

Investigation of residual stresses in sapphire plates after grinding and polishing

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The method of flexure determination was used to investigate the dependence of residual stresses on the depth of damaged layer in sapphire plates with a diameter of 100 mm after grinding and polishing. The flexure was determined from nonflatness of the control side of the plate using an interferometer of IT-200 type. The layer damaged in the process of grinding or polishing (of the tested side of the plate) was removed by chemico-mechanical polish. The removed value was established to an accuracy of 0.1 mg by weighing the plate before and after the treatment. The plate flexure was found to diminish with each removed layer, and to disappear after removal of 7 μm and 0.4 μm thick layer of the ground and polished samples, respectively. The most essential deformation caused by residual stresses was observed in the layers of the ground and polished plates with a thickness of 0.12–0.24 μm and 0.09–0.14 μm, respectively.

Методом определения прогиба исследована зависимость остаточных напряжений от глубины нарушенного слоя в пластинах сапфира диаметром 100 мм после их шлифования и полирования. Прогиб определяли по неплоскостности контрольной стороны пластины с помощью интерферометра ИТ-200. Съем нарушенного после шлифования или полирования слоя (тестовая сторона пластины) производили химико-механическим полированием (ХМП). Величину съема определяли взвешиванием пластины до и после ХМП с точностью 0.1 мг. Установлено, что с каждым снятым слоем прогиб пластины уменьшается, а при снятии слоя толщиной 7 мкм для шлифованного и 0.4 мкм для полированного образцов прогиб исчезает. Показано, что наибольшая деформация сапфира под действием остаточных напряжений наблюдается в слое толщиной 0.12–0.24 мкм для шлифованной и 0.09–0.14 мкм для полированной пластин.

1. Introduction

Due to its unique mechanical and optical properties, sapphire is widely used for the making of optical systems which operate under extreme conditions, substrates for light diode devices, etc. [1]. In most cases sapphire articles must comply with stringent requirements, such as high structure perfection, good quality of surface treatment, the absence of disturbed surface-adjacent layer, a surface roughness (R_a) of ~ 3 Å, a flatness (L) of ~ 5 μm, an optical quality of 20/10 according to the standard USA-MIL-0-13830. The latter is especially significant for substrates which undergo different kinds of treatment (cutting, grinding, polishing) in the process of their production, that results in the formation of a disturbed surface layer. This layer essentially influences the quality and, consequently, the properties of the final products, e.g. stability of epitaxial structures [2, 3], diminution of the lifetime of charge carriers [4], etc.

The obtaining of substrates with minimally disturbed layer and optimization of their treatment conditions require simple nondestructive methods for controlling the substrates at each treatment stage. Such

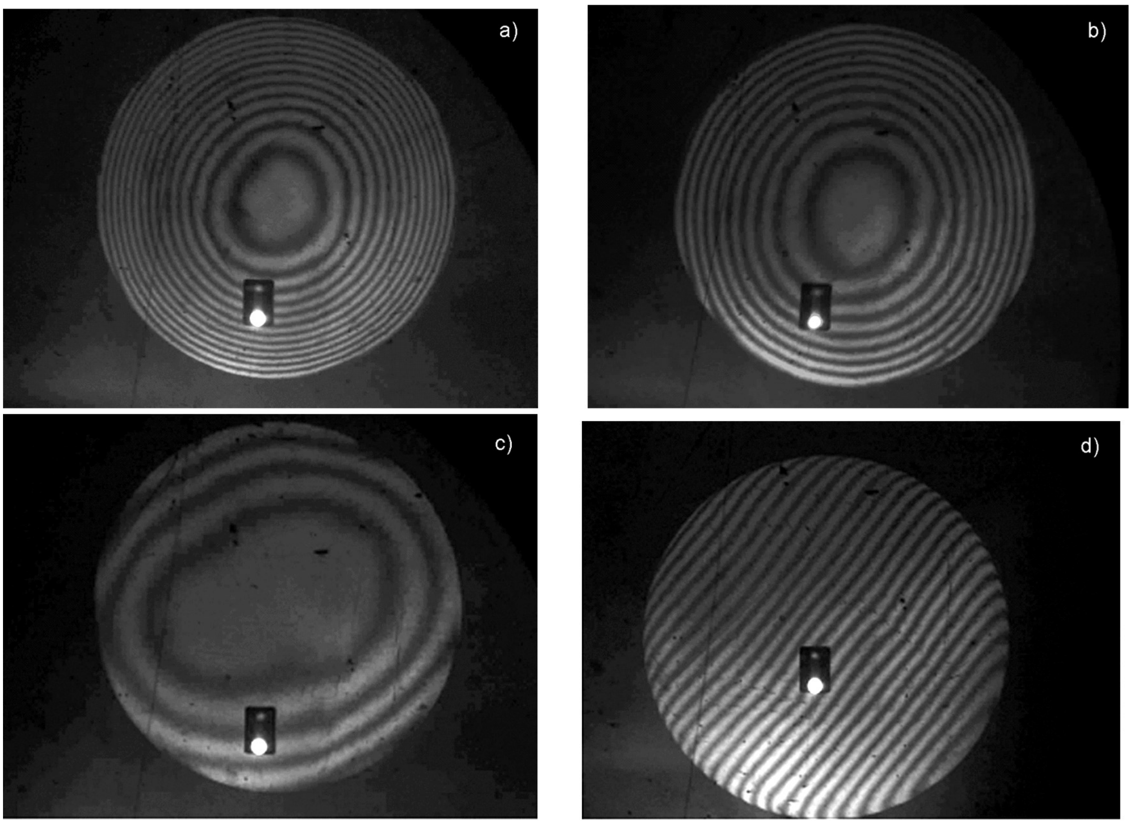

Fig. 1. Interference patterns of the control crystal surface: a — immediately after grinding the tested surface; b, c, d — at further CMP.

methods include direct measurements of flexure [5] or interferometric flatness determination [6]. As is known [7], the plates deflect if their two surfaces have been treated by different abrasives (the Twyman effect). This is caused by the stresses which arise in the process of treatment. So, by removing the disturbed surface-adjacent layer without producing additional stresses (e.g. by chemico-mechanical polish) one can control the flexure of the plate while measuring its flatness. This makes it possible to estimate the value of the disturbed layer and the residual stresses for each kind of treatment.

The present work is aimed at estimation of the depth of the disturbed layer and the residual stresses in sapphire substrates after grinding and polishing.

2. Experimental

The study was performed on 3.75 mm thick sapphire plates with a diameter of 100 mm. The plates were cut out of the crystals grown by the method of HOC in a furnace with carbon graphite thermal shields [8]. After cutting and grinding the crystals were annealed at 1800°C during 10 hours to remove mechanical stresses. Then one of the plate surfaces used for control was polished by means of a copper polisher with free abrasive ACH 1/0, to achieve a minimum non-flatness, and annealed once more during 10 hours at 1800°C. Thereat, the non-flatness of the polished surface did not exceed 1/3 of the Newton ring (0.09 μm) which was controlled by an interferometer of IT-200 type.

The investigation technique consisted in the following. After treating the tested surface of the sample (e.g. grinding with boron carbide of a preset grain size), the sample was weighed, and the interference pattern of the control surface was obtained. Then the method of chemico-mechanical polishing (CMP) was used for sequential removal of the layers from the tested surface of the sample by means of aerosil suspension. After each CMP procedure the sample was weighed, and the interference pattern of the control surface was taken. The removed layer thickness was determined from the difference between the sample's weight before and after CMP, the change in the flatness of the control plate surface was established from the number of the Newton

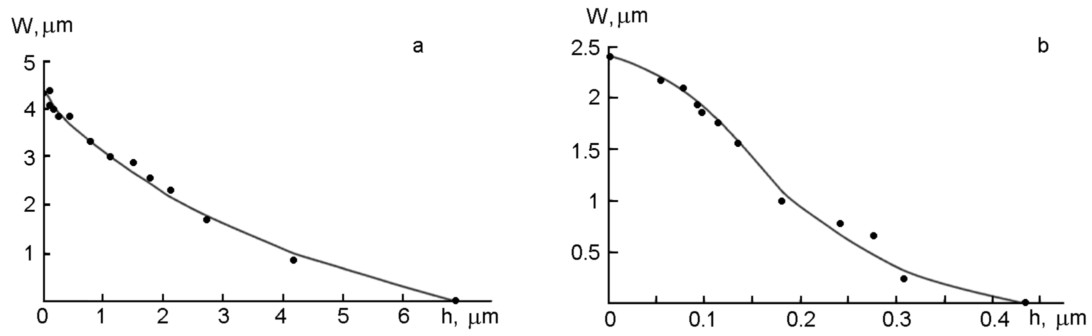

Fig. 2. Dependences of the flexure W of the ground (a) and polished (b) plate on the thickness of the removed surface-adjacent layer h.

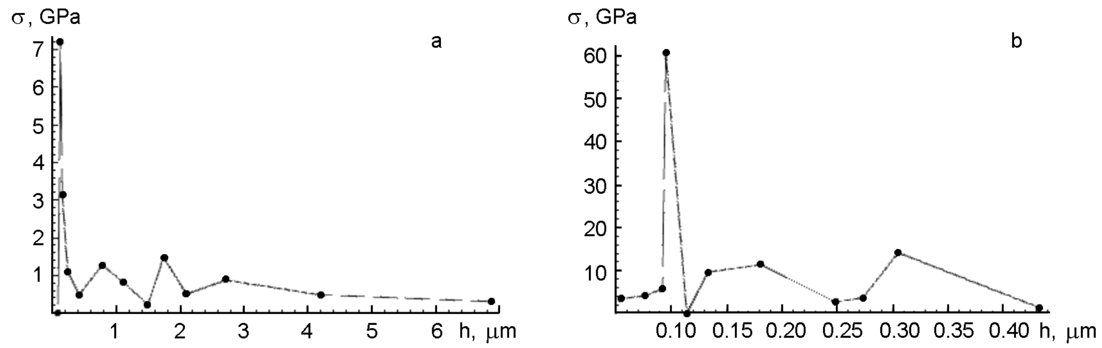

Fig. 3. Dependences of residual stresses σ in the ground (a) and polished (b) samples on the thickness of the removed surface-adjacent layer h.

rings. The thickness of the removed layer was calculated using the relation

$$h = \frac{4\Delta m}{\pi d^2 \rho}, \quad (1)$$

where Δm is the mass loss; ρ, the density of sapphire (3.98 g/cm^3); d, the diameter of the sample.

The loss of the mass of the tested sample was determined from the difference between its mass and the one of the standard sapphire sample of the same dimensions and mass which was used during all the experiments. Prior to the measurements, both samples were held in contact with each other on a massive metallic stand to equalize their temperatures. This allowed to minimize the hygroscopic effect and bring the mass measurement accuracy to ±0.1 mg. In view of a rather large diameter of the samples, the accuracy of determination of the removed layer thickness was ±30 Å.

3. Results and discussion

The flexure of the plate W was calculated from the relation $W = (\lambda/2)N$ (where $\lambda = 0.546$ μm, N is the number of the Newton rings on the control surface of the plate). Visually, i.e. without using a measuring facility, the changes of N can be revealed only to an accuracy of 1/3 of the ring, which corresponds to the accuracy of determination of the sample flexure W (about 0.1 μm).

The process of CMP was terminated when the interference pattern obtained after removing a layer of the sample surface showed the initial value (≤1/3 of the Newton ring) and remained unchanged.

Fig. 1 presents the interference patterns obtained after grinding of the sample with free boron carbide abrasive No.5 and removal of several layers by CMP.

Shown in Fig. 2 are the curves of the dependence of the flexure of ground (a) and polished (b) sample W on the thickness of the removed surface-adjacent layer h. One can see that the flexure of the plate decreases as the disturbed layer thickness diminishes. The thickness of this layer reaches 7 μm and 0.4 μm for the ground and polished sample, respectively.

The distribution and value of residual stresses in the disturbed layer may be estimated from the flexure of the plate after removal of the layer with the thickness h. If to consider the plate isotropic, the flexure

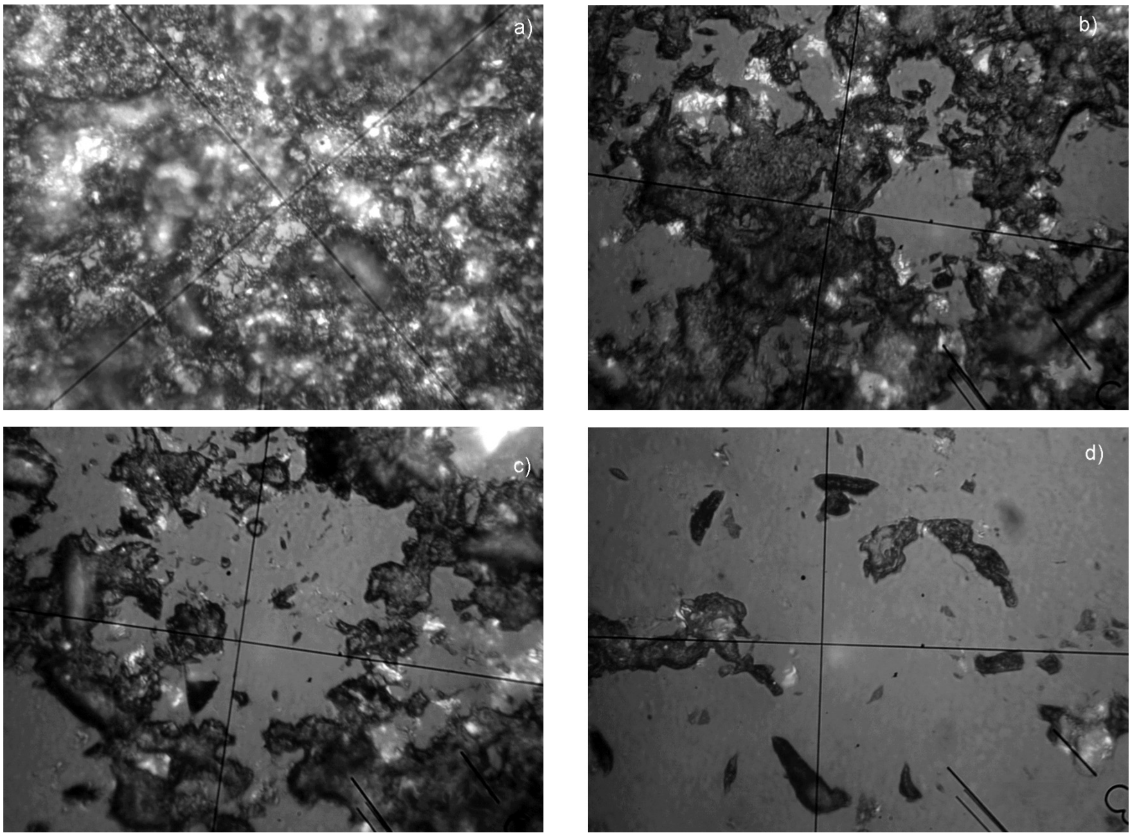

Fig. 4. Microphotographs of ground sapphire surface at layer-by-layer polishing of the disturbed layer: a — immediately after grinding; b, c — during polishing; d — after complete removal of the plate flexure (×300 magnification).

spherical, the stresses uniformly distributed over the surface, then for a flexure lesser than the thickness of the plate the relation between the flexure of the plate and the distribution of residual stresses in it will be expressed by the formula [9]:

$$\sigma(h) = -\frac{E}{3(1-\mu)r^2}\left[(d-h)^2\frac{dW(h)}{dh} - 4(d-h)W(h) + (4d-6h)W(0) + 2\int_0^h f(z)dz\right], \quad (2)$$

where E is the Young's modulus (350 GPa [1]); μ, the Poisson's ratio (0.28 [1]); r, the radius of the plate; d, the thickness of the plate; h, the depth of the disturbed layer. All the terms present in square brackets, except the first of them, may be neglected, since $h << d$.

Shown in Fig. 3 is the distribution of residual stresses depending on the thickness of the removed material. Despite the presence of several peaks on the curves $\sigma(h)$, only one of them, the highest, is to be paid attention to. The amplitude of all the other peaks is on the order of the error of dW/dh derivative calculation. Therefore, a conclusion concerning the character of the distribution of stresses in the ground and polished samples is to be made only after raising the accuracy of determination of the number of the Newton rings in the interference patterns at least by several fold.

The most essential deformation of the samples under the influence of residual stresses is observed in 0.12–0.24 μm thick layer of the ground plate and 0.09–0.14 μm thick layer of the polished plate. At further removal of the disturbed layer the stresses diminish and practically go to zero when the flexure disappears. Thereat, the value of stresses for the polished sample is higher in comparison with the one for the ground sample. This may be connected with the disturbed layer structure and different mechanisms of abrasive wear at grinding and polishing [10–12].

The surface of the ground sample is not smooth, so layer-by-layer polish at first removes the micro-relief peaks, and then these polished areas expand (Fig. 4a–d). The final point of the measurements (Fig. 4d) corresponds to completely removed plate flexure, but the surface is not smooth: it contains defects. Therefore, the disturbed layer is not completely removed.

The depth of the disturbed layer for mechanically polished sample estimated by the method of three-crystal X-ray diffractometry [13] in CuKα1 radiation is ~2 μm [14], which essentially exceeds the value to be assumed proceeding from Fig. 2b.

4. Conclusions

The stresses which arise in the substrate in the process of its treatment and cause its flexure are distributed in a thin surface-adjacent layer. The latter is almost completely removed during CMP, thereat its thickness is lesser in comparison with that of the disturbed surface-adjacent layer. So, this value can be determined only qualitatively. Nevertheless, such an estimation makes it possible to optimize the processes of sapphire treatment.

References

1. Encyclopedia of Sapphire, ed. by E.R.Dobrovinskaya, L.A.Litvinov, V.V.Pishchik, STC "Institute for Single Crystals", Kharkov (2004) [in Russian].
2. J.J.Nickl, W.Just, R.Bertinger, *Mater.Res. Bull*, **49**, 1413 (1974).
3. B.Beaumont, P.Vennegues, P.Gibart, *Phys. Stat.Sol.*, **227(b)**, 1 (2001).
4. A.I.Kurnosov, *Electronic Engineering, Semiconductor Devices*, **7(57)**, 3 (1970).
5. .W.C.Dash, *J.Appl.Phys.*, **29**, 228 (1958).
6. M.A.Okatov, E.A.Antonov, A.Baigodzhin et al., Reference Book for Optical Engineer, Politekhnika Publ., St. Petersburg (2004) [in Russian].
7. A.N.Kovalev, A.I.Kurtaikin et al., Surface Phenomena in Semiconductors, Nauka, Moscow (1976) [in Russian].
8. A.Ya.Danko, V.M.Puzikov, V.P.Seminozhenko, N.S.Sidelnikova, Technological Foundations of Leucosapphire Crystal Growth under Reductive Conditions, "ISMA", Kharkov (2009) [in Russian].
9. O.A.Privarnikov, V.N.Shevchenko, *Super-hard Mater., No.3, 52 (1987).*
10. D.V.Lotsko, Yu.V.Milman, N.M.Torchun, *Surface*, No.8, 136 (1984).
11. A.V.Stepanov, Foundations of Practical Crystal Strength, Nauka, Moscow (1974) [in Russian].
12. I.Ida, M.Sudzuki, M.Kadzuta, *Electr.Commun. Lab.Techn.I.*, **5**, 505 (1969).
13. V.F.Tkachenko, *Single Cryst. and Engin. Coll.*, **12**, 115 (1975).
14. V.F.Tkachenko, O.A.Lukienko, A.T.Budnikov et al., *Functional Materials*, **18**, 171 (2011).

Дослідження залишкових напружень у пластинах сапфіру після їх шліфування та полірування

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Методом визначення прогину досліджено залежність залишкових напружень від глибини порушеного шару у пластинах сапфіру діаметром 100 мм після їх шліфування та полірування. Прогин визначали за неплощинністю контрольної сторони пластини за допомогою інтерферометру ИТ-200. Знімання порушеного після шліфування чи полірування шару (тестова сторона пластини) проводили хіміко-механічним поліруванням (ХМП). Величину зйому визначали зважуванням пластини до та після ХМП з точністю 0.1 мг. Встановлено, що з кожним знятим шаром прогин пластини зменшуться, а при зніманні шару товщиною 7 мкм для шліфованого та 0.4 мкм для полірованого зразків прогин зникає. Показано, що найбільша деформація сапфіру під дією залишкових напружень спостерігається у шарі товщиною 0.12–0.24 мкм для шліфованої та 0.09–0.14 мкм для полірованої пластин.