

Thermochemical polishing of single-crystal HPHT-diamond substrates: surface analysis

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The results of single-crystal HPHT-diamond substrates treatment with the method of thermochemical polishing were analyzed. The main application of this method is — bringing the surface of the mechanically polished diamond substrates to the condition close to atomically smooth one. Using the method of optical profilometry, for the first time the results of the studies of the entire surface area (4×4 mm) of high-quality diamond substrates were obtained. It was shown that thermochemical polishing may considerably improve the morphological characteristics of diamond substrates up to the point that at 80–90% of their surface area the height differences will make less than 200 nm. The data obtained using the atomic-force microscopy method confirm the reduction in the surface roughness to the level of Ra (0.5–0.7) nm. The polishing process also leads to formation of cavities of various size and depth, distributed unevenly; as the homogeneity of the surface increases, their number may rise. The study results demonstrate the significant prospects of thermochemical polishing of diamond substrates for their industrial use in high-tech areas of microelectronics and micromechanics requiring flat surfaces with minimum roughness.

Keywords: HPHT-diamond, thermochemical polishing, mechanical polishing, surface roughness, planarity.

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Introduction

Diamond — is a very promising material for use in electronics [1,2], optics [3,4], as ionising radiation detectors [5–7] and heat sinks [8] in virtue of its unique physical properties. All these applications require a flat, even surface with the lowest roughness $Ra \approx 1–3 \text{ \AA}$ [9]. However, the diamond is known with its highest hardness, which presents a specific challenge when polished. Standard method of mechanical polishing (MP) require using a diamond powder on an iron disc rotating with high speed. Using this approach usually achieves the surface roughness level of units of nanometers (units of measurement Ra and Rq — arithmetic mean and mean square deviation of the profile heights, accordingly). Moreover, this method is characterized by strong anisotropy in relation to the polishing direction, and the mechanically treated surface is significantly striated, which limits its use in high-tech applications [10]. Therefore, MP is often used as primary treatment (grinding and polishing), and the finish polishing is done with other methods of precision surface treatment. Alternative polishing methods were proposed, which were

based on other physical principles: chemical reactions (chemical-mechanical polishing, plasma etching), graphitization (thermochemical polishing, polishing by dynamic friction), evaporation and knocking out of carbon atoms (laser polishing, ion spraying) and some others [9]. All methods have their advantages, disadvantages and physical limitations.

In 1953 paper [11] proposed a polishing method using a hot metal disc, which is now called thermochemical polishing (TCP). Active development of this method started in the end of XXth century and was carried out in papers [12–15]. The TCP method is based on the ability of certain metals (Fe, Ni, Mn, La, Ce etc.) to dissolve carbon when heated [16]. At the same time it is believed that metals catalyze transition of diamond in non-diamond forms of carbon (graphite and amorphous carbon), which in their turn diffuse into metal at high temperatures [17]. The force of metal interaction with carbon is determined mostly by fullness of d -sublevel. Thus, metals without electrons on it (for example, Cu, Zn) are relatively inert to carbon, metals with nearly filled d -sublevel

(for example, Ti, V) form carbides, and metals with several electrons (for example, Fe, Ni) dissolve carbon effectively. The latter also catalyze transition of diamond into graphite, therefore are used in TCP [16], the current studies also demonstrate the new opportunities of using Ni for TCP [18]. The literature contains some information on the effect of various compositions of a polishing disc on the diamond surface during TCP. Paper [12] showed that an iron disc shows better results than a nickel one. Cast iron and molybdenum discs do not have a noticeable effect on the polished surface.

TCP occurs during rotation (or sometimes in a static position) of a metal disc being slightly pressed at speeds not exceeding 10 rpm. The exposure and accordingly the removal is greater for the protruding parts of the crystal, which levels the surface. Minimum temperature, at which polishing is possible — 700 °C, but in this case the polishing speed is very low. At temperature of more than 950 °C all graphitized carbon has no time to be diffused in a disc, which causes its accumulation, therefore high temperatures are not used usually, or TCP is done in gaseous medium, where graphitized carbon produces volatile products (for example, CH_x in the hydrogen environment), and therefore is removed from the metal/diamond interface. Moreover, polishing is often done in two stages: at first at high temperature, and then at low one, to get rid of all remains of the graphitized carbon and achieve the highest quality of the surface [13]. Apart from temperature, another important factor that determines the TCP result is the composition of the atmosphere where it takes place. Polishing may be carried out in vacuum, hydrogen, inert gases and nitrogen. Polishing speed in this row drops from vacuum towards nitrogen. Under vacuum conditions the diamond polishing speed is the highest, since adhesion of the polishing disc and polished crystal increases at the expense of the lower pressure. Moreover, the residual oxygen in the chamber may additionally etch the diamond. In hydrogen the polishing speed is higher than in inert gases, since the first may react with the carbon dissolved in the polishing disc, which prolongs the achievement of the saturation state for the metal. The speed of polishing in nitrogen is lower than in inert gases, since nitrogen is absorbed by the surface of the metal plate and delays carbon diffusion [12]. Paper [14] proposed to use the transverse vibrations of the polishing disc to reduce strong friction with the polished specimen, which always occurs as a result of forming the adhesion transition layer at the diamond–metal interface. These vibrations „broke“ the specimen off the polishing disc. An important feature of TCP is absence of the significant effect of the crystal-lattice orientation on the end result [19]. This paper considers the TCP method and studies its capabilities to polish single-crystal diamond HPHT-substrates. Detailed analysis of geometry and roughness parameters of the diamond substrate was conducted after their treatment with the TCP method.

1. Study methods and specimens

This paper contains the results of study of nine single-crystal multi-sector HPHT-diamond substrates of type IIa, orientation $\langle 100 \rangle$, with dimensions of $4 \times 4 \times 0.5$ mm. The substrates were provided by LLC Scientific Production-HComplex „Almaz“, LLC „New Diamond Technology“ (Sestroretsk, St. Petersburg). All specimens were first exposed to MP, which has been finalized and is used on an industrial scale at these enterprises. Therefore, to demonstrate the MP results, photographs of the specific images are provided, which reflect the general features of the produced plates treatment. One may guarantee with high degree of accuracy the reproducibility of the MP results, since it is done using the same technology for all substrates on a polishing machine „DialitV Super Table™“ with a diamond powder of ACM 10/7 grade. TCP was carried out in LLC „KRISTALIN“ (Barnaul), on Mikrotom-3 unit with a rotary iron disc, in the hydrogen atmosphere, at temperature 750–800 °C. TCP for all specimens was carried out in somewhat different local conditions (duration of polishing, degree of specimen pressing). Study of the effect of each factor is beyond the goals of this paper. Morphology, roughness and planarity of the surface were studied by the atomic-force microscope (AFM) Integra-Aura in the resource center of „Microscopy and Microanalysis“ St. Petersburg State University, and on the optical profilometer Zygo ZeGage PRO HR in the laboratory „Diamond Microwave Electronics“ of RTU MIREA. This profilometer makes 3D-scans of the surface in a large area, and then combines the sections by binding method. The borders between the bound scans sometimes manifest themselves on the final image, but they are not a feature of the surface.

2. Results

2.1. Geometry of diamond substrates before and after TCP

2.1.1. Preliminary MP

Geometry of all studied substrates after MP is characterized by the deflection in the central part, with height difference from 1 to 3 μm. This feature is due to the standard mechanism to fix the substrates and process tooling exposed to uneven pressure. The latter causes the increased removal of the material from the central part of the substrate. Depending on the evenness of the pressure, diverse geometry of deflection is formed, however, within the process allowances. For example, at excessive but even pressure that the central part of the substrate is exposed to, a relatively isometric indent is formed (fig. 1, *a*). If fixation provides uneven pressure, beveled or even U-shaped indents may appear (fig. 1, *b*). In case when one of the holders provides much higher pressure, it is possible to produce a relatively smooth substrate with small height differences within the first hundreds of nanometers. Nevertheless, a

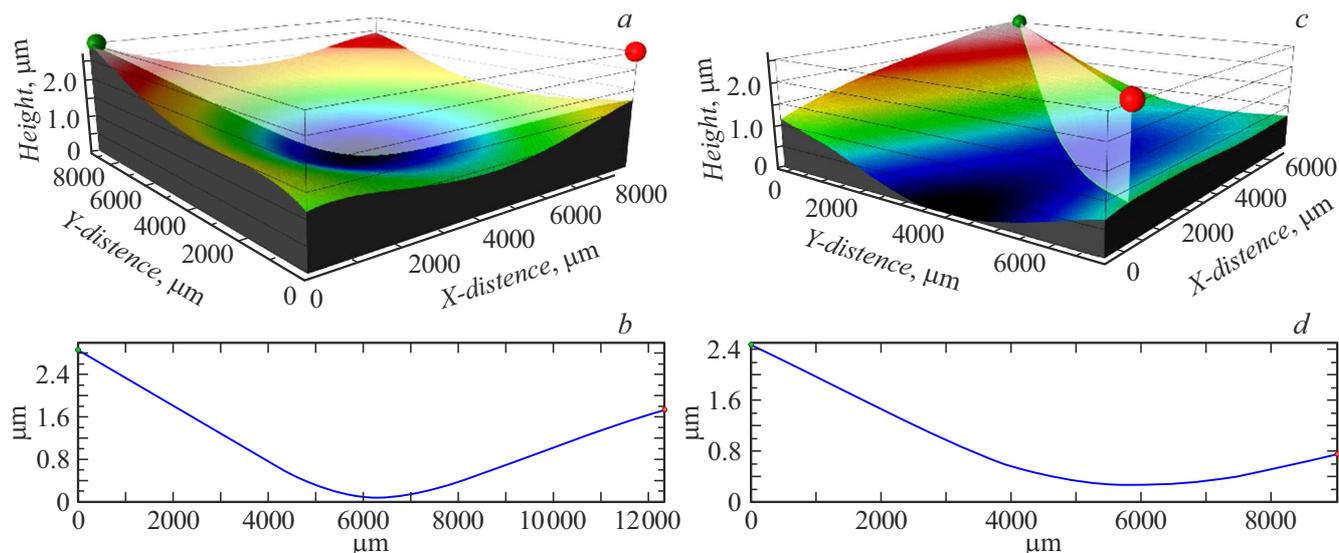


Figure 1. 3D-scans on the optical profilometer of the surface of diamond substrates after MP: *a* — isometric deflection in the central part of the substrate; *b* — profile along the line specified in fig. 1, *a*; *c* — U-shaped deflection in the central part of the substrate; *d* — profile along the line specified in fig. 1, *c*.

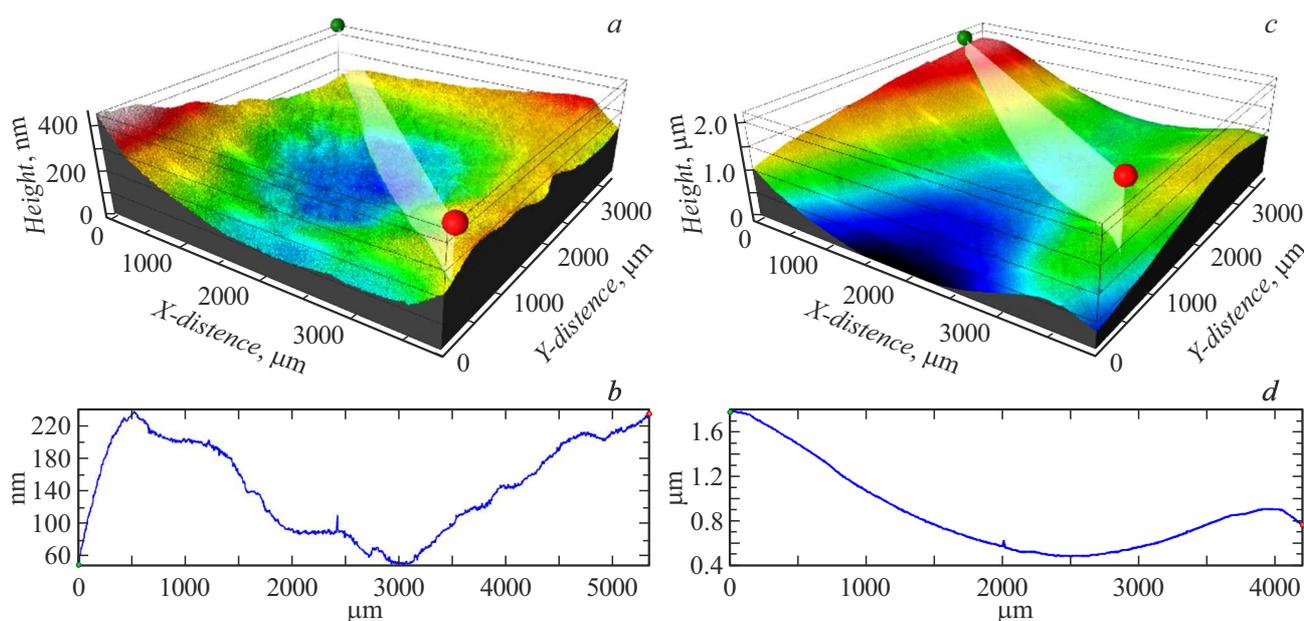


Figure 2. 3D-scans on the optical profilometer of diamond substrates after TCP: *a* — deflection in the substrate center (specimen 33725); *b* — profile along the line specified in fig. 2, *a*; *c* — beveled U-shaped indent (specimen 33892); *d* — profile along the line specified in fig. 2, *c*.

ledge of 1–2 micrometer will be formed on one of its corners.

2.1.2. TCP

After TCP of five diamond substrates, substantial changes were found in the surface geometry that were not specific for the used MP technology. Two specimens demonstrate significant reduction in the deflection, leveling of the surface

planarity, on another two the deflection changed to a convex central part, and one shows a convexity of the central part of up to 2 μm.

The following features may be identified for the plate deflection that emerged after TCP: 1) deflection located in the center of the plate, with uneven elevations towards the edges (specimen 33725, fig. 2, *a*), height difference of around 150 nm in the central part, 350 nm in the entire surface area; 2) beveled U-shaped deflection stretching in

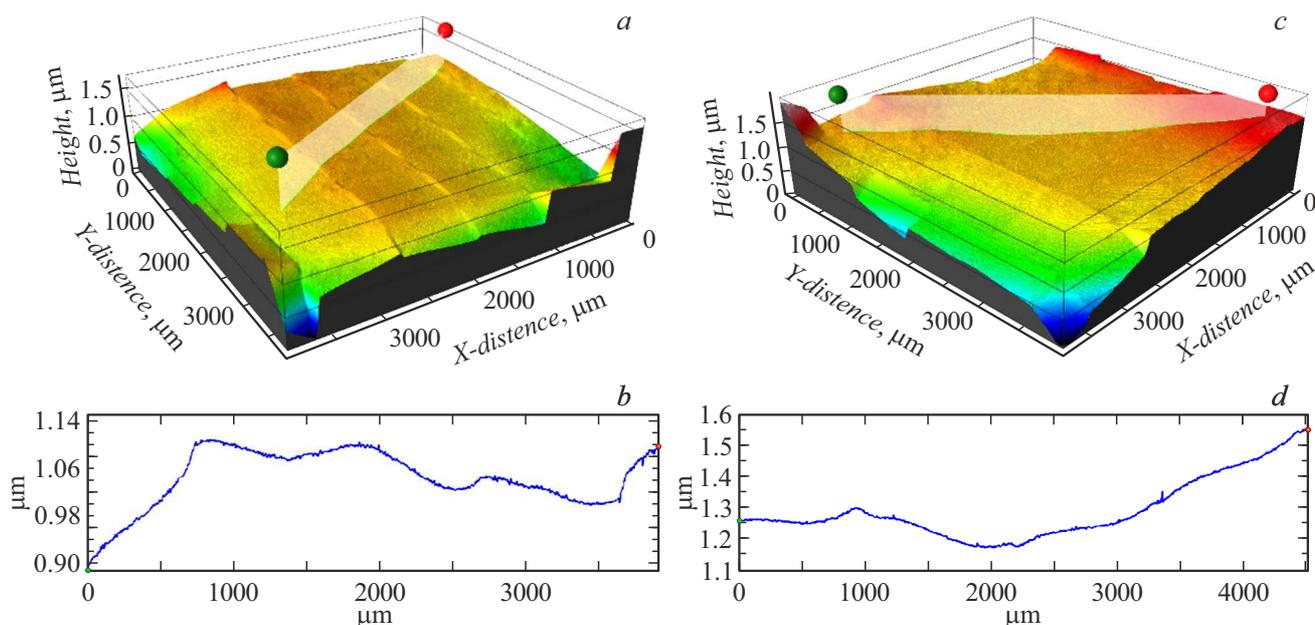


Figure 3. 3D-scans of diamond substrates on the optical profilometer after TCP characterized by a mild uneven elevation in the central part of the substrate and irregularities at its ends. *a* — specimen 35211, *b* — profile along the line, specified in fig. 3, *a*; *c* — specimen 40113; *d* — profile along the line specified in fig. 3, *c*.

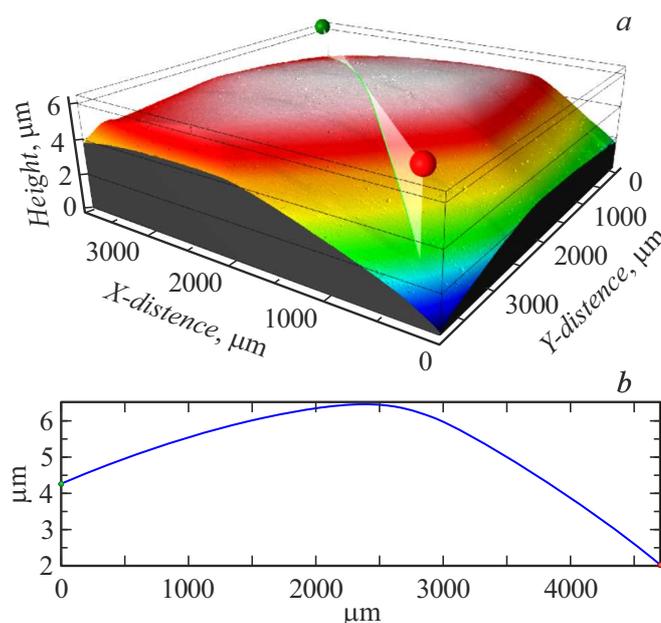


Figure 4. *a* — 3D-scan on the optical profilometer of the diamond substrate №44271 after TCP characterized by a significant elevation smoothly lowering towards edges; *b* — profile along the line specified in fig. 4, *a*.

the middle of the plate with height difference of $\sim 0.6 \mu\text{m}$ in the center of the plate reaching $1.8 \mu\text{m}$ on one of its edges (specimen 33892, fig. 2, *b*).

Two substrates with a mild uneven elevation in the central part of the plate there is a height difference of around 200 nm in the main surface area of the substrate (specimens 35211 and 40113, fig. 3). Irregularities that achieve the height difference of $1 \mu\text{m}$ are observed near the

edges of the diamond substrates №35211, 40113 and, most probably, they are related to the standard mechanism of plate fixation for TCP.

Geometry of one substrate (specimen 44271, fig. 4) differs significantly from the others: in its central part there is a significant elevation smoothly lowering towards the edges. Height differences at the edges vary from 2 to $6 \mu\text{m}$, while in the central part the difference is not more

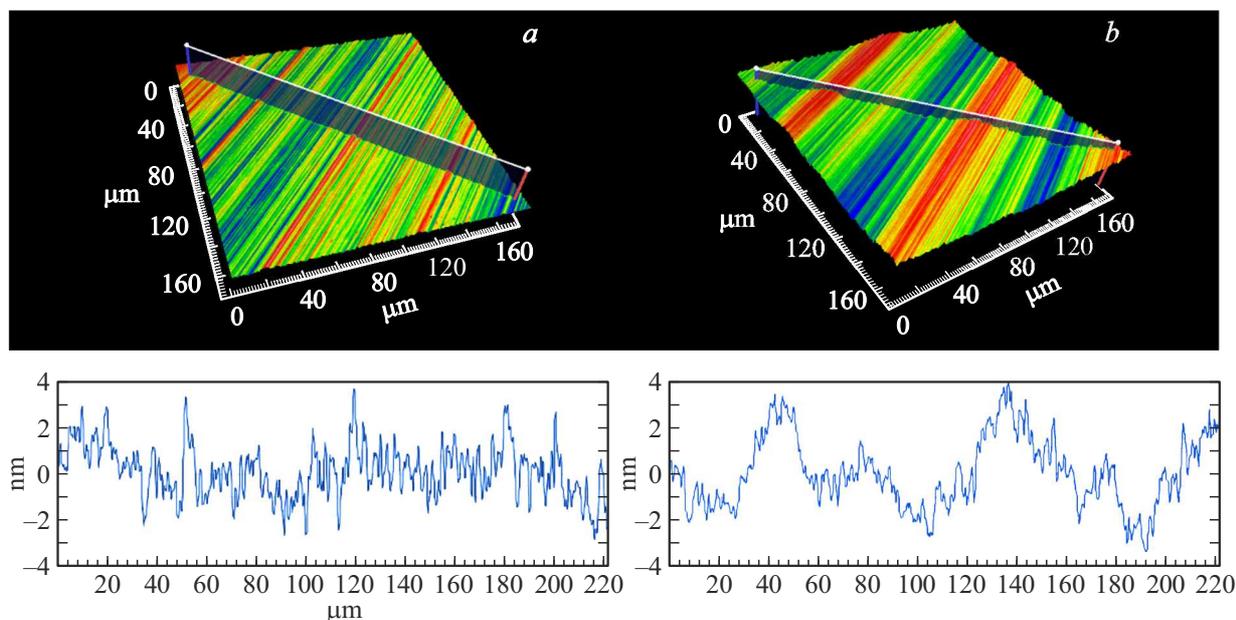


Figure 5. Detailed 3D-scans on the optical profilometer of diamond substrates after MP: *a* — relatively even shade lining with height differences on the profile of up to 7 nm; *b* — periodical shade lining with height differences of up to 8 nm.

than $1\ \mu\text{m}$. Besides, the surface of this substrate is scattered with multiple indents. Such significant change in the geometry may be related to the excessive duration of polishing.

2.2. Roughness of the surface of diamond substrates before and after TCP

2.2.1. Preliminary MP

High-quality MP provides for the surface roughness level of $Ra \sim 1\ \text{nm}$. Further reduction in the surface roughness with this method is possible, but requires a much more thorough approach. The surface after the used standard MP is significantly complicated with the parallel shade lining of various periodicity, which is seen well on the profile (fig. 5). Height differences reach 8 nm. It should be noted that the surface of any material after MP is characterized by higher stresses, abnormalities and areas with amorphous carbon formed by abrasive pressure in process of polishing.

2.2.2. TCP

The most important common feature of the surface of diamond substrates after TCP — absence of shade lining of MP or its significantly smoother nature with roughness of Ra (0.5–0.7) nm. Besides, sculptures appear on the surface: indents of round and oval shape, with dimensions from ~ 0.01 to $0.5\ \mu\text{m}$, depth from ~ 0.001 to $0.2\ \mu\text{m}$. Some of them are sometimes arranged in series. The table presents data on the quantitative and dimensional ratio of the indents in the local areas of the surface of the diamond substrates obtained when

studied by AFM method. Indents were isometric local areas of lowering in height by more than 5 nm. The number of indents in each cell of the table is specified for different AFM images with surface area of $100 \times 100\ \mu\text{m}^2$.

The surface of the diamond substrate №38577 (fig. 6) has shade lining that is much smoother and scarcer compared to the shade lining from the used MP. This shade lining may be both residual from mechanical polishing and formed due to irregularities of the polishing disc.

The surface of specimens 44060 (fig. 7) and 38577 is mostly homogeneous, the shade lining is hardly seen, however, the indents are more marked on them, being distributed very unevenly. It is seen especially well in fig. 6, *a*.

The only specimen with no indents is — 35211 (fig. 8). The local lows on the profile do not exceed 4–5 nm, however, the entire surface is coated by the shade lining clearly inherited from the mechanical polishing. This may indicate insufficient intensity or duration of thermochemical polishing. Nevertheless, the surface roughness reduced, and the height differences on the profile became around 3 nm. Moreover, this shade lining is more homogeneous than in the plates polished only mechanically, which are described in the beginning of section 2.2.

3. Discussion of the results

The TCP method provides significant effect on the formation of the surface geometry in the single-crystal diamond substrates. One may suggest that initially the geometry of the mechanically polished plates is inherited,

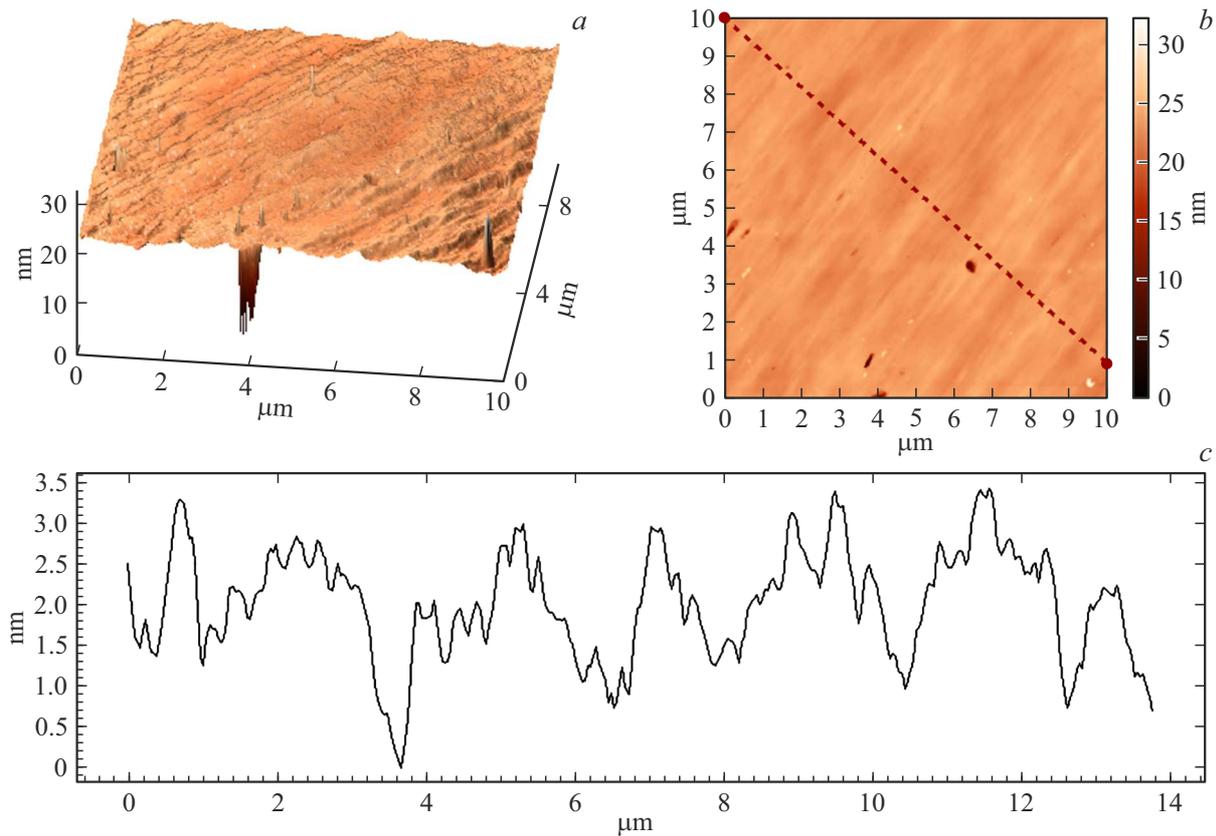


Figure 6. AFM images of the specimen 33892 surface after TCP: *a* — 3D image of its surface; *b* — 2D image of the surface shown in fig. 6, *a*; *c* — profile of heights along the line drawn in fig. 6, *b*.

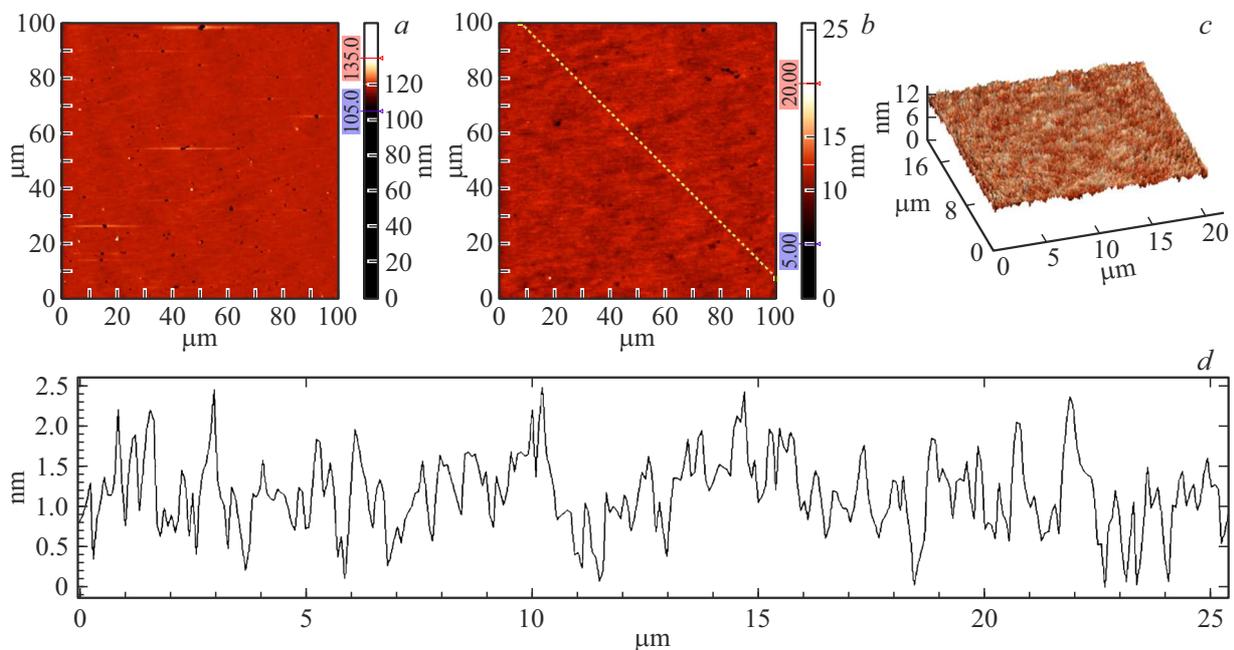


Figure 7. Images of the specimen 44060 surface made with AFM: *a* — 2D image of the area on its surface with size of $100 \times 100 \mu\text{m}$; *b* — 2D image of the area on its surface $20 \times 20 \mu\text{m}$; *c* — 3D image of the surface shown in fig. 7, *b*; *d* — profile of heights along the line drawn in fig. 7, *b*.

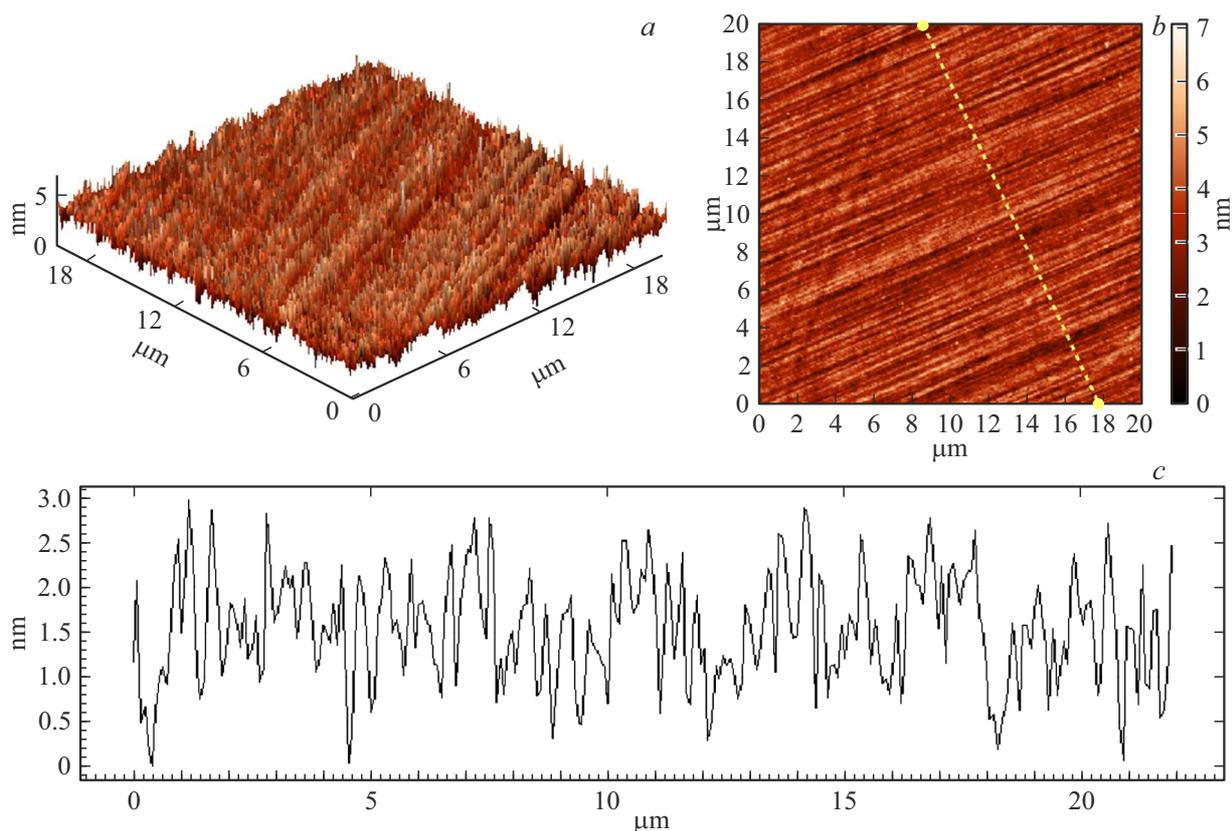


Figure 8. AFM images of the specimen 35211 surface: *a* — 3D image of its surface; *b* — 2D image of the surface shown in fig. 8, *a*; *c* — profile of heights along the line drawn in fig. 8, *b*.

however, in process of the material removal during TCP the surface is leveled, which to a large extent also depends on the process rigging. The process rigging for fixation, positioning and pressing of the diamond substrates affects rather substantially the geometry of the end surface. It is fair for the TCP method and for the MP method. The TCP also causes new features of the geometry. Some of the substrates are characterized by height differences not exceeding 200 nm on (80–90) % of the plate surface area. Using the TCP method, one may achieve a rather smooth surface, which is of interest for using this method in order to obtain the best planarity of the diamond substrates.

The most important common feature of the surface roughness of diamond substrates after TCP — absence of shade lining of MP or its significantly smoother nature and reduction of value Ra to the angstrom level. Usual height differences on the profile are 3 nm max., while for the mechanically polished substrates - around 8 nm. The thermochemical method of treatment results in indents. Based on the analysis of the tabular data, one may conclude that the roughness of the specimens polished thermochemically varies insignificantly in the surface area. On the other hand, the number and depth of the indents vary greatly. This is specific both for different diamond substrates and for one and the same substrate. The literature contains no single opinion about the indent formation mechanism.

In paper [12] the authors believe that the indents are formed by etching with gases in the atmosphere, where the polishing takes place (or with residual gases in case of polishing in vacuum). However, the other researchers [19] relate their formation to the higher concentration of catalyst—metals in some areas. Therefore, these indents may be formed using the same mechanism that is responsible for the polishing, but in their case the reaction continues regardless of the contact with the polishing disc, therefore it lasts longer.

Conclusion

This paper demonstrates the impact of the TCP method at the quality of the surface of single-crystal diamond substrates, and presents for the first time the profile charts of the entire surface area of the substrates after TCP. These results show that nearly in all cases the planarity of the diamond plates improves under TCP, the smoothest plates in 80–90 % of their surface area have the height differences not exceeding 200 nm. This method showed how to effectively remove/smoothen the shade lining inherited from the mechanical treatment with the reproducible values of the parameters of the surface roughness Ra 0.5–0.7 nm. However, in parallel to this, small isometric indents are formed, with no clear patterns in the surface area distribution and depth. Their number correlates positively

Quantitative and dimensional ratio of the indents in the local areas of the surface of diamond substrates polished thermochemically obtained when studied by AFM method

Specimen number	Surface roughness in the area	Quantity and depth of indents	
38577	$25 \times 25 \mu\text{m}$ $Rq = 0.891 \text{ nm}$; $Ra = 0.554 \text{ nm}$ $25 \times 25 \mu\text{m}$ $Rq = 1.165 \text{ nm}$; $Ra = 0.479 \text{ nm}$ $20 \times 20 \mu\text{m}$ $Rq = 1.138 \text{ nm}$; $Ra = 0.641 \text{ nm}$ $14 \times 14 \mu\text{m}$ $Rq = 0.909 \text{ nm}$; $Ra = 0.482 \text{ nm}$	98	44
		53 > 10 nm	19 > 10 nm
		23 > 20 nm	8 > 20 nm
		6 > 50 nm	3 > 50 nm
		Max 184 nm	Max 84 nm
44060	$25 \times 25 \mu\text{m}$ $Rq = 1.385 \text{ nm}$; $Ra = 0.666 \text{ nm}$ $25 \times 25 \mu\text{m}$ $Rq = 1.428 \text{ nm}$; $Ra = 0.668 \text{ nm}$ $60 \times 60 \mu\text{m}$ $Rq = 1.239 \text{ nm}$; $Ra = 0.598 \text{ nm}$ $55 \times 55 \mu\text{m}$ $Rq = 1.231 \text{ nm}$; $Ra = 0.602 \text{ nm}$ $100 \times 100 \mu\text{m}$ $Rq = 1.935 \text{ nm}$; $Ra = 0.716 \text{ nm}$ $60 \times 60 \mu\text{m}$ $Rq = 1.366 \text{ nm}$; $Ra = 0.739 \text{ nm}$ $20 \times 20 \mu\text{m}$ $Rq = 0.614 \text{ nm}$; $Ra = 0.460 \text{ nm}$	148	127
		55 > 10 nm	68 > 10 nm
		14 > 20 nm	28 > 20 nm
		Max 37 nm	Max 63 nm
		92	120
		37 > 10 nm	61 > 10 nm
13 > 20 nm	26 > 20 nm		
Max 76 nm	Max 119 nm		
		72	
		34 > 10 nm	
		14 > 10 nm	
		5 > 50 nm	
		Max 124	
35211	$20 \times 20 \mu\text{m}$ $Rq = 0.829 \text{ nm}$; $Ra = 0.639 \text{ nm}$ $25 \times 25 \mu\text{m}$ $Rq = 1.036 \text{ nm}$; $Ra = 0.732 \text{ nm}$ $25 \times 25 \mu\text{m}$ $Rq = 0.844 \text{ nm}$; $Ra = 0.679 \text{ nm}$	NA	
33892	$10 \times 10 \mu\text{m}$ $Rq = 0.636 \text{ nm}$; $Ra = 0.483 \text{ nm}$ $10 \times 10 \mu\text{m}$ $Rq = 0.755 \text{ nm}$; $Ra = 0.490 \text{ nm}$ $10 \times 10 \mu\text{m}$ $Rq = 0.629 \text{ nm}$; $Ra = 0.478 \text{ nm}$	47	71
		20 > 10 nm	38 > 10 nm
		9 > 20 nm	20 > 20 nm
		Max 85 nm	7 > 50 nm
		Max 70 nm	
		135	
		84 > 10 nm	
		48 > 20 nm	
		29 > 50 nm	
		Max 131 nm	

with the degree of the plate homogeneity, therefore, further research is necessary to optimize the polishing parameters.

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Conflict of interest

The authors declare that they have no conflict of interest.

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