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M. C. Willson, et al.

Monsanto Research Corporation
Dayton, Ohio

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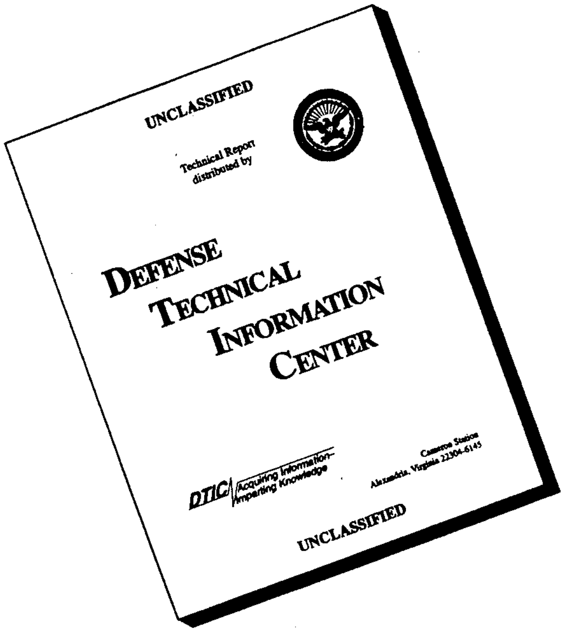

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by

M. C. Willson and C. E. Semler

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Dayton Laboratory
Dayton, Ohio 45407

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FOREWORD

The research described herein, which was conducted by Monsanto Research Corporation, Dayton Laboratory, was performed under NASA Contract NAS 9-8357. The work was done under the guidance of the Technical Monitor, Mr. D. G. Sauers, Supporting Development Branch, NASA Manned Spacecraft Center.

ABSTRACT

Sodium and potassium silicates were applied by drawing and dipping techniques to polycarbonate to provide a nonflammable coating. A polyurethane coating between the silicate and the substrate was required to achieve good adhesion. Polymeric films, usually polyurethane or polystyrene, were applied over the silicate to improve the long-time stability of the coating system. A limited number of organic-modified silica coatings were prepared but were significantly less effective than the alkali-silicate coatings in preventing flame damage to polycarbonate. Experimental glass compositions were formulated for plasma spraying studies from the alkali-B_2O_3-SiO_2, soda-lime-silica, and BaO-Al_2O_3-B_2O_3 systems. Commercial glasses were also employed. All plasma-sprayed coatings were too translucent to satisfy optical requirements in the intended application. Optically transparent pyrex coatings were applied to polycarbonate by RF sputtering, but substrate discoloration was observed in most cases. Most of the coatings prepared in the program were evaluated for flame resistance, light transmittance, optical uniformity and distortion, humidity and thermal shock resistance, vacuum and ultraviolet stability, and flexural and impact strength.

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TABLE OF CONTENTS

TABLE OF CONTENTS (Cont'd.)

LIST OF FIGURES

LIST OF FIGURES (Cont'd.)

LIST OF TABLES

LIST OF TABLES (Cont'd.)

SECTION I. SUMMARY

A. Alkali Silicates

Sodium and potassium silicates of varying K_2O/SiO_2 ratios (where K is either sodium or potassium) were applied to polycarbonate in order to render it nonflammable. The coatings were applied by both conventional doctor blade techniques and modified dip procedures. Early in the program, it was decided that a bond coating was necessary to provide good adhesion for the silicate layer. Consequently, a cryogenic urethane adhesive was selected for this purpose. In most cases the silicate coatings were air dried in a humid atmosphere to reduce the drying rate and minimize the possibility of stress cracking and warping. The hardened air-dried coatings take on glass like properties and require sanding and polishing to reduce optical distortion. Finally, in order to reduce the water loss and subsequent degradation of the silicate coating, a polymeric encapsulator material was applied over the silicate by dipping, drawing and spraying techniques. Commercially available polyurethane and polystyrene were selected on the basis of appearance and good performance during tests. The frosted appearance of the sanded silicate surface was eliminated by the polymer film.

The polycarbonate-polyurethane-silicate-polyurethane (PUSP) systems were evaluated on the basis of various tests considered important by NASA. Foremost of these tests were the optical transmittance, optical distortion, and flame impingement tests. The best PUSP systems were found satisfactory in each of these tests.

In addition, the PUSP system performed satisfactorily in thermal shock, flexural, vacuum, ultraviolet, impact, and humidity tests.

Due to the necessity of applying a flammable polymer moisture barrier over the silicate, some concern was raised over the possibility of the polymer flaming when impinged with a flame in an oxygen atmosphere. However, tests conducted at NASA indicated little flame propagation of the polymer film. Consequently, polymeric films were found to be acceptable.

B. Non Alkali-Silicates

Organic-modified silica coatings were prepared but were found to be significantly inferior to alkali-silicate coatings in providing flame protection to polycarbonate.

C. Plasma Sprayed Coatings

Extensive studies have been carried out using refined arc-plasma spraying techniques in order to develop a nonflammable, transparent coating for polycarbonate. Initially, simple chemical compounds were plasma sprayed and their melt characteristics observed. These materials showed very limited film formation. Consequently, the emphasis was shifted to the development of experimental glass compositions which could be formulated with the physical properties necessary for deposition by arc plasma techniques. In general, the experimental glasses were located in the following systems: alkali-B_2O_3-SiO_2, soda-lime-silica, or BaO-Al_2O_3-B_2O_3. In addition, several commercial glasses with the properties required to permit deposition by the arc plasma process were located. These experimental and commercial glasses were successfully plasma sprayed on polycarbonate, yielding uniform glass coatings suitable for non-optical-type uses, due to their translucent appearance. The particulate deposition characteristics inherent in the arc plasma process cause the translucent appearance because the resulting grain boundaries do not permit isotropic light transmission. As a result, the plasma-sprayed glass coating prepared in this program did not possess the optical qualities necessary for instrument dials. Such coatings might be of interest, however, for other components where coatings having abrasion-resistance, impact-resistance or other special resistance properties are required.

D. Sputtered Coatings

RF diode sputtering of transparent pyrex coatings on polycarbonate was performed. The overheating problem of this setup was basically overcome so that the polycarbonate would not be physically degraded. However, a slight brownish discoloration of the polycarbonate was always observed. Optically, the coatings (1000-2000 Å) retain their transparency but the substrate takes on a slight color.

SECTION II. INTRODUCTION

The objective of this experimental program was to develop a non-flammable transparent coating for polycarbonate, a material with good optical and impact properties, which is currently utilized in transparent visor and instrumentation applications. The work was divided into 3 main areas: 1) alkali silicates; 2) non-alkali silicates; and 3) plasma-spraying of glass. In addition, an effort was made to determine the feasibility of sputtering a nonflammable film on polycarbonate. The latter half of the contract period emphasized the alkali silicate coating approach.

SECTION III. RESULTS AND DISCUSSION OF RESULTS

A. Alkali Silicate Systems

1. Coating Fabrication.

a. Preparation of Bond Coatings. Both sodium and potassium silicates, because of their water content, are non-wetting when applied to a polycarbonate. Indeed, polycarbonate is used as a mold for producing silicate castings.

Coatings have been reportedly produced by treating plastic substrates with polyvinyl alcohol followed by application of the silicate in the form of a silicate-boric acid gel.

Transparent polyvinyl alcohol (PVA) films intended to serve as intermediate coatings formed on glass were well bonded whereas those formed on polycarbonate adhered poorly. Adhesion of silicate films to the PVA films was good with both substrates. No improvement in adhesion of PVA films to polycarbonate was obtained with a hydrolizing treatment of the polycarbonate prior to application of PVA as successfully employed with other substrates (Ref. 1).

Polyvinyl Butyral (PVB) film bonded to polycarbonate film by heating for 10 minutes at 200°F was also investigated as an intermediate coating. However, the optical quality was poor due to trapped bubbles and distortion.

A brief study was made using General Electric RTV108 silicone as an adhesive layer between the silicates and polycarbonate. The silicone coatings were applied with a spatula and air cured at room temperature. However, all coatings were found to have poor adhesion to the polycarbonate.

Initial polyurethane bond coating studies were made using a cryogenic polyurethane made by 3M Company designated Scothweld EC3515. The oven-cured specimens tended to form bubbles indicating the abnormal release of CO_2. Since aging of the urethane could have been a factor in this case, the 3M urethane was abandoned in favor of a urethane of known quality. No bubbles were observed after oven curing with the Dupont Adiprene L-100 polyurethane. A range of cure cycles were studied with the Dupont polyurethane is shown below:

CURE CYCLES FOR DUPONT ADIPRENE L-100 POLYURETHANE-COATED POLYCARBONATES

Cure Cycles	Cure Time, hr.	Temperature, °F	Atmosphere
1	5	212	Air
2	8	212	Air
3	32	212	Air
4	80	212	Air
5	96	212	Air
6	6	285	Air

No discernible difference was observed in polyurethane properties. In most cases, a MOCA hardner (4'4'-methylene-bis-2-chloroaniline) was used to accelerate the curing time of the polyurethane. In a few instances the MOCA was replaced by ethylene glycol. The curing time, however, was much longer in these cases. The urethane coating procedure consists of a preliminary air removal of the polyurethane to eliminate coating bubbles. This was accomplished by placing the polyurethane in an Erlenmeyer flask which is then evacuated to $\sim 10^{-4}$ torr. When the bubbling stopped, the hardner was mixed with the polyurethane (12.5 parts MOCA to 100 parts polyurethane by weight) and immediately drawn on 9" x 11" polycarbonate with an 8 inch Gardner knife. The polyurethane was drawn in thicknesses ranging from 2 to 10 mils. Generally, the coatings were transparent with minimal distortion. A summary of undercoating materials evaluated and subsequent results is shown in Table I.

b. Preparation of Silicate Coatings. Both sodium and potassium silicate exhibit high transparency over the visible spectrum. The silicates are commercially available from Philadelphia Quartz Company as a water-based solution in various Na_2O/SiO_2 or K_2O/SiO_2 ratios as shown below:

VISCOSITY OF ALKALI SILICATES (Ref. 2)

Silicate	Type	Na_2O/SiO_2	K_2O/SiO_2	Viscosity, cps @ 20°C
Sodium	K	2.87	-	960
Sodium	N	3.20	-	178
Sodium	S-35	3.74	-	222
Potassium	Kasil #6	-	2.11	1010

Table I

UNDERCOATING CANDIDATES

Type	Form	Cure Time, hr.	Cure Temperature, °F	Application Method	Substrate Treatment	Remarks
Polyvinyl alcohol	viscous liquid	1/6	220	Spatula	None	Poorly bonded
Polyvinyl alcohol	viscous liquid	1/4	220	Spatula	5 min. in hydrolizing solution	Poorly bonded
Polyvinyl Butyral	tacky film	1/6	200	Pressed	None	Bubbly distorted film
3M Scotchweld Polyurethane	viscous liquid	4	200	Doctor Blade	None	Bubbly film
Dupont Adiprene L-100 Polyurethane	viscous liquid	5-96	200	Doctor Blade	None	Transparent film

A variety of solution viscosities and coating properties can be obtained depending on this ratio. Generally, the as received solutions had the consistency of thin molasses. The silicate solutions were applied by both a drawing and a pouring technique. The poured coatings were made by simply pouring the silicate on the flat polyurethane/polycarbonate specimen and spreading it over the plate to achieve a uniform thickness. The drawn coatings were made using either a 4 inch or 10 inch doctor knife, set at the required thickness gap, to pull the solution over the polyurethane surface. Coatings were difficult to produce (with either technique) due to the nonwetting properties of the silicate solution on polyurethane. This problem was greatly alleviated by using the higher viscosity silicate solutions such as K sodium silicate and Kasil #6 potassium silicate. In a few instances, a fine mist of silicate diluted with water (1:1 by weight) was sprayed on the polyurethane surface and allowed to dry.

The heavier silicate was then applied to the sprayed silicate producing good wetting characteristics. The silicate coatings were then air dried in a high humidity environment (>60%) for several days until the coatings had hardened sufficiently for surface finishing.

Cracks and/or wrinkles may develop as the silicate drys depending on the silicate type, drying rate, and water content of the coating. Both K sodium silicate and Kasil #6 potassium silicate were initially found to give the most stable air-dried coatings. The majority of the silicate coatings were made from Kasil #6 potassium silicate because of its resistance to physical and optical degradation when exposed to varying humidity environments. Also, the drying rate must be low enough to allow water to volatilize slowly or cracking will result. If the amount of water retained in the specimen reaches a critical low, cracking and optical degradation will result. Table II summarizes the alkali silicate coating work. Post silicate application treatments and coating evaluation will be discussed in the succeeding sections.

c. Curing of Alkali Silicate Coatings. In order to produce a more stable alkali silicate coating that would be relatively insensitive to environmental changes, several curing methods were attempted. These were oven drying, chemical setting, and pressure curing. Little success was achieved because in most cases the optical properties of the coatings were severely degraded.

(1) Oven Drying Studies. Type K & N sodium silicate and Kasil #6 potassium silicate were subjected to various time-temperature cycles as shown in Table III. Initial studies were conducted using specimens which had been air dried for at least

Table II

PREPARATION OF SILICATE COATINGS

Spec. No.	Substrate			Undercoating			Protective Coating			Remarks
	Type	Size	Treatment	Type	Bake Time min.	Bake Temp, °F	Silicate	Type	Thickness, mils	
S-1	Glass Slide	1"x3"	None	None	---	---	N	Sodium	N.D.	Well bonded
S-2	Glass Slide	1"x3"	None	None	---	---	N	Sodium	N.D.	Transparent; well bonded
S-3	Polycarbonate	1"x1"	None	None	---	---	N	Sodium	N.D.	Bubbly; poor bond
S-4	Polycarbonate	1"x1"	None	None	---	---	N	Sodium	N.D.	Transparent, poor bond
S-5	Glass Slide	1"x3"	None	None	---	---	N	Sodium	N.D.	Transparent, well bonded
S-6	Glass Slide	1"x3"	None	Polyvinyl Alcohol	---	R.T.	-	None	N.D.	Transparent, well bonded
S-7	Glass Slide	1"x3"	None	Polyvinyl Alcohol	10	220	N	Sodium	N.D.	Bubbly; well bonded
S-8	Polycarbonate	1"x1"	None	Polyvinyl Alcohol	10	220	N	Sodium	N.D.	PVA poorly bonded to polycarbonate
S-9	Polycarbonate	1"x1"	5 min in hydrolyzing solution	Polyvinyl Alcohol	10	220	-	None	N.D.	PVA poorly bonded to polycarbonate
S-10	Polycarbonate	1"x1"	5 min in hydrolyzing solution	None	---	---	N	Sodium	N.D.	PVA poorly bonded to polycarbonate
S-11	Polycarbonate	1"x1"	10 min in hydrolyzing solution	Polyvinyl Alcohol	15	220	-	None	N.D.	PVA poorly bonded
S-12	Polycarbonate	1"x1"	10 min in hydrolyzing solution	None	---	---	N	Sodium	N.D.	Sodium Silicate poorly bonded
S-13	Polycarbonate	1"x1"	Dip in Acetone	Polyvinyl Alcohol	15	220	-	None	N.D.	Polycarbonate became gray after exposure to acetone
S-14	Polyvinyl Butyral	1"x1"	None	None	---	---	N	Sodium	N.D.	Well bonded; clear
S-15	Polyvinyl Butyral	1"x1"	None	None	---	---	N	Sodium	N.D.	Well bonded; cracked
S-16	Polycarbonate	1"x1"	None	Polyvinyl Butyral	10	200	N	Sodium	N.D.	Bubbles under polyvinyl butyral; well bonded
S-17	Polycarbonate	3"x3"	None	Polyvinyl Butyral	10	200	N	Sodium	N.D.	Bubbles under polyvinyl butyral; well bonded
S-18	Polycarbonate	3"x3"	None	Polyvinyl Butyral	10	200	N	Sodium	N.D.	Fewer bubbles
S-19	Polycarbonate	1"x1"	Grit blasted	None	---	---	N	Sodium	N.D.	Better bond; some diffusion at interface
S-20	Polycarbonate	1"x1"	Grit blasted	None	---	---	N	Sodium	N.D.	Better bond; some diffusion at interface
S-39A	Polycarbonate	1"x1"	None	3M Scotchweld® Polyurethane	---	R.T.	S-35	Sodium	N.D.	Transparent; a few cracks well bonded
S-40	Polycarbonate	1"x1"	None	Polyurethane	240	200	S-35	Sodium	N.D.	Transparent; a few cracks well bonded
S-41A	Polycarbonate	1"x1"	None	Polyurethane	240	200	Kasil 6	Potassium	N.D.	Transparent; a few cracks well bonded
S-42	Polycarbonate	1"x1"	None	Polyurethane	---	R.T.	Kasil 6	Potassium	N.D.	Transparent; a few cracks well bonded
S-43	Polycarbonate	1"x1"	None	Polyurethane	---	R.T.	N	Sodium	N.D.	Transparent; a few cracks well bonded
S-44A	Polycarbonate	1"x1"	None	Polyurethane	240	200	-	Sodium	N.D.	Transparent; many bubbles well bonded
S-45	Polycarbonate	1"x1"	None	Polyurethane	---	R.T.	Kasil 6	Potassium	N.D.	Transparent; well bonded
S-46	Polycarbonate	1"x1"	None	Polyurethane	---	R.T.	K	Potassium	N.D.	Transparent; well bonded
S-47A	Polycarbonate	1"x1"	None	Polyurethane	240	200	K	Potassium	N.D.	Transparent; many bubbles well bonded
S-48A	Polycarbonate	3"x3"	None	Polyurethane	240	200	Kasil 6	Potassium	N.D.	Transparent; many bubbles well bonded
S-49	Polycarbonate	3"x3"	None	Polyurethane	240	200	Kasil 6	Potassium	N.D.	Transparent well bonded
S-51	Glass Slide	1"x3"	None	None	---	---	Kasil 6	Potassium	N.D.	Transparent; some cloudiness few bubbles
S-50	Polycarbonate	1"x1"	None	Polyurethane	---	R.T.	Kasil 6	Potassium	N.D.	Sandwiched between urethane and polycarbonate mold; transparent no distortion
S53-65	Polycarbonate	1"x1"	None	General Elec. RTV 108 Silicone	---	R.T.	None	---	N.D.	Transparent, a few bubbles, poor adhesion of silicone
S51, 68-71, 75-78, 81-82, 110-111	Glass Slides	1"x1"	None	None	---	R.T.	Kasil 6	Potassium	N.D.	Transparent films, a few trapped air bubbles, made for chemical setting studies
S66-67, 72-74, 79-80, 83, 108-109	Glass Slides	1"x1"	None	None	---	R.T.	K	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for chemical setting studies

Cont'd Table II

Spec. No.	Substrate			Undercoating			Protective Coating			Remarks
	Type	Size	Treatment	Type	Bake Time min.	Bake Temp, °F	Silicate	Type	Thickness, mils	
S84-85, 90-91, 98-101, 112-113	Glass Slides	1"x1"	None	None	---	R.T.	N	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for chemical setting studies
S86-89, 96-97, 104-5, 114-115	Glass Slides	1"x1"	None	None	---	R.T.	Kasil 1	Potassium	N.D.	Transparent films, a few trapped air bubbles, made for chemical setting studies
S92-95, 102-103, 106-107, 116-117, 122	Glass Slides	1"x1"	None	None	---	R.T.	S-35	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for chemical setting studies
S128-129	Glass Slides	1"x1"	None	None	---	R.T.	Kasil 6	Potassium	N.D.	Transparent films, a few trapped air bubbles, made for oven drying studies
S130-131	Glass Slides	1"x1"	None	None	---	R.T.	K	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for oven crying studies
S132-133	Glass Slides	1"x1"	None	None	---	R.T.	N	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for oven drying studies.
S-126-127	Glass Slides	1"x1"	None	None	---	R.T.	Kasil 1	Potassium	N.D.	Transparent films, a few trapped air bubbles, made for oven drying studies
S134-135	Glass Slides	1"x1"	None	None	---	R.T.	S-35	Sodium	N.D.	Transparent films, a few trapped air bubbles, made for oven drying studies
S174-175	Polycarbonate	1"x1"	None	None	---	R.T.	Kasil 6	Sodium	N.D.	Transparent films, poorly bonded
S120-121, 123, 153-162, 171-172, 185, 187-188, 194	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	Kasil 6	Sodium	N.D.	Transparent, bubble-free films
S182, 194-196, 198-199, 201, 203, 207	Polycarbonate	1"x1"	None	Oven cured DuPont Adiprene 100 Urethane	72	212	Kasil 6	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bunded
S119-125, 204	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	K	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bonded
S136-138, 142-152, 163-169, 186	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	K	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bonded
S190-193, 197, 202, 205-206	Polycarbonate	1"x1"	None	Oven cured DuPont Adiprene 100 Urethane	---	R.T.	K	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bonded
S118	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	N	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bonded.
S122, 124	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	S-35	Sodium	N.D.	Transparent films, a few trapped air bubbles, well bonded
S176-181	Polycarbonate	1"x1"	None	3M Scotchweld Urethane	---	R.T.	Kasil 88	Potassium	N.D.	Transparent films, a few trapped air bubbles, well bonded
S230-232 S234-236	Polycarbonate	3"x3"	None	DuPont Adiprene L-100 Polyurethane	*	*	K	Sodium	~9-30	
S233	Polycarbonate	3"x3"	None	DuPont Adiprene L-100 Polyurethane	*	*	K	Sodium	~5	
S237-246	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	*	*	K	Sodium	~8-14	Oven drying and thermal shock studies
S247-255	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	8	212	Kasil 6	Potassium	~5-9	Oven drying and thermal shock studies
S256-257	Polycarbonate	3"x3"	None	DuPont Adiprene L-100 Polyurethane	8	*	K	Sodium	~19	Cold forming studies
S258-264	Polycarbonate	1"x1"	None	DuPont Adiprene L-100 Polyurethane	*	*	Kasil 6	Potassium	~8-11	Oven drying studies
S265-271	Polycarbonate	1"x1"	None	DuPont Adiprene L-100 Polyurethane	*	*	K	Sodium	13-23	
S272-276	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	*	*	K	Sodium	----	Cold forming studies
S277-281	Polycarbonate	1 1/2"x1 1/2"	None	DuPont Adiprene L-100 Polyurethane	*	*	Kasil 6	Potassium	~2-6	Oven drying studies
S282	Polycarbonate	3"x3"	None	DuPont Adiprene L-100 Polyurethane	*	*	Kasil 6	Potassium	----	Oven drying studies
S283-288	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	K	Sodium	~6	Oven drying studies
S289-294	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	Kasil 6	Potassium	~2-6	

Cont'd Table II

Spec. No.	Substrate Type	Substrate Size	Substrate Treatment	Undercoating Type	Undercoating Bake Time min.	Undercoating Bake Temp, °F	Protective Coating Silicate	Protective Coating Type	Thickness, mils	Remarks
8295	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	K	Sodium	----	Produced for future flame tests
8296	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	K	Sodium	~14	Produced for future flame tests
8297	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	K	Sodium	~12	Produced for future flame tests
8298	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	K	Sodium	~12	Produced for future flame tests
8299	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	Kasil 6	Potassium	~0.5	Produced for future flame tests
8300	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	Kasil 6	Potassium	----	Curved from substrate during draw down
8301	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	Kasil 6	Potassium	~9	Produced for future flame tests
8302	Polycarbonate	2"x2"	None	DuPont Adiprene L-100 Polyurethane	80	212	Kasil 6	Potassium	~11	Produced for future flame tests
8303-308, 321-323 330-332	Lexan Polycarbonate	2"x2"	None	None	RT Air Cured		K	Sodium	4-13	Oven drying studies
8309-314	Lexan Polycarbonate	2"x2"	None	None	RT Air Cured		Kasil 6	Potassium	4-13	Oven drying studies
8315-320	Lexan Polycarbonate	2"x2"	None	None	RT Air Cured		N	Sodium	6-39	Oven drying studies
8324-326 333-335	Lexan Polycarbonate	2"x2"	None	None	RT Air Cured		Kasil 6	Potassium	----	Oven drying studies
8327-329 336-338	Lexan Polycarbonate	2"x2"	None	None	RT Air Cured		N	Sodium	----	Oven drying studies
8355-361	Lexan Polycarbonate	2"x2"	None	None	5	212	K	Sodium	4-17	Flame tests; cold forming studies
8380-385	Lexan Polycarbonate	2"x2"	None	None	32	212	K	Sodium		Water repellent coating studies.
8393, 395-396	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	K	Sodium	22-26	Flame tests; cold forming studies
8398-400	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	K	Sodium	23-34	Cold forming studies
8401-402	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	Kasil 6	Potassium	19-21	Cold forming studies
8403-404	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	K	Sodium	35-73	Flame test; cold forming studies
8405-406	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	Kasil 6	Potassium	15-23	Cold forming studies
8433-434	1"x1" Polycarbonate	1"x1"	None	DuPont Adiprene L-100 urethane	RT Air Cured		K	Sodium	61-63	Adhesion studies
8435, 437, 440	1"x3" Glass Slide	1"x3"	None	DuPont Adiprene L-100 urethane	*	*	Kasil 6	Potassium	----	Future oven drying studies
8436, 438, 439	1"x3" Glass Slide	1"x3"	None	DuPont Adiprene L-100 urethane	*	*	K	Sodium	----	Future oven drying studies
8409-415 419	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	5	212	Kasil 6	Potassium	15-19	Protective coating studies
8418-420	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	32	212	Kasil 6	Potassium	15-16	Cold forming studies, protective coating studies
8441-445	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	Kasil 6	Potassium	23-25	Transmittance studies
8447	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	K	Sodium	----	Protective coating studies
8475	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	96	212	Kasil 6	Potassium	47	Protective coating studies
[illegible]	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	K	Sodium	20-49	Cold forming studies; protective coating studies
[illegible]	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	79	212	Kasil 6	Potassium	32	Cold forming, protective coating, and transmittance studies

Cont'd Table II

Spec. No.	Substrate			Undercoating			Protective Coating			Remarks
	Type	Size	Treatment	Type	Bake Time min.	Bake Temp, °F	Silicate	Type	Thickness, mils	
S493-494	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	80	212	Kasil 6	Potassium	30	Protective coating studies
S476-479	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	5	212	Kasil 6	Potassium	19-35	Protective coating studies
S480-488	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	5	212	K	Sodium	45	Protective coating and transmittance studies
S481, 483, 485	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	32	212	K	Sodium	----	Cold forming protective coating, and transmittance studies
S495-496 519-526	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	36-42	Cold forming studies; protective coating
S503-505 508-509	Lexan Polycarbonate	3"x6"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	18-39	Flame propagation studies
S497-502,	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	K	Sodium	20-77	Vacuum studies
S504, 506-507, 510	Lexan Polycarbonate	3"x6"	None	DuPont Adiprene L-100 urethane	72	212	K	Sodium	35-53	Flame propagation tests
S527-528 531	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	29-64	Protective coating studies
S532-540	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	15-35	Protective coating, U.V. stability, and weight loss studies
S541-560	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	24-35	U.V. stability and protective coating studies
S573-576	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	15-39	Protective coating studies
S580-582 583	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	17	Protective coating studies
S585-596	G.E. Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	16-25	Flexural strength and protective coating studies
S595	Lexan Polycarbonate	1/2"x3 1/2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	22	Flexural test
S596	Lexan Polycarbonate	1/2"x3 1/2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	23	Flexural test
S597	Lexan Polycarbonate	3"x6"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	19	Sent to NASA for flame propagation tests
S598	Lexan Polycarbonate	3"x6"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	17	Sent to NASA for flame propagation tests
S599	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	20	Sent to NASA for flame propagation tests
S600	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	13	Sent to NASA for flame propagation tests
S601	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	22	Sent to NASA for flame propagation tests
S602	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	20	Sent to NASA for flame propagation tests
S603	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	21	Sent to NASA for flame propagation tests
S604	Lexan Polycarbonate	2"x2"	None	DuPont Adiprene L-100 urethane	72	212	Kasil 6	Potassium	21	Sent to NASA for flame propagation tests
S613, 615, 616, 618	Polycarbonate	1 1/2"x1 1/2"	None	DuPont Adiprene L-100 urethane	50	200	Kasil 6	Potassium	2.5-9.0	Impact tests
S620, 623, 625 626	Polycarbonate	10"x10"	None	DuPont Adiprene L-100 urethane	50	200	Kasil 6	Potassium	9.5-16	Sent to NASA
S644, 643-648	Polycarbonate	1 1/2"x1 1/2"	None	DuPont Adiprene L-100 urethane	50	200	K	Sodium	31-46	Impact tests
S642, 649	Polycarbonate	1 1/2"x1 1/2"	None	DuPont Adiprene L-100 urethane	50	200	K	Sodium	28-36	U.V. stability

Table II

Spec. No.	Substrate Type	Substrate Size	Substrate Treatment	Undercoating Type	Undercoating Bake Time min.	Undercoating Bake Temp, °F	Protective Coating Silicate	Protective Coating Type	Thickness mils	Remarks
S650-655 660, 662	Polycarbonate	1 1/2"x1 1/2"	None	DuPont Adiprene L-100 urethane	50	200	Kasil 6	Potassium	N.D.	Impact tests
634, [illegible], 656-[illegible] 664, 666	Polycarbonate	10"x10"	None	DuPont Adiprene L-100 urethane	50	200	Kasil 6	Potassium	4-27	Sent to NASA
8669-672	Polycarbonate	10"x10"	None	DuPont Adiprene L-100 urethane	50	200	K	Sodium	17-27	Sent to NASA
867[illegible]	Polycarbonate	10"x10"	None	DuPont Adiprene 1-100	50	200	Kasil 6	Sodium	27	Sent to NASA
8675	Polycarbonate	10"x10"	None	DuPont Adiprene 1-100	50	200	Kasil 6	Sodium	6	Sent to NASA

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* Scotchweld EC 3515
R.T. = room temperature
All silicate supplied by Philadelphia Quartz Co.
Polyurethane: Scotchweld EC 3515
Polyvinyl Alcohol: Elvanol 70-05 (low mol. wt.)
Polyvinyl Butyral: Monsanto Company
MOCA Hardener: 4'4'-methylene-bis-2-chloroaniline

Tablo III

OVEN DRYING OF SILICATE COATINGS

Spec. Number	Substrate Type	Undercoating	Protective Coating				Coating Thickness mils	Appearance
			Type	Silicate	Bake Time, hr.	Bake Temp, °F		
120	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		R.T.*	N.D.	Transparent film
		Scotchweld			.5	100		No change
		Urethane			.5	150		No change
					.5	200		White translucent film
123	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		R.T.*	N.D.	Transparent film
		Scotchweld			.5	100		No change
		Urethane			.5	150		No change
					.5	200		Small scattered bubbles
128	Glass	None	Kasil 6	Potassium		125*	~19	Transparent film
					18	125		No change
					22	150		No change
					2	175		Transparent, no bubbles or cracks
129	Glass	None	Kasil 6	Potassium		125*	~19	Transparent film
					18	125		No change
					22	150		No change
					2	175		Transparent, no bubbles or cracks
171	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		R.T.*	~4	Transparent film
		Scotchweld			1	100		No change
		Urethane			21	125		Many small bubbles; good adhesion
172	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		R.T.*	~4	Transparent film
		Scotchweld			1	100		No change
		Urethane			21	125		Many small bubbles, good adhesion
174	Polycarbonate	None	Kasil 6	Potassium		R.T.*	N.D.	Transparent film
					1	100		No change
					21	125		No bubbles; poor adhesion
175	Polycarbonate	None	Kasil 6	Potassium		R.T.*	N.D.	Transparent film
					1	100		No change
					21	125		No bubbles; poor adhesion
186	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		119*	~15	Transparent film
		Scotchweld			28.5	119		Bubbles
		Urethane			16	198		Bubbles, white area, cracks
187	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		119*	~13	Transparent film
		Scotchweld			28.5	119		Bubbles, cracks
		Urethane			16	198		Bubbles, cracks, white area
188	Polycarbonate	EC 1535 (3M)	Kasil 6	Potassium		119*	~9	Transparent film
		Scotchweld			28.5	119		Bubbles, cracks
		Urethane			16	198		Bubbles, cracks, white area
195	Polycarbonate	DuPont	Kasil 6	Potassium		104*	~12	Transparent film
		Adiprene			19	104		Bubbles, transparent
		L-100 Urethane			16	118		No change
					8	126		No change
					19	158		Bubbles, cracks
196	Polycarbonate	DuPont	Kasil 6	Potassium		104*	~13	Transparent film
		Adiprene			19	104		Bubbles, transparent
		L-100 Urethane			16	118		No change
					8	126		No change
					19	158		Bubbles, cracks
203	Polycarbonate	DuPont	Kasil	Potassium		R.T.*	~5	Transparent film
		Adiprene			.5	125		No change
		L-100 Urethane			41	125		Cracks, no bubbles
					52	125		No change; removed
119	Polycarbonate	EC 1535 (3M)	K	Sodium	.5	100*	~39	Transparent film
		Scotchweld			.5	150		No change
		Urethane			~19	200		Large bubbles
130	Glass	None	K	Sodium		119*	N.D.	Transparent film
					18	125		No change
					22	150		No change
					2	175		No bubbles or cracks
131	Glass	None	K	Sodium	.5	100*	N.D.	Transparent film
					18	125		No change
					22	150		No change
					2	175		Cracks, no bubbles
192	Polycarbonate	DuPont	K	Sodium		R.T.*	~15	Transparent film
		Adiprene			2.5	100		Some edge cracks and bubble
		L-100 Urethane			21	150		No change
					26	180		No change, removed
202	Polycarbonate	DuPont	K	Sodium		R.T.*	~19	Transparent film
		Adiprene			.5	125		No change
		L-100 Urethane			41	125		Cracks, no bubbles
					52	125		No change, removed
118	Polycarbonate	EC 1535	N	Sodium		R.T.*	~24	Transparent film
		Scotchweld			.5	100		No change
		Urethane			.5	150		No change
					.5	200		Large bubbles, test terminated
132	Glass	None	N	Sodium		125*	~7	Transparent film
					18	125		No change
					22	150		No change
					2	175		Cracks, transparent
133	Glass	None	N	Sodium		125*	~16	Transparent film
					18	125		No change
					22	150		No change
					2	175		Cracks, transparent
122	Polycarbonate	EC 1535 (3M)	S-35	Sodium		100*	N.D.	
		Scotchweld			.5	150		Transparent film
		Urethane			.5	160		No change
					.5	170		No change
124	Polycarbonate	EC 1535 (3M)	S-35	Sodium		100*	N.D.	Transparent film
		Scotchweld			.5	100		No change
		Urethane			.5	150		Cracks and bubbles

Cont'd Table III

Spec. Number	Substrate Type	Undercoating	Protective Coating				Coating Thickness mils	Appearance
			Type	Silicate	Bake Time, hr.	Bake Temp., °F		
134	Glass	None	S-35	Sodium	 18 22 2	119* 125 150 175	~12	Transparent film No change No cahnge Cracks, transparent
135	Glass	None	S-35	Sodium	 18 22 2	119* 125 150 175	~13	Transparent film No change No change Cracks, transparent
176	Polycarbonate	EC 1535 (3M) Scotchweld Urethane	Kasil 88	Potassium	 17 7 25	110* 110 136 156	~3	Transparent film No change No change Microcracks, bubbles
180	Polycarbonate	EC 1535 (3M) Scotchweld Urethane	Kasil 88	Potassium	 17 7 25	110* 110 136 156	~8	Transparent film No change No change Microcracks, bubbles
S242A	2"x2"Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	K	Sodium	.5 .5 .5	100 150 208	~8	A few dispersed bubbles; slight removable haze
S243A	2"x2" Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	K	Sodium	.5 .5 .5	100 150 208	~8	A few dispersed bubbles shift removable phase
S247A	2"x2" Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	Kasil 6	Potassium	.5 .5 .5	100 150 208	~7	No bubbles; slight crazing
S249A	2"x2" Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	Kasil 6	Potassium	.5 .5 .5	100 150 208	~7	No bubbles; slight crazing
S260A	2"x2" Lexan Polycarbonate	DuPont** Adiprene L-100 Urethane	Kasil 6	Potassium	.5 .5 .5	100 150 205	~17	A few dispersed bubbles
S261A	2"x2" Lexan Polycarbonate	DuPont** Adiprene L-100 Urethane	Kasil 6	Potassium	.5 .5 .5	100 150 205	~23	A few dispersed bubbles
S265A	2"x2" Lexan Polycarbonate	DuPont** Adiprene L-100 Urethane	K	Sodium	.5 .5 .5	100 150 205	N.D.	Many bubbles
S266A	2"x2" Lexan Polycarbonate	DuPont** Adiprene L-100 Urethane	K	Sodium	.5 .5 .5	100 150 205	N.D.	Many bubbles
S287A	2"x2" Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	K	Sodium	.5 .5 .5	100 150 200	N.D.	Essentially no bubbles; slight removable haze
S294A	2"x2" Lexan Polycarbonate	DuPont Adiprene L-100 Urethane	Kasil 6	Potassium	.5 .5 .5	100 150 200	N.D.	Essentially no bubbles; crazed surface
S304	2"x2" Lexan Polycarbonate	None	K	Sodium	Room Temperature		5 mils	Initially acceptable (smooth and transparent) but cracked later.
S305	2"x2" Lexan Polycarbonate	None	K	Sodium	Room Temperature		10 mils	Initially acceptable (smooth and transparent) but cracked later.
S307	2"x2" Lexan Polycarbonate	None	K	Sodium	Room Temperature		20 mils	Initially acceptable (smooth and transparent) but cracked later.
S309	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	Room Temperature		5 mils	Initially acceptable (smooth and transparent) but cracked later.
S312	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	Room Temperature		10 mils	Initially acceptable (smooth and transparent) but cracked later.
S314	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	Room Temperature		20 mils	Initially acceptable (smooth and transparent) but cracked later.
S315	2"x2" Lexan Polycarbonate	None	N	Sodium	Room Temperature		5 mils	Initially acceptable (smooth and transparent) but cracked later.
S317	2"x2" Lexan Polycarbonate	None	N	Sodium	Room Temperature		10 mils	Initially acceptable (smooth and transparent) but cracked later.
S319	2"x2" Lexan Polycarbonate	None	N	Sodium	Room Temperature		20 mils	Initially acceptable (smooth and transparent) but cracked later.
S303	2"x2" Lexan Polycarbonate	None	K	Sodium	20	110	5 mils	Cracked; some bubbles; peeled off.
S306	2"x2" Lexan Polycarbonate	None	K	Sodium	20	110	10 mils	Cracked; Many bubbles; some peeling off.
S308	2"x2" Lexan Polycarbonate	None	K	Sodium	20	110	20 mils	Cracked; Many bubbles; some peeling off.
S310	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	110	5 mils	Cracked, a little peeling off.
S311	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	110	10 mils	Cracked; some bubbles; very little peeling off.
S313	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	110	20 mils	Cracked; few bubbles.
S316	2"x2" Lexan Polycarbonate	None	N	Sodium	20	110	5 mils	Cracked; peeling off
S318	2"x2" Lexan Polycarbonate	None	N	Sodium	20	110	10 mils	Cracked; some bubbles; peeling off.
S320	2"x2" Lexan Polycarbonate	None	N	Sodium	20	110	20 mils	Cracked; some bubbles; peeling off.
S321	2"x2" Lexan Polycarbonate	None	K	Sodium	20	150	5 mils	Cracked; some bubbles.
S322	2"x2" Lexan Polycarbonate	None	K	Sodium	20	150	10 mils	Cracked; many bubbles.
S323	2"x2" Lexan Polycarbonate	None	K	Sodium	20	150	20 mils	Cracked, many bubbles.

Cont'd Table III

Spec. Number	Substrate Type	Undercoating	Protective Coating				Coating Thickness mils	Appearance
			Type	Silicate	Bake Time, hr.	Bake Temp, °F		
S324	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	150	5 mils	Some cracks; few bubbles.
S325	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	150	10 mils	Some cracks; few bubbles.
S326	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	150	20 mils	Few cracks; many bubbles.
S327	2"x2" Lexan Polycarbonate	None	N	Sodium	20	150	5 mils	Cracked; few bubbles; some peeling off.
S328	2"x2" Lexan Polycarbonate	None	N	Sodium	20	150	10 mils	Cracked; some bubbles; some peeling off.
S329	2"x2" Lexan Polycarbonate	None	N	Sodium	20	150	20 mils	Cracked; many bubbles
S330	2"x2" Lexan Polycarbonate	None	K	Sodium	20	200	5 mils	Many bubbles.
S331	2"x2" Lexan Polycarbonate	None	K	Sodium	20	200	10 mils	Many bubbles.
S332	2"x2" Lexan Polycarbonate	None	K	Sodium	20	200	20 mils	Many small bubbles.
S333	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	200	5 mils	Coating white, opaque, and blistered.
S334	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	200	10 mils	Coating white, opaque, and blistered.
S-335	2"x2" Lexan Polycarbonate	None	Kasil 6	Potassium	20	200	20 mils	Coating white, opaque, and blistered.
S336	2"x2" Lexan Polycarbonate	None	N	Sodium	20	200	5 mils	Many bubbles.
S337	2"x2" Lexan Polycarbonate	None	N	Sodium	20	200	10 mils	Many bubbles.
S-338	2"x2" Lexan Polycarbonate	None	N	Sodium	20	200	20 mils	Many large bubbles.

N.D. - Not Determined due to degradation of the specimen

*Temperature at entry into oven

**These polyurethane undercoatings were room temperature air cured one day. In this case, a large proportion of the water contained in the silicate had evaporated prior to thermal curing. In most cases, bubbling was observed after short exposure to temperatures above 175°F and after long exposure to 100°-125°F. The absence of bubbling in the silicates (made on glass) indicated the possibility of some out-gassing of the polycarbonate/urethane. However, various specimens made with high temperature cured urethane undercoatings specimens 192, 194, 195, 196, 202, 203 did not appear appreciably better than those on uncured polyurethane. In most cases, bubbling was observed indicating the presence of water vapor inside the coating. Apparently, a watertight outer film forms during air drying and prevents the volatilization of water from inside of the coating.

Subsequently, a second study was conducted using specimens coated only with silicate. The specimens were placed in a preheated oven at 110°F. However, cracking and/or bubbling occurred in each silicate coating. It is apparent that water retention is critical in order to maintain a good coating. This can be observed with air-dried films which are allowed to remain in a low humidity environment as well as with oven-dried specimens. Cracking and fogging are observed when the water content of the coating falls below a minimum value. It does not appear that fluctuations in the oven-curing time can be used to improve this method.

(2) Chemical setting agents. A brief study was made to determine the feasibility of rendering the silicate film surface insoluble. Various silicate films were applied to glass slides and allowed to air dry. The chemical setting agents were applied from an aqueous solution (1 part H_2O to 1 part agent by weight) to the surface of the silicate. The results of this study are shown in Table IV. In most cases, the silicate surface was made either opaque or cloudy. The unaffected silicates were tested for water resistance and found to be soluble.

Table IV

EFFECT OF CHEMICAL SETTING AGENTS ON SILICATE FILM SURFACES

	Silicate Type				
Setting Agent	N Sodium Silicate	S-35 Sodium Silicate	K Sodium Silicate	Kasil 1 Potassium Silicate	Kasil 6 Potassium Silicate
Borax	Cloudy	Cloudy	Cloudy	Unaffected	Unaffected
$NaBO_2$	Unaffected	Cloudy	Unaffected	Unaffected	Unaffected
$MgSO_4$	White	White	Cloudy	Cloudy	Unaffected
$Al_2(SO_4)_3$	Cloudy	White	Cloudy	White	White

(3) Pressure Curing of Silicate Coatings. In order to observe the effect of pressure on the silicate coatings under investigation, a metallographic pressure curing chamber at Mound Laboratory, Miamisburg, Ohio, was utilized. This pressure chamber (of Monsanto design) was evacuated by a vacuum pump and then pressurized with argon. Although the pressure can be varied from 0-3000 psi, the pressure in the present work was maintained at 800 psi. The chamber temperature was held constant at 131°F by means of a Variac-controlled winding.

Two sets of samples were run in this pressure experiment. One set was exposed in the pressure chamber for 3 1/2 hours. Then the most promising specimens were prepared again and cured overnight. The results of these two runs are presented in Table V. The silicate liquids used in this work were Kasil 6, Kasil 1, Kasil 88, Kasil 6 + 0.2% Sterox, and Kasil 6 + 0.2% isopentylamine. Sterox and isopentylamine are surfactants and were included in this study hopefully to improve the wetting characteristics of the silicate liquid, giving a better bond to the substrate. In all cases, the silicate liquid was applied to either an uncoated or a polyurethane-coated polycarbonate substrate. Following the curing time in the pressure chamber, the samples were air dried overnight and subsequently dried overnight in a recirculating oven at 150°F. This drying caused deterioration of every sample, inducing more bubbles, milkiness, and some curling of the silicate coating.

The desired effect of this pressure mounting was to induce better bonding of the silicate liquid to the respective substrates. This effect was not observed; every coating was still soft after curing under pressure. Apparently, the pressure in the chamber was detrimental to the curing of the silicate coatings. All of the water in the silicate could not escape to allow complete curing of the coatings as a rigid film; hence, coatings remained in a plastic state.

d. Cold Forming and Polishing of Silicate Coatings. In most cases, further surface finishing was required to eliminate optical distortion. Two methods have been attempted to achieve this. The first method utilizes the cold flow properties of the silicate. Air dried silicates tend to increase in hardness with age because of water loss. After about 24 hours at 50% relative humidity, the coatings are hard enough to handle without leaving fingerprints. Under pressure, however, the films will flow. If a polycarbonate sheet with minimum optical distortion is placed on the silicate coating and pressure then applied, the silicate surface will conform to the polycarbonate and form an optically flat surface. Preliminary cold-forming studies were made using thick, type K, air-dried silicate coatings. Pressing time and

Table V

PRESSURE MOUNTED SILICATE COATINGS

Sample	Substrate		Undercoating	Coating			Appearance
	Type	Size		Type	Bake Hr	Temp °F	
S-212	Polycarbonate	1"x1"	none	Kasil 6	3 1/2	131	surface bubbles but smooth; soft, no bond
S-213	Polycarbonate	1"x1"	Polyurethane	Kasil 6	3 1/2	131	surface bubbles but smooth; soft, no bond
S-214	Polycarbonate	1"x1"	none	Kasil 1	3 1/2	131	few bubbles, smooth, poor bond
S-215	Polycarbonate	1"x1"	Polyurethane	Kasil 1	3 1/2	131	very bubbly; poor bond
S-216	Polycarbonate	1"x1"	none	Kasil 88	3 1/2	131	very bubbly; poor bond
S-217	Polycarbonate	1"x1"	Polyurethane	Kasil 88	3 1/2	131	some bubbles, but smooth; poor bond
S-218	Polycarbonate	1"x1"	none	Kasil 6 water Sterox	3 1/2	131	very bubbly; poor bond
S-219	Polycarbonate	1"x1"	Polyurethane	Kasil 6 water Sterox	3 1/2	131	many bubbles; better bond
S-220	Polycarbonate	1"x1"	none	Kasil 6 water isopentylamine	3 1/2	131	many bubbles; poor bond
S-221	Polycarbonate	1"x1"	Polyurethane	Kasil 6 water isopentylamine	3 1/2	131	many bubbles; poor bond
S-222	Polycarbonate	1"x1"	none	Kasil 6	17	131	some bubbles, poor bond; soft
S-223	Polycarbonate	1"x1"	Polyurethane	Kasil 6	17	131	more bubbles, better bond; soft
S-224	Polycarbonate	1"x1"	none	Kasil 1	17	131	some big bubbles; poor bond
S-225	Polycarbonate	1"x1"	Polyurethane	Kasil 1	17	131	many bubbles; poor bond

ram force were varied in order to determine their effect. The silicate coatings formed for a short time under high pressure tended to lose adhesion along the edges. Improved coating adhesion was obtained using a long pressing time and low pressure. However, the pressed coatings tended to experience surface changes after several days, indicating the presence of residual stresses. Additional coatings made from both K sodium silicate and Kasil 6 potassium silicate produced similar results. A summary of pressing conditions and results is shown in Table VI. Figure 1 shows the degree of distortion of a cold-formed coating when compared to the uncoated polycarbonate, urethane coated polycarbonate, and drawn silicate, urethane-polycarbonate system. The slight distortion of the grid lines for the cold-formed coating indicates the specimen is somewhat distorted probably due to residual stresses in the coating.

The second method involved surface polishing. The coatings were either placed on a rotating lap and sanded using an ethanol coolant or block sanded both manually and mechanically. The primary difficulty encountered was overheating and subsequent bubbling of the silicate. This problem was eliminated by carefully controlling the sanding rate.

The best results were obtained by block sanding with Luna Pregrinder 320 and 400 grit papers, respectively. The resultant coatings were translucent. When protective coatings of polymers were applied, the transparency of the silicate was recovered, yielding a transparent panel free of distortion.

e. Water Repellant Coatings for Alkali Silicates. Early in the program, the need to prevent the silicate from degrading due to humidity variations was observed. Various attempts, e.g., by oven drying, etc., were made to physically change the silicate. Finally, an attempt was made to apply a moisture barrier film over the silicate in order to both retain the water contained in the specimen and to prevent the absorption of water from the atmosphere. Usually the coatings were air dried until they were hard enough to be sanded. Immediately after sanding the protective coating was applied. Eleven commercial polymers were selected for use as possible water repellant films on silicate. These are shown in Table VII. Materials were selected for transparency, ease of application, and water vapor permeability.

The best results were obtained with DuPont Duco polystyrene and Hughes Chemical Co. Chemglaze polyurethane. The coatings produced were transparent with minimal bubbling or surface distortion. Some difficulty was encountered in attempting to produce quality coatings of Krylon 1302 and Aqua Guard acrylic. Apparently, both materials tend to crack the silicate soon after application. Crazing occurred in Owens Corning 650 glass resin coatings after dipping and air drying.

Table VI

COLD FORMING OF SILICATE COATINGS

Spec. No.	Type	Silicate	Ram Force, ton	Approximate Pressure, lb/in^2	Pressing Time	Appearance After
S236	K	Sodium	$\sim$8	$\sim$1777	$\sim$60 sec	Some wavy distortion and loss of coating adhesion on edges
S256	K	Sodium	$\sim$4	$\sim$888	$\sim$60 sec	Some wavy distortion and loss of coating adhesion on edges
S257	K	Sodium	$\sim$4	$\sim$888	$\sim$60 sec	Some wavy distortion and loss of coating adhesion on edges
S276	K	Sodium	$\sim$2	$\sim$500	$\sim$17 hrs	Good coating; essentially distortion-free
S273	K	Sodium	.5	$\sim$250	$\sim$ 1 hr	Good coating; essentially distortion-free
S275	K	Sodium	.5	$\sim$250	$\sim$24 hrs	Good coating; essentially distortion-free
S398	K	Sodium	800	200	24	Good distortion free film
S403	K	Sodium	5,000	1,250	1/12	Coating cracked
S395	K	Sodium	5,000	1,250	1/4	Coating cracked
S361	K	Sodium	10,000	2,500	1	Coating cracked
S400	K	Sodium	15,000	3,750	1/4	Coating cracked
S402	Kasil 6	Potassium	800	200	25	Good partically flattened film
S401	Kasil 6	Potassium	15,000	3,750	1	Good distortion free film
S495	Kasil 6	Potassium	400	100	48	100% distortion free; silicate flowed from surface resulting in a thin silicate coating
S468*	Kasil 6	Potassium	800	200	66	Flat silicate film
S469*	Kasil 6	Potassium	1,200	300	73	Distortion free; small area spalled from surface; silicate very thin due to flowing.
S459	Kasil 6	Potassium	2,000	500	1	Some residual distortion 90% distortion free
S458	Kasil 6	Potassium	2,400	600	1	75% distortion free; poor silicate bonding
S420*	Kasil 6	Potassium	4,800	1,200	24	Flat silicate surface
S481*	K	Sodium	400	100	100	100% distortion free; silicate very thin due to flowing
S465*	K	Sodium	800	200	1	$\sim$75% distortion free
S464*	K	Sodium	1,200	300	1	$\sim$75% distortion free; some silicate cracking
S466*	K	Sodium	1,600	400	1	$\sim$25% distortion free; several distorted areas
S461*	K	Sodium	2,000	500	1	$\sim$75% distortion free
S467*	K	Sodium	2,400	600	1	100% distortion free; silicate very thin

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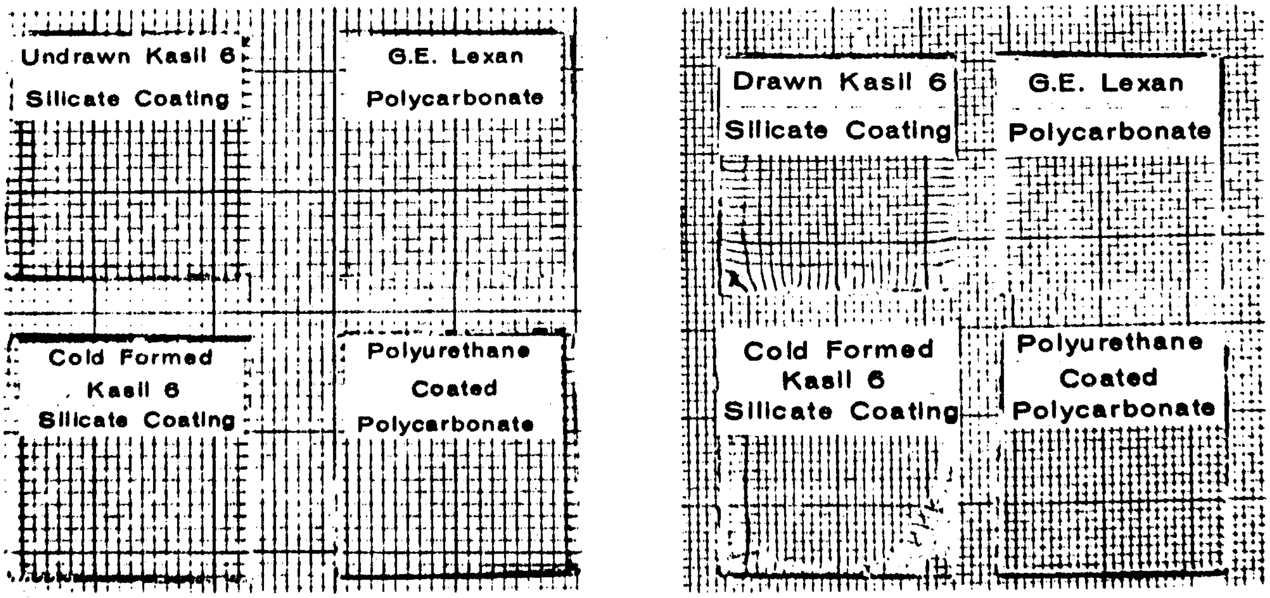
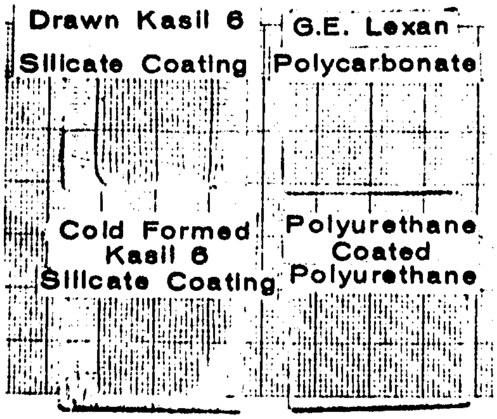
*Specimens coated after pressing with an enamel moisture resistant coating (Waterspar clear enamel).



Specimens Mounted on
Surface of Grid

Specimens Mounted 4 3/4
Inches From Grid



Specimens Mounted 9 3/4
Inches From Grid

Figure 1. Comparison of Distortion in Silicate-Urethane-Polycarbonate System.

Table VII

WATER REPELLANT MATERIALS FOR ALKALI SILICATE COATINGS

Material	Trade-name	Supplier	Application Method
Acrylic	Waterspar Clear Enamel	Pittsburgh Paints	Spray
Acrylic	Lucite 140	Dupont	Spray
Acrylic	Krylon # 1302	Krylon, Inc.	Spray
Acrylic	Aqua Guard	Micro Chem. Inc.	
Polycarbonate	Lexan	General Electric	Spray
Polystyrene	Styron 700	Dow Corning	Spray
Polystyrene	Duco	Dupont	Doctor Blade
Polyurethane	Adiprene L-100	Dupont	Doctor Blade
Polyurethane	Adiprene L-100	Dupont	Spray
Polyurethane	Chemglaze	Hughson Chemical Co.	Spray
Silicone	RTV	General Electric	Doctor Blade
Silicone	Glass Resin #650	Owens Illinois	Dipor Spray

A vital question at this time would be: How can a flammable polymeric film be used as a component in a system designed to act as a fire barrier? Others have observed that certain flammable materials when applied to a nonflammable material (such as silicate) are nonflammable in the form of a coating (Ref. 3). Initial studies conducted at NASA using a flame propagation test on several of our silicate specimens confirmed this fact. The polymer did not burn beyond the point of actual flame impingement. It is practical, therefore, to consider polymeric water-repellant coatings even though the overall polycarbonate-urethane-silicate-protective coating system is intended to be a fire protection coating. A summary of the protective coating studies is given in Table VIII.

2. Coating Testing and Evaluation.

a. Humidity Resistance. Eight silicate coated polycarbonate samples were used to observe the effect of a high humidity environment on these materials. The samples were exposed in a humidity cabinet, with the temperature, humidity, and percent weight change being noted at certain intervals. The samples were exposed for no less than 43 hours, with some test runs lasting 187 hours. The humidity test results are summarized in Table IX.

Initially, coating failures were experienced with both silicate types tested, regardless of the type of curing cycle (room temperature cured or thermally cured) used. Varying degrees of peeling, cracking, and whitening (of the silicate coating) were observed on all unprotected silicate coatings. Two samples were then coated with clear acrylic lacquer in order to observe its moisture resistance and compatibility with the silicate. These protected silicate coatings showed no degradation whatsoever, even after 187 hours in the humidity chamber. Figure 2 shows a comparison of the protected and unprotected silicate coatings after 187 hours at high humidity. The unprotected coatings (S-241 and S-285) are dried out and whitened, while the protected coatings (S-252 and S-286) remained unchanged.

b. Thermal Shock Resistance. The thermal shock resistance of two alkali silicate compositions (Type K [sodium] and Type Kasil 6 [potassium]) was checked. One specimen (alkali silicate on polycarbonate) of each composition was heated in an air oven to 150°F for approximately 30 minutes and then quenched in a Dewar cooled by liquid nitrogen to -90°F. The Dewar temperature was determined with a potentiometer reading the output of a copper-constantan thermocouple.

Table VIII

SILICATE PROTECTED WITH WATER REPELLANT FILMS

						Protective Coating			
Substrate Size	Spec. Nos.	Type	Silicate	Silicate Pretreatment	Silicate Thickness, mil	Type	Nominal Thickness, mil	Appearance	Application Method
2"x2"	S380-385	K	Sodium	None	N.D.	Waterspar clear enamel	N.D.	Transparent film	Sprayed
2"x2"	S412, 416, 419, 420	Kasil 6	Potassium	Buffed with wire-wheel	16-19	Waterspar clear enamel	1.5	Transparent film	Sprayed
2"x2"	S409, 415, 417	Kasil 6	Potassium	Sanded with grit-paper	15-20	Waterspar clear enamel	N.D.	Transparent film	Sprayed
2"x2"	S471, 473	Kasil 6	Potassium	Sanded with grit-paper	N.D.	Waterspar clear enamel	N.D.	Transparent film	Sprayed
2"x2"	S482	K	Sodium	Sanded with grit-paper	44	Waterspar clear enamel	<1	Transparent film	Sprayed
2"x2"	S483-485	K	Sodium	Sanded with grit-paper	N.D.	Waterspar clear enamel	<1	Transparent film	Sprayed
2"x2"	S487-488	Kasil 6	Potassium	None	N.D.	DuPont Adiprene L-100 Polyurethane	N.D.	Poorly adhered film	Sprayed
2"x2"	S447, 479	K	Sodium	None	N.D.	DuPont Adiprene L-100 Polyurethane	N.D.	Poorly adhered film	Sprayed
2"x2"	S470, 478	Kasil 6	Potassium	None	32, 19	DuPont Adiprene L-100 Polyurethane	~2	Transparent film	Drawn
2"x2"	S486	K	Sodium	None	20	DuPont Adiprene L-100 Polyurethane	~1	Transparent film	Drawn
2"x2"	S490	K	Sodium	None	24	DuPont Adiprene L-100 Polyurethane	~14	Transparent film	Drawn
2"x2"	S492, 496	Kasil 6	Potassium	None	25, 22	DuPont Duco Polystyrene	~1.5	Transparent film	Drawn
2"x2"	S480	K	Sodium	None	45	DuPont Duco Polystyrene	N.D.	Transparent film	Drawn
2"x2"	S497-498	K	Sodium	None	N.D.	DuPont Duco Polystyrene	N.D.	Transparent film	Drawn
2"x2"	S514	K	Sodium	None	39	DuPont Lucite 140	~5	Transparent film	Sprayed
2"x2"	S511	K	Sodium	None	57	Dow Styron 700	~6	Transparent; some crazed areas.	Sprayed
2"x2"	S516	K	Sodium	None	50	General Electric Lexan Polycarbonate	~4	Brittle grey film	Sprayed
2"x2"	S475-476	Kasil 6	Potassium	None	N.D.	General Electric RTV108 Silicone	~2.5	Poorly adhered grey film	Spatula
2"x2"	S489, 491	K	Sodium	None	N.D.	General RTV108 Silicone	~10	Poorly adhered grey film	Spatula
2"x2"	S527	Kasil 6	Potassium	400 grit paper sanded lightly	29	Krylon 1302	6	Transparent film some surface and bubble distortion.	Sprayed
2"x2"	S528, 532, 534	Kasil 6	Potassium	400 grit paper sanded flat	15-64	DuPont Duco Polystyrene	2	Transparent; a few bubbles.	Drawn
2"x2"	S535, 538	Kasil 6	Potassium	400 grit paper sanded lightly	35, 34	Aqua Guard**	3	Developed cracked areas in silicate.	Sprayed
2"x2"	S541, 546	Kasil 6	Potassium	400 grit paper sanded lightly	24, 30	Krylon 1302**	1	Developed cracked areas in silicate.	Sprayed
2"x2"	S549, 550	Kasil 6	Potassium	400 grit paper sanded lightly	33, 35	Owens Corning 650 Glass Resin	1	Crazed soon after application.	Dipped
2"x2"	S543, 573	Kasil 6	Potassium	400 grit paper sanded flat	29	Hughson Chemical Co. Chemglaze Polyurethane	1	Transparent coating; little optical distortion.	Sprayed
2"x2"	S574, 575	Kasil 6	Potassium	400 grit paper sanded flat	16, 24	DuPont Duco Polystyrene	1	Transparent optically distortion free coating; a few bubbles.	Drawn
2"x2"	S590-594	Kasil 6	Potassium	400 grit paper sanded flat	16-25	Hughson Chemical Co. Chemglaze Polyurethane	1	Transparent; little distortion.	Sprayed
2" x 3 1/2"	S595, 596	Kasil 6	Potassium	400 grit paper sanded lightly	22, 23	Hughson Chemical Co. Chemglaze Polyurethane	11	Transparent; little distortion.	Sprayed
2"x2"	S589	Kasil 6	Potassium	400 grit paper sanded flat	18	Hughson Chemical Co. Chemglaze Polyurethane	11	Transparent; little distortion.	Sprayed
2"x2"	S585, 587	Kasil 6	Potassium	400 grit paper sanded lightly	18, 19	Hughson Chemical Co. Chemglaze Polyurethane	1	Transparent; little distortion.	Sprayed
2"x2"	S586	Kasil 6	Potassium	400 grit paper sanded lightly	18	Hughson Chemical Co. Chemglaze Polyurethane	0.5	Transparent; little distortion.	Sprayed
2"x2"	S588	Kasil 6	Potassium	400 grit paper sanded lightly	18	Hughson Chemical Co. Chemglaze Polyurethane	<1	Transparent; little distortion.	Sprayed
2"x2"	S580-583	Kasil 6	Potassium	400 grit paper sanded flat	N.D.	Hughson Chemical Co. Chemglaze Polyurethane	1	Transparent; little distortion.	Sprayed
3"x6"	S597	Kasil 6	Potassium	400 grit paper sanded flat	19	DuPont Duco Polystyrene	2	Transparent; little distortion.	Drawn
3"x6"	S598	Kasil 6	Potassium	400 grit paper sanded flat	17	DuPont Duco Polystyrene	1	Transparent; little distortion	Drawn
2"x2"	S599, 601-604	Kasil 6	Potassium	400 grit paper sanded flat	20-22	Hughson Chemical Co. Chemglaze Polyurethane	1	Transparent; little distortion.	Sprayed

Cont'd Table VIII

Substrate Size	Spec. Nos.	Type	Silicate	Silicate Pretreatment	Silicate Thickness, mil	Protective Coating Type	Protective Coating Nominal Thickness, mil	Appearance	Application Method
2"x2"	S600	Kasil 6	Potassium	400 grit paper sanded flat	13	Hughson Chemical Co. Chemglaze Polyurethane	2	Transparent; little distortion.	Sprayed
1 1/2x1 1/2	S613	Kasil 6	Potassium	400 grit paper sanded flat	-	Hughson Chemical Co. Chemglaze Polyurethane	N.D.	Transparent; little distortion.	Drawn
1 1/2x1 1/2	S615	Kasil 6	Potassium	400 grit paper sanded flat	-	Hughson Chemical Co. Chemglaze Polyurethane	N.D.	Transparent; little distortion.	Dipped
10"x10"	S620	Kasil 6	Potassium	400 grit paper sanded flat	18	Hughson Chemical Co. Chemglaze Polyurethane	3.0	Transparent; slight haze.	Dipped
10"x10"	S623	Kasil 6	Potassium	400 grit paper sanded flat	10	Hughson Chemical Co. Chemglaze Polyurethane	7.0	Transparent; slight haze.	Dipped
10"x10"	S625	Kasil 6	Potassium	400 grit paper sanded flat	15	Hughson Chemical Co. Chemglaze Polyurethane	0.75	Transparent; slight haze.	Dipped
10"x10"	S626	Kasil 6	Potassium	400 grit paper sanded flat	15	Hughson Chemical Co. Chemglaze Polyurethane	2.5	Transparent; slight haze.	Sprayed
10"x10"	S634	Kasil 6	Potassium	400 grit paper sanded flat	20	Hughson Chemical Co. Chemglaze Polyurethane	4	Transparent; slight haze.	Dipped
10"x10"	S637	Kasil 6	Potassium	400 grit paper sanded flat	4	DuPont Duco Polystyrene	2	Transparent; slight haze.	Drawn
10"x10"	S657	Kasil 6	Potassium	400 grit paper sanded flat	6	Hughson Chemical Co. Chemglaze Polyurethane	6	Transparent; slight haze.	Dipped
10"x10"	S658	Kasil 6	Potassium	400 grit paper sanded flat	6	Hughson Chemical Co. Chemglaze Polyurethane	2	Transparent; less haze.	Dipped
1 1/2x1 1/2	S641-649	K	Sodium	400 grit sanded lightly	15-25	Hughson Chemical Co. Chemglaze Polyurethane	2	Transparent; less haze.	Dipped
1 1/2x1 1/2	S650-655 660, 662	Kasil 6	Potassium	400 grit sanded lightly	17-24	Hughson Chemical Co. Chemglaze Polyurethane	2	Transparent; less haze.	Dipped
10"x10"	S669	K	Sodium	400 grit sanded flat	29	Hughson Chemical Co. Chemglaze Polyurethane	-10	Transparent; little haze.	Dipped
10"x10"	S670	K	Sodium	400 grit sanded flat	29	Hughson Chemical Co. Chemglaze Polyurethane	-10	Transparent; little haze.	Dipped
10"x10"	S671	K	Sodium	400 grit sanded flat	19	Hughson Chemical Co. Chemglaze Polyurethane	-10	Transparent; little haze.	Dipped
10"x10"	S672	K	Sodium	400 grit sanded flat	27	Hughson Chemical Co. Chemglaze Polyurethane	-10	Transparent; little haze.	Dipped
10"x10"	S673	Kasil 6	Potassium	400 grit sanded flat	27	DuPont Duco Polystyrene	3	Transparent; some distortion.	Dipped
10"x10"	S664	Kasil 6	Potassium	400 grit sanded flat	10	Hughson Chemical Co. Chemglaze Polyurethane	~10	Transparent and clear	Dipped
10"x10"	S666	Kasil 6	Potassium	400 grit sanded flat	12	Hughson Chemical Co. Chemglaze Polyurethane		Transparent and clear	Dipped
10"x10"	S675	Kasil 6	Potassium	400 grit sanded flat	~6	Hughson Chemical Co. Chemglaze Polyurethane	~3	Transparent; unclear	Dipped

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Sprayed polyurethane coatings made using a 2 parts toluene--1 part polyurethane by volume mixture.

Dipped polyurethane coatings made using as received polyurethane

* Thickness after silicate pretreatment

** Micro Chemicals, Inc., Dallas, Texas

*** Krylon Inc. Norristown, Pa.

N.D.-Not Determined

Table III

HUMIDITY RESISTANCE OF SILICATE COATINGS

Spec. No.	Type	Silicate	Protective Coating Type	Time, hr.	Temp. °F	Relative Humidity	Percent Weight Change, %	Appearance
S236A	K	Sodium	None	0	---	--	0	Transparent, bubbled coating
S236A	K	Sodium	None	19	---	--	+5.5	No change
S236A	K	Sodium	None	26	---	--	+5.4	No change
S236A	K	Sodium	None	43	---	--	+6.4	No change; test terminated
S238	K	Sodium	None	0	100	98	0	Rough, cloudy film
S238	K	Sodium	None	19	100	98	+0.96	Transparent film; no haze
S238	K	Sodium	None	26	100	98	+1.41	Transparent film; no haze
S238	K	Sodium	None	43	100	98	+1.14	Transparent film
S238	K	Sodium	None	50	100	98	+1.27	No change
S238	K	Sodium	None	67	100	98	+1.90	No change
S238	K	Sodium	None	187	100	98	-	Coating failed along edges and peeled; test terminated
S241	K	Sodium	None	0	100	98	0	Transparent film; some surface haze
S241	K	Sodium	None	19	100	98	+1.0	Transparent, glassy film; no surface haze
S241	K	Sodium	None	26	100	98	+1.25	Transparent, glassy film; no surface haze
S241	K	Sodium	None	43	100	98	+1.04	Transparent, glassy film; no surface haze
S241	K	Sodium	None	50	100	98	+1.13	No change
S241	K	Sodium	None	67	100	98	+2.02	No change
S241	K	Sodium	None	187	100	98	-	Peeling; white areas and cracks; test terminated
S252	Kasil 6	Potassium	Acrylic Lacquer*	0	100	98	+0	Transparent, glassy film
S252	Kasil 6	Potassium	Acrylic Lacquer*	19	100	98	+1.0	No change
S252	Kasil 6	Potassium	Acrylic Lacquer*	26	100	98	+1.19	No change
S252	Kasil 6	Potassium	Acrylic Lacquer*	43	100	98	+1.33	No change
S252	Kasil 6	Potassium	Acrylic Lacquer*	50	100	98	+1.43	A few cracks and white areas
S252	Kasil 6	Potassium	Acrylic Lacquer*	67	100	98	+1.68	No change
S252	Kasil 6	Potassium	Acrylic Lacquer*	187	100	98	+2.06	No change; test terminated
S256A	Kasil 6	Potassium	None	0	---	--	0	Transparent, bubbled coating
S256A	Kasil 6	Potassium	None	19	---	--	+0.20	No change
S256A	Kasil 6	Potassium	None	26	---	--	+0.55	No change
S256A	Kasil 6	Potassium	None	43	---	--	+1.03	Cloudy along edges; very soft; coating removed
S285	K	Sodium	None	0	100	98	0	Transparent glassy film
S285	K	Sodium	None	19	100	98	+3.27	No change
S285	K	Sodium	None	26	100	98	+0.96	No change
S285	K	Sodium	None	43	100	98	+1.1	No change
S285	K	Sodium	None	50	100	98	+1.43	Small white spot; specimen ok
S285	K	Sodium	None	67	100	98	+1.92	No change
S285	K	Sodium	None	187	100	98	-	Coating failed; cracks around edges
S286	K	Sodium	Acrylic Lacquer*	0	100	98	0	Transparent film; some surface haze
S286	K	Sodium	Acrylic Lacquer*	19	100	98	-1.04	Transparent glassy film; surface haze diminished
S286	K	Sodium	Acrylic Lacquer*	26	100	98	+1.83	Transparent glassy film; no haze
S286	K	Sodium	Acrylic Lacquer*	43	100	98	+2.03	No change
S286	K	Sodium	Acrylic Lacquer*	50	100	98	+2.15	No change
S286	K	Sodium	Acrylic Lacquer*	67	100	98	+2.52	No change
S286	K	Sodium	Acrylic Lacquer*	187	100	98	+2.81	No change; specimen ok; test terminated
S290	Kasil 6	Potassium	None	0	100	98	0	Transparent, glassy film
S290	Kasil 6	Potassium	None	19	100	98	+1.11	Cloudy haze over surface; some loss of coating adhesion; coating removed
S290	Kasil 6	Potassium	None	26	---	--	-----	Cloudy haze over surface; some loss of coating adhesion; coating removed
S290	Kasil 6	Potassium	None	43	---	--	-----	Cloudy haze over surface; some loss of coating adhesion; coating removed

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*Pittsburgh Paints Waterspar Clear Enamel

NOT REPRODUCIBLE

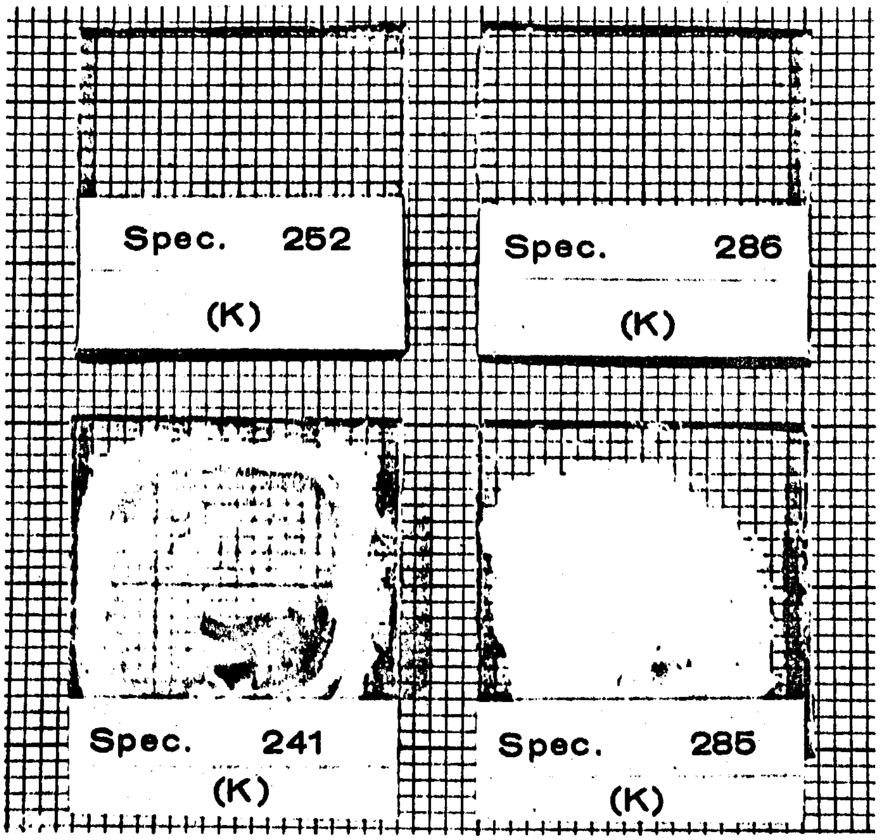


Figure 2. Appearance of Silicate Coatings After 187 Hour Exposure to High Humidity Environment

c. Flame Resistance. To determine the flame resistance of the alkali-silicate coatings on site, a test was devised for exposing the samples to an1800°-2000°F propane combustion flame in air. A schematic of the setup is shown in Figure 3. The test involved establishing the temperature of the flame with a Pt-Pt10Rh thermocouple and an optical pyrometer as a function of the distance from the substrate surface to the tip of the flame. Then by varying the substrate to flame distance, a preselected firing temperature could be achieved, and the burn-through time recorded. Using this procedure, flame tests were made to determine the flame protection properties of silicate-coated polycarbonate as a function of coating thickness. The specimens were evaluated by observing the time required for the flame to: (1) penetrate the silicate; (2) ignite the urethane and/or polycarbonate; and (3) penetrate the polycarbonate panel. Figure 4 shows two of the silicate specimens after the flame test. It is realized that this test is less severe than a comparable test in a pure oxygen atmosphere. However, because the silicate is noncombustible, it seems reasonable to assume that comparable silicate burn-through or failure times would be achieved, provided the temperatures were comparable. The results of flame tests performed on site are shown in Figure 5 and listed in Table X. As expected, the results show that the silicate burn-through time is increased as the coating thickness is increased.

The flame resistance standard set by NASA(Houston) called for the coating to withstand a direct flame of 1800°F for 45 seconds. As can be seen in Figure 5 this standard has been realized and surpassed by the silicate coatings developed in this laboratory. The best results on site were obtained on a 55 mil, Type K, sodium silicate coating which withstood 1800°F for ~150 seconds. The best result reported, on test samples sent to NASA(Houston) from this laboratory, indicates that a 25 mil silicate coating withstood 1800°F in a 6.2 psi oxygen atmosphere for 80 seconds without failing completely. These results are quite promising as they indicate that alkali silicate coatings can be produced which offer good flame resistance for polycarbonate substrates.

d. Optical Uniformity and Distortion. Federal Test Method 3041 was used for determining the optical uniformity and visual distortion of selected silicate-coated polycarbonate samples. According to this procedure, the optical characteristics of the samples are determined using a slide projector, with a transparent slide having two fine block lines (at right angles) ruled on it. The projector is placed 25 feet from a 5-foot square screen with a centered cross consisting of seven horizontal and seven vertical 1/16" lines intersecting at right angles in the center of the screen. With this setup, optical distortion is determined by placing the specimen close to and parallel with the screen, followed by

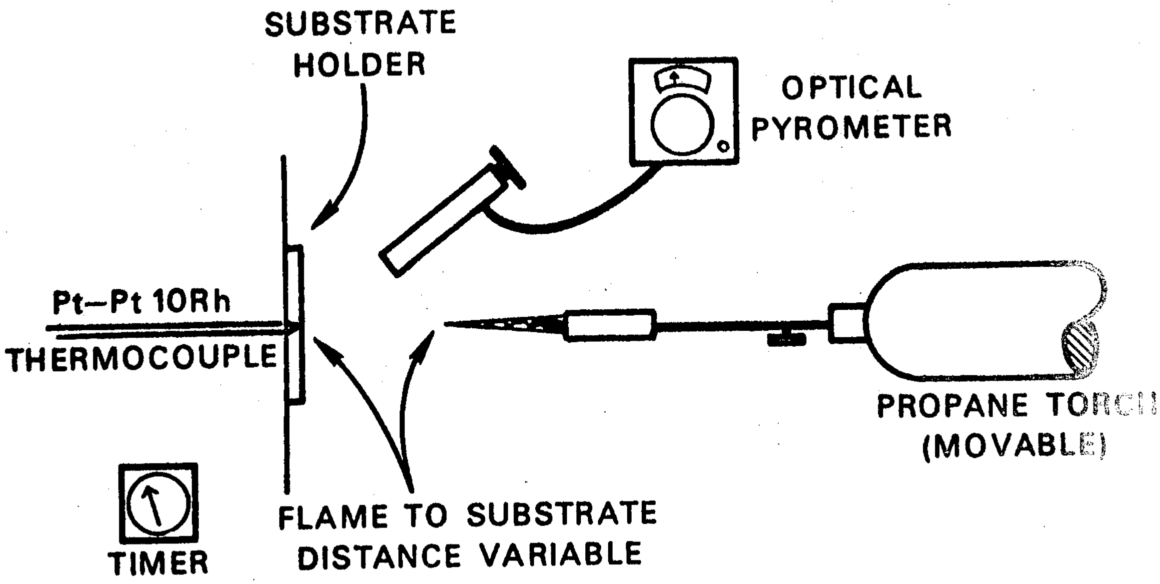


Figure 3. Top View Schematic of Flame Resistance Test

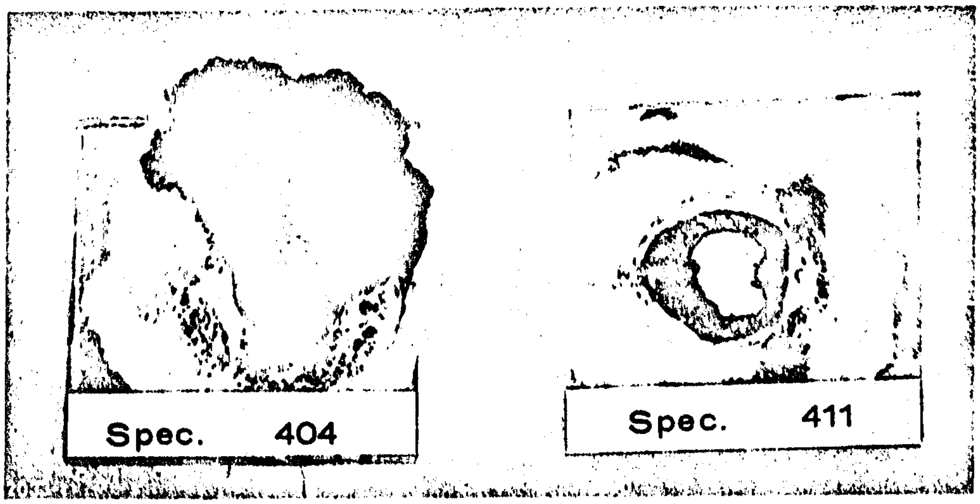


Figure 4. Appearance of Silicate Coatings After Exposure to 1800°F-2000°F Combustion Flame

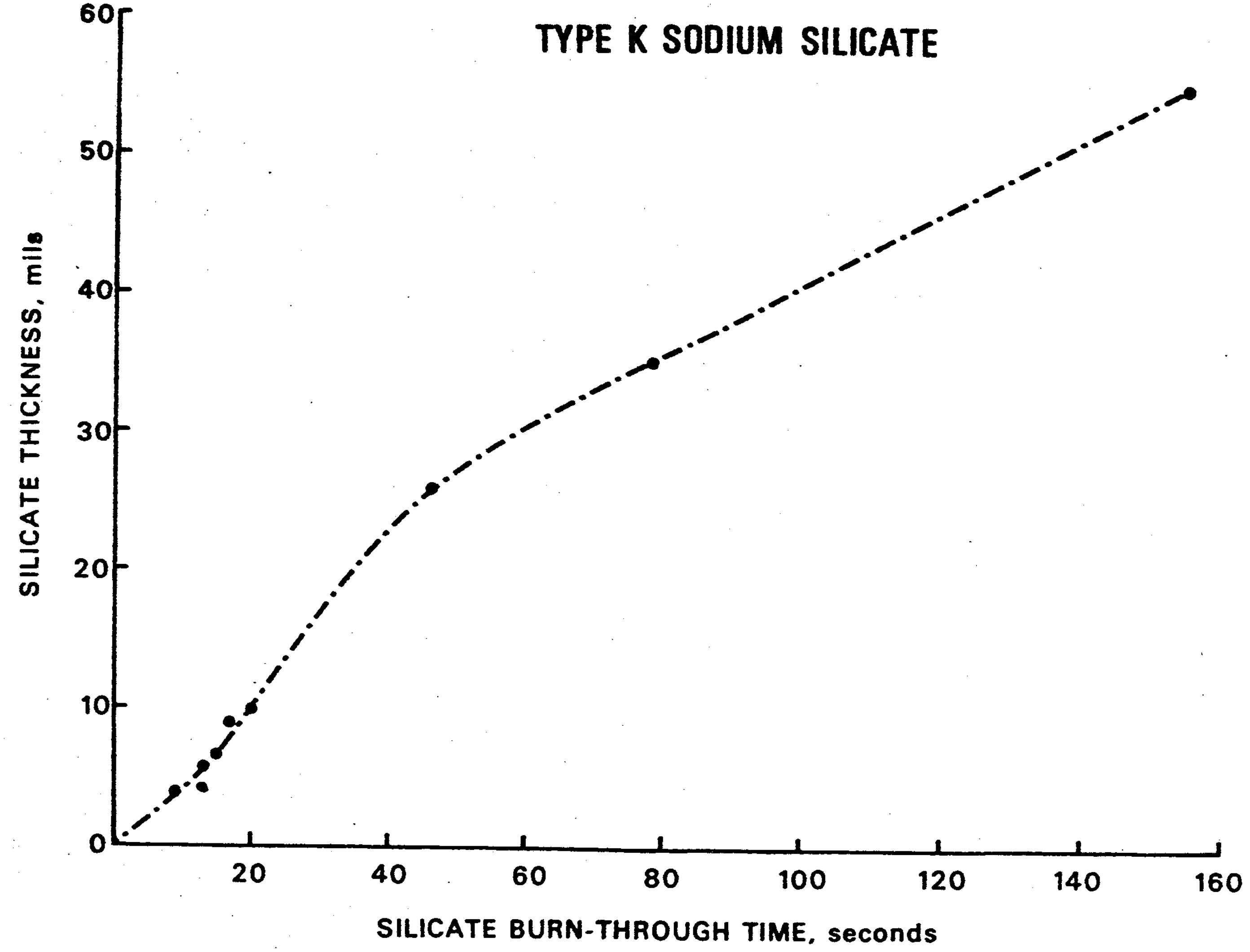


Figure 5. Graph of Flame Resistance Test (Propane Torch) Results for Type K Silicate Coated Polycarbonate Specimens

Table X

EFFECT OF A PROPANE TORCH FLAME ON SILICATE-COATED POLYCARBONATE[a]

Spec. No.	Silicate Coating Type	Silicate Coating Silicate	Coating Thickness, mils	Specimen Size, in^2	Silicate Burn Through Time, sec.	Polycarbonate and/or Urethane Ignition Time, sec.	Polycarbonate Burn Through Time, sec.
S355	K[b]	Sodium	3.9	2 x 2	~9	~20	~105
S356	K	Sodium	4.2	2 x 2	~13	~20	~93
S357	K	Sodium	5.7	2 x 2	~13	~23	~95
S358	K	Sodium	6.6	2 x 2	~15	~25	~90
S359	K	Sodium	9.1	2 x 2	~17	~25	~105
S360	K	Sodium	9.9	2 x 2	~20	~25	~90
S396	K	Sodium	26.	2 x 2	~46	~70	~200
S404	K	Sodium	35.	2 x 2	~78	~100	~245
S411	K	Sodium	55.	2 x 2	~150	~200	~365

[a] Flame temperature at point of impingement on test specimens was 1800-2000°F as determined via 90% Platinum - 10% Rhodium - Platinum thermocouple.

[b] Philadelphia Quartz Company

Note - All polycarbonate substrates were coated with Dupont adiprene L-100 urethane prior to silicate deposition.

movement of the specimen toward the projector. When light and dark shadows first appear in the shadow the distance from the screen is measured and recorded. This distance is indirectly proportional to the distortion caused by the specimen.

Optical uniformity is determined by placing the sample 12 inches from the front lens of the projector and parallel to the transparent slide (previously positioned and focused so that the sharply defined cross is coincident with the center cross ruled on the screen). While moving the specimen within its plane, the screen is observed for movement of the projected image of the cross. The specimen is rotated through 90° in its own plane and the test repeated. The maximum amount and nature or frequency of movement of the image is recorded.

By using the above-described procedures, a quantitative value for the optical uniformity and the optical distortion was assigned to selected polycarbonate samples. The resulting data are included in Table XI. Initial values (Part A) were obtained to establish upper and lower limits for comparison with later data. The results show a value of 8" for an optically perfect polycarbonate plate, while poor quality samples show readings near 0. Urethane-coated polycarbonate shows a drop in the optical distortion values to approximately 2 1/2". Therefore, as long as a urethane coating is used as the adhesive layer, the best distortion values will be in the 2"-3" range. For comparison with this range of values, further measurements were made (Part B). The results (show quite a few samples with the optimum distortion values) provided a rough indication of the quality of samples producible.

The optical uniformity values do not seem to provide any meaningful information which can be correlated with the coatings. As a result, this portion of the test method was not considered beyond the preliminary readings (shown in Table XI, Part C).

e. Light Transmittance. Tests were made to determine the light transmittance data, between 350 and 750 millimicrons, for polycarbonate/urethane/alkali silicate/protective polymer systems (PUSP systems). The data were obtained from a Perkin-Elmer Model 450 recording spectrophotometer. Figure 6 shows the data for uncoated polycarbonate, urethane-coated polycarbonate, and silicate/urethane/polycarbonate samples. Little effect is observed in the visible range, but the urethane film acts as a filter in the ultraviolet range. These data indicate that the alkali silicate transmits essentially 100% of the light energy. Figure 7 shows the light transmittance data as a function of wavelength for each of the PUSP specimens (polyurethane protective coating) listed. These samples show a range of transmittance

Table XI

Part A

OPTICAL DISTORTION VALUES FROM FEDERAL TEST METHOD 3041

Spec. No.		Sample	Distance
		Lexan circular plate	8"
		Lexan plate (commercial)	7 1/2"
		Urethane coated Lexan 1	2 1/4"
		Urethane coated Lexan 2	2 1/4"
S-235	K	Silicate/Urethane-coated polycarbonate	1 5/8"
S-284	K	Silicate/Urethane-coated polycarbonate	1 1/4"
S-243	K	Silicate/Urethane-coated polycarbonate	1 1/4"
S-242	K	Silicate/Urethane-coated polycarbonate	1"
S-256	K	Silicate/Urethane-coated polycarbonate	1"
S-275	K	Silicate/Urethane-coated polycarbonate	7/8"
S-287	K	Silicate/Urethane-coated polycarbonate	9/16"
S-296	K	Silicate/Urethane-coated polycarbonate	7/16"
S-257	K	Silicate/Urethane-coated polycarbonate	1/4"

Part B

Spec. No.		Sample	Distance
S-309	K#6	Silicate/Urethane-coated polycarbonate	1"
S-312	K#6	Silicate/Urethane-coated polycarbonate	1 1/8"
S-314	K#6	Silicate/Urethane-coated polycarbonate	2 1/4"
S-310	K#6	Silicate/Urethane-coated polycarbonate	3/4"
S-311	K#6	Silicate/Urethane-coated polycarbonate	< 1/2"
S-313	K#6	Silicate/Urethane-coated polycarbonate	< 1/2"
S-304	K	Silicate/Urethane-coated polycarbonate	7/8"
S-305	K	Silicate/Urethane-coated polycarbonate	2"
S-307	K	Silicate/Urethane-coated polycarbonate	2"
S-394	K#6	Silicate/Urethane-coated polycarbonate	1 1/2"
S-395	K	Silicate/Urethane-coated polycarbonate	2 1/2"
S-397	K#6	Silicate/Urethane-coated polycarbonate	1 3/4"
S-398	K	Silicate/Urethane-coated polycarbonate	2"
S-399	K	Silicate/Urethane-coated polycarbonate	1 3/4"
S-400	K	Silicate/Urethane-coated polycarbonate	2"
S-401	K#6	Silicate/Urethane-coated polycarbonate	3"
S-402	K#6	Silicate/Urethane-coated polycarbonate	3"
S-405	K#6	Silicate/Urethane-coated polycarbonate	1 1/2"
S-406	K#6	Silicate/Urethane-coated polycarbonate	2"
S-411	K	Silicate/Urethane-coated polycarbonate	3"

Part C

OPTICAL UNIFORMITY VALUES FROM FEDERAL TEST METHOD 3041

Spec. No.	Sample	Distance
	Lexan circular plate	1/8"
	Lexan plate (commercial)	1/16"
S-284	Silicate-coated polycarbonate	1/16"
S-275	Silicate-coated polycarbonate	N.O.*
S-296	Silicate-coated polycarbonate	1/4"
S-235	Silicate-coated polycarbonate	3/4"

* Not observable

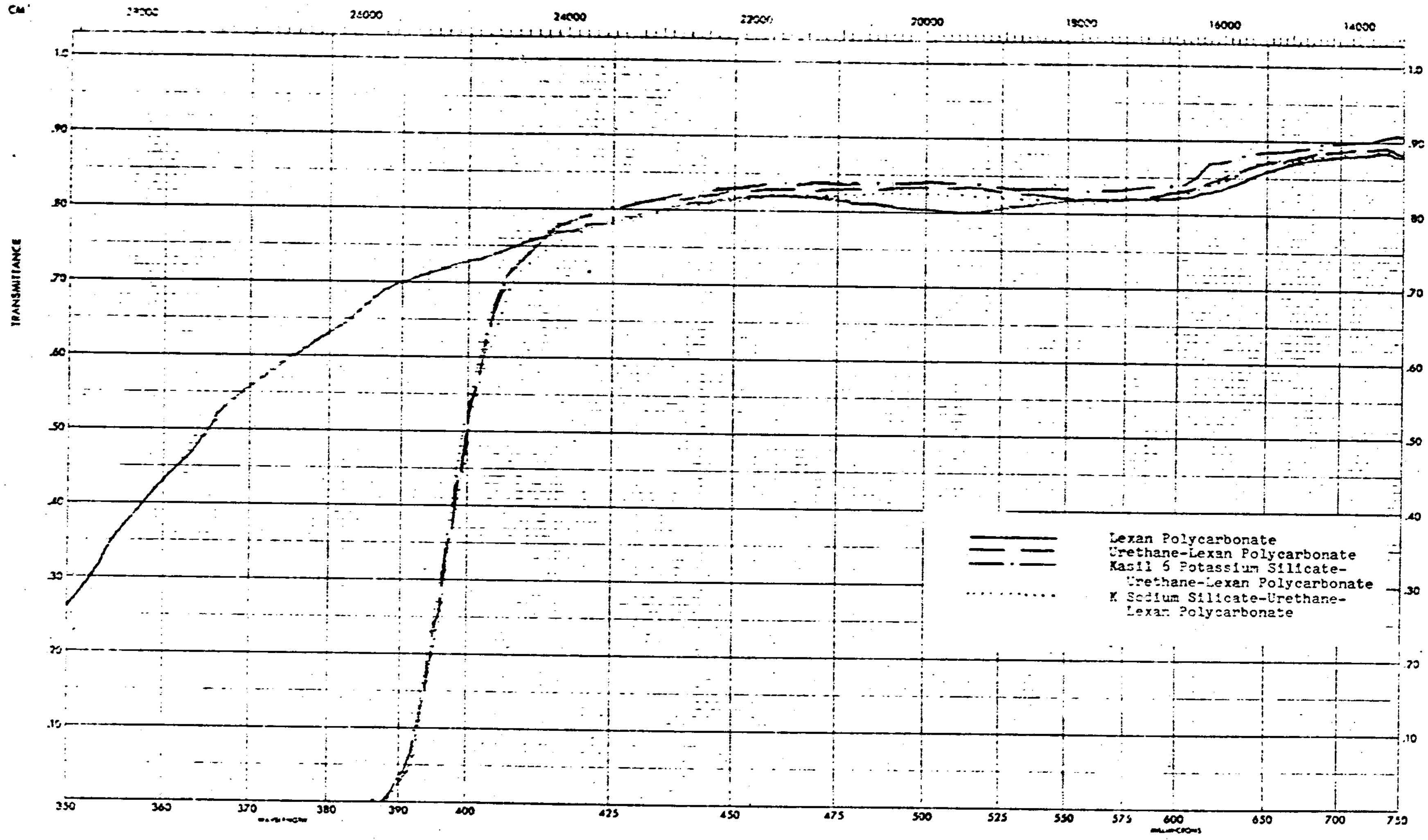


Figure 6. Light Transmittance of Silicate - Urethane - Polycarbonate Systems

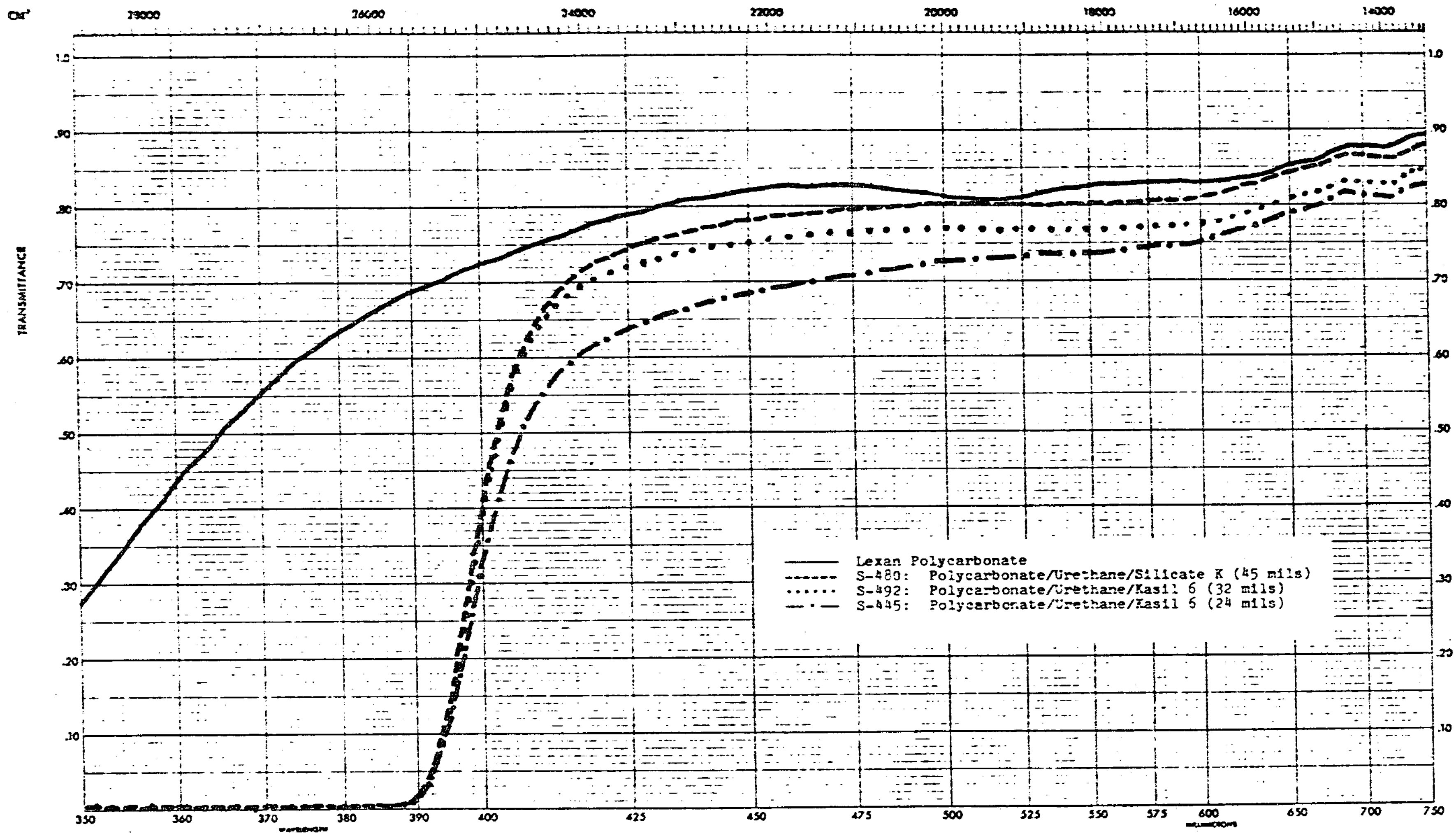


Figure 7. Transmittance of Alkali Silicate Systems

values, in the visible wavelength region, from approximately 50% to 88%. Optically, these coatings are quite satisfactory, but the protective coating tends to slightly reduce the transmitted light in the visible region. This conclusion is evident from a comparison of the curves for the silicate samples in Figures 6 and 7. This effect can be reduced by using the thinnest possible functional protective coating. Protective coatings of 1-3 mils have proved to be quite acceptable.

f. Vacuum Stability. In order to observe the effect of a high vacuum environment, selected alkali silicate film were exposed to a vacuum in the 10^{-5} torr range at room temperature for various periods. The details of these tests are included in Table XII. Both the acrylic enamel-coated and unprotected silicate coatings showed a tendency to craze after three hours at room temperature. Small cracks developed in the polyurethane-coated silicate specimen but the transparency of the sample was not appreciably affected. Polystyrene-coated silicate specimens showed little vacuum degradation even after 24 hours. Two samples (S-507 and S-510 in Table XII) remained in a vacuum of 6×10^{-5} torr for 24 hours. Initially these coatings showed no deviation, but, after a short time, the edges of the coating began to develop small cracks. Later, these cracks enlarged somewhat but did not traverse the sample, and hence did not constitute a failure of the coating. Some bubbles developed in the coating during the test, but the mechanism of bubble formation is not known for certain. In any case, these test results indicate that alkali silicate coatings will stand a hard vacuum for some time without complete failure.

g. Flexural Strength. The flexural properties of alkali silicate coatings were measured, according to ASTM Method D790, with a four point loading system. The flexural measurements were conducted on an Instron testing machine of 10,000 pounds capacity at a rate of 0.05 inches per minute. Results of these tests are contained in Table XIII. The tests were conducted in an environment of 73°F and 50% relative humidity.

The four-point loading was used to prevent localized compressive failure of the coating so that a valid measurement of the flexural strength and modulus of the specimen could be made. The shear strength between the coating and the polycarbonate was therefore determined by "flexural" tests in which the beam length is short compared to the thickness of the beam, and the coating is the outer fiber of the flexural beam. The shear strength of the bond between the coating and the polycarbonate can be determined by such a test, providing the shear strength is less than the tensile strength of the coating itself.

Table XII

VACUUM STABILITY OF ALKALI SILICATE SYSTEMS

Spec. No.	Type	Silicate	Protective Coating Type	Vacuum		Appearance
				Pressure, Torr	Time, hr.	
S414, 418	Kasil 6	Potassium	None	2×10^{-5}	0	Transparent film.
S414, 418	Kasil 6	Potassium	None	2×10^{-5}	3	Several crazed areas.
S414, 418	Kasil 6	Potassium	None	2×10^{-5}	12	Coatings entirely crazed and spalled; coatings removed.
S416	Kasil 6	Potassium	None	2×10^{-5}	0	Transparent film.
S416	Kasil 6	Potassium	None	2×10^{-5}	1/6	No change.
S416	Kasil 6	Potassium	None	2×10^{-5}	3	Crazed and translucent areas; coating removed.
S412, 415	Kasil 6	Potassium	Waterspar Acrylic enamel	2×10^{-5}	0	Transparent films.
S412, 415	Kasil 6	Potassium	Waterspar Acrylic enamel	2×10^{-5}	1/6	No change.
S412, 415	Kasil 6	Potassium	Waterspar Acrylic enamel	2×10^{-5}	3	Crazed and translucent areas; coatings removed.
S444	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	0	Transparent film.
S444	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	1/4	No change.
S444	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	3	Small crack near center; transparency unaffected; coating removed.
S410	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	0	Transparent film.
S410	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	4	A few small cracks; transparency still good.
S410	Kasil 6	Potassium	Dupont Adiprene L-100 Polyurethane	2×10^{-5}	20	Many fine cracks; good transparency.
S497-498	K	Sodium	Dupont Duco Polystyrene	2×10^{-5}	0	Transparent film; a few bubbles in the styrene protective coating.
S497-498	K	Sodium	Dupont Duco Polystyrene	2×10^{-5}	4	No change.
S497-498	K	Sodium	Dupont Duco Polystyrene	2×10^{-5}	24	A few bubbles appear entanged; good transparency.
S507	K	Sodium	Dupont Duco Polystyrene	6×10^{-5}	24	Some edge cracks; a few bubbles.
S510	K	Sodium	Krylon 1302 Acrylic	6×10^{-5}	24	Some edge cracks; a few bubbles.

Table XIII

FOUR POINT LOAD FLEXURAL STRENGTH OF ALKALI SILICATE COATINGS

Spl. No.	Thickness, mils	Width, mils	Length		5% Yield Strength, psi	Coating Deflection at		Modulus of Elasticity, psi x 10^6
			Major Span	Minor Span		Crack Initiation	Total Fracture	
S585	18	0.500	2.34	.78	7360	.03	.09	0.45
S586	24	0.512	2.34	.78	6800	.04	.13	0.34
S588	18	0.500	2.34	.78	7190	.04	.06	0.41
S589	16	0.500	2.34	.78	7350	.04	.07	0.38
S591	25	0.507	2.34	.78	6910	.04	–	0.29
S594	16	0.515	2.34	.78	6870	.04	0.1	0.35

The data obtained in the test described above were used to calculate the modulus of elasticity, according to the equation below:

$$E_b = \frac{L^3 m}{4bd^3} [3(a/L)-4(a/L)^3]$$

E_b = Modulus of elasticity

m = Slope of tangent to initial straight line portion of the load deflection curve

L = Support span

a = Distance from support to adhacent load point

The modulus of elasticity data in Table XIII, for the alkali silicate coatings, are comparable with the modulus values for uncoated polycarbonate. Because the modulus of elasticity values for the coating and the polycarbonate are similar, a bond failure would not be expected to occur under low stress conditions. Under high stress conditions, however, the silicate coating cracks.

h. Stability of Coatings After Ultraviolet Exposure. An Atlas weather-ometer was used to test the stability of alkali silicate coatings when exposed to an intense ultraviolet light source. The test involves mounting the silicate-coated polycarbonate samples vertically and rotating the sample holder in a circular path (30" diameter) around twin, glass-enclosed (protective glass dome) carbon arcs. The test chamber temperature rose to 155°F during the 22 hour exposure of these samples to the intense ultraviolet source. Table XIV indicates the spectral distribution of the Atlas enclosed violet carbon arc lamp (Ref. 4). The effects of this test on various PUSP systems are detailed in Table XV.

Table XIV

SPECTRAL DISTRIBUTION OF ATLAS ENCLOSED VIOLET CARBON ARC LAMP (Ref. 4)

Spectral Range Nanometers (10^{-9} meters)	Noon Summer Sunlight (Microwatts per Square Centimeter)	Twin Enclosed Arc (Microwatts per Square Centimeter)
Below 340 nm	1,040	112
340 - 400 nm	5,250	10,000
400 - 750 nm	59,800	20,900
750 - above nm	75,700	28,500
TOTAL	141,800	59,512

Table XV

ULTRAVIOLET STABILITY OF ALKALI SILICATE COATINGS

Spec. No.	Silicate Coating Type	Silicate Coating Thickness, mils	Protective Coating Type	Protective Coating Thickness, mils	Test Temperature, °F	Test Time, hr.	Appearance After
S493	Kasil 6	23	Dupont Duco Polystyrene	1	155	22	Transparent; light green cast.
S494	Kasil 6	32	Dupont Duco Polystyrene	1	155	22	Transparent; dark green cast.
S499	K	46	Dupont Duco Polystyrene	1	155	22	Transparent; green cast.
S500	Kasil 6	17	Krylon	2	155	22	Transparent; green cast.
S505	Kasil 6	17	Clear Enamel	1	155	22	Transparent; light green cast.
S512	K	64	Dupont Duco Polystyrene	5	155	22	Transparent; light green cast.
S515	K	23	Krylon 1302	4	155	22	Transparent; light green cast.
S523	Kasil 6	37	Krylon 1302	4	155	22	Transparent; light green cast.
S526	Kasil 6	38	Krylon 1302	1	155	22	Transparent; light green cast.
S532	Kasil 6	20	Dupont Duco Polystyrene	2	155	22	Transparent; light green cast.
S534	Kasil 6	15	Dupont Duco Polystyrene	2	155	22	Transparent; light green cast.
S541	Kasil 6	24	Krylon 1302	1	155	22	Essentially unaffected
S546	Kasil 6	30	Krylon 1302	1	155	22	Transparent; light green cast.
S642	K	28	Hughson Chemglaze Polyurethane	1	155	24	Transparent: light green cast.
S649	K	36	Hughson Chemglaze Polyurethane	1	155	24	Transparent; light green cast.

After the test, the samples appear transparent with a slight greenish color. This change in the visual properties of the test samples can best be illustrated by comparing the spectrophotometer graphs recorded both before and after the ultraviolet exposure. These data, recorded on a Perkin-Elmer Model 450 spectrophotometer, are shown in Figures 8 - 13.

The obvious conclusion to be gathered from these graphs is that the light transmission of these PUSP systems is lowered by exposure to the ultraviolet source. The protective coating on the silicate is affected by the ultraviolet source and causes a reduction in the light transmission of the samples. This was determined by peeling off the protective coating and noting the greenish discoloration of the film after exposure. The data indicate that the ultraviolet exposure has the greatest effect on polyurethane-coated samples and the least effect on polystyrene-coated samples.

i. Effect of Water Content on Silicate Properties. The percent water content of silicate coatings, after various stages of drying was determined. Assuming that only a negligible amount of water remains in a silicate coating after oven drying at 225°F, the silicate water content (immediately after application) was found to be 50.5%. The percent water content after oven drying at 100°, 125°, 150°, 175°, 200° and 225°F is shown in Table XVI. The hardness of the coatings varied from <6B pencil hardness (immediately after application) to a hardness (after 1 hour of 225°F) approaching that of SiO_2. It should be possible to predict the coating hardness if the percent of water is known. Also an optimum water content can be found which will yield coatings that are more resistant to cracking and wrinkling.

j. Impact Strength of the PUSP System. Impact tests were conducted using a Rhiehle pendulum impact tester. Both K sodium silicate and K psi 16 potassium silicate were selected as nonflammable protective layers. All specimens were made using a polyurethane bond coat over the polycarbonate, a silicate layer, and a polyurethane water repellent protective coating. The coatings were tested by impacting the specimens (having a 1 inch radius surface) at energies ranging from 15 to 45 ft-lbs. In addition, nine uncoated polycarbonate coatings were subjected to parallel tests. Initially a 45 ft-lb energy was employed. The results were determined on the basis of visual appearance and indentation diameter. The 1.5" x 1.5" coatings were mounted in a special fixture which allowed support over the entire area of the specimen. Normally on this device, the impact specimens are mounted so that they are supported only along the edges. However, the primary objective in this test was to determine the difference in impact strength between polycarbonate-urethane-silicate-protective (PUSP) coating systems and the original uncoated polycarbonate. A summary of test results is given in Table XVII.

In contrast, both of the PUSP specimens (K and Kasil 6 silicate) cracked through the polycarbonate at 45 and 30 ft-lbs. At less than 30 ft-lbs, only the silicate was shattered. The indentation diameter was less than that observed with uncoated polycarbonate at similar impact energies. The results appear to indicate a change in polycarbonate properties that is probably due to the 200°F cure cycle of the polyurethane bond coating applied to the polycarbonate. This can be rectified by using a lower temperature cure cycle (150°F) for a longer period of time.

The test results can also be explained by realizing the effect of the brittle silicate coating. Normally, the polycarbonate would absorb the impact energy by expanding via plastic deformation. However, the brittle silicate layer will absorb little energy and will tend to constrain the polycarbonate and not allow deformation resulting in the cracking of the polycarbonate.

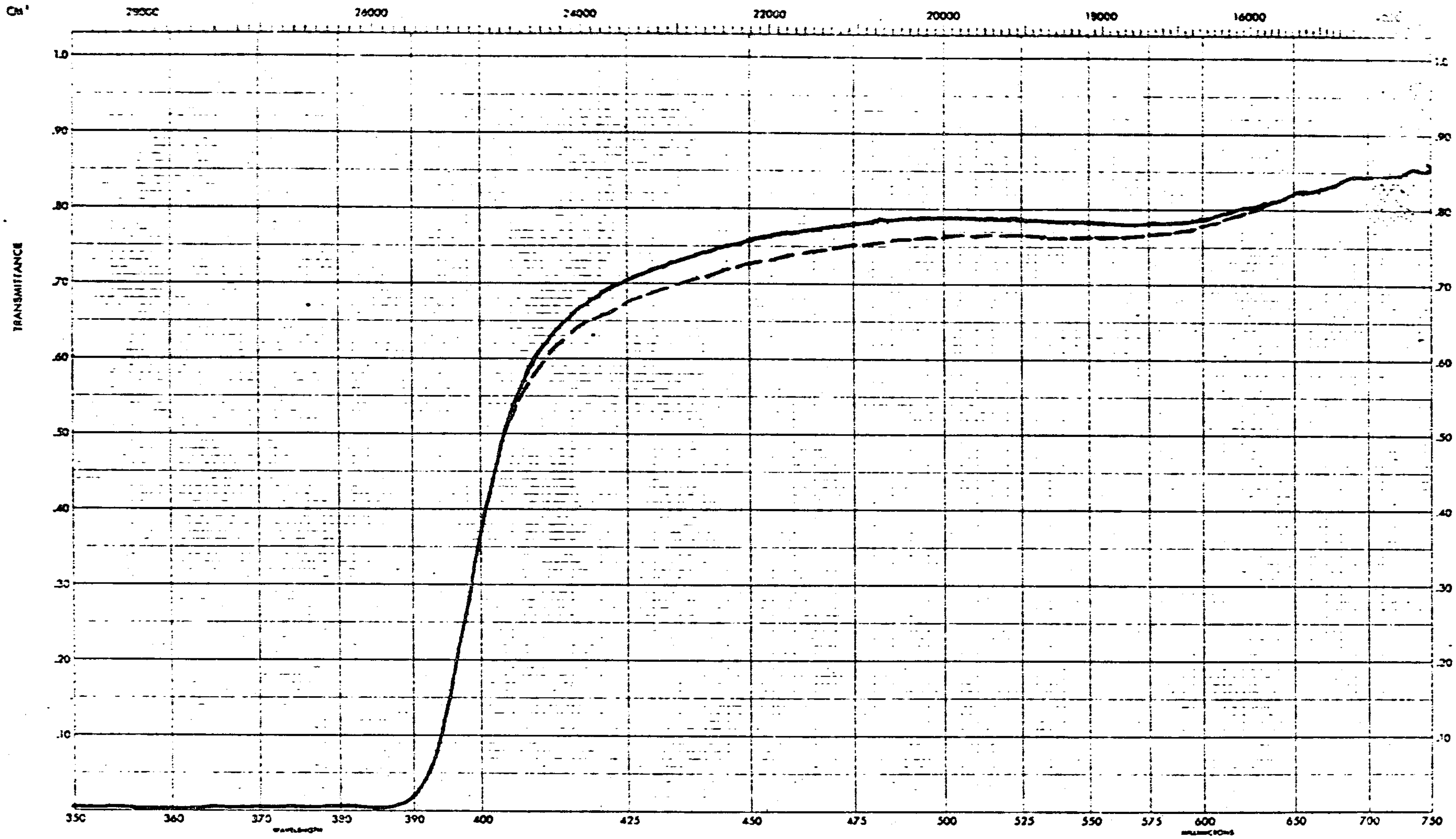


Figure 8. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 532.

——— Light Transmission Before Exposure

------ Light Transmission After Exposure

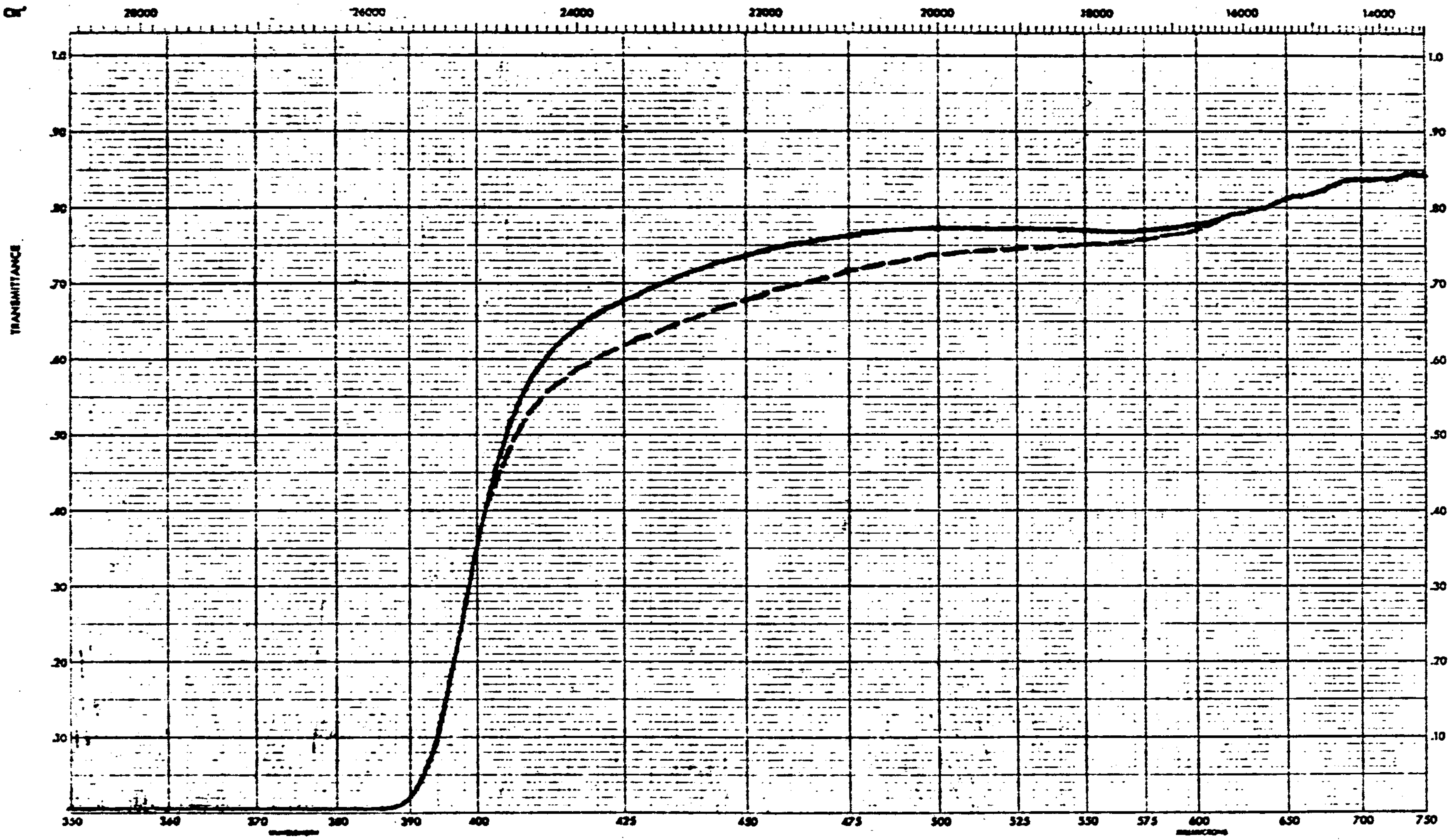


Figure 9. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 534.

______ Light Transmission Before Exposure

------ Light Transmission After Exposure

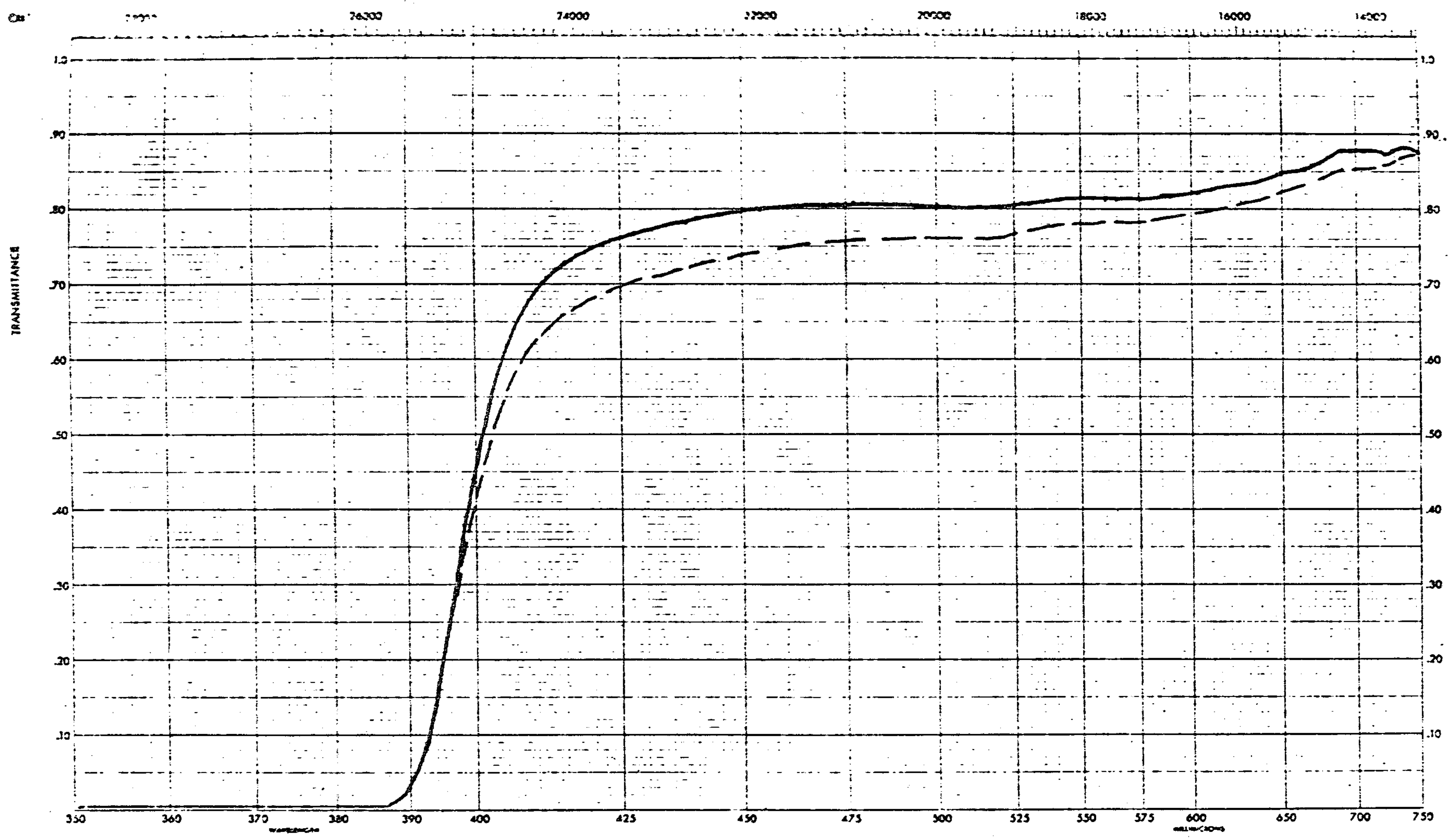


Figure 10. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 541.

──── Light Transmission Before Exposure

------ Light Transmission After Exposure

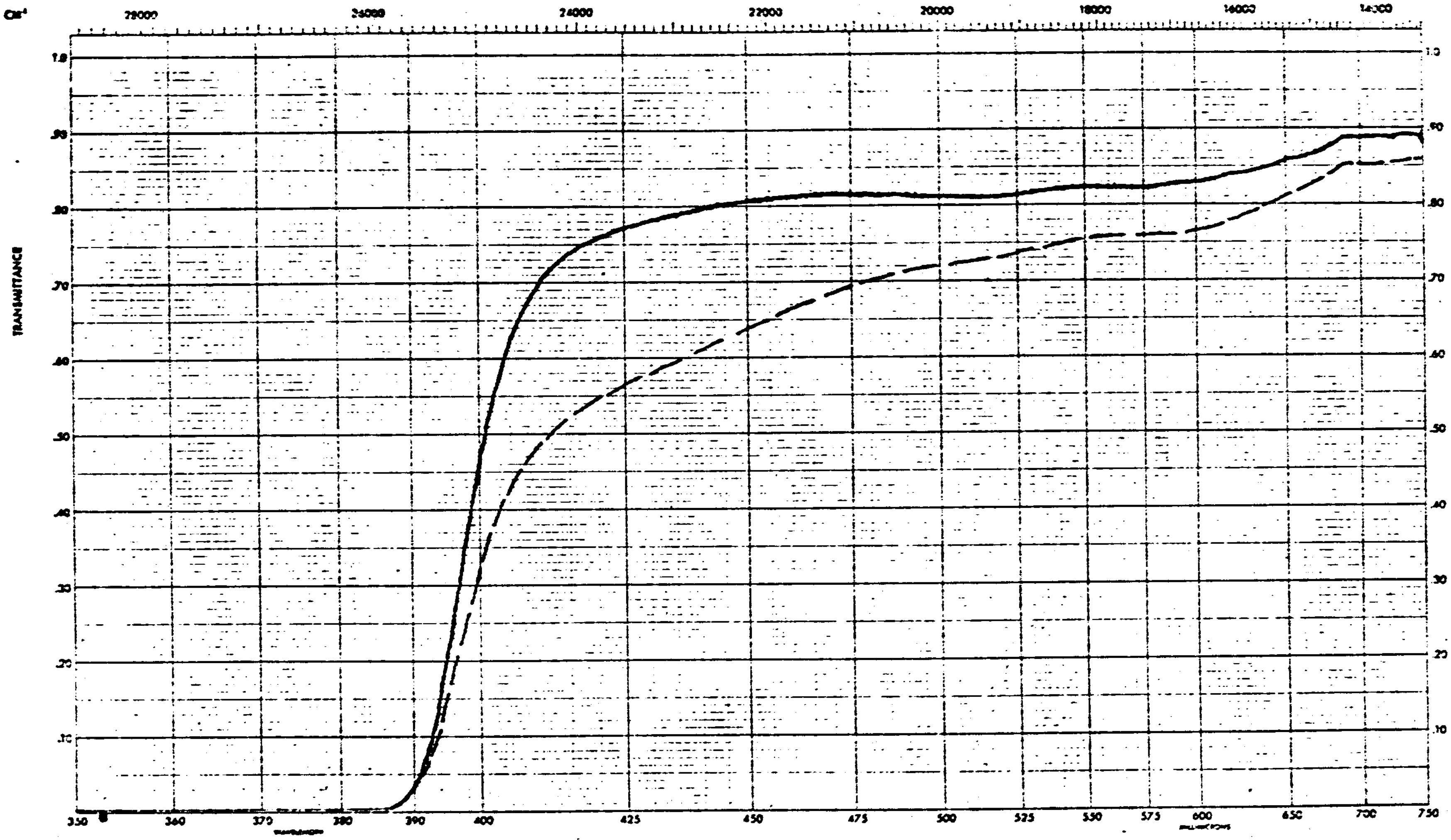


Figure 11. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 546.

———— Light Transmission Before Exposure

------ Light Transmission After Exposure

2 of 2

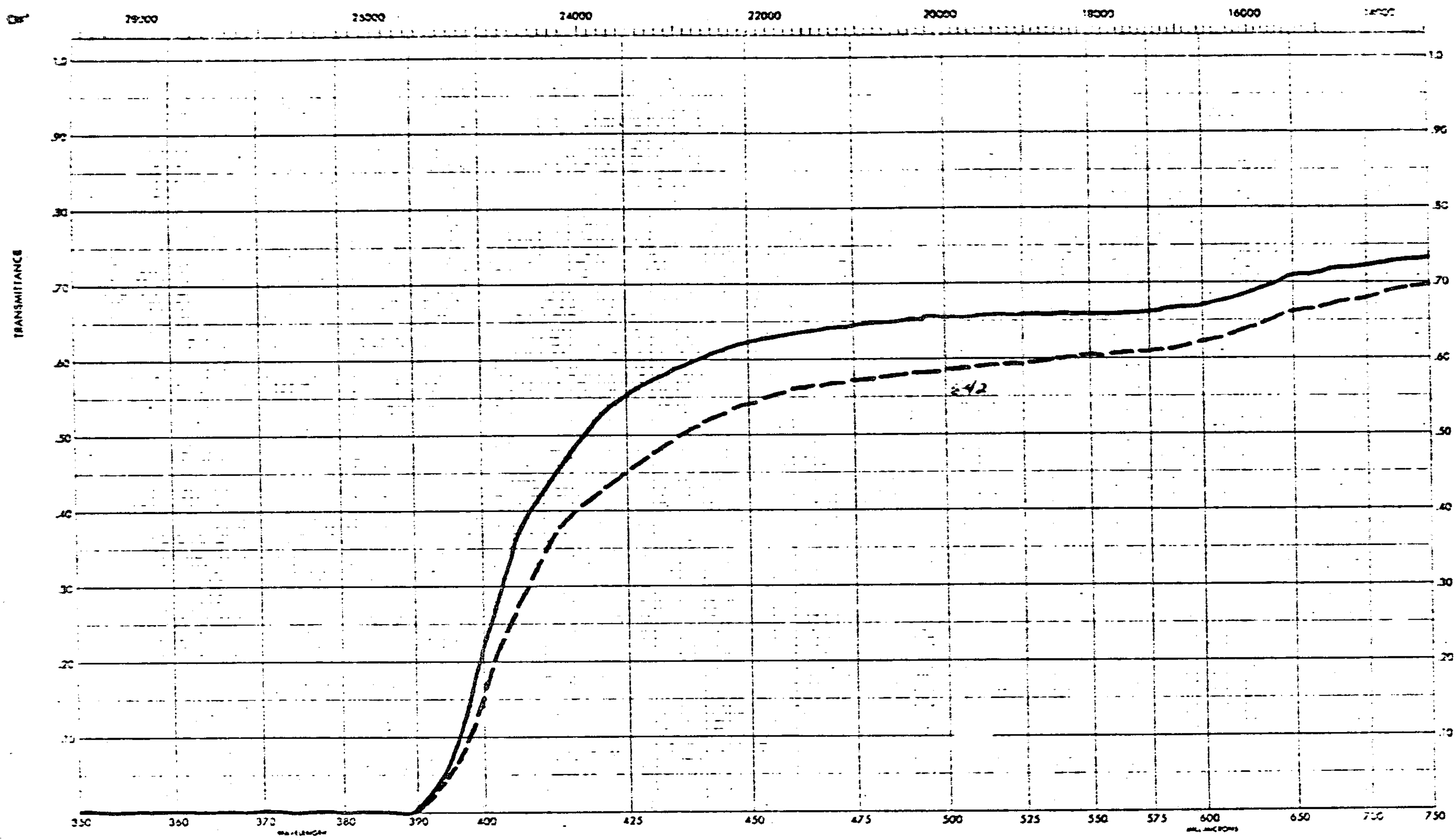


Figure 12. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 642.

——— Light Transmission Before Exposure

------ Light Transmission After Exposure

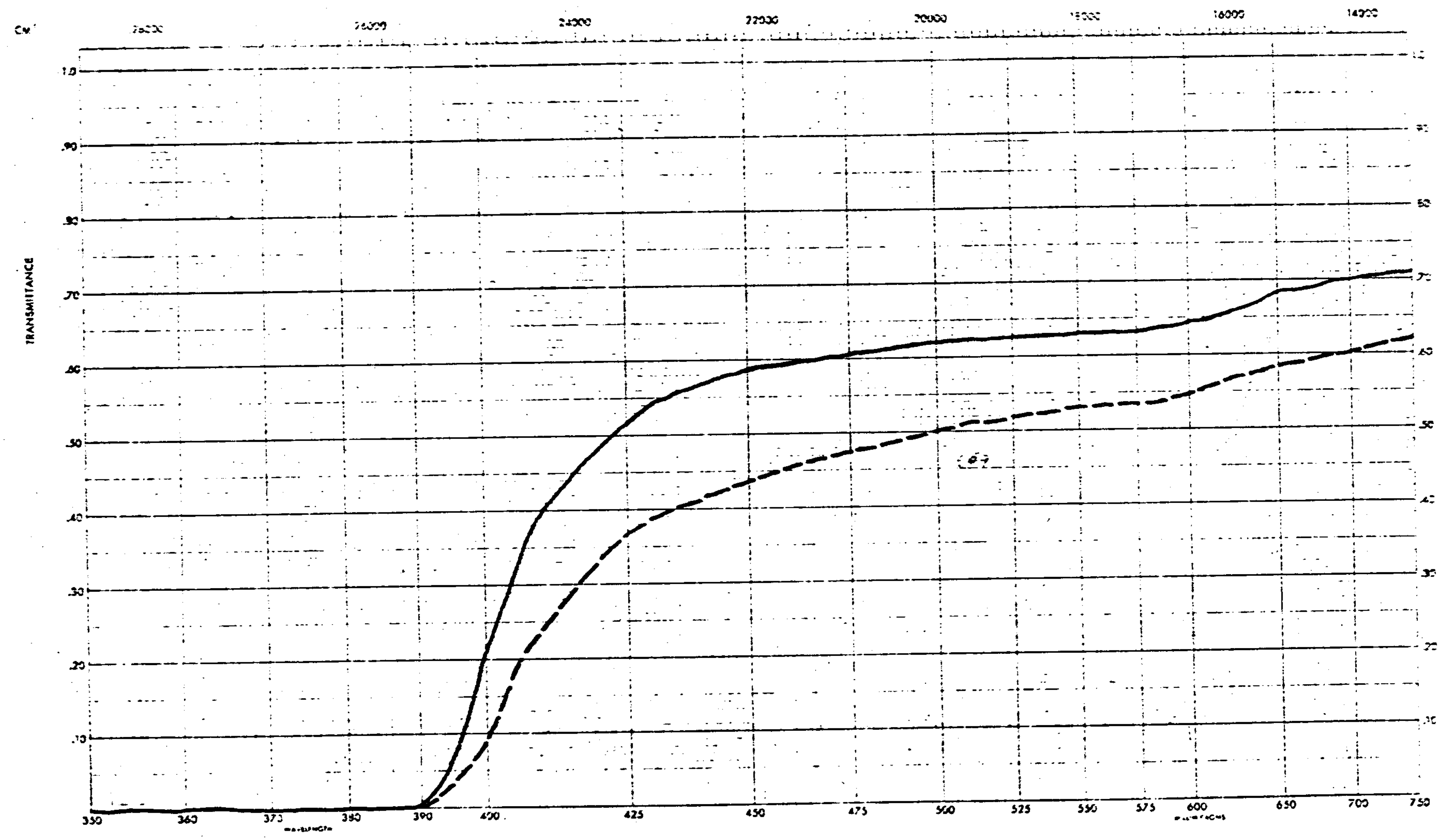


Figure 13. The Effect of Exposure to an Ultraviolet Source on the Light Transmission of Sample 649.

——— Light Transmission Before Exposure

------ Light Transmission After Exposure

Table XVI

EFFECT OF WATER CONTENT ON APPEARANCE OF SILICATE COATINGS

Spec. No.	Time, hr.	Temp., °C	Weight, g	% Water Content	Appearance
S-539	0	R.T.			Transparent film <6B hardness
S-540	0	R.T.	8.6905	50.5	Transparent film <6B hardness
S-539	1.5	100	-	-	No change
S-540	1.5	100	5.817	26.2	No change
S-539	1	125	-	-	A few bubbles
S-540	1	125	5.1324	16.3	A few bubbles
S-539	1	150	-	-	8H pencil hardness; more bubbles
S-540	1	150	4.8447	11.3	8H pencil hardness; more bubbles
S-539	2	175	-	-	Larger bubbles >9H pencil hardness
S-540	2	175	4.5240	5.02	Larger bubbles; >9H pencil hardness
S-539	1	200	-	-	Larger bubbles; wrinkling
S-540	1	200	4.3887	2.05	Larger bubbles; wrinkling
S-539	1	225	-	-	No change
S-540	1	225	3.2966	~0	No change

Table XVII

IMPACT TEST RESULTS

Sample No.	Silicate Type	Silicate Thickness, mils	Overall Specimen Thickness, mils	Impact Energy, ft-lbs.	Diameter of Indentation	Appearance
641	K	20	148	30 ft-lbs.	.5 in.	Cracked the glass, shattered the polycarbonate.
643	K	16	147	45 ft-lbs.	.6 in.	Cracked the glass, split PC.
644	K	14	145	20 ft-lbs.	.4 in.	PC slightly dented, coating had two cracks.
645	K	15	140	15 ft-lbs.	-	No visable damage due to striking edge.
646	K	17	147	15 ft-lbs.	.3 in.	Slight dent.
647	K	17	145	20 ft-lbs.	.375 in.	One crack in coating.
648	K	14	147	20 ft-lbs.	.4 in.	One crack in coating.
650	Kasil 6	17	147	45 ft-lbs.	-	Shattered, coating and PC.
651	Kasil 6	15	147	20 ft-lbs.	.35 in.	PC split, coating had few cracks.
652	Kasil 6	20	153	15 ft-lbs.	.3 in.	Two cracks in coating.
653	Kasil 6	22	154	30 ft-lbs.	.5 in.	Cracked and lifted some pieces from surface.
654	Kasil 6	19	155	15 ft-lbs.	-	Cracking in coating (some burst).
655	Kasil 6	21	151	20 ft-lbs.	.2 in.	
660	Kasil 6	20	150	20 ft-lbs.	.35 in.	Four cracks in coating.
662	Kasil 6	17	151	20 ft-lbs.	.4 in.	Two cracks in coating.
Uncoated PC	None	-	-	15 ft-lbs.	.4 in.	
Uncoated PC	None	-	-	15 ft-lbs.	.4 in.	
Uncoated PC	None	-	-	20 ft-lbs.	.45 in.	
Uncoated PC	None	-	-	20 ft-lbs.	.45 in.	All specimens dented. Increased in depth of dent with increase of force.
Uncoated PC	None	-	-	30 ft-lbs.	.5 in.	
Uncoated PC	None	-	-	30 ft-lbs.	.5 in. (small split)	
Uncoated PC	None	-	-	45 ft-lbs.	.65 in.	
Uncoated PC	None	-	-	45 ft-lbs.	.65 in.	
Uncoated PC	None	-	-	45 ft-lbs.	.65 in.	

B. Non-Alkali Silicates and Silica Coatings

1. Consideration of Approaches. Another approach to the fabrication of a water-insoluble, nonflammable, transparent coating on polycarbonate involved the use of non-alkali silicates and silica coatings. While insolubilization of the silicates is possible with the solution of salts containing di- and trivalent cations, the products found tended to be nontransparent. Silica coatings are both transparent and water insoluble.

The possible approaches to silica coatings that were considered are summarized below:

- Glass resins: These commercially-available resins are modified organic silicas.
- Hydrolyzed Ethyl Silicate: Slightly polymeric ethyl silicates such as ethyl silicate 40 can be hydrolyzed to a clear silica film.
- Acidified Potassium Silicate: Potassium silicate can be acidified and converted to polysilica acid that can plug to a clear silica casting. A weak acid can also be used to cause *in situ* conversion of silicate to silica coating as demonstrated by work at Goddard Space Flight Center.
- Silica Gel: Incorporation of a small amount of organic thickening agent in a commercially available silica gel allows film formation without precipitation of the silica as a powder.

The principle problems anticipated were differences in the expansion coefficient between polycarbonate and silica, shrinkage cracks in the inorganic layer found during water removal, and the difficulty of removing entrapped water from within a relatively thick silica film. The use of elastomeric urethane and multiple applications of silica was expected to alleviate this problem.

2. Modified Silica Coatings for Flame Retardation. Modified organic silica coatings appeared from screening tests to be significantly less effective than alkali-silicate coatings in preventing flame damage to a polycarbonate sheet (Table XVIII). In the screening test, a propane torch flame was impinged horizontally on the broad side of a specimen supported by a clamp on one side. The flame temperature was approximately 2000°F at the point of impingement.

These limited data indicated that the barrier coating on polycarbonate should preferably serve as an oxygen barrier (to prevent oxidation of the polycarbonate) and as a thermal insulator (to

Table XVIII

EFFECT OF A PROPANE TORCH FLAME ON UNCOATED AND COATED POLYCARBONATE SHEET[a]

Code	Coating	Polycarbonate Pretreatment	Coating Thickness, mil	Specimen Size, inches	Coating Failure		Initial Polycarbonate Softening Time, sec	Polycarbonate Melting Time, sec
					Time, sec	Mode		
62309-3	None	None	--	1 x 3	--	---	---	32
62330-4	None	None	--	1 x 3	--	---	<5	25
62330-6	None	None	--	1 x 3	--	---	<5	35
62298	None	None	--	2 x 2	--	---	5	40
62304-1	Glass	None[b]	35	1 x 3	1	Shattered	---	35
62304-2	Glass	None[b]	35	1 x 3	1	Cracked	---	60
62314-3	Glass	U[c]	35	1 x 3	8	Cracked[k]	55	70
62303	Glass Resin 650[d]	Flamed[e]	8	1 x 3	20	Cracked	---	35
62330-5	Glass Resin 650[d]	Flamed[e]	8	1 x 3	2	Peeled	<5	35
62309-4	Glass Resin 650[d]	Flamed[e]	9	1 x 3	--	---	40	42
62309-2	Glass Resin 650[d]	Flamed[e]	10	>1 x 3	3	Peeled	30	60[f]
62309-6	Glass Resin 650[g]	Flamed[e]	10	1 x 3	--	---	30	40
62330-2	Glass Resin 650[g]	U[c]	7	1 x 3	2	Flaked	20	49[h]
62330-3	Glass Resin 650[g]	U[c]	7	1 x 3	4	Flaked	35	42
62314-2	Potassium Silicate[i]	U[c]	20	2 x 2	36	Burned Through	>75	>75
62314-1	Potassium Silicate[j]	U[c]	30	2 x 2	37	Burned Through	>75	>75

---

a. Flame temperature at point of impingment on test specimens was approximately 1900-2000°F

b. Soft glass microscope slide clamped over polycarbonate

c. Polyurethane adhesive layer from Adiprene 1-100 isocyanate-terminated prepolymer and 4,4'-methylene bis(2-chloroaniline) (MOCA), less than 5 mil thick

d. Product of Owens-Illinois

e. Cleaned by passage through propane gas flame

f. Melting and sagging retarded by polymer outside of heated area on this specimen

g. Glass resin on reverse side of polycarbonate away from flame

h. Glass resin remained intact and helped prevent softened polycarbonate from dripping onto bench

i. Philadelphia Quartz, "K", ambient temperature cured (Specimen #230)

j. Philadelphia Quartz, Kasil 6, cured through 16 hours at 198°C (Specimen #186)

k. Urethane melted

retard melting and collapse of the polycarbonate). The alkali-silicate coatings serve both functions. They intumesced to form an inorganic foam that retarded heat transfer sufficiently to allow a coated specimen to withstand burning-through or excessive softening for more than 75 seconds. In contrast, silica or glass coatings served as oxygen barriers but did not greatly retard melting of the polycarbonate.

An uncoated piece of polycarbonate (1" x 3" x 0.131") started to burn within five seconds in propane flame but was self-extinguishing upon removal from the flame. The sample also started to soften and warp away from the flame within a few seconds. The polymer melted and flowed in the hottest area, and the whole piece melted and dropped to the bench in 25-35 seconds. A slightly larger piece (2" x 2") showed similar melting characteristics, but retained sufficient rigidity to avoid catastrophic melting failure slightly longer (40 seconds).

A 36-mil thick piece of soft glass attached to the polycarbonate retarded melting of the polymer for approximately another 30 seconds. Melting did occur within 60-70 seconds under the screening test conditions. The glass cracked in all cases, but if the pieces were held in their original position, no sign of charring of the polycarbonate occurred. When a polyurethane adhesive layer was used between the glass and polycarbonate, it was decomposed vigorously by the intense heat; and the gases evolved burned around the edges of the strip. In a separate experiment, the urethane lost all adhesive characteristics as it became fluid within 15 seconds when heated under ∿60 mil thick silica slide. The silica did not crack and the transmission of heat through the silica was still relatively fast.

The Glass Resin 650 generally showed poor adhesion to the polycarbonate. Under the stresses imposed by rapid heating, the 650 resin generally cracked quickly or peeled away from the substrate. Even when an intermediate polyurethane adhesive layer was used, the 650 resin popped off almost immediately upon contact of the flame with the coating. The adhesion of thick sections (8-10 mils) prior to heating was marginal on flame-cleaned polycarbonate. Over a polyurethane adhesive prime coating, the 650 resin was slightly cracked after room temperature drying, but adhesion was reasonably satisfactory. Although the 650 resin did not burn, heat transfer across a thin coating of 650 resin would be expected to be rapid. This product did not look attractive for further development.

The different initial softening data for the uncoated and 650 resin-coated polycarbonate are relatively insignificant and measure our inability to observe precisely the softening characteristics under the test method more than any significant difference in properties. Some slight shielding of the polycarbonate from direct flame infringement may have slightly delayed the apparent softening of the coated samples.

Although several routes to silica coatings were apparent, it was concluded that the alkali-silicate route was the most promising. The water sensitivity of these coatings may be most satisfactorily minimized by overcoating with a very thin water repellant coating such as an alkyl silane.

C. Plasma-Sprayed Coatings

Through the use of refined arc-plasma spraying techniques, extensive studies have been carried out in an effort to produce a nonflammable transparent coating on polycarbonate. Refined plasma spraying methods are necessary to avoid undesirable degradation of the polycarbonate.

In the arc-plasma generator, an inert or nonoxidizing gas (usually argon, helium, or nitrogen) is passed through a high-energy dc arc producing a very high-temperature, continuous plasma stream. The high-energy stream issues from the front nozzle of the generator, resembling an open welding flame. Powders may be injected into the plasma jet for controlled melting and deposition of a coating on the selected substrate. The construction of a plasma spray torch and the arrangement of the supporting equipment for production of plasma-sprayed coatings is shown in Figure 14. Using this equipment, various studies have been carried out in this laboratory involving a wide range of conventional and special metallic, ceramic, composite, and polymeric materials for numerous different applications. The development of a coating for polycarbonate by the arc-plasma process therefore seemed a logical extension of the technology based on preliminary plasma-sprayed particle studies.

1. Screening of Candidate Materials. The initial screening of materials emphasized the testing and evaluation of rather basic chemical compounds exhibiting a cubic crystal structure and/or desirable melt characteristics. The initial candidate materials included: $Y_2O_3 \cdot ThO_2$ (GE. Yttralox), $MgO \cdot Al_2O_3$ (spinel), MgO (periclase), CaO, CaF_2(fluorite), SiO (quartz), LiF, Al_2O_3 (corundum), B_2O_3 and sodium borosilicate glass. These candidate spray powders were exposed to 100% relative humidity conditions at 36°F for 21.5 hours. With the exception of Al_2O_3, CaO, and sodium borosilicate glass, most of the materials showed reasonably good resistance to this environment as indicated by their practically nil weight gain. However, Al_2O_3, CaO, and sodium borosilicate glass showed significant weight increases of 3.1, 7.4, and 10.2%, respectively. Secondly, the candidate materials were evaluated by routine particle impact studies. In this procedure, the plasma torch is passed rapidly across a series of microscope slides mounted at various distances from the torch nozzle. A rudimentary diagram of this test is shown in Figure 15. Various torch/electrode/nozzle combinations are studied

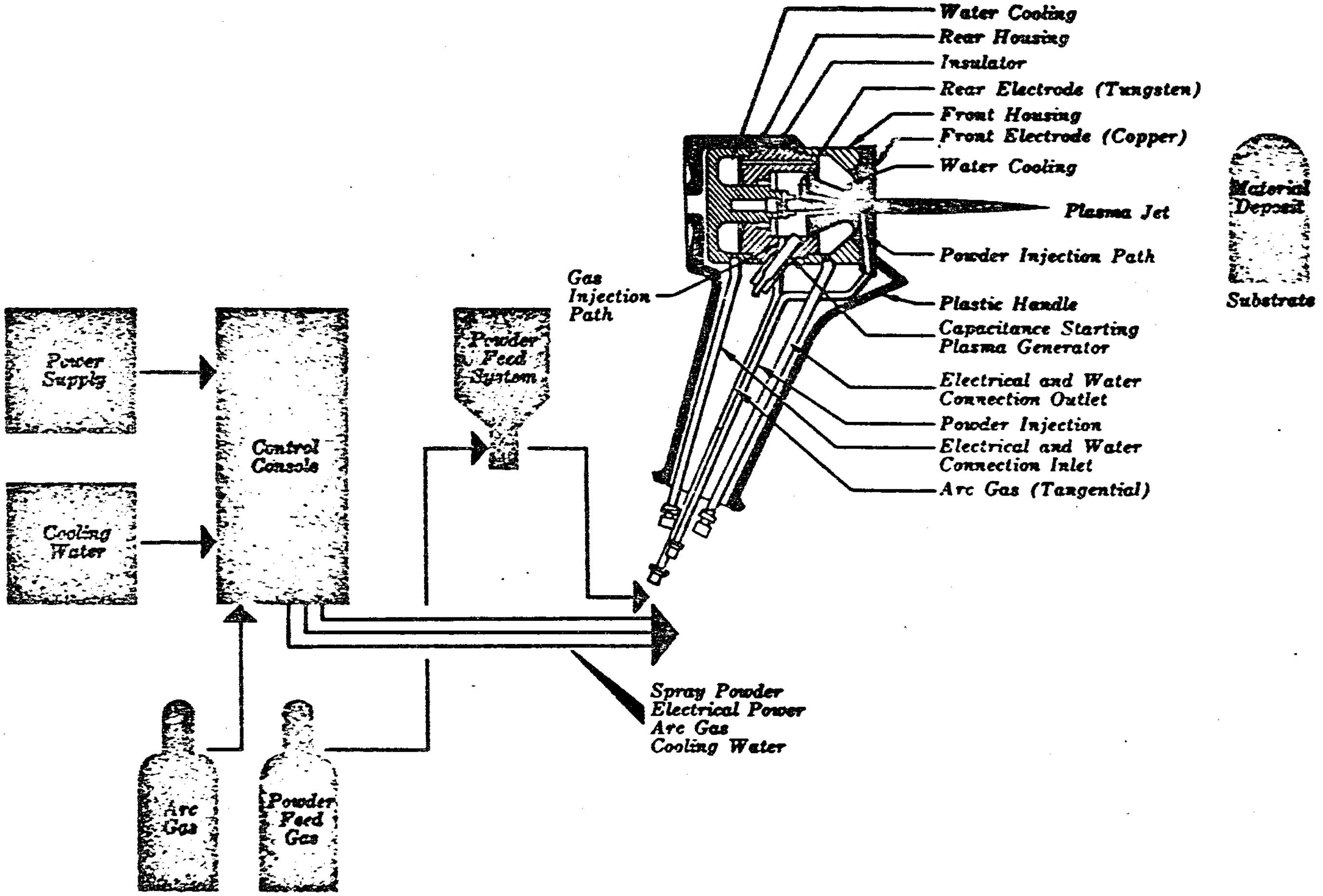


Figure 14. Arrangement of Equipment for Production of Plasma-Sprayed Coatings, showing Details of the Plasma Spray Torch with Standard Argon Electrodes.

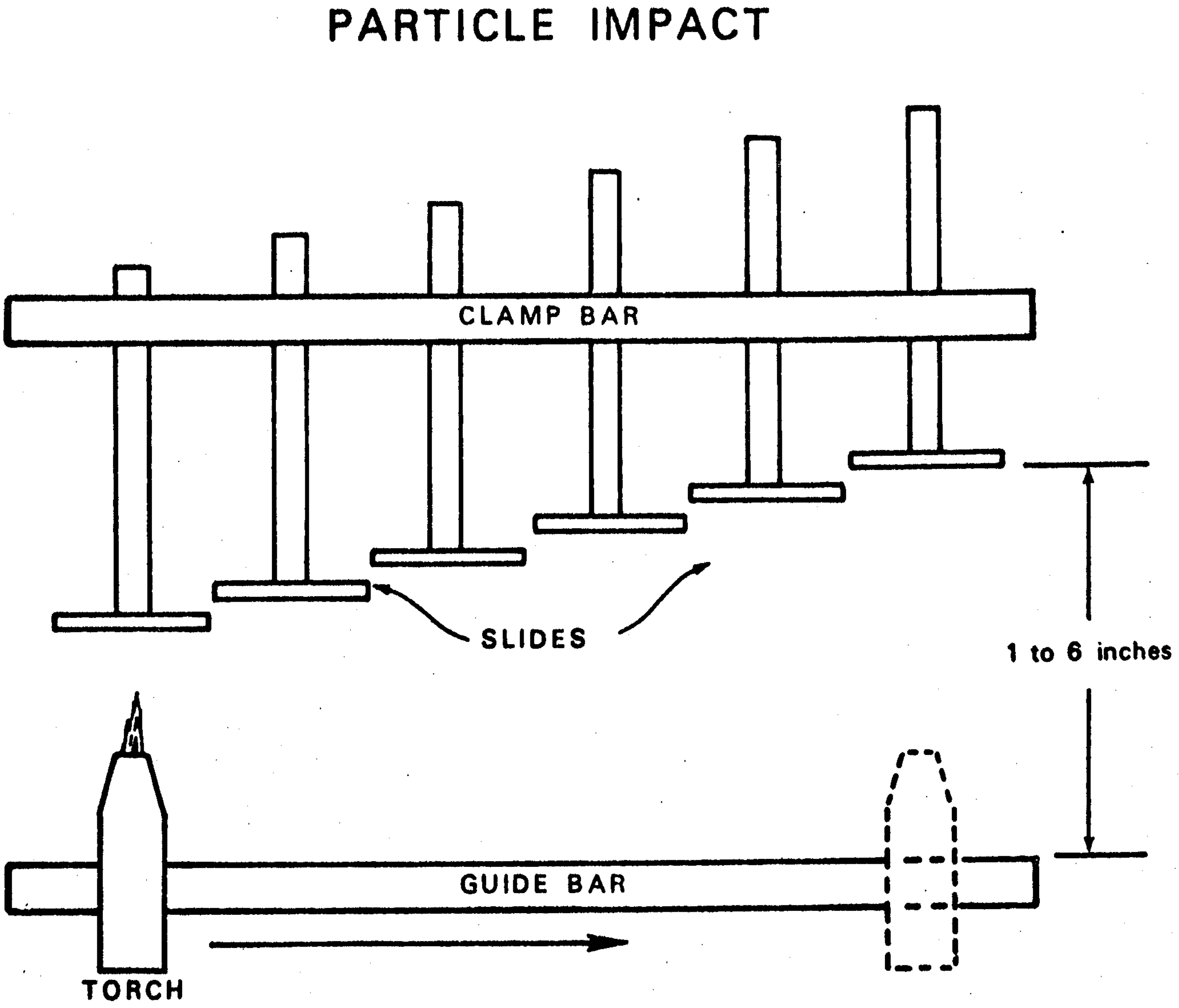
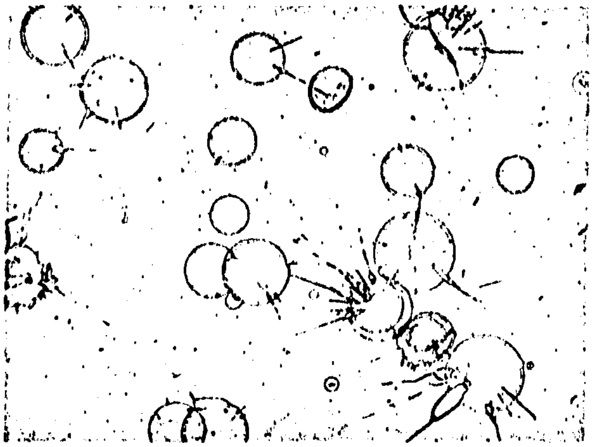

PARTICLE IMPACT

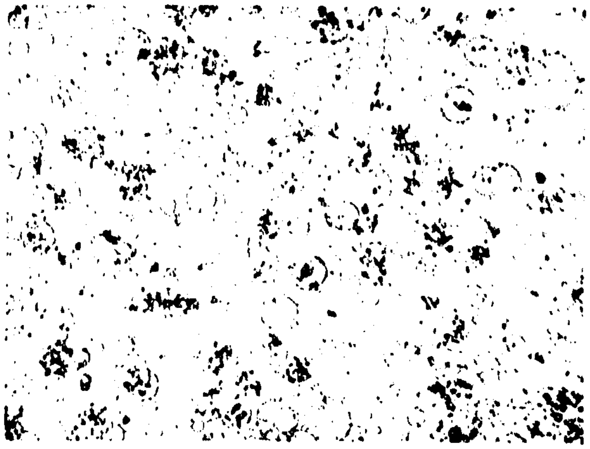
Figure 15. Schematic Arrangement of Equipment for Observing Melting Characteristics of Spray Powders at Various Torch Conditions

with selected arc gas types or mixtures. Microscopic examination of each glass slide indicates the degree of melt and flow for each material at one set of conditions. Evaluation of a whole range of conditions establishes base plasma spraying conditions for each material. The general appearance of these particle impact slides, using different materials, is shown in Figure 16. The results of these particle impact studies are contained in Table XIX.

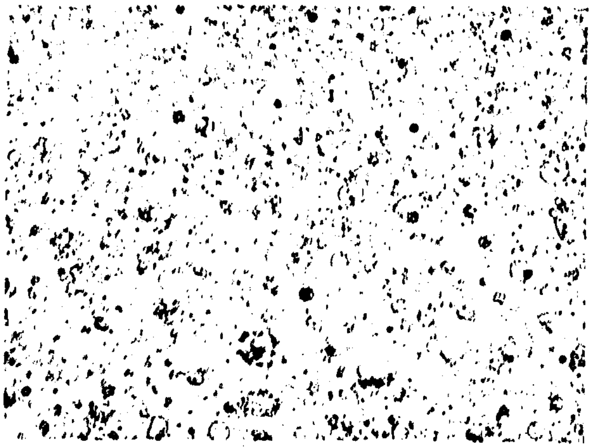
2. Survey of Glass-Forming Systems. After the initial screening of various chemical compounds showed limited promise, the experimental approach was redirected to find several glass compositions that would permit the coating of polycarbonate by arc plasma techniques. In order to achieve this goal, it was necessary to find a glass composition with the appropriate physical characteristics, i.e., transparency, colorless, chemical stability, and low viscosity at the liquidus temperature. It was necessary therefore, to evaluate several glassy systems and their modifications to obtain the required properties. Preliminary information indicated that soda-borate and soda-lime-silica glasses might meet the necessary conditions. Soda-borate glass, however, showed no promise due to its very poor chemical stability and optics. Figure 17 (Ref. 5) shows the phase equilibrium relations in the portion of the system Na_2O-CaO-SiO_2 that is of interest to glass technology. Glasses along the devitrite ($Na_2O \cdot 3CaO \cdot 6SiO_2$)-sodium disilicate ($Na_2O \cdot 2SiO_2$) boundary curve are much too viscous for plasma spraying. Emphasis, therefore, was shifted to compositions in the devitrite field with liquidus temperature between 900°C and 1050°C. Optically, the soda-lime-silica glasses are excellent but they tend to be very viscous causing the retention of bubbles and inhibiting complete mixing. Experimental work was conducted to prepare a soda-lime-silica glass with better flow characteristics. The replacement of Na_2O by CaO results in considerable improvement in chemical durability, reduction of thermal expansion coefficient, and an increase in the melt viscosity. Rawson (Ref. 6) states that higher CaO and lower SiO_2 decreases the melt viscosity. However, too much CaO gives a glass that is difficult to melt and sure to devitrify. Too little lime yields a glass with poor chemical durability but with a low melting point. Also, the addition of small amounts of B_2O_3 to promising compositions in the soda-lime-silica systems was considered (Ref. 7). Such a modification was incorporated in a series of experimental glasses which fall basically in the alkali-B_2O_3-SiO_2 system. Several glasses in the system BaO-Al_2O_3-B_2O_3, and modifications made therefrom, were evaluated. The basic ternary compositions were taken from the work of Hirayama (Ref. 9). Their general location in the ternary diagram is shown in Figure 18. Because of the refractive index and flux properties of cryolite (Na_3AlF_6), a series of samples was prepared based on the cryolite-refractory oxide system with some additives to enhance the melt characteristics. Other more specialized glass compositions were



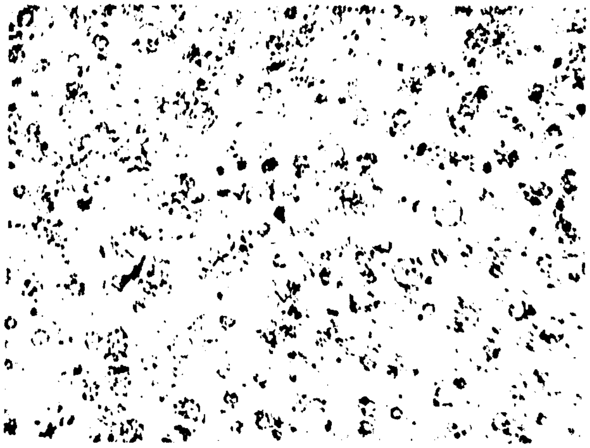
90% Y_2O_3-10% ThO_2 200-X



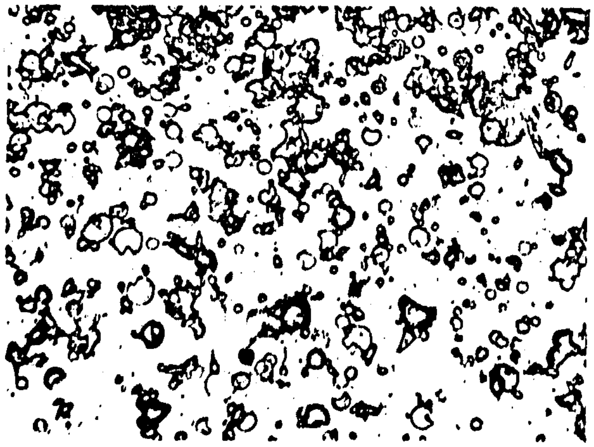
$MgO \cdot Al_2O_3$ (Spinel) 100-X



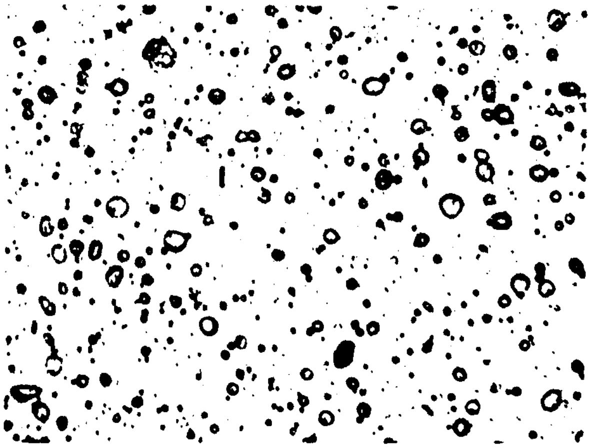
CaO 100-X



MgO 100-X



Sodium Borosilicate (Glass)
100-X



SiO_2 (Quartz) 200-X

Figure 16. Plasma-Sprayed Transparent Particles

Table XIX

PARTICLE IMPACT STUDIES OF INITIAL CANDIDATE MATERIALS

Spec. No.	Sprayed Material	Supplier	Mesh Size	Comments
P1-12	Quartz	General Electric	-400	Transparent particles; limited flow
P-13-24	Quartz	General Electric	-400	Improved flow
P-25-36	Quartz	General Electric	-400	Transparent; partially flowed particles
P37-48	Quartz	General Electric	-400	Transparent; partially flowed particles
P49-60	Quartz	General Electric	-325 +400	Transparent; partially flowed particles
P63-98	Quartz	General Electric	-325 +400	Transparent; partially flowed particles
P99-125	Quartz	General Electric	-400	Transparent; partially flowed particles
P126-152	CaO	Baker & Adamson	as received	Transparent; flowed particles
P153-155	CaO	Baker & Adamson	as received	Transparent; flowed particles
P479-526	CaO	Baker & Adamson	-400	Transparent; flowed particles
P156-182	MgO	Cerac 1281	-325	Transparent; flowed particles
P183-209	MgO	Cerac 1281	-325	Transparent; flowed particles
P212-239	$MgO \cdot Al_2O_3$ (spinel)	Cerac 1118	-325	Transparent; flowed particles
P431-478	$MgO \cdot Al_2O_3$ (spinel)	Cerac 1118	-325	Transparent; flowed particles
P240-243	Sodium Silicate	Fischer Scientific	-100 +250	Transparent; flowed particles
P244-249	Sodium Borosilicate	Fischer Scientific	-325	Transparent; limited flow
P250-252	Sodium Borosilicate 10% CaO	Fischer Scientific	-325	Transparent; limited flow
P527-574	CaF_2	J. T. Baker	-400	Many unmelted particles
P575-622	CaF_2	J. T. Baker	-400	Many unmelted particles
P623-670	B_2O_3	Matheson	-400	Transparent; some coalesced particles
P671-718	B_2O_3	Matheson	-400	Increased coalescence
P258-281	Al_2O_3	Du Pont Colloidal	as received	Transparent; flowed particles
P282-305	Al_2O_3	Du Pont Colloidal	as received	Transparent; flowed particles
P308-355	$Na_2B_4O_7$	Mallinckrodt	unsized	Transparent; flowed particles
P360-383	$Na_2B_4O_7$	Mallinckrodt	unsized	Transparent; flowed particles
P387-430	$Na_2B_4O_7$	Mallinckrodt	unsized	Transparent; flowed particles

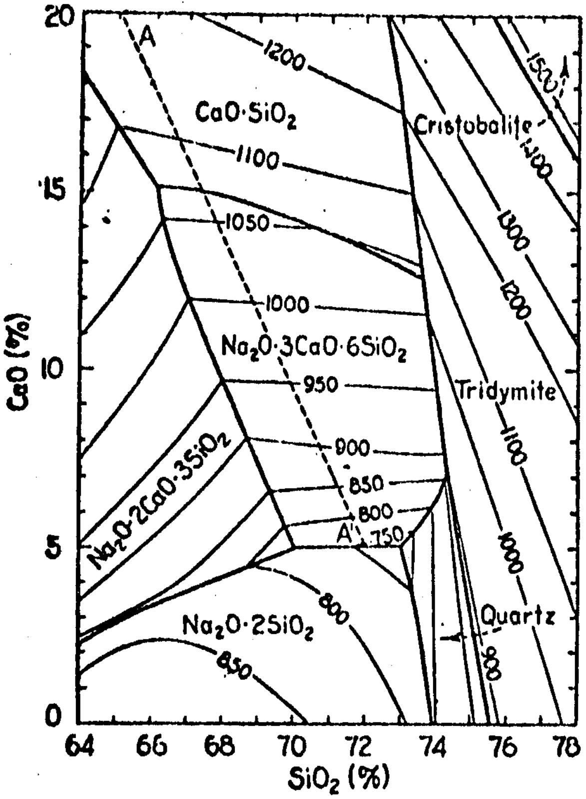


Figure 17. The portion of the ternary system Na_2O-CaO-SiO_2 of interest to glass technology, according to Morey (1930); weight percent of Na_2O obtained by subtracting sum of CaO plus SiO_2 from 100.

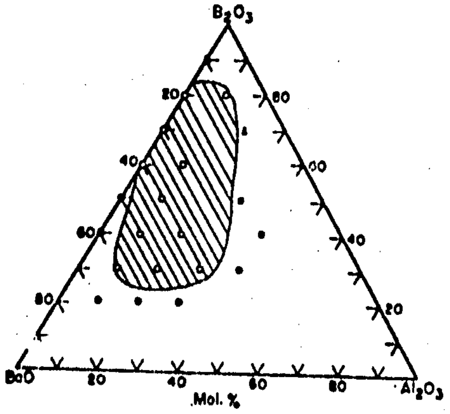


Figure 18. Glass-forming portion of the System BaO-Al_2O_3-B_2O_3.

○ clear glass
● devitrified
▲ immiscible

obtained from published papers (i.e., Ref. 10), abstracts, and patents (i.e., Ref. 11). Beyond the evaluation and modification of experimental glasses, the materials available on the commercial market were scanned. Several glaze and enamel frit compositions with promising properties, were chosen from those available for testing by arc plasma methods.

3. Plasma Spraying of Glass Compositions.

a. Preparation and Appearance of Coatings. Generally, the experimental glasses studied were located in one of the following systems: alkali-B_2O_3-SiO_2, soda-lime-silica, BaO-Al_2O_3-B_2O_3, cryolite-based glass, and several specialty glasses (i.e., Refs. 10 and 11). The detailed compositions of the thirty glasses evaluated during this program are shown in Table XX. Each of the compositions was prepared in small batches (10 grams) for observation of the melt properties and optical qualities. Then the promising compositions were prepared in 400 gram batches for evaluation by particle impact studies. Glasses 8, 16, and 20 were prepared in large batches, and their particle impact data are shown in Table XXI.

In addition to the experimental glasses, five commercial glasses were selected for particle impact studies because of their low fusion points. The results of these particle impact studies are contained in Table XXII. Using the optimum conditions determined in the particle impact studies, coatings (ranging in thickness from 2-10 mils) of glasses 16 and 20, Pemco 1729 and 1730, and Al_2O_3 (conditions determined previously) were applied directly onto 2" x 2" Lexan polycarbonate as follows:

8 samples (2123-2131) of Al_2O_3
16 samples (2132-2139) of Glass 20
8 samples (2140-2143) of Glass 16
12 samples (2144-2149) of Pemco 1730
12 samples (2150-2155) of Pemco 1729

The coatings were uniform and well-bonded to the polycarbonate, except for the small areas where glass globules were deposited or where spalling occurred.

Optically, the coatings were translucent-to-opaque, varying with the glass type and the coating thickness. Figure 19 illustrates the optical character of the samples in transmitting a pattern and light, as compared with uncoated Lexan polycarbonates. The light source is below the samples and grid. All of the samples show some definition of the grid pattern in transmitted light. The clarity depends upon the thickness and quality of the coatings. Figure 20 illustrates the optical character of the samples in

Table XX

EXPERIMENTAL GLASS COMPOSITIONS

Sample	Composition		Comments
Glass 1	SiO_2	4.0	Soft by 800°C, but not fluid at 950°C. Resultant glass is greenish but transparent.
	B_2O_3	2.5	
	K_2CO_3	1.5	
	BaO	1.5	
	Na_2CO_3		
Glass 2	B_2O_3	5.0	Soft by 800°C, pourable at 950°C. Resultant glass is bluish. Even with its high B_2O_3 content this glass is stable and warrants further modification.
	SiO_2	2.0	
	K_2CO_3	1.5	
	BaO	1.5	
Glass 3	B_2O_3	4.0	Fluid by 750°C, pourable at 800°C. Transparent glass, but greenish. The fluid nature is excellent but chemical stability is very poor.
	SiO_2	1.0	
	Na_2CO_3	2.0	
	K_2CO_3	1.5	
	BaO	1.5	
Glass 4	PbO	6.837	Composition from British Patent #1,041,945. Very fluid, but yellow opaque. No further interest
	B_2O_3	1.428	
	Al_2O_3	0.204	
	ZnO	1.531	
Glass 5	B_2O_3	3.0	Pourable at 950°C. Transparent glass but greenish-brown hue. Chemical stability is poor.
	SiO_2	1.0	
	Na_2CO_3	2.0	
	K_2CO_3	2.0	
	BaO	2.0	
Glass 6	SiO_2	7.3	Not completely melted after 8 hrs @ 1000°C. Optically clear, but viscous. Poor mixing due to the viscosity.
	$CaCO_3$	1.338	
	Na_2CO_3	3.33	
Glass 7	SiO_2	6.92	Melted pretty good after 8 hrs @ 1000°C. Optically clear, but viscous.
	$CaCO_3$	0.894	
	Na_2CO_3	4.408	
Glass 8	B_2O_3	4.0	Excellent properties, clear and pourable after 4 hrs @ 950°C. So good that 400 g batch was prepared for particle impact study.
	SiO_2	3.0	
	K_2CO_3	2.2	
	BaO	1.5	
Glass 9	B_2O_3	3.0	Very viscous, not pourable. Bluish color after 4 hrs @ 950°C.
	SiO_2	4.0	
	K_2CO_3		
	BaO	1.5	
Glass 10	B_2O_3	0.136	Clear glass, but very viscous, not pourable after 4 hrs @ 950°C. The composition taken from G. W. Morey (1932).
	SiO_2	6.826	
	$CaCO_3$	0.88	
	Na_2CO_3	4.349	
Glass 11	B_2O_3	1.0	Not pourable after 6 hrs @ 1000°C. Bluish color. Poor optical properties.
	SiO_2	5.0	
	$CaCO_3$	2.677	
	Na_2CO_3		
Glass 12	B_2O_3	2.0	Good optical and physical properties. Pourable after 6 hrs @ 1000°C. Greenish color, but transparent.
	SiO_2	4.0	
	$CaCO_3$	3.569	
	Na_2CO_3	3.419	
Glass 13	B_2O_3	1.0	Poor glass. Not pourable and opaque.
	SiO_2	5.0	
	$CaCO_3$	2.677	
	K_2CO_3	3.66	
Glass 14	B_2O_3	2.0	Poor glass. Not pourable and opaque.
	SiO_2	4.0	
	$CaCO_3$	3.569	
	K_2CO_3		
Glass 15	B_2O_3	8.0	Bubbled up at ∼700°C; Mostly melted by 850°C, but still some unmelted material at 1000°C. White, opaque, and not pourable, so no further interest.
	Al_2O_3	1.0	
	BaO	1.0	
Glass 16	B_2O_3	4.0	No bubbling observed; All melted by 1000°C. Clear and readily pourable, so may be feasible for plasma spraying.
	Al_2O_3	1.0	
	BaO	5.0	
Glass 17	B_2O_3	0.72	Shows good melting properties; Very fluid at ∼950°C, but melted below 700°C. Looks promising for plasma spraying.
	Al_2O_3	2.05	
	P_2O_5	4.45	
	SiO_2	0.13	
	Na_2CO_3	3.897	
	Li_2CO_3	0.916	
Glass 18	B_2O_3	3.5	Some bubbling. Good melting properties; Very fluid at ∼950°C. Appearance not optimum because of a slight greenish color.
	Al_2O_3	1.0	
	BaO	4.5	
	Na_2CO_3	1.709	
Glass 19	B_2O_3	4.0	Less bubbling than Glass 18. Good melting properties; very fluid at ∼950°C. Good appearance; clear and colorless. Looks promising for plasma spraying.
	Al_2O_3	0.8	
	BaO	4.4	
	Na_2CO_3	0.8	
Glass 20	B_2O_3	3.5	Evidence of melting at ∼700°C. Colorless and pourable at 950°C; clear. Looks promising for plasma spraying.
	Al_2O_3	1.0	
	BaO	4.5	
	K_2CO_3	1.466	

Cont'd of Table XX

Sample	Composition		Comments
Glass 21	B_2O_3 Al_2O_3 BaO K_2CO_3	4.0 0.8 4.4 1.173	Evidence of melting at ~800°C. Essentially colorless and pourable at 950°C; clear. Fritted sample slightly more whitish. Might be promising for plasma spraying.
Glass 22	B_2O_3 Al_2O_3 BaO PbO Na_2CO_3	3.5 1.0 4.0 1.0 0.855	Evidence of melting at ~800°C. Essentially colorless and pourable at 950°C. Looks promising for plasma spraying.
Glass 23	B_2O_3 Al_2O_3 BaO PbO Na_2CO_3	3.0 0.7 3.6 2.0 1.196	Evidence of melting at ~700°C. Higher lead; yellowish color, but still essentially clear. Pourable at 950°C. Plasma spraying could be tried if others fail.
Glass 24	B_2O_3 Al_2O_3 BaO PbO K_2CO_3	3.5 1.0 4.0 1.0 0.733	Evidence of melting at ~800°C. Colorless and pourable at 950°C. Looks promising for plasma spraying.
Glass 25	SiO_2 Na_3AlF_6	9.0 1.0	No melting by 1000°C.
Glass 26	Al_2O_3 Na_3AlF_6	9.0 1.0	No melting by 1000°C.
Glass 27	$CaCO_3$ Na_3AlF_6	15.89 1.1	No melting by 1000°C.
Glass 28	BaO Al_2O_3 B_2O_3	6.684 0.888 2.428	Shows good properties. Melted at 950°C and quite fluid; readily pourable. Colorless.
Glass 29	SiO_2 Na_3AlF_6	7.978 2.795	Evidence of sintering, but no melting by 1000°C.
Glass 30	SiO_2 Na_3AlF_6 B_2O_3 Na_2CO_3	6.0 2.0 1.0 1.0709	Melted by 1000°C, but still rather viscous. Colorless and white regions observed due to lack of mixing.

Table XXI

PARTICLE IMPACT STUDIES OF EXPERIMENTAL GLASS COMPOSITIONS

Glass	Sample	Arc Gas Flow	Current	Electrodes	General Appearance
#8	1688-1735	0.5-2.0	200-800	SG-3	Rounded and irregular transparent particles; some unmelted. Limited adherence and flow.
#8 (-400)	1736-1780	0.5-2.5	200-800	SG-1B(M)*	Small rounded or irregular, transparent particles. Limited adherence and flow.
#16	1897-1923	1.5-2.5	200-600	SG-1B(M)	Rounded and irregular, clear particles. Some adherence and coalescence. Limited film formation.
#16	1924-1950	1.5-2.5	200-600	SG-1B(C)**	Multi-sized, rounded, clear particles. Pretty good melting with some flow and coalescence in several.
#16	2020-2046	1.5-2.5	200-600	SG-3	Multi-sized, round, clear particles. Little adherence and no flow.
#20	1958-1984	1.5-2.5	200-600	SG-1B(C)	Multi-sized, round and irregular, clear particles. Limited flow, coalescence, and adherence.
#20	1991-2017	1.5-2.5	200-600	SG-3	Multi-sized, round and irregular, transparent or translucent particles. Limited adherence and flow, with little coalescence

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* (M) = Metals Nozzle
** (C) = Ceramics Nozzle

Table XXII

PARTICLE IMPACT STUDIES OF COMMERCIAL GLASS COMPOSITIONS

Glass	Sample	Arc Gas Flow	Current	Electrodes	General Appearance
Pemco H-5456	843-869	1.5-2.5	200-600	SG-1B(M)*	Discrete, irregular, rounded, transparent particles. Poor adherence. No coalescence.
Pemco Pb-63	780-806	1.5-2.5	200-600	SG-1B(M)	Transparent particles; some unmelted. Poor adherence. No coalescence but some flow.
Pemco Pb-63	807-842	1.5-2.5	200-600	SG-1B(C)**	Rounded or stringy, transparent particles. Poor adherence and generally no coalescence. Some samples at higher power show good melting and limited coalescence.
Pemco Pb-63	870-881	1.5-2.5	400-600	SG-1B(C)	Transparent, irregular masses. Limited adherence but some coalescence.
Pemco Pb 545	909-926	1.5-2.5	400-600	SG-1B(M)	Few particles. Yellowed film with no adherence.
Pemco Pb 545	930-956	1.5-2.5	200-600	SG-1B(C)	Rounded or irregular, transparent particles. Poor adherence and no coalescence. Tendency to turn yellow.
FERRO 3819 (-100)	1216-1263	0.5-2.0	200-800	SG-3	Multi-sized, transparent particles; rounded and irregular. No adherence or coalescence.
FERRO 3819 (-325)	1264-1311	0.5-2.0	200-800	SG-3	Rounded, transparent particles with white film (unmelted). No adherence or coalescence.
Pemco 1729 (-100)	1313-1360	0.5-2.0	200-800	SG-3	Clear, rounded particles with increased power. Some flow therefore adherence and coalescence observed.
Pemco 1729 (-325)	1361-1420	0.5-2.0	200-800	SG-3	Mainly small, rounded, transparent particles. Some adherence. Yellowed film forms at higher power.
Pemco 1729 (-325)	1421-1456	0.5-2.0	200-600	SG-1B(M)	Small, rounded, transparent particles. Limited flow, coalescence, and adherence.
Pemco 1729 (-325)	1457-1459	0.5-2.0	200-600	SG-1B(M) Shr-N_2	Round and iregular, transparent particles. Some flow, coalescence, and adherence.
Pemco 1730 (-100)	1502-1546	0.5-2.5	200-600	SG-1B(M)	Multi-sized, clear, rounded particles. Some flow, coalescence, and adherence.
Pemco 1730 (-325)	1547-1591	0.5-2.5	200-600	SG-1B(M)	Clear, round, transparent particles. Limited adherence and flow.
Pemco 1730 (-100)	1595-1639	1.0-2.0	200-800	SG-3	Multi-sized, round and irregular, transparent particles. Some adherence but limited flow.
Pemco 1730 (-325)	1640-1687	0.5-2.0	200-800	SG-3	Round, transparent particles. Some adherence but limited flow.

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* (M) = Metals Nozzle
** (C) = Ceramics Nozzle

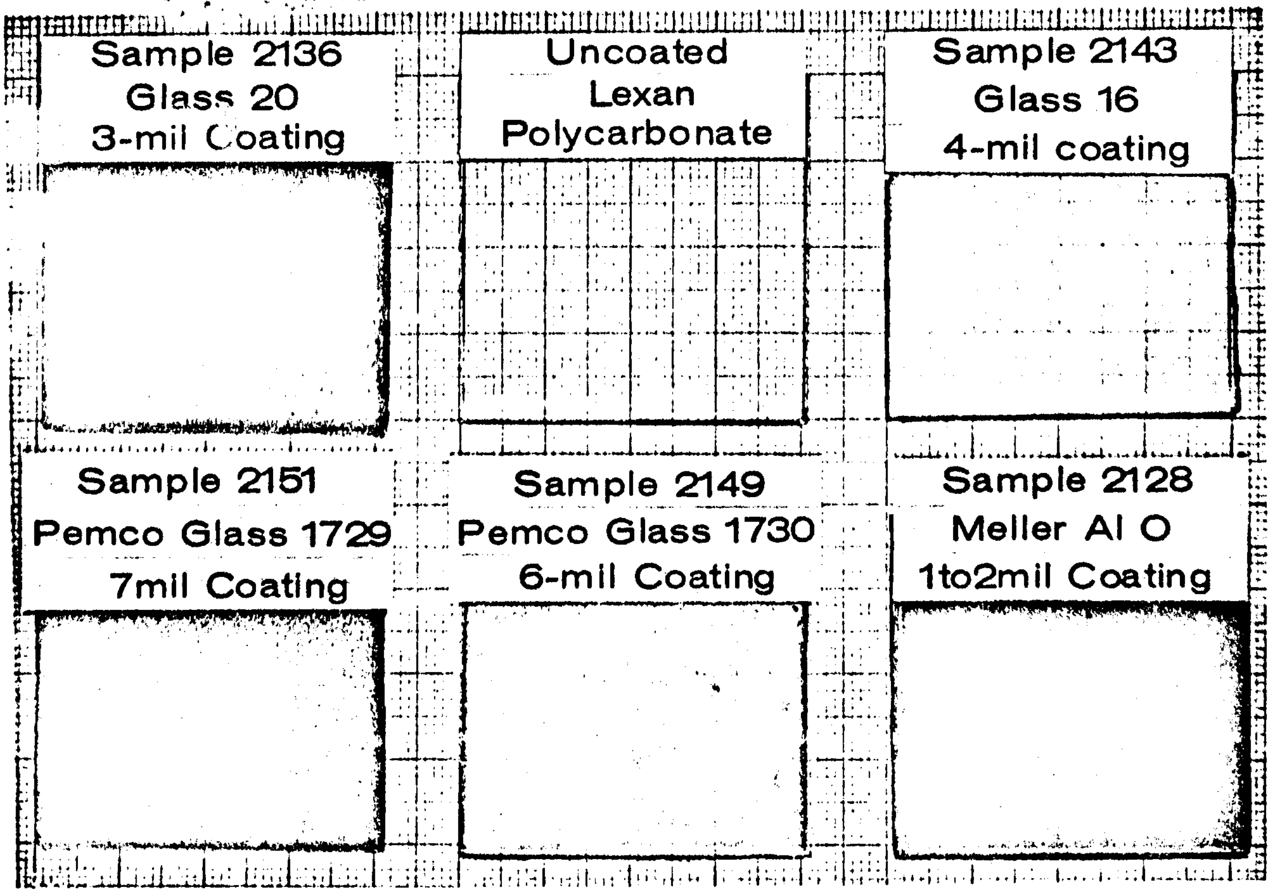

Figure 19. Illustration of Transmitted Light Properties of Plasma-Sprayed Glass Coatings. (Light Source is Below the Samples and Grid.)

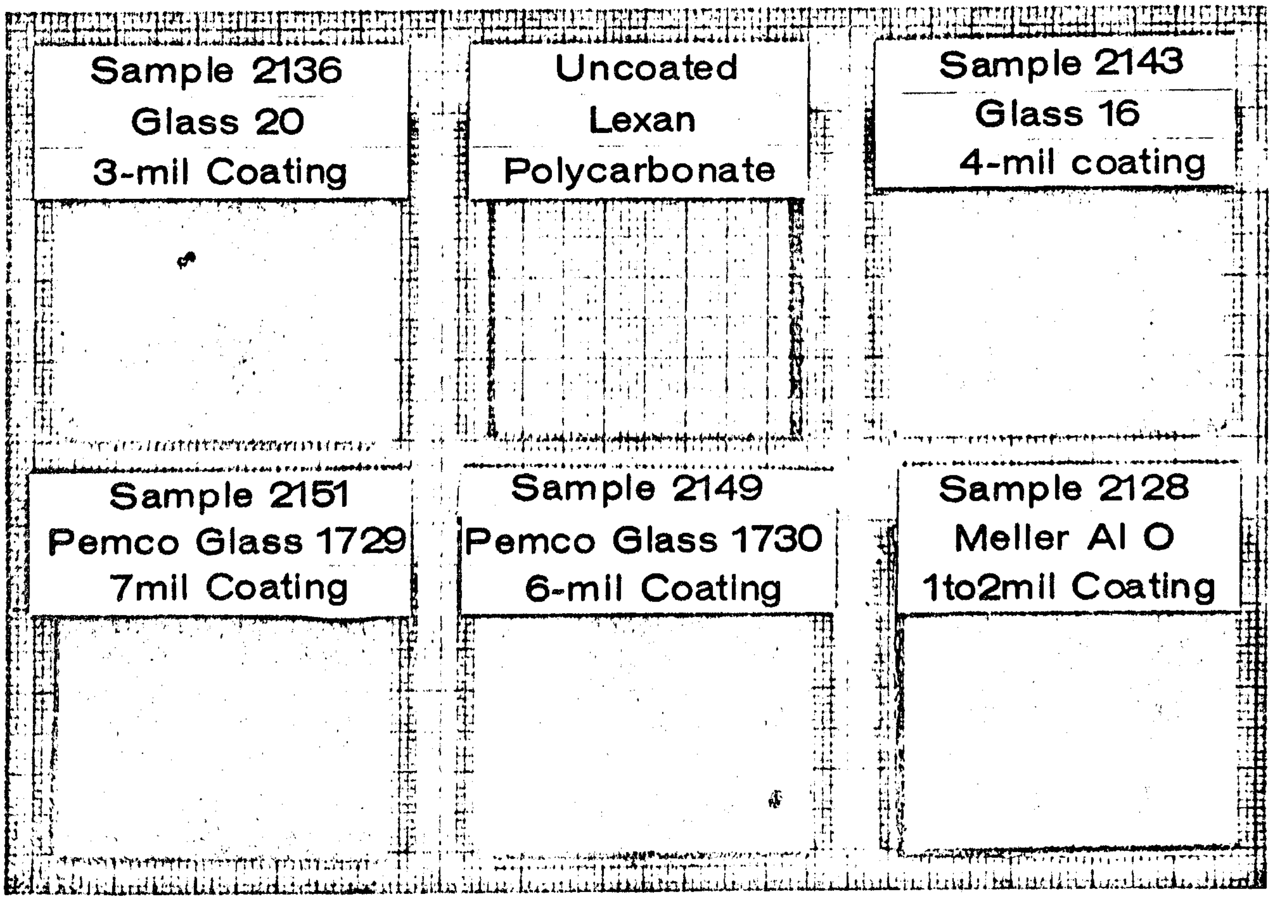

Figure 20. Illustration of Reflected Light Properties of Plasma-Sprayed Glass Coatings. (Light Source is Above the Samples and Grid.)

reflected light; the light source is above the samples and grid. Sample 2143, Glass 16, 4-mil coating, is the only coating, showing definition of the grid. The other coatings are more whitish and opaque, and do not permit penetration of reflected light.

b. Light Transmittance. Using a Perkin-Elmer Model 450 ultraviolet, visible and near-infrared spectrophotometer, light transmittance data were obtained for several plasma-sprayed glass coatings on polycarbonate. The results shown in Figure 21. compare the transmittance of glass-coated polycarbonate with uncoated Lexan polycarbonate. These data experimentally confirm the visual observations; the glass coatings show translucent-to-opaque optical properties. As expected, the film thickness affects the transmittance as shown by the Pemco 1730 sample. The 8-mil coating has up to 5% transmittance, but the 10-mil coating shows 1% or less transmittance. In addition, the chemical composition of the glass appears to be a factor. Glasses 16 and 20 are both basically barium aluminum borate compositions, but glass 20 has potassium carbonate added. The transmittance data show glass 16 has up to 20% light transmission, but glass 20, with thinner coatings, shows no better than 2-4% light transmission. Light transmission properties depend upon the thickness and chemical composition of the coating.

c. Flame Propagation Studies. Using the flame test equipment described in Section 2C, plasma-sprayed coatings of alumina and Pemco glass were tested at 1000°C. The results indicate that such coatings offer only limited thermal resistance to an impinging flame. In the case of the alumina, after exposure to the flame, the coating cracks in $\sim$10 seconds, and the polycarbonate burns through after $\sim$65 seconds. This cracking (due to thermal shock) of the coating results in a drastic loss in flame resistance as the polycarbonate is then exposed to the direct flame. In the case of the Pemco glasses, the coating melts in $\sim$10 seconds, and the polycarbonate burns through after 30 seconds. The performance of these glasses is indicative of the problem in formulating a glass for plasma spraying that offers flame resistance. In order to plasma spray a transparent glass coating on polycarbonate, the glass must be very low melting and have a very fluid melt. As shown by the translucent Pemco glass coatings (which melted in the flame test), even if a transparent glass coating is prepared by formulating a very low melting, fluid composition, the flame resistance of such a coating would probably be extremely limited.

4. Selective Melting of Plasma-Sprayed Glass Coatings by Laser. An attempt was made to optimize the optical quality of these plasma-sprayed glass coatings on polycarbonate by selective melting with a CO_2 laser. It was hoped that the glass coating would absorb enough energy to melt with the polycarbonate remaining transparent to the laser energy. This was not the case, however, because

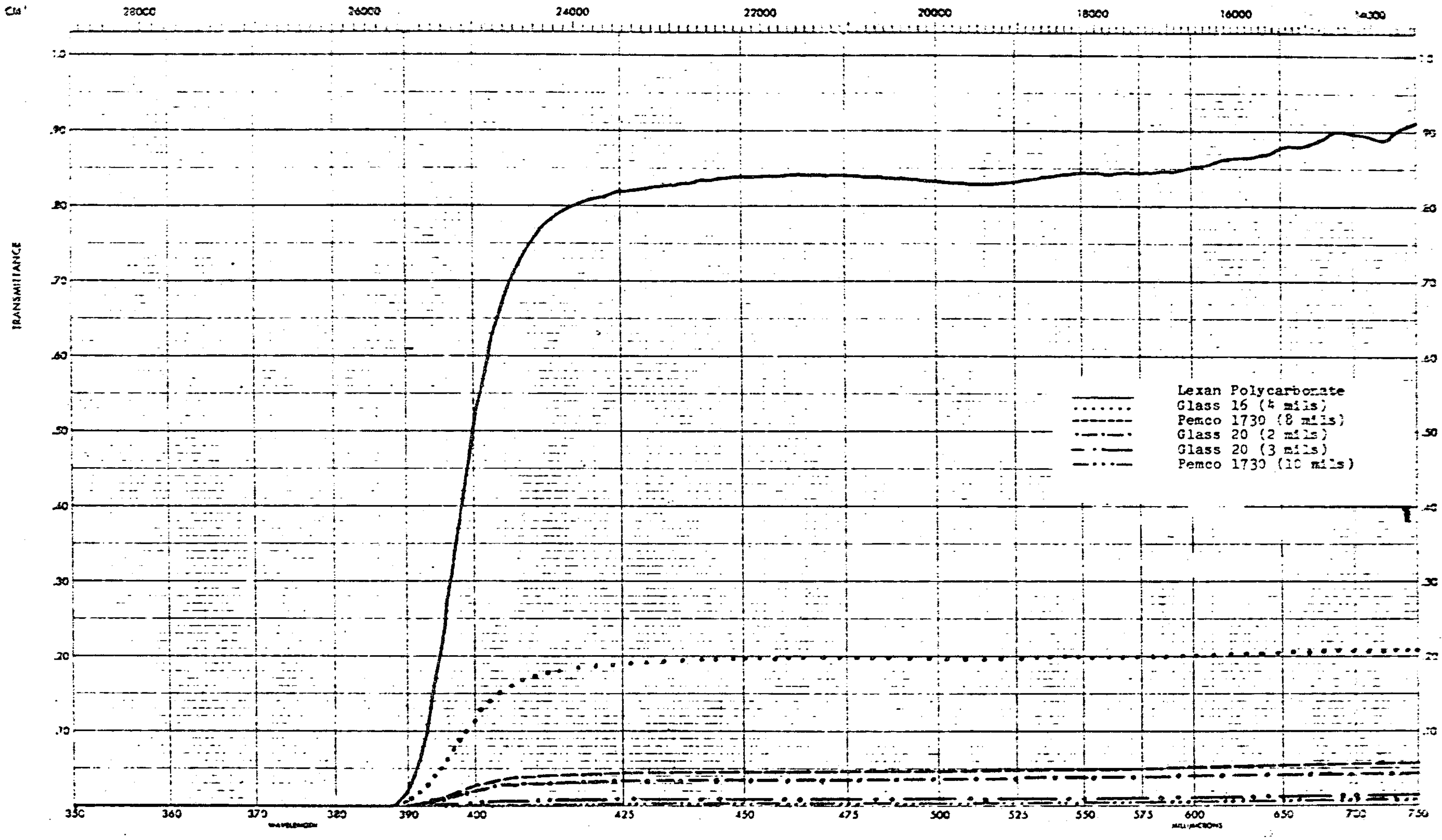

Figure 21. Transmittance of Plasma-Sprayed Glass Coatings on Polycarbonate

enough heat apparently was absorbed and retained in the coating to cause heat-up and deterioration of the polycarbonate substrate. Rather than melting uniformly to produce a transparent, grain boundary-free film, the surface charred and the coating spalled.

D. RF Sputtered Coatings

An RF sputtering capability (diode arrangement) was developed in the last months of this contract. The research was aimed toward establishing the procedures for depositing and actually preparing transparent coatings on polycarbonate. An overall view of the RF sputtering-vacuum system utilized in this work is shown in Figure 22. Figure 23 shows the various components of the sputtering module including: the baseplate-collar, with feed-throughs; moveable, water-cooled anode; Pirani gauge; pyrex spacer; and moveable RF tuning network.

In order to establish the operational procedure for the sputtering module, a metallic target was used initially because a metal coating facilitates observation of the coating process. The target chosen for the preliminary runs was tantalum, because it easily deposits a uniform, mirror-like coating which simplifies the coating thickness determination with an interferometer (thallium light source). One portion of the substrate being coated is masked-off to provide a step for the thickness measurement. In this way, the coating thickness can be correlated with the power settings and deposition time, permitting experimental curves to be drawn for reproducibility of the coating thickness. Tantalum was deposited with no special problems on glass, alumina, ferrite, stainless steel, and polycarbonate.

Transparent Pyrex has been successfully deposited on glass, stainless steel, and polycarbonate. The pyrex on polycarbonate samples required some experimentation to reduce the heating effects on the polycarbonate. Initially, when sputtering under the same conditions used to deposit tantalum on polycarbonate, the deposition of pyrex on polycarbonate resulted in wrinkling and bubbling of the polycarbonate surface due to overheating. This overheating problem was solved by manipulation of the inherent sputtering variables. Similar techniques should be usable for any other transparent, dielectric material to be deposited directly on polycarbonate.

Although transparent coatings of pyrex were deposited on polycarbonate without any physical deformation of the substrate, a light brown discoloration of the substrate occurred. Even though the power level for these runs was set at a minimum, the substrate still discolored. These results indicate that the discoloration of

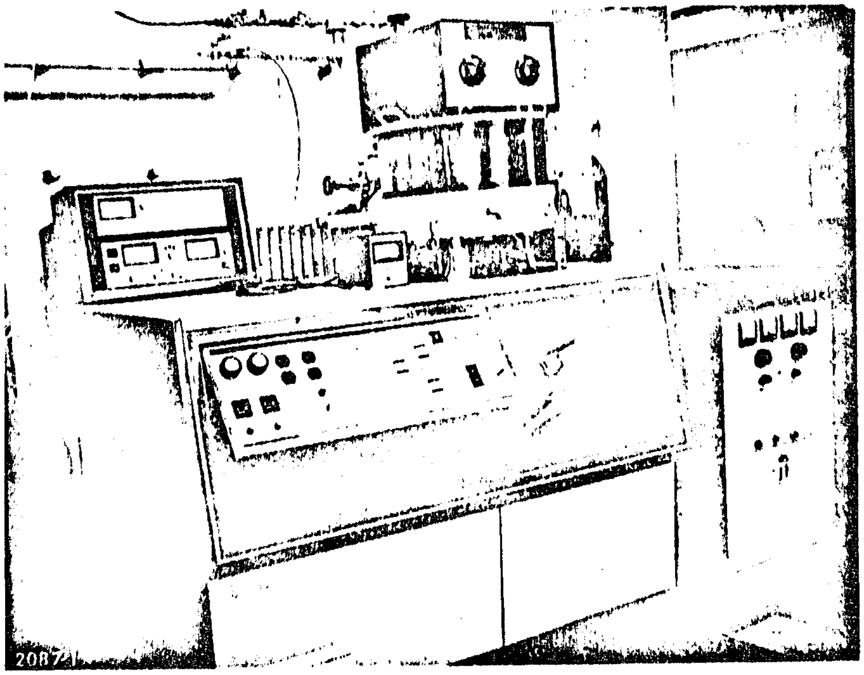

Figure 22. An Overall View of the RF Sputtering Module (SM-8500) Mounted on a Veeco 775 Vacuum System

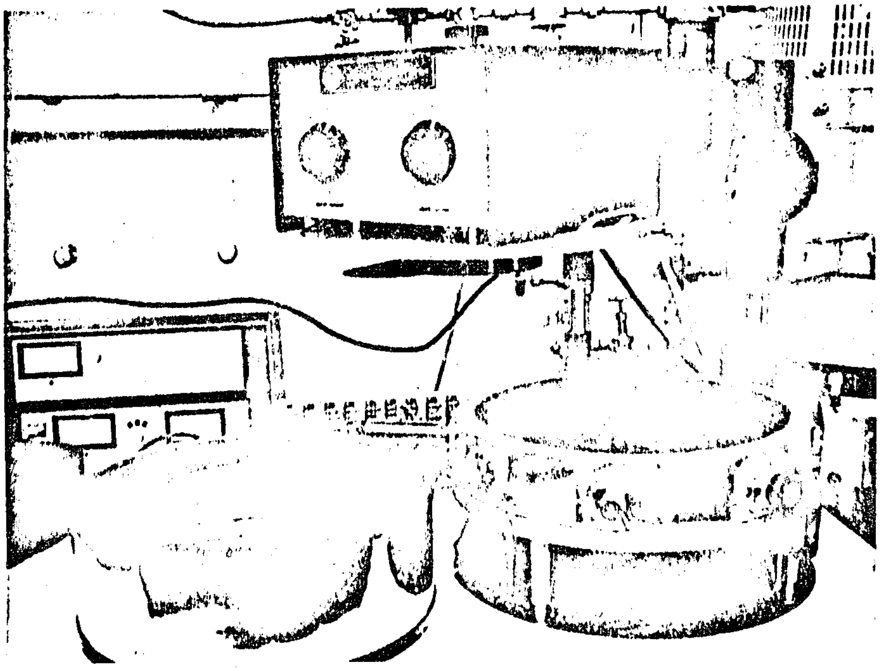

Figure 23. Individual Component of the RF Sputtering Module (SM-8500)

polycarbonate is an inherent problem due to the heat generated in our sputtering module. This slight discoloration of the polycarbonate does not, however, constitute a complete failure of the sample because the sample remains wholly transparent. It is believed that this discoloration problem could be overcome by utilizing radial sputtering. The use of the radial sputtering procedure for the deposition of alumina directly on polycarbonate is more feasible because it is a much lower temperature process.

Attempts to deposit alumina on a silicate-coated polycarbonate resulted in severe bubbling and cracking of the silicate coating upon exposure to the sputtering media for only a few seconds. Slight changes in the sputtering procedure could not sufficiently quell the overheating problem. Initial efforts to deposit alumina directly on polycarbonate were not successful due to the adsorbed water in the target and the resultant outgassing. Heat treatment of the alumina target is underway to remedy this situation.

SECTION IV. CONCLUSIONS

A. Alkali and Nonalkali Silicate Coatings

Polyurethane coatings perform satisfactorily as hard coatings on polycarbonate for improving the adhesion of alkali silicates.

Coatings made from type K sodium silicate and Kasil 6 potassium silicate were superior to other silicate types. Of the two, Kasil 6 potassium silicate appeared to have superior physical properties such as resistance to cracking and wrinkling. In all cases, however, a protective outer film is necessary to prevent silicate degradation.

Both polyurethane and polystyrene were found to produce transparent distortion free protective films provided the silicate surface had been properly ground to eliminate silicate surface irregularities.

The polycarbonate-urethane-silicate-protective (PUSP) coating system performed satisfactorily when evaluated on the basis of thermal shock stability, flame resistance, flame propagation humidity resistance, light transmittance, flexural strength and impact strength. However, long term stability of physical properties has not been determined beyond several months. Some darkening of the protective film was encountered in ultraviolet stability tests indicating a degradation of the polymer. The PUSP systems appear to be relatively unaffected by vacuum down to 10^{-5} torr for 24 hours. However, the effect of lower pressures has not been determined.

B. Plasma-Sprayed Glasses

The particulate nature of the arc-plasma deposition process is the basic reason for degradation of the optical properties of these glass coatings. The plasma torch emits molten particles which solidify on the substrate as distinct units having grain boundaries, causing refraction and reflection of light and resulting in the translucent or opaque appearance of the coating. The original idea that a transparent ceramic or glass coating could be applied by plasma technology was based on preliminary particle impact studies which showed clear, well-melted particles. However, as a coating is built up (thickened) with the present materials, the particulate nature of the coating has an increasingly detrimental effect on the optics of the system. It is likely that a transparent, plasma-sprayed glass coating could be developed, but, in so doing, there would be some sacrifice of thermal protection because the starting composition must be very low melting in order to enhance the fludity of the molten glass and thereby permit better coalescence to minimize grain boundary formation. It is possible that a polymer + glass plasma-sprayed coating would permit optimization of the optics of these systems, but the extent of the thermal protection to be offered by such a coating is not known. Although the plasma sprayed coatings prepared in this program did not possess the optical qualities necessary for instrument dials, such coatings might be of interest for other components where coatings that are abrasion-resistant, impact-resistant, etc., are required.

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