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# Anti-reflection Coatings Resistant to High Energy Laser Radiation

Optical properties of single, double and triple anti-reflection films have been examined, with the emphasis on their resistance to laser-induced damage ($\lambda = 1.06$ μm). The average damage thresholds for anti-reflection have been determined.

## 1. Introduction

The following types of anti-reflection coatings are most commonly used: a single anti-reflection film of optical thickness $L(\lambda_0/4)$, a double layer of the type $H(\lambda_0/4)$ $L(\lambda_0/4)$, and a triple layer of the type $L(\lambda_0/4)$ $H(\lambda_0/4)$ $L(\lambda_0)/4$, where $L$ and $H$ denote the materials, the respective refractive indices of which are relatively lower and higher than that of the substrate [1]. The investigations carried out in our Laboratory [2] have shown that calcium fluoride is characterized by poor mechanical resistance and low refractive index, its value depending upon the applied technology of evaporation, whereas thorium fluoride shows a medium refractive index and an excellent mechanical and chemical resistance. Magnesium fluoride has been for long time applied to anti-reflection coatings. Examinations of optical properties of rare earths oxides, ytterbium oxide [3] and gadolinium oxide [4] have shown that their indices of refraction (1.9-2.9), and mechanical and chemical resistance are high. The following types of anti-reflection layers have been selected for the investigation of their resistance to laser-induced damage: single ($CaF_2$ and $MgF_2$), double ($Gd_2O_3$-$MgF_2$ and $Yb_2O_3$-$MgF_2$) and triple ($MgF_2$-$ThF_4$-$MgF_2$) layers. The paper [5] which was published in the course of our studies dealt with the same type of triple layers deposited on quartz substrates.

## 2. Experimental Part

For anti-reflection films the BK-7 glass was used as a substrate, because of its refractive index being approximately equal to that of neodymium glass. The films were evaporated on optical wedges (25×35 mm, 5° wedge angle) in order to eliminate the light reflected from the back side of the glass.

Prior to evaporation the glass was carefully cleaned chemically (polished with a fine powder, then washed in running water, water solution of acetic acid and alcohol, consecutively) and dried by centrifugation. Thereupon the substrate was cleaned in vacuum by a 10 minute ionic bombardment; a needle valve has been applied to maintain a constant air pressure $10^{-2}$ Tr. All films were deposited in the Edward's 19E-7 vacuum unit. This unit does not offer possibility of heating the substrate during evaporation. During preliminary evaporations the substrate was heated to 250°C, it appeared that the temperature lowers down to 180°C during evaporation process lasting typically for a few minutes. In order to assure a constant temperature of the substrate during the condensation an additional heat supply system has been applied. Under the above conditions, the substrate was maintened at the level of 250°C for all coatings, except for the $CaF_2$ layer, which was deposited on a substrate at the temperature 110°C.

The pressure during evaporation was held at $1\times10^{-5}$ Tr (except for $Yb_2O_3$ layers, where $p = 1\times10^{-4}$ Tr). The substrates were rotated at the rate $n = 20^{\text{rew}}/\text{min}$ during ionic bombardment, cleaning and evaporation. Resistance heaters were employed to evaporate the materials. Boats of wolfram sheets were used for $Yb_2O_3$ and $Gd_2O_3$ materials, while $ThF_4$ and $MgF_2$ were evaporated from molybdenium boats. Tantalum boats were applied for $CaF_2$.

Optical thickness of the layers during the process of evaporation was controlled photometrically using a narrowband interference filter ($\lambda_{max} = 500$ nm).

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The reflection coefficients of antireflection films were measured 24 hrs after the layers had been taken out from the vacuum chamber. The measurements were taken by means of a special reflectance attachment adjusted to the VSU-1 Zeiss spectrophotometer. This unit designed by Dr Wilk was constructed in our laboratory [7]. Relative reflection coefficients of coatings, as reffered to those of the optic wedge without any coating, were measured for perpendicular incidence of the light beam. The refractive index of the substrate as well as the reflection coefficient in the used spectral range were known. Next, the absolute values of the reflection coefficient $R$ were determined. The measurement accuracy of the reflection coefficient amounted to 0.01%. In order to determine the changes in reflectance caused by the ageing process the layers were remeasured after 5 months and 15 months, respectively. In the meantime the samples were stored under normal atmospheric conditions. It has been stated that maximal changes in the reflectivity $R$ were less than 0.1% for all films. Preliminary investigations of the film resistance to the laser irradiation were conducted at the Institute of Physics, University of Poznań. The films were irradiated several times with a laser beam of 2 kW power and $\lambda = 1.06$ μm. No damages of the surface were detected visually. The same films were then examined at the Quantum Electronic Institute, Military Academy in Warsaw. A scheme of the measuring system is shown in Fig. 1.

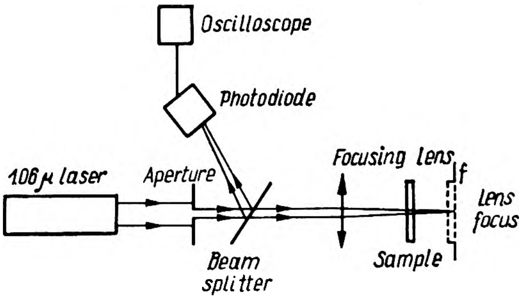

Fig. 1. Diagram of the apparatus used for the damage treshold determination

The applied neodymium laser ($\lambda = 1.06$ μm) generated giant pulses of the energy (0.3–0.5) J of 30 ns duration. The light beam was focused by a lens of 15 cm focal length. The films deposited on the wedges were positioned perpendicularly to the laser beam and shifted along the beam axis enabling to alter the pulse power surface density on the sample. Both pumping conditions and geometry of the setup during the examinations were constant. In investigations described in [5] the films examined deposited on the plane parallel plate, were placed in the focus of a lens ($f = 6$ m), whereas the laser beam power was changed by Schott filters. The sample was placed at the angle 17° with respect to the laser beam.

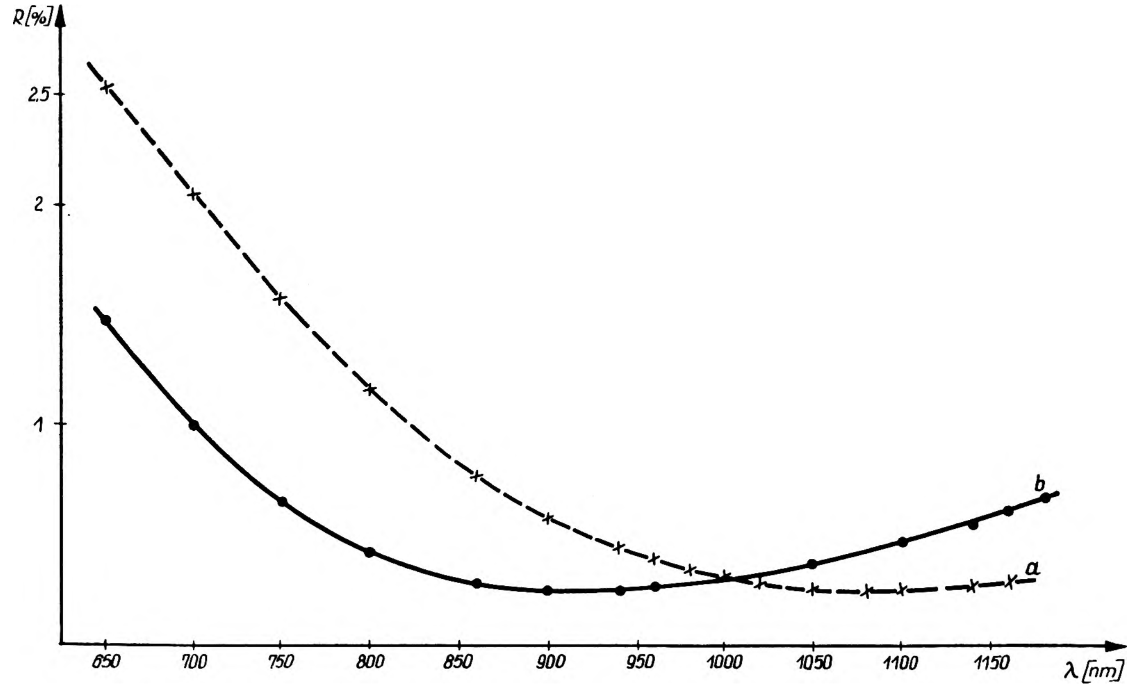

Fig. 2. Reflectance $R$ versus the wavelength for single $CaF_2$ antireflection coatings. Films ($a$, $b$) were obtained by the same evaporation process

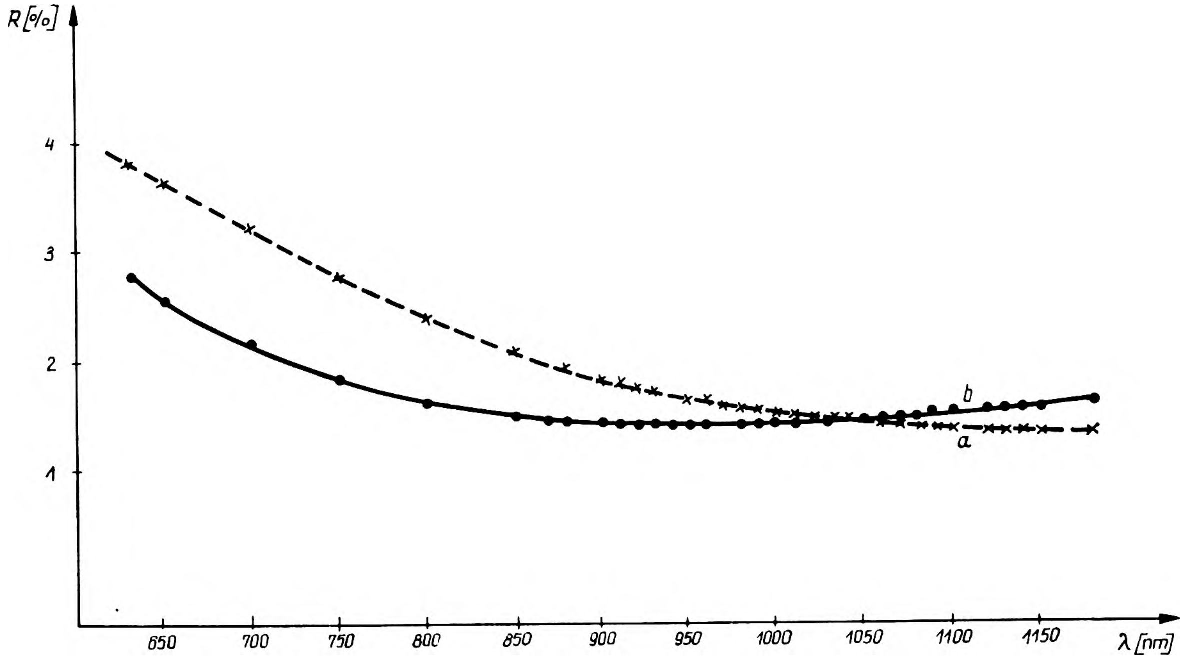

Fig. 3. $R = f(\lambda)$ for $MgF_2$ antireflection coatings. Films *a*, *b* were obtained by the same evaporation process

## 3. Results and Discussion

Spectral characteristics of reflection coefficients *R* for single $CaF_2$ and double $Gd_2O_3$-$MgF_2$ anti-reflection films are shown in Figs. 2–4. The spectral curves *a* and *b* in Fig. 2 refer to the $CaF_2$ films obtained from the same evaporation process. Analogical curves for $MgF_2$ are given in Fig. 3. From Figs. 2 and 3 it may be seen that the films differ among one another in thickness, despite the rotation of plate holder with samples. Nevertheless, $\Delta\lambda$ the reflection curves presented in the graphs for single anti-reflection films are relatively flat within the wavelength range about $\Delta\lambda = 150$ nm, when compared to the spectral characteristics of the film.

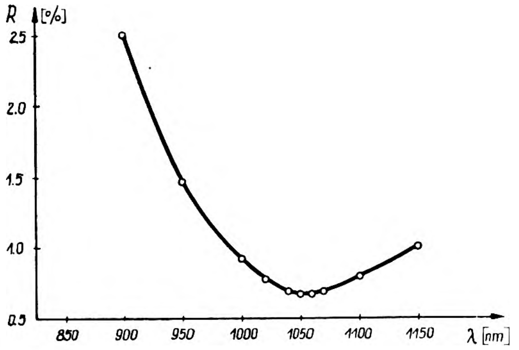

Fig. 4. $R = f(\lambda)$ for double $Gd_2O_3$-$MgF_2$ anti-reflection coating

| Anti-reflection film | Base | Coefficient of reflection $\lambda = 1.06\mu m$ | Damage threshold $MW/cm^2$ |
|---|---|---|---|
| $MgF_2$ | BK-7 glass | 1,4% | 500 |
| $MgF_2$ | neodynilium glass | 0,7% | 250 |
| $CaF_2$ | BK-7 glass | 0,25% | 800 |
| $Cd_2O_3$-$MgF_2$ | BK-7 glass | 0,7% | 700 |
| $Yb_2O_3$-$MgF_2$ | BK-7 glass | 2% | 300 |
| $MgF_2$-$ThF_4$-$MgF_2$ | BK-7 glass | 0,3% | 500 |

The spectral curve of a double-layer $Gd_2O_3$-$MgF_2$ coating (Fig. 4) shows that reflectance rises much more steeply toward longer and shorter wavelength than it does that for a single-layer film. Numerical values of the reflectance for the examined anti-reflection coating (at $\lambda = 1.06$ $\mu$m) and the determined thresholds are presented in Table. The films were irradiated repeatedly (4–8 times) with a laser beam at different regions of their surfaces. First detectable damage of the film manifested by a visible breakdown plasma was stated when the sample was placed at 1 cm distance to the focus. The average surface density value of the pulse power, corresponding to the threshold damage, has been determined by measuring the laser pulse energy, its duration and the irradiated area of the film. From Table it follows that the highest average damage threshold occurs for the single $CaF_2$ and double

$Gd_2O_3$-$MgF_2$ anti-reflection films. A high resistance of anti-reflection $CaF_2$ films to the laser-induced damage compared with its low mechanical resistance is somewhat surprising. In recently published paper [6], concerned with the resistance of thin films to laser radiation ($\lambda = 0.6943$ μm) the stated damage threshold for $CaF_2$ films ranged within 50 J/cm²–300 J/cm². No other details, however, have been given.

The damage threshold for single $MgF_2$ and triple $MgF_2$-$ThF_4$-$MgF_2$ anti-reflection films, determined in our laboratory, are lower than those given in [5]. Although we have applied another measuring system, it seems to be most probable that the discrepancy of the results is mainly due to differences in technology applied to film production. In paper [5] electron gun and quartz substrate have been used. The influence of the substrate on the properties of films are significant. From Table it follows that the resistance of anti-reflection $MgF_2$ films, deposited on a wedge made from neodymium glass, is by 50% lower than that of identical films but deposited on a glass substrate. Microscopic observations have shown that the irradiation of films deposited on neodymium glass induced the damages of both film and substrate. In the case of film deposited on glass no damage of the substrate was observed.

The investigations carried out in our laboratory have allowed to state good resistance to radiation not only in single $CaF_2$ but also in double $Gd_2O_3$-$MgF_2$ anti-reflection films.

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## Couches antiréfletchissantes résistantes à l'influence du rayonnement laser

On a étudié les propriétés optiques des couches antiréflechissantes simples, doubles et triples ainsi que la résistance de ces couches à l'influence d'un faisceau laser ($\lambda = 1{,}06$μm). On a déterminé les seuils de défaut moyens pour les revêtements antiréflechissantes respectifs.

## Просветляюще покрытия, стойкие к воздействию лазерного излучения

Проведены исследования оптических свойств однослойных, двухслойных и трехслойных пресветляющих покрытий, а также стойкости этих покрытий к воздействию лазерного пучка ($\lambda = 1{,}06$μм). Определены средние пороги повреждений для отдельных просветляюших покрытий.

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