REFLECTIVE METAL COATINGS*

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ABSTRACT

Over seven hundred specimens of cold rolled or stainless steel have been coated with selected plastic formulae consisting of the general species polyurethanes, epoxies and silicones. Coating by the dip technique has been found most satisfactory. Of the 700 specimens over 150 have been coated with films of gold, copper, silver or aluminum and values of the relative reflectance, adherence and radiation absorption of the films have been measured. The relative reflectances, measured with a photometer over the wavelength region of approximately 0.5 to 2.5 microns and indicated as the relative reflectance of the specimen to that of a gold coated glass standard, have been shown to compare favorably with similar films respectively deposited on glass. The relative reflectance of silver films on polyurethane substrates was highest and the relative standing of the other metals in descending order was copper, gold and aluminum. The relative reflectance of a given metal varied with the type of plastic coating on which it was deposited in the descending order polyurethane, silicone and epoxy. Typical relative reflectance values found for gold on each material respectively were 0.96, 0.86 and 0.84; the value on glass was 0.995.

Adherence measurements, made by a headed pin technique, revealed that the order of adherence was reversed from that of the reflectance, i.e., adherence values were in the descending order aluminum, gold, copper and silver. Adherence of the metal films to the epoxy, Maraset 617-C, was superior to that to polyurethane coatings. Films of aluminum passed the "scotch tape test" when deposited on epoxy or silicone coatings but did not perform as well on polyurethane. Gold films adhered well to the epoxy, Maraset 617-C. When the film adherence value was above approximately 550 psi the film normally would pass the scotch tape test.

For aluminum films deposited on glass the relative reflectance increased slightly as the film thickness was increased over the range 1500 to 25,000 angstroms. For similar films on an epoxy coating, however, the relative reflectance decreased from 0.85 to 0.69 as the film thickness was increased over the range cited above.

Radiation absorption measurements of 25 coated steel specimens, supported in a vacuum calorimeter, confirmed in general the photometer

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measurements with the exception that aluminum films placed second instead of fourth in heat shielding effect. Remaining problems are reflectance and radiation absorption measurements over the spectral range 1 to 15 microns and the development of methods for improving the adherence and durability of the metal films.

INTRODUCTION

The conquest of space by the use of ballistic missiles and satellites has presented scientists with the problem of temperature control in zones where heat transfer is predominantly by radiation. This event has revealed that a dearth of information exists with regard to the absorption, reflectivity and emissivity of constructional materials in the near infrared region of the electromagnetic wave spectrum, about 1 to 15 microns.

One method of temperature control for an instrument in a heat radiation field is to place it in a closed container whose surface has known optical properties.

The fabrication of a container entirely of a material of known and desired optical properties, such as gold, or silver, is impractical because of considerations of weight, economy, mechanical strength, and the cost of mechanically polishing the surface. On the other hand, a coating technique whereby the surface could be given desirable optical properties appeared feasible.

A suggested method of simply controlling the optical properties of a container was to coat the container with an adherent plastic film of high gloss and subsequently to coat the plastic with a metal film of the desired optical parameters. However, the selection of a plastic, its method of application, and the selection of the coating metal in order to provide coatings of both the proper optical properties and sufficient durability was not so simple. This required further information concerning the materials, the methods of application and experimental evidence on reflectivity, adherence, and film aging when exposed to expected extremes of temperature and pressure. This paper describes progress of work planned to increase information in these areas.

PLASTIC COATING THE METAL SUBSTRATES

General

In order to attain desired standards of strength and durability preliminary experiments were conducted with steel substrates of a convenient size, 1" x 1-1/2" x 1/32", about the size of one-half of a glass slide commonly used in microscopy. The plastics studied were those selected for low vapor pressure and high temperature resistance. These included selections from the species of epoxies, silicones and polyurethanes, varied as to formula in an effort to approach the desired conditions of adherence, high gloss and durability. These were applied by spraying, dipping, brushing or flowing on. However, dipping proved to be the more satisfactory method and was the one predominantly employed.

Metal films were applied to the surface of the plastic film by the vacuum evaporation method. Metals employed were gold, silver, copper and aluminum.

Adherence measurements of the plastic to the metal and of the metal film to the plastic were conducted by a headed pin technique; and reflectance measurements of similar films were made by means of a photometer registering over the approximate wavelength range 0.5 to 2.5 microns. Subsequently, the rate of temperature rise of specimens supported in a radiation field in vacuo was determined. These experiments are outlined in detail in the subsequent paragraphs.

Substrate Cleaning and Coating

Substrates initially used were hot-rolled mild-steel sheet but undesirable surface non-uniformities resulted in a change to cold rolled steel and subsequently to stainless steel sheet. A few specimens of aluminum and copper were also coated. A number of cleaning techniques were tried and the following one was selected as satisfactory.

(1) Vapor degrease the substrates.
(2) Scrub substrates in Kyro EO (detergent) and rinse in cold tap water.
(3) Dip for 10 seconds in 2-1/2 molar ferric chloride.
(4) Rinse in cold tap water.
(5) Rinse in boiling tap water for 5 minutes.
(6) Dip in acetone.
(7) Repeat vapor degreasing.
(8) Dry the substrate in air at room temperature.

Immediately after cleaning, substrates were coated with a plastic material by spraying, brushing, flowing on or by dipping. After a few specimens were examined visually for surface gloss the dip method was selected as generally superior. One to four coats proved satisfactory. Film drying was conducted in a drying chamber with a filtered air supply; this was constructed of plywood and plexiglass and is exhibited in Figure 1. It allowed the fabrication of dust free specimens.

Coating Experiments

Five epoxy resins were employed as coating materials. These resins were: the Shell products Epon 1001, Epon 834 and Epon 815 and two products made by Food Machinery and Chemical Co., Oxiron 2001 and Oxiron 2002. The Shell epoxies were used with various combinations of resin, catalyst, solvent, and curing agent; they were found to give grainy and matte surfaces in all tests. The Oxirons were also mixed with various concentrations of resin, catalyst, curing agents and solvents. In tests where solvents were not used, the Oxiron mixtures were brittle, and in runs using solvent they were tacky and did not cure.

Three silicone resins were examined. These were General Electric's products SR-53, SR-82 and SR-111. Of the three products, SR-111 possesses the more desirable properties. SR-53 was too thin to be practicable for use, even after six dips of resin. It also formed a matte surface. SR-82 did not appear suitable as a coating agent; it formed small droplets which did not adhere to either steel or plastic over steel. SR-111 gave a smooth thick glossy coat which was somewhat softer than polyurethane coatings. It withstood temperatures of 500°F for two hours, but it has not been tested for longer periods of time. This resin was found to have the desirable characteristics of good adherence to metal substrates, high gloss, hardness, and heat resistance. A distinguishing feature of the silicone resin is its ease of application. Substrates can be coated with this material at room temperature directly with the commercial formula.

A silicone varnish and an acrylic coating were tried as coating materials. They were Dow Corning's #997 Silicone Varnish and Zac Lac, an acrylic hardboard coating. The varnish, at concentrations of 25% and 50% solids, remained soft after being heated at 300°F for 24 hours. Studies conducted here have shown that a concentration of 25-50% solids is needed to mask the surface of the substrate. The Zac Lac gave an even, smooth coat but cracked and blistered when exposed to a temperature of 425°F for 1-1/2 hours.

A polyurethane, Sy 627-119, marketed by the Ferbert Shorndorfer Co., and consisting mainly of du Pont's hylene isocyanates, proved to be an excellent agent. This plastic has been tried with several polyester leveling agents (at concentrations of 1-2 pph) at 25% and 50% solids, using ethyl acetate and xylene in equal mixtures as solvent thinners. The 25% solids dip gives a more level coat than the 50% solids dip. This material was applied by dipping the slide into the liquid plastic. The method is simple and gives smooth even coatings when suitable materials are used.

The third satisfactory plastic was an epoxy resin, Maraset 617-C, produced by the Marblette Co. This plastic is composed of two components which must be mixed at a temperature of 125-135°C. The resin must be applied to hot substrates at approximately the same temperature. Except for the inconvenience occasioned by the necessity for the higher temperature, this resin was satisfactory as a coating material both as to adherence and gloss.

DEPOSITION OF METAL FILMS

Metal films were deposited by a normal vacuum evaporation process in the vacuum system exhibited in Figure 2. For best adherence of the metal films to the plastic substrates cleaning of the plastic film after drying was found desirable; the method most commonly used is outlined below.

(1) Scrub specimen with a pipe cleaner in hot (70-90°C) Alconox solution (approximately 1 teaspoonfull of the powdered commercial detergent Alconox to 1 liter of distilled water).

(2) Rinse specimen in hot running tap water.*
(3) Rinse specimen in hot (70-90°C) distilled water.
(4) Rinse specimen in Methanol.
(5) Dry with hot air.

Films of gold, silver, copper and aluminum were deposited on selected plastic coated specimens and some films were deposited directly on glass. Film thicknesses were generally about 3,000 angstroms, but limited studies of films over the range 1500 to 25,000 angstroms were conducted.

Metal coated specimens were examined for adherence of the metal film to the plastic and for the reflectance of the film.

Adherence Measurements

General

Adherence measurements were made of some 122 metal film specimens deposited by evaporation onto plastic coated metal substrates. These consisted of 33 gold films, 20 silver films, 20 copper films and 26 aluminum films. Measurements were made both by the scotch tape test, which is essentially a go or no go test, and a headed pin test. The former consists of pressing a length of scotch tape firmly against the film; then, pulling it rapidly away from the substrate to see if the film is removed. The latter consists of gluing or soldering a brass headed-pin to the metal film and withdrawing the pin from the film in a direction normal to the film by use of a tension apparatus. The tension at failure of the joint is recorded. Approximately five measurements were made for each film. Eastman 910 adhesive was found to be satisfactory for a bonding agent to gold, silver and copper but not to aluminum. For aluminum the joint broke most frequently at the glue interface and failed to remove the film.

Headed pins were soldered to some films with a low melting solder, Cerroseal (50% tin - 50% indium with a melting point of 118°C) and removed as described. However, if soldering is used it is best to overcoat the metal film to be examined by a second film such as evaporated copper or nickel before the specimen is removed from the vacuum chamber. The second film prevents damage to the first film by solution in the tin of the solder, in the case of gold and silver, and promotes solderability in the case of aluminum. Alloys of lower melting temperatures than Cerroseal such as Woods Metal or related members of this series may also be useful but have not been used thus far in this work.

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*For the epoxy Maraset 617-C, an additional two steps were taken between steps (2) and (3) above. These were: a dip in hot chromic acid followed by a rinse in hot running tap water. This procedure appeared to increase considerably the adherence of the metal film subsequently deposited.

The complete details of each measurement including substrate, plastic coating, and cleaning methods were recorded. A summary of the measurements is shown in Table I.

Gold Films

Examination of the first section of Table I, devoted to gold, reveals that gold films were deposited on plastic coatings of three general species; 18 were deposited on the epoxy Maraset 617-C; 34 on polyurethanes; 2 on silicones and 2 on Oxiron, another epoxy. Only 11 films passed the scotch tape test; the remaining 45 did not. Of the eleven, 7 were on Maraset 617-C, 1 on polyurethane, 2 on silicone (the only two measured) and one of the two on Oxiron. Films which did pass the test exhibited tensile stresses for removal >550 psi on Maraset 617-C. The tests on the silicone and Oxiron were lower but too few to be significant. Changes in cleaning techniques for the polyurethane coatings did not improve adherence values to a marked degree, nor sufficiently for the films to pass the scotch tape test.

It is evident that the gold adhered best to the epoxy Maraset 617-C. The wide scatter of the data is characteristic of such adherence tests. However, the higher values agree with findings by the scotch tape method.

Silver Films

The adherence of 20 silver films was measured. Adherence values obtained were distinctly less than for gold. No specimen survived the scotch tape test. A summary of the data is shown in Table I. Adherence of the silver film to the polyurethane coating is less than to the epoxy.

The failure of the silver to perform as well as gold in the adherence measurements was surprising and deserves further review for full substantiation.

Copper Films

A summary of the adherence measurements of 20 copper films are exhibited in Table I. It will be noted that copper films on Maraset 617-C, in general, passed the scotch tape test whereas those on polyurethane did not.

Aluminum Films

Adherence measurements of 20 aluminum films were made. A summary of these are listed in Table I. All aluminum films deposited on silicones or the epoxy, Maraset 617-C, passed the scotch tape test. Those on polyurethane failed it.

The adherence measurements to the aluminum films by the headed pin method are not considered valid since the bond usually broke between the pin and the film. Overplating and soldering measurements were also poor

TABLE I

SUMMARY OF ADHERENCE MEASUREMENTS OF METAL FILMS ON PLASTIC COATED STEEL SUBSTRATES

Plastic Overcoat	Tensile Measurements (psi)			Remarks
	Low Value	High Value	Average Value of Five Highest	
		GOLD FILMS		
Epoxy, Maraset 617-C	224	933	729	Passed Scotch Tape Test
Epoxy, Oxiron	265	556	410*	- - - - - - - -
Polyurethane	75	521	503	Failed Scotch Tape Test
Silicone*	316	429	373	Passed Scotch Tape Test
		SILVER FILMS		
			(only four values)	
Epoxy, Maraset 617-C	234	449	325	Failed Scotch Tape Test
Polyurethane	143	326	263	Failed Scotch Tape Test
		COPPER FILMS		
Epoxy, Maraset 617-C	87	675	575	Passed Scotch Tape Test
Polyurethane	143	581	404	Failed Scotch Tape Test
		ALUMINUM FILMS		
Epoxy, Maraset 617-C		No Useful Data		Passed Scotch Tape Test
Polyurethane		No Useful Data		Failed Scotch Tape Test
Silicone	204	796	629	Passed Scotch Tape Test

*Only two measurements.

since the second coat was applied after exposure to air; this caused fracture between the aluminum film and the overcoating film.

Relatively high values of adherence to the silicone material were obtained (up to 796 psi). However, there appeared to be a chemical reaction between the aluminum film and the silicone coating accompanied by a loss of gloss.

Additional adherence measurements of the aluminum films must be made in order to obtain realistic data. It is probable that the film-substrate adherence value is greater than 550 psi since all films except those on polyurethane passed the scotch tape test.

Reflectance Measurements

The Photometer

For the reflectance measurements a photometer sensitive over the approximate wavelength range 0.5 to 2.5 microns was constructed. The photometer and its optical path are exhibited in Figure 3. A plywood box, coated on its interior with flat black paint, encloses the optical path. The source is a quartz infrared lamp of the pencil type mounted in a cylindrical parabolic reflector, a polished, gold-plated copper sheet. During measurements, the source voltage is set at 40 volts with a variac and remains on continuously. For this source voltage, the detector temperature stabilizes at approximately 27°C. Measurements are begun after temperature stabilization and after the proper optical adjustments are made. A given specimen under test is exposed to radiation from the source and is adjusted axially for maximum reflected energy to the detector, a Kodak Ektron detector of the type N-2. An indication of the integrated radiation arriving at the detector is obtained by measuring the detector's resistance with a wheatstone bridge. A typical series of measurements is as follows:

	Detector Resistance (ohms)	Resistance Symbol
Source off	6.10×10^5	R_1
Source on, no specimen	6.08×10^5	R_2
Source on, standard specimen adjusted axially for maximum radiation intensity at detector	3.50×10^5	R_3
Source on, specimen being compared with standard adjusted axially for maximum radiation intensity at detector	3.60×10^5	R_4

There are a number of methods by which a figure of merit of relative reflectance for a coated specimen may be calculated using combinations of the measured detector resistance values cited above. However, all of these except one exhibited poor repeatability for the calculated values utilizing measurements conducted at different times.

By utilizing the expression $1 - \frac{R_4 - R_3}{R_2 - R_3}$ a repeatable relative reflectance was obtained. This quantity, simplified to the form $\frac{R_2 - R_4}{R_2 - R_3}$ and termed "relative reflectance", increased as the reflectance increased and varied over the approximate range zero to unity. Furthermore, this value could be expressed in terms of percent relative reflectance simply by multiplying it by 100.

This method of calculation gives a relative figure of merit for a given specimen with respect to a standard reflector. The standard is a gold coated microscope slide with approximately 3000 angstroms of gold deposited on it by evaporation. Some specimens examined were better than the standard and gave a value greater than unity for the relative reflectance. A value greater than unity means that the specimen directs more energy to the detector than does the standard.

Usually, four measurements of $(R_4 - R_3)$ were taken successively and the average value was recorded. The denominator $(R_2 - R_3)$ was found to be essentially constant at 2.58×10^5 ohms, and this value was used for all calculations of the relative reflectance data tabulated or plotted. The uncertainty in the average measurements is about one percent.

Reflectance measurements of over 183 plastic coated metal specimens overcoated with metal films were made with the photometer. These consisted of approximately 71 specimens coated with gold films, 22 specimens coated with silver, 23 coated with copper and 67 coated with aluminum. The substrates were predominantly plastic coated steel or stainless steel, but a few were glass.

Gold Coated Substrates

Some 71 gold coated specimens were examined; 32 of these were made after a standard relative reflectance measurement technique had been established and are used as the basis for this discussion.

The data obtained can best be interpreted from the graphical representation of Figure 2. Here the ordinate axis represents the relative reflectance as determined by the method outlined above; the abscissa represents the number of the particular specimen, 1 to N, plotted at equally spaced positions.

The specimens examined were coated with three types of plastics, polyurethane, silicone, and epoxy. These are identified by the plotting point code on the graph. Two entries of the reflectance of gold plated glass slides are also shown to exhibit the accuracy of the 100 percent line.

It is immediately discerned, in spite of considerable scatter of points, that the polyurethane coats are superior to those of the epoxy, Maraset 617-C. In fact, the average reflectance is 0.960 for the polyurethane compared to 0.836 for the epoxy coated units. The silicone value, for only two units tested, was 0.860. The gold coated glass value, for two units also, was 0.997.

Somewhat unusual are the three points above unity; however, it must be remembered that the unity value is not absolute but arbitrary, and it is to be considered as a reference line only. At the same time it is apparent that some coatings on plastic may be as good or better than those on glass, dependent on coating conditions and the thickness of the film.

Silver Coated Substrates

A similar plot of the data obtained for 23 silver coated substrates is exhibited in Figure 3. The pattern of behavior of the gold coated substrates is virtually repeated except that more data points are located above the unity line. This appears to indicate that silver is a better reflector of radiation in the wavelength region 0.5 to 2.5 microns than is gold. This is of course borne out by data in the literature.*

Copper Coated Substrates

Data for 22 copper coated specimens are exhibited in Figure 6. A similar pattern of behavior for the various coatings is exhibited. In this case, the relative position of average values is almost identical to that of the gold coated specimens. It is interesting to note the relative reflectance of a specimen of polished bulk copper is recorded as 1.05 compared to copper coated glass specimen. This is probably a function of metal thickness as discussed subsequently.

Aluminum Coated Specimens

Data for 67 aluminum coated specimens are exhibited in Figure 5. The pattern of behavior previously revealed is repeated. The relative reflectances of aluminum plated units appear to be slightly below those obtained for similar specimens respectively of the other three metals. Data for a larger series of silicone specimens are exhibited. The group of points near the bottom of the figure represent films which seemed to have reacted in some manner with the aluminum films on deposition; these films were discolored and were dull in appearance to the eye.

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*W. E. Forsythe, Smithsonian Physical Tables, Ninth Revised Edition, p. 552, Smithsonian Institute, Washington, D.C. (1954).

Comparison of Metal Platings

From the plotted data and by averaging all points except those obviously out of the general data group, Table II was assembled. Here the relative standings of the metals and the plastic coated reflectors may be determined. Values for bulk metals are also shown. It is evident that the plastic coatings, which were studied, were more satisfactory as reflectors in the descending order polyurethane, silicone, epoxy and that the glass bases were only slightly better than the polyurethane. One combination, silver on polyurethane, is better than silver on glass. Likewise, the order obtained from the tables for the metals is in the descending order silver, copper, gold, aluminum; although, copper and gold were almost equal within the limit of error. Of course one must remember that the limited range of the study (to only 2.5 microns) will give misleading information when one must consider the entire range 1 to 15 microns.

Variation of Reflectance with Film Thickness

It has already been noted that polished bulk copper gave a reflectance ratio in the photometer of 1.05 for a gold plated glass standard and may be compared to 1.01 for a copper plated glass standard.

A series of specimens of plain glass and steel substrates coated with Maraset 617-C were coated with aluminum films in the thickness range 1500 angstroms to 25,000 angstroms. The thicker films had to be made by multilayer evaporation exposing the sample to air between every coating thickness of about 5000 angstroms. Hence, the thicker films had up to four exposures to atmosphere.

A plot of the data obtained for the relative reflectance of coated glass specimens is shown on an expanded scale in Figure 8. Since there were only two points for each thickness there is considerable scatter of the data. However, a positive slope of reflectance is evident. Coincidentally or otherwise a reflectance of unity is reached at the 2.5 micron thickness range, the wavelength of the longest radiation employed. Although admittedly the data are insufficient for strong argument, a definite trend toward greater relative reflectance with greater thickness appears to be established - as might be expected.

On the other hand, as shown in Figure 9, a line plot with a negative and larger slope was found for the reflectivity versus thickness data for evaporated aluminum films on the epoxy, Maraset 617-C. This particular plot has points taken from single films made during the same time as the glass specimens of Figure 6. In addition, four points are added at 2750, 3000, 13,300 and 18,000 angstroms, respectively, representing averages of four or more specimens, each made without glass specimens present. Hence, there are enough data to insure validity of the findings. In addition, the films lost their bright appearance to the eye as the thickness increased. However, x-ray diffraction examinations of the films on the two substrates failed to reveal significant differences in film structure. At first it was considered that the negative slope

TABLE II

SUMMARY OF RELATIVE REFLECTANCES OF VARIOUS METAL FILMS AND METALS

RELATIVE REFLECTANCE*

METAL FILMS

Substrate (Plastic Coating)	Gold Est. Val.	Gold Calc. Val.	Silver Est. Val.	Silver Calc. Val.	Copper Est. Val.	Copper Calc. Val.	Aluminum Est. Val.	Aluminum Calc. Val.
Polyurethane	0.975	0.96	1.03	1.037	0.975	0.979	0.95	0.947
Maraset 617-C	0.84	0.83	0.845	0.855	0.86	0.845	0.80	0.809
Silicone	0.86	0.86	-	-	-	-	0.89	0.897
Glass	0.995	0.997	1.01	1.009	1.01	1.012	0.99	0.987

BULK METALS

Metal	Substrate from Shop, Uncleaned	Polished
Steel	0.02	0.867**
Aluminum	0.807	0.895
Copper	0.04	1.05

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*Relative Reflectance $\frac{R_2 - R_4}{R_2 - R_3}$. R_2 is the detector measurement with source on and no specimen in the holder. R_3 is the measurement with source on and the standard inserted and adjusted. R_4 is the measurement with the source on and the new specimen inserted and adjusted.

**This value appears to be somewhat high for steel.

might be attributed to surface defects or roughness increasing with film growth, as is commonly observed for thick electroplated films. However, the fact that films on glass did not behave in this manner cast doubt on the correctness of this conjecture. An alternate explanation is that the heat of evaporation released damaging exhalations from the plastic substrate. Some reaction between silicone coated substrates and aluminum films were observed in a similar situation. A third explanation may be optical interference effects, but more data are needed to identify the reason.

MEASUREMENTS OF RADIATION ABSORPTION IN VACUO

Apparatus

A radiation calorimeter was constructed which consisted of a 2000 liter beaker, coated on its interior with gold; a quartz-faced infrared heat lamp; a high vacuum system and two thermocouples. The device was assembled by inverting the beaker over the lamp. Two small holes in the bottom of the beaker permitted access to the radiation field. A thermocouple was inserted through one of the holes. The specimen under test, soldered with tin to a second thermocouple which supported it, was inserted through the other hole. Comparison of variously coated specimens was made by taking temperature versus time measurements, after turning on the lamp, at a pressure of 2×10^{-5} mm of Hg. The lamp filament was set by an optical pyrometer at a color temperature of 1250°C. The radiation on the specimen is the integrated emission from the lamp which extends over the visible spectrum to somewhat above 2 microns in the infrared and is repeatedly reflected within the gold plated beaker.

Temperature Versus Time Measurements

A total of 25 steel drill-rod specimens (1/8" in diameter by 2-1/2" in length) were prepared and tested with the calorimeter as described. Of these, 21 were coated with polyurethane SY 627-119 and subsequently metallized, 2 were coated with polyurethane only, and 1 was initially tested in the uncoated condition and then coated with soot and tested. The 21 metallized specimens consisted of 8 coated with gold to thicknesses of 2000 and 4000 angstroms, 6 coated with silver to 2000 and 4000 angstroms, 3 coated with copper to 2000 angstroms, and 4 coated with aluminum to 2000 angstroms. These data are presented more specifically in Table III.

Figure 10 shows typical plots of temperature versus time data obtained. Measurements were taken at 50 second intervals for each specimen over a specimen temperature range from approximately 25°C to 200°C. The time interval (about 650 seconds) for a metallized specimen to reach 200°C was about 3 times as great as that for the uncoated specimen and 5 times greater than for the same specimen coated with soot. From the figure, it can be seen that for a specimen temperature of about 200°C the corresponding temperature of the thermocouple in the radiation field was 350°C.

TABLE III

SPECIFICATIONS OF DRILL ROD SPECIMENS EXPOSED TO RADIATION IN VACUUM CALORIMETER

Specimen (Code)	Metal	Approximated Metal Thickness (Angstroms)	Number of Specimens	Plastic Coating
Au-1-R, . . ., 5-R	Gold	2000	5	Polyurethane, SY 627-119
Au-6-R, . . ., 8-R	Gold	4000	3	"
Ag-1-R, . . ., 3-R	Silver	4000	3	"
Ag-4-R, . . ., 6-R	Silver	2000	3	"
Cu-1-R, . . ., 3-R	Copper	2000	3	"
Al-1-R, . . ., 4-R	Aluminum	2000	4	"
P-1 and P-2	None	-	2	"
R-1	None	-	1	None
R-1-Blackened with Soot	None	-	1	None

For the various specimens tested, the ratio of the required time for a given specimen to reach 200°C to the time for the black rod to reach 200°C was determined. These ratios versus the specimen number, 1 to N, are plotted in Figure 11. An alternate method of exhibiting the data is to plot the time rate of temperature change in degree centigrade per second; this is obtained by extrapolating the slope of the calorimeter temperature versus time curves. These data are exhibited in Figure 12. As may be seen, there is a considerable scatter of the results. Upon examination of the specimen with a microscope, it was noticed that the metal films had thin areas; in such areas, the aluminum and silver films appeared dull and discolored as if some sort of reaction had occured. Those films which showed the greatest quantity of thin area corresponded to points on Figure 12 which indicated high absorption. The cause of the thin areas over the film surface was poor evaporation filament geometry or failure to rotate the specimen during evaporation.

The amount of data obtained is insufficient to make any final comparison between variously coated specimens. However, if the widest scattered points (Nos. 1,4,12,14,16 and 18) are neglected, Figure 12 reveals that the maximum protection in descending order for the various metals was silver, aluminum, gold and copper; the corresponding slope averages in °C per second are 0.251, 0.272, 0.289, and 0.315. This differs from the photometer results where the similar order was silver, gold, copper and aluminum.

SUMMARY

Steel specimens coated by dipping with a plastic of the polyurethane type and further coated with either of the metals silver, copper, gold or aluminum reflect light in the wavelength region 0.5 to 2.5 microns virtually as well as similarly coated glass surfaces. The order of relative reflectance from high to low is the order of the metals as named above. On the other hand, similar metal substrates coated with resins of an epoxy or silicone base reflected electromagnetic radiation of the same wavelength range distinctly less well.

The order of adherence of the metal films to the plastic coated substrates was the reverse of the order of reflectance both as to type of metal and as to type of plastic coating, i.e., the decreasing order of adherence of the metal films to the plastic coated specimens was aluminum, gold, copper and silver; and of the plastics, for specific metal, the order was epoxy, silicone and polyurethane. The only pairs passing the scotch tape test were aluminum films on an epoxy and on a silicone, gold on an epoxy, and copper on an epoxy. The value established as passing or failing the scotch tape test was approximately 550 psi.

The relative reflectance for aluminum films deposited on glass increased to a small degree with thickness of the aluminum film in the range 1500 to 25,000 angstroms. In contrast to this, the relative reflectance of similar films on an epoxy coated steel degraded with thickness. This effect is considered highly interesting by the investigators, but the reason for it has not yet been established.

Radiation absorption measurements of specimens supported in a vacuum calorimeter gave data which confirmed the relative heat radiation shielding value of the various metal films for plastic coated metal surfaces. The single disagreement in the order of protection, the position of aluminum films moved up from fourth to second position, may be possibly ascribed to differences in the spectral range utilized in the two methods or the small number of specimens examined.

More data covering the entire spectral range 1 to 15 microns will be required to resolve the precise relative standing of the metal films in the applied form. However, the properties of the films sufficiently resemble that of the bulk metal to accept the bulk metal measurements as correct with regard to relative position.

The metal films applied to steel substrates overcoated with polyurethanes gave marked heat radiation shielding. Remaining problems are reflectance and radiation absorption measurements over the entire range 1 to 15 microns and the defining of methods to improve the adherence and durability of the metallic films.

Figure 1 Chamber for Dust Free Drying

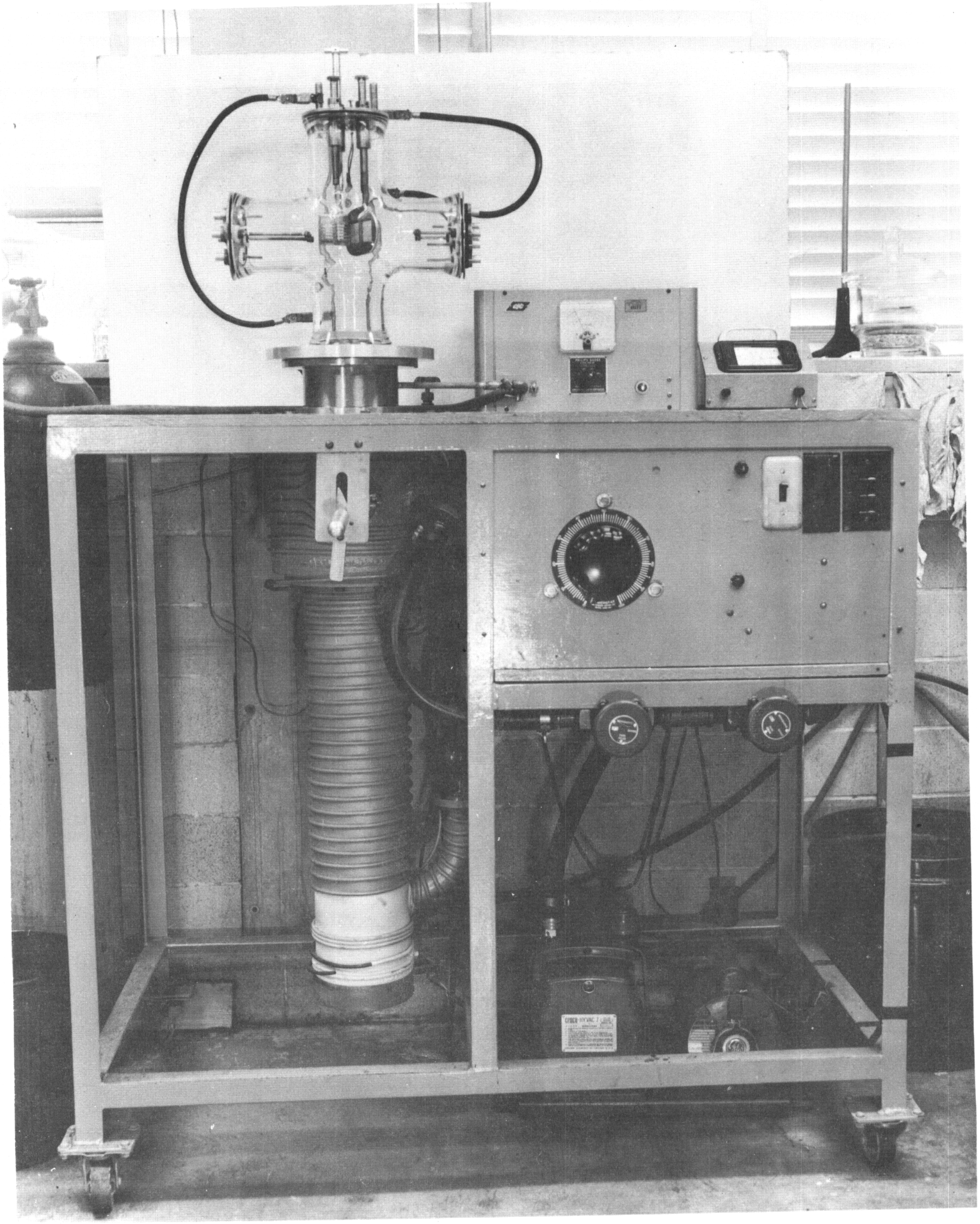
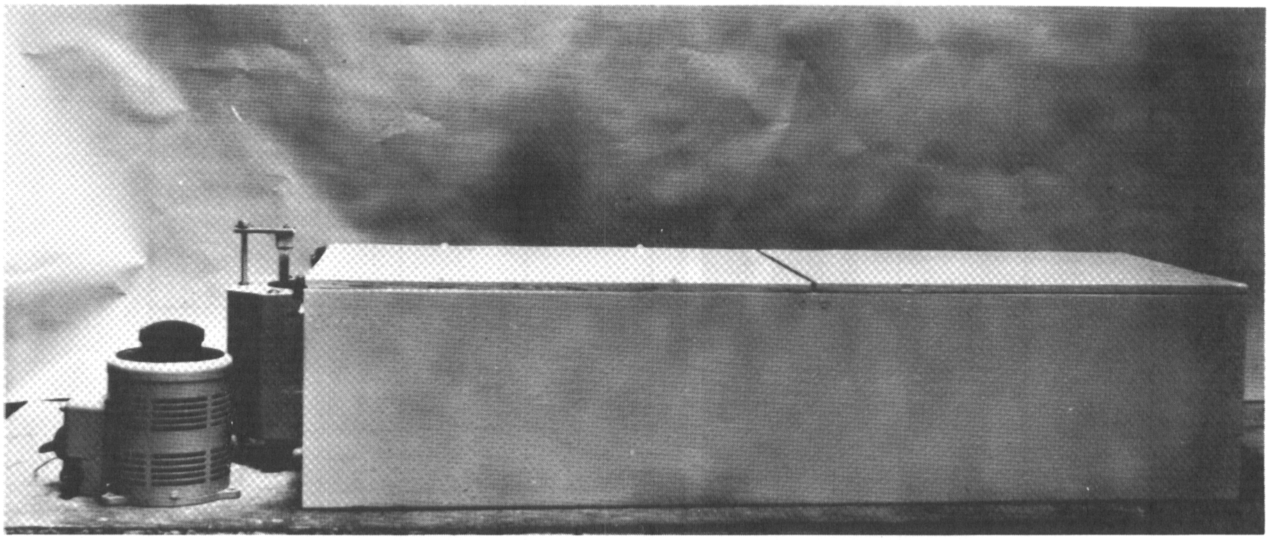
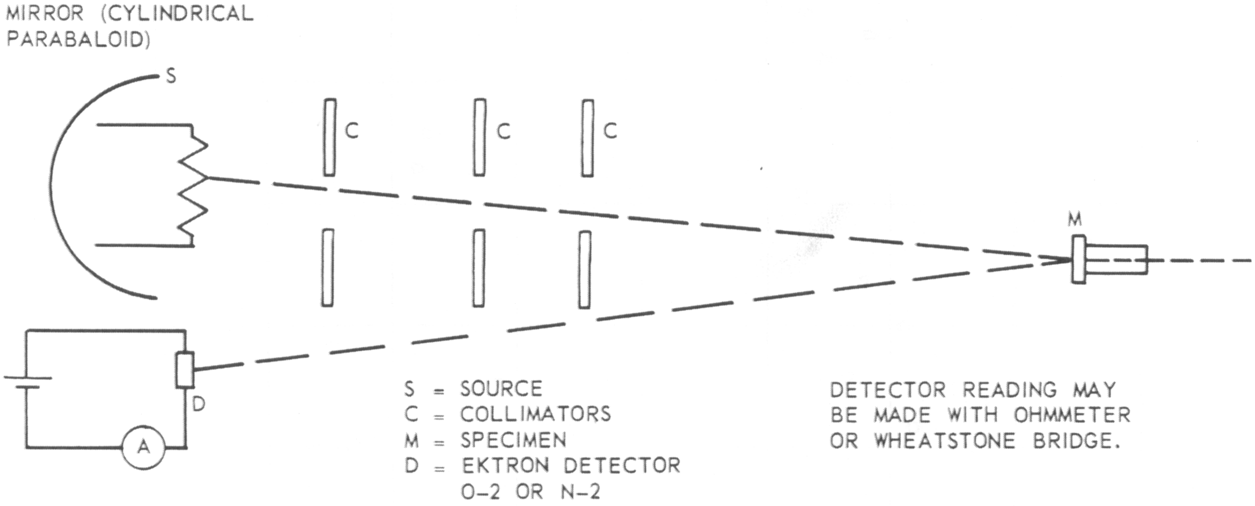


Figure 2 Vacuum System for Depositing Thin Reflective Films



A. Photometer



B. Optical Path

Figure 3 Infrared Comparative Photometer of Approximate Range 0.5 to 3 Microns.

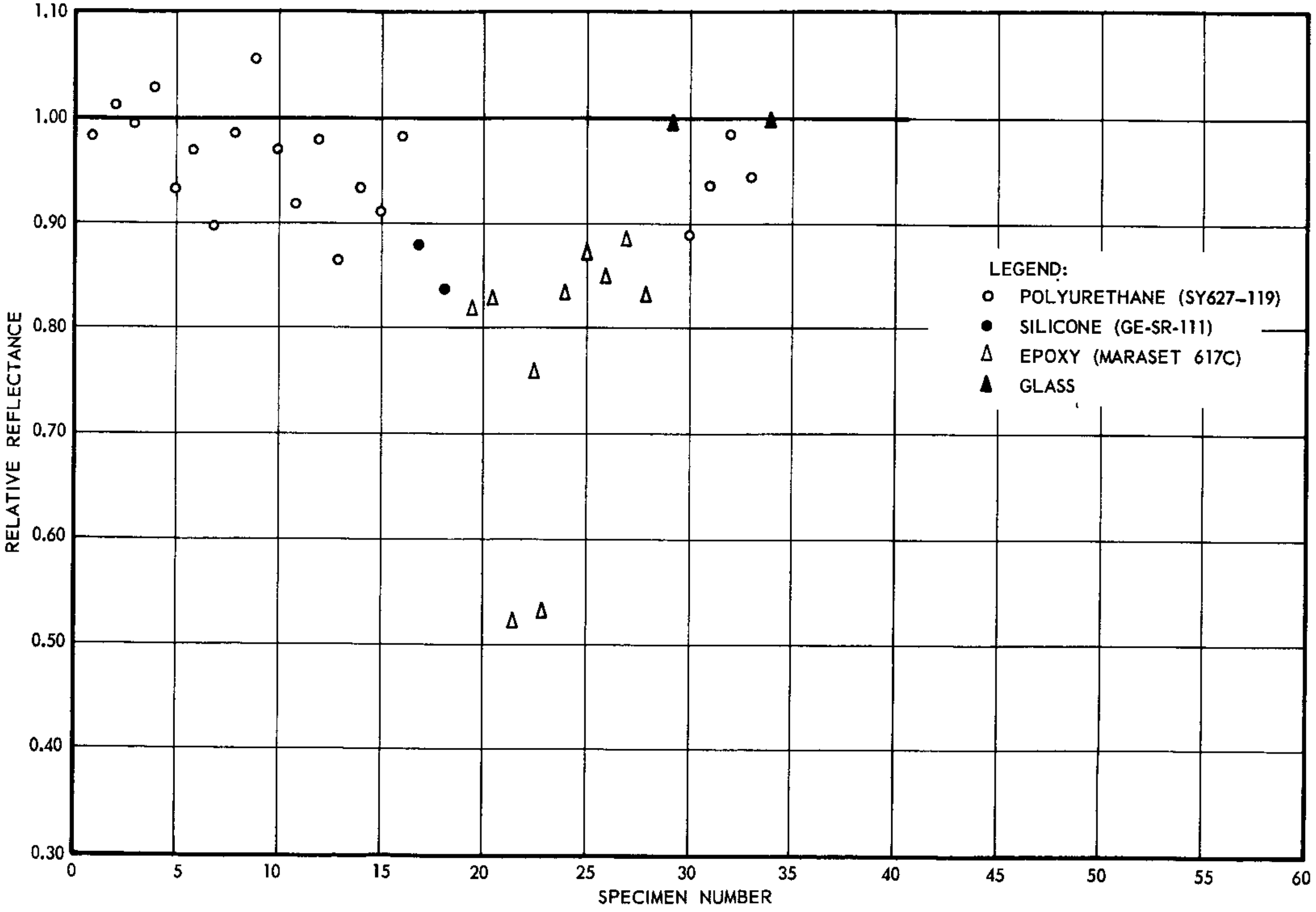


Figure 4 Plot of Reflectance Data for Gold Films Deposited on Plastic Coated Steel Substrates.

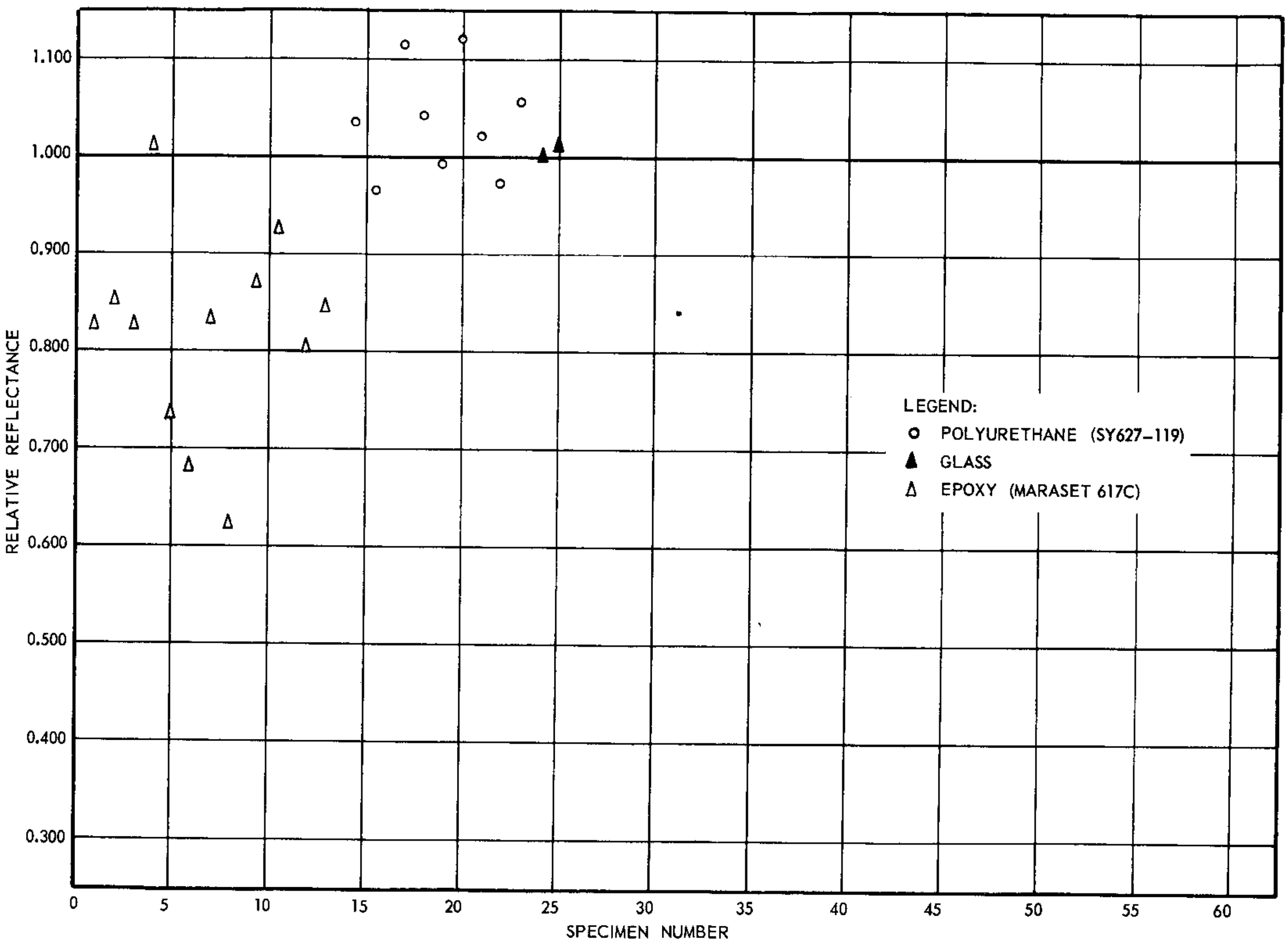


Figure 5 Plot of Reflectance Data for Silver Films Deposited on Plastic Coated Steel Substrates.

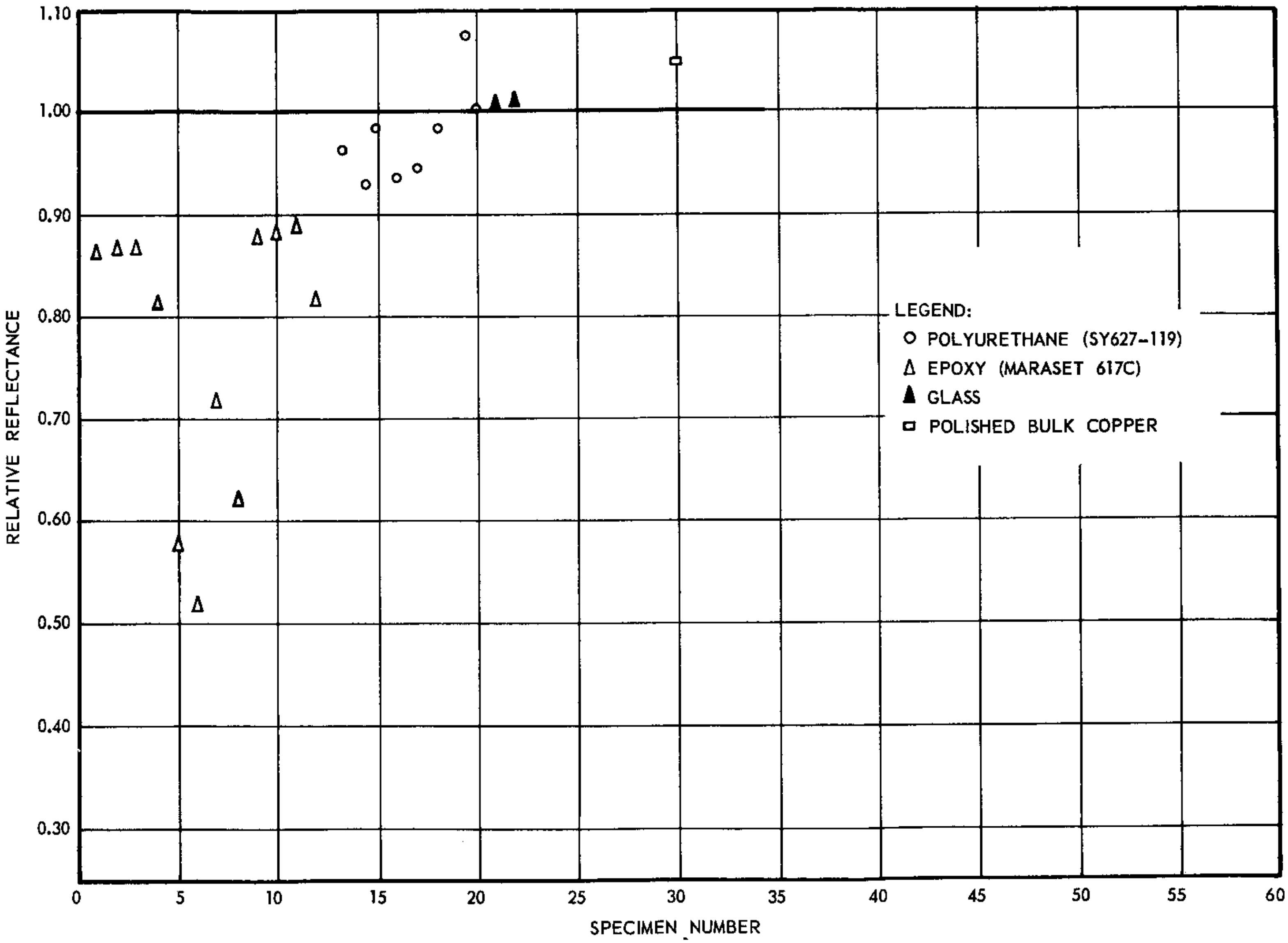


Figure 6 Plot of Reflectance Data for Copper Films Deposited on Plastic Coated Steel Substrates.

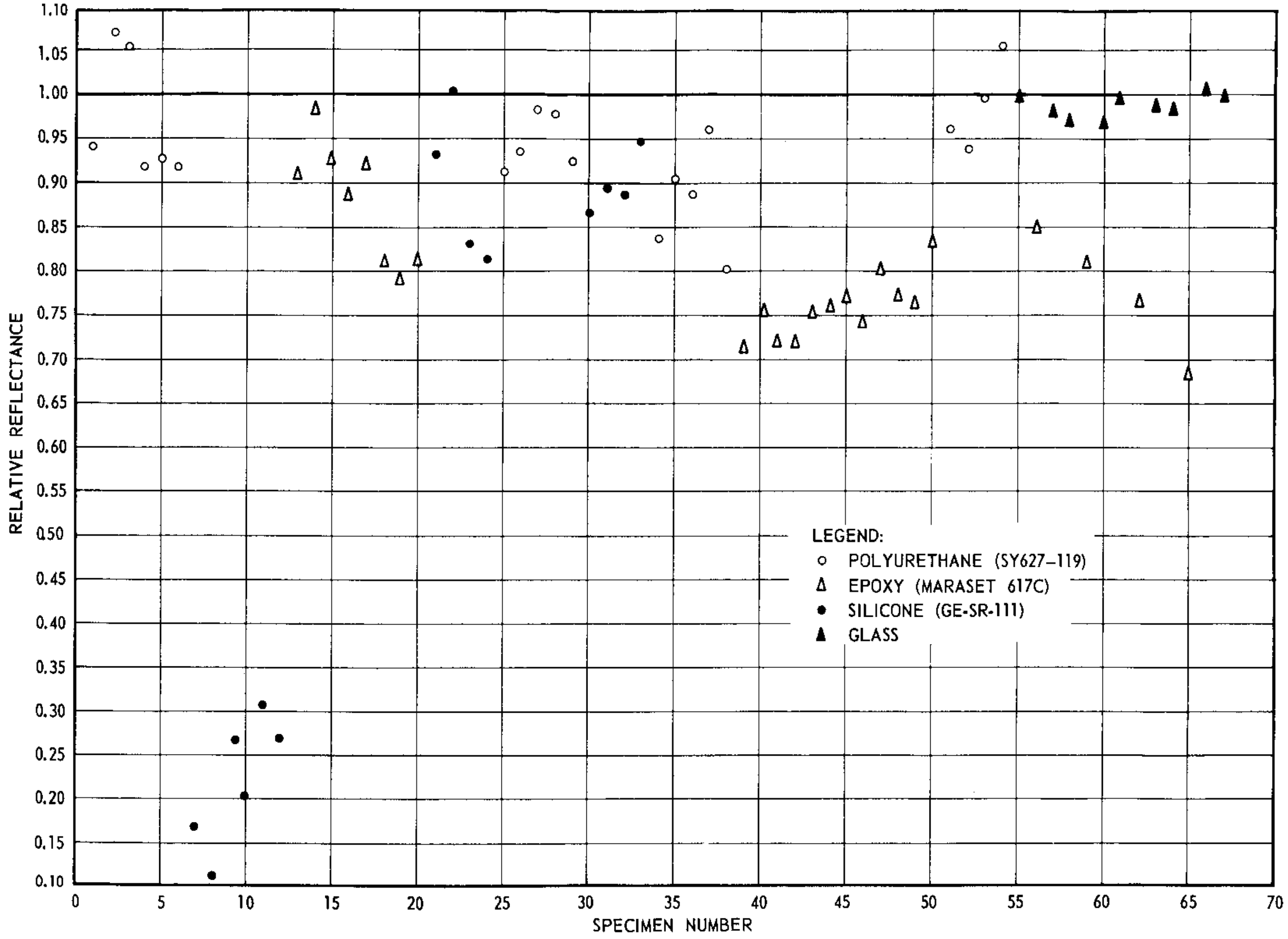


Figure 7 Plot of Reflectance Data for Aluminum Films Deposited on Plastic Coated Steel Substrates.

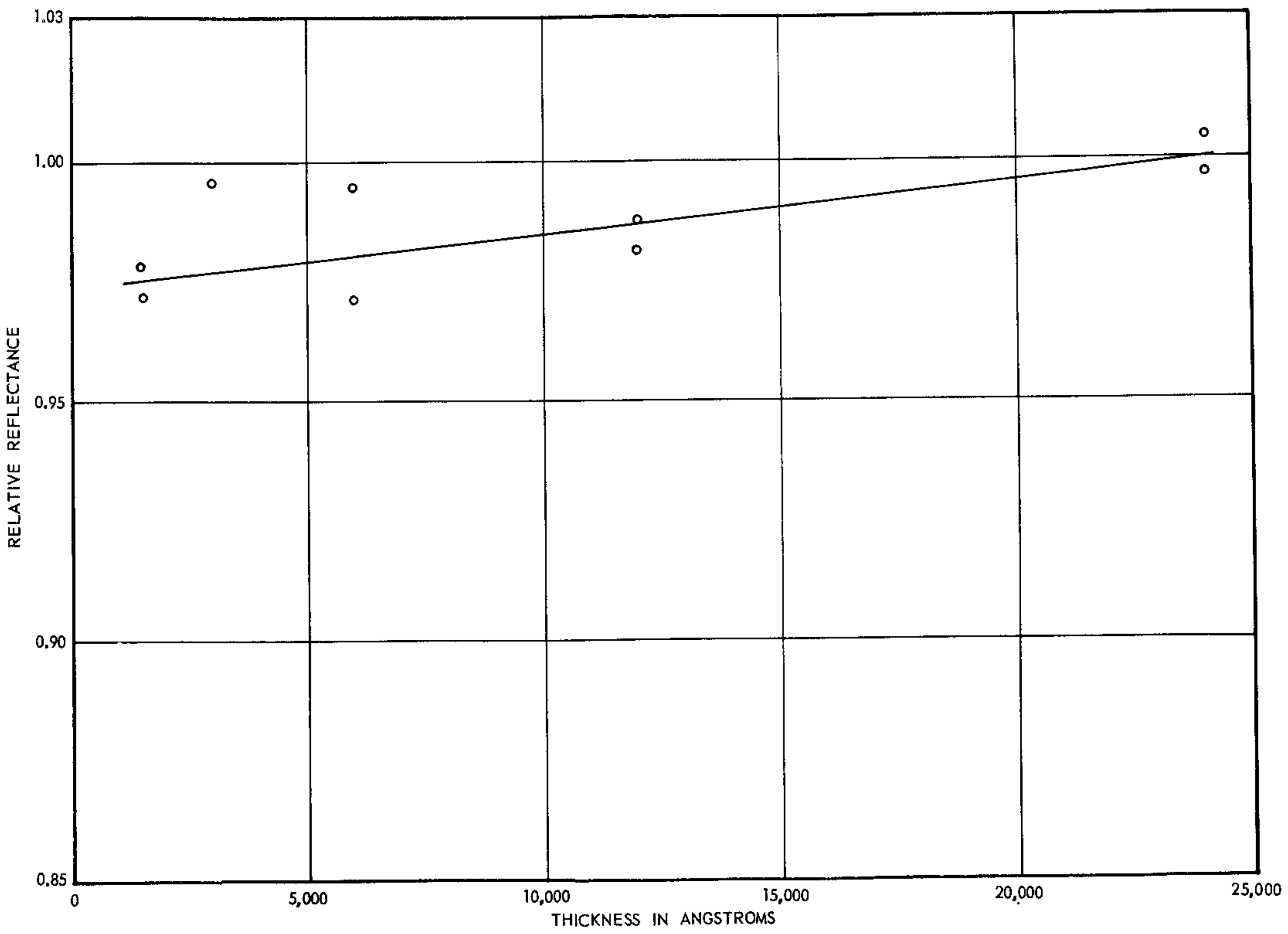


Figure 8 Data Showing Variation of Reflectance With Metal Film Thickness for Aluminum Films on Glass Substrates

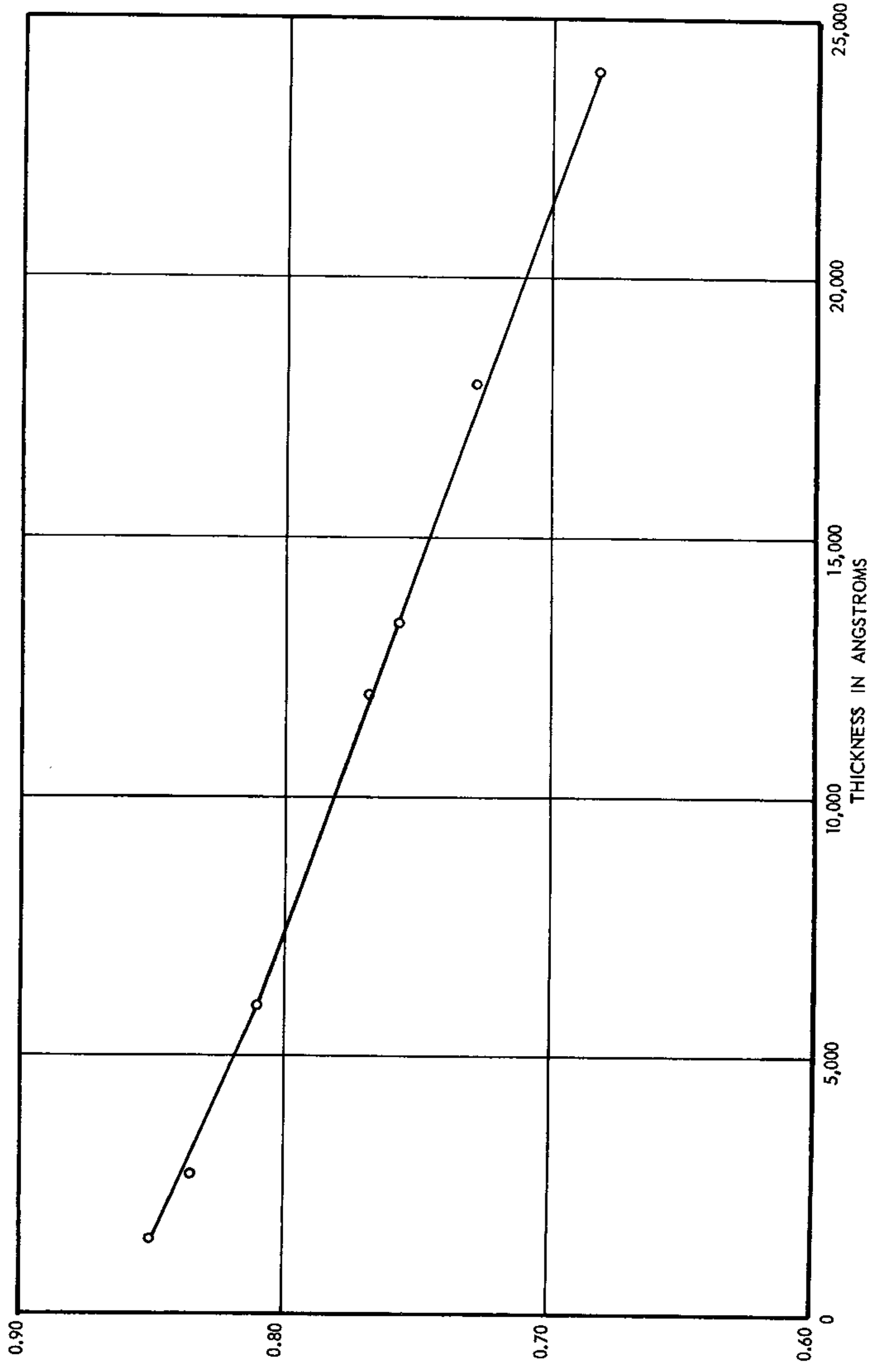


Figure 9 Data Showing Variation of Reflectance with Metal Film Thickness for Aluminum Films Deposited on Epoxy Coated Steel Substrates.

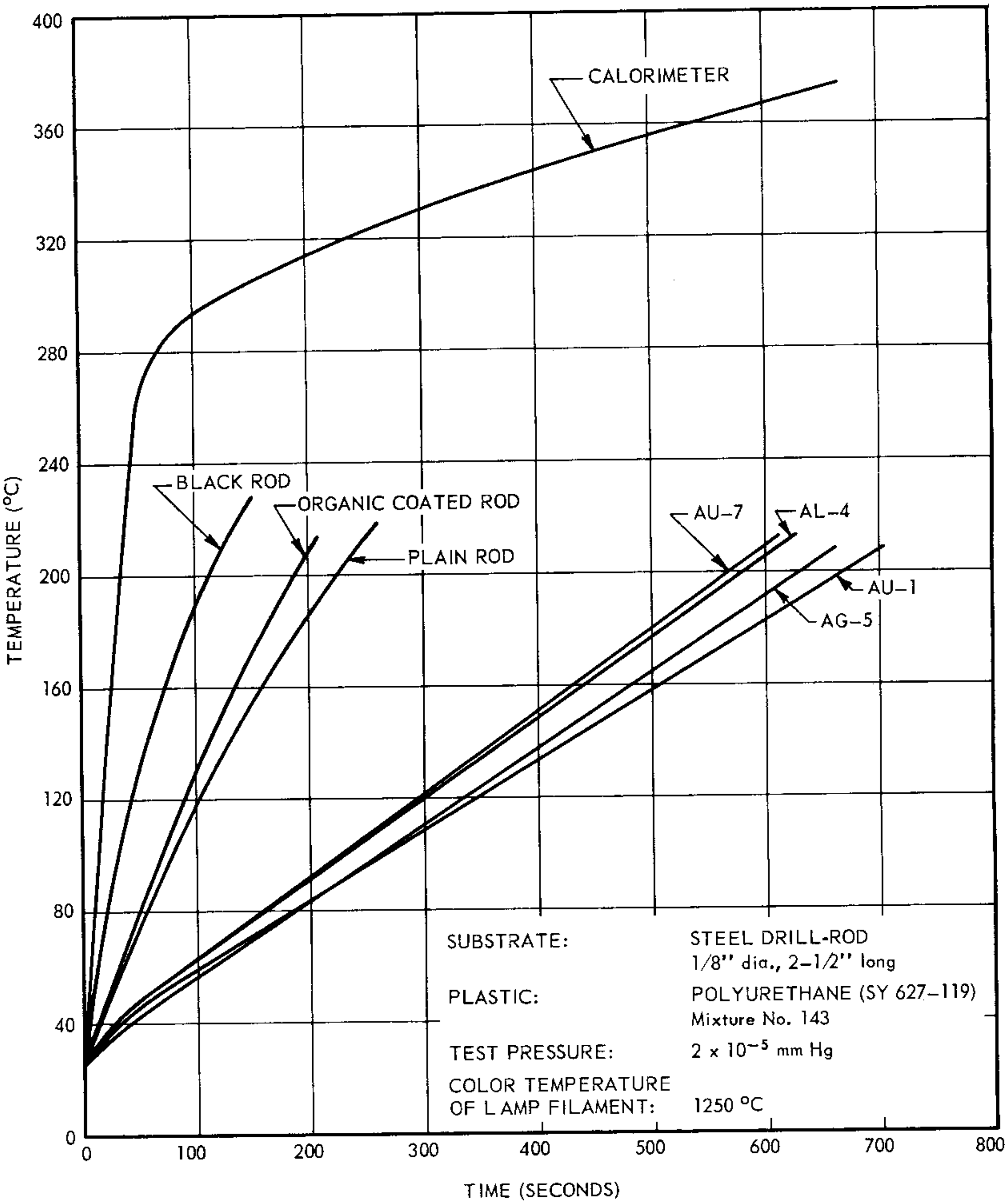


Figure 10 Typical Plots of Temperature Versus Time Data Made With the Radiation Calorimeter.

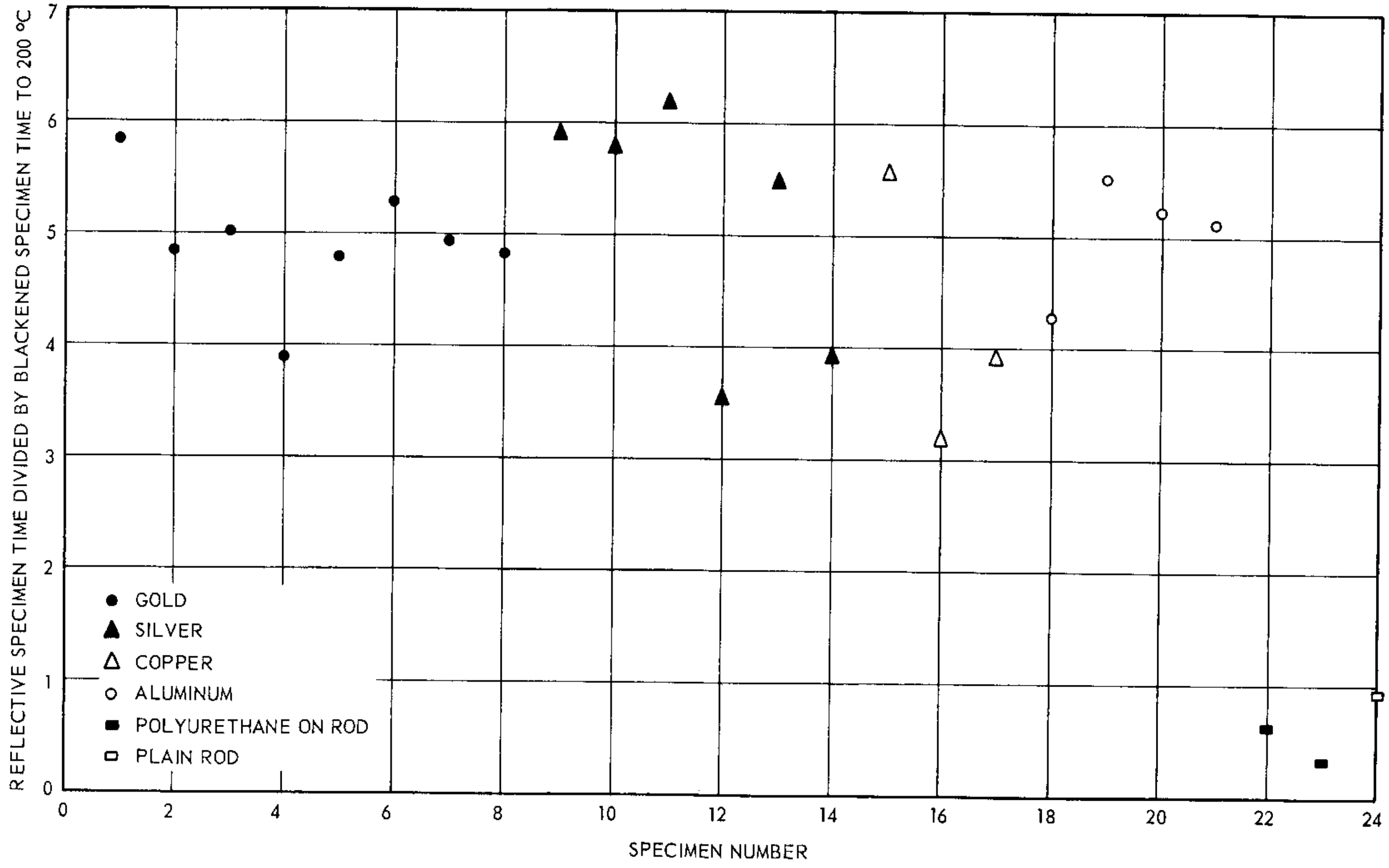


Figure 11 Ratio of Reflective Specimen Time to Blackened Specimen Time at 200°C

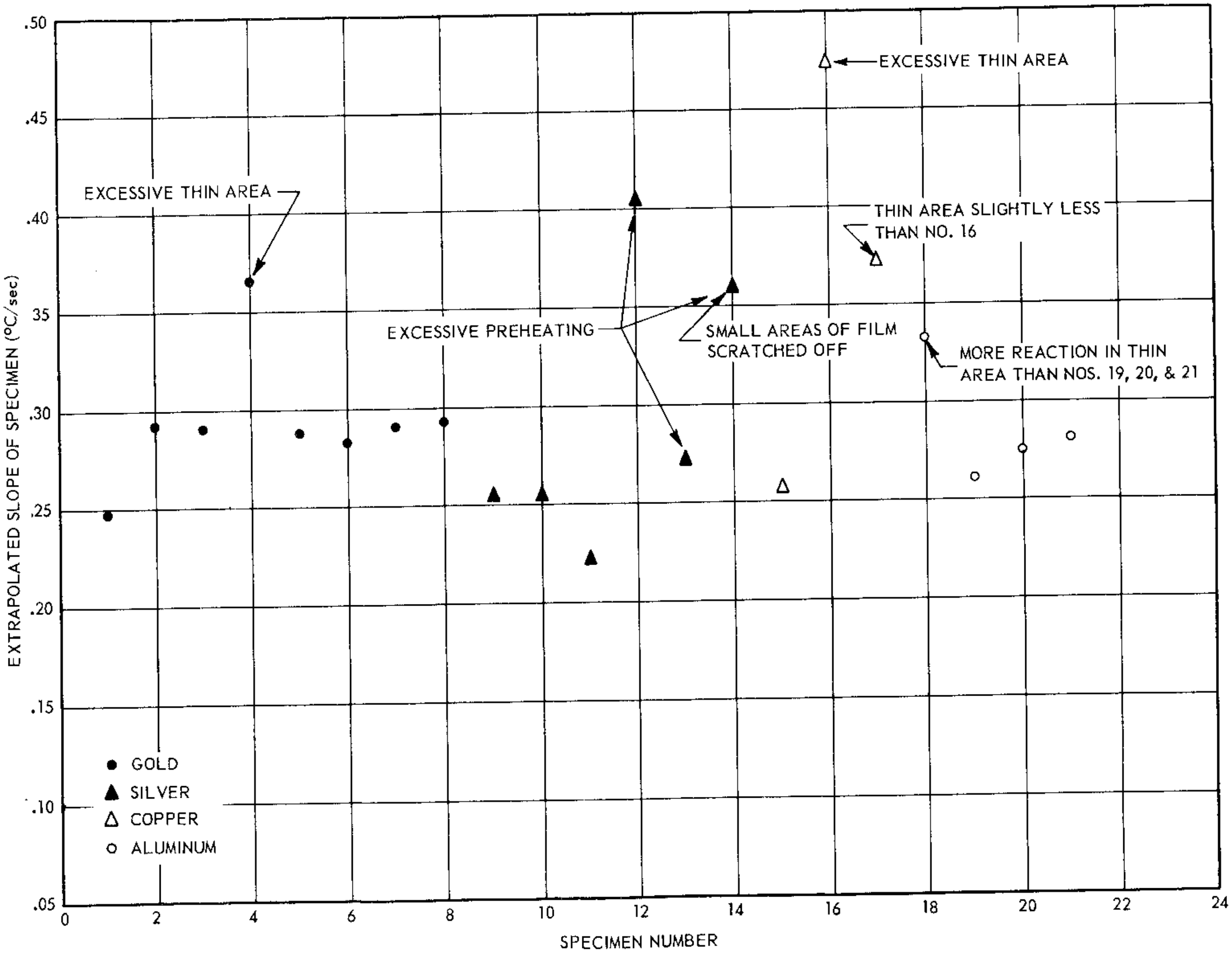


Figure 12 Time Rate of Change of Specimen Temperature for Variously Coated Specimens