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Technical Report No. A-0094

INVESTIGATION OF COATING PLASTIC LENSES WITH SPUTTER-DEPOSITION PROCESS

Final Report

June 1, 1971

Department of the Army
Edgewood Arsenal
Edgewood Arsenal, Maryland 21010

Contract DAAA15-70-C-0269

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PHYSICS TECHNOLOGY LABORATORIES, INC.

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13. ABSTRACT

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FOREWORD

This research and development program was funded under Contract No. DAAA15-70-C-0269, Edgewood Arsenal, during the period from January 14, 1970, through June 1, 1971. This period included an extension of ninety days with Physics Technology Laboratories, Inc. subsidized effort.

SUMMARY

During the term of this contract coatings from 500 to 10,000 Å thick of SiO_2 and Al_2O_3 were deposited on polycarbonate lenses. Techniques were established for depositing transparent, glass-like coatings without introducing changes in the visual properties of the lenses. One result of the program was the discovery that the present "mar resistance" test is not applicable to testing of optical coatings. The technical objectives of the effort were met with the exception of an anticipated parametric study. In the final stages of the investigation, effort was applied to the preliminary design for mass production equipment. The results of this investigation provide sufficient information to enable the prediction of production rates.

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TECHNICAL OBJECTIVE:

To investigate the coating of plastic lenses through the sputter-deposition process for added protection of the lenses against scratching, marring or agent penetration in order to improve the operational use of respiratory protective systems. The contract involves experimental, developmental or research work.

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AIMS:

To develop experimental procedures and techniques of sputtering to coat plastic lenses and collect data on test analysis of coated lenses with conclusions and recommendations, and provide a preliminary design for mass production set-up time, cost and production rates.

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I. INTRODUCTION

In the past several decades extraordinary advancements have been achieved in synthetic materials. Developments in plastics have been so successful that such materials have found optical applications as lenses. Although, when compared to glass, plastic is much cheaper and more shatter resistant, it is also much softer and, consequently, susceptible to scratching and marring. Such difficulties can be overcome by increasing the hardness of the lens. However, as hardness is increased, flexibility and shatter resistance are compromised. Consequently, a design paradox exists as the lens material must be hard enough to resist scratching and marring, and soft enough to provide flexure and shatter resistance. Because much of the scratching is caused by abrasion of dust particles as the lenses are cleaned in the field, and because much of the dust is probably SiO_2, scratch resistance can be attained only if the lenses are at least as hard as glass. However, such hardnesses far exceed an acceptable range for flexure and shatter resistance.

A method for circumventing the design paradox is provided by advances in thin film physics. With these advances, two materials can be combined in such a way as to exploit the advantages of both materials and neutralize their disadvantages. For example, a solution to the lens problem would involve deposition of a hard thin film (like sapphire or quartz) upon soft plastic. It is the nature of thin film properties that the deposited hard layer will attain much of the ductility and flexibility of the soft plastic substrate while retaining its own hardness to enhance scratch- and mar-resistance. This report describes the application of sputtering for the attainment of these hard films.

Sputtering, a relatively new technology, accomplishes the deposition of thin films by placing the substance (target) in a vacuum and generating a plasma (a space charge neutral ionized gas, usually argon). If the target is a conductor or semiconductor, a negative potential applied to the target will cause it to be bombarded by positive ions from the plasma. For each ion striking the target, a number of neutral target atoms will be expelled from the target surface (number or yield varies as a function of target material and ion energy). Sputtered neutral atoms are thus deposited on a substrate, atom layer by atom layer, to provide remarkable control over material thickness and extremely high bonding strength.

Dielectric materials can also be sputtered by backing the target with two conductive D-plates, across which an RF potential is applied. As one plate swings negative, the portion of the dielectric in contact with the plate becomes polarized to produce an apparent negative charge on the dielectric surface exposed to the plasma. This negative charge causes positive ion bombardment from the plasma to sputter neutral dielectric atoms. As the plate becomes positive, electrons are drawn from the plasma to neutralize target surface charges so that the sputtering cycle may be repeated.

One interesting advantage of sputtering, not directly applicable to this program, is that unique alloys or composites can be formed by sputtering several different target materials simultaneously onto a substrate. Quantity of each material in the alloy can be controlled accurately by alteration of target potential or target size. Because the deposited surfaces are built up atom layer by atom layer, there is no requirement for one material to be soluble within another to formulate unique alloys.

Sputtering is extremely flexible, not only in the variety of materials compatible with the process, but also with the variation in techniques that can be applied. For example, we often sputtered the substrate prior to deposition so that the coating is applied to atomically clean surfaces. In addition, low rate sputtering of the substrate can be continued throughout the deposition process (bias sputtering), activating the surface (without elevating substrate temperature) and causing epitaxial growth. The basic sputtering process, with its atom layer by atom layer deposition, accounts for extremely high bond strengths to parent materials. The additional features of atomically clean substrates and activated surfaces (all in the same production step) provides bond strengths unheard of with conventional coating processes.

II. BRIEF DESCRIPTION OF THE SPUTTERING PROCESS

In recent years the technique of laying down thin films had been limited primarily to evaporation and chemical decomposition techniques. Recent development in the field of sputtering, i.e., the removal and deposition of thin films by ion bombardment of the prime material, has opened up entirely new and advanced methods and compositions that are now possible in all fields where thin films are required. Such thin films are being used extensively in microelectronic circuitry, in optical filters and coatings, in surface protection requirements, as well as other advanced scientific applications. It is believed that numerous advances and applications of thin films in all of these areas are possible through further research and development in sputtering techniques.

Sputtering can be described most simply as the emission of target particles under the impact of neutral or charged particles on a target surface. There exist two sputtering processes commonly referred to as "physical" and "chemical" (or reactive) sputtering. Chemical sputtering arises whenever a reactive ion and the target material form volatile compounds. Physical sputtering on the other hand is caused by a collision process between incident ion and the lattice atom. The resultant sputtered material in both cases provides the material from which thin films are formed.

The proposed method of formation of special surfaces has the additional advantage of being prepared under high purity conditions. The work is done in vacuo which precludes the normal contamination encountered in the atmosphere. Newly formed surfaces can be considered to be as clean as the bulk material from which they are formed. Even the presence of monolayers of impurities can be eliminated. Hence, the entire process lends itself to sterilized or sterilizable surfaces.

The sputtering technique can be used to deposit high temperature materials. In the preparation of graphite surfaces, for example, pure graphite or compounds consisting in part of graphite can be formed by simultaneously sputtering from several materials. The resultant films formed by multi-sputtering can be of any combination of materials and entirely new solid state compounds or composites are possible. W. R. Sinclair of Bell Telephone Laboratories has produced complex oxide system films (binary, ternary, etc.) by sputtering carbides in an oxygen atmosphere.

Even though the process of sputtering has been used in the past for thin film deposition it has only recently been possible to sputter at pressures in the submicron range and under high purity conditions. Initially, diode sputtering was used as a crude source, later followed by more refined triode systems wherein the sputtering voltage can be varied independent of the plasma density. Presently, a third generation method* has been developed which is a RF-generated** plasma and DC/RF sputtering complex. This arrangement eliminates all possibilities of sputtering holders or bell jar components, and does not require any filaments, hence, enhancing purity. Any material, conductive or dielectric, can be sputtered with rare gases as well as reactive gases such as oxygen.

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* A commercial sputtering unit developed and patented by PTL is being manufactured and marketed by Varian Associates. Pictures of this apparatus are included in the Appendix.

** Radio frequency.

Review of the Deposition of Various Materials by Sputtering:

In the research and development effort of laying down thin films with specified crystal structures, stoichiometry, and other physical parameters, a well controlled and reproducible method is mandatory. A great deal of work has been done in the field of evaporation and chemical vapor deposition of thin films in vacuum but both methods have certain limitations. One of the newest and least explored techniques of thin film deposition is that of sputter deposition and bias sputtering. The literature over the last several years has included many preliminary studies of the use of the sputtering technique, the results of which show the tremendous possibilities which the method has to offer.

To review only a few of these studies, we refer to the work by the researchers at IBM. Maissel and Schaible[1] published a paper in 1955 in which they described the use of bias sputtering for activating the sputter deposited materials at the substrates. The bias sputtering used in proper sequence was found to provide excellent adhesion of films to glass and other type substrates. Bias sputtering, when applied to the substrate, provided a cleaning action which enhanced the purity of surface by the selective removal of contamination on the surface. Furthermore, bias sputtering, when properly applied, had a predominant effect on activating the surface layers so that atoms migrated to tightly bound sites in the lattice, hence, insuring epitaxial growth. The IBM researchers [2, 3] carried out further work on RF sputtering of insulators and were able to show that SiO and SiO_2, boron nitride, alumina, pyrex 7740, teflon, and many other complex dielectric materials, could be properly deposited by sputtering.

Jorgenson and Wehner[4] also reported work on the deposition of thin film dielectrics by RF sputtering and found it to be readily accomplished if controlled in the proper manner. Jorgenson and Wehner showed, for example, that sputtered SiO_2 on quartz substrate had a much higher radiant energy transmission than evaporated quartz, and, furthermore, had almost a constant light transmission between 300 and 900 μm.

Work has been reported on the deposition of the various alloys such as nickel-chromium films. Pratt, Weintraub and Wage[5] of the U.S. Army Electronics Laboratories, Fort Monmouth, made a study entitled, "Comparison of Some Physical and Chemical Properties of Vacuum Evaporated and Sputtered Nickel-Chromium Films." In a step-by-step comparison of evaporated versus cathodic systems they came to the following conclusions: (1) In the vacuum evaporation process nickel-chromium ratio varies with the sheet resistivity, whereas sputtered films had ratios which tended to remain more nearly like the starting bulk material; (2) The temperature coefficients of the resistance of sputtered films were more constant and equal to the bulk material than evaporated films; (3) The film density of the sputtered films remained relatively constant and nearly as high as the starting material, whereas vacuum evaporated film density decreased appreciably. This study gives a fairly good indication that the sputter deposition technique will provide homogeneous films with more reproducible physical characteristics of the bulk material than the vacuum evaporation method.

An early paper by Wolsky, Shooter and Zdanuk[6] entitled, "The Sputtering of Compounds," demonstrated the use of sputtering to reproduce intermetallic compound semiconductors in thin film form. They found that GaSb could be sputtered and reproduced in physical characteristics if proper control conditions were maintained.

Under an Air Force research contract Krikorian and Sneed[7] have done a great deal of work on cathodic sputtering of thin films. They have investigated the differences of evaporated and sputtered thin films as related to crystal structure, epitaxial growth, resistivity, and substrate types. It was this research team who proposed for the first time a "triple point" in the nucleation and growth of germanium thin films on single crystals as a function of temperature and deposition rate. They found that if the temperature is kept high enough a single crystal will always form, yet if the temperature is lowered the deposition rate must be lowered to form single crystals. However, there was a certain T/R (temperature/rate) value at which only polycrystalline deposits would be formed and below a second T/R value the deposition would be completely amorphous. These researchers have not, to our knowledge, applied the use of bias sputtering to enhance singly crystal formation at low substrate temperatures, or used steps to produce organic thin films.

Again in the field of alloy deposition, Smith[8] made deposits of nickel-iron films by cathodic sputtering and electro-depositions. Even though his work was brief in duration, he was able to show that the sputtered thin film technique provided many controllable parameters to obtain the proper magnetic thin films he was striving for.

In a study of the sputtering rates of semiconductors, work was done by Laegried, Wehner, and Meckel[9] on germanium and silicon. The results of this study showed that the control of deposition rates could be well predicted if one knew the bombarding ion energy and the current density. In the evaporation technique for growing thin films in vacuum, the control of the deposition rate is very difficult and cannot be varied over the wide ranges possible by sputtering.

The March 1966 issue of the Transactions of the Metallurgical Society of AIME includes several papers on sputtering of thin films of metals, dielectrics, and semiconductors for numerous applications. The properties and structure of thin silicon films sputtered on amorphous fused quartz substrates has been reported by Kumagai, et al[10]. They obtained some degree of crystalline silicon when deposited on substrates with tempera-trues between 50°C and 400°C. They did not use bias sputter. (It is believed that concurrent bias sputtering nucleation and growth from one point would result in the desired form.)

The effects of purity on silicon epitaxial growth are discussed in a paper by Jona[11]. He used low energy electron diffraction to study the early stages of epitaxy, and noted the effects of crystal planes as well as contamination on the growth process.

Recently, experimentation has been done by Herzog et al[12] wherein sputtering has been used as a source for mass spectrometry. They have found that whole microcrystalline units of material can be sputtered using KeV argon bombardment. This suggests that the composition of such sputtered heavy molecules reflects the crystalline structure of the parent material and hence may be transported to a substrate without destroying the stoichiometry of the material.

III. EXPERIMENTAL PROCEDURE

The experimental effort in the deposition of hard coatings on polycarbonate was done in an RF/RF-DC Sputtering System. In this system the plasma is generated and maintained by the use of an induction coil driven by radio frequency of approximately 2 to 20 mc/sec. (This information is available in more detail in the appendix.) The sputtering targets of pyrex, quartz, etc., are mounted on the "D" plates of the second RF circuit. These plates are driven by capacitance-coupled radio frequency and it is at this (target) surface that the sputtering takes place.

The sputtering head is mounted in a 4-inch diffusion-pumped system that has a water-cooling baffle just above the pump. The upper chamber consists of a 9-inch diameter aluminum chamber with an extension of a pyrex bell jar with the same diameter. (Photograph 1 with the indicated system parts is provided here.) The pumping system has a capability of 400 liters/sec speed and most of the tests were made at background pressures of 1 to 5 x 10^{-6}mm Hg. The working gas pressures of argon or oxygen vary between 5 x 10^{-4} and 5 x 10^{-3}mm Hg.

The arrangement of the system includes two intermediate meshes between the target area and the lenses substrate location. These meshes are used to repel the plasma from the substrate region and to suppress the bombardment of the lenses with high energy electrons. Appropriate geometrical configurations have also been used at the targets to reduce secondary electron emission and acceleration towards the lens surfaces.

Experiments were made by sputtering with an ion beam. Sputtering is caused by bombarding target surfaces with energetic ions. Conventionally, ions are made available by exciting a plasma. Bombardment can be effected by charging the target negative with respect to plasma, thereby attracting the positive ions. However, there are alternate sources for ions. One such source is an ion beam. The greatest advantage of an ion beam is that there is no plasma in the target-substrate region to cause heating. A second advantage is that the substrate can be exposed easily to the ion beam for a short period, prior to deposition, to sputter-clean the substrate. In addition, bombarding ions in an ion beam can be accelerated to high energies (10,000 electron volts with equipment on hand at PTL) to increase sputtering yield. An experiment was conducted with a polycarbonate substrate. An extremely hard, transparent and well adhering film was formed with this method. It is believed that this technique should be evaluated further because: (a) It will eliminate the source of heat, which has caused the problems previously discussed, and (b) it may provide better protective films than those deposited by conventional sputtering techniques.

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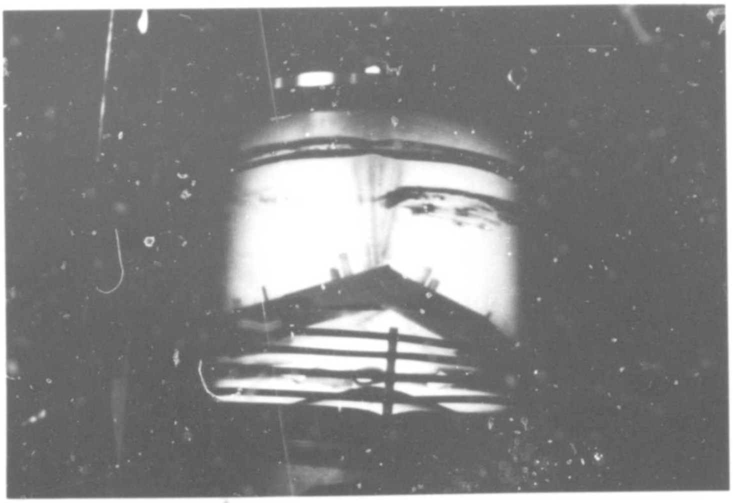

Photo. 1 - Sputtering Chamber

The following candidate coating materials were used as sputter targets: (1) Al_2O_3 because of its extreme hardness; (2) SiO_2 because of its relative high hardness and desirable qualities as a chemical barrier; (3) PYREX #7740 with a composition of 81% SiO_2, 2% Al_2O_3, 13% B_2O_3 and 4% Na_2O. PYREX #7740 was selected because of relative hardness (slightly less than quartz), desirable qualities as a chemical barrier and reduced cost when compared to quartz and alumina. This represents a slight deviation from GSC-1 glass (85% SiO_2, 10% B_2O_3, 5% Al_2O_3). However, the PYREX #7740 possesses desirable temperature survival characteristics and is much more readily available.

During the study, it was established that all substrate materiasl are compatible with vacuum. However, all materials are sensitive to heat. With polycarbonate, heat apparently causes substrate expansion sufficiently dissimilar from the deposited layer that when the substrate cools and contracts, the thin film shatters. This was eliminated by moving the substrates far away from the sputtering targets and by operating with low intensity plasmas. Water-cooled heat sinking was used, but did not appear effective.

Vinyl is compatible with vacuum. However, application of heat causes severe outgassing which prohibits successful deposit and destroys the substrate. The vinyl appears extremely sensitive and every step taken with the conventional sputtering equipment has failed.

Silicon rubber is the most resistant to heat of the three substrates. However, heat causes sufficient expansion to permit penetration of the depositing atoms deep into the substrate pores. Although this provides extremely high bond strength, it also causes the formation of a rough surface which reduces transparency.

IV. OPERATING CONDITIONS

The procedure followed in experimentally producing the films involved:

A. Modification of vacuum equipment to handle the gas load introduced into the chamber by the lens material. The aluminum bell jar system shown in Photograph 2 was used for the successful deposit of clear, thick films of SiO_2 and Al_2O_3. The pumping equipment consists of a 4-inch diameter oil diffusion pump backed by a mechanical pump. A water-cooled baffle is installed above the diffusion pump to provide an optically dense trap for molecules from the pump area. It was found that lenses were affected by the presence of backstreaming molecules and by outgasses solvent materials under sputtering discharge conditions. It was therefore necessary to eliminate backstreaming and to provide adequate pumping speed at the chamber to insure removal of the products of outgassing.

B. Determination of the conditions under which sputter-deposited film can be produced on lens material.

Conditions such as RF discharge power setting, target voltage and current, target to lens spacing, background pressure, gas used, target material, and lens cleaning procedure all have important consequences relating to the film properties. The procedure and conditions that produced durable films of SiO_2 and Al_2O_3 on lenses were as follows:

(1) Cleaning Lenses.

The lenses were cleaned in distilled water to which Alconox was added: 15 grams of Alconox to 2 liters of water at 80°C. (Alconox is distributed by Van Waters and Rogers, Inc.) The lenses were then rinsed in distilled water and dried with heated air.

(2) Glass slides were cleaned by the same procedure and placed in the vacuum chamber with the lenses. The slides were weighed before placing them in the chamber and again at the conclusion of the sputtering run. Their increase in weight served as an indication of the amount of material deposited during the run.

(3) The RF discharge conditions under which clear films were produced were 1850 volts 250 ma output from a source operating on a frequency of 10 - 20 megahertz. The RF power was delivered to a 2-turn coil 12 inches in diameter inside the vacuum chamber. This RF coil serves to ionize a fraction of the gas used for sputtering.

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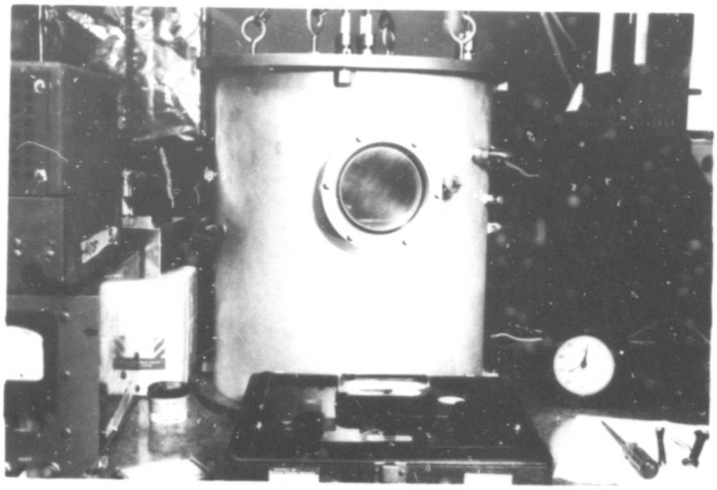

Photo. 2 - Vacuum System

(4) Target voltage was varied for different runs over the range 1000 volts to 3000 volts. The target voltage, also at 10 - 20 megahertz was supplied to targets in two configurations. Early in the program two targets were used in a configuration that placed the plane of one target at 90 degrees to the other. This design is capable of the highest deposition rate.

The alternate configuration places the two targets, actually one disc of target material coated on the non-sputtered side with a metal film except for an uncoated strip across the diameter, on two "D" shaped target holders. The RF voltage is applied to the targets with one "D" receiving the maximum negative amplitude when the other is at the maximum positive amplitude. Because the RF signal varies at 10 - 20 megahertz the target material receives electrons, from the ionized gas within the chamber, at a rate that is sufficient to eliminate charge build-up on the target surface. Therefore the ions that drift to the vicinity of the target are accelerated to the target by the applied RF field with sufficient energy to sputter target material.

(5) Target to lens spacing.

Lenses were coated when placed as close as 2 inches from the targets and as far away as 14 inches. The material deposited is reduced as the target-to-substrate distance increases in the ratio

$$wt_2 = \sqrt{2}\, d_1/d_2\ wt_1$$

where d_1 = distance of nearest substrate (lens)

d_2 = distance of farthest substrate

wt_1 = amount of material deposited per unit area at d_1

(6) Background pressure.

The gas pressure was set by considering the trade-off between the increased ion density at higher pressures and the longer mean free paths of sputtered material at lower pressure. Reasonable deposition rates are achieved at pressures of the order of 5×10^{-3} Torr.

(7) Gas used.

Argon was used as the sputtering gas because it is inert, available, and has high atomic weight and is therefore efficient in sputtering. Oxygen was used as the sputtering gas in order to improve the stability of the sputtered films. Additional work must be done to fully evaluate the properties of the sputtered films in terms of adhesion and elimination of stresses. The disadvantage of using oxygen is that the deposition rate is less than half that when argon is used.

V. THE LIMITATION OF THE "MAR RESISTANCE" TEST*

One of the major questions raised during the test procedure was the validity of the "mar resistance" test. In the early part of the program, the mar resistance tests which included the dropping of #80 carborundum grit on the lenses was found to etch SiO_2, Al_2O_3, and glass deposits made on the lenses. The etching which resulted from this mar test was found to be almost as bad as occurred when the uncoated polycarbonate was subjected to the falling grit. It came to our attention that we should attempt to see how this mar resistance test affects actual solid surfaces. Hence, we applied the test to a thick disc of SiO_2. We followed this with alumina (Al_2O_3), Pyrex glass (microscope slide), silicon slab and the plastic lenses. In every case the results indicated that all of these surfaces etched severely. It would seem that our search for material which would coat the lenses and which would be resistant to this test is without a doubt an impossible task.

We then attempted to determine if those grit materials to which the actual lenses would be exposed would have the hard quality of the carborundum used in the test. There is no doubt that most sand and other silicates would indeed have this hardness and, hence, marring of the surfaces may be inevitable. This led us to consider if this test was valid.

We, therefore, at the final stages of the program when this was eventually considered, believe that it may be necessary to use a different mar resistance test such as the weighted-scratch test described by Holland and other authors. In any event, we feel that some of our tests actually were not appropriate for determining positive results in this program.

A set of samples including all of the materials listed above, plus plastic lenses coated with SiO_2 and Al_2O_3 are shown in the attached Photograph 3. (These actual samples have been mounted and are being included as part of this final report for the direct inspection by the contract personnel.)

During the testing period, the freezing/boiling thermal shock test indicated that none of the coatings shattered. The room temperature impact test showed that the thin film coating also remained intact as long as the impact was not great enough to break the plastic lens itself. Many deposits were seccessful in forming good layers without fisual distortion and apparent light transmission and hazing.

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* Standard Mar Resistance Test, Method 1093, part of Red. Test Method Std. No. 406 (formerly Fed. Spec. L-P-4066).

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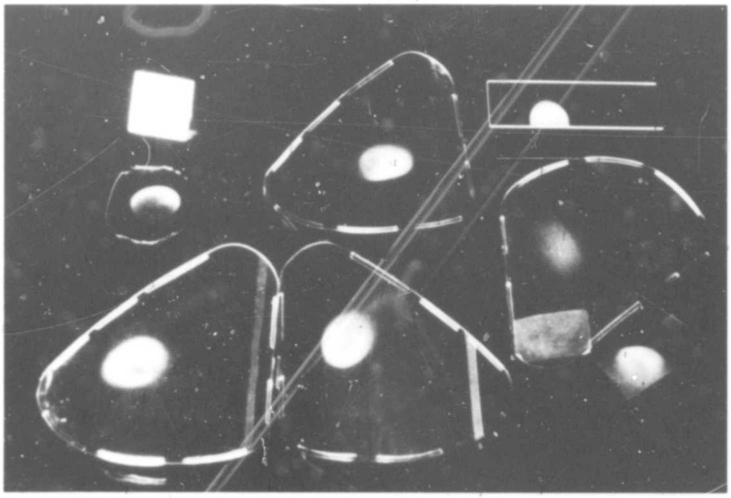


Photo. 3 - Coated Lens Samples

VI. RESULTS

In the initial phase of the contract the following conclusions were reached:

(1) All substrates are compatible with vacuum.

(2) All substrates are sensitive to temperature.

   a. Good films have been deposited on polycarbonate by by placing the substrate far away from the target and operating at low plasma intensities.

   b. Silicon rubber survives plasma temperatures. However, expansion permits infusion of deposit into material pores, clouding the substrate. The resultant surface is lint-free.

   c. Vinyl outgasses severely when subjected to heat and no deposits have been successful. (Deposits have not yet been made with the ion beam technique upon vinyl.)

(3) Sputtering with an ion beam provides a very promising technique.

   a. Plasma is removed from the substrate-target chamber, eliminating the source of heat.

   b. Sputter cleaning of the substrate is performed simply.

   c. The technique may provide better films than possible with other methods and it is as practical for mass production.

(4) The most suitable type of protective layer material has been determined to be Al_2O_3.

   a. Al_2O_3 was the hardest material considered.

   b. Al_2O_3 provided the clearest deposits.

   c. Deposition rate of Al_2O_3 was about the same as the other materials evaluated.

   d. Al_2O_3 accounted for the lowest materials cost.

The effort in Phase II was used to investigate some of the film deposited on polycarbonate.

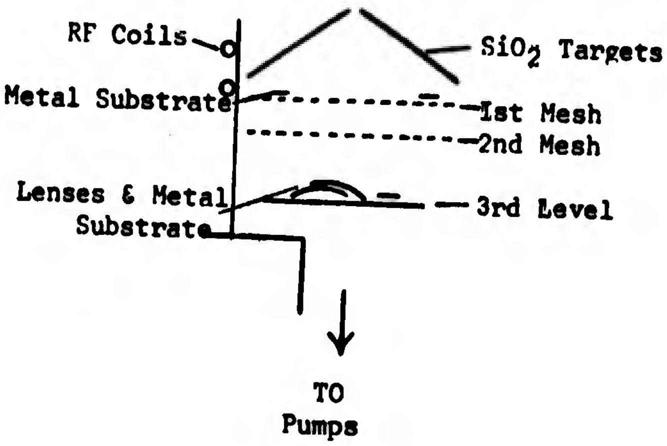
In addition to routine experimentation, experiments include thermal shock testing during which coated lenses were reduced to 0°C and then placed directly into boiling water. The results showed no deterioration of the sputter-deposited film. Lenses were also subjected to abrasive tests by dropping #80 carborundum grit from a height of 36 inches. This was a severe test that scratched both the coated and uncoated lenses; however, subsequent microscopic examination revealed the depth of scratches on unprotected plastic was two to three times greater than that of the scratches produced in the sputter-deposit protected films.

During the later stages of the contract we found that the deposition of silicon oxides in argon atmospheres had a deficiency of oxygen. The films obtained were brownish in color indicating SiOx, where x is less than 2. (SiO is light brown.)

We then attempted sputtering runs using oxygen for the sputtering gas. It is known that this procedure can be used to replace oxygen in oxide films where normally, under rare gas deposits, some oxygen is lost during the process. Both plastic lenses and metal strips were used as substrates, and a run made at 1000 eV and 0.55 amps of sputtering power.

During these runs we could see the SiO_2 being deposited on the metal strips placed in the top portion of the sputtering chamber. As the clear SiO_2 layers built up there appeared the color changes common to reflections characteristic of various wave lengths of the optical spectrum. Hence, as the layers built up through 1/4 wave-length depths the colors went from reddish to green, to bluish hue. This showed that good clear quartz layers were being formed on the top layer of the target.

The arrangement of the experiment was as shown here:



The lenses were placed low in the chamber to provide distance and mesh barriers between the plasma and the lenses. This reduced heating as well as fast electron bombardment of the lenses.

We again attempted the same run, but with the lenses near the target. We were unsuccessful, however, in keeping the plasma away from the lenses and a resulting etched layer appeared on the lenses. A new attempt was made with additional potentials placed on the meshes, but with the same unsuccessful results.

The earlier run looked quite good with deposits of 4300 Å. Tests were run fro peeling using the tape test. Cold and hot tests were made and the films stayed on. Drop tests of #80 grit were made to determine etching, but the results were inconclusive. These lenses were subsequently sent to Edgewood Arsenal for further testing.

Since these tests were made we have attempted to obtain faster, better deposits of SiO_2 by oxygen sputtering. We believe we have a possible successful process but have not yet received test information from Edgewood on the lenses we sent to them. We will also try to deposit the SiO_2 on the silicone rubber.

VII. PRODUCTION FEASIBILITY

The work performed during the feasibility study of this contract showed conclusively that coatings could be applied at sufficient sputtering rates to allow for the mass production of sputter coating lenses at reasonable cost.

Sputtering rates of approximately 1000 Å per minute are readily available for most materials used in the feasibility study (i.e., quartz, SiO, Al_2O_3, etc.). Using this rate of deposition and allowing 12 lenses to be processed, side by side (in parallel), on a conveyor belt through a continuous sputtering chamber, then 12 lenses could be coated per minute per target area used in the sputtering chamber. Therefore, using a sputtering target 36 inches in effective sputtering length would allow the conveyor to flow at a speed of 36 inches per minute--or coating 12 lenses in series by 12 in parallel. The overall rate of lenses coated would be 144 per minute (each lens is 3 inches in length) or 8,640 per hour. An excellent rate of production per machine. Since both sides must be coated for maximum effectiveness, it is readily seen that over 1000 lenses may be coated per hour.

VIII. PROBLEMS ENCOUNTERED AND SUGGESTED SOLUTIONS

Several surmountable problems delayed the results in this experimental effort which we list and discuss below.

A. Plastic surface/coating compatibility:

An unexpected problem came to light early in the investigation in the excessive outgassing of the polycarbonate under simultaneous vacuum and heat environmental conditions. Polycarbonate has a low vapor pressure at room temperature, but when the plasma from sputtering increased the temperature above 80°C, the emission of occluded gases from the plastic prevented successful coating applications. This problem was eventually overcome by the isolation of the plasma and fast electrons from the substrates by using grids, extended spacing between target and substrate, and/or by lowering the sputtering power input.

B. Film deposition rate:

As mentioned above, it was necessary under some conditions to lower the sputtering power input to reduce deleterious heating in the plastic substrates. Since the deposition rate goes almost linearly with the power input, this power reduction subsequently lowered the deposition rate. This problem was and can be further overcome by proper target to substrate spacing and geometry. Larger targets, for example, at extended stand-off distances would accomplish deposition rates of several thousand angstroms per minute of deposit thickness without undue heating.

C. Mar testing techniques:

The grit-drop test to determine abrasive resistance did not seem to be a reasonable testing procedure. It turned out that the grit when dropped as prescribed on a thick sheet of SiO_2 or Al_2O_3 caused etching and hazing of the surface. In like manner, the thin films deposited on the lens were also etched. We see no solution to such etching without assuming new exotic film materials (other than silicon oxide sand particles on silicon oxide surfaces), or the assumption that the abrasive particles are less hard than the surface material. Is the etching granule substance encountered generally as hard as the #80 carborundum used in the Mar Resistance Test?

These were the major problems encountered and the subsequent solutions applied.

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IX. RECOMMENDATIONS

Although the feasibility program ran out of funds before a firm and valid process was derived, sufficient data and success were achieved to determine that sputter coatings were indeed possible for application to plastic lenses. Several changes in future programs are necessary before valid results may be firmed for a reproducive process.

a. The testing procedure appears to be inapplicable to the study. It is recommended that more valid or determinative tests be developed to prove out the coatings and compare same to uncoated lenses.

b. It is recommended that a development program be established to select one of the materials investigated (i.e., Al_2O_3) and to coat several hundfed lenses under various processing parameters; submit these test species to various test procedures and center in on the optimum process.

c. Evaluate the development program as outlined in b. above and determine the practical application of producing the lenses on a productive basis.

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