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REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION: Unclassified	1b. RESTRICTIVE MARKINGS
2a. SECURITY CLASSIFICATION AUTHORITY	3. DISTRIBUTION/AVAILABILITY OF REPORT: Approved for public release; distribution unlimited.
AD-A223 053	5. MONITORING ORGANIZATION REPORT NUMBER(S): ARO 23794.1-MS-F
6a. NAME OF PERFORMING ORGANIZATION: SUNY/Albany; 6b. OFFICE SYMBOL (If applicable): Physics	7a. NAME OF MONITORING ORGANIZATION: U. S. Army Research Office
6c. ADDRESS (City, State, and ZIP Code): 1400 Washington Ave, Albany, N.Y. 12222	7b. ADDRESS (City, State, and ZIP Code): P. O. Box 12211, Research Triangle Park, NC 27709-2211
8a. NAME OF FUNDING/SPONSORING ORGANIZATION: U. S. Army Research Office; 8b. OFFICE SYMBOL (If applicable):	9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER: DAAL03-86-G-0058
8c. ADDRESS (City, State, and ZIP Code): P. O. Box 12211, Research Triangle Park, NC 27709-2211	10. SOURCE OF FUNDING NUMBERS: PROGRAM ELEMENT NO. / PROJECT NO. / TASK NO. / WORK UNIT ACCESSION NO.

11. TITLE (Include Security Classification): Army Science and Technology Fellowship: Physics of Materials

12. PERSONAL AUTHOR(S): Lanford, William A.

13a. TYPE OF REPORT	13b. TIME COVERED	14. DATE OF REPORT (Year, Month, Day)	15. PAGE COUNT
Final	FROM 1/7/86 TO 1/31/90	May 31, 1990	4

16. SUPPLEMENTARY NOTATION: The view, opinions and/or findings contained in this report are those of the author(s) and should not be construed as an official Department of the Army position, policy, or decision, unless so designated by other documentation.

17. COSATI CODES		
FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS (Continue on reverse if necessary and identify by block number): Thin film, protective coatings, glasses

19. ABSTRACT (Continue on reverse if necessary and identify by block number)

This is an Army Science and Technology Fellowship on the physics of materials. A computer controlled plasma assisted CVD chamber was designed and built. This system was then used to fabricate thin film coatings on different glasses, including ZBL glass (which transmits in the infared) and on models for historic stained glasses. Studies of the mechanism of the reaction between water and historic stained glass indicate that the reaction mechanism may be different from what had been expected.

DTIC ELECTED

20. DISTRIBUTION/AVAILABILITY OF ABSTRACT: ☐ UNCLASSIFIED/UNLIMITED ☐ SAME AS RPT. ☐ DTIC USERS	21. ABSTRACT SECURITY CLASSIFICATION: Unclassified
22a. NAME OF RESPONSIBLE INDIVIDUAL	22b. TELEPHONE (Include Area Code) / 22c. OFFICE SYMBOL

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete.

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ARMY SCIENCE AND TECHNOLOGY FELLOWSHIP: PHYSICS OF MATERIALS

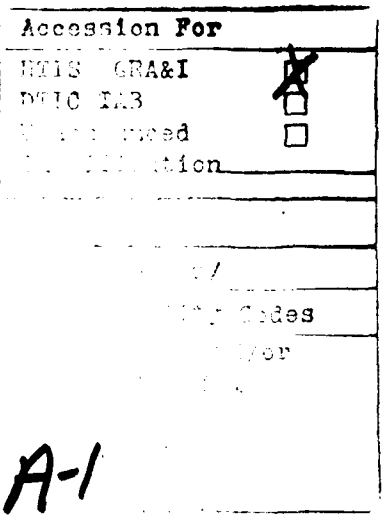
FINAL REPORT

WILLIAM A. LANFORD

MAY 30, 1990

U.S. ARMY RESEARCH OFFICE

CONTRACT NUMBER: DAA03-86-G-0058



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OVERVIEW

This project is an ARO Fellowship. Training of graduate students in Materials Physics was a primary goal. This was to be accomplished by having the Fellow work on the design, the building and the de-bugging of a computer controlled plasma assisted CVD chamber. The central idea behind the particular project was to make use of logically controlled thinfilm deposition system to fabricate thinfilms consisting of many sub-layers. These multilayer thinfilms could then be used both to study the transients in the deposition process and to make composite thinfilms for coatings (diffusion barriers, durable coatings, etc.).

After building this system, studies of the properties of some thinfilm coatings on glass have been undertaken. This has led the research project into the study of the reaction between water and glass. Data from this work indicates that the reaction mechanism may be very different from what had been expected based on studies of glasses of other compositions.

PLASMA ASSISTED CVD CHAMBER

This chamber is a rather standard capacitively coupled RF plasma assisted CVD chamber. However, this chamber is under logical control and monitoring. For example, the computer controls the gas flow, the substrate temperature, the RF power (on or off) and the deposition time. By controlling the deposition conditions, this chamber is capable of fabricating thinfilms consisting of many different (CVD) sublayers.

The design and building of this chamber took the majority of time of the ARO Fellow under this program. The equipment need to build this chamber was purchased under a differ (but related) research contract funded by IBM Corporation.

The first films deposited in this chamber were hydrogenated amorphous carbon films which were studied as possible protective coatings for the new long wavelength transmitting ZBL glass or for historic stained glasses.

HALIDE GLASSES

About 10 years ago, a new glass forming family of materials was discovered. A typical member of this family is a mixture of Zr, Ba, and La fluorides (ZBL glass). While there are many composition variations, members of this family share the following interesting features. First, they transmit light from the visible light to light of 8-10 microns in wavelength. Second, these glasses react rather quickly with water, severely limiting their application. Because of the many potential interesting applications in infared optical systems of these glasses, there is considerable interest in developing coatings for these glasses that will preserved its unique optical characteristics and at the same time provide effective protections of environmental water. Thin film amorphous carbon is a natural candidate. It is easily made by plasma assisted CVD from methane and it is both hard and chemically inert.

Experiments consisting of comparison of two samples of ZBL glass, one coated with amorphous carbon and one not coated, both treated in water under identical conditions showed that the coated sample was much less affected by the attach by water. Indeed, in some areas of a sample, the protection seemed essentially perfect. However, repeat experiments have shown that it is difficult to make defect free

coatings. More studies are needed of the stress in these films, of the adhesion between these films and the substrate, of the conformality of the films, and of the optical clarity of these films (they tend to be slightly brownish).

HISTORIC STAINED GLASS AND THE REACTION BETWEEN WATER AND GLASS

The second glass for which an inert easily applied coating is needed is historic stained glass. In many cases these glasses have survived for 1000 years with little deterioration. However, the industrialization of Europe over the past 50 years have changed the environment so drastically that many will not survive another 20 - 30 years with protective steps in the very near future.

This is actually a much broader question than may seem. Many different glass compositions were used (over time, different locals) and the atmospheric conditions vary greatly. We have begun to study the mechanisms of the reaction between water and glass modeled to simulate some of the historic glasses with the idea that we can provide long term protection with a plasma CVD coating.

The glass studied most intensively to date is a mixture of SiO_2, CaO and K_2O. Based on a large number of other studies, it is to be expected that the attack of this glass by water (or aqueous solutions) will be led by an ion exchange and interdiffusion reaction where K-ion from the glass exchange and interdiffusion with hydronium ions from water. To study this reaction mechanism, we have exposed samples of this glass to water (both as a vapor and as a liquid) and subsequently measured the elemental profiles near the surfaces of these treated glasses. Surprisingly, this model historic stained glass behaves very differently from other alkali-silicate glasses studied. For example, the reaction rate is very different *in vapor vs water* and there is no indication that the ion exchange reaction outlined above occurs in this glass.

This is a very interesting result which we are pursuing and which may have a rather general importance to the general question of the reaction between water and glass. Because of this and because of the general interest in the historic stained glass problem, we are beginning to collaborate with the Fraunhofer Institute for Silicate Research, Wurzburg, West Germany and have received notice that NATO will provide some future support for this work.

PUBLICATIONS

To date, none of the above work has been published. Both the work on coating of ZBL glass and on the mechanism of reaction between water and glass are being prepared for publication.

SCIENTIFIC PERSONNEL

W. A. Lanford, Principal Investigator, no support from this contract.

Ellen Berning, Fellow, Masters Degree in Physics, 1987.

Josh Shapiro, Research Assistant, now working toward a Ph.D. in Physics at Harvard.

Anne Weiner, now on the staff of the Chicago Museum of Science and Industry.

Daniele Cherniak, Fellow, Ph.D. 1990.