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INTEGRATED HELMET-MOUNTED SIGHT/DISPLAY PROGRAM:
Coating Technology

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JULY 1979

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FOR THE COMMANDER

CHARLES BATES, JR.
Chief
Human Engineering Division
Aerospace Medical Research Laboratory

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# CONTENTS

<table>
<thead>
<tr>
<th>SECTION I</th>
<th>INTRODUCTION</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.1 Purpose of Document</td>
<td>1-1</td>
<td></td>
</tr>
<tr>
<td>1.2 Statement of Work</td>
<td>1-1</td>
<td></td>
</tr>
</tbody>
</table>
| 1.3 Program Objective  
  Major Tasks | 1-1 |

<table>
<thead>
<tr>
<th>SECTION II</th>
<th>SUMMARY OF TASK RESULTS</th>
<th>2-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.1 Optics on Plastics</td>
<td>2-1</td>
<td></td>
</tr>
</tbody>
</table>
| 2.2 Variable Density  
  State-of-the-Art Technology Survey  
  Variable Density Concepts  
  Vendor Survey -- Variable Density | 2-5 |
| 2.3 Durability and Abrasion  
  Vendor Evaluation  
  In-House Laboratory Development and Testing | 2-11 |
| 2.4 High Absorption  
  Substrate Absorption  
  Beamsplitter Absorption | 2-15 |

<table>
<thead>
<tr>
<th>SECTION III</th>
<th>CONCLUSIONS</th>
<th>3-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>3.1 Optics on Plastics</td>
<td>3-1</td>
<td></td>
</tr>
<tr>
<td>3.2 Variable Density</td>
<td>3-1</td>
<td></td>
</tr>
<tr>
<td>3.3 Durability and Abrasion</td>
<td>3-2</td>
<td></td>
</tr>
<tr>
<td>3.4 High Absorption</td>
<td>3-3</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>SECTION IV</th>
<th>RECOMMENDATIONS FOR CONTINUING WORK</th>
<th>4-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>4.1 Optics on Plastics</td>
<td>4-1</td>
<td></td>
</tr>
<tr>
<td>4.2 Variable Density Visor Development</td>
<td>4-2</td>
<td></td>
</tr>
</tbody>
</table>
| 4.3 Durability, Abrasion Resistance and  
  Environmental Protection | 4-2 |
| 4.4 High Absorption | 4-3 |

<table>
<thead>
<tr>
<th>SECTION V</th>
<th>TECHNICAL DISCUSSION</th>
<th>5-1</th>
</tr>
</thead>
</table>
| 5.1 Optics on Plastics  
  5.1.1 Technical Literature Survey  
  5.1.2 Vendor Survey  
  5.1.3 Laboratory Studies -- Optics on Plastic | 5-1  
  5-6  
  5-6 |
First Generation 5-10
Second Generation 5-11
Third Generation 5-11
Fourth Generation 5-12
Fifth Generation 5-12
Proposed Sixth Generation 5-13

5.2 Variable Density
5.2.1 State-of-the-Art Technology Survey 5-20
5.2.2 General Status of Candidate Approaches 5-20

5.3 Durability and Abrasion Resistance 5-45
5.3.1 Introduction 5-45
5.3.2 Nature of the Overcoating Problem 5-46
5.3.3 In-house Testing and Evaluation of Overcoat Techniques and Materials 5-48

5.4 High Absorption 5-56
5.4.1 Evaporation of "Metal Lux" Materials 5-56
5.4.2 Tinted Plastics for Visors 5-56
5.4.3 Ghost Ratios for Tinted Acrylic 5-58

SECTION VI REFERENCES 6-1

APPENDIX
ATTACHMENT NO. 1 "OPTICS ON PLASTICS--VENDOR SURVEY LETTER"
ATTACHMENT NO. 2 STATUS REPORT--"TRICHRIOCS ON PLASTICS"
ATTACHMENT NO. 3 ABRASION RESISTANT COATINGS FOR PLASTIC OPTICS
ATTACHMENT NO. 4 VISOR HANDLING AND CLEANING INSTRUCTIONS
ATTACHMENT NO. 5 GHOST RATIOS FOR VARIOUS COATING AND TINTED ACRYLICS USED IN IHMS/D VISORS
ILLUSTRATIONS

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-1</td>
<td>Special Techniques are used to Electron-Beam Evaporate Optical Thin Films on Plastics</td>
<td>2-4</td>
</tr>
<tr>
<td>2-2</td>
<td>Cary 14 Spectrophotometer is One of Several Instruments used to Evaluate Variable Density Concepts</td>
<td>2-10</td>
</tr>
<tr>
<td>2-3</td>
<td>Differential Interference Microscopy is used to Evaluate Abrasion Tests of Plastic Materials</td>
<td>2-14</td>
</tr>
<tr>
<td>2-4</td>
<td>Parabolic Dome Formed from High Absorption Tinted Acrylic Material -- Prior to Cutting Out IHMS/D Visor</td>
<td>2-17</td>
</tr>
<tr>
<td>4-1</td>
<td>The Thin Film Laboratory is Completely Equipped to Deposit Thin Films of any Metal, Semiconductor, Glass, or Ceramic</td>
<td>4-4</td>
</tr>
<tr>
<td>5-1</td>
<td>Hemispherical Acrylic Aircraft Transparency Used to House Guidance and Target Designator Equipment</td>
<td>5-3</td>
</tr>
<tr>
<td>5-2</td>
<td>IHMS/D Visor with Inconel/Aluminum Reflecting Spots</td>
<td>5-8</td>
</tr>
<tr>
<td>5-3</td>
<td>Reflectance of Opaque Mirrors on Acrylic Sheet</td>
<td>5-9</td>
</tr>
<tr>
<td>5-4</td>
<td>Optical Monitor Setup Used to Measure Transmission Through the Beamsplitter Spot on the IHMS/D Visor</td>
<td>5-14</td>
</tr>
<tr>
<td>5-5</td>
<td>IHMS/D Visor with Opaque Reflector Spot and Partially Transmitting Metal Filter Spot</td>
<td>5-16</td>
</tr>
<tr>
<td>5-6</td>
<td>IHMS/D Visor Spot Measurement Setup in Thin Film Laboratory</td>
<td>5-17</td>
</tr>
<tr>
<td>5-7</td>
<td>Characteristics of 11-Layer Trichroic Coating on Tinted Acrylic</td>
<td>5-19</td>
</tr>
<tr>
<td>5-8</td>
<td>Cary 14 Transmission Scan of Polarizing Sheet</td>
<td>5-21</td>
</tr>
<tr>
<td>5-9</td>
<td>Polarizing Film as Received, and Stretch Laminated to a CR-39 Plastic Lens</td>
<td>5-22</td>
</tr>
<tr>
<td>5-10</td>
<td>Photochromic Fabrication Techniques</td>
<td>5-26</td>
</tr>
</tbody>
</table>
5-11 Photochromic-Coated Acrylic Paraboloid Showing Moisture Reactions Which Can Occur if the Photochromic is not Overcoated for Protection

5-12 Vacuum Former and Epoxy Mold Used to Form Thin Photochromic-Coated Plastic Sheet

5-13 Photochromic-Coated Sheet with Hard Overcoat -- Formed in Vacuum Former at Too High a Temperature

5-14 Photochromic Sheet with Hard Overcoat -- Formed in Vacuum Former at Proper Temperature

5-15 Vacuum-Formed Photochromic Paraboloid with Moisture Bubbles

5-16 Vacuum-Formed Photochromic Paraboloid with Fewer, Smaller, Moisture Bubbles

5-17 Photochromic Glass Ophthalmic Blank

5-18 Effect of Voltage on Transmission of Electro-Optic Particle Oriented Fluid Test Filter

5-19 Liquid Crystal Numeric Display

5-20 Army XM 73 Rocket Sight Modified for PLZT Variable Reticle Pattern Generator

5-21 Craze Markings on Abcite-Coated Acrylic Sheet Vacuum Formed into Paraboloid

5-22 Low-Energy D-C Ion Sputtering System

5-23 Thermal Evaporation System Used to Evaporate Metal Lux Materials

5-24 Cary 14 Transmission Scans of Various Tinted Acrylics
### TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>5-1</td>
<td>Properties of Markopolo™ MPS-1</td>
<td>5-25</td>
</tr>
<tr>
<td>5-2</td>
<td>Military Specifications Relevant to Visor Coatings</td>
<td>5-55</td>
</tr>
<tr>
<td>5-3</td>
<td>Ghost Ratios for Various Visor Configurations</td>
<td>5-60</td>
</tr>
</tbody>
</table>

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v
SECTION I
INTRODUCTION

1.1 PURPOSE OF DOCUMENT

This document provides an overall summary of work performed between 15 May 1972 and 15 June 1973 on the "Coating Technology" portion of the IHMS/D contract, No. F33615-72-C-0420/PZ0002, Phase II, Paragraph 3.2.3.1.5 of the Revised Technical/Cost Proposal (Ref. 1).

1.2 STATEMENT OF WORK

Phase II was to "encompass the construction of several improved models of the IHMS/D that will serve to define problems, anticipated performances, hardware options, and final system design of the Mark II, Mod 0 (Model 8) IHMS/D."

Paragraph 3.2.3.1.5 of the Statement of Work, entitled "Coating Technology," involves conducting a survey of the state of the art in coatings, and obtaining and evaluating samples of candidate coatings including variable-density filtering for the visor for possible introduction into the Mark II, Mod 0 (Model 8) program.

1.3 PROGRAM OBJECTIVE

The program objective was to investigate new materials, processes, deposition techniques, and parameters required to improve and enhance the performance characteristics of optical coatings for the Model 8 system.
Major Tasks

Optics on plastics - State-of-the-art technology study and vendor survey

Variable density - Vendor survey, state-of-the-art technology study, and laboratory development

Durability and Abrasion - Vendor evaluations, laboratory development and testing

High Absorption - Laboratory coatings development
SECTION II
SUMMARY OF TASK RESULTS

2.1 OPTICS ON PLASTICS

A five-year technical literature survey (1968-1972) was run on the general subject, "Optics on Plastics." Survey results showed that plastic optics are only recently coming into prominence and that most work to date has been concerned with improving plastic materials and developing fabrication techniques for making optical quality plastic substrates.

A letter survey requesting information on technical capabilities to deposit optical films on plastic substrates was sent out to 85 vendors of optical coatings. The vast majority of those responding denied any interest, experience, or capabilities in coating plastic substrates. The few that did respond in the positive indicated either experience or future interest in depositing antireflective films, metal filters, and simple, low-reflectance beamsplitters on plastics. No one advertised or indicated any experience in successfully depositing the more complex multilayer dichroics, trichroics, band-pass filters, or interference filters.

The following laboratory studies were conducted:

- **Adhesion of Thin Films to Acrylic** - Test depositions of many different metals, ceramics, and glasses onto both clear and tinted acrylic showed that Inconel 600 and CeO₂ were the metallic and dielectric films which displayed the greatest adherence to acrylic and which could best be expected to act as adhesive undercoats for optical films.
• Model 7 Reflective Spot and Beamsplitters - Tests showed that thin Inconel/opaque aluminum and thin Inconel/semi-opaque aluminum films could be effectively used as the mirror and beamsplitter spots on Model 7 visors.

• Development of High-Reflectance Opaque Mirrors - To further improve the percent reflectance and physical durability of front surface, opaque mirrors used in the IHMS/D optics train, a special, multilayered materials system composed of Cr/Al/\(\text{MgF}_2/\text{CeO}_2\) was developed and incorporated into some of the later Model 7 units.

• Development of Optical Sensor - A relatively simple but highly-efficient optical sensing system was devised to monitor the optical transmission of beamsplitter films as they were being deposited on acrylic test pieces. The photodiode/light source was subsequently used to monitor the beamsplitter depositions on all Model 7 visors.

• Visor Rework Capability - Techniques were developed to chemically strip and recoat all of the optical coatings which were developed for use on the Model 7 and Model 8 visors.

• Trichroic Test Depositions - Several tests were made to determine difficulties to be expected in depositing high-reflectance trichroic coatings on acrylic and polycarbonate visors. Results of these tests verified the expected adhesion and stress crazing problems that were anticipated, and provided a basis on which recommendations were made to AMRL regarding the need for a Model 8 trichroic development program.
As noted above, several of the thin film coatings, deposition techniques, and fabricating procedures developed during the course of this program were incorporated directly into Model 7 units. The program also offered several important technical directions which will be used on forthcoming Model 8 units.
Figure 2-1. Special Techniques are used to Electron-Beam Evaporate Optical Thin Films on Plastics
2.2 VARIABLE DENSITY

State-of-the-Art Technology Survey

A five-year technical literature survey (1968-1972) was conducted on the general subject, "Variable Density (Transmissivity) Concepts." The survey was long and difficult because of the vast number of key words and potential applications that had to be searched out. Any and all concepts were to be considered, no matter how far-fetched or unusual they might be.

The net result of the survey was a compilation of approaches, the more appropriate of which are listed below.

Variable Density Concepts

The following concepts were examined for their potential use in obtaining an IHMS/D visor with variable density see-through capabilities for day-night applications.

- Dual sunshield (single or variable density)
  - Sun glasses
  - Retractable visor
  - Snap-on visor
  - Louvered sunshield (high-angle cut-off)
  - Conformal sunsheet (tinted Saran)
  - Cross-polarized laminate sheets
  - Graded density filter (linear or circumferential)
Photochromics
- Visor material
- Visor laminate
- Visor coating
- Visor insert (organic, glass)

Electro-optic fluids (herapathite - dipole crystals)
- Visor sandwich
- Visor insert

Liquid crystals (nematic-types oriented liquids)
- Visor sandwich
- Visor insert

Electro-optic ceramics (PLZT)
- Visor insert

Assuming that the simplest approach to obtaining a day-night visor capability is the best approach, then some form of dual sunshield is a leading candidate, followed by photochromics, electro-optic (particle-oriented) fluids, liquid crystals, and finally electro-optic ceramics.

If either visor fabrication or overcoating techniques could be developed, then use of a single visor made of photochromic material appears to be the simplest method of obtaining variable transmissivity which is compatible with current IHMS/D designs.

If sun/cloud or other relatively fast response requirements are imposed on the variable density visor, then the more difficult and complex techniques using electro-optic fluid or liquid crystal sandwich visors will be required.
Use of electro-optic PLZT ceramic inserts, while capable of extremely fast response characteristics, is not viewed as a likely candidate approach for IHMS/D applications unless nuclear flash requirements are imposed.

Vendor Survey -- Variable Density

The vendor survey was run using telephone and letter requests to obtain technical literature and information relating to capabilities and approaches to making variable density optics. Results of the survey revealed the following generalized information:

**Dual Sunshield** -- Many manufacturers produce a wide variety of tinted optics and special tinted materials which could be used in making a single-density dual sunshield. Samples of various tinted materials, "loudered" light control sheet, and polarizing material were acquired and examined.

**Photochromics** -- Only one manufacturer of photochromic glass was found and consulted. Unfortunately, only glass disks (used for ophthalmic lenses) are available and then only in relatively small diameters.

Two manufacturers of photochromic organic materials were also contacted. Both would provide either coating liquids or as-coated cellulose acetate butyrate. One vendor was especially willing to sell material and to contract for custom development work on coating color and response characteristics, and development of coatings on acrylics.

Sample quantities of both the coating liquid and photochromic-coated sheet stock were obtained for in-house testing. Flow-coating tests on parabolic acrylic domes revealed the problems with coating uniformity and dusting defects that would have to be contended with to obtain high-quality optical coatings. Vacuum forming tests were also run on the photochromic sheet
stock, the test results of which served to illustrate the criticality of such parameters as time, temperature, moisture content, and severity of radius.

**Electro-Optic (Particle-Oriented) Fluids** -- As of mid-1971, two vendors manufactured and sold particle-oriented fluids. One of the vendors was willing to sell the material outright--the other required a license agreement from the purchaser. Development of these materials is continuing by at least one of the vendors to further reduce the required operating voltages while increasing the optical contrast.

Honeywell has in the past made test cells and alphanumeric displays using both electro-optic particle-oriented fluids and liquid crystals. Test results showed certain advantages of each material; basically, the electro-optic fluids made better variable density filters than did liquid crystals.

**Liquid Crystals** -- A multitude of vendors sell liquid crystal solutions for do-it-yourself projects, and sell liquid crystal displays as components or complete instruments.

A simple numeric display was purchased and used for in-lab demonstrations. See-through clarity of the nematic crystal cell was not especially good and served to illustrate the optical scattering problem typically encountered with these solutions. Like electro-optic particle-oriented fluids, the major problems involved with using liquid crystals evolve around the need for a highly-specialized dual (sandwich) visor configuration and associated power supply.

Liquid crystals are generally considered second choice behind particle-oriented fluids for the variable density visor application.

**Electro-Optic Ceramics** -- Two major companies are involved in the fabrication of PLZT electro-optic ceramics. One of them is the original developer...
of the material—Sandia Corporation. The other company is Honeywell Inc. who is at present the only company which successfully manufactures and sells the material in production quantities.

PLZT is manufactured by Honeywell's Government and Aeronautical Products Division (G&APD) (Ceramic Center). As a result, there has been considerable in-house development of and experimentation with this material for various display applications. While PLZT is currently used for nuclear flash goggles and will certainly find many future uses in a planar operational mode, it is not expected that the material will be useful for general HMS/D visor purposes unless used in conjunction with glasses or goggles.
Figure 2-2. Cary 14 Spectrophotometer is One of Several Instruments used to Evaluate Variable Density Concepts
2.3 DURABILITY AND ABRASION

Vendor Evaluation

Three basic technologies which can be used to apply protective organic overcoats to visors were investigated and evaluated.

- Liquid coating
- Vapor phase polymerization
- Thin film deposition

Liquid Coating -- Eighteen vendors of liquid coatings were contacted and their products evaluated for applicability to solving the visor protection problem. The material most widely accepted as the standard of the industry was DuPont's Abcite. Other proprietary organic coatings (including Owens-Illinois T-650) are also candidate materials for the IHMS/D visor.

Vapor Phase Polymerization -- Three major techniques were found to be potentially applicable to the durability problem.

- Pyrolysis (Union Carbide's "Parylene")
- Ultraviolet polymerization (G. E. Process)
- Glow discharge polymerization

The Parylene process was examined, but initially rejected because of a tendency to yellow under intense sunlight. With the switch to tinted acrylic visors, this may not be a problem and the process should be reconsidered in the future.
Both the ultraviolet and glow discharge polymerization processes are in relatively new stages of development. Little physical property data are available for comparison with the more standard liquid coatings. These processes are definitely attractive, however, especially from the standpoint of their inherent abilities to deposit thin films of controlled, uniform thicknesses.

**Thin Film Deposition** -- These techniques include thermal evaporation and RF ion sputtering, and again fall into the general category of advanced technologies with promising futures and potential for IHMS/D applications.

**In-House Laboratory Development and Testing**

**Liquid Coatings** -- Honeywell's GAP Division has extensive capabilities and experience in the development and application of liquid organic coatings. A short-term feasibility program was run using these facilities to test and evaluate three specific silicone coatings and five different primer coatings.

The best results were had with Dow Corning X-1-2900 coating over Dow Corning DC1205 primer. The coating was slightly harder than Abcite and showed excellent potential for improving the durability and abrasive resistance of acrylic optical components.

One of the most significant in-house test results was that a coating which may be readily applied to bare acrylic may physically destroy an optical coating which was previously deposited on that same acrylic. Special care must be taken to select organic overcoats which do not contain solvents or other constituents that react with or soften the acrylic visor.

**Vapor-Phase Polymerization** -- Cost estimates to deposit Parylene C and N on acrylic test panels were obtained from a local vendor, but the tests were not run because of lack of time on the current program.
No samples of ultraviolet or glow discharge polymerization were obtained, but a Honeywell-funded program to develop a facility and technical capability to deposit thin organic films was initiated early in 1973. At present, a vacuum chamber, high intensity ultraviolet source and other associated equipment has been obtained and is presently being set up in the GAP Division Thin Film Laboratory.

**Thin Film Deposition** -- Deposition tests were made in the Thin Film Laboratory using RF ion sputtering to deposit Teflon on various substrates. A decision was subsequently made, however, to discontinue deposition of the organic materials until such time as the aforementioned special vacuum system could be set up and used solely for the deposition of organic films.
Figure 2-3. Differential Interference Microscopy is used to Evaluate Abrasion Tests of Plastic Materials
2.4 HIGH ABSORPTION

The goal of this part of the Coatings Technology program was to develop more efficient means of changing the balance of absorption/reflection of the IHMS/D visor while maintaining transmission at about 1 percent.

Two basic approaches were taken:

- Maximizing absorption of the substrate
- Maximizing absorption of the beamsplitter film

**Substrate Absorption**

A cursory survey of plastics vendors showed that a relatively wide selection of either gray- or bronze-tinted acrylic was available off-the-shelf in transmission ranges down to a nominal 13 percent $T$ at 5500Å. Special low transmissivity material could be made on a minimum order basis with appropriate lead time.

Samples of more than 15 grades of tinted acrylic down to a transmission of 7 percent were obtained and evaluated on a Cary 14 spectrophotometer.

Test depositions of various metal and dielectric thin films on the tinted acrylic showed the material to behave generally the same as clear acrylic and that beamsplitter films could be chemically stripped from the tinted acrylic without any apparent degradation to the plastic surface.

One of the biggest benefits derived from using tinted acrylic is that the ghosting effect can be greatly reduced by virtue of the intrinsic absorptance of the visor. Computer computations of the ghosting ratios for various tinted
material/beamsplitter/anti-reflective (AR) combinations also revealed that satisfactory ghost ratios could be obtained on a tinted visor/beamsplitter without the use of an AR outercoating.

All IHMS/D visors delivered under the current program have subsequently been made of tinted acrylic.

Beamsplitter Absorption

Dielectric beamsplitter thin films have low optical absorption (around 2 percent A). Metallic filter thin films have higher absorption values (around 20 percent A). The goal of this task was to deposit films with absorptances in the 40-80 percent A range.

A series of special high absorption evaporation materials called "Metal Lux" were obtained from Italy through a U.S. vendor who typically coats sunglasses. Because of the success with the tinted acrylic material, however, work on test-depositing these high absorption materials was minimized.
Figure 2-4. Parabolic Dome Formed from High Absorption Tinted Acrylic Material -- Prior to Cutting Out IHMS/D Visor
3.1 OPTICS ON PLASTICS

The development of techniques to deposit optical coatings on plastic materials has not been emphasized to any great extent in the past, mainly because industrial efforts have concentrated more on developing fabrication techniques and new materials for making better optical quality plastic substrates. While simple, low-number-of-layer optical coatings are currently available and are being used on Model 7 units, the more difficult multi-layered coatings are still under development and will likely be available before 1976 along with overcoating techniques and materials which will physically protect the optical coating without seriously degrading optical quality.

Greater emphasis will also be seen on expanding the use of plastic optics to take advantage of low-cost producibility, physical toughness, and resistance to breakage.

A continuing effort in thin film coatings on plastics will be required to maintain helmet sight and display projects on an advanced state-of-the-art level.

3.2 VARIABLE DENSITY

Several techniques are available for obtaining a true, variable density visor. Crossed-polarizing sheets could be used in conjunction with a mechanical rotation device. Visors or sunshields could be made using photochromic materials. Dual sandwich-structure visors could be made using either electro-optic particle-oriented fluids or liquid crystals. Electro-optic ceramic disks could be inserted into visors in the critical areas.
Of all these techniques, the simplest technique which would be compatible with current IHMS/D designs is the photochromic approach. If color neutrality or response time is of more importance and if additional power supplies and photosensors can be tolerated, then more complex visors incorporating electro-optic particle-oriented fluids is considered the second choice.

For each of the above technologies, there will necessarily be some amount of fabrication development effort required to make a variable density visor which is compatible with and does not hinder the performance of an IHMS/D system.

3.3 DURABILITY AND ABRASION

The problem of improving durability and abrasion resistance of optical quality plastic components has only recently been given a great deal of consideration and study. As usual, military requirements have initiated programs which are currently producing some new materials and new coating technologies.

Aircraft transparencies made of acrylic and polycarbonate are now being overcoated with liquid organic coatings for improved durability. These same coatings may not, however, be quite as useful on extremely high-quality optical components where coating uniformities are important. While the best of the current coatings may improve the durability of bare visors, these same coatings may require some special process variations in order for them to be used to protect optically-coated visors.

Several new techniques for depositing ultra-thin organic coatings are also currently under development at Honeywell and elsewhere. It is expected that IHMS/D visor applications will benefit from these programs in the near future.

With but a few exceptions, the durability and abrasion-resistance requirements for IHMS/D applications have been culled from several existing Mil specs which are generally inadequate and relatively unapplicable to the IHMS/D visor.
3.4 HIGH ABSORPTION

The use of tinted acrylic for IHMS/D visors has effectively reduced ghosting effects and spurious reflection conditions which previously existed on early IHMS/D units.

Eventually, transmissivity of the visor will be controlled solely by selecting a grade of tinted acrylic with the proper absorption. At that time, the beam-splitter film on the visor will be made entirely of dielectric materials and deposited on the visor in such a fashion so as to make the coating a very high-efficiency trichroic which has its maximum reflectance peaked for a P1 CRT phosphor at 5300Å.
SECTION IV
RECOMMENDATIONS FOR CONTINUING WORK

4.1 OPTICS ON PLASTICS

- A strong, ongoing effort should be made to replace glass optical components in the IHMS/D system with plastic optical components.

- The results of the "trichroics on plastic" program currently being funded by AMRL should be incorporated into next-generation visors and into other plastic optics being used in conjunction with a CRT.

- A refined and more accurate optical transmission monitor should be developed to enable deposition of beamsplitter spots on visors to accuracies approaching ±0.1 percent at 1 percent T.

- Future IHMS/D programs should consider purchasing a special order lot of low-transmission (1-5 percent T) acrylic sheet for use with high-reflectivity dielectric beamsplitters and trichroic coatings.

- Future IHMS/D programs should carefully follow the advancements being made in the development of new materials and fabrication techniques for aircraft transparencies, and incorporate these developments into advanced IHMS/D units.
4.2 VARIABLE DENSITY VISOR DEVELOPMENT

- A continuing effort should be maintained to define and build prototype (flyable) IHMS/D systems which incorporate at least a dual density and eventually a true variable density visor.

- Because of the simplicity of materials and because of the multitude of design and fabrication options, the concept of cross-polarized sheets should be further studied as a means of obtaining variable density in the range 0-30 percent T.

- Because of the multitude of design and fabrication options and because of the relative compatibility to current IHMS/D designs, photochromic concepts should be explored on a more practical basis with priority being given to experimental forming of visors from photochromic sheet, and overlaying of visors with thin photochromic film.

- While electro-optic particle-oriented fluid and liquid crystal concepts are considered more complex means of obtaining variable-density visors, some effort should nevertheless be made to translate the results of a current AMRL/double-visor program into a design which meets IHMS/D requirements.

4.3 DURABILITY, ABRASION RESISTANCE AND ENVIRONMENTAL PROTECTION

- A long-range program must be initiated to test new materials and fabrication techniques which are required to protect the optical surfaces and optical coatings on IHMS/D visors. Both liquid organic coating and vapor-phase deposition organic
coating techniques should be tested and evaluated, and the best process put to immediate use. The goal of the program would be to improve general cleanability, durability, abrasion resistance, and solvent protection of both bare visors and visors with optical coatings.

4.4 HIGH ABSORPTION

- Future IHMS/D programs should consider special ordering especially low transmittance (1-5 percent) acrylic material for use in conjunction with dielectric beamsplitter coatings on visors.

- High-strength plastic materials (e.g., polycarbonates) which are also available in various grades of tint should be considered for future use on IHMS/D applications where improved strength/weight ratios are required.
Figure 4-1. The Thin Film Laboratory is Completely Equipped to Deposit Thin Films of any Metal, Semiconductor, Glass, or Ceramic
SECTION V
TECHNICAL DISCUSSION

5.1 OPTICS ON PLASTICS

5.1.1 Technical Literature Survey

Using the search facilities of the Honeywell Systems and Research (S&R)/Government Aeronautical Products (GAP) library, a literature search encompassing a 5-year period (1968-1972) was run on the general subject of "optical coatings on plastic materials."

The following information resources were searched:

- Applied Science and Technology Index
- DDC TAB -- (Defense Documentation Center Technical Announcement Bulletin)
- Government Reports Index and Announcements (Formerly HSDRGR -- United States Government Research and Development Reports)
- International Aerospace Abstracts
- NASA STAR -- (Scientific and Technical Aerospace Reports)
- Physics Abstracts

The survey discovered remarkably few technical papers or articles on the subject. What information was found tended to divide the topic into three basic sub-categories -- Substrates, Protective Overcoats, and Optical Films. These are discussed below.
Plastic Optics Substrates -- While the advent of plastic optical components has been predicted for several decades, it has only been within approximately the past 5 years that the optical quality of as-molded plastic substrates has been good enough to match the as-polished quality of glass substrates. Advancements in high-volume injection molding technology and the invention of new processing techniques such as "press polishing" are now enabling plastic optics to take advantage of their lower cost and lighter weight.

From a materials development standpoint, new advancements in chemical processing of the raw materials are improving the optical properties of the finished products. For instance, the optical transmission of clear polycarbonate now approaches that of methyl methacrylate (acrylic), and the reduced number of impurities in polystyrene reduces the yellowish tint that previously prevented this polymer from being a very attractive candidate for low-cost optics. The use of ultraviolet stabilizers in polycarbonate (e.g., G.E.'s Lexan 303), the introduction of new plastic materials such as transparent injection-molded ABS (acrylonitrile butadiene styrene), NORYL (G.E.) and many other developments are all contributing to the increased interest in the use of plastic optical substrates.

Another of the current driving forces for development of these materials is the Air Force's need for more durable, higher temperature materials for aircraft transparencies, canopies, radar domes, etc., (Figure 5-1). New fabrication techniques involving lamination of polycarbonate to acrylic take advantage of the temperature capability and strength of the polycarbonate, with the chemical resistance of the acrylic -- but this combination is still temperature limited. Because of the base material limitations, overcoating with organic materials may improve the temperature capabilities only slightly, and then only for short-duration exposures.

It appears that a more satisfactory solution to the high-temperature problem will soon come from the development of new transparent plastics with
Figure 5-1. Hemispherical Acrylic Aircraft Transparency Used to House Guidance and Target Designator Equipment
exceptionally high heat distortion temperatures (around 260°C), such as 3-M's polyarylsulfone and Astral 360.

**Protective Overcoatings for Plastic Optics Substrates** -- Because plastics are inherently softer, more scratch-prone and less organic solvent resistant than glass optics, there is an obvious need to improve these properties, and the literature being published today tends to illustrate and highlight this need.

While technical approaches such as surface irradiation and glow discharge polymerization/cross-linking are being studied, the greatest emphasis is being heavily placed on developing organic overcoatings which can protect the substrate. Two of the best known of these overcoatings are DuPont's Abcite and Owens-Illinois' T-650. There are, however, literally hundreds of other coatings systems most of which are "proprietary" formulations and which are widely advertised as abrasion- and solvent-resistant coatings for plastics.

Unfortunately, the vast majority of these coatings function only for physical/chemical protection and are usually applied in such a manner -- technique and thickness -- that they are not useful for optical applications where coating uniformity and distortion-free clarity are prerequisites.

Several of the coatings, notably Abcite, however, can be applied in a uniform enough manner to permit their use as a protective overcoat on optical substrates. Abcite is presently being used on several types of flight visors.

**Optical Thin Films on Plastic Substrates** -- Almost without exception, all the literature found to date deals with optical coatings which are of minimal thickness or low number of quarterwave layers -- anti-reflective films of one, two or three layer thicknesses, thin (non-opaque) metal filters, opaque metal mirrors, dielectric overcoats for improved solvent/abrasion resistance, and multilayer dielectric films for relatively low reflectance (~50 percent R) beamsplitters.
The main reason for the thickness limitation is that most plastics are very prone to stress, solvent, and heat crazing and are considerably less compatible with the usual optical film materials than are glass, ceramic or quartz substrates. Commonly used dielectric thin films such as MgF\textsubscript{2}, TiO\textsubscript{2} are also badly mismatched with plastics from the standpoints of thermal expansion and physical hardness. The net result of this hybriding of organic and non-organic materials, is that both the chemical and physical compatibilities are generally lacking and become more obviously so as the non-organic film being deposited on the organic substrate increases in thickness.

Overcoating the plastic substrate with an organic coating that is physically harder than the substrate provides for some improvement in the durability and abrasion resistance of the deposited film, but there is usually not enough benefit to enable the optical film to compete with an identical film deposited on hot glass. An anti-reflective film deposited on Abcite-coated acrylic, for instance, may be as durable and abrasion resistant as the same film deposited on room-temperature glass, but not as good as the same film deposited on hot glass.

Overcoating the optical film with an organic coating has two difficulties:

**First**, many of the organic overcoats are solvent systems which react somewhat with the plastic substrate. If there are pinholes or craze-cracks in the optical film or edge areas where the substrate is exposed, the interface between the optical film and the substrate will be chemically attacked -- resulting in crazing and lifting of the film.

**The second problem** deals with the destruction or interference of the previously achieved optical properties. Unless the organic overcoat is deposited to precise thicknesses so that it enhances or contributes to the desired optical characteristic, it will likely reduce or eliminate the efficiency of the optical film. As an example, thick organic coatings
over quarter-wave MgF₂ will eliminate the anti-reflective surface — but deposition of precise multiple half-waves of Teflon over the MgF₂ will somewhat maintain the reflectance at a low level (until cumulative film absorption becomes too great.)*

5.1.2 Vendor Survey

Attachment No. 1 is a sample form letter sent out to more than 85 vendors of optical coatings. Only two or three companies indicated any interest or experience in coating multilayer optical films on plastic substrates.

One of the companies which has recently advertised quite heavily on their plastics coating capability admitted they did not want to coat more than a 5-layer system. Problems with crazing and film integrity are the reasons for failure of the higher-order number of layer films.

5.1.3 Laboratory Studies -- Optics on Plastic

Adhesion of Thin Films to Acrylic -- A brief series of thin-film test depositions were made to determine which metal and dielectric materials would adhere most strongly to clear and tinted acrylics.

Unlike metal/glass and metal/ceramic couples where pure chromium is most often used as a bonding aid, the materials which exhibited the strongest adhesion to acrylic were Inconel 600 (a Ni-Cr-Fe-Cu-Si-Mn-C alloy) and cerium dioxide (CeO₂).

*Note: The subject of organic thin films deposited to precise thicknesses by ultraviolet polymerization, glow-discharge polymerization, evaporation, ion sputtering and vapor-phase pyrolysis will be discussed briefly in the following section on Durability and Abrasion.
With the exception of a couple of early IHMS/D visors, all of the metal-reflecting mirror and partially transmitting beamsplitter spots deposited on the inside visor surfaces were made of Inconel/aluminum thin films.

Figure 5-2 shows a tinted acrylic visor with the opaque Inconel/aluminum spot on the upper left corner of the visor, with the semi-opaque (nominal 0.8 percent T) Inconel/aluminum spot on the lower right.

**Development of High-Reflectance Opaque Mirrors** -- In order to further boost the reflectivity of the opaque Inconel/aluminum mirrors, a series of tests were made to develop an enhanced metal mirror system which would be compatible both with combiner glasses and acrylic visors.

The following materials systems were tested:

- Al
- Ag
- Al/MgF\(_2\)
- Al/MgF\(_2\)/CeO\(_2\)

Figure 5-3 shows the reflectance curves for the two overcoated combinations. While the reflectance of silver is generally quite good, it was believed that the silver on acrylic is too sensitive to environmental attack and tarnishing (even if overcoated), and that the more passive and potentially more durable system using opaque aluminum overcoated with multiple \(\lambda/4\) layers of MgF\(_2\) and CeO\(_2\) was a better selection for the IHMS/D application. Because the index of refraction for metals such as aluminum is a complex number, special equivalent \(\lambda/4\) optical thicknesses are required to compensate for the k value (extinction coefficient for phase charge) so that the resultant coating produces constructive interference and enhanced reflectivity.
Figure 5-2. IHMS/D Visor with Inconel/Aluminum Reflecting Spots
Figure 5-3. Reflectance of Opaque Mirrors on Acrylic Sheet
Continued development is expected to raise the reflectance curve for this type of opaque reflector from the present 92-94 percent reflectance to 95-98 percent reflectance.

Development of Optical Sensor -- During the course of the IHMS/D program, the following metal filter (beamsplitter) coatings were required to be deposited in the inside visor surfaces.

<table>
<thead>
<tr>
<th></th>
<th>Transmission</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Summer 1972</td>
<td>70% ± 5%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>50% ± 5%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>30% ± 5%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>10% ± 2%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>5% ± 1%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>1% ± 0.5%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>1% ± 0.2%</td>
<td>transmission</td>
</tr>
<tr>
<td>March 1973</td>
<td>+ 0.0%</td>
<td>transmission</td>
</tr>
<tr>
<td></td>
<td>1% - 0.2%</td>
<td>transmission</td>
</tr>
</tbody>
</table>

Since the sensitivity of an optical monitor is inversely proportional to the transmission, the difficulty involved in producing these films increased geometrically. The difficulties were, in the past year, overcome to some extent by making improvements in our home-made monitoring system. The following sketches illustrate the various iterations and modifications which were made.

First Generation

Quartz crystal monitors of the type shown rack-mounted in Figure 4-1 were initially used and were satisfactory as long as transmission tolerances were relatively loose (±5%). The crystals were placed as near to the substrate as possible during deposition.
Second Generation

A Cds photocell and light bulb source were used to measure the transmission through a flat sample sheet located adjacent to the visor.

Third Generation

A micro-volt-ammeter was used in place of a less accurate millammeter to improve the sensitivity of measurement.
Fourth Generation

Fixtures were revamped to permit measurement of transmission through the actual beamsplitter as it was being deposited on the visor.

Fifth Generation

Because of the curvature of the visor it was necessary to align the source accurately with the detector to improve rim-to-rim reproducibility. A collimating lens system was added to the barrel of the source to achieve this improvement.
With the above setup, filters can be deposited to 1 percent ± 0.5 percent T with 80-90 percent success.

Figure 5-4 is a photograph of the current fifth generation setup. The source and detector tubes (bottom and top) are located on either side of the visor (which has already been coated with the mirror and beamsplitter spots). A remote crystal oscillator head can also be seen at 5 o'clock to the visor.

**Proposed Sixth Generation**

To achieve transmission accuracies at 1 percent ± 0.2 percent T and eventually to 1 percent ± 0 -0.2 percent T, it will be necessary to make the several additional improvements shown in the following schematic.

**Improvements:**

No. 1 - Silicon detector; stable, not as temperature sensitive.
No. 2 - Reference sample holder; eliminates angle problem associated with hand-held reference, opens possibility of using bandpass filter for dielectrics.
No. 3 - Light baffles; prevent uncollimated light reaching the detector.
No. 4 - Alignment fixture; cuts setup time, insures maximum sensitivity.
No. 5 - Better collimation, increases sensitivity.
Figure 5-4. Optical Monitor Setup Used to Measure Transmission Through the Beamsplitter Spot on the IHMS/D Visor
Visor Spot Measurements -- After depositing the partially transmitting metal filter/beamsplitter spot on the visor using the previously mentioned optical sensor, the visor would be removed from the system. It would be visually examined for quality and uniformity (Figure 5-5), and remeasured in air using a diffused light source, Spectra Brightness Spot Meter/Power Supply and Photo Research Corp. footlambert meter. Figure 5-6 illustrates this test setup.

There was excellent agreement between the readings obtained with this setup, the optical monitor in the vacuum system, and the highly accurate Cary 14 spectrophotometer.

Visor Rework Capability -- A simple solution of 1-5 percent HF in de-ionized water was found to chemically strip most of the metal and ceramic thin films deposited on acrylic surfaces. While there is no apparent degradation seen after initial stripping, repeated stripplings (three or four times) appear to eventually degrade the optical surface quality.

Trichroic Test Depositions -- In visor-projected helmet mounted displays, the optical system is susceptible to interference from ambient light if the incoming light aligns with the optical path inside the visor. The result is a significant loss of image contrast and a resultant degradation of image readability. In addition, metal mirror and filter spots of the type currently used on the IHMS/D program have an upper limit of reflectivity -- around 70-90 percent R for the metal filter spot. An additional benefit in image brightness and contrast could be obtained if each reflecting surface could be improved to around 95-99 percent.

In early 1973, Honeywell proposed that a combination of trichroic coatings on the visor and other optical surfaces could solve the problem. While it was not possible to fully explore trichroics on the current program, some work did show the feasibility of the approach and also pointed out several technical thin-film deposition problems. Trichroic test depositions were made on both acrylic and polycarbonate visors.
Figure 5-5. IHMS/D Visor with Opaque Reflector Spot (Upper Left) and Partially Transmitting Metal Filter Spot (Lower Right)
Figure 5-6. IHMS/D Visor Spot Measurement Setup in Thin Film Laboratory
Figure 5-7 is a transmission curve of one of these coatings.

Following discussions with AMRL, a written proposal was made to the Air Force to develop trichroic coatings on acrylic material (Ref. 2). This program was subsequently funded by the Air Force and is currently in progress.

The status of this program as of June 15, 1973 is summarized in the Appendix of this report -- Attachment 2.
Figure 5-7. Characteristics of 11-Layer Trichroic Coating on Tinted Acrylic
5.2 VARIABLE DENSITY

5.2.1 State-of-the-Art Technology Survey

All of the information resources previously listed in Section 5.1.1 were also used in searching for information on variable density concepts.

5.2.2 General Status of Candidate Approaches

5.2.2.1 Dual Sunshield -- The use of crossed polarizers appears to be the most attractive and feasible of the dual sunshield approaches. Polarizing film and sheet stock is readily available in many different sizes and thicknesses, and it would seem that either manual or motor-driven techniques could be used to rotate one of the polarizer sheets through 90 degrees to obtain essentially complete light attenuation.

Sample sheets of thin polarizing films were obtained from Marks Polarized Co. and measured on the Cary 14. The film (without its backing sheet) had a transmission of 26 percent T. Uncrossed pair dropped the transmission to 16 percent T -- and crossing the sheets reduced the transmission to 0 percent T at 5500Å, with only a bit of U-V (approximately 0.4 percent T) being transmitted at 4000Å (see Figure 5-8).

One means of applying polarizing film to curved surfaces is shown in Figure 5-9, which shows a vendor-supplied lens of CR-39 plastic with a thin polarizing film stretched and essentially "glued" over the convex surface.

It is possible that this technique could be used to apply a film to the outside of an IHMS/D visor, then used in conjunction with another polarizing sheet which is rotated to effect variable transmissivity.
Figure 5-8. Cary 14 Transmission Scan of Polarizing Sheet
Figure 5-9. Polarizing Film as Received, and Stretch Laminated to a CR-39 Plastic Lens
Other "variable density" concepts examined included:

- **3-M's Louvered Light Control Film** - A cellulose acetate butyrate material which closely resembles a miniature venetian blind windowshade. The film is actually a composite laminate material with a 10-mil-thick "louver" sheet sandwiched between two 5-mil-thick cover sheets. The louvers are typically spaced on 10 mil centers. The total thickness of the light display (control) sheet can be varied, as can the size, spacing and angle of the louvers. The material can be vacuum formed. Typical applications of the material include cutting off the overhead incident light on the front of CRT displays, and cutting out high-angle light on ski goggles.

- **Isomet's Electronically Tunable Optical Filter** - An all solid-state device which has the unique capability of changing its optical transmissivity according to the frequency of an applied electrical signal. Unfortunately for the IHMS/D application, the filter does not change the overall transmission level within the visible spectra, as much as it merely shifts the wavelength at which its transmission is a maximum.

5.2.2.2 **Photochromics** -- Photochromic materials are, with the exception of photochromic glasses, extremely complex organic compounds which are only recently (since 1960) being thoroughly enough understood to be considered commercially marketable products. The range of chemistry, composition, characteristics, and properties is unusually broad, making it difficult to derive a single product that is useful for many types of applications. Photochromic sheet is commercially available in at least one standard color and thickness which uses cellulose acetate butyrate as a substrate and whose photochromic coating is subsequently overcoated with another organic film.
for protection against moisture and abrasion. Photochromic coating material is also available commercially for do-it-yourself coaters.

The advertised properties of one commercially available material, Markopolo Type MPS-1 (Marks Polarized Co.) are shown in Table 5-1.

In-house observations of this material indicated the return or relaxation times are not as quick as advertised, but rather three or four times longer.

One of the more current photochromic materials is mercury dithizone (diphenyl thiocarbazone). The photochromic behavior of this compound has only recently been examined in detail. The normal form has a strong absorption band in the range of 4900-5300°A. Irradiation in this band produces an intermediate with strong absorption centered around 6000°A. The rate of back reaction is highly dependent on the presence of another molecule serving as a proton bridge. Although mercury dithizone does not react with a high quantum yield, high optical densities can be produced with sufficient intensity of light. They show good photochromical stability if activated by visible light. A potential problem with this material is that there is little attenuation of the infra-red region, so that precautionary measures might have to be taken to avoid the possibilities of retinal burn.

Another company which has shown some current interest in photochromic materials is American Cyanamid Co. Very little information was found, however, except that they apparently have made some aircraft transparencies for a commercial airline.

**Fabrication of Photochromic Visors**

Figure 5-10 illustrates eight different techniques that could be considered in using photochromic materials to achieve variable density visors. Of these techniques, four basic processes emerge:
Table 5-1. Properties of Markopolo\textsuperscript{tm} MPS-1*

<table>
<thead>
<tr>
<th>Property</th>
<th>Description</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Transparent State</strong></td>
<td>Low Intensity Light</td>
<td>Yellow-Orange</td>
</tr>
<tr>
<td></td>
<td>Average White Light</td>
<td>$30%$</td>
</tr>
<tr>
<td></td>
<td>@4500 A</td>
<td>$0.1%$</td>
</tr>
<tr>
<td></td>
<td>@6000 A</td>
<td>$85%$</td>
</tr>
<tr>
<td><strong>Light \to Dark</strong></td>
<td>Moderate Sunlight</td>
<td>$30 \to 9%$ (10 seconds)</td>
</tr>
<tr>
<td></td>
<td>Bright Sunlight</td>
<td>$30 \to 4%$ (10 seconds)</td>
</tr>
<tr>
<td></td>
<td>Yellow-Orange \to Neutral Gray</td>
<td></td>
</tr>
<tr>
<td><strong>Dark \to Light</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$65%$ of Original $%T$ in 15 seconds</td>
<td></td>
</tr>
<tr>
<td></td>
<td>$90%$ of Original $%T$ in 90 seconds</td>
<td></td>
</tr>
<tr>
<td><strong>Repeatability</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Several thousands of cycles (minimum)</td>
<td></td>
</tr>
<tr>
<td><strong>Temperature Stability</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$185-195^\circ C$ maximum limit</td>
<td></td>
</tr>
</tbody>
</table>

*Marks Polarized Company

2079-SR3
Coating Formed Parts -- A small quantity of photochromic coating liquid was obtained from Nuclear Research Associates and tested in-house. Test results showed that special coating techniques would have to be developed to obtain a uniformly thick, defect-free, optical quality coating on a formed acrylic visor. Overcoating materials and techniques would also be required to protect the moisture-sensitive photochromic coating. Figure 5-11 shows the type of permanent damage that moisture and fingerprints can cause to a photochromic-coated acrylic paraboloid which has not been overcoated for environmental protection.
Figure 5-11. Photochromic-Coated Acrylic Paraboloid Showing Moisture Reactions Which Can Occur if the Photochromic is not Overcoated for Protection
A test was also run at a vendor's shop whereby another parabolic dome (uncut visor) was flow-coated with photochromic material -- again, it was evident that another technique would be required to obtain the desired coating thickness and uniformity.

**Forming Coated Sheet**

Sample panels of photochromic sheet stock were also special-ordered from the same vendor in the following configurations:

1) Nine inches x 18 inches x 0.030 inch and 18 inches x 18 inches x 0.030 inch cellulose acetate butyrate sheet coated on both sides with orange photochromic coating.

2) Same as No. 1 except overcoated with proprietary protective coating.

3) Eighteen inches x 18 inches x 1/8 inch acrylic sheet coated both sides with orange photochromic coating and overcoated with protective coating.

4) Eighteen inches x 18 inches x 1/8 inch acrylic sheet composed of two pieces 18 inches x 18 inches x 1/16 inch acrylic sheet previously coated with orange photochromic coating on one side, then laminated together to form 18 inches x 18 inches x 1/8 inch sheet with photochromic material on inside.

The nine inch long 0.030-inch sheets were used for simple photochromic demonstrations. The 18 inch by 18 inch x 0.030-inch sheets were used for vacuum forming tests to determine the effect of radii and forming temperatures on the photochromic material. **Figure 5-12 shows the vacuum former and epoxy mold used in these tests.**
Figure 5-12. VacuumFormer and EpoxyMold Used to Form ThinPhotochromic-CoatedPlasticSheet
Test results indicated that forming temperatures will have to be absolutely minimized and that short radii (less than 3/4 inch) will have to be avoided -- especially if the sheet is overcoated. Figure 5-13 shows the whitish discoloration and severe crazing and tear markings which result if the coated sheet is not formed using the proper time/temperature conditions. Figure 5-14 shows the same part formed at a lower temperature.

Additional tests, using the vacuum-forming technique developed by Honeywell for forming IHMS/D visor paraboloids, were also made on the thin acetate butyrate photochromic material. Tests results, while encouraging, indicated the need to properly prebake the sheet before forming so that moisture bubbles would not destroy the optical quality. Figure 5-15 illustrates the specular bubbles which arise on vacuum forming a hard-coated photochromic sheet which was not adequately prebaked. Figure 5-16 shows the same type sheet which was formed at a lower temperature. Moisture bubbles are fewer and smaller but the paraboloid was incompletely formed.

The thicker 1/8-inch acrylic sheets with photochromic outer coat and hard overcoat were also vacuum formed. Test results again showed the need to prebake the sheet before forming. There was also an indication that the coating became lighter in color (increased transmission) after forming. The 1/8 inch-laminate sheets with photochromic on the inside were to be made on a development basis by the vendor, but to date, have not yet been received. It is expected that placing the photochromic on the inside will avoid some of the problems with sharp radii, will maintain thickness uniformity of the photochromic during forming, and will perhaps reduce somewhat the deleterious effects of temperature during forming.

Laminating Film on Formed Part -- The approach recommended by one of the photochromic vendors was to apply a photochromic film to a visor in the same manner in which the polarizing sheet was applied to the CR-39 optics blank shown in Figure 5-9.
Figure 5-13. Photochromic-Coated Sheet with Hard Overcoat -- Formed in Vacuum Former at Too High a Temperature
Figure 5-14. Photochromic Sheet with Hard Overcoat -- Formed in Vacuum Former at Proper Temperature
Figure 5-15. Vacuum-Formed Photochromic Paraboloid with Moisture Bubbles
Figure 5-16. Vacuum-Formed Photochromic Paraboloid with Fewer, Smaller, Moisture Bubbles
The basic approach would be as follows:

1) Form acrylic paraboloid (or visor)
2) Clean surface
3) Form photochromic foil or film (4-5 mils thick)
4) Chemically soften photochromic and stretch over dome
5) Allow photochromic to reharden
6) Spin-coat hard organic overcoat over photochromic for environmental protection

The basic reason for not applying both coating materials to the dome as liquids, is that spin or flow coatings are very thin (< 0.1 mil) and that in order to maintain humidity protection, either the photochromic or the protective overcoat must be at least several mils thick.

Extruding Photochromic Sheet -- There is a possibility of extruding a photochromic-doped acrylic sheet. The job would probably require two vendors -- one to provide the photochromic materials and process technology -- the other to provide the extrusion equipment.

- Prototype extrusions require 50-100 pound batches of acrylic (enough to fill extrusion drums).
- Over 20-inch wide sheet is considered production equipment. Standard production width is 50 inches wide.
- Having once obtained an extruded sheet (the surface finish of which would be relatively coarse), the sheets would be cut into the 24-inch x 24-inch squares (size required to vacuum form visor paraboloids), then hot-pressed against mirror surfaces to obtain higher-quality surface finishes.
Photochromic Glass

As mentioned earlier in this report, there are some photochromic glass materials available in industry -- notably Corning Glass Works' Photogray and Photosun ophthalmic blanks.

Like PLZT electro-optic ceramics, this material would have to be used as inserts in a visor. A sample blank is shown in Figure 5-17.

5.2.2.3 Electro-Optic (Particle-Oriented) Fluids -- Particle-oriented fluids are suspensions of sub-micron sized needle-like dipole crystals suspended in an electrically insulating carrier fluid, which when polarized in an electric field, become oriented in such a fashion so as to block out light being transmitted through the cell. One of the advantages this material has over most conventional liquid crystals is that light passing through the cell is not scattered or made diffuse.

As of mid-1971 only two companies were marketing particle-oriented fluids. The current state of the art is such that the primary interest in these materials is for display applications where the competing technology is liquid crystals.

Honeywell has made up alpha-numeric displays using these materials, and has found them to be generally comparable to nematic liquid crystals. The following comparisons can be made:

- Power density - Liquid Crystals (LC) higher
- Contrast uniformity - LCs less dependent on thickness
- Operating voltage - LCs lower
- Contrast - Equivalent
- Resolution - Equivalent
Figure 5-17. Photochromic Glass Ophthalmic Blank
- Viewing angle restrictions - None for Electro-Optic (EO)
- Temperature range - EO passes Mil Spec
- Lifetimes - Equivalent
- Response time - LC faster
- Use for variable filters - EO better

A simple test filter was also made up during the present program using Varad V-100 (Marks Polarized Co.). Purpose of the cell was to check out the transmission characteristics at different applied voltages. The Cary 14 was used to scan the cell as various voltages were applied in 5-volt increments between 0-45 v at 3500 Hz.

Figure 5-18 shows the resultant family of transmission curves.

Although activation of the fluid is frequency dependent, the optimum frequency is about 3 KHz. There was essentially no change seen in the transmission curve at 45v in varying the frequency between 3000-4000 Hz.

The biggest problem with using particle-oriented fluid technology for the IHMS/D application is that the visor, which is already a critical optical element, must now be made more complex by making it into a sandwich structure composed of two conformal visors, precisely spaced just a few mils apart, containing a protective overcoat and transparent conductor electrodes over their entire mating surfaces, and sealed at the edges to contain a fluid which normally attacks or dissolves acrylcs and polycarbonates. A power supply and associated controls, optical feedback sensors, etc., must also be incorporated into the IHMS/D system.

5.2.2.4 Liquid Crystal Fluids -- With but a few differences, liquid crystal methods of obtaining variable density light control are basically the same as those described for electro-optic particle-oriented fluids. The biggest
Figure 5-18. Effect of Voltage on Transmission of Electro-Optic Particle Oriented Fluid Test Filter
objection, again, is the complexity of the dual-visor sandwich structure, its sensitivity to gap spacing and the associated hardware needed to operate the cell.

Figure 5-19 illustrates a simple numeric liquid crystal display procured from Compar Corp. The display was used for general visual evaluation of see-through. Light scattering and localized mottling appeared to characterize the display.

Because of the optical scattering effects seen when using liquid crystal as a transmission filter, liquid crystals are considered inferior to particle-oriented fluids for this application. There is, however, a technique, the "Field Effect," whereby unidirectional rubbing of the inside electrode surfaces can cause nematic-type liquid crystals to align in a preferred orientation to effectively increase contrast and reduce optical scattering.

Another major problem with using either liquid crystals or particle-oriented fluids for a flight visor application is that they do not operate in a fail-safe mode; e.g., if the power goes out, the filter goes black (opaque).

5.2.2.4 Electro-Optic Ceramics -- Materials such as lanthanum-doped lead zirconate-lead titanate (PLZT) are a recent development (1970-71) in electro-optic ceramic materials.

To use PLZT as a transmission filter, it is necessary to take a disk of the hot-pressed material, optically polish both sides, deposit an interdigitized array of transparent conducting electrodes on one surface, and sandwich the disk between orthogonally-crossed polarizers. Without any applied voltage, the crossed polarizers effect a zero transmission condition. With a voltage applied to the electrodes, an electric field is produced in the material transverse to the optical axis, causing induced birefringence in the PLZT which
Figure 5-19. Liquid Crystal Numeric Display
rotates the polarization of the light and effectively enables light to be transmitted through the second polarizer. Response characteristics are fast — going from approximately 30° T to 0.1° T in less than 50 microseconds, and down to 0.003° T in 500 microseconds.

Because of the fast response times, PLZT has been used to make nuclear flash goggles. Unfortunately, there are several basic problems with using this material for IHMS/D.

- PLZT is a brittle ceramic which has size limitations, and as such would have to be used as an insert into the visor.
- PLZT, like particle-oriented fluids and liquid crystals, requires a power supply for operation. Crossed polarizers are also needed.
- PLZT operates in a planar mode, making incorporation into a visor a difficult task.

It is expected that forthcoming applications will mainly use PLZT in welder's goggles-type devices and in flat arrays such as is proposed for futuristic, thin, wall-hanging TV sets.

Current state-of-the-art PLZT development work is noted as being concentrated in two major areas --

- Improvement of optical properties, mainly by using higher purity materials to reduce material defects and increase see-through clarity, and through use of anti-reflective transparent conductor electrodes.
Development of slurry casting techniques to increase the size of disks and plates larger than can be made by hot-pressing.

One of Honeywell's current applications of this material is the moving reticle heads-up rocketsight display shown in Figure 5-20.
Figure 5-20. Army XM 73 Rocket Sight Modified for PLZT Variable Reticle Pattern Generator
5.3 DURABILITY AND ABRASION RESISTANCE

5.3.1 Introduction

The four basic categories of flight visors are

- Standard Flight Visor -- no optical coatings
- Standard Flight Visor -- with optical coating(s)
- Helmet Sight Visor -- no optical coatings
- Helmet Sight/Display Visor -- with optical coating(s)

Whether the above visors be made from acrylic or from polycarbonate, they each share a relatively equivalent problem with durability, abrasion resistance, and solvent resistance. The fact is that most plastics are easily scratched and marred, and are otherwise susceptible to physical damage which can degrade the optical quality of the plastic surface.

In the case of the standard flight visor (uncoated), any scratches or marrings of the surface can be readily buffed and polished out using a mildly abrasive polishing compound and damp cloth. The abrasion resistance of this category visor can also be improved by overcoating the visor with a harder, organic coating.

If the standard flight visor is coated with, for instance, a thin gold film for improved IR reflectance and protection against intense solar radiation, then the gold-coated visor must immediately be overcoated with a hard organic coating. Because of the thinness of this protective overcoat, no subsequent polishing or buffing would be permitted.

In both of the previous cases, neither the mechanical polishing nor application of the protective overcoat degrades the general see-through quality of the visors.
For helmet sight visors there is a high need to maintain the special optical quality and figure of the visor inner surface. Visors which reflect a simple cross-hair from an intense tungsten light source may not need any optical coatings to enhance the visor reflectivity. In this case, any minor scratches or marrings on the visor may sometimes be removed with polishing compounds using extremely light and uniform polishing technique. Protective organic overcoats may not be used to protect the visor unless they can be applied in such an especially uniform and defect-free manner so that the optical image is not distorted or distracted, and so that see-through and prismatic deviation problems do not arise.

If the helmet sight visor needs an optical coating to enhance the surface reflectivity, or if the helmet sight/display visor needs special optical coatings to enhance contrast, brightness, and definition of a CRT image, then:

- No buffing or polishing of the optical coatings is allowed
- The optically coated visor must be used as is, or protective overcoating material and techniques must be developed to protect the optical coatings.

5.3.2 Nature of the Overcoating Problem

There are two major aspects to the overcoating problem:

- Solvent resistance of the visor must be improved so that various organic solvents and solutions typically encountered around the general flight environment and in various cleaning agents will not chemically attack the visor.
The abrasion resistance of the optically coated visor must be improved to withstand the rigors of usage and handling and to permit more effective and more frequent cleaning.

The solvent resistance problem arises from the fact that plastics such as acrylic are readily attacked by most organic solvents such as acetone, trichloroethylene, alcohols, etc. Even a short-term exposure of acrylic to a mild solvent such as ethyl alcohol or methyl alcohol can lead to softening of the plastic surface. Overcoating of acrylic with thin-film dielectric materials does impart solvent resistance to the visor, but pinholes in the film and edge effects around the visor edges and bolt holes can still be susceptible to solvent attack. Organic overcoats applied by dipping, spinning, or spraying are more effective solvent barriers than are vacuum-deposited thin films.

The basic durability problem arises from the fact that acrylic is a relatively soft substrate material and that, regardless of how hard the deposited optical film is, if the film is thin enough it can be "scratched" and "abraded"; i.e., the underlying substrate deformed sufficiently to cause collapse and localized fracture of the film. A crude analogy is the apparent durability of a thin film of ice on hard concrete - versus - the apparent lack of durability of a thin film of ice on a soft mud field. A better example can be seen by comparing a thin film of chromium deposited on a glass surface, with the same film deposited on acrylic. The chromium film on glass can be rubbed, scrubbed, and severely abraded--while the same film on acrylic is readily "rubbed off."

Chemical reactivity of the deposited film with the substrate is, of course, an important factor which must also be considered. Unfortunately, there is a relatively low propensity for most metals to stick to organic surfaces. Even oxide getters such as Cr and Al find little chemical attraction to polymer surfaces.
5.3.3 In-house Testing and Evaluation of Overcoat Techniques and Materials

5.3.3.1 Forming of Hard-Coated Material -- Vacuum-forming tests were run to determine the feasibility of forming paraboloids (basic visor shape) using hard-coated plastic sheets.

The tests previously mentioned in paragraph 5.2.2.2, where hard-coated photochromic sheet stock was formed, is one example of these tests. As shown in Figure 5-13, unless forming time and temperatures are correct and unless radii are minimized, there is a strong probability that the hard overcoat will tear, crack, and otherwise be stretched to the point of failure.

Figure 5-15 showed moisture bubbles which can occur on forming hard-coated photochromic sheet. While this problem can also be experienced on uncoated sheet, it serves to show that preconditioning or bake-out of the base sheet is an important process step if the overcoated sheet is to be subsequently formed.

Because of the reputation of Abcite as an outstanding hardcoat material, it was decided to attempt to vacuum form a test paraboloid from Abcite-coated acrylic sheet. The results of this test are shown in Figure 5-21. As can be seen, the extremely hard overcoating cracked and crazed. The vendor data sheets on Abcite declare that Abcite can be drape formed at 275°F, but only for single-contours not exceeding a 4-inch radius. While our single test is certainly not conclusive, it appears indicative of the probability that hard-coated sheets of acrylic cannot be formed into certain parabolic shapes for IHMS/D applications.

5.3.3.2 Thin-Film Protective Overcoatings -- Glass optical substrates can be coated with optical thin films which are extremely hard and durable. The coatings are usually composed of λ/4 layers of glass and ceramic materials, deposited by thermal/e-beam evaporation or ion sputtering.
Figure 5-21. Craze Markings on Abcite-Coated Acrylic Sheet Vacuum Formed into Paraboloid
The films are hard and durable for three major reasons:

1) The film/substrate hardneses are relatively compatible, so that the substrate provides a physically strong and supporting base for the coating.

2) The film/substrate chemical and physical properties are relatively similar so that adhesive bonds are strong and thermal expansion mismatches are minimized.

3) The substrate can be heated to an elevated temperature (200-400°C) during deposition to assist chemical bonding of the deposited film and to achieve its most stable and physically dense molecular structure.

Plastic substrates do not lend adequate support to a hard optical film -- the substrates are readily deformed and depressed, which results in stress crazing of the plastic surface and cracking or spalling failure of the deposited film. Plastic substrates are for the most part chemically incompatible with the optical films so that adhesive bonding is weak. Finally, plastic substrates cannot be heated to a temperature significant enough to assist in formation of hard films.

Tests run in the Thin Film Laboratory showed that some benefit could be achieved by overcoating a simple aluminum front surface mirror on acrylic with a dielectric overcoat, but that the improvement was only minimal compared to the durability of the same coating on glass substrates. Typical abrasion resistance of aluminum on acrylic would be 20 cheesecloth rub at 1/2 lb. force, compared to 50 rub/1 lb. for Inconel/Al/MgF₂/CeO₂ on acrylic.

Simply increasing the thickness of the dielectric overcoat film did not further improve the abrasion resistance, but instead made a highly stressed coating which was more susceptible to crazing.
Reference 3 reports work performed by another company which attempted to increase the durability and abrasion resistance of polycarbonate plastic lenses by ion sputter-depositing 500-10,000Å thick films of SiO₂, Pyrex, and Al₂O₃ over the optical surface. The conclusions to their work showed that even mild substrate heating (to 80°C) produces outgassing of the plastic which effectively destroyed the film being deposited, and that while 1000Å Al₂O₃ seemed to be the best overcoat film, they concluded that the "mar resistance test" was "invalid" due to "etching" effects.

Their report indicates that the particular test consisted of dropping No. 80 carborundum grit onto the lens surfaces and visually examining for scratches and defects. Test results showed that the grit "etched" both the plastic and the ceramic thin film overcoats.

It is interesting to speculate that the "etching" effect was probably stress crazing which produces a fine network of cracks -- and that this test did provide a valid indication of the film's physical integrity. In fact, these craze marks must be prevented from forming before the overcoating can be considered useful.

The "Trichroics on Plastics" program currently being run by Honeywell has been successful in determining some of the major deposition parameters which influence crazing of both the plastic substrate and the ceramic overcoat film. To date, we have been successful in eliminating the stress crazing in multi-layer ceramic films up to 13,000Å thick. Details on the current status of this project are presented in Attachment No. 2 of this report.

A brief series of test depositions was previously made using dc ion sputtering to deposit thin films of metals on acrylic test pieces. Results showed that temperature effects due to surface scrubbing of the argon ions would be a major problem, and that the test pieces would have to be entirely removed from the plasma and shielded from cathode radiation to prevent heating of the substrates.
Figure 5-22 shows the sputtering system used in these tests.

5.3.3.3 Organic Protective Overcoatings

NOTE: The in-house work performed on development and evaluation of abrasion-resistant organic coatings for the IHMS/D application is reported in Attachment No. 3 of this document.
Figure 5-22. Low-Energy D-C Ion Sputtering System
5.3.3.4 Military Specifications for Visors -- Several Military Specifications are used to define the durability and abrasion resistance of various flight visor configurations. In general, these specifications rely on other existing military specifications (which pertain to optical coatings on glass substrates) for testing of various coatings on plastic visors.

Unfortunately, the various coatings referred to are used on standard flight visors and do not necessarily have to be of the same high optical quality as visors used for a IHMS/D.

Adhesion of most coatings (whether on glass or plastic) is usually tested via an "adhesive tape" test.

Abrasion resistance of optical coatings on glass is usually tested by rubbing the surface with either "cheesecloth" or an "abrasive eraser". Optical coatings on plastic are usually tested only with cheesecloth. Other tests for "haze", "mar resistance," and "abrasion" using Taber abraders, steel wool, powdered cleanser, falling grit, calibraser wheels, etc., are also used for evaluating thick organic coatings on plastic.

A brief summary of applicable military specifications is presented in Table 5-2.

5.3.3.5 Visor Handling and Cleaning Instructions -- A set of instructions and materials to be used in cleaning and handling helmet sight/display visors was prepared earlier by Honeywell. Attachment No. 4 to this report contains a copy of these instructions.
### Table 5-2. Military Specifications Relevant to Visor Coatings

<table>
<thead>
<tr>
<th>Military Specification No.</th>
<th>Title</th>
<th>Adhesion</th>
<th>Abrasion</th>
</tr>
</thead>
<tbody>
<tr>
<td>MIL-M-13508B</td>
<td>&quot;Mirror Coating Process, Front Surface Aluminized, for Optical Elements&quot;</td>
<td>Slow tape</td>
<td>50 rub cheese/lb.</td>
</tr>
<tr>
<td>MIL-C-675A</td>
<td>&quot;Coating of Glass Optical Elements (Anti-Reflection)&quot;</td>
<td>Visual flaking, peeling, or blistering</td>
<td>Abrasive eraser 20 strokes/2.0-2.5 lb.</td>
</tr>
<tr>
<td>MIL-C-14806A</td>
<td>&quot;Coating, Reflection Reducing, for Instrument Cover Glasses and Lighting Wedges&quot;</td>
<td>Visual flaking, peeling, or blistering</td>
<td>Abrasive eraser 20 strokes/2.0-2.5 lb.</td>
</tr>
<tr>
<td>MIL-L-38169</td>
<td>&quot;Lenses, Goggle and Visor, Optical Characteristics, General Specification for&quot; (Class 3 only -- gold coated)</td>
<td>Fast tape</td>
<td>Abrasive eraser 20 strokes/2.0-2.5 lb.</td>
</tr>
<tr>
<td>MIL-V-22272B</td>
<td>&quot;Visors, Protective, Helmets&quot; (Class 3 only -- gold coated)</td>
<td>Fast tape</td>
<td>Abrasive eraser 20 strokes/2.0-2.5 lb.</td>
</tr>
<tr>
<td>MIL-C-XXXXXX</td>
<td>&quot;Coatings, Visor Polycarbonate, Flying Helmet&quot;</td>
<td>Fast tape over scribed area</td>
<td>CS10F calibrase wheel 100 cycles/500gms</td>
</tr>
<tr>
<td>MIL-V-43511 (GL)</td>
<td>&quot;Visors, Flying Helmet, Polycarbonate&quot; (Class 1 and 2 only -- no coatings)</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>MIL-E-12397B</td>
<td>&quot;Eraser, Rubber-Pumice (For Testing Coated Optical Elements)&quot;</td>
<td>---</td>
<td>---</td>
</tr>
</tbody>
</table>
5.4 HIGH ABSORPTION

5.4.1 Evaporation of "Metal Lux" Materials

To determine whether clear visors could be coated with a highly absorbing optical film, it was decided to acquire some sample quantities of a special series of evaporation materials used for coating sunglasses.

Working through American Optical Co. in the U.S., three grades of materials were ordered from their supplier in Italy. The materials ordered were

- Gray Lux M776A (brown)
- Grisolux M797E (light gray)
- Rovilux M778 (blue)

Several test depositions were made using the vacuum evaporation system shown in Figure 5-23. Alumina crucibles and tungsten filaments were used to contain and heat the powdered materials. Test results did not show as high an absorption as was expected, and work was discontinued in favor of the tinted acrylic approach.

5.4.2 Tinted Plastics for Visors

The primary material used for IHMS/D visors is tinted acrylic (neutral gray) in a 1/8-inch sheet which has approximately 14 percent transmission at 5500 Å.

Acrylic was originally selected for use in helmet mounted sight and display programs because of its availability, ease of formability, and relatively good solvent resistance. It was selected over polycarbonate because several years ago when clear visors were used, the acrylic had better clarity and less
Figure 5-23. Thermal Evaporation System Used to Evaporate Metal Lux Materials
tendency to yellow than did polycarbonate. Only recently has polycarbonate been made with the same high transmission and ultraviolet stability as acrylic.

Now, however, IHMS/D systems use tinted acrylic visors to reduce intrinsic ghosting and to enhance pilot comfort in bright lighting conditions. Again, tinted acrylics were selected because of past experiences with clear acrylic and because at least one vendor (Rohm and Haas) offered a complete line of solar control acrylics ("Plexiglas") in two shades -- neutral gray and bronze (see Figure 5-24). Standard tints range from 16-76 percent transmission at 5500Å. Lower transmission shades down to 7 percent were obtained and measured on this program, but are considered special order (minimum quantities) materials by the vendor.

The largest supplier of polycarbonate is General Electric whose "Lexan" is available in two standard tints (smoke and bronze) but only one standard transmission (50 percent). Other shades of lower transmissivity are available on special order.

5.4.3 Ghost Ratios for Tinted Acrylic

IHMS/D applications cannot tolerate ghost images in displays where fine detail and contrast are required. To eliminate ghosting effects on the visor, one or more of the following optical techniques must be used:

- Place AR coating on outside visor surface
- Increase reflectance of inner surface
- Increase absorption of visor material

The three basic models have been examined in detail experimentally. In each model, dramatic improvements in ghosting ratio...
Figure 5-24. Cary 14 Transmission Scans of Various Tinted Acrylcs
reflectance of primary image can be achieved, but from fabrication cost and visor durability points of view, simply increasing the absorptance of the visor material is an extremely effective technique.

Sample calculations of the three models and families of ghost ratio curves can be found in Attachment No. 5 of this report.

Table 5-3 summarizes a few of the ghost ratio curves.

Table 5-3. Ghost Ratios for Various Visor Configurations
(Taken from curves in Attachment No. 5)

<table>
<thead>
<tr>
<th>Acrylic Tint (%T)</th>
<th>Dielectric Reflecting Coating (%R)</th>
<th>AR Coating (%R)</th>
<th>Ghost Ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>92</td>
<td>---</td>
<td>---</td>
<td>1</td>
</tr>
<tr>
<td>50</td>
<td>---</td>
<td>---</td>
<td>4</td>
</tr>
<tr>
<td>10</td>
<td>---</td>
<td>---</td>
<td>110</td>
</tr>
<tr>
<td>92</td>
<td>10</td>
<td>---</td>
<td>3</td>
</tr>
<tr>
<td>50</td>
<td>10</td>
<td>---</td>
<td>18</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
<td>---</td>
<td>310</td>
</tr>
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<td>95</td>
<td>---</td>
<td>9,500</td>
</tr>
<tr>
<td>50</td>
<td>95</td>
<td>---</td>
<td>38,000</td>
</tr>
<tr>
<td>10</td>
<td>95</td>
<td>---</td>
<td>&gt;100,000</td>
</tr>
<tr>
<td>92</td>
<td>---</td>
<td>0.5</td>
<td>9</td>
</tr>
<tr>
<td>50</td>
<td>---</td>
<td>0.5</td>
<td>33</td>
</tr>
<tr>
<td>10</td>
<td>---</td>
<td>0.5</td>
<td>830</td>
</tr>
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<td>92</td>
<td>10</td>
<td>0.5</td>
<td>25</td>
</tr>
<tr>
<td>50</td>
<td>10</td>
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<td>100</td>
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<tr>
<td>10</td>
<td>10</td>
<td>0.5</td>
<td>2,500</td>
</tr>
<tr>
<td>92</td>
<td>95</td>
<td>0.5</td>
<td>70,000</td>
</tr>
<tr>
<td>50</td>
<td>95</td>
<td>0.5</td>
<td>&gt;100,000</td>
</tr>
<tr>
<td>10</td>
<td>95</td>
<td>0.5</td>
<td>&gt;&gt;100,000</td>
</tr>
</tbody>
</table>
SECTION VI
REFERENCES


ATTACHMENT NO. 1
"OPTICS ON PLASTICS--VENDOR SURVEY LETTER"
October 20, 1972

Gentlemen:

I am interested in obtaining information in regard to your capabilities in the field of optics on plastics. Do you have brochures, catalogs or printed material in any of the areas detailed below?

- Protective (anti-abrasive) organic optical coatings
- Single layer thin film vacuum or sputter deposited coatings
- Multilayer thin film coatings
- Antireflective coatings
- Reflective coatings
- Beam splitters
- Band pass filters
- Photochromics
- Fabrication of plastic optical components

In lieu of prepared printed material, if it is not available, I would appreciate any information you may be kind enough to provide.

Sincerely,

Joseph L. Stevens
Materials Engineering
Mail Station R3636

JLS/jaj
ATTACHMENT NO. 2
STATUS REPORT--"TRICHRROICS ON PLASTICS"
DEVELOPMENT OF TRICHOIC COATINGS ON PLASTIC

PROGRAM OBJECTIVE

Work on this AMRL project commenced 3 April 1973. Original goals were to develop a functional trichroic optical coating on tinted acrylic material which had at least 85 percent reflectance at the P1 phosphor wavelength (5300 Å) when viewed at 37.5 degrees and which could be used as the reflecting surfaces on IHMS/D Model 8 visors.

SUMMARY OF RESULTS

As of 2 July 1973, more than 34 thin-film depositions containing over 81 different test samples (flats) have been made in the course of this program. Two sets of thin-film materials systems have been tested and depositions made on seven types of substrate materials.

Two trichroic films with slightly different attributes have been developed. They are:

- Thirteen-layer TiO₂/MgF₂ (on acrylic)
  - 98% R
  - ±750 Å half-band at 50% R
  - Passes MIL-M-13508B tape test
  - Fine microcrazing on surface
  - Physically stable at 52°C
AT2-3

- Thirteen-layer TiO$_2$/SiO$_2$ (on acrylic)
  - 90% R
  - ±650 Å half-band at 50% R
  - Will not pass tape test
  - No microcrazing as deposited
  - Physically unstable at 52°C

- Tests have been run which show quite conclusively that the major factor preventing successful deposition of the trichroic film is the propensity of plastics to craze under conditions of stress and temperature.

- The first-layer TiO$_2$ film is responsible for almost all of the temperature rise seen at the plastic surfaces during deposition.

- The microcrazing of the TiO$_2$/MgF$_2$ films represents a stress-relieved condition which is more physically stable and stronger than a TiO$_2$/SiO$_2$ film which is not microcrazed.

CONCLUSIONS

- The work to date on this program constitutes a significant technical advancement in the state-of-the-art of depositing complex optical coatings on plastics.

- To determine whether the microcrazing is detrimental to the optical performance of an IHMS/D system, it will be necessary to coat several visors and run a more complete optical and human factors laboratory evaluation.
• Although work to date has utilized flat test samples, it is expected that all deposition parameters and techniques can be readily transposed for use on curved-visor substrates.

• Additional work should be done to further minimize thermal/stress effects on the two trichroic systems developed to date -- also, another high index of refraction material with evaporation temperature lower than TiO$_2$ should be investigated.

• Continued development of the trichroic film on plastic will result in more reflective and more durable visor coatings.

RECOMMENDATIONS

• Test visors of both clear and tinted acrylic should be coated with a trichroic which exhibits microcrazing in order to determine whether this effect degrades visual see-through or distortion.

• The program to develop a trichroic coating on plastic visors for IHMS/D should continue -- the goals being to deposit films with reflectances greater than 99 percent, and which films are physically stable at maximum service temperatures and durable enough to withstand rigorous cleaning.
TECHNICAL DISCUSSION

Major variables investigated in this program included:

- Substrate materials
- Substrate cleaning
- Substrate bake-out
- Glow discharge exposure
- Deposition materials
- Deposition temperature
- Deposition rates
- Partial pressure \( O_2 \)
- Location of \( O_2 \) source
- Adhesion of films to substrate
- Third material pre-layer
- Number of layers (film thickness/stress)
- Time interval between layers
- Substrate temperature

A brief discussion of each of these variables follows, with an explanation of why they are critical to the development of trichroic coatings on plastics.
Substrate Materials

Substrate materials tested include:

- Plexiglas "G" acrylic (tinted) 80
- Plexiglas II acrylic (clear) 80
- Plexiglas 55 acrylic (clear) 80
- Plexiglas II acrylic (clear) overcoated with Abcite 80
- Polycarbonate 140
- Polysulfone 170

Other materials considered for future tests include:

- Polyarylene 290
- Polyarylsulfone ---
- Astral 360 260

The tinted and clear acrylics were of primary interest to this program. Other materials were tested only to determine if plastics with higher heat distortion or maximum service temperatures could be used to alleviate the thermal/stress crazing problem.

No crazing was experienced with glass substrates. A small but noticeable lessening of the crazing was seen in clear acrylic over tinted (13% T) acrylic -- probably due to the lower radiation absorption in the clear material.
Tests on the higher-temperature plastics were inconclusive, and should be retested along with the three remaining high-temperature plastics to determine whether the higher thermal stabilities, heat distortion temperature, hardresses, and strengths can influence or reduce the crazing problem.

Substrate Cleaning

A standard cleaning procedure used throughout the program consisted of:

- Twelve-hour soak in 3 percent detergent solution
- Swab with cotton balls (3 percent detergent)
- Deionized water rinse
- \( \text{N}_2 \) blow dry

The adhesive backing on the clear and tinted acrylics is water soluble, and the cleaning process listed above is an adequate procedure. The "Abcite" coated acrylic does not have a water-soluble adhesive backing and requires a petroleum ether swab in the cleaning process.

Substrate Bake-Out

Immersion of acrylic in water for a 24-hour period resulted in a sample weight increase of 0.16 percent. Either 38°C/7 hours or 66°C/1 hour air bake will drive off this absorbed water.

Because the bake-out appeared to worsen the resulting craze patterns of a deposited film, air baking was not used as a standard procedure. Figure 1 illustrates the effect of 77°C and 93°C prebakes on a 9-layer \( \text{TiO}_2/\text{MgF}_2 \) film.
a. Nine-Layer TiO\textsubscript{2}/MgF\textsubscript{2} (Run C-3102) Standard Clean, 85x

b. Nine-Layer TiO\textsubscript{2}/MgF\textsubscript{2} (Run C-4099) Standard Clean plus 77°C/1 Hour Air Bake

c. Nine-Layer TiO\textsubscript{2}/MgF\textsubscript{2} (Run C-3102) Standard Clean plus 93°C/1 Hour Air Bake, 85x

d. Nine-Layer TiO\textsubscript{2}/MgF\textsubscript{2} (Run C-3102) Standard Clean, plus 5-Minute Glow Discharge

Figure 1. Effects of Cleaning on Micro Crazing
Glow Discharge Exposure

Glow discharge exposure of acrylic substrates immediately prior to deposition of multilayer TiO$_2$/MgF$_2$ films was tested, but results were generally negative.

The glow discharge had two basic effects:

- It increased the size of the craze networks (see Figure 1)
- It caused reduced adhesion of the TiO$_2$/MgF$_2$ films in the acrylic

It may be stated that the glow discharge permitted the films to remain on the substrates in a more intact but highly stressed condition.

Glow discharge preconditioning was not used as a standard procedure throughout the test program.

A Honeywell-designed laser scatterometer was used to relate optical scattering to reflectivity of crazed trichroic films. Results showed that a TiO$_2$/MgF$_2$ coating deposited on an acrylic substrate with standard cleaning, had significantly lower scatter than nearly equivalent films deposited on pre-baked or glow discharge cleaned substrates.

Figure 2 illustrates a schematic of the laser scatterometer, and Figure 3 shows an X-Y plot of the scatter curves taken from three trichroic test samples.
Figure 2. Schematic of Honeywell Laser Scatterometer.
Figure 3. Laser Scatterometer Data, Trichroc Films on Acrylic
Deposition Materials

Two of the materials systems most commonly used to deposit optical films on glass were tested on this program. They are:

- TiO$_2$/MgF$_2$
- TiO$_2$/SiO$_2$

The TiO$_2$/MgF$_2$ system represents an excellent spread (differences) in high/low refractive indices. The higher the spread of values, the lower the number of layers required to obtain a given reflectance value. It was our hope to minimize the number of layers and thereby minimize the associated stress problems.

The TiO$_2$/SiO$_2$ system is perhaps most commonly used in industry to deposit trichroic coatings on glass and quartz, and was therefore included in the test program.

Other systems which should be tested in a future program should include first-layer materials which have melting/evaporation points lower than TiO$_2$, and possibly a less durable but easier-to-evaporate system such as ZnS/cryolite.

Deposition Temperature

The intention was to minimize thermal effects of the deposition and to maintain the plastic substrates at as low a temperature as possible. For this reason, almost all of the depositions were made at room temperature.
Some work was done, however, to super-cool the substrate prior to deposition by placing the substrate against a liquid-nitrogen-cooled plate. Results of the test were generally negative, with the trichroic film undergoing heavy crazing. It may be, however, that a lesser amount of cooling would have been beneficial.

**Deposition Rates**

Deposition rate tests using e-beam evaporation techniques were run on both the TiO$_2$/MgF$_2$ and TiO$_2$/SiO$_2$ systems. The following results were found:

- **TiO$_2$** -- High power, fast rates caused excessive heating of the substrates and hindered stoichiometric formation of the proper oxide state. Extremely low rates produced an obviously less durable film with poor adhesion. Moderate rates (10-15 Å/second) were best.

- **MgF$_2$** -- Deposition rates were not too critical. Rates near 30-40 Å/second were best.

- **SiO$_2$** -- Deposition rates were not critical as far as substrate heating was concerned, but faster rates again hindered stoichiometric formation of the SiO$_2$. (Note: The deposition of SiO$_2$ entailed sublimation of SiO in a partial pressure of O$_2$). Rates around 20-40 Å/second produced best results, although it was obvious that there was a considerable amount of SiO in the film.
Partial Pressure \( \text{O}_2 \)

Both the \( \text{TiO}_2 \) and \( \text{SiO}_2 \) films were sensitive to \( \text{O}_2 \) partial pressures during deposition. The technique used was to first evacuate the deposition chamber to the \( 10^{-6} \) torr range, then back-fill with high-purity \( \text{O}_2 \) to \( 8 \times 10^{-5} \) torr when the deposition was started.

Initial tests attempted to minimize temperature effects by minimizing the time during which the substrate was exposed to source radiation. To facilitate a fast deposition, the \( \text{O}_2 \) pressure was maintained throughout deposition of both the \( \text{TiO}_2/\text{MgF}_2 \) and \( \text{TiO}_2/\text{SiO}_2 \) films. It is not known whether magnesium oxyfluoride was formed and if it did, whether or not it was detrimental or beneficial. This is one variable that will have to be examined in a future program.

Location of \( \text{O}_2 \) Source

Contrary to the belief that a gas released into a pumped vacuum system is universally dispersed throughout the entire chamber, our tests showed that placement of the \( \text{O}_2 \) source is important.

If the \( \text{O}_2 \) source was placed near to and pointing at the substrate surface to be coated, the reflectance of the deposited film could be as much as 5 percent higher than if the \( \text{O}_2 \) source was located at the base of the chamber near the e-gun source.

Adhesion of Film to Substrate

Film/substrate adhesion was evaluated by "tape test" using a commercially available transparent tape.
In general, the TiO$_2$/MgF$_2$ coatings would pass a slow-to-moderately fast tape pull. The TiO$_2$/SiO$_2$ coatings would not pass even a slow pull.

It was concluded that, because the first layer is identical (TiO$_2$) in both systems, the adhesive strengths were related to the presence or absence of film stresses. Thus, the TiO$_2$/MgF$_2$ film which crazes during deposition is physically stable after deposition and is able to pass some level of tape test. On the other hand, the TiO$_2$/SiO$_2$ films are not crazed after deposition, are likely in a highly stressed condition, and when subjected to additional tape lifting stresses, exhibit adhesive failure.

This viewpoint is also reinforced by the fact that the TiO$_2$/MgF$_2$ films do not further craze on subsequent air bake at 52°C/20 hours, whereas a TiO$_2$/SiO$_2$ film which was previously craze-free will experience catastrophic crazing on heating to 52°C/20 hours.

Figure 4 illustrates the type of microcrazing which is typical of a TiO$_2$/MgF$_2$ trichroic on acrylic.

Figure 5 shows a TiO$_2$/MgF$_2$ film which has been partially stripped by tape to reveal the crazed plastic surface. This type of visual information has convinced us that it is the plastic surface which crazes first, thus causing the overlaying film to also craze in a replica fashion.

No changes in craze size or shapes are seen on heating the TiO$_2$/MgF$_2$ films to 52°C/20 hours.

Figure 6 shows a TiO$_2$/SiO$_2$ film after 52°C/20 hours heating. Before heating, the film/substrate surface was perfectly flat and craze-free.
Figure 4. Stress Crazing of 9-Layer TiO$_2$/MgF$_2$ Film on Acrylic, 85x

Figure 5. Crazed Acrylic Surface Revealed by Tape Pulling TiO$_2$/MgF$_2$ Film (85x)
Figure 7. Comparison of Cary 14 Reflectance Curves for TiO$_2$/MgF$_2$ and TiO$_2$/SiO$_2$ on 2074 Plexiglas Trichroics
Future work should be aimed at increasing the reflectance curves of the TiO$_2$/MgF$_2$ system to greater than 99 percent. It is expected that 17- or 19-layer coatings will be required.

**Time Interval Between Layers**

Initial tests emphasized speed and no time lag between layers. This is a standard approach by most optical coatings vendors who deposit trichroics on glass and quartz.

Perhaps the most significant technical finding of the current program is that a definite time interval between layers is required to reduce crazing of the coatings.

Our tests definitely showed that there should be at least a 4-minute interval between layers. Figures 8 and 9 show the benefits of this time lag between layers in increasing the reflectance curve for the TiO$_2$/MgF$_2$ system.

It became obvious at this point that temperature effects were manifesting themselves in subtler ways than the simple and more obvious signs of crazing. With no time interval between layers, the greater-number-of-layer coatings will become increasingly more crazed and subsequently produce lower reflectance values.

**Substrate Temperature**

A small iron-constantan thermocouple was placed through the side of an acrylic test sample with the bead near the surface to be coated. Temperature measurements made during the course of several depositions revealed the following information:
Figure 6. Nine-Layer TiO$_2$/SiO$_2$ Film and Acrylic Substrate After 52°C/20 Hours in Air Bake
Third Material Pre-Layer

Both CeO$_2$ and MgO $\lambda/4$ layers were deposited on the acrylic surface in a test to determine whether a third pre-layer would enhance adhesion of the TiO$_2$ film. While CeO$_2$ is normally an excellent bonding aid to acrylic, the addition of the TiO$_2$, MgF$_2$, and SiO$_2$ overlayers was apparently stressy enough to overcome the acrylic/pre-layer bond strength. If anything, the deposited trichroics displayed more extensive and finer crazing.

Number of Layers (Film Thickness/Stress)

On one hand, it is important to minimize the number of layers so that film stresses can also be minimized. On the other hand, it is important to maximize the number of layers so that a maximum reflectance of the trichroic can be obtained.

In both the TiO$_2$/MgF$_2$ and TiO$_2$/SiO$_2$ systems deposited on acrylic, there is a certain maximum number of layers that can be deposited without crazing -- above which the crazing commences, becomes more and more severe, and eventually destroys the entire film. About six or seven layers of TiO$_2$/MgF$_2$ can be deposited before crazing starts; and about 13 layers can be deposited before the reflectance value starts to drop. For TiO$_2$/SiO$_2$, crazing can be avoided up to 13 layers, but as stated previously, the film is extremely stressy and unstable. The reflectance for the 13-layer TiO$_2$/SiO$_2$ system is also not as good as the reflectance for the 13-layer TiO$_2$/MgF$_2$ system because of the narrow spread of index of refraction values.

A composite family of reflectance curves for 5, 9, and 13-layer TiO$_2$/SiO$_2$ trichroics is shown in Figure 7, along with an equivalent 13-layer TiO$_2$/MgF$_2$ trichroic.
Figure 8. Effects of Time Interval Between Layers of 9-layer TiO$_2$/MgF$_2$ Trichroic on 2074 Plexiglas.
Figure 9. Effects of Time Interval Between Layers of 11-layer TiO₂/MgF₂ Trichroic on 2074 Plexiglas
Neither the SiO\(_2\) nor MgF\(_2\) depositions showed a significant temperature rise on the acrylic surface.

TiO\(_2\) is responsible for nearly all of the temperature rise seen on the acrylic surface.

The faster the deposition rate (higher e-beam power levels), the higher the resultant temperature rise for a given coating thickness.

The higher the starting substrate temperature, the greater the temperature rise for any given deposition rate.

Typical values for single-layer depositions are in the following tabulation:

<table>
<thead>
<tr>
<th>Material</th>
<th>Deposition Rate ((\frac{1}{\text{sec}}))</th>
<th>Initial Substrate Temperature ((^\circ\text{C}))</th>
<th>(\Delta T) Increase ((^\circ\text{C}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>(\lambda/4) MgF(_2)</td>
<td>30-40</td>
<td>22</td>
<td>1</td>
</tr>
<tr>
<td>(\lambda/4) SiO(_2)</td>
<td>20-40</td>
<td>22</td>
<td>1</td>
</tr>
<tr>
<td>(\lambda/4) SiO(_2)</td>
<td>20-40</td>
<td>50</td>
<td>2</td>
</tr>
<tr>
<td>(\lambda/4) TiO(_2)</td>
<td>10-15</td>
<td>22</td>
<td>20</td>
</tr>
<tr>
<td>(\lambda/4) TiO(_2)</td>
<td>10-15</td>
<td>45</td>
<td>30</td>
</tr>
<tr>
<td>(\lambda/4) TiO(_2)</td>
<td>15-20</td>
<td>50</td>
<td>40</td>
</tr>
</tbody>
</table>

A 4-minute interval between layers (and especially after the TiO\(_2\) layers) is the minimum time required for temperature stabilization. Five or six minutes is probably better.
An example of the temperature rise without a time interval is shown in Figure 10 for a 4-layer TiO$_2$/SiO$_2$ deposition.

A similar chart for the last three layers of a 7-layer TiO$_2$/SiO$_2$ system with 4-minute intervals is shown in Figure 11.

Towards the end of the current program, a new optical evaluation tool was used to gain more information on film stresses. The tool was a Twyman-Green laser interferometer (6328Å). First, an acrylic test piece was acquired which had a surface flatness on the order of 1-3 fringes. Using the laser interferometer with camera attachment, a photograph was taken of the test piece. Figure 12 shows a typical photo of the interference pattern.

Having documented the part's flatness, a 2-layer TiO$_2$/SiO$_2$ was deposited onto the part and another photograph taken of the new flatness pattern. Results showed that the deposition of the film had caused the acrylic to go out of flat -- concave at the deposited surface -- and that the part was now only 10-20 fringes flat.

The conclusion was reached that the laser interferometer could be a valuable tool for measuring deposited film stresses.
Figure 10. Temperature Rise on Acrylic Surface Due to Deposition of 4-layer TiO₂/SiO₂ Film -- No Time Interval
Figure 11. Temperature Rise on Acrylic Surface Due to Deposition of 7-layer TiO$_2$/SiO$_2$ Film -- 4-minute Intervals
Figure 12. Photograph of Acrylic Test Piece as Examined with a Laser Interferometer
ATTACHMENT NO. 3
ABRASION RESISTANT COATINGS FOR PLASTIC OPTICS
(HELMET SIGHT/DISPLAY VISORS)

- Feasibility Study -
ABRASION RESISTANT COATINGS FOR PLASTIC OPTICS
(HELMET SIGHT/DISPLAY VISORS) - FEASIBILITY STUDY

Prepared for:
Aerospace Medical Research Laboratory
Attn: AMRL/HER (FY9378)
Contract: F33615-72-C-0420/PZ0002
Phase II Paragraph 3.2.3.1.5
Wright-Patterson AFB, Ohio 45433

Prepared by:
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Minneapolis, Minnesota 55413
SECTION I
INTRODUCTION

1.1 PURPOSE OF DOCUMENT

This document is a final report of a feasibility study on visor durability and abrasion resistance, performed by the Honeywell GAP Division's Plastic Laboratory under the "Coatings Technology" portion of the IHMS/D contract No. F33615-72-C-0420/PZ0002, Phase II Paragraph 3.2.3.1.5 of the revised Technical/Cost Proposal.

1.2 STATEMENT OF WORK

Phase II was to "--encompass the construction of several improved models of the IHMS/D that will serve to define problems, anticipated performances, hardware options, and final system design of the Mark II, Mod O (Model 8) IHMS/D."

Paragraph 3.2.3.1.5 of Phase II was entitled "Coating Technology", and involved conducting a survey of the state-of-the-art in coatings, and obtaining and evaluating samples of candidate coatings, including variable density filtering for the visor to possibly introduce it into the Mark II, Mod O (Model 8) program. One specific task of the "Coatings Technology" program involved studying the visor's durability and abrasion resistance, which is the subject of this report.
1.3 PROGRAM OBJECTIVE

The program was to evaluate several state-of-the-art coatings which might be used on IHMS/D visors to add durability, and abrasion and solvent resistance. Another aspect of the program was to examine the feasibility of using an in-house facility to apply the abrasion-resistant coatings to IHMS/D visors.

1.4 MATERIALS TESTED

**Primers:**
- XZ-8-5066, epoxy-silane, Dow Corning
- DC-1205, epoxy-silane, Dow Corning
- A-1100, gamma aminopropytriethoxysilane, Union Carbide
- MC7134-01, epoxy-polyamide, Honeywell Inc.
- MS6293H, epoxy-amine, Honeywell Inc.
- MS6020H, epoxy-amine, Honeywell Inc.

**Coatings:**
- X-1-2900, silicone resin, Dow Corning
- XR-4-3143, silicone, Dow Corning
- E1847-85-4, silicone, Dow Corning
- T-650, silicone, Owens Illinois
- XD-8157L, Zwitterion, Dow Chemical
- Abcite (Abcite coated acrylic', DuPont

**Miscellaneous:**
- MS6626, ethyleneglycolmonobutylether
- n-Butyl alcohol, AR grade
- Methyl alcohol, AR grade
- Xylene, AR grade
- Freon 113, DuPont
- TT-I-735, isopropyl alcohol
Based on the limited investigation of hard coatings to protect plastic optics, such as the IHMS/D visor, several conclusions can be made:

1. An in-house facility is feasible for applying hard coatings to protect plastic optics such as the IHMS/D visor. However, this feasibility study and other similar efforts indicate that a considerable amount of developmental effort, equipment, and time will be required to accomplish this task.

2. Because of the subjective nature of the tests used, clear-cut comparison between coatings was difficult. Further effort with coatings should be expanded to make use of objective test procedures to facilitate coating comparison. However, of the six coatings investigated (XR-4-3143, E1847-85-4, X-1-2900 and T-650 silicones, XD-8157L Zwitterion, and Abcite), Abcite showed the best all around properties. The silicone coatings were roughly of equivalent hardness, with the T-650 being slightly softer and the XR-4-3143 and X-1-2900 slightly harder than Abcite. The E1847-85-4 coating was the least hard and least abrasion resistant of all the coatings. Silicone coatings required a primer for adhesion. Coating uniformity was a major problem encountered in this investigation.

3. Five primer systems were developed and evaluated to improve the adhesion of silicone coatings (XR-4-3143 and X-1-2900). The primer which appeared to work the best with X-1-2900 silicone was DC1205 epoxy-silane. According to the manufacturer, the XR-4-3143 silicone coating is no longer available.
4. This cursory evaluation of hard coatings showed that there may be a number of coating materials available which can be developed for protecting plastic optics. In this brief study, a number of promising materials were found. It is expected that further, more detailed investigation of hard coatings may provide still more promising candidates for protection of plastic optics.

5. Application of a uniform coating free of particulate contamination was not achieved in this study, regardless of the precautions. The use of conventional dip coating equipment resulted in rippled coatings due to equipment vibrations. Better equipment may have alleviated this problem. Dip coating in a semi-clean area and filtering the coating through 5-micron filters reduced but did not eliminate particulate contamination on the coated item. Better filtration and processing in a high-grade clean room may alleviate this problem.
SECTION III
RECOMMENDATIONS

1. Since this limited investigation uncovered a number of promising coatings, additional effort would be expected to expand the number of coating candidates available for further development. Further investigation of hard coatings for protecting plastic optics is therefore recommended.

2. The feasibility of using any of the hard coating candidates on typical plastic optics parts should be determined by comparison with the Abcite material and process. The cost of either licensing a process, purchasing the service or development of an in-house capability should be compared.
High transparency plastics are being used with increasing frequency for windows, plastic optics, visor and similar applications. These materials usually perform their function quite well, whether it be for low-cost optics, impact resistance, to lighten weight, or any one of a number of other applications. Most of these materials have a very low resistance to abrasion. Very hard, clear plastic coatings to protect the softer plastic part are being used with greater frequency.

Materials and processes to provide hard plastic coatings must be compatible with the surface to be coated. For acrylic surfaces, this would mean using a cure temperature not exceeding 170°F. Also, the coatings must be thin, uniform, and free of particulate contamination.

Acrylic plastic visors used by the helmet sight program are of course subjected to handling typically given to similar pilot visors. A hard protective coating on the helmet sight visor and other plastic optic systems would provide abrasion protection.

A proprietary coating by DuPont called Abcite has been considered. It is believed to be applied as a 5-micron film and is considered by some to be the best of those available; however, its highly proprietary nature makes it unattractive for immediate use. The study reported herein investigated alternative hard coating techniques and materials for potential IHMS/D use. The goal of the study was to gain experience with the materials, their application processes, and their limitations.
SECTION V
PROCEDURE, RESULTS AND DISCUSSIONS

5.1 LITERATURE SEARCH

There has been considerable interest in hard coatings to protect plastics. Therefore, it is not surprising that a fairly large number of companies have developed hard coatings. A cursory search of vendors of hard plastic coatings which are intended for or could be used to protect plastics revealed no less than 13 companies and 15 candidate materials. (See Table 5-1.)

Vacuum deposited coatings were not considered in the feasibility study since the materials deposited require facilities which were unavailable at the time. Also, most of the literature reviewed on this subject (Union Carbide's Parylene process and General Electric's line-of-sight process) indicated that the films produced would not be hard enough. However, it should be pointed out that vacuum depositing coatings may be the best way to deposit and control ultra-thin coatings for visor protection. A greater literature search effort is needed in this area. Some literature on this subject may be found in the bibliography.

5.2 MATERIAL SELECTION

Because of funding limitations, only a few coatings could be tried. One of the more commonly used coatings for protecting plastics from abrasion is Owens Illinois T-650. This material was therefore believed to be a candidate for visor coating. Previous experience with XR-4-3143 from Dow Corning showed this material to have an exceptional hardness and to be amenable to thin film. Material adhesion at one time was a problem, which
Table 5-1. Hard Coatings for Protecting Plastics

<table>
<thead>
<tr>
<th>Company Name</th>
<th>Material Designation</th>
</tr>
</thead>
<tbody>
<tr>
<td>American Optical</td>
<td>AO-50M</td>
</tr>
<tr>
<td>Ball Chemical Company</td>
<td>MBT</td>
</tr>
<tr>
<td>Dow Chemical Company</td>
<td>XD-8157L</td>
</tr>
<tr>
<td>Dow Corning Corporation</td>
<td>XR-4-3143, X-1-2900</td>
</tr>
<tr>
<td>DuPont</td>
<td>Abcite</td>
</tr>
<tr>
<td>Fortin Plastics</td>
<td>Fortin</td>
</tr>
<tr>
<td>General Electric</td>
<td>Lexan MR-400</td>
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<tr>
<td>Gray Cote Company</td>
<td>Gra-Cote</td>
</tr>
<tr>
<td>Mark-Sion Polarized Corporation</td>
<td>Proprietary</td>
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<tr>
<td>Mitsubishi Rayon</td>
<td>Ultra MR, MR</td>
</tr>
<tr>
<td>Omnitech</td>
<td>Omnitech</td>
</tr>
<tr>
<td>Owens-Illinois</td>
<td>T-650</td>
</tr>
<tr>
<td>Rohm and Haas</td>
<td>Abrasion Resistant Coating 360</td>
</tr>
<tr>
<td>Sierracin Corporation</td>
<td>Sierracin 311</td>
</tr>
</tbody>
</table>

was solved by using the right primer. This material was therefore included as a candidate. After development work on this coating had been done, it became no longer available. A replacement material, X-1-2900, was then used in its place as a visor coating candidate. Another candidate coating was also selected for evaluation. This coating, designated XD-8157L by Dow Chemical, was not available for sampling. However, the manufacturer agreed to coat test samples with it without charge. The last coating used was Abcite. This material is of course not available as a coating, except under the exclusive licensing arrangement from DuPont. Abcite-coated acrylic is available and was, therefore, used for comparison.
5.3 EFFECT OF POLYMER CHEMISTRY

It is interesting to note the effect of chemistry on the properties required in a hard organic coating for protecting softer plastics. Generally, linear polymers tend to be soft and somewhat stretchy. Examples of linear polymers are polyethylene, polypropylene, poly(paraxylylene) (Parylene N and C). Some linear polymers appear to be hard but are far too soft for the Helmet Sight visor. Examples of apparently hard, but actually soft linear polymers are polyacrylate, polycarbonate and polysulfone.

Generally, polymers which are cross-linked tend to be harder than linear polymers. The greater the degree of cross-linking in the polymer, the greater the hardness of the polymer within its polymer class. Examples of cross-linked polymers are epoxies and phenolics. Many other polymer types are amenable to varying degrees of cross-linking, making possible plastic materials with wide latitude of hardness such as polyurethanes and silicones.

Silicone polymers pose some rather interesting contrasts in physical properties. They are widely known as soft, rubbery (RTV) materials. These of course are basically of the linear variety. Less widely known are cross-linked silicones. This class of materials is peculiar in that polymers possessing extreme hardness, as well as low surface tension are possible at high cross-link density. It is not surprising then that a number of the hard coatings for protecting plastics from abrasion are high cross-link density silicones. Virtually all of the silicone coatings evaluated for this feasibility study were of this class. These included Owens Illinois T-650, Dow Corning XR-4-3143 and X-1-2900 and to a lesser degree of cross-linking Dow Corning E1847-85-4.

Another interesting candidate coating for the visor was the high cross-link density Zwitterion polymer (XD-8157L) recently developed by Dow Chemical. This coating may be applied from a water solution, thus avoiding many of the incompatibility problems with mutual optical coatings resulting from solvent base coatings. Through heating, the polymer cross-links to a hydrophobic hard coating.

2079-SR3
5.4 SAMPLE PREPARATION

The approach used in this work was that of developing sufficient technology to effectively use a hard coating on a typical visor. Many experiments were performed with the various hard coatings selected for test. Emphasis was placed on the X-1-2900 system since it appeared to show the most promise for the feasibility test. A large portion (approximately one third) of the effort was required for the development of a primer system for the hard coatings. These efforts were carried to the point where a single hard coat/primer system showed promise on acrylic slides. The tentative processing techniques were used to apply the hard coat/primer system to two full-sized helmet visors. Evaluation of the coated visors concluded the feasibility study.

Test Substrates

Samples of the candidate coatings were evaluated on tinted acrylic substrates. Tinted acrylic was cut into slides of 1/8 inch x 1 inch x 3 inch. The edges of the substrates were sanded smooth and the corners rounded. Protective paper was peeled from the substrate. Examples of coated substrates are shown in Figure 5-1.

Substrates were cleaned by a three step sequence using isopropyl alcohol, detergent, and Freon 113. They were immersed in isopropyl alcohol for a few minutes, gently brushed with a camel hair brush, and then rinsed with clean isopropyl alcohol. The substrates were then immersed for a few minutes in a detergent solution consisting of five percent Sep-Ko in deionized water, followed by a rinse with deionized water. They were then immersed in Freon 113 for a few minutes and allowed to dry in a laminar flow booth.

To facilitate the readability of the report and eliminate unnecessary repetition, primer and coating formulations are listed in Appendixes A and B.
Figure 5-1. Hard-Coated Tinted Acrylic "Slides" Ready for Evaluation
5.5 TESTS PROCEDURES

Coatings were evaluated for hardness, abrasion resistance and adhesion. These tests were primarily used as a means of identifying gross differences between materials. The tests were in no way meant for anything other than providing a quick method of differentiating desirable characteristics from less desirable characteristics. All of the physical tests performed were subjective, and are described below.

**Abrasion Resistance**

Surfaces were rubbed with an eraser for a total of 20 strokes under moderate pressure. The eraser used was that from an Acro-Rite #2 pencil. Relative resistance to abrasion was judged by the degree of base left after removal of eraser debris.

**Hardness**

Hardness was evaluated by indenting the coated surface with a sharp scalpel (Propper #23 blade) under 15X magnification. After indentation, the coating was scraped with the blade of the scalpel at 90 degrees to its surface. Relative hardness was judged by the resistance of the coating to indentation and scraping.

**Adhesion**

Adhesion was the least subjective test. It was checked by cross-cutting through the coating with a scalpel (Propper #23 blade). Scotch #250 tape was placed over the cut and firmly pressed onto the coating. The tape was
then quickly jerked from the coating at 90 degrees to the surface. Any loss of coating adhesion was recorded as a failure.

**Thickness**

In early experiments coating thickness was visually observed under 15X magnification. Later tests made use of a clutch micrometer accurate to 0.0001 inch, as well as an electronic film thickness measuring device (Permascope, Model EC).

Data from evaluating the coatings is summarized in Table 5-2.

**HARD COATINGS**

**XR-4-3143 with Various Primers**

_Epoxy-Polyamide_ --Several slides were primed with a 20-percent solids solution of an epoxy-polyamide coating (MC 7134-01) by dip coating at a two inch/minute withdrawal rate. The coating was allowed to air-dry five minutes, then partially cured seven to eight minutes at 150°F. When cooled to room temperature, the slides were coated with XR-4-3143 by dip-coating at a two inch/minute withdrawal rate. The coating was cured for one hour at 170°F.

The top coat of the coated slides attacked the primer. This was observed as cloudiness between the primer and top coat, as well as a disruption of the primer film. Microscopic examination of the coated slides showed that the prime coat was thicker than the top coat. Abrasion resistance testing of the three surfaces showed the XR-4-3143 to be the most resistant, the acrylic next and the primer the least resistant to abrasion. The hardness of the
Table 5-2. Summary of Results from Protective Hard Coatings for Plastic Optics

<table>
<thead>
<tr>
<th>Coating</th>
<th>Primer</th>
<th>Abrasion Resistance (Eraser)</th>
<th>Adhesion (Cross-Cut Tape)</th>
<th>Hardness (Scalpel Cut)</th>
<th>Coating Uniformity</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>XR-4-3143 Silicone</td>
<td>MC7134-01 Epoxy-Polyamide (20%)</td>
<td>1</td>
<td>1</td>
<td>10</td>
<td>5</td>
<td>Very soft primer</td>
</tr>
<tr>
<td></td>
<td>MS6020H Epoxy-Amine (10%)</td>
<td>1</td>
<td>1</td>
<td>5</td>
<td>9</td>
<td>Soft primer</td>
</tr>
<tr>
<td></td>
<td>DC1205 Epoxy-Silane (5%)</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3</td>
<td>None</td>
</tr>
<tr>
<td>T-650 Silicone</td>
<td>XZ-8-5066 Epoxy-Silane (5%)</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>4-5</td>
<td>None</td>
</tr>
<tr>
<td>X-1-2900 Silicone 1</td>
<td>A-1100 Silane (1.5%)</td>
<td>3</td>
<td>2</td>
<td>3</td>
<td>3</td>
<td>Thick coating</td>
</tr>
<tr>
<td></td>
<td>DC1205 Epoxy-Silane (1%)</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3-10</td>
<td>None</td>
</tr>
<tr>
<td></td>
<td>XZ-8-5066 Epoxy-Silane (1%)</td>
<td>10</td>
<td>10</td>
<td>--</td>
<td>6-10</td>
<td>No adhesion</td>
</tr>
<tr>
<td></td>
<td>A-1100 Silane (1%)</td>
<td>10</td>
<td>10</td>
<td>--</td>
<td>9-10</td>
<td>No adhesion</td>
</tr>
<tr>
<td></td>
<td>DC1205 Epoxy-Silane (1%)</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>3</td>
<td>None</td>
</tr>
<tr>
<td>E1847-85-6 Silicone</td>
<td>None</td>
<td>8</td>
<td>1</td>
<td>7</td>
<td>6</td>
<td>Coating too soft</td>
</tr>
<tr>
<td>XD-8157L Zwitterion</td>
<td>(Applied by Dow Chemical)</td>
<td>4</td>
<td>1</td>
<td>4</td>
<td>4</td>
<td>Thick coating</td>
</tr>
<tr>
<td>Abcote</td>
<td>(Abcote coated acrylic supplied by DuPont)</td>
<td>1-2</td>
<td>1</td>
<td>1-2</td>
<td>1</td>
<td>None</td>
</tr>
</tbody>
</table>

1. X-1-2900 used at 40% solids.
2. X-1-2900 used at 50% solids, prediluted catalyst.

Results are based on a scale of 1-10 where values approaching unity are more desirable than those approaching 10. All ratings are strictly subjective. Controlled laboratory analysis should be used to further compare materials.

Three surfaces showed a similar correlation. Adhesion of the top coat to the primer was good, but due to the relative softness of the primer and its excess thickness, the top coat could be peeled from the primer by moving the primer (see Table 5-2). Two things became apparent: (1) the primer should be applied in a film much thinner than the top coat; and (2) the primer, if applied in a finite thickness, should be much harder than that attainable from an epoxy-polyamide. Amine-cured epoxies are generally harder than polyamide-cured epoxies. The following experiment used an amine-cured epoxy primer.

Epoxy-Amine -- Several slides were cleaned as before and coated with a 10 percent solids solution of an epoxy-amine (MS6020H) at a 4 inch/minute withdrawal rate, air-dried for 60 minutes, and cured 16 hours at 170°F. Observation showed that the primer had "crawled." This condition is symptomatic of amine-cured epoxies. Information on the compatibility of the primer and
hard coat was needed, so the slides were used regardless of the discontinuities of the primer. The primed slides were dip coated at 4 inches/minute with XR-4-3143 which was then cured two hours at 200°F.

Examination of the coated slides under 15X magnification showed the primer to be much thinner than that of the previous test, but still well in excess of that required. No loss of adhesion was observed. The abrasion resistance of the acrylic substrate was roughly equivalent to the coating. A similar correlation in hardness was observed. (See Table 5-2.)

"Crawling" of an epoxy-amine coating can sometimes be corrected by increasing the molecular weight of the amine. This is accomplished by reacting a quantity of the epoxy resin with an excess of the amine. Thus, the next test was carried out using a prepolymer amine-epoxy primer.

Slides were coated with the prepolymer amine-epoxy solution as before. The primed slide was coated as previously described with XR-4-3143. Evaluation of the surfaces for adhesion, abrasion resistance and hardness showed no change from the previous test. Visual examination revealed a minor improvement in film continuity. Effort on this primer system was abandoned.

During the series of three tests just described it became apparent that minute particulate contamination grossly affects the appearance of the coatings. While most of the handling and application of coatings was done in a laminar flow booth, the coating films were contaminated by very small particulate contamination. Obviously the hard coating material was very sensitive to contamination.

Epoxy-Silane -- Several slides were cleaned in the usual manner and primed with DC 1205 epoxy-silane primer at a 5 percent solids by dip-coating at a 4 inch/minute withdrawal rate. Another set of slides were primed with XZ-8-5066 epoxy-silane at 5 percent solids in the same manner. Both primers
AT3-19

were allowed to air-dry 30 minutes, and cured for 45 minutes at 170°F. The primed slides were coated with XR-4-3143 by dip-coating at 4 inches/minute, followed by curing for 2 hours at 170°F. The coated slides were evaluated for abrasion resistance and adhesion. (See Table 5-2.)

The adhesion of XR-4-3143 to the primed acrylic slide was found to be good. Abrasion resistance of the coating was the same as previously noted in other tests. The primers could not be evaluated for abrasion resistance since there was no evidence of a film thick enough for testing. Both primers showed "rainbow" effect fringes typical of films in the micron thickness range. It was learned that XR-4-3143 was no longer available, but that a new material X-1-2900 was recommended as a substitute. To effect a low temperature cure, X-2504 catalyst was recommended. Samples of this material were ordered from Dow Corning.

T-650 With A-1100 Silane

A hard coating for plastics developed by Owens-Illinois was purported to be one of the better ones available. This material was designated T-650, and was to be used with a silane adhesion promoter for best results. Several slides were cleaned in the usual manner and primed with a one percent A-1100 amino-functional silane in de-ionized water. The primer was air-dried 60 minutes, and then force-dried for 2 hours at 170°F. The primed slides were dip-coated with a 30 percent solids solution of T-650 at a 4 inch/minute withdrawal rate. The coating was air-dried 15 minutes, then cured for 48 hours at 170°F.

Abrasion resistance and adhesion were determined in the usual manner and compared to the epoxy-silane XR-4-3143 systems. The T-650 coatings was judged harder and more abrasion resistant than the acrylic, but less than the XR-4-3143. It had good adhesion to primed acrylic. The T-650 coating
appeared to be much thicker than the XR-4-3143 coatings (see Table 5-2). Further work with this coating would be to produce thinner films. However, no further work with T-650 had been planned until after evaluation of X-1-2900. This material was expected to have a hardness and abrasion resistance equivalent to that of XR-4-3143 which, as indicated above, was better than that of the T-650. Another reason for not pursuing development of the T-650 material was the relatively long high-temperature cure required. This type of cure would be incompatible with acrylic optics because of the potential risk of dimensional changes in plastic optics during thermal exposure.

X-1-2900 Tests

Use with Various Primers -- A series of tests was planned for evaluating the X-1-2900/X-2504 coating. Several acrylic slides were cleaned in the usual manner. One slide was primed with a one percent solids solution of DC-1205 epoxy-silane by dip-coating at a withdrawal rate of 4 inches/minute. The primer was air dried 30 minutes then cured for 45 minutes at 170°F. Another slide was primed with a one percent solution of XZ-8-5066, air-dried for 30 minutes, and then cured for 45 minutes at 170°F. A third slide was primed with a one percent solution of A-1100 in n-butyl alcohol, air-dried for 30 minutes, then force dried for 10 minutes at 170°F.

All three primed slides were coated with X-1-2900/X-2504 without dilution at a withdrawal rate of 4 inches/minute. The coating was cured 10 hours at room temperature followed by post-curing 2 hours at 170°F. The coated slides were evaluated for hardness, abrasion resistance and adhesion in the usual way.

Test results showed that the X-1-2900 coating was much too thick to be of use for plastic optics. Shrink marks indicated excessive thickness, and adhesion was only fair. The coating showed the same degree of hardness as XR-4-3143. Further development was needed to reduce the film thickness.
Effect of Solvent on Coating Uniformity -- The solids content of the undiluted coating was found to be 80 percent. That of the XR-4-3143 was 50 percent. The 80 percent coating would require solvent reduction for thinner films. The next series of tests describes the effect of reduced solids content on the coating performance of X-1-2900.

Several slides were cleaned in the usual manner and primed with one percent solids solutions of XZ-8-5066, DC-1205 and A-1100, and cured as noted previously. The primed slides were dip-coated at a withdrawal rate of 4 inches/minute with a 40 percent solids solution prepared by dilution in a ratio of one-to-one by weight with methyl alcohol. The coating was cured for 16 hours at room temperature then post-cured for 2 hours at 170°F.

Adhesion and abrasion resistance was evaluated in the usual manner (see Table 5-2). Visual observations were performed under 15X magnification. The X-1-2900 coating had good adhesion to acrylic primed with DC-1205, and poor adhesion to acrylic primed with either XZ-8-5066 or A-1100. Abrasion resistance correlated with that of XR-4-3143. The coating had an orange peel surface. This was puzzling since this condition is seldom observed with low solids coatings applied by dip coating. This problem can often be solved by adding a small quantity of a high-boiling solvent.

Several slides were cleaned in the usual manner and primed with one percent of DC-1205, and air-dried for 30 minutes by dip coating at 4 inches/minute. The primer was air-dried for 30 minutes, then cured for 45 minutes at 170°F. Half of the slides were coated with a 50 percent solution of X-1-2900 by dip-coating at 4 inches/minute. The other half of the slides were coated in the same manner with a 50 percent solution of X-1-2900 which had been modified with 2 percent xylene by weight. All coatings were cured 16 hours at room temperature then postcured 2 hours at 170°F.
Coated slides were examined for an orange peel effect. There appeared to be no significant difference between the slides coated with xylene-diluted material and those coated in the usual way as far as the orange peel problem was concerned.

**Effect of Catalyst Dilution** -- All details involved in the preparation and application of the coating were reviewed. It had been noted that the addition of X-2504 catalyst to X-1-2900 resin produced stringing at the points of contact of the two liquids. The stringing disappeared with continued stirring. This effect is usually the result of rapid polymerization at the point of catalyst/resin contact. The results may or may not show up in the applied film. In this situation it was believed that the orange peel effect may have been the result of stringing from the impact of resin and catalyst. It was believed that dilution of the X-2504 catalyst prior to addition to X-1-2900 resin may alleviate the orange peel problem.

Several slides were cleaned in the usual manner and coated with one percent DC-1205 epoxy-silane primer by dip coating. The primer was cured 45 minutes at 170°F. The X-2504 catalyst was diluted with methyl alcohol in a ratio of 0.5 g catalyst to 60 g alcohol. The diluted catalyst was added to 100 g X-1-2900 resin to give a 50 percent solids solution. Primed slides were dip coated at 4 inches/minute, cured for 16 hours at room temperature, and post-cured for 2 hours at 170°F.

Adhesion and abrasion resistance of the coatings were compared to XR-4-3143 coated slides. Visual observations were made under 15X magnification. The X-1-2900 coating prepared by predilution of the catalyst showed none of the orange peel condition noted previously. The use of xylene as a latent solvent/leveling agent had no benefit. The coating was adherent and exhibited abrasion resistance equivalent to the XR-4-3143 coating. (See Table 5-2.)
Effect of Solids Content on Coating Thickness -- Another problem was to correctly determine coating thicknesses. Up to this point, coating thickness had been determined by measuring the difference between coated and uncoated slides by visual observation and also with a clutch micrometer accurate to 0.0001 inch. It had been determined that the thickness of the 1/8 in x 1 in x 3 in acrylic slides varied by as much as 0.0003 inch. Thus, coating thickness measurements were subject to inaccuracies by the degree of slide thickness variations. This was not a critical problem at this time but one which required a solution so that both solids content of the coating and dip coating rate could be established for a known coating thickness. This could be accomplished by using aluminum panels as the coating substrate and an electronic film thickness measuring device.

A series of chromate treated (MIL-C-5541, Class 1A) aluminum panels were cleaned with isopropyl alcohol. The panels were primed with one percent DC-1205 by dip-coating at a 4 inch/minute withdrawal rate. The primer was air-dried for 30 minutes at room temperature, then cured for 45 minutes at 170°F. Coatings of X-1-2900/X-2504 were prepared by pre-diluting the catalyst with methyl alcohol to get 40, 50, 60 and 70 percent solids solutions. The coatings were applied to the primed aluminum panels by dip-coating at 4 inches/minute cured for 16 hours at room temperature then post-cured 2 hours at 170°F. Coating thickness measurements were made with the Permascope Model EC. These data are found in Table 5-3.

Effect of Primer Cure, Coating Solids Content and Dip Rate -- Additional tests were run to determine the effect of primer cure, dip-coating, withdrawal rate, and coating solids content on coating thickness. Slides were cleaned in the usual manner and primed with one percent solids DC-1205 as previously described. The primer cures used varied from 45 minutes to 48 hours at 170°F. Solutions of X-1-2900 prepared by the catalyst dilution technique were prepared in 12.5, 25 and 50 percent solids concentrations.
Table 5-3. Coating Thickness Variations as a Function of Solids Content for X-1-2900/X-2504

<table>
<thead>
<tr>
<th>Solids Content, %</th>
<th>Thickness MILS</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>0.02</td>
</tr>
<tr>
<td>50</td>
<td>0.12</td>
</tr>
<tr>
<td>60</td>
<td>0.20</td>
</tr>
<tr>
<td>70</td>
<td>0.35</td>
</tr>
</tbody>
</table>

Coatings were applied by dip coating at withdrawal rates of either one inch/minute or 4 inches/minute, cured 16 hours at room temperature and post-cured 2 hours at 170°F.

Adhesion and abrasion resistance were evaluated in the usual manner. Thickness of the coatings was also determined (see Table 5-4). Visual observation showed a negligible degree of orange peel.

Abcite-coated acrylic was subjected to adhesion abrasion resistance and hardness tests and was checked in the usual way. Although all tests showed Abcite to be an excellent coating, it did appear to be very slightly less hard and slightly less abrasion resistant than the X-1-2900 silicone coating. However, it should be carefully noted that these tests were strictly subjective. Further evaluation of these materials under more closely controlled conditions may show different results. These data are summarized in Table 5-2.

**E1847-85-4 Tests** -- A 25 percent solids solution of E1847-85-4 silicone coating was prepared by dilution in an equal volume ratio with xylene. The diluted coating was applied directly to clean acrylic slides by dip coating at
AT3-25

a 4 inch/minute withdrawal rate. The applied coating was cured for 12 hours at 170°F. Hardness, abrasion resistance and adhesion were checked in the usual manner.

The E1847-85-4 coating was found to be equivalent to acrylic in hardness, adhered well and was about as abrasion resistant as acrylic.

**Plastic Optics Coating** -- Scrap visors were coated with X-1-2900 over DC-1205 primer using the process controls which showed the most promise. One visor was cleaned in the usual manner and dip-coated with a one percent solution of DC-1205 at 4 inches/minute. The primer was cured for 90 minutes at 170°F after a 30-minute air-dry in the laminar flow booth. A 50-percent solids solution of X-1-2900 prepared by the catalyst dilution technique was applied by dip-coating at 4 inches/minute, cured for 16 hours at room temperature, then post-cured for 2 hours at 170°F. Adhesion, abrasion resistance and hardness were evaluated in the usual way.

Coating on the front of the visor showed the expected good adhesion, abrasion resistance and hardness. Coating on the back side of the visor showed poor adhesion. Since the history of the visor was not known, it was concluded that the lack of adhesion on the inside surface was due to some processing sequence prior to the cleaning stage for coating. Visual examination of the coating showed gross particulate contamination.

A second scrap visor was obtained which had a known history. It was coated in the same way as that of the first visor, except that the coating materials were filtered through a 5-micron filter prior to use.

Adhesion, abrasion resistance of the coating on both sides appeared to be good. Visual examination revealed far less particulate contamination, but still an undesirable amount.
Table 5-4. Effect of Primer Cure, Coating Dip Rate and Coating Solids Content for X-1-2900 Coating and DC-1205 Primer

<table>
<thead>
<tr>
<th>Primer Cure Hrs. 3 170°F</th>
<th>Solids Content %</th>
<th>Dip Coating Rate in/min</th>
<th>Cure Time/Temp. Hrs. 3 RT Hrs. 3 170°F</th>
<th>Adhesion Tape</th>
<th>Abrasion Resistance</th>
<th>Thickness, MILS Top Bottom</th>
</tr>
</thead>
<tbody>
<tr>
<td>3/4</td>
<td>50</td>
<td>4</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.25 0.25</td>
</tr>
<tr>
<td>3/4</td>
<td>25</td>
<td>4</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.15 0.15</td>
</tr>
<tr>
<td>3/4</td>
<td>12.5</td>
<td>4</td>
<td>48</td>
<td>None</td>
<td>None</td>
<td>0.05 0.05</td>
</tr>
<tr>
<td>1-1/2</td>
<td>50</td>
<td>1</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.2 0.2</td>
</tr>
<tr>
<td>2</td>
<td>50</td>
<td>1</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.2 0.2</td>
</tr>
<tr>
<td>6</td>
<td>50</td>
<td>1</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.2 0.2</td>
</tr>
<tr>
<td>48</td>
<td>50</td>
<td>1</td>
<td>48</td>
<td>OK</td>
<td>OK</td>
<td>0.2 0.2</td>
</tr>
</tbody>
</table>
SECTION VI
TECHNICAL CONCLUSIONS

Of the six coatings evaluated, Abcite has the greatest all around potential for meeting all of the requirements for plastic optics. However, both Dow Corning X-1-2900 silicone and Owens Illinois T-650 silicone show considerable promise for protecting plastics optics from abrasion. One of the most significant advantages in the use of Abcite is that it reportedly is applied as a 5-micron thick film. The other materials are used at about 25 - 65 microns. Thinner coatings would be expected to be less likely to interfere with front surface mirrors and other optical systems than thicker coatings. The coatings, ranked in order of decreasing abrasion resistance are X-1-2900, Abcite, T-650 and XD-8157L. The XR-4-3143 silicone was equivalent to X-1-2900 in abrasion resistance, but is no longer available. Pursuit of the high cross-linked silicones, vapor-deposited coatings, and other coatings for protecting plastic optics would require (1) an investment of funds for equipment to apply them in thin, contaminant-free, uniform films, (2) manpower funding, and (3) fairly long development lead times. However, the benefits of a successful development could mean freedom from licensing fees, royalty fees, and a shorter turn-around time.
SECTION VII

BIBLIOGRAPHY


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Wright, A. N., Ultraviolet Photopolymerization Symposium, General Electric, September 13, 1972.


G. E. Deposits Ultra Thin Film, Plastics Design and Processing, August 1967.

New Polymers and Associated Vapor Deposition Process Provide High Integrity Thin Insulating Films, Insulation, April 1965.
A number of primers were believed to have potential for the visor application. The primer systems consisted of MC7134-01 epoxy-polyamide, MS6020H epoxy-amine, MS6293H epoxy-amine, XZ-8-5066 epoxy-silane, DC-1205 epoxy-silane, and A-1100 silane.

**MC7134-01 EPOXY-POLYAMIDE**

This coating essentially consists of a 42 percent solids solution of an epoxy, such as Epon 1001, and a polyamide, such as Versamid 115, in a complex blend of solvents. The solvent blend is primarily xylene, with small portions of isopropyl alcohol and other aromatic solvents. Since a very thin coating would be impossible to achieve at this solids content, the material was reduced in solids to 20 percent with xylene.

**MS6020H EPOXY-AMINE (MODIFIED)**

The base material consists of an epoxy resin (Epon 815) and an amine (Piperidine) as a 100 percent solids material primarily intended for use as an impregnant. To obtain a coating with less color, Dow epoxy resin DER 332 was substituted for Epon 815. To facilitate the application of very thin films, the material was reduced to a 10 percent solids solution with xylene.

Another modification of the material was prepared to reduce "crawling" of the coating during cure. This involved the preparation of an amine functional prepolymer. The prepolymer was prepared by reacting excess piperidine...
with DER 332 epoxy resin over a period of several days at room temperature. This mixture was then used to catalyze the appropriate amount of DER 332 epoxy resin and was reduced to 25 percent solids with xylene.

MS6293H EPOXY-AMINE

This material essentially consists of Epon 828 epoxy resin and \( p, p' \) methylenedianiline. It is prepared as a 100 percent solids material and used primarily as an impregnant. Attempts to reduce this material with solvents were abandoned because of solubility problems.

XZ-8-5066 EPOXY-SILANE

This is a primer composed of Dow epoxy DER 330 and Dow Corning silane Z-6020 (N-B-aminoethyl-\( \gamma \)-aminopropyl trimethysilane) diluted to 10 percent solids with either Dowanol PM (propyleneglycol monoethylether). This primer was prepared by Dow Corning and reduced to 5 percent and 1 percent solids with isopropyl alcohol to facilitate the application of a thin coat.

DC-1205 EPOXY-SILANE

This is a primer composed of Dow epoxy DER 667 and Dow Corning silane Z-6020 diluted to 5 percent solids with Dowanol PM. This primer was prepared by Dow Corning and reduced to 1 percent solids with isopropyl alcohol to facilitate the application of a thin coat.
A-1100 SILANE

This material is prepared in an acidified water solution at 1 percent solids. About 0.1 percent by weight glacial acetic acid is added to deionized water to give a solution with a PH of 3.0 to 4.5. To the acidified water solution is added 10 percent by weight silane with stirring. Agitation is continued until the solution clears (about 5 minutes). The solution is then diluted with deionized water to give a 1 percent silane solution.

Another preparatory method was also used with A-1100 silane. This was prepared by combining one percent by weight silane and n-butyl alcohol for use with the T-650 coating.
APPENDIX B
COATING DEVELOPMENT - FORMULATIONS

It should be understood that basic polymer development (i.e., changes made to monomers and or polymers) was not attempted with the coatings. This was considered well beyond the scope of the feasibility study. Instead, techniques for using the coatings were developed. In the case of the X-1-2900 coating, the technique developed for using it is critical. For without it, the coating was found to be of little value.

XR-4-3143

This is a one-component siloxane polymer which cures to a highly cross-linked state. It was developed by Dow Corning for use as a scratch and abrasion resistant material for protecting plastics. Its major disadvantage has been poor adhesion. Dow Corning reported fair adhesion is attainable with epoxy primers. However, since the major use of the coating was for exterior applications, and since epoxy primers yellow in outdoor exposure, little was done to develop the system's potential. Use of the material on this program was in the as-received condition, as well as a diluted version. This material is no longer available and was replaced by X-1-2900.

X-1-2900

This is a two-component siloxane polymer consisting of X-1-2900 silicone resin and X-2504 silicone hardener. The coating prepared in accordance with the manufacturer's recommendations has limited usefulness since it
cannot be applied in a uniform thin film. A simple, but very important step in the preparation of this material was developed. The procedure requires that the catalyst (X-2504) be diluted with methyl alcohol prior to combining it with the X-1-2900 silicone resin. The ratio of components used to provide a 50-percent solids solution was 0.5 grams of X-2504 silicone catalyst in 60 grams methyl alcohol with stirring. This solution is then added to 100 grams of X-1-2900 silicone resin with stirring. Solutions of 40-percent solids were prepared in a manner similar to that described above.

**T-650**

This material is a two-component, highly cross-linked silicone polymer prepared by combining 4.5 grams of 500-15 catalyst with 100 grams T-650 silicone resin with stirring. This solution was diluted with 52 grams of a solvent blend consisting of 50 grams n-butyl alcohol and 2 grams of xylene to provide a 30-percent solids coating.

**E1847-85-4**

This is a high cross-link density oxime cured, one-component siloxane coating. It is furnished as a 50 percent solids solution but was reduced to 25 percent solids with xylene.

**XD-8157L**

This is a highly cross-linked Zwitterion coating which was applied to acrylic substrates by Dow Chemical from a water solution.
Abcite

This is a proprietary coating developed by DuPont. It is available under license. Abcite coated acrylic is available from DuPont.
ATTACHMENT NO. 4
VISOR HANDLING AND CLEANING INSTRUCTIONS
VISOR HANDLING AND CLEANING INSTRUCTIONS

HANDLING

• Handle coated visor with extra special care
• Avoid touching optical surfaces
• Do not scratch, bump or abrade optical surfaces
• Handle and lift visor using release knob or visor edges
• Store helmet to prevent accidental damage to visor

CLEANING

The optical properties of a coated visor may be degraded if brought into contact with such solvents as gasoline, jet fuel, oils and greases.

DO NOT use standard polishing pastes, rags, flight suit sleeve, etc. to "clean" visor.

If necessary, visor may be cleaned using the following procedures:

DUST, LOOSE DEBRIS

• Blow off visor with aerosol air or,
• Carefully dust off visor with camel's hair brush

DIRT, MUD

1. Rinse or lightly "cottonball"-swab visor with clean tap water.
   DO NOT use hot water on visor. Take special care not to scratch optical coatings.

2079-SR3
2. Allow visor to air dry

3. If visor dries with water spots, rerinse using clean deionized water and again allow to air dry.

FINGERPRINTS

- Swab with cottonball soaked in petroleum ether. (CAUTION: Petroleum ether is highly volatile and flammable...keep away from heat.)

MINOR GREASES, OILS

1. Attempt to remove using cottonball soaked in petroleum ether.

2. If grease or oil smudge still remains, reswab with cottonball soaked in 3% detergent solution...rinse in cool or room-temperature tap water...rinse in deionized water...air dry. (NOTE: DO NOT USE DETERGENT IF OPTICAL FILMS ARE ALUMINUM.)
VISOR CLEANING KIT...CONTENTS

This kit contains the following materials to be used for cleaning helmet sight visors:

1. **Detergent Concentrate** - "Micro" detergent; product of International Products Corp., Trenton, New Jersey.

2. **3% Detergent Solution** - 3% detergent concentrate in clean-tap or deionized water. (NOTE: Two large (8-oz) bottle cap-fulls of concentrate in one large (8-oz) bottle of water will make a 3% solution.)

3. **Petroleum Ether (AR Grade)** - **CAUTION:** Petroleum ether is highly **VOLATILE** and **FLAMMABLE**. Keep away from heat!

4. **Deionized Water** - Use either deionized or distilled water to provide residue-free rinse.

5. **Cottonball Swabs** - Use to provide soft, scratch-free swabbing.

6. **Q-Tip Swabs** - Use only to remove smudges not taken off through use of cottonball swabs.

7. **Camel's Hair Brush** - Use to remove dust and loose debris.

8. **Canned Air** - Aerosol spray can of clean, filtered air... used for blowing loose particles of debris off visor.
ATTACHMENT NO. 5

GHOST RATIOS FOR VARIOUS COATING AND TINTED ACRYLICS USED IN IHMS/D VISORS
EXAMPLE CALCULATION:

FOR 50% T TINTED ACRYLIC
(ASSUME 4%R/SURFACE)

\[
\text{GHOST RATIO} = \frac{R_1}{R_2^{*}} = \frac{R_1}{(1 - R_1)^2 (T_A)^2 (R_2)}
\]

\[
= \frac{0.04}{(1 - 0.040)^2 (0.5)^2 (0.04)} = 4.4
\]

Model 1. Ghost Ratio Calculation Tinted Acrylic Visor --
No Coatings

2079-SR3
EXAMPLE CALCULATION:

FOR 10% T TINTED ACRYLIC
10% R REFLECTIVE COATING

\[ \frac{R_1}{R_2} = \frac{R_1}{(1-R_1)^2 (T_A)^2 (R_2)} \]

\[ = \frac{0.1}{(1 - 0.1)^2 (0.1)^2 (0.04)} = 309 \]

Model 2. Ghost Ratio Calculation Tinted Acrylic Visor with Reflective Coating
EXAMPLE CALCULATION:

FOR 30% T TINTED ACRYLIC
40% R REFLECTIVE COATING
0.5% R AR COATING

$\text{GHOST RATIO} = \frac{R_1}{R_2^*} = \frac{R_1}{(1 - R_1)^2 (T_A)^2 (R_2)}$

$= \frac{0.5}{(1 - 0.5)^2 (0.3)^2 (0.005)} = 4.444$

Model 3. Ghost Ratio Calculation Tinted Acrylic Visor with Both Reflective and Antireflective Coatings
Models 1 and 2. Ghosting Ratio for Various Tinted Acrylics and Reflective Coatings

2079-SR3
Model 3. Ghosting Ratio for Various Tinted Acrylics Reflective and AR Coatings

2079-SR3