to unique tribological properties of tetrahedral amorphous carbon (ta-C) films [1,2] and great progress in producing ta-C coatings on an industrial scale, their applications are of increasing demand especially in automotive industry [3]. For the auto industry, the problem of energy consumption due to friction is a topical one, as one-third of the fuel energy is used to overcome friction in the engine, transmission, and brakes [4]; so to reduce the fuel consumption, friction in various vehicle components needs to be reduced. One of efficient ways to improve friction and wear properties of hard materials and coatings is laser surface texturing (LST) [5] which can be applied to produce a variety of surface micro-dimples functioning as reservoirs for liquid or solid lubricants and traps for wear particles [6–15]. Frictional performance of hard ta-C and other diamond-like carbon (DLC) films can additionally benefit from the laser-induced structure modification (graphitization)

of the surface layers [13–17] as the surface graphitization was suggested as a lubrication mechanism of diamond and DLC films [18–22].

Recent achievements of laser interference patterning of ta-C films in the regime of surface graphitization showed different friction performance of the films of different thickness varied from ~80 nm (nanopatterning) [14] to 2.5 μm (micropatterning) [13,15]. For nanopatterning, the friction of periodic graphitized surfaces (sliding against a 100Cr6 steel ball) was decreased by ~50% compared to the original film [14], that was attributed to a reduced area of contact between the frictional partners. On the other hand, micropatterning of 2.5-μm-thick films showed that the laser-induced graphitization (surface swelling) resulted in an increased friction due to the ball sliding on softer graphitized amorphous carbon [13,15]. To clarify the effect of surface graphitization on the friction properties of laser-patterned DLC films we proposed [23] to use a technique of friction force microscopy [24,25] for imaging of frictional forces both in the laser-graphitized swellings and original film surface on the micro and nanoscale. Interestingly, we found lower friction forces in laser-graphitized regions during scanning of a Si tip over

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laser-micropatterned areas [23], opposite to the results of tribological tests reported in [13,15].

The structure of the laser-modified DLC surface is that of nanocrystalline graphite as follows from most previous studies of laser-irradiated DLC films [8,13–16,26 and refs. therein]. In the structure analysis, visible Raman spectroscopy is usually applied to study the evolution of the  $sp^2$  phase in amorphous carbon films in dependence on deposition and post-growth treatment parameters [26–31]. Typical post-deposition annealing temperatures are limited by the values of  $\leq 1300$  °C [27,28], while local laser-induced graphitization of the near-surface regions of micrometer-thick ta-C film gives a unique opportunity to study structural changes as a function of temperature varied from the graphitization temperature to the sublimation temperature, i.e. from 1000 to 4000 K [15]. Little data is still reported of detailed studies of the influence of high surface temperatures on Raman characteristics of ta-C films (such as the position and width of the D and G peaks, the  $I(D)/I(G)$  ratio) and their correlation with the model of Raman spectra in disordered and amorphous carbon [30], except for recent papers of UV laser graphitization and delamination of ultra-thin ta-C films [32] and Raman examination of laser-ablated spots [13].

In the paper we report on laser surface modification of micrometer-thick ta-C films in the regime of single-shot irradiation with UV laser pulses. First, we present experimental results on surface relief changes and new data of the Raman spectra evolution during gradual increase of the pulse energy and transition from pure surface graphitization to incipient ablation regime. Second, we focus on further study of the friction properties of laser-graphitized ta-C surface using friction force microscopy and wear-resistant diamond-coated tips (instead of soft Si tips). In addition, we present a new data of using contact-mode atomic force microscopy (AFM) in the force modulation mode [33,34] for imaging the local surface elasticity of laser-graphitized ta-C films. The contact AFM data obtained for the surface grating of graphitized microstructures have evidenced the lower friction forces between a diamond-coated probe and film surface, and lower stiffness in the laser-graphitized regions.

## 2. Experimental details

Tetrahedral amorphous carbon films were deposited onto steel substrates using the Laser-Arc-technology [35]. After deposition the ta-C coatings were mechanically polished to obtain the resulting roughness of  $R_a \approx 4$  nm. The thickness of the mechanically polished ta-C film was 2  $\mu\text{m}$ ; the film was characterized by the Young's modulus of  $\sim 450$  GPa, hardness of 45 GPa, and density of 2.8 g/cm<sup>3</sup> [13]. The Young's modulus was measured with a laser surface acoustic wave technique [36] and the hardness was determined from the Young's modulus by multiplication with the factor 0.1, as reported elsewhere [35].

Micropatterning of the ta-C films was performed with a KrF excimer laser (model CL-7100 Optosystems Ltd., 248 nm wavelength, 20 ns pulse duration) in an optical projection scheme with a linear demagnification of 1:10. The laser spot size was in the range of 3–4  $\mu\text{m}$  (width)  $\times$  100  $\mu\text{m}$  (length), the laser intensity distribution was uniform over the laser spot. The laser-produced one-dimensional surface relief gratings represented arrays of parallel micro-swells with a period of 8 to 10  $\mu\text{m}$ . The laser surface patterning was performed under single-shot irradiation conditions, i.e. each laser spot area was irradiated by one laser shot, being repeated after each successive translation step equal to the period of the fabricated micropattern.

The surface profile analysis of laser-irradiated areas was performed using a high-resolution white-light interferometry (WLI) technique, which allowed the laser-induced surface relief changes to be measured with vertical resolution of 0.1 nm and lateral resolution of 0.45  $\mu\text{m}$ .

Laser-induced structural modifications of the ta-C films were studied by means of Raman spectroscopy (Ar<sup>+</sup> laser, excitation wavelength  $\lambda = 514.5$  nm). The Raman spectra were recorded using a triple

spectrometer Jobin Yvon S3000 in a microconfiguration, with a laser spot radius of 2  $\mu\text{m}$ . The spectral resolution was 2  $\text{cm}^{-1}$ .

Surface relief and microfriction properties of laser-patterned ta-C films were studied with SPM Solver P47 (NT-MDT) and atomic force microscope of the NTEGRA Spectra system (NT-MDT) using the lateral force mode (LFM) which allowed to image the surface topography and friction forces during one scan [24,25]. Diamond-coated AFM probes with a spring constant of 1.4 N/m were used. The LFM measurements were carried out in air at relative humidity RH = 50% and room temperature  $T = 25$  °C. In the force modulation mode (FMM), scanning of the AFM tip over the surface was performed simultaneously with a vertical periodic motion of the tip, enabling the local surface elasticity to be imaged together with imaging the surface topography [33,34]. The FMM imaging of the laser-patterned ta-C films was carried out in air at RH = 50% and  $T = 25$  °C using silicon V-shaped cantilevers under average load of  $\sim 1$   $\mu\text{N}$ .

## 3. Results and discussion

### 3.1. Single-shot graphitization and ablation with UV laser pulses

In this section we report on single-shot graphitization and ablation characteristics of the 2- $\mu\text{m}$ -thick ta-C film based on the analysis of surface relief changes in the laser-irradiated spots. For the ta-C film under study (characterized by  $sp^3$  content of 70%), the surface graphitization starts at  $T_{gr} \approx 1000$  K and the ablation starts at the sublimation temperature  $T_{sub} \approx 4000$  K [15]. On the laser energy scale these two processes are separated, with the surface graphitization starting at lower fluences. The laser graphitization of DLC films is accompanied by surface swelling in laser-graphitized regions due to reduction of material density (as discussed in detail elsewhere [16]), whereas during ablation the surface level is decreased due to material removal. Competing with each other, both the graphitization and ablation processes contribute to the resulting surface level of laser-irradiated spots on the ta-C film surface. Fig. 1 shows how the surface level or swelling height ( $h_s$ ) of the laser spots changes, relatively to the original surface, with increasing laser fluence ( $E$ ) during transition from the 'pure' surface graphitization to the 'graphitization + ablation' regime under single-shot irradiation conditions. The surface graphitization of the ta-C film starts at  $E_{gr} \approx 0.2$  J/cm<sup>2</sup> and the graphitization rate gradually increases with fluence, resulting in reaching the maximum swelling height of  $h_s = 390$  nm at  $E \approx 0.8$  J/cm<sup>2</sup>. Further increase of the laser fluence leads to lowering of the swelling height due to incipient ablation at  $E_{abl} \approx 1.2$  J/cm<sup>2</sup> ( $h_s = 370$  nm), so that at  $E > E_{abl}$  the graphitization occurs together with the material ablation in the laser spots. The ablation rates increase with fluence, and at  $E_{abl} \approx 10$  J/cm<sup>2</sup> the swelling height "returns" to the zero level ( $h_s = 0$ ) which means that the ablation and graphitization rates become comparable during single-shot irradiation.

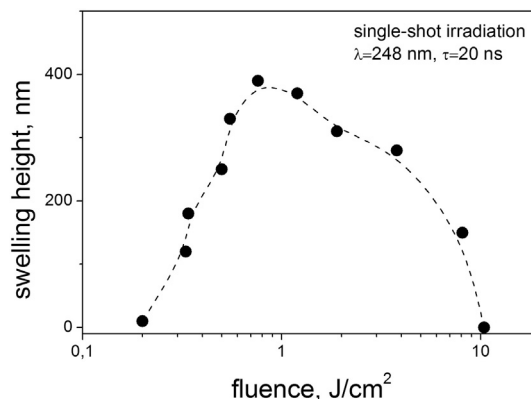


Fig. 1. Swelling height vs laser fluence for laser spots obtained during single-shot irradiation of the 2- $\mu\text{m}$ -thick ta-C film with UV laser pulses of 20-ns duration.

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