Research Article

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Super-hard coatings deposited under conditions of the dynamic shock wave and measurement features of control parameters during production

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Abstract: Super-hard coatings are of increasing scientific interest as they allow synthesizing materials with unique physical and chemical properties for further application in the industry at high-speed processing or tooling. This study is aimed to investigate tetrahedral amorphous carbon (ta-C) films, as well as the dependence of the pulsation parameters on the irradiation parameters and the potential of such pulsations to be further applied in practice. The article shows that ta-C films are completely amorphous and have up to 85% of sp3 bonds. Deposited films are characterized by high compression stresses ranging within 8-10 GPa. The possibilities of reducing these stresses by thermal and pulsed laser annealing have been examined. The formation of ripples in super-hard ta-C films were studied by applying femtosecond laser pulses with a wavelength of 775 nm, an average wavelength of 387 nm, and the pulse duration of 150 fs. Obtained results demonstrated that the orientation of the pulsations on smooth surfaces is perpendicular to the vector of the electric field of the linearly polarized laser beam. Besides, the period of pulsation reduces with decreasing the laser wavelength and/or increasing the angle of laser beam incidence on the substrate.

Keywords: tetrahedral amorphous carbon, pulsed laser deposition, relaxation, stresses, laser pulse, super-hard materials

1 Introduction

To date, super-hard materials with hardness of >40 GPa are actively explored as the requirements for harder materials in many industries have increased recently. All objects created by the human have undergone at least one type of industrial processing with cutting, drilling, and forming its components. The equipment used in these processes is either hard or coated with hard materials for better wear resistance. Besides, the heightened interest in the equipment of longer lifetime has prompted related industries to use super-hard rather than hard materials for practical objectives. Numerous studies have been performed to explore new super-hard materials due to the disadvantages of the traditional ones, such as cubic boron nitride (c-BN) and diamond [1], which are expensive and require extreme pressures and temperatures to be synthesized. So far, the most promising candidates have been the transition metal compounds with light elements (C, B, O, and N) [2–5]. However, most of these compounds (e.g., WC, HfN, or TiN) are neither super-hard nor easy to synthesize, and their production often requires high-pressure and high-temperature conditions [6–8]. Among them, only dense diborides of transition metals have been proved to be super-hard and relatively easy to obtain.

Usually, the diamond is considered as the most incompressible substance known. However, the search for new super-hard materials with elastic properties and hardness comparable with those of diamonds has been of high research interest over the past few decades, which is important from both a scientific and technological point of view. The research in super-hard materials focuses on synthesizing new super-hard materials and making theoretical forecasts with an emphasis on studying the hypothetical phases. The elastic properties of the materials, such as volume modulus, shear modulus, Young’s modulus, and Lamé elastic modulus, are determined by microscopic interactions, where hardness, strength, and yield strength are directly related to the adhesion forces between atoms. Unlike strength and yield strength, hardness has an ambiguous definition, usually described as the resistance of a material to imprinting or scratching by another material. Thus, hardness values depend on the geometry of nozzles in pressing or scratching the device, the applied load, the

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test method, and the experience of the person involved in the measurement. Therefore, the dependence of hardness on both microscopic and macroscopic properties of the material is of particular interest [9, 10].

Super-hard composite thin coatings have the potential to improve the cutting and mechanical properties of the material and increase its wear-resistance [11]. Hence, they constitute an active research area. Super-hard composite thin coatings have numerous design solutions like composite multilayer coatings, superlattice coatings, nano-sized coatings, etc. Many factors are to be considered at developing this type of coatings, namely, the interface volume, the size of crystallites, the thickness of one layer, surface and interphase energy, texture, epitaxial stress and strain, etc. All these factors depend significantly on the choice of materials, deposition methods, and process parameters. Commonly, enhanced hardness properties can be achieved through the dimensional and structural effect and deposition method. In this new field, numerous tasks require solutions, theoretical processing, and experimental validation. Among others, these include the influence of synthesis conditions like a shock wave at the deposition of material particles on the substrate [12]. Depending on the dynamics of shockwave propagation and overlapping, the ordering of the deposited particles in three-dimensional space can be secured, which results in uniform film growth and reduction of internal stresses. That, in turn, may degrade the tribological properties and lead to the rapid destruction of the coating [13, 14]. In this paper, an important aspect and novelty of discussion is the technological innovation of imposing interference electromagnetic fields for the thin film deposition of super-hard alloys based on existing deposition methods [15–17]. Change in frequency and interference patterns allows achieving variability in film structure and, consequently, in mechanical properties of coatings.

The term “wavy structures” describes periodic surface structures that self-organize as a result of laser irradiation of solid materials. Their direction depends on the direction of the electric field vector of a linearly polarized laser beam. The period of pulsation depends on the laser wavelength and the angle of the laser beam incidence. Wavy structures are formed near the fluence ablation threshold, and its formation on different materials is described in the work of Bonse et al. [14]. As reported in the study of Gnilitskyi et al. [13], the occurrence of these structures is related to interference between the incoming laser beam and the beam component scattered along the substrate surface (“surface scattered wave”) [13]. Such wave structures are generalized by the term “LIPSS” (laser-induced periodic surface structures). In the past, some LIPSS types have been classified and distributed into different groups [15]. This study presents the findings of the laser irradiation pulses appearance.

1.1 Aims

The research aimed to examine the effect of material processing on the reduction of wear and friction. To achieve this and to determine optimal parameters, studies have also been carried out on the formation of wave structures in materials from steel 1.2990, tungsten carbide, and tetrahedral amorphous carbon (ta-C). Thus, the ta-C is a specific phase of amorphous carbon film with a high percentage of sp3 bonding that ranges between 70 and 85%, which was produced using pulsed laser deposition and shows a high hardness of 55 to 65 GPa [18].

2 Methods

Carbon films were deposited using the PLD method in a high-vacuum system. For ablation, an excimer KrF laser with a laser pulse energy of 1.2 J, a pulse duration of 30 ns, and a maximum repetition frequency of 50 Hz was applied. Graphite of 99.9% purity was used as a target material. The laser beam moved in a spiral with a constant vector velocity over the target. Residual gas pressure during deposition was 1.104 Pa.

Before deposition, the substrate was bombarded for about 1 minute by a beam of Ar + ions (energy 700 eV, current density 150 AA/cm² on the substrate) to remove impurities.

The films were deposited on solid semiconducting Si substrates. The temperature of the substrate was measured using a thermocouple attached to the substrate surface. Thermal annealing was performed by heating samples immediately after deposition using a resistive heating element.

In experiments with pulsed laser annealing, the same excimer laser beam used for ablation was directed onto the substrate. Its cross-sectional area on the substrate surface was 2 cm². All annealing experiments were performed in a vacuum.

The film stress was determined by the Stoney equation [19] from the bending of silicon substrates caused by deposited films. The bend was measured after deposition with the DEKTAK profiler [20]. For the in-situ measurements, 400-Am-thick Si substrates of 50 mm length and 5 mm width were clamped one-sided to the substrate holder. The
film thickness needed for measuring stress was determined based on the reflected intensity of the laser beam.

For examining wavy structures, mostly the automated high precision micromachining station of fs-laser was used [18]. The femtosecond laser system allows processing materials with the focusing technique, where the average wavelength of the fs-laser system is 775 nm, the maximum pulse energy is 1 MJ, the pulse duration is 150 fs, and the constant pulse repetition frequency is 1 kHz. The fs-laser beam is focused on the sample surface using a 100 mm lens for these studies.

## 3 Results

### 3.1 Deposition of ta-C films

During deposition, the hydrogen-free films of amorphous carbon obtained with PLD can contain diamondlike bonds (sp3) in the range of 0-85% and, accordingly, have low or high hardness depending on the temperature of the substrate and the energy density (fluence) of the laser on the target. It has been established that the fluence must be more than 6 J/cm², and the substrate temperature must be less than 90°C to enable formation of super-hard carbon films with a high sp3 (ta-C) content. Thus, the essential parameter to be considered is the average kinetic energy of the film-forming particles. Calorimetric measurements have shown that it varies between 30-100 eV with a fluence of 6-30 J/cm² (Figure 1). In this range, the sp3 content comprises 70-85%.

At lower values of kinetic energy and fluence, the sp3 content decreases accordingly. Excess of the substrate temperature over 90°C leads to the decrease of the sp3 content regardless of the laser fluence.

The maximum deposition rate of the ta-C films was 120 nm/min, where the laser fluence on the target was 10 J/cm², the laser beam cross-section on the target surface was 3.5 mm², and the pulse frequency was 50 Hz. With these parameters, the average temperature of the substrate increases to about 708°C during deposition, which might be associated with the energy supply of film-forming particles. Consequently, the ta-C films can be produced without cooling the substrate.

### 3.2 Voltage reduction by thermal and pulse laser annealing

After deposition, the internal compressive stress of the ta-C films was quite high amounting to 8-10 GPa, which resulted in laminating films with thicknesses ranging from 200 to 400 nm. The film thicknesses required for stress calculation were determined from the reflected intensity of the
measuring laser beam at a wavelength of 670 nm. Therefore, to obtain films of higher thickness, these stresses must be reduced or avoided. The voltage can be completely removed by thermal annealing within 5 minutes at 600°C. Such method was also applied in the present research.

As can be seen from Figure 2, the residual voltage depends on the temperature and annealing time. A minimum annealing temperature of 600°C and the minimum annealing time of a few minutes are required for almost complete stress relaxation. The sp3 content is, thus, retained, as well as the diamondlike properties of the ta-C films. To grow thick ta-C films within the micrometer range, sublayers of several hundred nanometers thick must be applied and annealed sequentially. Thus, the ta-C films of several micrometers thick were produced on Si and WC hard metal substrates with the thickness of the layers of 200 nm.

3.3 Investigating the ripples formation with perpendicular incidence angle

Ripples formation was examined as a function of laser pulses and laser fluences amount during the movement of the laser beam across the surface using a constant pulse distance for each test field to observe wave formation over large areas.

It has been established that the ripple would be much more distinctly oriented in the direction perpendicular to the electric field vector if the laser beam was scanned over the entire surface of the material (Figure 3a). Thus, the orientation of the ripple is independent of the scanning direction. Optimal fluence, at which the waves are well oriented and expressed, and no significant removal of the material is noticed, was equal to 3.61 J/cm² for the ta-C films 2 μm thick.

The distance between pulses, most suitable for the formation of ripples on large areas, was about 15 μm at a laser spot radius of 20 μm, which corresponds to the overlap of neighboring areas of the spot by about 57%. This overlapping of pulses was found to be the most appropriate regardless of the spot size itself. The same overlap of 57% was also used for doubling the laser frequency. The pulsation period was 330 nm (see Figure 3b). The most suitable fluence of the laser pulse was found to be 14.25 J/cm².

4 Discussion

A new method of stress relaxation in ta-C films [21–23] has been applied, which concerns pulsed laser annealing performed during deposition with a second laser or alternately with the deposition process. The paper of Pfeiffer et al. [16] presents the results of stress changes in three different ta-C films 150 nm thick after pulsed laser annealing. It is noteworthy that the stresses significantly reduced only after 200
laser pulses of 30 ns duration (i.e., relaxation occurs for a short time), the same way in experiments performed in this research. The residual voltage decreases with increasing laser energy density from about 3 GPa at 110 mJ/cm² to 1 GPa at 165 mJ/cm² and almost to 0 GPa at 180 mJ/cm².

The temperature fields induced in the ta-C/Si systems by laser irradiation calculated using a numerical computer program are described in the work of Drescher et al. [24]. The temperature-dependent material constants of silicon were taken from the studies of Drescher et al. [24] and Morath et al. [25]. The ta-C optical constants at 248 nm were even: refractive index, extinction, and absorption factor was 3.0, 0.35, and 1.8*105 cm⁻¹, respectively, and density amounted to 3.3 g/cm². Specific heat capacity was calculated according to the Debye theory with the Debye temperature of 1770 K. For thermal conductivity, the value 9.2 102 W/cm/K was used, which was measured on ta-C films with pulsed laser deposition [26]. Also, the Gaussian dependence of the laser pulse on time was applied with the pulse duration of 30 ns.

According to these calculations, the maximum temperature during one pulse in the ta-C film with a thickness of 150 nm on Si is 763 K at a laser energy density of 110 mJ/cm² and 1053 K at an energy density of 180 mJ/cm², at which the minimum tension is reached. Besides, at a constant fluence, the maximum temperature during one pulse relatively strong depends on the thickness of the film, only to a thickness of about 300 nm, after which it stops due to the low thermal conductivity of ta-C. It might be also a reason for the low penetration depth of temperature fields in the film. Thus, only about 50 nm is heated almost evenly. The temperature required for complete relaxation is assumed to be 180 K higher than that required for conventional annealing. However, for the annealing experiments, 150 nm thick films were used. As the temperature gradient of about 200 K is between the upper and lower parts of the film and full relaxation requires all parts of the film to be involved, the temperature required for complete relaxation is within the range of conventional annealing. On the other hand, the temperature exceeds 650 K only for 40 ns. and 400 K only for 230 ns. per impulse, i.e., the relaxation process in pulsed laser annealing takes several tens or several hundred microseconds, which is much faster than at conventional annealing. The results show that the process is determined mainly by thermal processes. Also, high heating and cooling rates of about 1010 K/s necessary for stress relaxation lead to faster exchange processes and reordering of atoms compared to maximum rates of less than 1 K/s during thermal annealing.

The contents of sp3 bonds in the ta-C films, deposited at a fluence density of 16 J/cm², has been established to be 80.3% for the non-annealed film and 82.4% for the film with pulsed laser annealing. Thus, the sp3 content of the films does not decrease as a result of pulsed laser annealing, which corresponds to the measured properties. Whether the slight increase is significant requires further research.

Mechanical properties are preserved as well. According to results presented in the study of Weissmantel et al. [11], the deposited films have unusually high hardness and Young’s modulus values, which were measured on several 12 Am thick films and obtained on solid metal WC substrates. At that, the hardness is ten times lower compared to Young’s modulus. This is probably due to the presence of particles with dimensions of several hundred nanometres in the films. This assumption is confirmed by the relatively large scattering of ten values measured at different points on the same sample. The highest values of these single measurements performed in the particle-free film area were 85-95 GPa.

Both thermal and pulsed annealed ta-C films demonstrate good adhesion to Si and WC hard metal substrates. The high values of critical loads under which the film is damaged during a scratch test are important for industrial applications. The damage to the film was detected by recording the sound radiation during the test and, afterward, visually in the optical microscope.

In all investigated materials, the ripples begin to form already as a result of surface irradiation by one laser pulse, provided that the laser fluence is above a certain threshold and depends on the material. If one or two pulses are applied, the wave structure in ta-C is formed partially and preferably perpendicular to the electric field vector of a linearly polarized laser beam. However, they are also formed near the ruptures consisting of ta-C films made of particles associated with deposition but have a circular shape.

With further increase in the number of laser pulses, the orientation of the generated pulsations becomes more pronounced in the direction perpendicular to the field vector, even if they still have a round shape near the ruptures. It appears that the ripples act as scattering centers for the incoming laser beam. It should be noted that the ripples were formed directly in 2 μm thick ta-C films, and the center of the laser spot melts in the case of ten pulses due to the incubation effects.

Measured periods of pulsations, which are below the average laser wavelength of 775 nm, slightly depend on the material (Table 1) but almost do not depend on the density of the stream and the number of laser pulses. From available literature [27], the refractive index of the material is known to change in the irradiated region when photons interact with the material. This laser-induced refractive index ni, which differs slightly for each material, can be calculated...
Table 1: The dependence of the measured periods of pulsations on the average laser wavelength, density of the stream, and the number of laser pulses

| Material          | Period of wave structures $S_0$, nm | Laser-induced refractive rates $n_i$ |
|-------------------|-------------------------------------|------------------------------------|
| ta-C              | 667                                 | 1.162                              |
| Steel 2990        | 648                                 | 1.196                              |
| Tungsten carbide  | 706                                 | 1.098                              |

from the pulsation period by the following equation (1) [27]:

$$n_i = \frac{\lambda}{S_0}$$

(1)

Thus, $S_0$ is the period of wave structures at normal incidence, and $\lambda$ is the wavelength of the incident laser beam.

4.1 Formation of pulses at inclined incidence

Assuming that pulsation forms due to interference between the incoming laser beam and the surface scattered wave, as suggested in the work [28], the period of pulsation should be changed by using the inclined incidence of laser radiation on the sample surface. The scheme of this so-called "surface scattered wave" model showing the interference of the incoming and scattered beam is shown in Figures 3, 4. Scattering can occur in two directions (forward and reverse) and that the refractive index caused by the laser is significant [29]. The period of the ripple can be calculated by the equation (2):

$$S^* = \frac{\lambda_2}{1 \pm \sin \beta} = \frac{\lambda}{n_i \pm \sin \beta}$$

(2)

In performed experiments, the polarization of the laser beam was always parallel to the incident plane. If it was perpendicular to the polarization, the ripple period did not change with the change of incidence angle. The measured periods were compared with those calculated for the $S^+$ type according to the model, where a good agreement between theoretical and experimental values was revealed.

The group Sadowski et al. [30] studied the influence of technological parameters of the device for direct laser sintering of metal. The paper [30] described that the small power of a beam (40 W) does not provide enough energy to maintain a continuous laser melting line Inconel 718. It has been shown that medium power beams (100 W and 150 W) result in better and more uniform scanning lines with minimal voids and smaller melt volumes. The research by Stoia et al. [31] revealed that the mechanical properties of a sample depend on the laser incidence angle and the orientation of the sample in space. Therefore, the technological parameters of the laser unit in similar experiments are worth examining in the future.

4.2 Conclusions

The ta-C films were produced at high growth rates of up to 120 nm/min by the PLD method. Full relaxation of stresses can be achieved by thermal and pulsed laser annealing. Stress relaxation occurs quickly with pulsed laser anneal-
ing, and the process itself requires several tens to hundreds of microseconds. Super-hard ta-C films can be produced on WC hard metal substrates without stress, several micrometers thick, and with good adhesion using the described method in alternation with the deposition process.

In all the studied materials, wavy structures are formed perpendicular to the electric field vector of the incident femtosecond laser beam. Besides, when the laser beam falls obliquely on the sample surface, the ripple period can be reduced or increased (interference patterns of S- and S-type) according to the “surface-scattered wave” model. Wavy structures on ta-C coated steel surfaces improve friction and wear characteristics.

Conflict of Interests: The author declares no conflict of interest regarding the publication of this paper.

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