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R.F.SPUTTERED OPTICAL THIN FILMS
Vabljeno predavanje, SD-88, Nova Gorica

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Key Words: Optical Thin Films, Dielectric Thin Films, Thin Film Deposition, RF Sputtering

ABSTRACT: After a general review of the technology, three applications will be presented:

1. Co-sputtering of optical thin films
2. Sputtering of oxides for I.R. applications
3. Thickness trimming of optical films by sputtering

We have investigated the Co-sputtering of two dielectric materials with refractive indeces as widely different as possible with the aim of obtaining both homogeneous films with any intermediate refractive index and inhomogeneous films with predetermined profiles.

The results obtained by co-sputtering CeO_2-SiO_2 and TiO_2-SiO_2 are presented. The availability of sputtered films of SiO_2, TiO_2,CeO_2 and Y_2O_3 for I.R. applications, allows to get coatings more resistant and more stable to the environmental stresses. The results obtained are presented together with the characterizations of films with special care to the refractive index, absorption and laser damage thresholds under cw and pulsed laser radiaton (1.06 μm and 10.6 μm). Finally, an original process allowing the thickness adjustement of any optical film (either by trimming or thickening) by the sputtering technique is presented. The results obtained with this process are discussed.

R.F. NAPRŠEVANJE OPTIČNIH TANKIH PLASTI

Ključne besede: optične tanke plasti dielektrične tanke plasti, nanašanje tankih plasti, RF naprševanje

POVZETEK: Po splošnem pregledu tehnologije bomo predstavili tri konkretne uporabe le-te:

1. Sočasno naprševanje optičnih tankih plasti
2. Naprševanje oksidov za uporabo v IR območju
3. Spreminjanje debeline optičnih filmov z naprševanjem in odprševanjem

Proučevali smo sočasno naprševanje dveh dielektričnih materialov s kolikor se le da različnima lomnima količnikoma. Namen je bil izdelati, bodisi homogene filme s katerokoli vmesno vrednostjo lomnega količnika, bodisi nehomogene filme z vnaprej določenim profilom lomnega količnika.

Predstavljamo rezultate dosežene, s sočasnim naprševanjem CeO_2- SiO_2 in TiO_2 materialov. Možnost uporabe napršenih filmov SiO_2, TiO_2, CeO_2 in Y_2O_3 v IR območju omogoča izdelavo pokritij, ki so bolj stabilna in odporna na vplive okolja. Predstavljamo dosežene rezultate hkrati s karakterizacijo filmov; le-ta obsega meritve lomnega količnika, absorpcije in praga poškodb pri obsevanju s CW in pulsno lasersko svetlobo (1.06 μm in 10.6 μm). Na koncu predstavljamo izvirno tehniko nastavitve debeline katerekoli optične plasti (bodisi tanjšanje oz. debelenje) z naprševanjem. Komentiramo rezultate, dosežene s takim postopkom.

1. INTRODUCTION

The increasing demand in the field of thin film optical treatments, which has developed during last few years, has made necessary the need to realize structures with ever increasing complexity and with high level of performance both from the optical point of view as well as resistance to the environmental conditions and use. The use of sputtering technologies, in the R.F. diode as well as R.F. magnetron configurations, for the realization of optical films, has enabled in these last few years to widen the spectrum of materials which can be easily used (with particular regards to refractory oxides) and to obtain films which are more compact and adherent. This paper describes three new applicatios of sputtering to the realization technologies of optical thin films.

(1) The realization of optical films with predetermined refractive index with continuity between the extreme values of 1.45 and 2.4 by means of co-sputtering.

(2) The use of sputtered oxide films having a high refractive index in mid-I.R. (5-12 μm)

(3) Trimming the thicknesses of the optical films by means of sputteretch and sputter deposition in cases where the film thickness was different to the projected value.

2. EXPERIMENTAL

This work has been carried out on a sputtering system equipped with three targets 8" in diameter, one of which is of the magnetron type, and a 3 KW (13.5 Mhz) R.F. generator. The water cooled substrate holder consists of a circular rotating (0-10 turns/minute) electrode beneath the targets and acts as an anode during the deposition, with possibility of etch-sputtering and bias-sputtering. The matching network enables simultaneous sputtering from two or three targets by dividing the power received from the generator. The system is also supplied with a radiant heater for heating the substrates. The vacuum system consists of 500 l/sec turbo-molecular pump with the necessary backing rotary pump. The dynamic control of the optical thickness of the deposited films is carried out by a photometer.

This device enables two types of measurements, the first approach enables observation of the reflectance variations directly on the sample in motion during the film growth at a wavelength which is suitably chosen, and the second in which the deposition cycle is interrupted, but not the vacuum cycle, the reflectance is mesured at different wavelengths on the stationary sample.

3. CO - SPUTTERED OPTICAL FILMS

One of the problems most commonly encountered in the design of new optical structures resides in the limited choise of refractive indices available, especially when the mechanical characteristics and resistance to treatment by laser radiation are particularly stringent. In the past this limitation was overcome by attempting to realize optical film obtained by mixing two or more materials by means of co-evaporation technique with thermal sources.

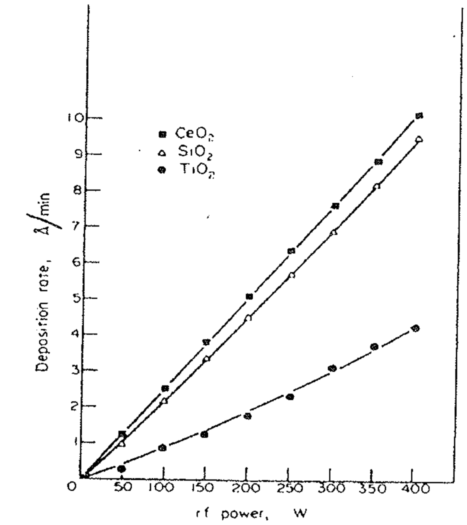

Figure 1: Sputtering rate as a function of the rf power at the cathode for TiO_2, CeO_2 and SiO_2

This method however requires a complex apparatus for the control of the evaporation rates of the individual sources and does not offer sufficient reproducibility nor can it be adapted to industrial processes. In the solution proposed by us the substrate to be treated is placed on a circular rotating anode, beneath two targets which emit simultaneously. Actually it is not strictly a proper co-deposition since the substrates are alternatively exposed to the two sources. Since the thickness of the single layer is of the order of Å, the mixture abtained acts as perfectly homogeneous (ref.3). The pairs of materials experimented by us are CeO_2+SiO_2 and TiO_2+SiO_2. As the sputtering atmosphere mixtures of Ar and O_2 have been used as well as pure Ar, at a total pressure equal to 5 to $10x10^{-3}$ torr. The deposition rate of the single material was simply adjusted by controlling the power supplied to the targets and the related selfpolarization voltages.

As an example we show in fig 1 the calibration curves for TiO_2, CeO_2 and SiO_2.

Homogeneous films

In the case of homogeneous films with predetermined refractive index, our attention was focused on the repeatability attainable under realistic working conditions. We felt this was the real test for the usefulness of the proposed technology. Because of the dispersion of all materials in question (particularly TiO_2 and CeO_2) the refractive index must be determined always at the same wavelength λ, in our case generally 5500 Å. Films with a targeted optical thickness equal to $\lambda/4$ were deposited on Corning microsheets 0211, BK7 and sapphire. Both refractive index and actual optical thickness were determined by measuring the transmittance of the samples before and after deposition as a function of the wavelength.

Shown in Figure 2a and b is the refractive index of the mixtures SiO_2-TiO_2 and SiO_2-CeO_2 as a function of the volume percentage of SiO_2. Marked with the shaded area is the scattering of results obtained for the refractive index. It is apparent from Figure 2 that the scattering of values is higher for mixtures SiO_2-CeO_2 than for mixtures SiO_2-TiO_2. This is likely to be due to the strong dependence of the refractive index of CeO_2 films on the substrate temperature. Although thickness monitors were actually used in this experiment, it was found that the same reproducibility could be obtained for the refractive

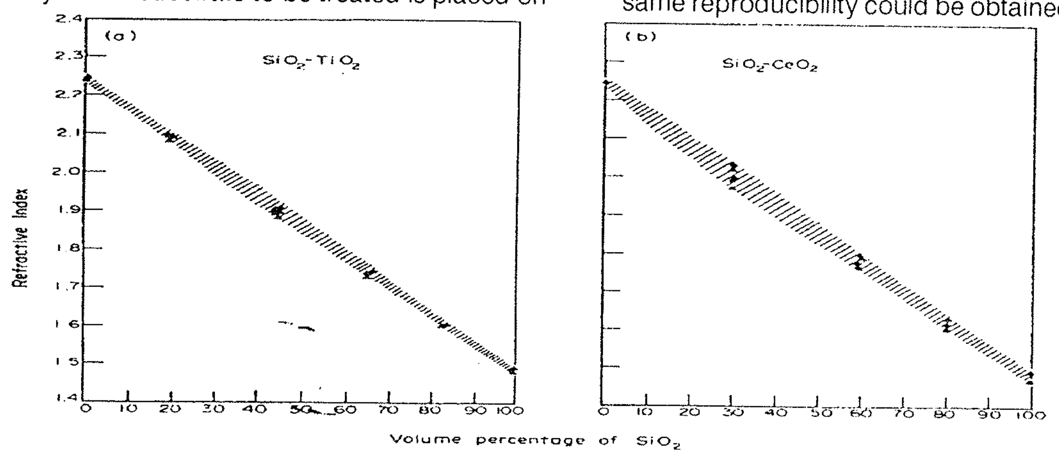

Figure 2: Refractive index of co-sputtered films as a function of the volume percentage of SiO_2: (a)SiO_2-TiO_2; (b) SiO_2-CeO_2. The shaded area points to the range of values obtained.

Table 1: **Co-sputtered optical films**

Couple	% of SiO_2	SiO_2 power W	H index power W	Total rate Å/min	$\bar{n}$ average	n_{MIN} - n_{MAX}	
	0	0	400	4.1	2.24	2.23-2.24	Ultimate pressure $<10^{-6}$ torr. Sputtering atmosphere 6mtorr of pure Ar
	30	85	400	5.8	2.00	1.99-2.00	
SiO_2-TiO_2	50	175	400	8	1.87	1.86-1.88	
	66	360	400	12.5	1.75	1.74-1.76	
	85	400	175	11.2	1.62	1.61-1.62	
	100	400	0	9.5	1.49	-	
	0	0	260	6.6	2.23	2.21-2.24	
SiO_2-CeO_2	30	130	260	9.4	2.00	1.97-2.02	
	60	400	260	16	1.82	1.79-1.83	
	80	400	85	12	1.65	1.63-1.67	
	100	400	0	9.5	1.49	-	

index by controlling time and rf power at the cathode, provided all other parameters were kept constant. However, in this case, reaching a good vacuum ($<10^{-6}$) before deposition and keeping the discharge atmosphere free from reactive gases is especially essential because even small percentages of such gases (particularly O_2) could affect in an appreciable way the deposition rate of all materials in question. The conditions of our experiments are summarized in Table 1 which gives also the maximum dispersion obtained for the refractive index.

Inhomogeneous films

In the case of inhomogeneous films, because of difficulties involved in determining the refractive index profile on the deposited film, we chose to start from the simplest case of a linear variation. Starting from the refractive index of the substrate (1.53) the refractive index of the film was made to change linearly up to a given higher value with a total thickness of the film much higher than the wavelength of interest.

The apparent refractive index of the substrate was thereby modified. We made use of the experience gained with homogeneous films to determine the values to be used for the process parameters. Displayed in Figure 3 is the variation with time of the power delivered to the SiO_2 and CeO_2, cathodes together with a list of the values used for the main parameters. The total physical thickness of the film was 2μm. In Figure 4 we have reported the profile targeted for the refractive index, ranging from 1.53 to 2.24. Marked with dotted lines are the reflectivity at normal incidence for a bulk material with refractive index equal to 2.24 and the reflectivity of the untreated substrate. While the apparent refractive index of the treated sample is clearly changing, oscilations due to interface effects are still clearly visible. We feel these could be reduced by modifying the profile as follows: the corners at the two ends of the sloping profile should be smoothened, the refractive index should be kept at its high value for an appreciable thickness (of the order of 1 μm) and the total thickness of the film should be increased.

With this technique we have performed, in the research as well as small scale production stage, numerous types of optical treatments. All the co-sputtered films have passed the environmental tests related to the standards MIL 13508B and MIL 675A for adhesion, abrasion, thermal cycles, salt sprays and humidity cycles. As an example we report in figure 5 the design layout of an Ar-W treatment (large band anti-reflectant in the visible and at 1.06μm).

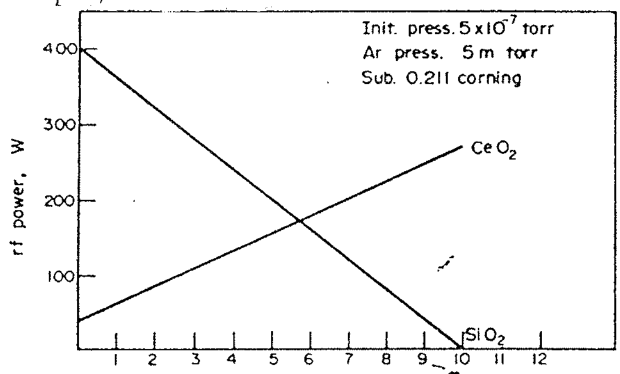

Figure 3: **Time dependence of the power delivered to SiO_2 and CeO_2 cathodes during the deposition of an inhomogeneous film.**

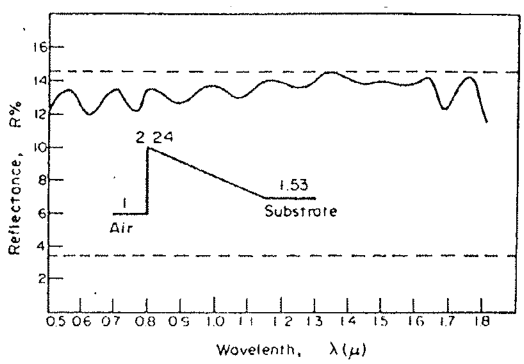

Figure 4: **Reflectivity of an inhomogeneous film. The upper dotted line marks the reflectivity of a bulk material with refractive index equal to 2.24. The lower one the reflectivity of the uncoated substrate.**

4. SPUTTERED OXIDES FOR THE MID I.R. (5-12μm)

The use of oxides for the realization of optical films in the visible or near infrared deposited by means of thermal sources, generally electron-gun, has produced several years ago a net improvement in mechanical characteristics and environmental resistance of the film. The difficulty in obtaining completely oxidised films, even with reactive depositions, gives rise to absorbing layers with low laser damage threshold. The use of sputtering

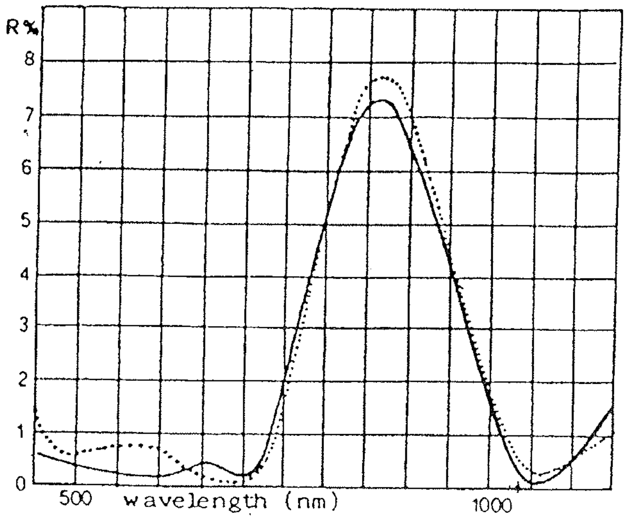

Figure 5: **Optical filter (AR 500-700 nm+AR 1.06μm) with cosputtered films.**
DESIGN INDEX (1060 nm)
Substrate 1.6
1 ° film 1.71 cosputtered
2 ° film 1.45
3 ° film 1.751 cosputtered
4 ° film 1.954 cosputtered
5 ° film 1.45
AIR

and the refining of thermal deposition techniques, in the last few years have clearly improved the optical characteristics of the oxide films. Our aims has therefore been that of optimizing and characterizing the films of sputtered oxides for uncommon application in the mid-I.R., aiming above all for the application at 10.6 μm (wavelength of CO_2 lasers). The materials taken into consideration are CeO_2, TiO_2 and Y_2O_3. In the table 2 all the optimal deposition parameters for the films, which have been deposited on Germanium substrates with an optical thickness $\lambda/4$ of 10.6 μm are reported.

FILM DEPOSITION PARAMETERS				
Power density	6.7	3.2	3.9	W/CM2
Polarization voltage	1100	700	850	V
Residual pressure	1	1	1	10^{-5} torr
Ar partial pressure	7	7	7	10^{-3} torr
O2 partial pressure	0	5	20	10^{-4} torr
	Y2O3	CeO2	TiO2	

The films realized have been tested for the laser damage threshold at 10.6 μm (pulsed and cw) and at 1.06 μm (pulsed). In figures 6, 7 and 8 the values obtained are reported. From an observation of the absorption coefficient and damage threshold values, it is possible to obtain the usefullness of these oxides up to 10.6 μm. In fact while the damage thresholds are comparable with the materials which are traditionally used at 10μm these latter are decisively more unstable, softer and less adherent than the oxides deposited by us.

For the co-sputtered films SiO_2+CeO_2 it has been valued, on a structure of thickness $\lambda/4$ on a BK7 glass substrate, the laser damage threshold for 1.06 μm laser impulses of 30 nsec duration. In figure 9 this behaviour is reported as a function of the refractive index of the film (SiO_2 damage threshold is up to 3 GW/cm^2)

5.TRIMMING THE THICKNESS OF THE OPTICAL FILMS

Since the dynamic control of the thickness of optical films often proves to be a problem, especially in those cases in which a high degree of accuracy is required, according to the present most advanced techniques one makes use of, after a precise measurement, a reoptimization of the realization design on the basis of the characteristics of the films already deposited. The method proposed by us enable us to overcome this difficulty by carrying out adjustements either by addition (sputter-deposition) of the thickness of the deposited film even after it has been exposed to the atmosphere, or by subtraction (etch-sputtering). While the eventual addition of material is also possible by the thermal evaporation techniques, trimming by thinning is carried out only by means of sputtering. This thinning is carried out by inverting the polarity in the system in such a way that the substrate holder becomes the cathode of the structure. This procedure is carried out with a very low power density and in this way the thinning is found to be extremely controllable. In this stage much care is needed to ensure that foreign sputtered material does not deposit itself by direct redeposition or back- scattering in to the surface of the substrate causing contamination which is capable of drastically reducing the damage threshold of the treatment. The uniformity in thinning is generally more than necessary since in the worst we are dealing with reduction of the order of 10% of the total film thickness, for which a 10% disuniformity would result in a maximum of 1% deviation from the projected value. However in the case of a total removal of a very thick or multi-layered film, in order not to change the planarity of the substrate, a much more elevated erosion uniformity is necessary which is easily attainable on small surfaces but for large surfaces movement of the sample is required. Very interesting results in this field have been obtained in a study stage with Ion-Beam etching.

The damage threshold by laser irradiation of structures adjusted by ion beam etching proves to be the same as that of un-trimmed films. This technique applied to production cycles of optical components for laser application has anabled us to rise the yield, as far as optical specifications are concerned, from 70% to values better than 95%.

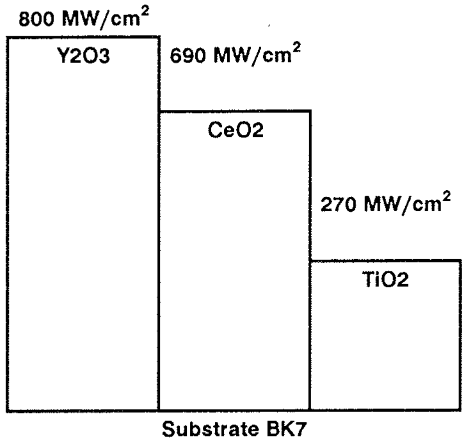

Figure 6: Laser damage treshold (1.06 μm), pulse length 30 nsec

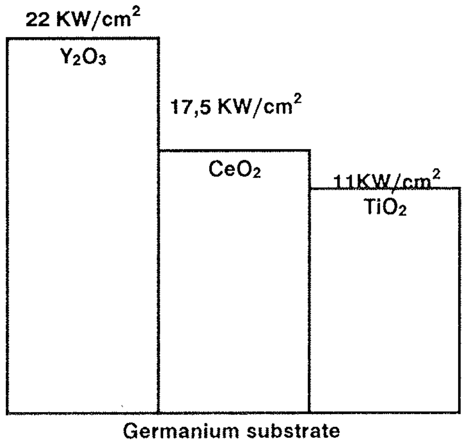

Figure 7: Laser damage treshold (CO_2 cw)

6. CONCLUSION

The co-sputtering system for optical films proposed by us enables the ralization of homogeneus films with predetermined refractive index between the two starting materials, with a reproducibility of ± 0.01 with ideal mechanical characteristics and high laser damage threshold. This enables us to greatly simplify the design stage and to obtain better optical response with smaller number of films. Furthermore we have demonstrated the usefullness of hard oxides (CeO_2, TiO_2, Y_2O_3) in the mid I.R. with good laser damage threshold at 10.6 μm and very good at 1.06 μm and excellent mechanical characgeristics. The trimming of the film thickness by means of sputtering also allows an easy realization of very critical components and a net increase of the production yields.

In conclusion the results achieved confirm sputtering as an extremely ductile and reliable technique for the realization of optical films for laser and sophisticated optics.

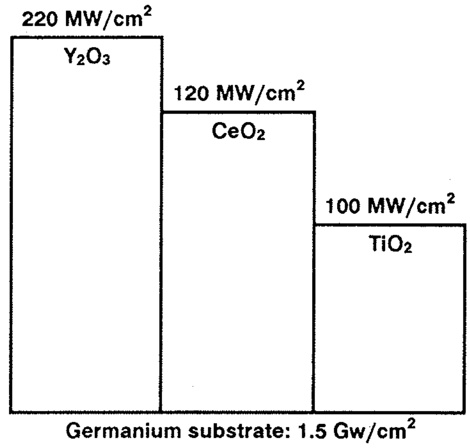

Figure 8: Laser damage treshold (TEA)
LASER PARAMETER
Wavelength 10.6 μm
Peak power 2 MW
Pulse length 75 nsec
Frequency 50 pps
Exposition time 5 sec

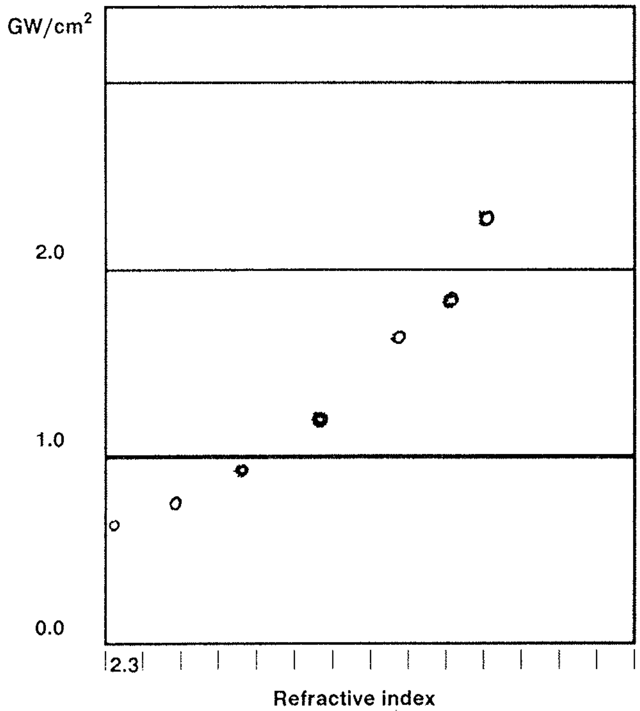

Figure 9: Laser damage treshold of cosputtered film

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