Considerations concerning the realization and testing of optical coatings for high-power lasers at 1.06 μ m

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In this paper some recent results in producing and testing of optical coatings for high-power Nd:glass lasers at 1.06 μ m are presented. High reflective (HR), partial reflective (PR) and antireflective (AR) coatings have been investigated and a satisfactory damage threshold (of about 1 Gw/cm² for a 17 ns pulse length) was obtained. Experimental data are presented and discussed taking into consideration a large number of parameters concerning the materials and their properties, the structure of the coatings and the technological background.

1. Introduction

It is well known that in a laser system the coatings of the optical components represent one of the weakest parts. In order to obtain reliable and efficient high-power solid state lasers highly resistant optical coatings are indispensable. This task is realized in at least two stages, i.e., first, to develop reproducible methods for obtaining such coatings, and second, to develop respective testing methods. In both the stages a considerable number of parameters should be maintained within the controlled limits. Sometimes, both in technology and testing, there remain some uncontrollable factors, leading to the undesired dispersion of results, which affect their correct interpretation. This may explain very diversified results obtained even in testing of similar samples (the same structure and the same technology) as it was demonstrated by round-robin experiment concerning identical Balzers coatings [1].

The purpose of our research was to obtain three types of coatings, namely high reflective $(>99^{\circ}/_{0})$, partial reflective (near $60^{\circ}/_{0}$), and antireflective $(<1^{\circ}/_{0})$ ones for the use in high-power Q-switched Nd : glass lasers at 1.06 µm. Our research programme for obtaining such coatings is presented in Sect. 2. Section 3 deals with the experimental method and procedure for testing the damage threshold of the coatings. In Sect. 4 the results of the tests, the performances obtained and some comments are presented. The concluding remarks are given in Sect. 5.

2. Research programme for obtaining resistant optical coatings

The task of the present study was to obtain the following main characteristics of the coatings:

- spectral characteristics within the desired range,

- damage thershold in Q-switched, 1.06 μ m wavelength, high enough for long life operation of the coatings,

- resistance to noxious mechanical and environmental conditions in order to make possible a prolonged operation in normal atmosphere and cleaning without special precautions,

- good reproducibility of all the mentioned characteristics in different batches of the coatings and different moments of testing.

In order to implement our technology to a large-scale production a special attention was paid to the last two points.

The research was realized in three steps. In the first step optical $\lambda/4$ monolayers of different materials were obtained; in order to establish their influence on the damage threshold of the layers the experimental conditions were varied. These results were used for preliminary selection of the evaporation conditions.

In the second step the three kinds of coatings (HR, PR, AR) were obtained, using the appropriate materials and conditions found in the first step. The range of the selected conditions was maintained quite large because of different optical and mechanical behaviour of the materials in the multilayer stack [2]. This situation is similar as regards the laser beam damage threshold values [3], [4]. Consequently, several interferential structures were obtained in different evaporation conditions and their damage threshold served as a merit figure for selecting the best results concerning both the structure/materials and the evaporation conditions.

In the third step the reproducibility of the characteristics of the final coatings (representing the solution) was studied. During three years, the same type of HR, PR, and AR coatings were obtained in minimum two or three different batches and tested in the same experimental setup for damage threshold measurement.

The main experimental conditions applied in the research are the following. The BK 7 optical glass (VEB Jenaer Glaswerk, GDR) was used as substrate. The samples were prepared in the form of plane plates of 25 mm diameter and 5–7 mm thick, polished on both their surfaces. The polishing procedure corresponds either to conventional optics (rms roughness of 120 Å) or to laser optics (rms roughness of about 10 Å) requirements. Roughness was measured with a Talystep instrument (Taylor Hobson, England). Before deposition the samples were cleaned with ethanol; no other type of cleaning (ultrasonic, chemical, etc.) was used.

The following oxides prepared especially for optical purposes were used as the evaporation materials: SiO_2 , Al_2O_3 , ZrO_2 , Ta_2O_5 , TiO_2 , TiO_2 , TiO. The coatings were obtained by electron beam evaporation, the optical thickness being controlled by a photometric method. In order to obtain stoichiometric compounds, the oxygen

atmosphere was controlled during evaporation. In the first step of the research the main attention was given to the following controllable evaporation conditions: the condensation rate, the partial pressure of the oxygen and the temperature of the substrate [3]. The condensation rate varied from 3 to 14 min/layer ($\lambda/4$ layers, $\lambda = 1.06 \mu$ m), the partial pressure of the oxygen was $10^{-2}-5.10^{-2}$ Pa, the temperature of the substrate: 100–300°C. In the second step several classical interferential structures were subject to experiments: (*HL*)⁸*H* for HR; *HLH*, 2*H*(*LH*)², (*HL*)²*H* for PR, $k_1 Hk_2 L$, *LLL*, *LML* for AR. For HR and PR coatings a barrier and/or an overcoating layer was used [3–6].

3. Testing equipment and procedure

The setup for damage threshold testing consists of a YAG: Nd laser with the known and long-term stability beam parameters, a focusing lens, a set of fixed callibrated attenuators located in front of the lens, a sample holder with fine xy translation stages, a fast detection system and an energy meter for monitoring the wave form and the energy of the laser beam; the associated equipment for the inspection of sample after its irradiation is also included.

The main parameters of the laser are the following:

- mode of laser operation: Q-switched, TEM₀₀, polarized linearly,
- spatial and temporal profile of the laser beam: near Gaussian,
- spot size on the sample (FW $1/e^2$): 0.1 mm,
- pulse length (FWHM): 17 ns,
- focal length of the lens: 500 mm,
- laser beam energy: 7 mJ,
- pulse repetition frequency: 2 Hz,
- peak of the maximum fluence in the test zone: 45 J/cm²,
- pulse to pulse reproducibility of the maximum fluence: $\pm 5^{0}/_{0}$.

As a rule the laser is operated at a fixed pumping energy and both the output energy and the pulse shape are monitored. The longitudinal and transversal beam profiles are measured before and after each experiment using a photoelectric method with $\sim 10 \ \mu m$ spatial resolution; during the experiment the transversal spatial profile is occasionally recorded on a black paper.

The rise time of the detection system is < 0.4 ns. The accuracy of the calibrated attenuators is not worse than $3^{0}/_{0}$. During the experiments only the attenuators are changed and the sample is moved using the translation stages.

The preparation of sample before testing comprised an identification and a cleaning procedures. In the first procedure each sample was assigned two letters and a number. For monolayers the letters indicated the type of coating and the material, while the number corresponded to the evaporation conditions. For the final solution of the coatings the letters indicated the type, while the numbers corresponded to different batches. For cleaning, a fine cloth and pure ethanol was used just before the sample irradiation. All the tests were performed in normal

laboratory conditions. The samples were located in the middle of the confocal zone behind the focusing lens, normal to the beam. The testing sites formed a line along a diameter of the sample, the distance between sites being 1 mm; the first and the last three sites were subject to 25, 1, 10 and 50 pulses, respectively, at the highest fluence, in order to have a marking on the sample. Supplementary notations on the sample edge were used to identify each end. Along the diameter the sites were irradiated from lower to higher values of the fluence. For each fixed attenuation the numbers of sites were: 1 (for the data from Tab. 1) and 5 (for the data from Figs. 1–4). The numbers of laser pulses on each site were: 50 (for the data in Tab. 1), and 20 (for the data in Figs. 1–4).

A visible spark observed during irradiation was the criterion for the definition of damage. After irradiation a visual inspection was made, using either a naked eye or a $100-400 \times$ magnification microscope or a $80 \times$ magnification phase contrast microscope.

4. Results and discussion

The main results are presented in Figs. 1–4 and Tables 1 and 2. The normalized data for the damage characteristics were based on the long-term reproducibility of the peak fluence, which was verified by using some samples as "standards" at different moments of the damage testing experiment.

In Figures 1-4 the probabilities of damage vs normalized fluence (or normalized power density) are presented. The zero and $100^{0}/_{0}$ levels correspond to the non-damage (ND) case and to the damage (D) case, respectively. The normalized damage threshold $f_{\rm th}$ is defined as

$$f_{\rm th} = \frac{1}{2}(f_{\rm ND} + f_{\rm D})$$

where f represents either the normalized fluence or the normalized power density

$$f = \frac{F}{F_{\max}} = \frac{I}{I_{\max}},$$

 F_{max} and I_{max} being expressed by the formulae:

$$F_{\text{max}} = 2E/(\pi w^2) = 45 \text{ J/cm}^2,$$

 $I_{\text{max}} = F_{\text{max}}/\tau = 2.6 \text{ GW/cm}^2$

where: E – laser energy before attenuators, w – beam waist radius (at $1/e^2$ intensity points) in the test zone, τ – laser pulse length (FWHM).

Due to cumulative errors a factor of 2 is estimated in the absolute value of the quoted peak maximum fluence F_{max} or peak maximum power density I_{max} .

Figure 1 presents the damage characteristics for monolayers of SiO_2 , ZrO_2 and TiO_2 in different experimental conditions, corresponding to the first step of the research.





Fig. 1. Damage characteristics for $\lambda/4$ monolayers (corresponding to the first step of the research): **a** - SiO₂, **b** - ZrO₂, **c** - TiO₂

For TiO₂ monolayer there occurs a strong dependence of the damage threshold on the evaporation conditions. As an example, for MT 10 $f_{th} < 10^{-2}$, while for MT 11 it is about 1.4×10^{-1} . A spread of the damage threshold is observed for samples obtained under the same evaporation condition. For example, for MZ 4 $f_{th} = 1.2 \times 10^{-1}$ and for MZ 8 $f_{th} = 1.5 \times 10^{-1}$. For SiO₂ monolayer $f_{th} \ge 0.7$.

Type of coating	Design	Materials		Peak power density for non-damage after 50 pulses
HR	S (HL) ⁸ H2L A	$H = \mathrm{TiO}_2, L$	= SiO ₂	I ₀ *
	$S 2H(LH)^2 A$	$H = \mathrm{TiO}_2, L$ $H = \mathrm{TiO}, L$	$s = SiO_2$ $s = SiO_2$	I ₀ /4 I ₀ /4
	S HLH A	$H = \mathrm{TiO}_2, L$	= SiO ₂	I ₀ /4
PR	S HLH2L A	$H = \mathrm{TiO}_2, L$ $H = \mathrm{TiO}, L$	$a = SiO_2$ $a = SiO_2$	I ₀ /4 I ₀ /4
	S 2LHLH A	$H = \mathrm{TiO}_2, L$	= SiO ₂	$I_{0}/4 - I_{0}/2$
	$S (HL)^2 H A$	$H = Ta_2O_5, L$ $H = ZrO_2, L$	$u = SiO_2$ $u = SiO_2$	I ₀ /2 I ₀ /2
	$S 2L(HL)^2 H A$	$H = \operatorname{ZrO}_2, L$	= SiO ₂	<i>I</i> ₀ *
	$S (HL)^2 H2L A$	$H = \operatorname{ZrO}_2, L$	$L = SiO_2$	I _o
	$S k_1 H k_2 L A$ $k_1 > 1$ $k_2 < 1$	$H = \mathrm{TiO}_2, L$	$L = SiO_2$	I ₀ /4
AR	S LLL A	MgF ₂ , Si	iO ₂	I ₀ /4
	S LML A	$M = Al_2O_3, L$	= SiO ₂	<i>I</i> ₀ *
	SILA	SiO ₂		2.5 I ₀

Table 1. Damage properties of the coatings tested in the second step of the research $(I_0 = 1.0 \text{ GW/cm}^2)$

* indicates the final solutions.

Table 1 presents the damage properties of the coatings studied in the second step of the research. We see that the AR coating of the type S|LML|A, which is relatively easily obtained experimentally, exhibits a high resistance in the laser beam. As to the PR coatings we note the improvement of the resistance in the laser beam when a $\lambda/2$ barrier or overcoating SiO₂ layer is used.

The damage characteristics for the final solution are presented in Figs. 2-4 for HR, PR and AR coatings, respectively.



Fig. 2. Damage characteristics for the final solution of HR coating: \mathbf{a} – obtained and measured in 1983, \mathbf{b} – obtained and measured in 1985



Fig. 3. Damage characteristics for the final solution of PR coating: \mathbf{a} – obtained and measured in 1983, \mathbf{b} – obtained and measured in 1985

For HR coating the batches HR 1, 2, 15, and 18 have the same evaporation conditions for TiO_2 as for the monolayer batch MT 10. The same is valid for ZrO_2 in the PR coating, batches PR 1, 5, 7, 12, 15, 17, 18, 23 and the monolayer batches MZ 4 and 8.

From Figures 1-3 it follows that the damage threshold for HR and PR coatings is higher than that obtained in general for the monolayer of the highest index material used in these coatings [4]. Two different damage characteristics in Fig. 4b



Fig. 4. Damage characteristics for the final solution of AR coating: \mathbf{a} – obtained and measured in 1983, \mathbf{b} – obtained and measured in 1985

may be explained by a different roughness of the substrate used. A higher damage threshold is obtained for a lower rms roughness.

Finally, as to the damage assessment, we note the following: each time a spark was observed during irradiation, a microscopic change of the surface was observed for monolayers (TiO_2 , ZrO_2), HR and PR coatings; for AR coatings microscopic damages were observed even at the fluence levels at which the spark was not observed.

In Table 2 we present the characteristics of the final solution for HR, PR and AR coatings and their damage threshold, expressed in peak power density as it is usually done in the literature.

The data for damage threshold are taken from Figs. 2-4. The spread of the

Type of coating	Reflectance at 1.06 μ m (⁰ / ₀)	Peak power density for damage threshold $I_{\rm th}$ (GW/cm ²)		
HR	≥ 99.5	1.1–1.2		
PR	60 ± 5	0.79-1.2		
AR	≤ 0.75	0.53–1.2		

Table 2. Characteristics of the final solution (third step of the research)

values for AR coatings is explained mainly by different roughness of the substrates. The spread for PR coatings could be attributed to the uncontrolled factors during the evaporation or the measurements.

5. Conclusions

Reliable, high damage thresholds for HR, PR and AR coatings were obtained when using a high-power Q-switched Nd:glass lasers at 1.06 µm. A research programme for obtaining resistant optical coatings is being developed, taking account of a large number of parameters concerning materials and their preparation, the structure of the coatings and the technological background. An experimental setup was constructed and a procedure for testing such coatings developed. Reproducible results from tests of different batches were obtained. A damage threshold of about 1 GW/cm² for a 17 ns laser pulse was obtained for all types of coatings.

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Рассуждения о реализации и тестировании оптических покрывал для лазеров высокой мощности при 1,06 µm

Представлены новейшие результаты полученные в реализации и тестировании оптических покрывал для высокой мощности лазеров из неодимового стекла. Исследованы покрывала сильно отражающие (HR), частично отражающие (PR) и антиотразительные (AR) и получен удовлетворительный порог повреждения (около 1 Gw/cm² для импульсов 17 ns). Представлены и обсуждены экспериментальные данные, принимая во внимание большое количество параметров касающихся материалов и их свойств, структуры покрывал и вида применяемой технологии.