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# On the nature of changes in the optical characterization produced in sapphire on its irradiation with a pulsed powerful stream of hydrogen ions

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**Abstract** Changes in the optical characteristics in synthetic sapphire specimens produced by microsecond pulse irradiation with a stream of hydrogen ions of energies ranging up to tens keV have been observed. Data on decrease in the optical reflection, measured within the wavelength range of 200–900 nm, are presented. This characterization is compared with the data received by optical and atomic force microscopy as well as by lattice structure analysis performed with X-rays. The measurements indicate that the changes of optical parameters are not a consequence of absorption increase and/or sapphire decomposition. They result from modifications of the morphology and structure of surface layer of the sapphire samples, induced by irradiation.

**Key words** optical characteristics • irradiation • fast ion stream • surface modification • morphology

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## Introduction

Problem of changes in the optical characteristics of transparent inorganic compositions (e.g. sapphire samples) after their irradiation with high-energy streams of hydrogen ions is of importance for aerospace engineering and nuclear fusion technology. In space experiments, sapphire can be used in various devices, which are positioned upon external surfaces of space vehicles. Any changes of the sapphire optical properties, and first of all its transparency and reflectivity, are connected with the influence of many factors occurring within the open space, e.g., micrometeorites, high energy protons, radiation belts of Earth, cosmic rays, solar wind or solar plasma. An inherent part of the radiation constitutes hydrogen ions of energy ranging from keV to several tens of keV [1, 2]. An obvious issue is the determination of a contribution of each mentioned radiation to the damage of optical parts.

In nuclear fusion devices, sapphire plates can be used as reactor chamber windows for measuring and/or monitoring plasma parameters, and for laser beam leading into the chamber volume. In the process of thermonuclear plasma "burning", the parts made of sapphire can be exposed to a continuous or pulsed beams of neutrons from nuclear fusion reactions, $\alpha$-particle streams, X-rays and $\gamma$-radiation, as well as to ions of hydrogen and its isotopes. The energy value of the latter particles results from plasma temperature and acceleration mechanisms inherent to plasma of the particular devices, it lies again within the range from units of keV up to tens of keV [3, 7, 9]. In this study, we investigated the role of fast ions in the modifications of sapphire optical characteristics, as well as the nature of such changes.

## Materials, irradiation conditions and methods of investigation

The main material used in our investigations was a synthetic sapphire, manufactured in the form of disks of 15 mm in diameter and 5 mm in thickness. Each disk had one surface polished and this surface was the subject to irradiation. The rear surfaces of the samples were ground in order to decrease the contribution to the reflection coefficient of the analyzed surface.

As a source of the hydrogen ion stream we used a plasma injector – IBIS facility, operated at the Andrzej Soltan Institute for Nuclear Studies, Otwock-Świerk by Warsaw, Poland. The IBIS device was equipped with an electrode system of the RPI (Rod Plasma Injector) type and a fast-acting electromagnetic gas valve. The system consisted of a set of two coaxial electrodes of 200 mm in length and of 90 mm and 130 mm in diameter, respectively. Each electrode was composed of 32 thin molybdenum rods oriented axially and distributed symmetrically around the cylindrical electrodes. The gas valve was placed at the symmetry axis, but it could puff the chosen working gas into the inter-electrode gap. After the gas puffing, with a selected time delay $\tau$ (when an appropriate gas pressure was achieved in the inter-electrode region), the main discharge could be initiated by the application of a high-voltage pulse from a condenser bank. The experiments were performed mostly at the initial charging voltage $U_0 = 30$ kV corresponding to the bank energy $E_0 = 44$ kJ. Under the chosen experimental conditions that device generated ion streams with an ion energy distribution extending up to 60 keV, and having a maximum at several keV. The pulse duration of the stream was about 1 μs. Hydrogen was used as a working medium. Sputtering of the electrodes of the IBIS device was negligible.

Specimens were placed at a distance of 30 cm from the electrode endings. An energy density of the ion stream varied from shot to shot within the limits from 3.4 to 5.7 J/cm$^2$. The power flux on the sample surface was *ca*. $5 \times 10^6$ W/cm$^2$. The irradiation process was performed during two successive steps. At the very beginning, the virgin samples were investigated optically. After that, the specimens were irradiated within the IBIS facility by a series of 10 pulses, with intervals between consecutive shots equal to about 1 min. Then, optical characteristics of the samples was measured again. After those measurements a new series of 10 pulses was applied at the same IBIS operational mode, and then we performed the next optical measurements. The ion fluxes, as obtained by the samples during successive series of shots, were estimated to reach $5 \times 10^{16}$ and $10^{17}$ ions/cm$^2$, correspondingly.

The percentage of the light reflected from the irradiated surface within the wavelength range 200–850 nm (sometimes extended up to 900 nm) has been considered as the main optical characteristics of the specimens. In our measurements we used a Shimadzu UV-2101PC Spectrometer (produced by the Shimadzu Corporation, Tokyo 101, Japan), working in the reflection regime. A spectral resolution of the device, as determined by the slit width, was 5 nm. Its photometric accuracy in the transparency regime was equal to ±0.3%, and the photometric reproducibility in the same regime was ±0.1%.

A collimated beam of light of the chosen wavelength was reflected from a mirror, and after passing the way of 6 cm it fell (through a 5-mm-diameter diaphragm) upon the surface of the sample under investigation. The angle of incidence of the beam on the surface was 5°. The distance from the diaphragm to the specimen surface could be changed within the limits of 0–5.7 mm (thus from the mirror to the sample in the range 6–11.7 mm). After the reflection from the sample, the beam is coming to the second mirror positioned at the same distance (6 cm) from the diaphragm. Then, passing the distance of 14 cm, it is coming to the entrance of the photomultiplier. All measurements were carried out for different distances between the diaphragm and the specimen.

After the investigation of a spectral dependence of reflectivity of the surface irradiated in two steps, the surfaces were studied first with an optical microscope, i.e. by means of the method of probing adequate namely to the optical material. Then, the use of an atomic-force microscope (AFM) gave information about the morphology of the irradiated specimens with a much higher spatial resolution than in the previous case. In particular, these investigations provide us with the data on the extent (abyssal profile) of the damages, which were produced as a result of radiation and thermal interaction of the beam. Later on, the X-ray diffraction structure analysis supplied data on the material, present on the surface and its lattice parameters as well.

## Results and discussion

### Spectral dependence of the reflection coefficient

The wavelength dependence of the reflection coefficient was investigated both for the polished unirradiated surface (virgin sample) and for the rear (ground) side of the specimen. For the second surface it was measured (in fact the coefficient of scattering) for the sapphire disc turned this side to the probing beam of light and positioned at a distance of 4.8 mm from the diaphragm. This distance was chosen by taking into account that the thickness of the sample (5 mm before irradiation) is about the same.

First, we performed a comparison of the spectral dependences of the reflection coefficient *R%,* as measured for the virgin polished surfaces, with the values computed from the formula valid for an angle of incidence close to normal:

$$R\% = \{(n-1)/(n+1)\}^2 \times 100. \quad (1)$$

Using the table data for the refraction index of sapphire *n* [10], we observed small differences. However, if one takes into account a contribution of the light scattered by the second (rear) surface of the disc to the over-all reflection coefficient (i.e. subtracting the lower curve from the upper one), it practically removes these differences.

Secondly, for our samples a considerable decrease in the reflection coefficient was obtained after the irradiation. For example, a very sharp fall in the ranges 200–250 nm and 400–850 nm was observed after the first irradiation step (see Fig. 1a). The second irradiation step (Fig. 1b) resulted in a progressive decrease in *R%* within the 200–400 nm range, and the maximal fall took place at the extreme left wing of the UV range, where the coefficient

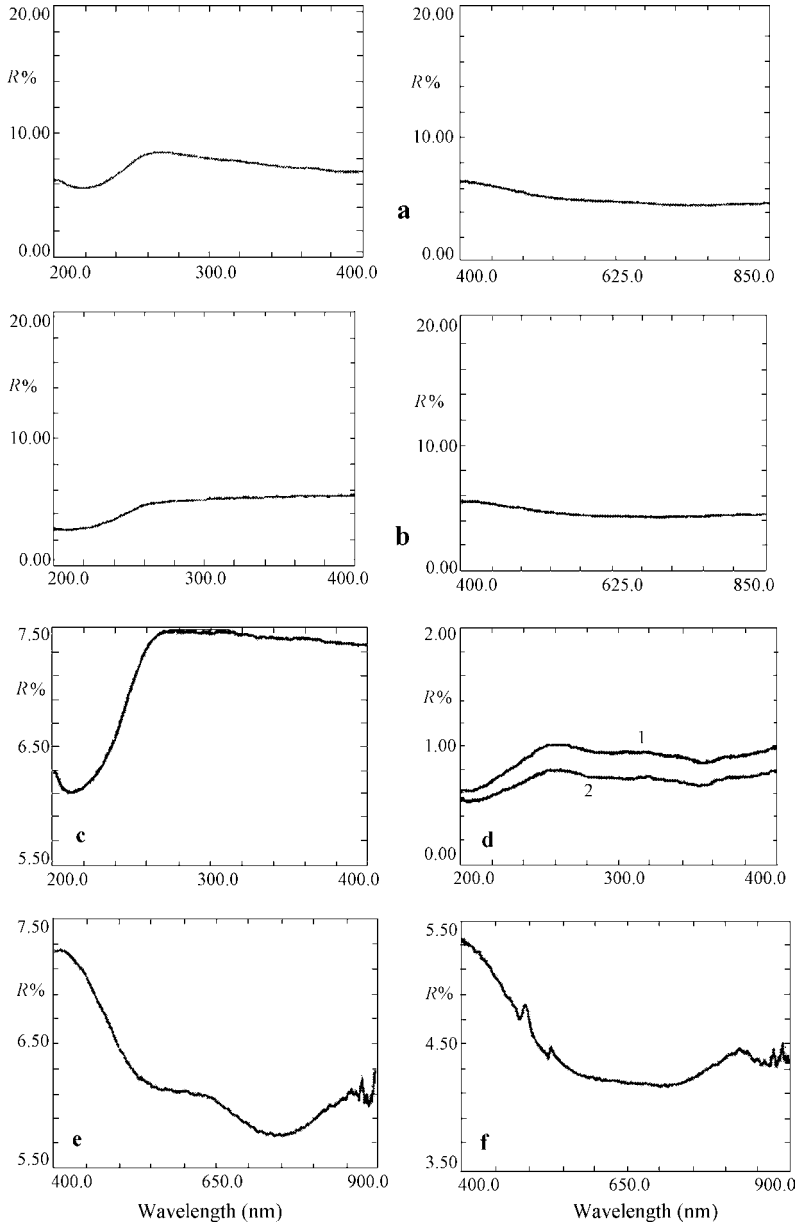


**Fig. 1.** a – Spectral dependence change of the reflection coefficient after the first step of irradiation as a function of the wavelength; b – the same after the second irradiation step; c – the same for the front surface of specimen irradiated during two steps, as measured in the UVV range at a distance from the specimen to the diaphragm equal to $h = 0$; d – the same dependence for the rear matted (ground) surface, as measured at two distances to the diaphragm equal to $h = 0$ and $h =$ 4.8 mm; e – the same dependence for the front surface as measured in the VIR range at a distance of $h = 0$ from the specimen to the diaphragm; f – the same dependence measured in the VIR range at a distance of 5.7 mm between the front surface and the diaphragm.

fell down to 3. In principal, the decrease in the reflection of the samples might be a consequence either of a light absorption inside the surface layers, or of an increase in scattering of light by the irradiated surface due to some changes in its morphology.

More precise examination of the wavelength dependence of *R%*, whose examples are presented on a larger scale in Fig. 1c, 1d, 1e, and 1f, demonstrates interesting variations (peaks and dips) at certain wavelength values.

In the strict sense, a comparison of properties of the volatile species emitted from the surface with those of solid material left or deposited on the sample after irradiation cannot be used as an argument in the physical interpretation of our results. But there are many speculations in the literature (see e.g. [8]) that between the above species one can find sapphire decomposition products. So we have made a collation of the reflection coefficient dependence on the wavelength with the known from the literature absorption bands of the elements and their combinations, which might be created during the sputtering of a sapphire.

The products of sputtering and thermal evaporating processes of a sapphire during their time-of-flight and after their deposition have been investigated in works collected in a book [12] by atomic absorption or emission measurements, with mass spectrometry or with secondary ion mass spectroscopy, by means of neutron activation analysis, by the help of matrix isolation spectroscopy, as well as with a variety of spectroscopic methods (UV-visible, IR, Raman, and ESR). By this way, it appears possible to determine the qualitative and quantitative characteristics of practically all the products of sputtering (ions, atoms and molecules) before their recombination and chemical transformations usually taking place during their deposition upon the substrate.

All the studies mentioned above have shown that, under the action of hydrogen ions of keV energy range onto a sapphire, in the sputtering products during their time-of-flight, besides the atoms and molecules, characteristic for the steady-state thermal evaporation (Al, O, $O_2$, AlO, $Al_2O$, $Al_2O_3$), also not-steady-state molecules such as

$Al(O_2)_2$, $O_3$, $AlO_3$, AlH, AlOH type and some others are present. Spectral dependences of the absorption coefficients of these steady-state and not-steady-state components in UV and VIR ranges are added up [8]. Collation of these curves with graphs, received during our measurements of the reflection coefficient (Fig. 1c, 1e, 1f), shows that no correlation between the literature data on the absorption bands of the above-mentioned compositions and the dips recorded in the spectral distributions of $R\%$ for our specimens can be seen. Moreover, inside some wavelength intervals, where we might expect an absorption increase because of the relatively vast amount of a product made during the sapphire sputtering by protons, just in contrary a peak in the reflection $R\%$ is observed. Such a situation takes place e.g. near 400 nm (25.000 $cm^{-1}$), as shown in Fig. 1e.

This conclusion is supported also by the fact of the perceptible quantitative change of $R\%$ at a relatively small (5.7 mm) increase in the distance (compared with 20 cm) from the specimen to the photomultiplier (see Fig. 1f), which may not be possible for the absorption coefficient. Hence, it seems that we should not expect any substances different from a sapphire upon the irradiated specimen surface, and we have to admit that not the absorption but scattering can be responsible for the results received.

## Centers of light scattering

First of all, since in our experiments the "reflection" coefficient is decreased after irradiation by the value comparable with the original magnitude for the virgin polished surface, it is clear that the scattering cross-section of the centers must be high (i.e. circa $\pi a^2$, where $a$ is the characteristic size of the scattering center), and the scattering "quasi-particles" should be "packed" relatively tightly upon the samples surface. As it is well known, the scattering cross-section might be high only in two cases: first – at the resonance scattering, when the scattered light wavelength is close to that natural for the illuminated substance, and secondly – in the case of the so-called Mie-type scattering [4]. The first case must be excluded since we have shown before that the absorption within the investigated wavelength range does not play a substantial role. Consequently, we deal with the Mie scattering, i.e. the scattering by the quasi-particles having the size $a$ comparable with (or larger than) the wavelength $\lambda$ of the scattered light.

Such scattering particles may have a refractive index $n$ either not much different from the corresponding value $n_1$ for the environment (due to affinity of their densities or because of the flattened shape of it in the beam direction) or showing a strong difference. In the first case – of the so-called scattering on "soft" particles – the process will proceed mainly in the forward direction. In the second case, it will depend on the ratio $n/n_1$. When this ratio is approaching infinity, the particles are "reflecting". If this were the case (e.g. at the reduction of metallic Al from $Al_2O_3$ during its irradiation), the measured "reflection coefficient" should be increased very sharply. But this is not shown by the results of our experiment.

It is evident that one may expect the surface radiation defects of two types – bulges of the material itself or its inflation with creation of hollow inner regions (blisters). In any of these cases, the quasi-particles will be soft, and we may expect a preferable scattering in the forward direction. At such an effect the "reflection coefficient" measured in the reflectometric experiment will be decreased, as can be seen in Fig. 1.

In this situation one has to distinguish cases when the size of the scattering particle is much larger than the light wavelength or approaches this value. In the former case, scattering would be a refraction of the light near the particle borders, but an interrelation between the characteristic size of the particles and a spectral dependence of the reflection coefficient cannot be deduced. In general, it is possible only to ascertain the fact that the surface becomes rough with $a >> \lambda$. Here, a decrease in the reflection coefficient should be seen, as compared with the mirror-like one, and it should be monotonous one within the whole wavelength range. Such a general fall in the reflectivity $R(\lambda)$ throughout the whole range is fixed in Fig. 1. Moreover, a small change (by 5.7 mm) of the distance from the irradiated surface to the diaphragm (see Fig. 1e $\rightarrow$ Fig. 1f) has resulted in a rather considerable (by $\approx$30%) decrease in the "reflection" coefficient. Taking into account the geometry of the verification test experiment and the percentage of the $R\%$ fall, one can also estimate that such "particles" upon the surface have radius 1 $\mu m << a << 1$ mm, and that they are numerous.

The situation $a \rightarrow \lambda$ is of main interest for us because in such a case the scattering cross-section should show maxima correlated with the particles radius. This means that in our experiment certain minima in the spectral distribution of $R\%$ (the Mie resonances) should appear, corresponding to the cases:

$$na/\lambda \approx i, \text{ where } i = 1, 2, 3\ldots \quad (2)$$

On the basis of such resonance events it would be possible to estimate the characteristic sizes of the inhomogeneous regions. Indeed, if one does not take into account a non-monotonous variation (hump) of $R(\lambda)$ in the range near 260 nm, which exists everywhere including also a non-irradiated surface (being intrinsic for the refraction index of sapphire itself), then several valleys near certain wavelengths could be seen. Analysis of the graphs similar to Fig. 1 shows that these valleys might correspond to the surface peculiarities with the characteristic sizes as follows: 200–240 nm, 700–750 nm, 520–580 nm, 490–530 nm and 800–850 nm. The conclusions about the scattering character of the spectral distribution of $R\%$ are also supported by analogous examinations of the backsides of the specimens (Fig. 1d), where these variations correspond to the details of the rough ground surfaces.

In this way, on the basis of the data presented one can conclude that the decrease in the light reflection from the irradiated sapphire specimens results from the morphology changes of their surface layers.

## Morphology and structure defects of the sapphire irradiated surface

In connection with the optical peculiarities mentioned above, direct investigations of morphology and structure defects of the irradiated surface by means of microscopic

methods became of particular interest. Figure 2 represents a 500-fold magnification of the sapphire surface after the second step of its irradiation. One can easily note the presence of many bright spots upon the dark background. This means that we deal here with the "cat effect" observed in the light reflected from a very uneven surface. Indeed, imagine that the original polished surface of our specimen is mainly undisturbed in result of its irradiation, and only few dispersed defects appeared on it. If it were the case then in the picture seen with a microscope in the reflected light, then we should see some dark spots upon the bright background. We have here the opposite case. Thus, the first conclusion, which may be derived from the analysis of Fig. 2, is that the surface (previously polished) became entirely uneven. It looks, namely, as multiple bright spots, because in any surface defect one can find zones oriented perpendicularly to the direction of observations, which consequently reflect the light very strongly.

One can make a rough estimate of characteristic dimensions of the resulted defects from the size of the light spots, which appears to be about 5–10 μm. Thus, the first conclusion about the appearance on the surface of some formations having the size $a >> \lambda$, derived from the reflectometric investigations, has found a firm confirmation here in optical microscopy.

An important issue is the nature of the observed defects. Since they have the reflecting zones, the creations constitute most likely bulges or inflations, not valleys. This conclusion finds its support in the microscopic image of some artificial valleys, which were made on the surface by means of a diamond pyramid during the micro-hardness measurements. These valleys look like black spots not reflecting the light. So, we may suppose that the main defects of the surface layers are the structure damages looking like radiation blisters.

The irradiated surface layer seems to be weakly tied up with the base material. This conclusion was made because of the character of the scratch made by the diamond pyramid. Unevenness and chips, as observed along the scratch length, manifest themselves as evidence of the low adhesion of the irradiated surface layers of sapphire. In this situation, it seems quite plausible that during the irradiation process, with our relatively high flux-density ($>10^6$ W/cm$^2$), the melting and crystallizing processes take place upon the surface layer of sapphire, which are accompanied by the appearance of thermal stresses and creation of the structure defects.

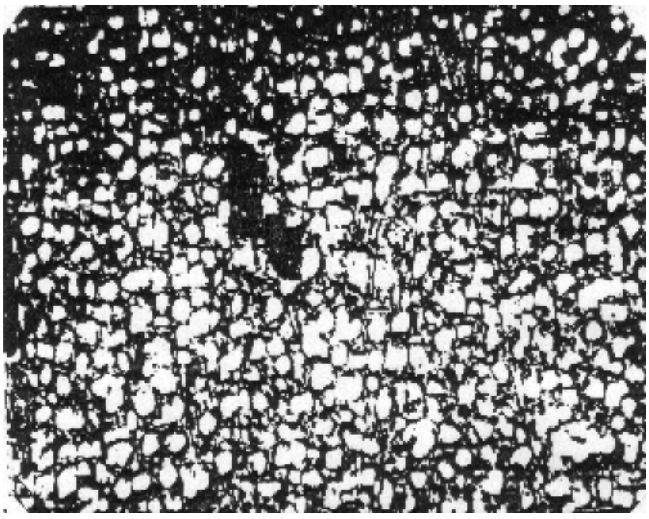

**Fig. 2.** Image of the surface of specimen after the second irradiation step, obtained with an optical microscope at 500x magnification.

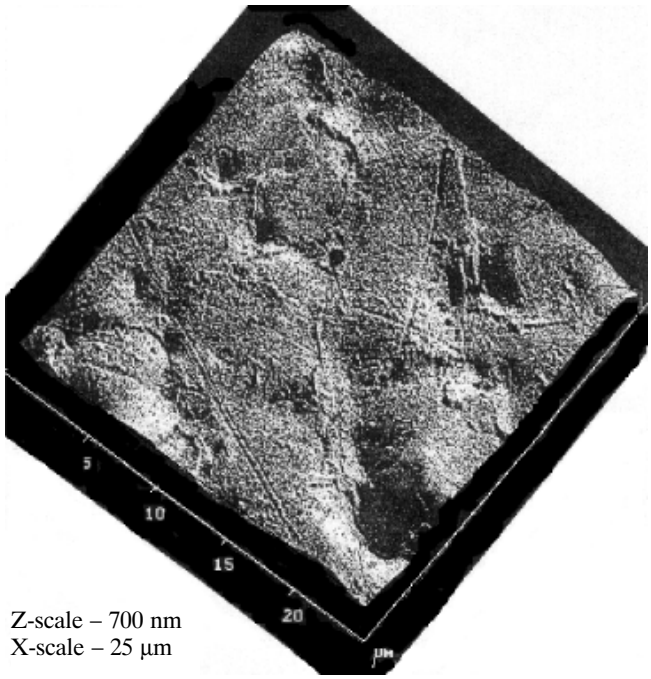


**Fig. 3.** Three-dimensional image of part of the double-irradiated surface of specimen, of dimensions 25*25 mm$^2$, obtained with AFM.

More detailed examination of the sapphire surface has been performed by means of atomic-force microscopy (AFM). As an example, an image of the area surface of the irradiated specimen having dimensions 25*25 mm$^2$, which was obtained with AFM, is presented in Fig. 3. In this Figure, a highly uneven surface of the sample can be easily seen, and one can observe the following characteristic defects:

- inflations of the blister type, with a characteristic size of about 7 μm;
- craters with a parapet (e.g. in the lower angle), with a characteristic size of 4 μm;
- pores without a parapet, having characteristic sizes of 0.2–0.7 μm.

These observations also confirm our previous conclusions, made during the reflectometric examination and optical microscopy, since characteristic dimensions of the surface defects, as observed in AFM, coincide with the dimensions determined with the above-mentioned methods. Surface of the pore walls and the adjacent area show traces of melting.

Figure 4 represents an AFM image of the irradiated area fragment which belongs to a single blister. The surface adjacent to the pore (or crater) appeared to be covered by small spherical inflations having two typical sizes: 0.05 μm (overall fine ripples) and 0.2–0.3 μm (positioned on the figure mainly along the crack). They can be identified with micro-blisters and pores produced by the discharge of some gas component from the bulk.

### Mechanisms of the creation of defects inside the surface layer of irradiated sapphire specimens

Figure 4b presents a graph of the irradiated surface relief. The visible dips and valleys correspond to the craters and pores, whereas peaks represent parapets. One can see that for the craters the ratio of the depth of valleys (having relatively flat bottoms) to the diameter is 1:50, and for the pores (having a conical shape) this aspect ratio approaches 1:1.

Some of the craters have parapets indicating that they are created by the process of throwing away a liquid upon the solid surface. An example of the crater of such a kind can be seen in Fig. 4a, in the center of the blister. The height of the parapet, as determined by a contrast change, is >0.1 μm.

The conical character of the pores reminds some pinhole structures appearing in metals during implanting them with non-soluble gases at high temperatures. Their creation is explained there by coming to the surface (through structure defects) some gases, implanted or produced in the bulk material during its irradiation. In the case of sapphire samples, such forming of gases may implant hydrogen and oxygen. This results from investigations of sputtering and evaporation of the sapphire plates at high temperatures [5, 6, 8, 12].

This examination shows that the nature of craters and pores, produced on the irradiated surface of sapphire, is different. We have made an evaluating computation of the temperature and its distribution within a sapphire specimen for a single pulse of 10-keV protons, with an energy density

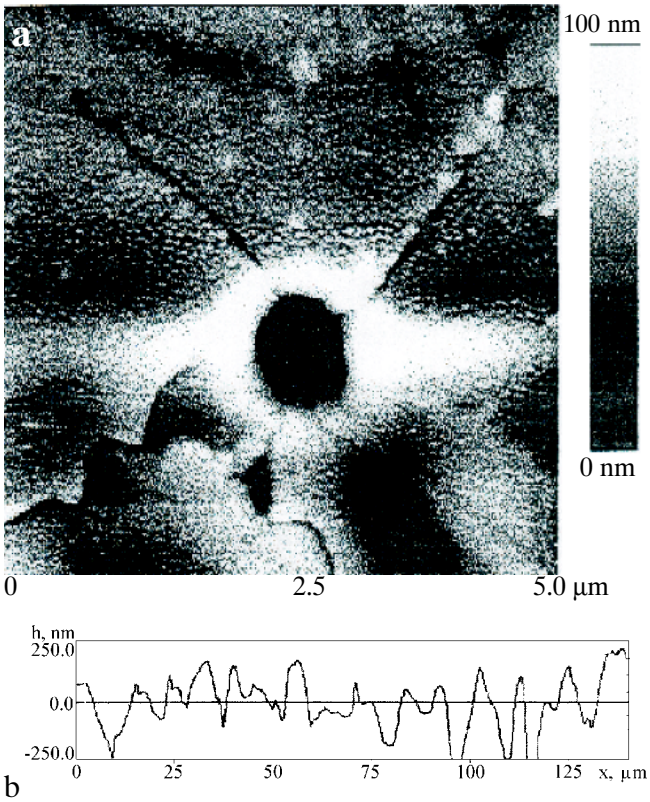


**Fig. 4.** a – Surface of a blister; one can identify a crater with a parapet and some pores and micro-blisters; on the right, the collation of the height scale with the contrast of the AFM image is presented; b – relief of the irradiated surface of specimen obtained with AFM.

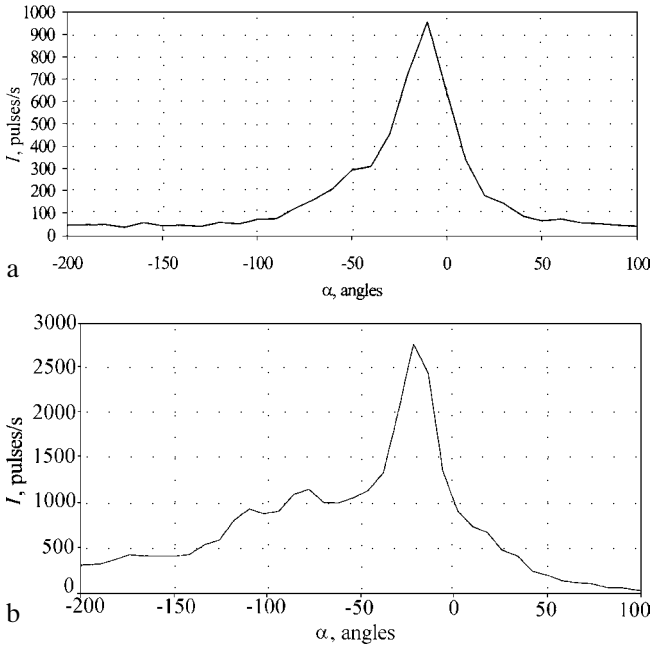


**Fig. 5.** a – Dependence of the X-ray reflection coefficient on the angle of rotation of virgin specimen, obtained with a double-crystal X-ray spectrometer applied for the investigation of the state of the bulk crystal under the radiation-damaged layer; b – the same dependence investigated after two irradiation steps.

flux on the surface equal to 5 J/cm$^2$, and for three probing pulse durations: 1 μs, 0.5 μs, and 0.1 μs, respectively. It should be mentioned that under the stationary irradiation of solids with ions of non-soluble gases, the temperature of blister creation does not exceed ≈0.5 $T_{melt}$ [11], where $T_{melt}$ denotes the melting temperature. From our calculations it can be deduced that with 1-μs pulse the depth of the melted layer of sapphire cannot exceed 0.2 μm. This value is approximately of the order of the projected range of the hydrogen ions (protons) of energy 10 keV in the material (which is equal to 0.13 μm, as it was computed from the numerical model described in [13]).

Thus from one side we had the above-mentioned assertions that the composition of specimens after irradiation remains original i.e. of a sapphire nature (see data of reflectometry). From the other side, we performed the discussion concerning the production of the gas phase from the sapphire sample, due to its decomposition during irradiation (see data of AFM). Therefore, it seemed reasonable to investigate the lattice structure of the near-surface layer. This was done with a diffractometer using the phase analysis of the surface layer in an X-ray grazing incidence mode and with a double-crystal X-ray spectrometer applied for the investigation of the state of the bulk single crystal under the irradiated-damaged layer. Figure 5 represents the results: 5a – for the virgin sample, 5b – for the irradiated surface. One can formulate the following observations:

- No characterizations (e.g. lattice parameters) connected with a material different from the sapphire are manifested themselves in the irradiated surface and in the bulk crystal as well.
- The diffractometer has not demonstrated any diffraction lines within the irradiated surface, and this fact gives evidence in favor of the amorphous state of the layer surface.

– The rotation curve for the reflection from the 204 facet shows that after the irradiation the bulk crystal demonstrates some increase in this curve within the range of small angles. It usually results from the appearance of distortions within the single-crystal layer, lying under the damaged surface. In such a case, the lattice parameter of the bulk single crystal is increased, that may be a consequence of the appearance of defects of the internodal type.

All these observations affirm the fact that sapphire after irradiation remains its original composition. All products of its decay created during irradiation, e.g. the gas phase, which produces morphological changes on the specimen surface, are not presented within the solidified layer.

## Summary and conclusions

The main results of the studies described in this paper can be summarized as follows:

– It has been found that under the action of pulsed (lasting ~1 μs) streams of hydrogen ions with keV-range energies, and characterized by an energy flux of about 5 J/cm$^2$, the reflection coefficient of the polished sapphire surface decreases in a relatively wide spectral interval from UV, through VR to near IR (200–900 nm).
– It has been shown that the decrease in the reflection coefficient of a single sapphire crystal surface results from the light scattering upon structural defects and morphological changes of various types, which are formed in the surface layer of $Al_2O_3$. It is not due to the light absorption in the near-surface layer.
– The creation and evolution of defects inside the near-surface layer determine physical nature of the morphology changes in the specimen surface. It is accompanied by the processes of melting, evaporation, physical and chemical sputtering, as well as by accumulation of the atomic hydrogen and gas products of the sapphire decomposition inside the irradiated layer during irradiation.

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