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THIN FILM RESEARCH AND DEVELOPMENT

by

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FOREWORD

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ABSTRACT

The objective of this effort was to investigate materials and techniques for applying antireflectance coatings having the properties of good adhesion and scratch resistance to acrylic plastics. The acrylic plastic used in helicopter windshields has a high degree of reflectivity of the impinging sun's light rays. This is a major threat to helicopter survivability since these rays are readily observed by enemy ground personnel.

CONTENTS

<i>Section</i>	<i>Title</i>	<i>Page</i>
	FOREWORD	iii
	ABSTRACT	v
1	INTRODUCTION	1-1
2	ANTIREFLECTION COAT	2-1
	2.1 Antireflection Coatings in General	2-1
	2.2 Single Layer Coatings.	2-1
	2.3 Multilayer Coatings	2-2
	2.4 Materials and Techniques	2-5
	2.4.1 Floating Film on Water.	2-5
	2.4.2 Chemical Disposition of TiO ₂	2-7
3	GLOW DISCHARGE POLYMERIZATION	3-1
	3.1 Technique.	3-1
	3.2 Glow Discharge Experiments	3-1
	3.2.1 Film Thickness Measurements	3-4
	3.2.2 Glow Discharge Materials	3-6
4	RESULTS AND DISCUSSION.	4-1
5	CONCLUSIONS AND RECOMMENDATIONS	5-1
6	REFERENCES.	6-1

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1. INTRODUCTION

This report describes the effort to investigate materials and techniques for applying antireflectance films to acrylic plastic substrates. Acrylic plastic is used in helicopter windshields and results in a high degree of reflectivity of the impinging sun's light rays. This is a major threat to helicopter survivability since these reflected rays are readily observed by enemy ground observers.

Much work has been done in the field of thin film antireflection coatings for optical systems. These coatings have been restricted to materials which lend themselves to the techniques of vacuum deposition technology. These techniques are costly and time consuming and are not applicable for rapid field repair of large surfaces such as helicopter windshields.

Thin films have been successfully produced as reverse osmosis membranes for the desalination of sea water using a technique of floating films on a water surface.¹ Film thicknesses of 1000 to 5000 Å can be produced with materials having lower refractive indices than the acrylics. This report describes the application of these films as antireflection coatings.

Thin films (500 Å) of TiO_2 have also been successfully coated on contact lenses.² TiO_2 was applied to acrylic during this program and evaluated as a material for multi-layer antireflection coatings.

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2. ANTIREFLECTION COAT

2.1 ANTIREFLECTION COATINGS IN GENERAL

It has been well established in the literature that an effective single layer antireflection coating to produce zero reflection must meet the following criteria for any given wavelength of normal incident light:

$$n_f = \sqrt{n_o n_s} \quad \text{and} \quad d_f = \frac{\lambda}{4}$$

where:

- n_f = Refractive index of film
- n_o = Refractive index of surrounding medium
- n_s = Refractive index of transparent substrate
- d_f = Film thickness
- λ = Wavelength of light

2.2 SINGLE LAYER COATINGS

In order to meet these criteria for an acrylic plastic substrate of refractive index 1.52, a film of refractive index 1.23 and thickness 1250 Å would be required for light of wavelength 5000 Å. While it is a relatively easy chore to obtain required film thickness, we are quite limited in obtaining coatings with refractive indices lower than 1.338. Table 1 gives a list of candidate materials which can be deposited on acrylic substrates using methods other than vacuum deposition. It illustrates the limitations imposed upon us for low refractive index materials.

We can calculate the total reflectance of material light for a single layer coating (thickness = $\frac{\lambda}{4}$) by:

$$r_{\lambda/4} = \left(\frac{n_o n_s - n_f^2}{n_o n_s + n_f^2} \right)^2$$

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This equation gives us the percent light reflected for normal incidence, and shows that we must content ourselves with applying coatings to give minimum reflections.

The lowest refractive index material in Table 2-1 is FEP with $n = 1.338$. Choosing a quarter-wave layer of this material on an acrylic substrate, we find the reflectance minimum to be 0.6% at 5000 Å wavelength. This value is one-half that found for Magnesium Fluoride ($n = 1.38$) @ 5000 Å wavelength.

The deposition of FEP as a thin film has not been reported, but this material should be investigated. Thin films of polyvinylidene fluoride (Kynar) were successfully deposited using polymer solutions cast on water. This material has an index of refraction $n = 1.42$. This gives us a reflectance minimum of 1.97% at 5000 Å. This represents a little more than half that achieved for the acrylic alone (4.7%).

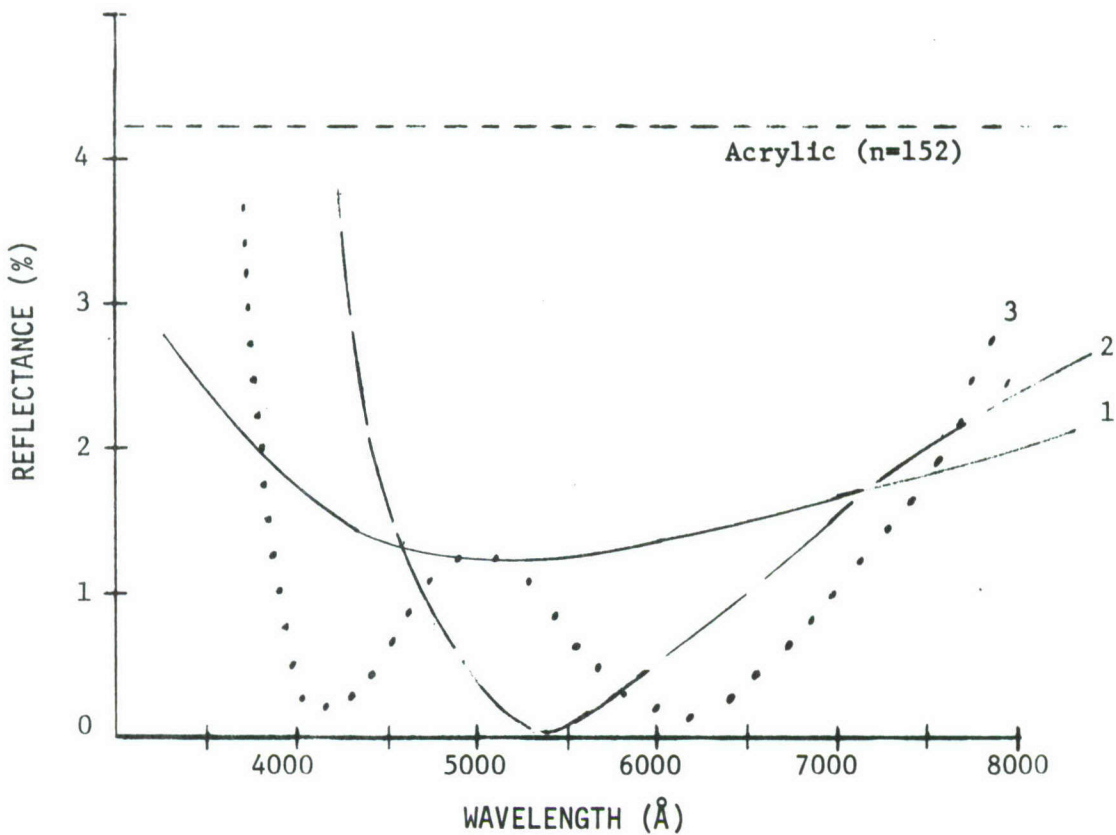
2.3 MULTILAYER COATINGS

Since we are after minimum reflections over a visual spectrum range of 3500 Å to 7500 Å, single layer coatings appear to be inadequate. The reflectance curve rises sharply for single coatings on either side of the 5000 Å median.

An effective method of lowering and/or broadening the minimum reflectance range has been through the use of multilayer thin film coatings. Thelen³ has reported refractive index values for two layer systems that produce zero narrow reflectance bands and wide minimum reflectance bands. The refractive index of these materials approximate candidate materials chosen by us for this program; i.e., Kynar ($n = 1.42$) and TiO_2 ($n = 2.30$). Figure 2-1 illustrates Thelen's bands. The coating producing zero reflectance at 5500 Å is a "V" type coating and gives a narrow band reflection reduction between 5000 Å and 7000 Å. This is too narrow for our use. The "V" type coating is produced using quarter-quarter wave film thicknesses. The broadening of the minimum reflectance band is achieved with a "W" type coating which inserts a half wave film between the top low

Table 2-1. Candidate Materials for Acrylic Substrates

Material	n
Acrylic	1.52 ($\sqrt{n} = 1.23$)
Cellulosic	1.50
Epoxy	1.55 - 1.61
FEP	1.338
Polycarbonate	1.586
Polystyrene	1.60
Polyurethane	1.50 - 1.60
Silicone	1.43
Polyvinylchloride	1.52 - 1.55
Polyvinylidenechloride	1.60 - 1.63
Polyvinylidene fluoride	1.42
Titanium Dioxide	2.30



Refractive Index of Substrate $n_s = 1.52$

- Curve (1) Single Layer $n_f = 1.38$ (quarter wave)
- Curve (2) Double Layer $n_{f1} = 1.38$
"V" Type $n_{f2} = 2.30$ (quarter wave)
- Curve (3) Double Layer $n_{f1} = 1.38$ (quarter wave)
"W" Type $n_{f2} = 2.00$ (half wave)

Figure 2-1. Reflectance of Various Coatings

refractive index coating and the substrate (see Figure 2-1).

Three and four layer systems have been shown to be extremely effective in lowering and broadening the reflectance band. One such system as reported by Thelen is shown in Figure 2-1.

While Table 2-1 does not show materials of similar refractive indices as shown in Figure 2-2 a model could be set up to approximate the curve.

2.4 MATERIALS AND TECHNIQUES

Candidate materials for single layer systems were chosen from the classes of polyurethanes and polyvinylidene fluorides since these materials readily form into thin films using the polymer solution on water technique. Titanium Dioxide was chosen for its ability to be chemically deposited onto the acrylic substrate and as a candidate for multilayer systems. The best film forming materials and recommended solvents are shown in Table 2-2:

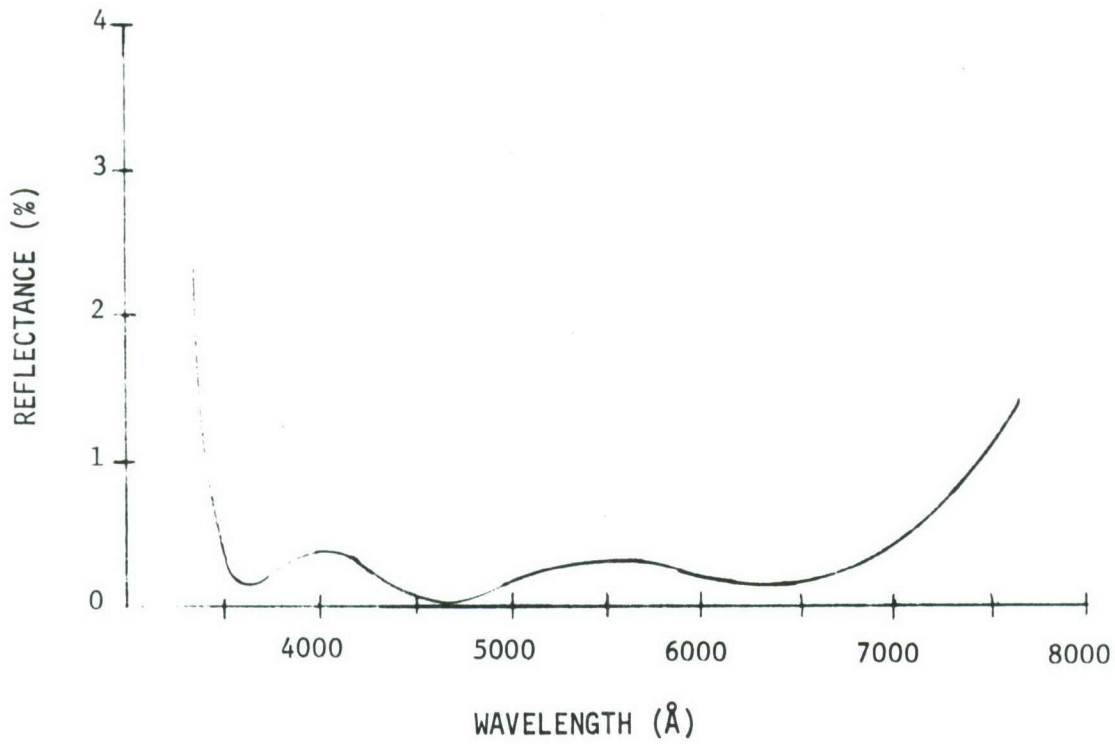
Table 2-2

Material	Solvent	n	Film Thickness (Å)
Estane 5710 (urethane)	Cyclohexanone	1.50	1000→ 5000
Kynar (vinylidene fluoride)	Cyclohexanone	1.42	1000→ 5000
TPT (TiO ₂)	Hexane	2.30	500→ 1000

2.4.1 Floating Film on Water

Methods¹ have been previously developed to cast thin films 1000 to 5000 Å thick by spreading a polymer solution on a water surface. These films were used as reverse osmosis membranes for the desalination of seawater. It had been found during this development that generally 10% solutions of resins in solvents not miscible with water gave uniform and relatively pinhole free thin films.

The polymer solution is slowly poured onto the water surface and spread by hand until the appropriate thickness is obtained.



Refractive Index of Substrate $n_s = 1.52$
 Quarter Wave Film Thickness

- (top) $n_{f_1} = 1.38$
- $n_{f_2} = 2.2$
- $n_{f_3} = 2.43$
- $n_{f_4} = 1.887$

Figure 2-2. Reflectance of Four Layer Coatings

To obtain consistent results with these films it was necessary to ensure that the polymer solution and water bath were free of extraneous materials. A number of samples prepared in our casting bath contained impurities and hence gave lower reflectance values due to dispersion errors. We were plagued with this problem throughout the program. Time did not permit resolving the problem.

Acrylic samples were cleaned with mild detergent, thoroughly rinsed, and then inserted into the casting film bath under the formed film lying on the water's surface. The Estane and Kynar films were cast using cyclohexanone as the solvent. The acrylic sample was raised from under the above formed film until it lifted the film off the water's surface and was adhered firmly to it. It had been found from previous experience that better adhesion is obtained when the substrate is completely wetted by the water bath. After removal from the bath, the samples were allowed to dry slowly at room temperature.

2.4.2 Chemical Disposition of TiO_2

The technique employed involves the chemical deposition of thin, transparent titanium oxide films by the hydrolysis of tetraalkyl orthotitanates deposited on the substrate from solution. This process was first developed by DuPont and others to act as an adhesion promoter in the bonding and laminating fields.

The hydrolysis of the tetraalkyl titanate is a step-wise process. The complete hydrolysis of thin films occurs rapidly in air of ordinary moisture content to form a clear coating.

From previous experience it was determined that a dipping process was the most satisfactory. The prime requisites for such a process are humidity control and absolute uniform withdrawal rates; both are necessary for optically clear and uniform coatings.

Acrylic samples to be coated were cleaned with mild detergent and thoroughly rinsed. They were then immersed in a 6% concentration of tetraisopropyl titanate (TPT) in Hexane. Prior studies had shown this

concentration to give optimum results in regard to optical clarity and adhesion.

Again time did not permit optimizing equipment to achieve best results. Coatings which were produced with this method gave cloudy appearances at first, due to improper humidity conditions and withdrawal rates. Subsequent specimens produced had slight fringe patterns developed within the films due to withdrawal problems. These samples, however, were adequate to show the feasibility of such coatings for use with multilayer coatings.

3. GLOW DISCHARGE POLYMERIZATION

3.1 TECHNIQUE

The technique for coating the acrylic slides with a glow discharge polymer consists of evacuating the discharge cell down to 5 microns or less, filling the cell with monomer vapor to a pressure of 1 mm Hg and applying an alternating voltage at 20 KHz to the electrodes to initiate the glow. Then the voltage is lowered to the minimum value required to sustain the glow and the vapor is pumped through continuously.

In a classic glow discharge, the polymer deposits directly on the electrodes. By using screen electrodes and mounting the acrylic slides behind the screens, it is possible to collect the polymer directly on the slides. After the slides are coated on one side, they are turned around in the electrode assembly and coated on the second side.

3.2 GLOW DISCHARGE EXPERIMENTS

A high vacuum glassware system for glow discharge experiments was assembled and consisted of a discharge cell, thermocouple gauge, cold traps, diffusion pump, oil pump and external power supply. The discharge cell, shown in Figure 3-1, had an inlet for introducing monomer vapor, an electrode assembly, pressure gauge and two press seal Kovar leads connected to a Precision Electronics audio frequency amplifier and Hewlett Packard audio oscillator. The voltage was measured with a Simpson meter and the current by a Sensitive Research radio frequency milliammeter. The electrode assembly, shown in Figure 3-2, was constructed from blocks of teflon and acrylic plastic. Two parallel screen electrodes, 2.5 X 3 in. were set 1 cm apart. Acrylic plastic slides, 2 X 3 X 1/16 in. were placed 1/8 in. behind the electrodes by means of a teflon spacer.

The initial experiments were conducted using copper screen electrodes

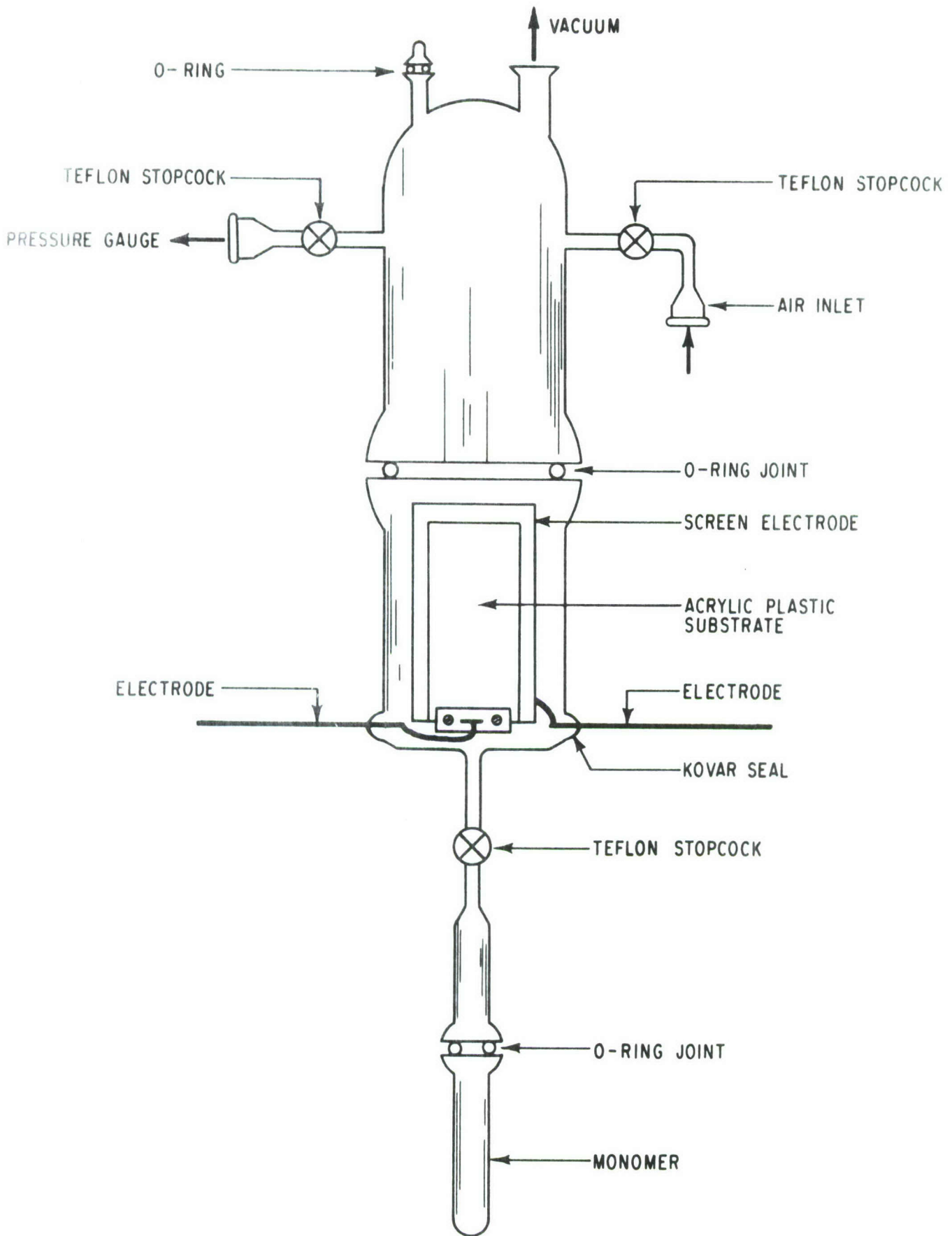


Figure 3-1. Glow Discharge Cell

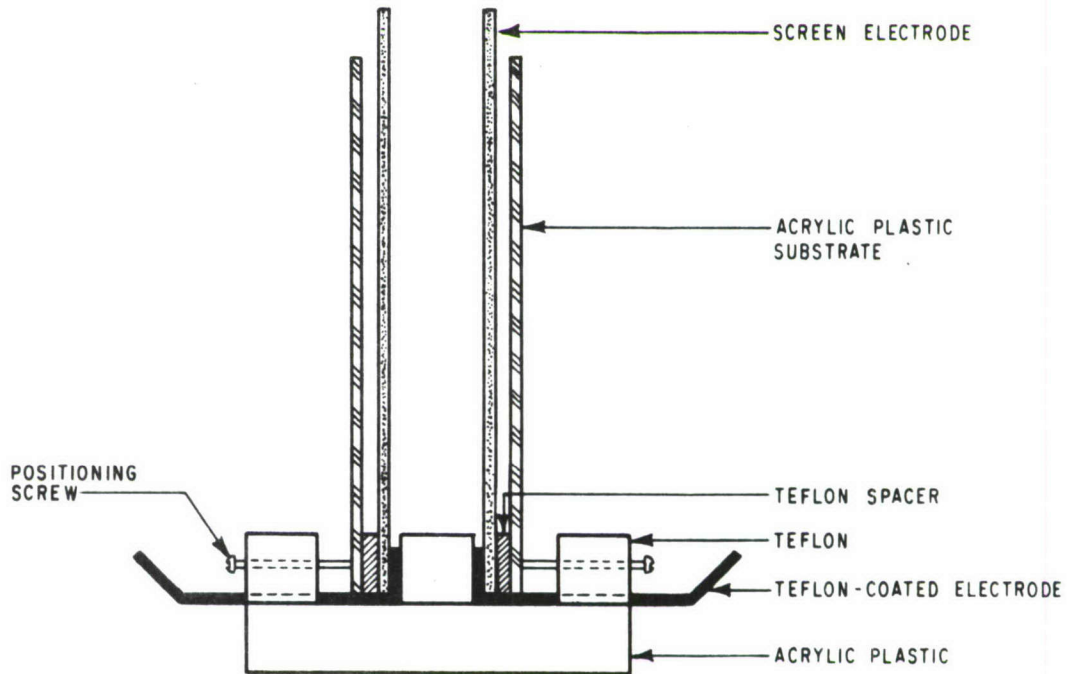


Figure 3-2. Electrode Assembly for Acrylic Slides

and monomers such as perfluoroalkane-70 and perfluoroalkane-115. The uniformity of the coating depended on the gauge of the screens and their precise parallel alignment with respect to the slides. Copper screens proved to be too flexible.

Some experiments were carried out using stainless steel screens. They drew too heavy a current, producing a heat build-up which caused warping of the slides. The optimum choice was brass. By using wide mesh, stiff brass screen electrodes, slightly larger in area than the substrates, it was possible to coat the slides uniformly without damaging temperature rises. Subsequent experiments were directed toward preparing films 1500 Å in thickness by varying the duration of the glow discharge. Discharges longer than one minute produced cloudy or highly colored films. Thin, colorless films were obtained with discharges of 30-60 seconds' duration.

Usually, the lower portion of the acrylic slide was masked off with tape during the glow discharge experiment, then later removed so as to have a sharp boundary for thickness measurements.

3.2.1 Film Thickness Measurements

Film thicknesses were determined by optical interferometry. In this procedure, the substrate resting on a levelling stage, is examined under a microscope fitted with a Watson 8 mm interference objective with an illuminating tube for a monochromatic light source, in this case, a sodium lamp. Variations in the vertical height of the specimen will produce a lateral deflection in the fringe pattern. The fringes are shifted by one fringe for each half wavelength of height. For sodium light, the wavelength is 0.59 microns.

The fringe patterns of several samples of glow discharge films on acrylic slides were photographed. The thickness of the films was determined by measuring the fringe shift on the photographs. The data for the best films coated on both sides is reported in Table 3-1.

Thin films from the glow discharge polymerization of perfluoro-

Table 3-1. Thickness Measurements of Glow Discharge Films on Acrylic Slides

Sample No.	Side A	Side B
1	2600 Å	1850 Å
2	1410 Å	1410 Å
3	2100 Å	1650 Å
4	955 Å	1360 Å

alkane-115 were also collected on polished NaCl or KBr plates to enable spectroscopic examination of these films for the purpose of identification and determination of their refractive index.

The infrared spectrum of one of these films, shown in Figure 3-3, exhibits a single broad band at 1210 cm^{-1} indicative of C-F stretching. There is no appreciable absorption at frequencies higher than 1350 cm^{-1} which is a feature of fully fluorinated hydrocarbons.⁴

Interference fringes can often be observed in the infrared spectrum of thin films. The number of fringes between the first and last maximum (or minimum) counted gives rise to a formula from which the refractive index can be calculated if the thickness is known or can be determined by another technique.⁵ Preliminary work in this area indicated that the glow discharge films were too thin for this application so this approach was abandoned.

3.2.2 Glow Discharge Materials

The following materials were used as received:

1. Perfluoroalkane-70 (boiling range $70-80^{\circ}\text{ C}$, n_D^{20} 1.2796, PCR 01-12310-04);
2. Perfluoroalkane-115 (boiling range $115-195^{\circ}\text{ C}$, n_D^{20} 1.3197, PCR 01-12320-01);
3. Perfluoroalkane-195 (boiling range $195-225^{\circ}\text{ C}$, n_D^{20} 1.3220, PCR 01-12330-07);
4. Acrylic plastic, Rohm and Haas.

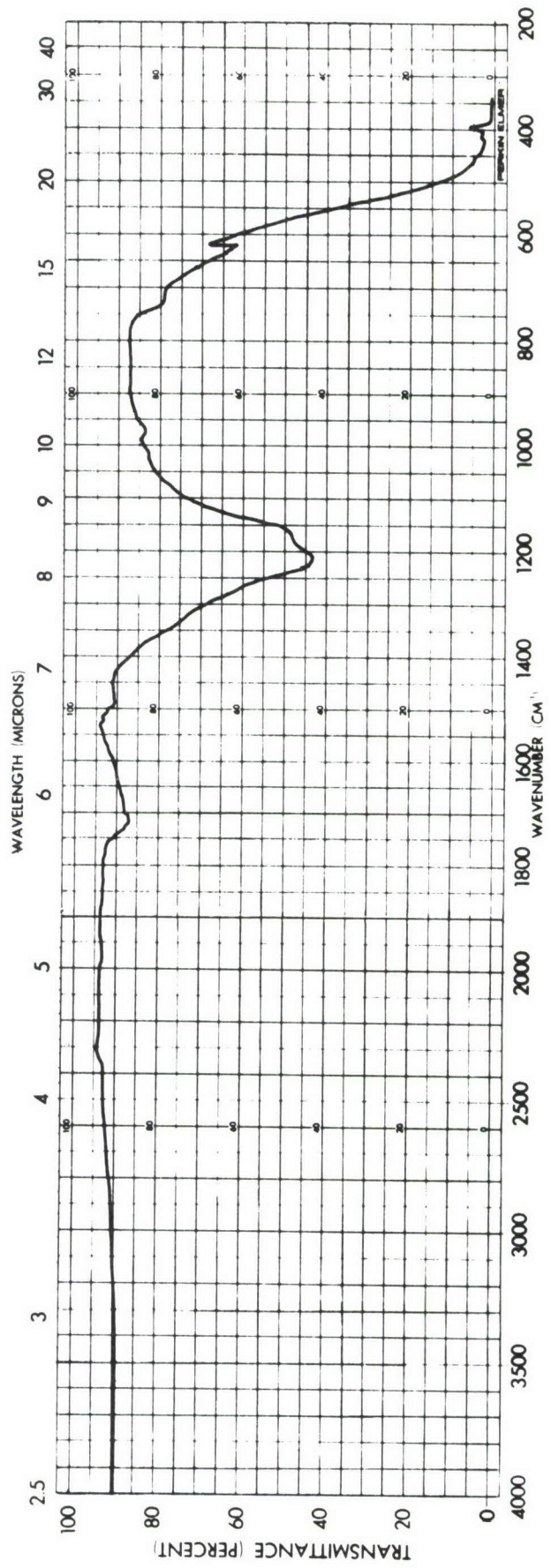


Figure 3-3. Infrared Spectrum of G. D. Polymer of Perfluoroalkane - 115

4. RESULTS AND DISCUSSION

Time and funds for this program were exhausted before an adequate number of samples could be prepared and tested to determine the acceptability of present process techniques and materials.

From the samples that were prepared, however, we were able to determine:

- Single layered samples of Kynar ($n = 1.42$) exhibited reflectance values of 2% at normal incidence and withstood the eraser and adhesion tests;
- Single layered samples of Estane ($n = 1.50$) did not produce adequate bond strength to the acrylic substrate;
- Both films of Kynar and Estane exhibited dispersion losses which were due to "dirt" in the casting system;
- The TiO_2 coatings proved to have excellent abrasion and scratch resistance as well as excellent adhesive strength to the substrate. The fringe pattern interference, however, prevents the production of a two layer system.

The results of tests on these samples were inconclusive.

Most of the effort in the glow discharge (G.D.) portion was directed toward 1) refining the design of the discharge cell and electrode assembly so that the cell could accommodate 2" X 3" acrylic slides and monomer vapor would flow through the electrode gap, and 2) determining the type of screen electrode which would allow a uniform film to collect on the acrylic slides. The designs shown in Figures 3-1 and 3-2 are the result of that effort. The best electrode material was found to be stiff brass screen since the use of other materials resulted in warping of the acrylic slides from elevated electrode temperatures or the formation of non-uniform films. Uniform, clear, colorless films with thicknesses ranging from 955 Å to 2600 Å were deposited on both sides of 2" X 3" acrylic slides and were shown by infrared spectroscopy to contain only C-F bands.

Attempts to determine the refractive index of these films were unsuccessful. However, the refractive index of G.D. films cannot necessarily be predicted from the refractive index of the starting material because it is known that G.D. polymers differ chemically and physically from the polymers produced by other chemical procedures from the same monomer. An example of this is teflon.⁶ The films deposited on acrylics by the glow discharge of perfluoroalkanes did not have acceptable adhesion or scratch-resistant characteristics. Other monomers subjected to this process might meet these criteria such as other fluorinated chemicals or metallorganics.

Recently a group at Bell Telephone Laboratories reported the preparation of optically clear thin polymer films from vinyltrimethylsilane and hexamethyldisiloxane by glow discharge techniques and they suggest that it may be possible to vary the refractive index of a film after it has been deposited by heating it in an oxygen atmosphere.⁷

5. CONCLUSIONS AND RECOMMENDATIONS

Previous work on thin films to reduce reflection from transparent substrates indicates that a system is needed to quickly and inexpensively apply these coatings. The results of the work on this program indicate that means of refinement be used to produce good "dirt"-free films. This may necessitate the use of clean room conditions for their production. Also an apparatus for absolute uniform withdrawal of samples being chemically deposited with TiO_2 is required.

A further investigation into the deposition of FEP ($n = 1.338$) films is required. There is presently an FEP film (2 mil thick) being manufactured as a pressure sensitive adhesive which utilizes a clear silicone ($n = 1.43$) as the adhesive. This may prove to be the basis for a multi-layered system. Preliminary results with this material gave high dispersion losses, therefore interfering with light transmission. Methods, however, could possibly be developed to insure clear flat surfaces.

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