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Antireflection coating of diamond elements of power optics for CO_2 lasers

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Abstract. **A technology of deposition of a single-layer PbF_2 coating, antireflecting for the CO_2 laser radiation, on diamond substrates is developed. As substrates, use is made of plates of polycrystalline CVD diamond. The transmission coefficient of diamond plates with two-side coating attains 98.5%–99%. The measured laser-induced damage threshold of such plates under normal environment conditions for the pulsed ($\tau \sim 100$ ns) and continuous-wave radiation of CO_2 lasers amounts to 250 and 4 MW cm^{-2}, respectively. These values are comparable with the thresholds of the diamond substrate damage. The increase in the damage threshold of the AR coating under continuous-wave irradiation to 5.5 MW cm^{-2} is found in the case of preliminary multiple irradiation of samples by weaker laser pulses. This effect can be explained by the pollutant removal from the surface of the film without its damage (laser cleaning effect).**

Keywords: *antireflection coating, resistance to radiation damage, diamond optics, CO_2 laser.*

1. Introduction

To satisfy the requirements of rapidly developing laser engineering it is necessary to reduce the mass and dimensions of lasers and other components with a simultaneous increase in power and reduction of laser radiation divergence. To solve these problems new optical materials are required with significantly improved performance. In this context, synthetic diamond is one of the most interesting materials, for which the commercial production technologies have been actively developed since the end of the 20th century.

Diamond possesses unique optical [transparency range from the near ultraviolet to millimetre waves, except the region of background absorption (2–6 μm)] [1, 2] and thermophysical (thermal conductivity above 20 W cm^{-1} K^{-1}) [3] properties. Due to the record-breaking heat conduction, diamond can endure much greater radiation loads than traditional optical materials [4–9]. These facts in combination with up-to-date facilities of synthesis of large-size polycrystalline diamond plates using the method of chemical vapour deposition (CVD) [10, 11] open new perspectives of using diamond as the main material for elements of power optics in high-power lasers, e.g., beam splitting plates, exit windows, lenses and diffractive optical elements (DOEs). In particular, this is true for CO_2 lasers, which are the highest-power sources of pulsed and continuous-wave radiation in the mid-IR range [9, 12, 13].

However, the high refractive index of diamond under normal conditions leads to considerable losses of radiation energy due to Fresnel reflection (up to ~30% from both sides of the substrate). The use of microstructuring of the diamond surface [14–16] or the application of antireflection thin films on it [9, 17–20] allows the reduction of such losses to a few percent. These methods are not free of disadvantages. The fabrication of surface AR microstructures is difficult for initially structured diamond surface, e.g., for the surface of a DOE. In interference AR coatings, they commonly use multilayer and multicomponent films to get minimal reflection losses. However, their radiation resistance considerably decreases with increasing number and thickness of layers [21, 22]. The choice of the film composition is determined by the refractive index of the AR-coated material (substrate) and its optical characteristics in the operating range of laser radiation wavelengths. In this case, it is also necessary to consider adhesive and thermophysical characteristics of the coating. Essential difference of the latter from the characteristics of the substrate material can lead to the damage of the coating due to the effect of heat fields under laser exposure [20–22] or even fatigue damage of the coating due to internal stresses in the process of storage.

The aim of the present study was to make a single-layer AR coating for diamond optical elements affected by high-energy pulsed or continuous-wave radiation of a CO_2 laser having the wavelength 10.6 μm and to study the radiation resistance of this coating.

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2. Technology of deposition and optical properties of the coating

The design of AR coatings, as well as the numerical modelling of their spectral characteristics was carried out using the software packages OptiLayer (M.V. Lomonosov Moscow State University, Russia) and MCalc Multilayer Calculation (Detlef Arhilger, Germany).

The coating was deposited by means of electron-beam evaporation in high vacuum using a SYRUSpro 710 system (LEYBOLD OPTICS, Switzerland). The film growth was monitored using an OMS-5000 spectrophotometric system (LEYBOLD OPTICS, Switzerland). The deposition rate was traced using a quartz crystal microbalance system (INFICON, Switzerland). To improve the coating quality and adhesion, the substrates were heated before deposition and then processed by an ion beam (argon ion cleaning).

The spectral characteristics of the diamond plate with an AR film coating were controlled with a Spectrum 100 Optica IR Fourier spectrometer (PerkinElmer, USA). The measurement of optical constants (refractive and absorption indices) was performed using an IR-VASE ellipsometer (J.A. Woollam, USA).

For power optics of IR lasers ($\lambda \leqslant 8$ μm) oxide-based coatings [18, 19, 22] are commonly used. In the longer-wavelength range the absorption coefficient of oxides considerably grows [23], which is an obstacle for their use in the power optics of CO_2 lasers. The fluoride-based coatings are transparent in the mid-IR spectral range (to 15 μm) and have good adhesion properties. Such coatings also possess resistance to climatic conditions and minimal internal stresses on a diamond substrate, which is of particular importance in their use as elements of power optics.

As a film-forming material for the AR coating the lead fluoride (PbF_2) was chosen, for which the measured refractive index at the wavelength λ = 10.6 μm of the CO_2 laser radiation is $n_{AR} = 1.55-1.56$ [in the absence of noticeable (measurable) absorption]. For the lead fluoride coating the value of n_{AR} practically ideally satisfies the condition of minimal reflection loss for a single-layer quarter-wave AR coating $n_{AR} = \sqrt{n_d n}$, where $n_d = 2.4$ and $n = 1$ are refractive indices of the diamond plate and the environment (air), respectively. Figure 1 presents the calculated dependence of the reflection coefficient on the wavelength in the mid-IR range with the dispersion of the refractive index of the film and the substrate taken into account.

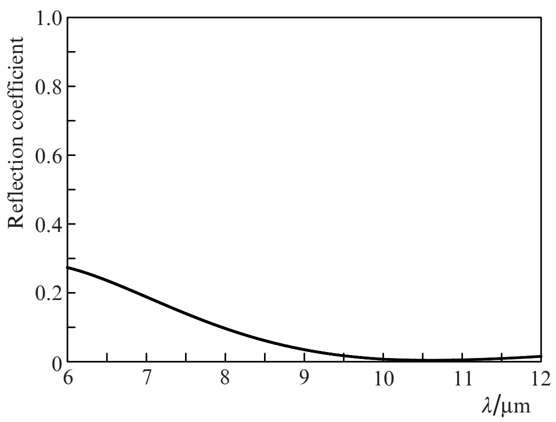

Figure 1. Calculated reflection spectrum of a single-layer AR coating deposited on both sides of a CVD diamond substrate.

The quarter-wave AR coating of PbF_2 was deposited on both sides of a polished polycrystalline diamond plate having a diameter of 1 cm and a thickness of 600 μm. The transmission spectra of the plate in the mid-IR range measured before and after the deposition of the film are shown in Fig. 2.

From Fig. 2 it is seen that the transmission coefficient reaches its maximal value 98.5% in the wavelength region 10–11 μm [spectrum (*2*) in Fig. 2], which corresponds to the wavelength generated by a CO_2 laser. The variation of the wavelength both to long-wavelength and to short-wavelength regions of the mid-IR range leads to a considerable increase in optical radiation losses. The measurement of the transmission coefficient using the CO_2 laser radiation with the fixed wavelength 10.6 μm at low intensities of radiation, known not to damage the coating, yielded even more optimistic value of 99% ± 0.5%. Thus, at the expense of the single-layer AR coating the transmission coefficient of the diamond plate at λ = 10.6 μm was increased by more than 30% (relative to the initial value for the uncoated diamond plate, equal to 68.5%). The measured losses R caused by reflection from the plate surfaces with an AR coating did not exceed 0.5%.

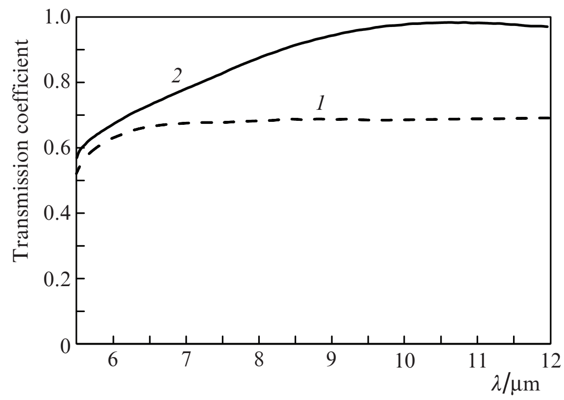

Figure 2. Transmission spectra of a diamond plate (*1*) before deposition of an AR film and (*2*) with the double-side AR coating of PbF_2.

3. Study of radiation resistance of the AR coating

In the experiments on the study of radiation resistance of the PbF_2 coating on a diamond plate, we used a CO_2 laser operating in both continuous-wave and pulsed oscillation regimes. The pulsed regime was implemented using the same CO_2 laser as in Ref. [20], where the resistance of a multilayer broadband coating on a diamond plate was studied, described in detail in Ref. [24]. The laser pulse consisted of a spike having the duration 85–90 ns at the half-maximum level, followed by a mildly sloping part with the duration up to 1 μs. The front spike contained up to 70% of the total pulse energy.

As a source of continuous-wave laser radiation, we have chosen a TL technological laser (JSC 'Laser Technology Centre', Russia). The intracavity aperture used in the laser to improve the radiation quality allowed obtaining the radiation close to a single-mode one with an output power up to 1100 W. The laser power was measured with a calorimeter built-in in the laser scheme. Since in the process of experiments the diamond plate was not cooled, the maximal time of exposure to the laser beam did not exceed one second to avoid the damage due to thermal deformations.

The techniques for measuring the parameters of CO_2 laser radiation, incident on the diamond plate and passed through it, as well as the techniques for performing the experiments on the laser impact, in general, repeat those described in Ref. [20].

The radiation was focused on the surface of the diamond sample by means of spherical ZnSe lenses having the focal lengths 127 and 63.5 mm (for pulsed regime of irradiation) and KCl lens having the focal length 93 mm (for continuous-wave regime of irradiation). In the first case the waist size of the laser beam at the $1/e^2$ level in the lens focus amounted to 400 and 200 μm, respectively, which allowed extreme radiation intensities of 150 and 600 MW cm^{-2}. In the case of cw radiation focusing (into a spot having the diameter 160 μm), the maximal intensity was equal to 5.5 MW cm^{-2}.

The signs of coating damage were recorded using optical methods both in the process of laser irradiation and after it. In the first case, the beginning of the damage process was detected by the reduction of the transmission coefficient of the plate and/or by the change of the radiation intensity distribution in the spot of a probe beam of cw radiation from He–Ne laser. The probe beam passing through the region affected by the CO_2 laser was projected onto a screen so that the characteristic size of the laser spot amounted to ~7 mm. Even a minor damage of the coating film caused diffraction of the probe radiation with $\lambda = 0.63$ μm, leading to the appearance of a black point having the size of 0.5–1.0 mm, surrounded by a bright aureole, in the initially uniform light spot. After the effect of the CO_2 laser radiation, the condition of the AR coating and the bulk diamond was checked using an optical Axiotech Vario microscope (Carl Zeiss, Germany). The spectra of optical transmission in the mid-IR range (5–14 μm) of the exposed plate were also measured. The spectral characteristics were monitored both in the regions subjected to intense laser irradiation and beyond them (for the intact diamond plate with the double-side coating). The measurements of IR spectra in the studies of the coating radiation resistance were carried out using an IFS-113v vacuum IR Fourier spectrometer (Bruker, USA) with the microscope that allowed the optical transmission spectra to be measured for the diameter of a probe radiation beam as small as 40 μm, which was appropriate to the conditions of our experiment.

4. Results of experimental studies of radiation resistance of the AR coating and their discussion

Pulsed regime. The first series of experiments was carried out with the lens having $f = 127$ mm which has been previously used in our studies of radiation resistance of a broadband multilayer AR coating of diamond [20]. It was found that even at maximal CO_2 laser radiation intensities available under these conditions (up to 150 MW cm^{-2}) no signs of damage of the PbF_2 film and the diamond itself can be observed. Further studies in this regime were performed with a smaller focal length of the lens ($f = 63.5$ mm) that allowed one to attain the radiation intensity up to 600 MW cm^{-2}.

Under the pulsed impact of CO_2 laser radiation, the threshold of the near-surface plasma appearance and the change of spatial intensity distribution of the probe He–Ne laser radiation was detected at $I_{th}^{pulse} = 250$ MW cm^{-2}. However, there were regions of the irradiated surface, where the appearance of plasma glow near the focusing spot was not accompanied by any changes in the intensity distribution of the probe He–Ne laser radiation up to 500–600 MW cm^{-2}. Optical microscopy of the diamond plate surface revealed the presence of considerable symmetric damage of the PbF_2 film both in the presence of recordable changes in the spatial intensity distribution of the probe radiation at $I \approx I_{th}^{pulse}$ (Fig. 3a) and in the absence of such changes at $I \approx$ 600 MW cm^{-2}.

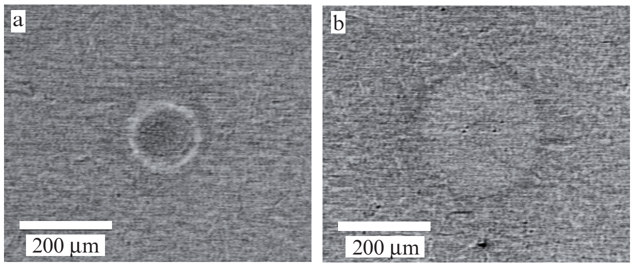

Figure 3. Microphotographs of damaged regions of the AR coating irradiated by a single CO_2 laser pulse with I = (a) 300 and (b) 550 MW cm^{-2}.

Then we measured the transmission coefficient of the plate with a double-side AR coating as a function of the number of incident pulses with the intensities exceeding I_{th}^{pulse}. The impact of a single laser pulse with $I \approx 300$ MW cm^{-2} reduced the transmission coefficient from the initial value 99% ± 0.5% to 96% ± 0.5%. Increasing the number of pulses with the same intensity, we observed no additional change in transmission. No visible changes with the increased number of incident pulses was also fixed in the character of the coating damage as compared to that presented in Fig. 3a. Noticeable changes were observed only when the intensity of laser radiation was increased to a maximal available value of 600 MW cm^{-2}. Figure 4 presents the dependence of the transmission coefficient of the diamond plate with the AR double-side coating on the number of CO_2 laser pulses with $I \approx 600$ MW cm^{-2}. The dashed line in Fig. 4 corresponds to the transmission coefficient 68.5% of the diamond plate without an AR coating.

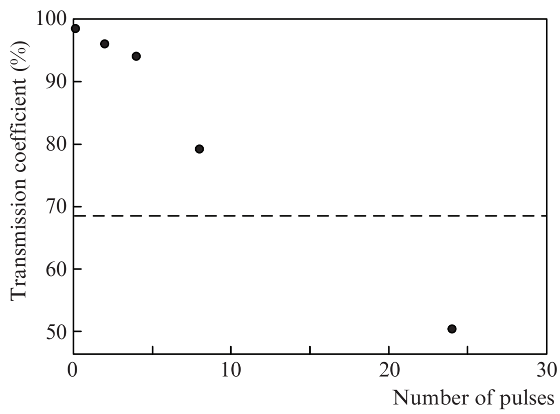

Figure 4. Dependence of the transmission coefficient of the diamond plate with a double-side PbF_2 coating on the number of laser pulses with $I \approx 600$ MW cm^{-2}. The dashed line corresponds to the transmission coefficient of the diamond plate without an AR coating.

When the number of laser pulses having the chosen intensity is increased to 24, we observed not only the coating damage, but also the damages in the bulk diamond, which reduced the transmission coefficient below the initial level of uncoated diamond plate, i.e., the effect of accumulation of damages, also known as optical fatigue, took place.

Continuous-wave regime. In the process of studying the radiation resistance of the AR coating with the continuous-wave CO_2 laser, the intensity of irradiation was varied from 100 kW cm^{-2} to the maximal possible value under the conditions of our experiment (5.5 MW cm^{-2}). The time interval τ of laser exposure, chosen in the range 0.6–1 s, was controlled using a mechanical gate installed at the output of the CO_2 laser. The condition of the coating and the bulk diamond was monitored after each laser impact by means of an optical microscope. A gradual increase in the radiation intensity has shown that at $I_{th}^{CW} = 4.2-4.4$ MW cm^{-2} the damage of the AR coating of the diamond plate occurs in 75% of all cases.

A microphotograph of a typical damage region, obtained by the optical microscope, is presented in Fig. 5. It draws attention that the film damage zone is asymmetric, in contrast to the case of pulsed irradiation. The initial spatial distribution of the laser radiation intensity in the focusing spot was symmetric with respect to the beam axis and close to Gaussian, as in the case of pulsed radiation. The dark spot shifted up from the centre of the irradiation area in Fig. 5 (the characteristic size of the damage region being 160 μm) corresponds to the region of a maximal damage of the film surface. We believe that this asymmetry can arise because the inhomogeneities of the film itself, the defects of the initial diamond surface, as well as the dust particles from air and the layer of surface adsorbate can serve as epicentres of the damage process [25].

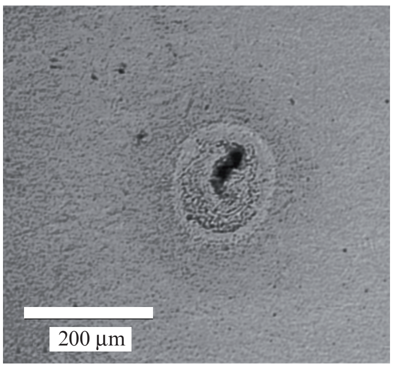

Figure 5. Microphotograph of the damaged region of the AR coating irradiated by a 10.6-μm continuous-wave CO_2 laser at $I = 4.5$ MW cm^{-2} and $\tau = 0.6$ s.

Experimentally we detected considerable influence of a surface adsorbate (apparently, a mixture of water and particles) on the process of the AR coating damage under continuous-wave irradiation. At first, the tested sample was shifted from the focal plane in the direction opposite to the laser beam propagation, towards the lens. Then, by moving the sample gradually back to the focal plane, the characteristic size of the laser beam spot at the surface of the diamond plane was reduced from 1 mm to the initial 160 μm. Smoothly varying the radiation power and the beam spot size, we managed to reach the intensity of 5.5 MW cm^{-2}, which was maximal possible in our experiments. In this case, no signs of the AR coating damage and bulk diamond was found. We associate this fact with laser cleaning of the film surface [25] in the subthreshold regime.

Figure 6 shows the transmission spectra of the areas of a coated diamond plate, where the damage of an AR-coated film was observed [spectra (*2*) and (*3*)], and the spectrum of the region preliminarily treated using the laser cleaning method, after the impact of the radiation with I = 5.5 MW cm^{-2} [spectrum (*4*)]. For comparison, Fig. 6 also shows the spectrum of the AR-coated intact plate [spectrum (*1*)]. Spectrum (*2*) in Fig. 6 corresponds to the region of AR coating damage, shown in Fig. 5. From Fig. 6 it is seen that the damage of the film leads to noticeable loss of radiation at the operating wavelength 10.6 μm [spectra (*2*) and (*3*)], and that after laser cleaning the laser irradiation with $I > I_{th}^{CW}$ does not affect the spectral characteristics of the AR coating [spectrum (*4*)]. Hence, the difference between spectra (*2*) and (*3*) for the damage zones, obtained under close experimental conditions, is probably due to the presence of surface adsorbate and particles at the intact nonirradiated surface. The reduction of the transmission coefficient below the level of polycrystalline diamond without the AR coating (horizontal dashed line) in the case of $I = 4.5$ MW cm^{-2} [spectrum (*3*)] is most probably explained by the scattering and absorption of radiation both at the defects of the damaged PbF_2 film and at the possible defects in the diamond substrate.

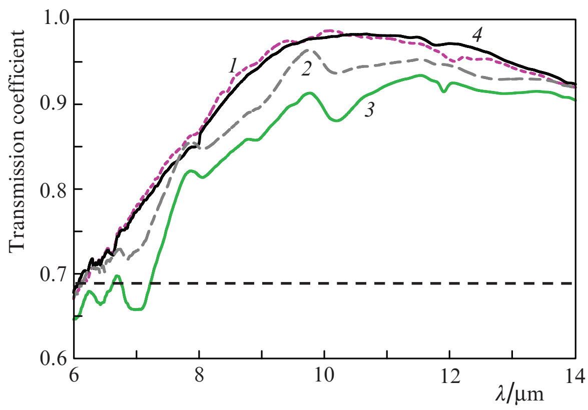

Figure 6. Transmission spectra of the diamond plate: (*1*) before the action of the continuous-wave CO_2 laser; (*2*) and (*3*) for the damaged regions of the film after laser irradiation with $I = 4.4$ ($\tau = 0.8$ s) and 4.5 MW cm^{-2} ($\tau = 0.6$ s), respectively; (*4*) after the laser cleaning procedure followed by laser irradiation with $I = 5.5$ MW cm^{-2} ($\tau = 1$ s).

We estimated the coating temperature T in the centre of the irradiated spot at the surface of the plate, basing on the solution of the heat conduction equation [26] (without allowance for nonlinear absorption). The highest temperature variations are achieved if we assume that all losses of radiation observed experimentally in the AR-coated element (~0.1%) with the reflection $R \leqslant 0.5\%$ taken into account are due to the optical absorption in a ~1-μm-thick coating. In this case the model of surface heat source is implemented with heat removal mainly into the diamond substrate [the thermal conductivity of diamond $k_d \geqslant 20$ W cm^{-1} K^{-1} significantly exceeds that of $PbCF_2$ ($k = 0.014$ W cm^{-1} K^{-1} [27])]. This assumption yields a maximal change of temperature $\Delta T \leqslant 100$ K both for pulsed and for continuous-wave laser regime, which is much lower that the melting temperature of PbF_2 ($T \approx 1100$ K). Thus, under our conditions the laser heating cannot cause the damage of the coating. Apparently, the key role in real processes of absorption of laser radiation energy and heat removal from the laser-affected region should be played by the defects of the diamond plate itself or by the local exfoliation and inhomogeneities of the coating [28], as well as by the surface aqueous adsorbate and particles from air, surrounding the diamond element [25]. This explanation also agrees with the spread of damage thresholds detected in our experiments with pulsed laser impact (see Fig. 3).

The damage threshold of a single-layer AR coating determined experimentally under the pulsed CO_2 laser irradiation is comparable with that of polycrystalline diamond [6–9, 14] and significantly exceeds that of the multilayer coating intended for the wavelength range 8–12 μm [20], also comprising PbF_2, ($I \geqslant 250$ MW cm^{-2} instead of $I = 50$ MW cm^{-2}). In the case of continuous-wave irradiation, the radiation resistance of the single-layer film is also higher, but the increase in the threshold intensity is less expressed ($I \geqslant$ 3 MW cm^{-2} instead of $I = 4-5.5$ MW cm^{-2}, respectively). We relate this fact to a considerable increase in the beam spot size in the present studies of radiation resistance under the continuous-wave irradiation (from 35 to 160 μm), which affects the processes of absorption and heat removal from the region of laser impact, determined by the defects of the diamond sample with an AR coating.

5. Conclusions

We have demonstrated the prospects of using single-layer quarter-wave PbF_2 films as AR coatings for diamond optics of high-power CO_2 lasers operating in both the pulsed and continuous-wave regimes. The damage threshold intensities of such AR-coated films appeared to be comparable with the radiation resistance of diamond plates themselves [6–9, 14]. They also do not yield to the laser damage thresholds of subwavelength surface structures [15], possessing at the same time a number of advantages (simplicity of fabrication technology, the possibility of application to nonplanar surfaces of optical elements, etc.).

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