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A PROTECTIVE GLASS COATING FOR PLASTIC OPTICAL COMPONENTS

June 1975

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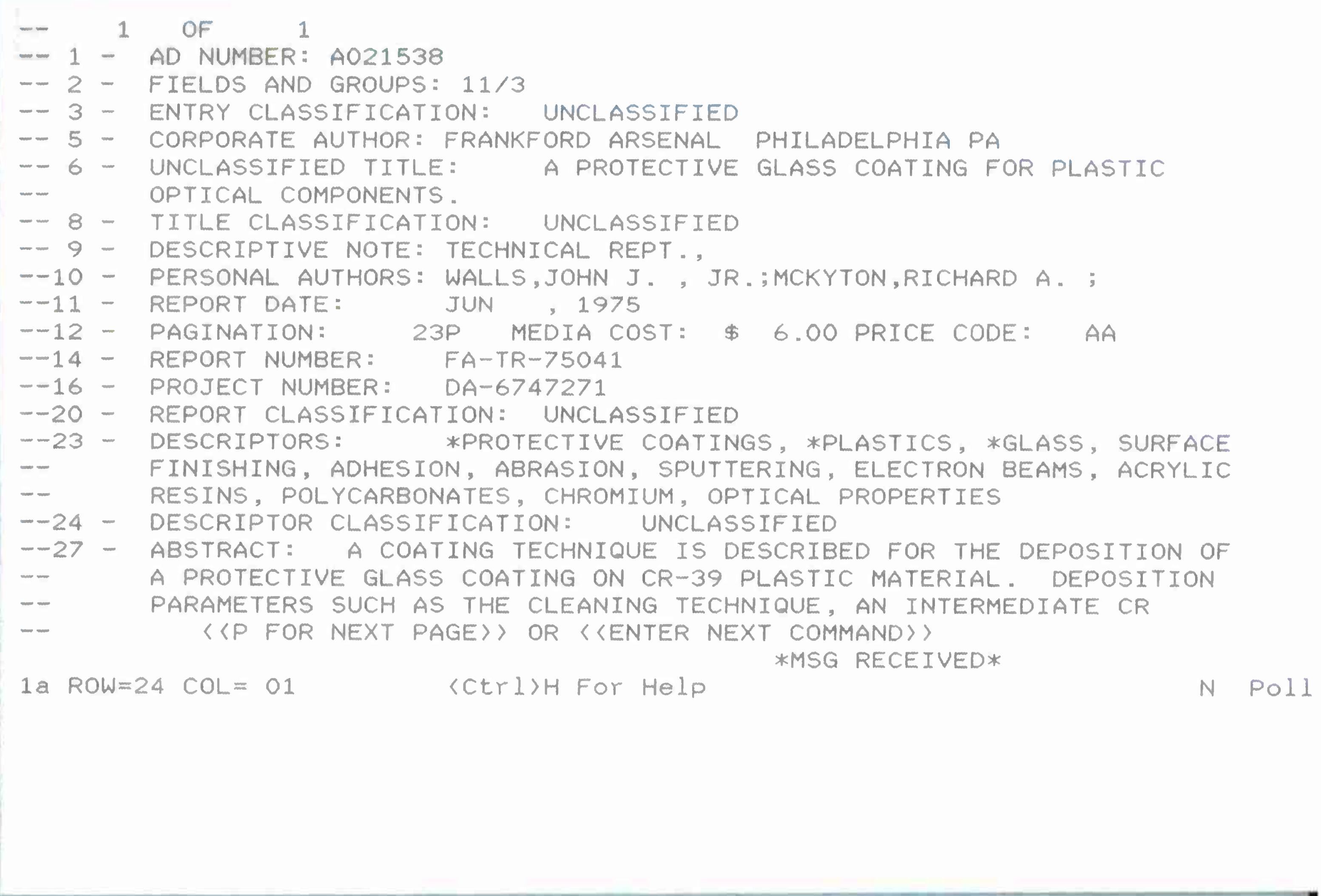

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A coating technique is described for the deposition of a protective glass coating on CR-39 plastic material. Deposition parameters such as the cleaning technique, an intermediate binding layer, and the thickness of the glass coating have a significant effect on the adherence and abrasion resistant properties of the coated plastic. The optical and physical properties of the coated plastic are presented as are several applications of this coating.

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INTRODUCTION

One of the major problems associated with the use of plastic materials for military applications is the vulnerability of the optical components to surface damage. Although there is a range of hardness values for plastics, they all exhibit hardness values several orders of magnitude below the optical glass materials. Several techniques have been developed to give the plastic surface some degree of protection. Some methods make use of organic films for protection such as glass resins (silicon compounds) and fluorocarbon type materials. Although there is a definite improvement in surface protection they still will not pass the military eraser abrasion test per MIL-C-48497. Other methods using the process of vacuum vaporization of inorganic film materials have been to date used with moderate success and sometimes prohibitive in cost. A problem normally associated with this technique is the use of high substrate temperatures that are needed to achieve the crystallization of the deposited film material. The high temperatures that are used to produce hard coatings cannot be used on plastic materials because of their low melting and deformation temperatures. Thus lower deposition temperatures are used on the plastic materials, resulting in films with hardness values much lower than can be achieved on glass materials. In summary, although some advances have been made to protect plastic surfaces from abrasive damage, the protection is not adequate for military type applications.

It is the purpose of this investigation to study and define a coating process that can be applied to plastic optical components and give the components a surface protection that is capable of functioning within a military environment. The plastic materials considered for possible use as optical components included acrylic, polycarbonate, and CR-39 (Allyl diglycol carbonate). Several film materials were deposited using vacuum vaporization, sputtering, and a hybrid of both techniques. From this study the best combination of both plastic and protective coating was selected and used on applications such as a plastic window for a tank periscope and also on ophthalmic lenses.

METHODS AND PROCEDURES

Samples were prepared using both sputtering and electron-beam evaporation methods. The sputtering study was conducted by Battelle Memorial Institute-Pacific Northwest Laboratories (see appendix) and the electron-beam evaporated samples were prepared and investigated at Frankford Arsenal.

Sputtering[1]

The two sputtering techniques used for depositing coatings on the plastics were the diode and triode methods. One of the basic problems associated with the diode method is the generation of high substrate

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[1] Maissel & Glang, "Handbook of Thin Film Technology", McGraw-Hill, 1970, Chapters 3 & 4.

temperatures by ion and electron bombardment. It was necessary to use a low deposition power in order to maintain reasonable temperatures on the plastics during the coating process. The coating materials evaluated on both acrylic and CR-39 plastics were: SiO_2, ZrO_2, and Al_2O_3. A total of thirty coating runs was performed utilizing the diode sputtering method. All the samples were cleaned with a detergent soap and were subsequently subjected to an additional cleaning using a sputter-etch cycle for one minute at 50 watts. The deposition times were varied between one minute to thirty minutes with deposition powers ranging between 50 watts to 300 watts.

A triode sputtering approach was evaluated using a Balzer Sputron II system. The advantage of the triode system over the diode method lies in the reduction of high energy electrons bombarding the substrate. Reactive sputtering was used to deposit Al_2O_3 films on CR-39 plastic. A total of ten coating runs was made at various power levels. The sputtering deposition times varied from ten minutes to sixty minutes.

Electron Beam Evaporation

It was decided to use Schott glass 8329 for the protective coating material because it can be deposited to relatively thick layers thus providing good abrasion and chemical resistant properties. In addition, this material can be deposited without using special techniques such as reactive evaporation. The material is designed so that it can be evaporated using an electron-beam gun source without concern for material fractionation which could cause a highly absorbing film. The glass material was evaporated using an Airco-Temescal electron-beam gun system. All coatings were deposited at a pressure in the high 10^{-6} torr to low 10^{-5} torr range. The thickness of the films was controlled using an inhouse designed optical thickness monitor.

During the course of this investigation it became evident that the three significant factors involved in depositing a film with good adherence and abrasion resistant properties are: the cleaning technique, an intermediate binding layer, and the thickness of the coating.

Cleaning Technique

The normal cleaning methods that are used for glass materials are not necessarily applicable to plastic materials. When the plastics were cleaned using a detergent and subsequently subjected to ultrasonic cleaning, the surface of the plastic produced a beaded water effect. A surface is normally considered clean if a sheet of water effect can be produced on the surface. In order to achieve this effect an additional cleaning step was used. The samples were exposed to a D.C. glow discharge for various times and positions of substrates with respect to the glow ring. The voltage was fixed at 1.4KV and the chamber pressure was maintained at 35 microns. The samples near the high density plasma (glow ring) started to turn yellow in a very short time (approximately 30 seconds). Spectral transmission curves of these samples indicated a 7% loss of light in the blue spectral region for a plasma exposure of one minute. However, by placing the samples in a less dense plasma, a minimum change in absorption resulted (less than 1%). Subsequent exposure of these samples to water resulted in a sheet of water effect on the surface thus indicating a clean surface had been achieved.

Intermediate Binding Layer

The need for a binding layer developed after several coating runs of the glass film on the three types of plastics indicated a definite film bonding problem. The glass coatings on both the acrylic and polycarbonate materials were easily stripped away using the tape test per MIL-M-13508. The coating on the CR-39 plastic was able to pass the tape test, however, when exposed to a high humidity condition the film started to show definite signs of deterioration and in some cases crazed and peeled off completely. To circumvent this problem a technique conventionally used for bonding noble metals to glass was used. This consists of depositing an intermediate layer of a metal such as chromium. This layer forms a strong oxide type bond to the glass and a coulombic bond to the noble metal. The same approach was used for bonding the glass coating to the plastic substrate. A determination was made as to what thickness of chromium will give maximum transmission and still maintain the required bonding strength. A study revealed that a 0.25% increase in the surface reflectance by the chromium film was adequate to produce a strong film bond.

Thickness of Coating

The thickness of the glass coating turned out to be a very significant factor in determining the abrasion resistant properties of the coating. Coatings of different thicknesses were applied to the plastics with and without the chromium underlayer. The samples were evaluated for their abrasion resistant properties using the eraser abrasion test per MIL-C-48497. Optical film thicknesses varied from one wavelength at 0.55um to ten wavelengths at 0.55um. All the coatings were deposited at ambient temperatures.

PERFORMANCE EVALUATION

Sputtered Coatings

There were two major problems associated with the diode sputtering technique. One problem is the excessive heating of the plastic, the other is the discoloration of the plastic due to the heavy electron bombardment which causes a polymerization effect. Although these effects were reduced at lower power levels, they still existed at power levels as low as 50 watts. Both the SiO_2 and ZrO_2 films had high absorption characteristics and were unacceptable from a users standpoint. These films did exhibit some improvement in their adherence properties but had very poor abrasion resistant properties. The Al_2O_3 films had acceptable absorption and adherence properties but again showed poor abrasion properties. At least six samples were given a very thin layer of Al_2O_3 by sputtering then another full-wave was deposited by electron-beam deposition. These samples also had poor abrasion resistant properties. These results were consistent for all of the three types of plastics used.

Electron Beam Evaporation

Acrylic

An attempt was made to deposit a glass film directly to the plastic. The standard cleaning procedure and plasma cleaning discussed earlier were used. After the 8th quarter-wave of film thickness was deposited, the coating started to show signs of stress and started to craze. Additional attempts were made to pre-treat the surface of the plastic with silica gel and also a glass resin solution. These techniques did not improve the adherence or abrasion resistant properties of the coating. Finally, a chromium underlayer was used with an overcoat of a full wave of the glass coating. Poor adherence of the chromium film to the acrylic resulted. This process was repeated several times. In each case a poor chromium to acrylic bond was observed. The films were optically clear but could be abraded very easily using the eraser abrasion test.

Polycarbonate

Basically the same results obtained with the acrylics were also observed with the polycarbonate material. However, unlike the acrylics there were some cases where the glass coating was deposited with and without a chromium layer and was able to pass the tape adherence test. The thicknesses of the glass coatings were limited to approximately three full-waves since above this value the coatings would go into stress and craze when exposed to moisture. The optical characteristics of the glass coatings did not degrade the base characteristics of the polycarbonate plastic.

CR-39

Physical Properties - In order to determine the optimal thickness for the glass coating several coatings were deposited to given thicknesses. These coatings were evaluated for their damage resistance by four primary tests: tape adherence (MIL-M-13508), eraser abrasion (MIL-C-675), alcohol cleaning, and water solubility for twenty-four hours at ambient temperature. Tables 1 and 2 show the results obtained with and without a chromium underlayer. The results for the glass coating without a chromium underlayer indicate that although it can

Table 1. Environmental Performance of Glass Coating on CR-39 as a Function of Thickness (Without Chronium)

Opt. Thickness (@0.55um)	Adherence (MIL-M-13508)	Abrasion (MIL-C-675)	Alcohol Wipe	Water (24 hours)
1 wave	No effect	scratched	No effect	small surface damage
2 waves	" "	"	" "	" " "
4 "	" "	slight improvement	" "	" " "
5 "	" "	a very few light scratches	" "	" " "
6 "	" "	a very few light scratches	" "	" " "
7 "	" "	one or two scratches	" "	" " "
8 "	" "	one or two scratches	" "	film removal
9 "	" "	one scratch	" "	" "

Table 2. Environmental Performance of Glass Coating on cr/CR-39 as a Function of Thickness (With Chronium)

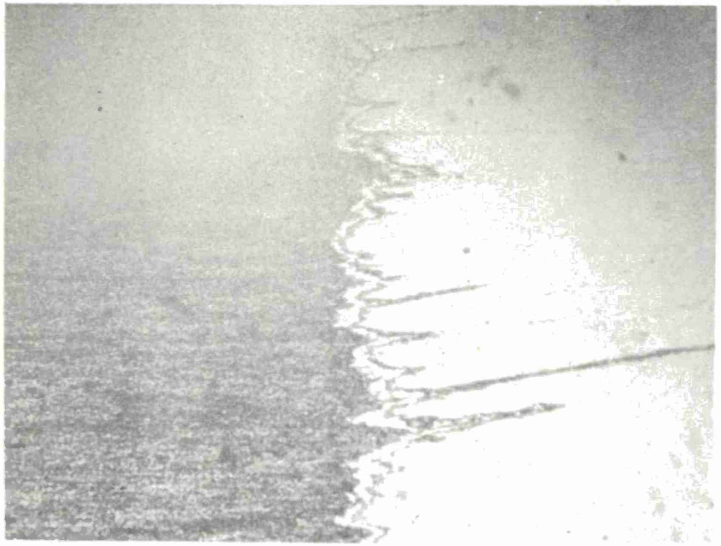
Opt. Thickness (@0.55um)	Adherence (MIL-M-13508)	Abrasion (MIL-C-675)	Alcohol Wipe	Water (24 hours)
cr + 1 wave	No effect	scratches	No effect	No effect
cr + 5 waves	" "	fine scratches	" "	" "
cr + 8 "	" "	few scratches	" "	" "
cr + 9 "	" "	No damage	" "	" "
cr + 10 "	" "	No damage	" "	" "

pass the tape test the bond is still weak enough to be damaged by a moisture condition. Also, there is a sign that film stress is developing as the film thickness increases since the thicker films were easily removed with the water test. Another result that is obvious is the increasing abrasion resistant properties as the thickness of the glass coating increases. The coatings with the chromium underlayer exhibit the same properties as the coatings without the underlayer with one exception; the film bond has definitely been improved and no damage was experienced when exposed to a moisture condition. The coated plastic was also subjected to miltary temperatures and no damage resulted. In fact, a sample was placed in a liquid nitrogen bath and experienced no coating separation.

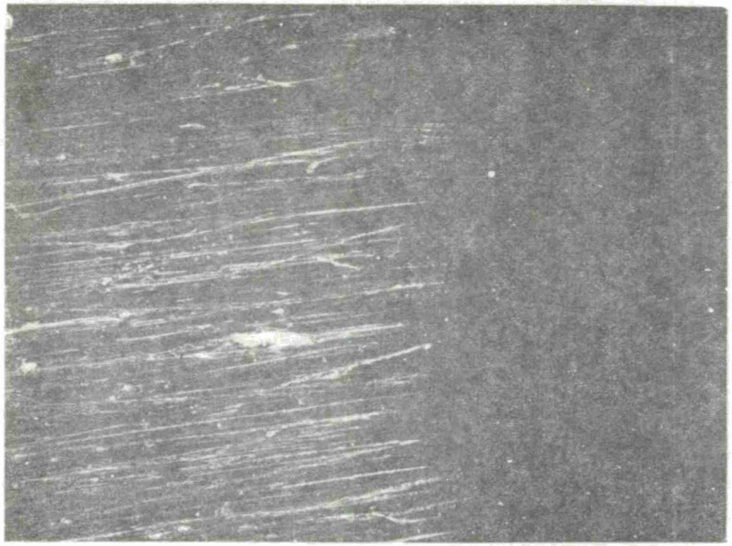
To demonstrate the abrasion resistant properties of the coating, half of the surface of a sample was coated with the glass. The sample was then subjected to 10, 50, 100, and 1000 strokes using the eraser abrasion test. Shown in Figure 1 are the pictures taken at the uncoated/coated boundary. The uncoated area (left side) is progressively destroyed until after 1000 strokes it has no specular characteristics at all. The coated area has only a few scratches after 1000 strokes. Scanning electron microscope pictures (300X, 3000X, and 10,000X) were taken of a coated and uncoated surface that was subjected to the eraser test per MIL-C-675 (20 strokes). The uncoated surface (Figure 2) shows signs of gross deterioration whereas the coated sample (Figure 3) has a scratchless surface and a fine grain structure (white dot in picture is an identification mark).

In order to compare the abrasion resistant properties of this coating with materials presently available, an abrasion/transmittance test was devised. The test consisted of abrading each sample for a given number of strokes then measuring the transmittance in the abraded area and repeating the process to determine the transmittance degradation as a function of the number of strokes. A Cary 14 spectrophotometer set at 0.55um was used to measure the transmittance characteristics The materials tested include: acrylic, polycarbonate, CR-39, abcite coated acrylic, glass resin coated polycarbonate, a Japanese coating on CR-39 (Toome-Lite), and the Frankford Arsenal glass coating on CR-39. Figure 4 indicates the results obtained by this method.

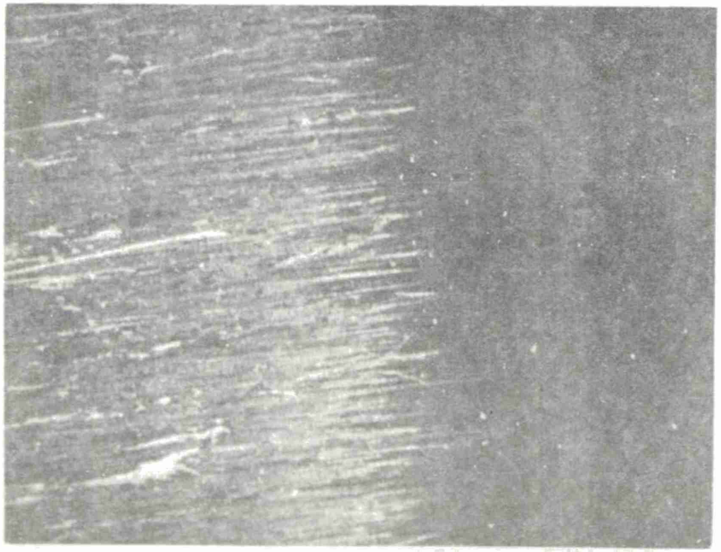
Optical Properties - The spectral transmittance of a coated and uncoated sample was measured and is shown in Figure 5. The sample was coated on both sides, and as indicated from the transmittance curve has experienced very little change in transmission compared to the uncoated sample. To the eye, the transmission color of the sample is neutral as is the surface reflection. Pictures of the coating shown in Figure 3 reveal a fine grain type structure with no light scattering problem.



1000 abrasion strokes

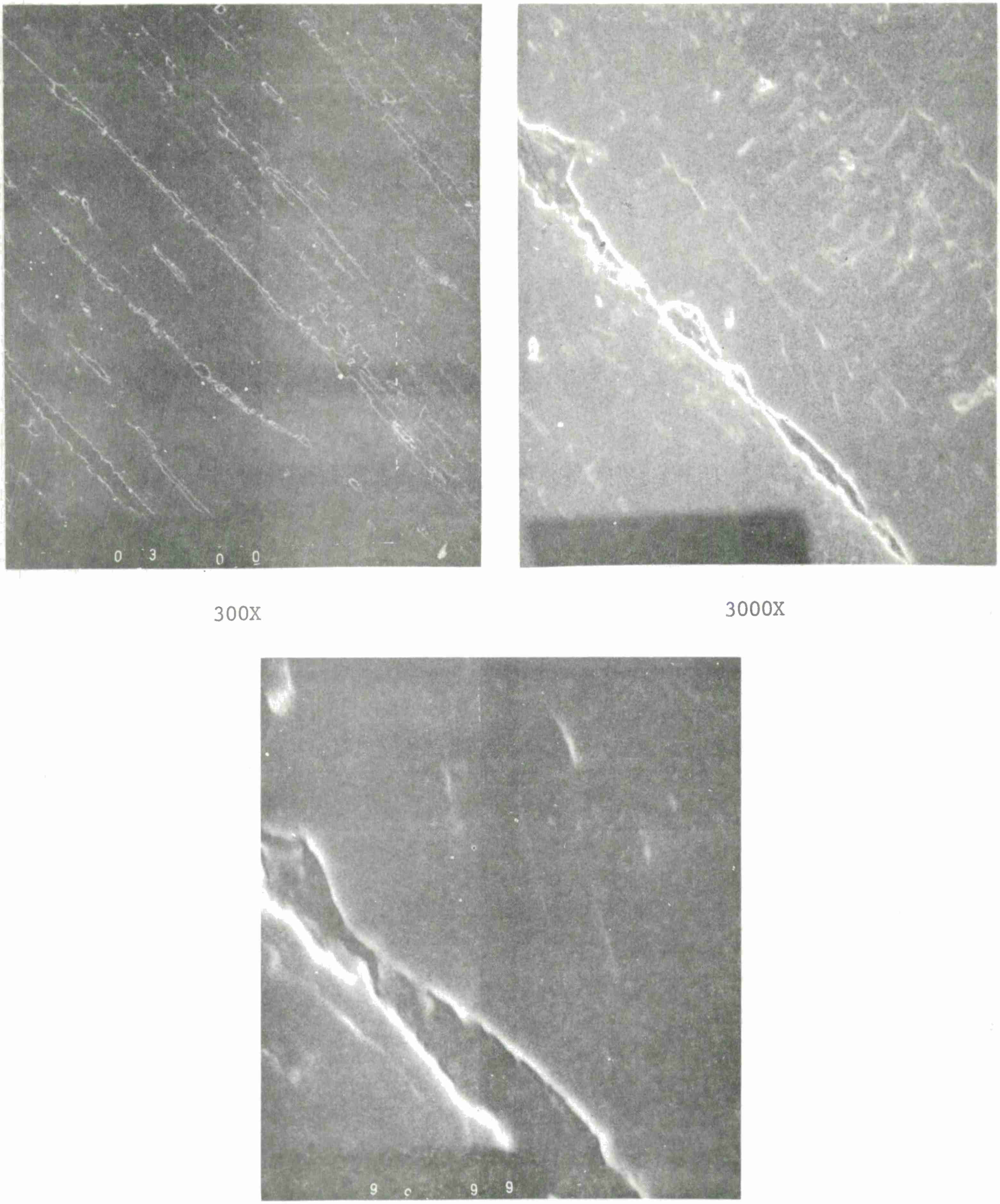


50 abrasion strokes



100 abrasion strokes

10 abrasion strokes

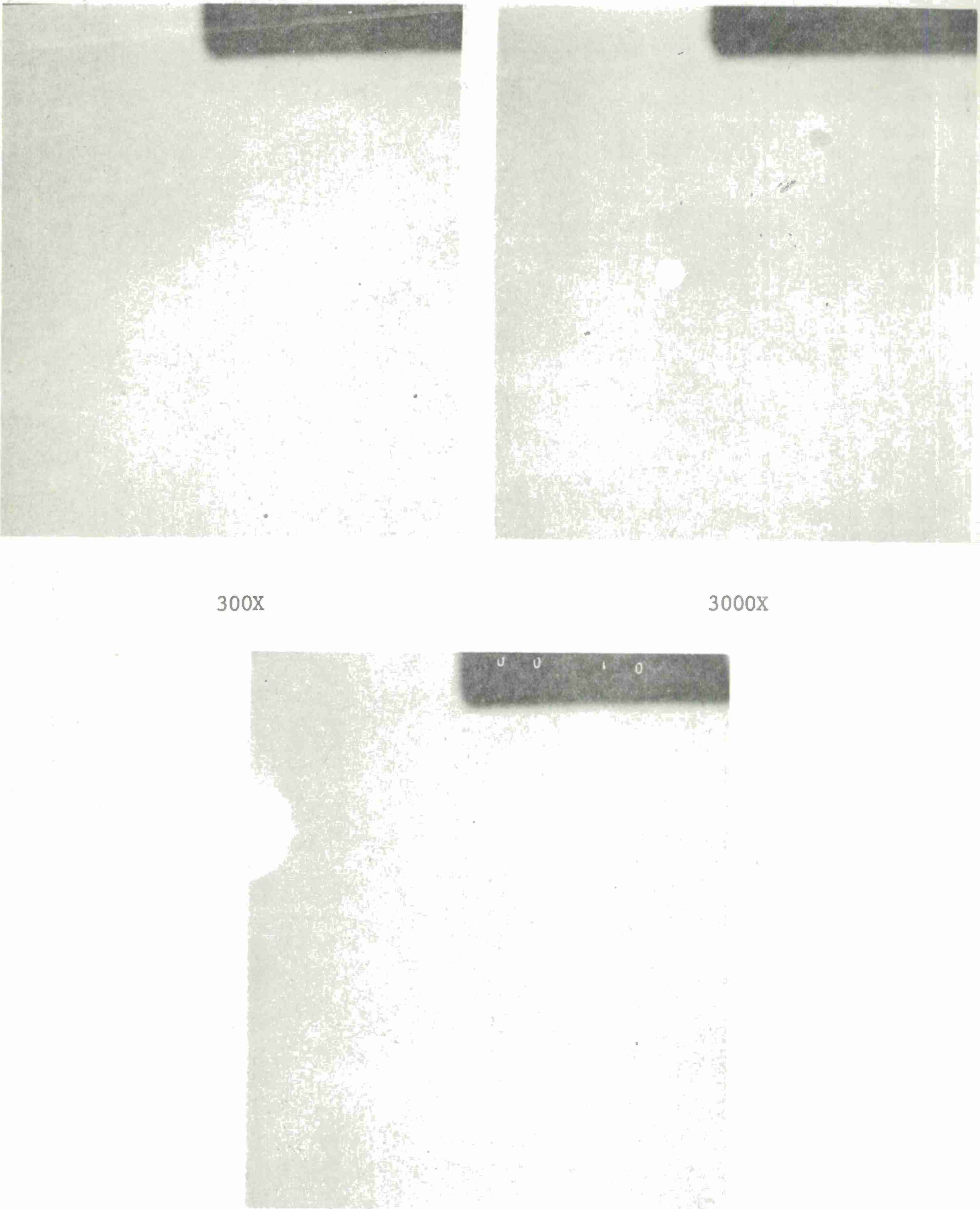


300X

3000X

10,000X

Figure 2. Damage of CR-39 (Uncoated) after Abrasion Test



300X

3000X

10,000X

Figure 3. Electron Microscope Pictures of Glass Coating on CR-39

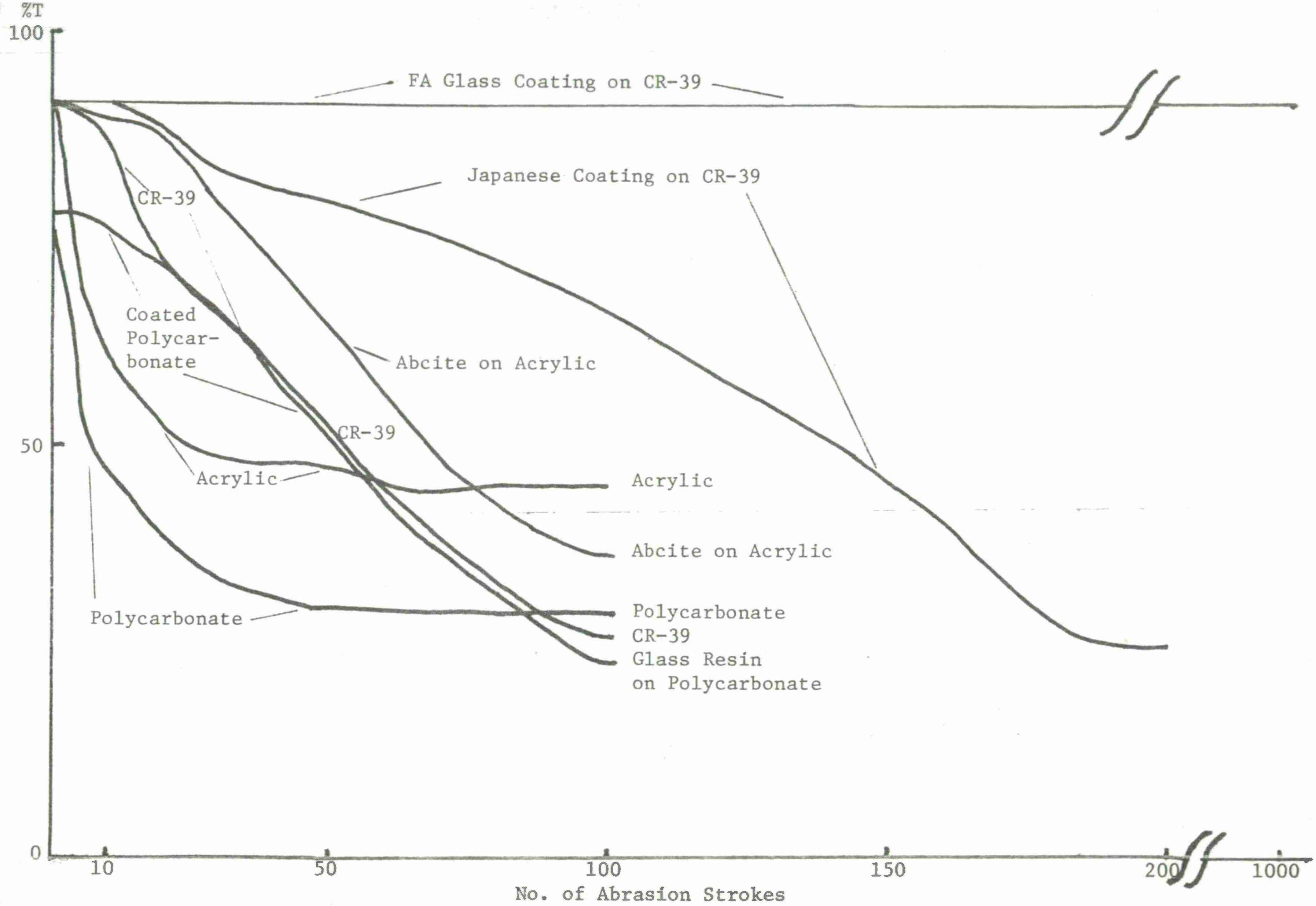


Figure 4. Transmission vs. No. of Abrasion Strokes

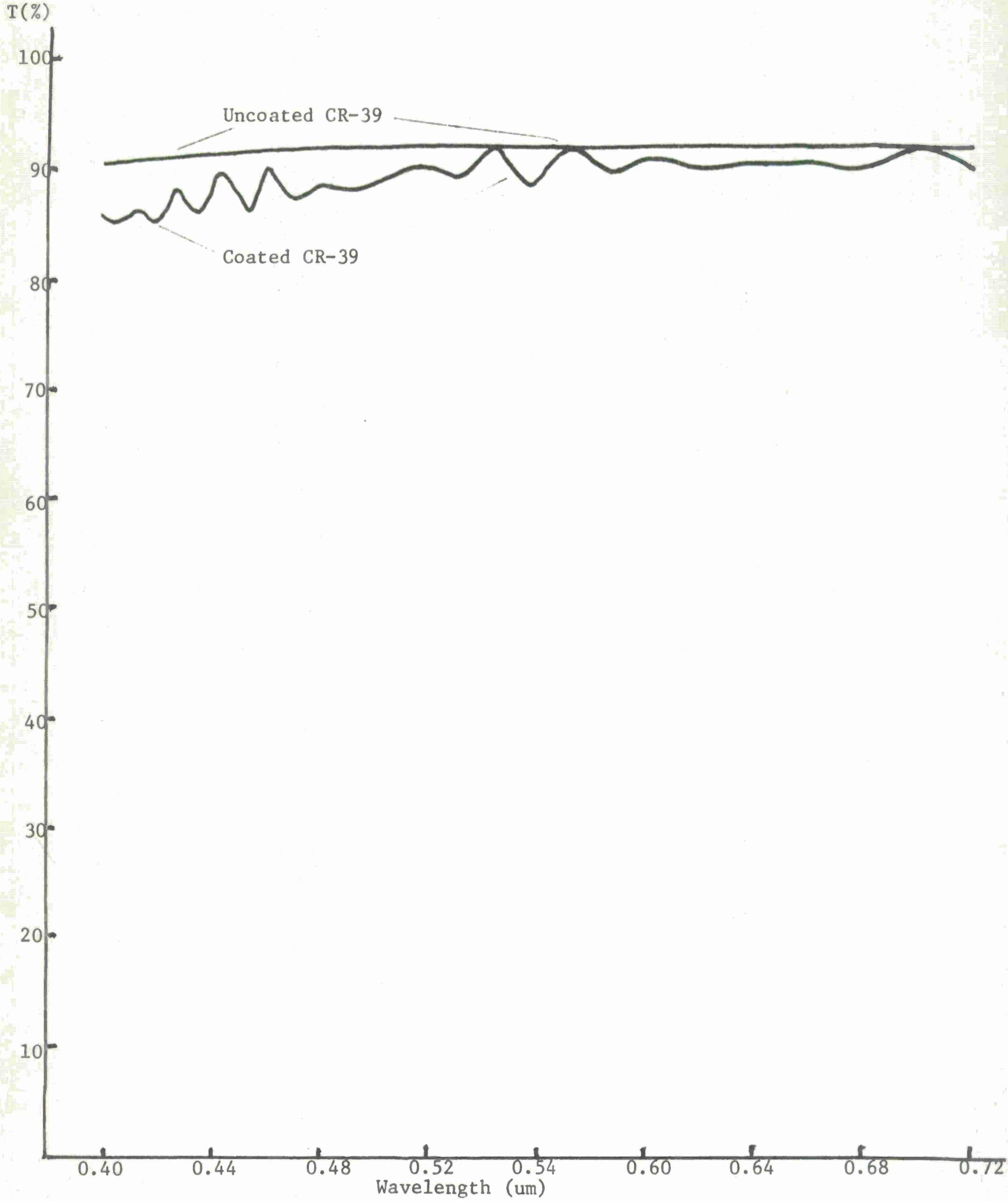


Figure 5. Transmission of Glass Coated CR-39

APPLICATIONS

In order to demonstrate the feasibility of this coating for military and commercial applications, two items were selected for coating: the windows and mirrors for the M-17 tank plastic periscope and a pair of ophthalmic lenses. The M-17 plastic periscope has two glass windows and two glass mirrors bonded to an acrylic periscope block. Under extreme military temperatures some periscopes have a tendency to experience a delamination problem at the glass/acrylic boundary. This is primarily due to a significant difference between the expansion coefficients of the glass and acrylic materials causing a high stress condition. In order to eliminate this problem it was proposed that a glass coated CR-39 window plate be used as a substitute material. Since the expansion coefficients of these two plastic materials are similar, a very low stress condition should be present at the cemented boundary. The size of the mirrors and windows to be coated is approximately 3 x 8 x 1/8 inch thick. The mirrors were coated with a chromium underlayer followed by an aluminum coating and finally a half-wave coating of the glass material. Coating the windows presented some difficulty due to CR-39 absorption of the radiant energy from the electron beam gun heated glass source. Some warping of the thin plastic plate was experienced. Process modifications had to be developed to reduce the warpage to an acceptable level. It should be noted that this plastic plate was thin enough to be flexed easily. The windows and mirrors were bonded to the acrylic periscope and are presently undergoing field tests. A pair of CR-39 ophthalmic lens blanks were coated on both sides with the glass coating and subsequently cut to size for framing. No damage or resolution change was observed from either the coating process or cutting operation. Illustrated in Figure 6 are some of the items coated. In the foreground is the tank periscope window followed by the ophthalmic lenses and watch crystal and the last item is the tank periscope mirror.

Other applications that seem prime candidates for the glass protective coating include: instrument cover plates e.g. aircraft instruments, compasses, watch covers. Also, windows and aircraft windshields that experience abrasive damage from wiper blades should be improved considerably if treated with this process.

CONCLUSIONS

Initial laboratory evaluation tests of the glass coated CR-39 plastic indicate that it can function in a military environment and enhance the abrasion resistant properties of the plastic surface considerably.

Special care must be taken when coating samples whose thicknesses are thin enough to be flexed easily. Warping could result from the plastic absorption of radiant energy from the electron beam source.

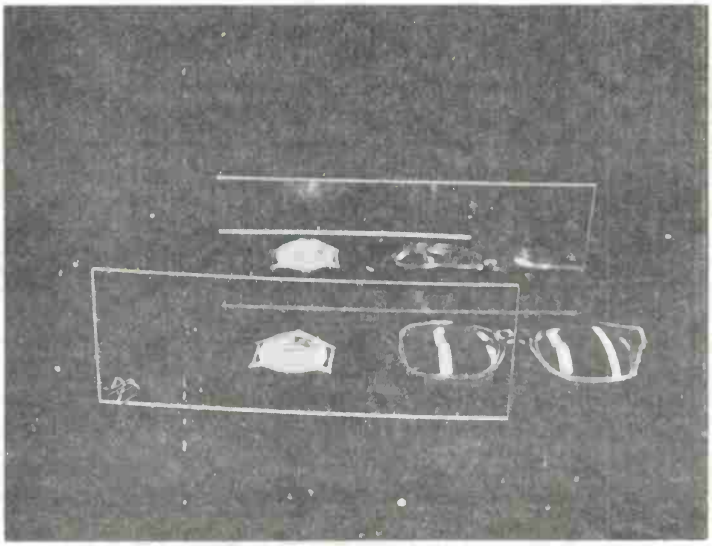


Figure 6. Glass Coated Plastics

Both high transmission and resolution of the plastic component have been maintained.

The use of plastic as a substitute material for glass reduces the weight by approximately one-half as well as provides a safer environment for its use.

Cost savings may also be realized on plastic items that presently have short-life cycles due to their poor abrasion resistant properties.

RECOMMENDATIONS

Due to some enhancement of the adherence properties of films on both acrylic and polycarbonate materials using the sputtering approach it is very probable that additional studies on the surface stabilization of these plastics would result in durability characteristics similar to the glass coated CR-39 plastic.

It is recommended that serious consideration be given to the implementation of this protective coating to applicable military components.

APPENDIX

Final Report

EVALUATION OF SPUTTERING A PROTECTIVE COATING ON PLASTIC COMPONENTS

CONTRACT NO. 300A00738

to

Frankford Arsenal
Philadelphia, PA

April 1974

BATTELLE MEMORIAL INSTITUTE
PACIFIC NORTHWEST LABORATORIES
P. O. Box 999
Richland, Washington 99352

EVALUATION OF SPUTTERING AS A PROCESS TO DEPOSIT A PROTECTIVE COATING ON PLASTIC COMPONENTS

Contract No. 300A00738

to

Frankford Arsenal

INTRODUCTION

Plastics and acrylics are used for many optical components under various conditions. One of the problems in utilizing these materials is that they scratch easily degrading the component and many times making it unusable. In an effort to minimize damage due to scratching, sputtered deposition was evaluated as a means to deposit protective coatings onto plastics. The following materials were evaluated: SiO_2, ZrO_2 and Al_2O_3. Three different methods of deposition were evaluated: diode sputtering, electron beam evaporation and triode sputtering. The basic problems experienced with diode sputtering was excessive heating of substrate due to ion bombardment. The diode sputtering-electron beam deposition provided highly adhering films on the plastic but they had questionable scratch resistance. The triode sputtering did deposit good film but again the scratch resistance was poor. The net results of all of these tests indicated that any surface protection was poor. It was very difficult to determine whether the scratch resistance had been improved over the basic material. It appeared that the deposited films had the characteristics of hard dielectrics but the soft underlayer allowed the hard deposit to easily be scratched.

Diode Sputtering

The diode sputtering system used was MRC Model SEM-8620. This unit has a J-etch design which provides a bias control of the power level to either the target, normally the upper unit, or the substrate holder, normally the lower unit. This arrangement allows sputter cleaning of the substrate prior to deposition and sputter bias to the substrate for control of film properties. One of the drawbacks of this system is that the substrate does receive heavy electron bombardment and some ion bombardment. The net effect to the plastic substrate was overheating. This presented considerable problems in depositing on to the plastic substrates. Extremely low power levels had to be used in order not to damage the substrates due to heating. In addition to this, it appeared that the heavy electron bombardment caused discoloration of the plastic substrates. Efforts to deposit silicon dioxide and zirconium dioxide resulted in highly absorbing film on the substrate. It was difficult to differentiate if it was the films that had a high absorption character to them or if the plastic dielectric interface was the area of high absorption. It was noted that by decreasing the power level the degree of absorption was greatly reduced. At power levels on the order of 50 watts the absorption was quite low and almost acceptable. In evaluating aluminum oxide the absorption was controllable at the low power levels. A total of 30 runs utilizing the diode sputtering method were made. Twenty-four of these runs were done utilizing diode sputtering alone. Six of these utilized diode sputtering to deposit a very thin layer to achieve good adherence to the substrate and then deposition by electron beam evaporation onto the substrate. In the six runs in which the electron beam evaporation was utilized the material in all cases was aluminum oxide.

Deposition Parameters

The substrates were cleaned utilizing a detergent soap dried and inserted into the chamber. After the system was pumped down to a low 10^{-5} torr range the system was backfilled with argon and oxygen. To assure a clean substrate the substrates were sputter-etched for one minute at 50 watts. The system was then set with a small bias (voltage) on the substrate and depending upon the run the power varied from 50 watts to 300 watts. The deposition time varied from one minute to 30 minutes. Table I summarizes the deposition parameters. Utilizing the above parameters under no conditions did the silicon dioxide depositions result in an acceptable film. The resultant film was always an absorption level but it did indeed decrease with the power level showing the least absorption for the 50 watt runs. The deposition parameters for aluminum oxide were basically the same. It was found that power levels of 50-100 watts utilizing aluminum oxide did result in acceptable films from an absorption standpoint.

Thermal Heating Evaluation

In an effort to determine if the thermal heating was a problem causing high absorption for the silicon dioxide depositions, samples of Cr-39 and the acrylics received from the Arsenal were placed into an oven for evaluation. The substrates were cycled by 50°F steps in a temperature controlled oven from room temperature of 400°F. During each step the substrates were evaluated to determine if surface had been affected and discoloration could be observed. No discoloration was observed on the surface during any of the tests. From this it is concluded that there must be some type of a chemical interaction due to the high electron bombardment during deposition. Based on this information and our past experience with triode sputtering we felt that this method held promise. Basically, triode sputtering does not bombard the substrates with high electron energies or ion bombardment. The material is sputtered from a central target and then is deposited on to the substrates. Normally with triode sputtering, high adherence is achieved with controlled heat at the substrate. See Appendix A for details on Triode Sputtering Operation.

Triode Sputter

Equipment used for this evaluation was a Balzer Sputron II. This work was performed at the Balzers headquarters in Santa Ana, California. A total of 10 runs were made varying the various parameters. Basically, what it involved in varying the parameters on the sputron is the power level or sputtering current. Table II summarizes the parameters used. Reactive sputtering was used in this experiment. The target material was aluminum. Oxygen was back-bled into the system oxidizing the aluminum resulting in aluminum oxide deposit on the substrates. At no time was discoloration or absorptive deposit observed in the deposited films. From the limited

Table I. Diode Sputtering Parameters

System pressure $\leq 5 \times 10^{-5}$ torr

Backfill - 1μO_2 the $\pm$ 06.5μ with Ar

Sputter pressure 6.5μ

Sputter etch 1-3 min.

Forward power 50-300 watts

Reflected power ∿0

Bias voltage 0-50V

Deposition time 1-30 min.

Table II. Triode Sputtering Parameters

Pressure 8×10^{-4} Ar then $1 \times 10^{-3} O_2$

Sputter current $1^{-1.2}$A

Sputter voltage 850 to 1200

Filament current 70A

Arc current 18-30-A

Arc voltage 50-60V

experimentation that was done it appeared that a sputtering current of 1.2 amps and an oxygen back-bleed of 1.05×10^{-3} torr oxygen was utilized. The argon pressure in the chamber was 8×10^{-4}. One of the advantages of the triode sputtering is that lower chamber pressures can be used. The resultant films had high adherence and acceptable thicknesses but poor damage resistance. It appeared that the same results which had been experienced with diode sputtering were still present, namely, that the surface of the dielectric surfaces appeared to be hard, but as pressure was applied to them the soft underlying surface would deform resulting in damage to deposited film. Sputtering time varied from 10 minutes to 60 minutes. Damage resistance of these substrates was evaluated also at Frankford Arsenal during one of my visits. And as was witnessed by John Walls the damage resistance appeared to be about the same as the uncoated substrates.

Summary

From the limited work done under this program it appears that sputtering does provide a mechanism for adherence of dielectric films on plastics. It does not provide the damage resistance necessary to protect the plastic from damage due to handling and normal use of optical components.

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