AR COATING WITH HIGH DAMAGE THRESHOLD
ON SiO_2 GLASS

C. Kanamori
S. Nakajima
H. Toratani
K. Nakagawa
T. Izumitani

Hoya Optics, Inc.
3400 Edison Way, Fremont, CA 94538

MASTER

September 12, 1983

Lawrence
Livermore
National
Laboratory

NOTICE

PORTIONS OF THIS REPORT ARE ILLEGIBLE.

It has been reproduced from the best available copy to permit the broadest possible availability.

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

NOTICE

Work performed under the auspices of the U.S. Department of Energy by the Lawrence Livermore Laboratory under contract number W-7405-ENG-48.

This document was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, expressed or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise, does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

HOYA OPTICS, Inc.

UCRL 15583

3400 Edison Way, Fremont, California 94538 PHONE (415) 490-1880 TELEX 172-647

AR COATING WITH HIGH DAMAGE THRESHOLD
ON SiO₂ GLASS

Final report

LINL PO# 2531205

Sept. 24, 1983

Principal investigators

C. Kanamori
S. Nakajima
H. Toratani
K. Nakagawa
T. Izumitani

Report prepared by :

H. Toratani

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

HOYA OPTICS, Inc.

3400 Edison Way, Fremont, California 94538 PHONE (415) 490-1880 TELEX 172-647

INTRODUCTION

In the previous investigation, low reflective and almost uniform coating have been prepared on silica glass substrates. However, the laser damage threshold of the coating was rather low. It is suggested that the low threshold is attributed to the carbon residue in the film and/or the contamination on the substrate surface. After the last report, we have evaluated the uniformity of AR treated films on silica glass substrates and also examined the several methods which were expected to increase the damage threshold.

In this report, the following experimental results are shown.

- 1) Uniformity of AR coatings on Optosil and Sprasil provided by LLNL.
- 2) Relation between cleaning methods and properties of AR coatings.
- 3) Baking and sintering conditions of gel-derived films.
- 4) Heat treatment of AR coating in O_2 flow.

EXPERIMENTAL RESULTS

Uniformity of AR coating

Wave front distortion of LLNL silica glass substrates, sintered sol-gel coatings and AR treated coatings were measured by interferometric procedure. Analysis of fringe pattern by ZAPP were given in Fig.1, 2 and 3. Reflective wave front distortion of substrates increased by $\lambda/8 \sim \lambda/170$ after sintering and also increased by $\lambda/13 \sim \lambda/125$ after AR treatment(leaching). It is concluded that the uniformity of AR coating except the edge of substrate is better than $\lambda/5.5$ ($\lambda=6238\text{\AA}$), at least.

Relation between cleaning and AR coating

For the purpose of removing the contamination, the various cleaning procedures of substrates were examined. However, it was observed that the optical properties of AR coating strongly depend on the cleaning procedure of substrate. This suggests that the surface states of substrates affect the properties (film structure, porosity, etc.) of coated films.

The reflective spectra of AR coating to which the following cleaning procedures were applied were classified into two types, as shown in Fig.4.

- [1] Detergent - H_2O - H_2O - H_2O - IPA - IPA - IPA - Vapor drying
- [2] RBS 25 - RBS 25 - H_2O $\rightarrow$ continue to procedure [1]
- [3] HF - H_2O $\rightarrow$ continue to procedure [1]
- [4] $\text{NH}_4\text{F} \cdot \text{HF} + \text{HNO}_3$ - H_2O $\rightarrow$ continue to procedure [1]

Reflective spectra of type A in Fig.4 were observed for the AR coating on the substrates cleaned by the procedure [1]. That of the type B were observed

HOYA OPTICS, Inc.

3400 Edison Way, Fremont, California 94538 PHONE (415) 490-1880 TELEX 172-647

in the case of the procedures [2], [3] and [4]. These facts were independent of the leaching time for AR treatment. Each type of reflectivity spectra was simulated by the two layers graded index film model (for A) and single layer graded index film model (for B) as shown in Fig.5. We have not enough data to explain the reason why such difference in the reflectivity arise, at present. However, the following result may give one usefull suggestion, that is the reflective spectra of type B were often observed in the case of long sintering time and higher sintering temperature compared with that of normal condition.

Baking and sintering condition

It was suggested by LLNL that the low damage threshold of our previos samples may be due to the residual carbon in the film. In order to confirm this possibility, we have prepared thr coated films which were sintered in O_2 gas and air flow at 350 - 370°C for 5 - 10hrs. The optical properties of AR coatings prepared from these coated films were not so different from that by normal procedure. The effect of O_2 gas flow in sintering will be clarified by the damage threshold measurement in LLNL.

Heat treatment of AR coating

AR films obtained by leaching were re-heated in O_2 gas and air flow in order to burn out the residual carbon. As the result, the film decreased in thickness and the reflectivity slightly increased but was kept less than 0.5 %. The effect of this treatment on the damage threshold will be also clarified by the LLNL damage experiment.

HOYA OPTICS, Inc.

3400 Edison Way, Fremont, California 94538 PHONE (415) 490-1880 TELEX 172-647

SUMMARY AND FUTURE PLAN

Table 1 and 2 show the preparation conditions and reflective properties of AR coatings. The effect of the cleaning of substrates, O_2 gas flow in sintering and re-heating of AR films in O_2 gas flow on the improvement of damage threshold must be clarified by the damage measurement in LLNL.

The technique of the low reflective and uniform coating on silica glass substrate has been developed. However, with respect to the reproducibility of AR film having the same optical properties, there are several problems, that is, the aging condition of coating solution, the sintering condition and the leaching condition. We should make the experiment for improving the damage threshold and the reproducibility of AR coating.

Supplement

We will send the damage test samples of 14 pieces which are listed in Table 1 and 2.

Fig. 1 Reflective wave front distortion of AR coating on Optosil (# 3632)

Fig.2 Reflective wave front distortion of AR coating on sprsil (# 3588)

2/5/3

Sprasil

3588

2/5/3

2/17/0

Optosil

3632

2/19/4

Fig. 3

Transmittive wave front distortion of AR coated substrates

Fig.4 Two types of reflective spectra of AR coating

Fig.5 Simulation of AR coating by graded index layer model

TABLE 1 LIST OF AR-COATED SAMPLES (substrates HOYA prepared)

Sample No.	Cleaning	Solution age(days)	Sintering	AR treatment	Sintering after leaching	Reflectivity		
						$R_{\min}$	$\lambda_{\min}$	$R_{3\omega}$
9061	neutral solution	12	350°C, 7h	85°C, 6h	350°C, 7h, air	0.42%	335nm	0.42%
9063	"	"	"	85, 16	350, 5, O ₂	0.16	330	0.21
9144	"	20	350, 12	85, 8	none	$R \approx 0.7\%$ (330< λ <800)		
9147	"	"	350, 7, air	85, 6	330, 10, O ₂	0.46%	365nm	0.49%
9181	"	11	"	85, 8	none	0.21	350	0.21
9182	"	"	350, 5, O ₂	85, 8	330, 10, O ₂	0.47	370	0.56
9183	"	"	350, 7, O ₂	85, 10	none	0.10	370	0.21
9188	HF	"	350, 5, O ₂	85, 8	"	0.33	340	0.33
9203	neutral solution	13	350, 10, O ₂	85, 12	"	0.54	360	0.54

TABLE 2 LIST OF AR-COATED SAMPLES (substrates LLNL provided)

Sample No.	Cleaning	Solution age(days)	Sintering	AR treatment	Sintering after leaching	Reflectivity		
						$R_{\min}$	$\lambda_{\min}$	$R_{3\omega}$
3632	neutral solution	12	350°C , 7h	85°C , 20h	none	$\leq 0.42\%$	$\leq 330\text{nm}$	0.43%
3588	"	"	"	85°C , 11h	350°C , 3h, O ₂	0.45%	330nm	0.48%
3640	"	20	350°C , 12h	85°C , 4h	none	0.67%	630nm	0.74%
3589	"	13	370°C , 5h, O ₂	85°C , 12h	none	0.65%	500nm	0.91%
3591	"	13	350°C, 10h, O ₂	85°C , 36h	350°C , 3h, O ₂	0.62%	420nm	0.75%