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EM STUDY OF CeO_2 COATING OF LASER OPTICAL COMPONENTS

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The article deals with electron microscopy study of structural properties, stability, and ageing effects of CeO_2 monolayers, widely used as a coating material for laser optical components. Thermal treatment for 1^h at 550°C and 6 months of atmospheric influence has shown no effect on the structure, grain size or packing density of CeO_2 films.

1. Introduction

Dielectric films of rare earth oxide CeO_2 appear transparent in the visible and near infrared spectral region up to 12 μm and this region is potentialy covered by their application[1,2,3,4].

Among CeO_2 complex film systems, four-layer antireflexive coating combinations in the visible and near infrared consisting of $CeO_2 + MgF_2$ (for glass of refractive index 1.52)[3], are well known. Antireflexive coating combinations for typical IR material, such as Ge is for example: Si + CeO_2 + MgF_2, which increases transmitivity of Ge — plate almost twice[3]. MgF_2 appears to be a standard outer layer compatible with all other materials[1,2,3]. For this reason it is widely used for antireflective as well as for reflective coatings.

Reflective multilayers normaly consist of alternating films of high and low refractive index (such as $CeO_2 + MgF_2$) multilayer of high index contrast used for laser mirrors, beam splitters etc. Metal — dielectric complexes are useful as protective and reflectance enhancing coatings, especially on Al-mirrors. Al-CeO_2 combination is effective reflector in the visible and near ultraviolet spectral region[3].

Obviously, stability, stress resistance, and structural homogeneity of CeO_2 layers, for their application in coating of laser optical components — is of primary importance. The principal question here is dependence of optical properties (transmitivity) on the structural properties: grain size, packing density, etc.

We have undertaken the study of CeO_2 structural behaviour depending on thermal treatment, ageing effects and conditions of vacuum deposition — to elucidate this problem from the structural point of view.

2. *Experimental results*

CeO_2 high index films, in our experiments, were evaporated in vacuum 10^{-5} — 10^{-6} mm Hg, in the machine VARIAN NRC 836, with automatic deposition controller VARIAN ADS 200.

For the reason of studying CeO_2 films, layers were not evaporated in combination with other materials, but separately, in monolayers. Films were deposited on glass and NaCl substrates at normal temperature, and than exposed to atmospheric conditions during 6 months. It is sufficiently long time to see if the influence of very rough conditions on film's structure takes place.

CeO_2 monolayers of the thickness of 26 mm and 46 nm were studied by transmissional electron microscopy as well as by electron diffraction methods, after thermal treatment in the electron microscrope.

Films were heated for 30 minutes at 400 °C, and than for 1^h at 550 °C. Main morphologic and diffraction characteristics of the films, can be summarised as follows:

1. CeO_2 films deposited on glass substrate, according to electron diffractions were found amorphous — on diffractograms, besides central diffussive halls some very slight ring corresponding to interplane distances have been found. Layers have been found continuous without contrast differences, for both film thicknesses. (26 nm and 46 nm).

2. CeO_2 films deposited on NaCl substrate (transparent in IR), were found to be cubic crystalline structure, with the grain size between 12 — 15 nm in both films. Interplane distances are in good agreement with ASME tables (see TABLE I.). There is, however, a slight difference in preferentional orientation of the layers of 26 nm in comparison with the layers of 46 nm. Thinner layers exhibit preferentional orientation (001) CeO_2/(001) NaCl and (100) CeO_2/(100) or/(110) NaCl. This is, obviously, effect of the influence of substrate crystalline structure, whose effect decreases as the films thickness increases.

3. Thermal treatment in electron microscope at very high temperatures (550°C) practically has no any effect on morphology and diffraction pattern of CeO_2 films, which is a direct evidence of stability of CeO_2 layers. For illustration see Fig. 1 (a, b) Fig. 5 (a, b). No changes of crystalline structure, grain size, or packing dens-

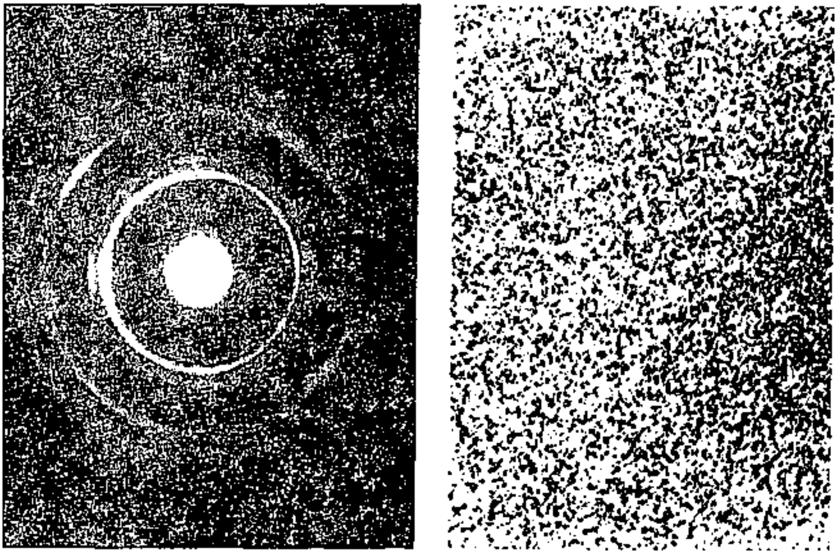

Fig. 1(a,b) : CeO_2 — 26.8 nm/NaCl, 86000 × magnification

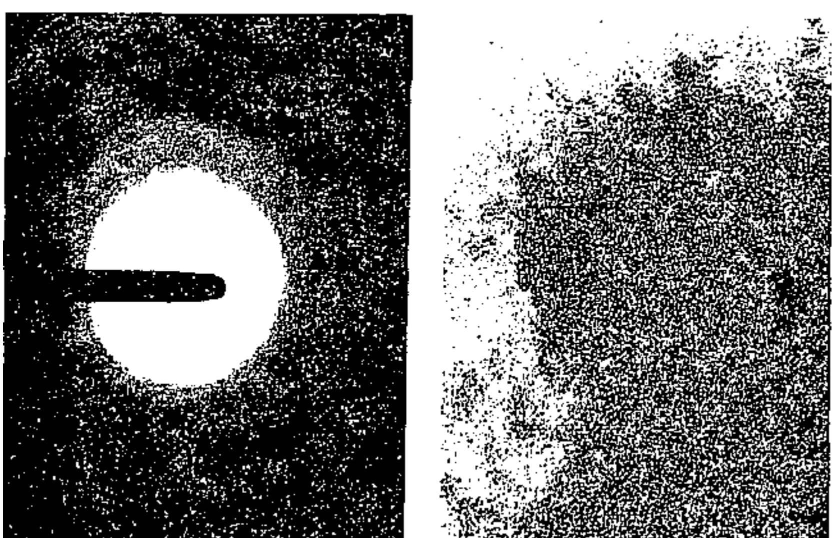

Fig. 2(a,b): CeO_2 — 46.6 nm/glass, 86000 × magnification

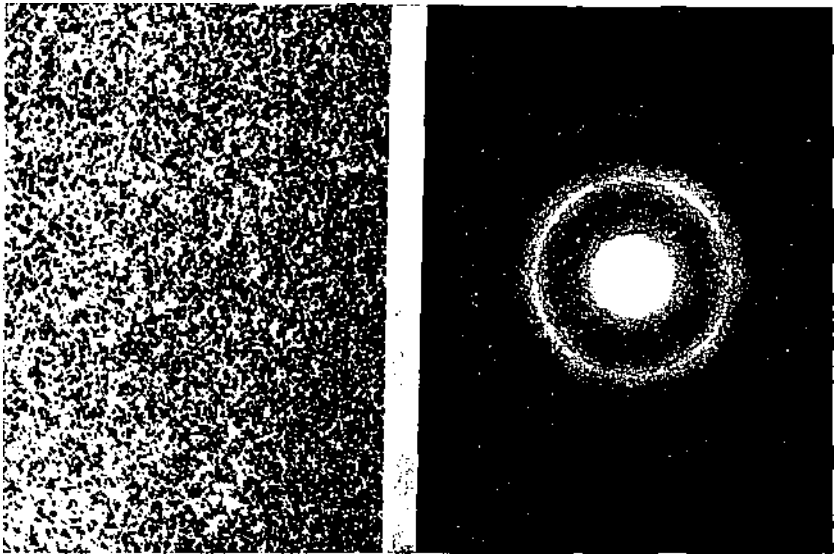

Fig. 3(a,b):CeO_2—46.6 nm/NaCl, 86000 × magnification

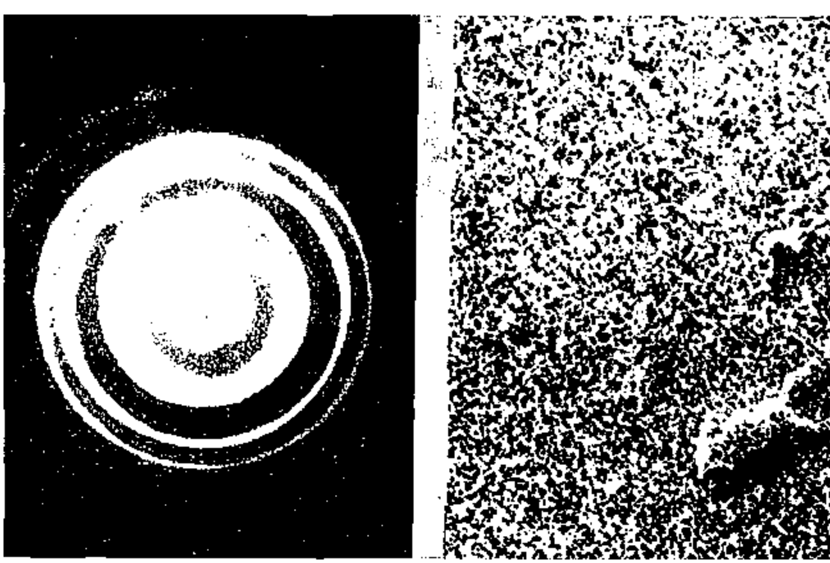

Fig. 4 (a,b):CeO_2—46.6 nm/NaCl, 30 minutes on 550°C, 86000 × magnification

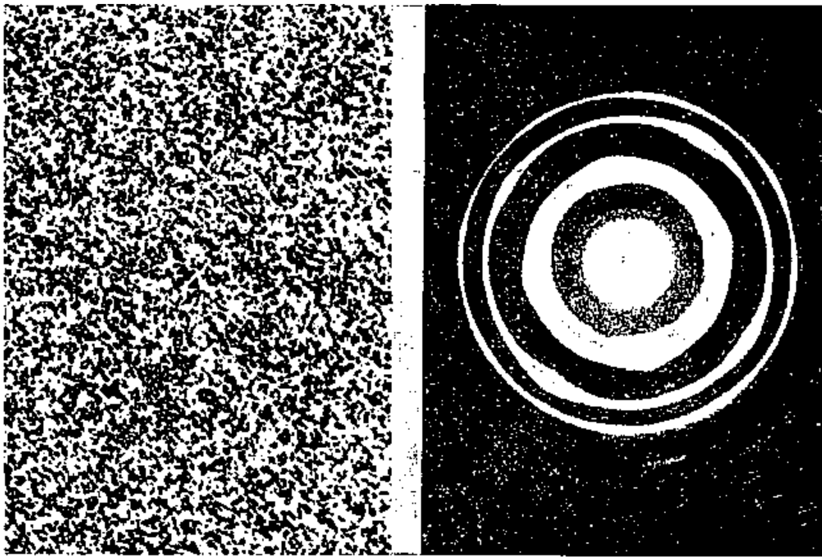

Fig. 5(a,b): CeO_2—26.8 nm/NaCl, 30 minutes on 550°C, 86000 × magnification

TABLE 1.

Experimental		ASTM	
d (nm)	I_{rel}	d (nm)	hkl
0.312	very strong	0.3142	(111)
0.271	strong	0.2706	(200)
0.1922	strong	0.1913	(220)
0.1634	strong	0.1632	(311)
0.1558	slight	0.1562	(222)
0.1344	slight	0.1353	(400)
0.1245	slight	0.1241	(331)
0.1208	very slight	0.1220	(420)
0.1104	slight	0.11044	(422)
0.11041	slight	0.10412	(511)

Interplane distance for CeO_2

sity under thermal treatment has been observed, which is in agreement with previously reported observations[1,2,3]. All the effects of residual stress, for CeO_2 films in comparison with ZnS, cryolite and even SiO_2 dielectric films, treated in the same way[5,6] — are almost negligible. Strictly speaking, ZnS films, for example, were completely destroyed after submition to the same treatment.

A comparative study of transmission (optical) and structural properties of CeO_2 films is now under progress.

3. Conclusion

Although our study presents only preliminary results, it can be said that CeO_2 coatings of laser optical components fulfill all important criteria: transparence, refractive index, homogeneity, hardness and stress resistance, as well as stability on atmospheric influences and ageing effects.

It is possible that other rare earth oxides, less investigated than CeO_2 are as good or even better coating materials than CeO_2, and represent very promissing field of investigation.

Acknowledgment

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ISPITIVANJE TANKIH SLOJEVA CeO_2 POMOĆU ELEKTRONSKOG MIKROSKOPA

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U članku su opisana ispitivanja tankih slojeva CeO_2 pomoću elektronskog mikroskopa, da bi se ustanovili utjecaji termalnog tretiranja i efekata starenja. Filmovi su bili podvrgnuti atmosferskom utjecaju kroz 6 mjeseci, a zatim termički tretirani u elektronskom mikroskopu na 400°C kroz 30 min., plus I^h na 550°C. Nikakve strukturne promjene, promjene veličine zrna ili gustoće pakovanja nisu primjećene.