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GLASS COATING OF ELECTRON-BOMBARDMENT ION-THRUSTER GRIDS BY ELECTRODEPOSITION

by

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and Dale E. Niesz

prepared for

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James Sovey, Project Manager

BATTELLE
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16. Abstract

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ABSTRACT

Parameters affecting the electrodeposition of Corning Code 1723 glass powder to yield controlled fired-coating thicknesses on ion thruster grids were investigated. A set of conditions was determined for producing a fired-coating thickness of 0.041 to 0.051 centimeter, reproducibly, on 30 centimeter-diameter grids. In processing subsequent to the deposition of the coating, defects developed which were in part attributed to the glass used, the preparation of the coated grid for firing, and the firing temperature. Some comparisons were made of the behavior, in processing, of Code 1723 and Code 7052 glass.

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by

John W. Lennon, John E. Reichelderfer,
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SUMMARY

The feasibility of applying glass coatings by the electrodeposition process to ion thruster grids having been established, it was desirable to define the parameters for reproducibly coating grids with a given coating thickness. Coating trials were conducted, using a water suspension of Corning Code 1723 glass powder, and the following variables were studied: particle size of glass, applied voltage, deposition current, deposition time, area of electrodes, and distance between electrodes. Additionally, the following glass-water suspension parameters were controlled or monitored: specific gravity, temperature, pH, and electrical conductivity.

Initial coating studies on molybdenum sheet or 7.5 centimeter-diameter grids enabled the establishment of conditions for coating 30 centimeter-diameter grids to yield a fired-coating thickness between 0.041 and 0.051 centimeter. These conditions were:

Glass particle size – 50 percent finer than 13 microns

Suspension characteristics

Temperature	20 C
Specific gravity	1.21
pH	10
Electrical conductivity	$3.8\text{-}4.6 \times 10^5$ ohm^{-1} cm^{-1}
Electrode spacing	12 centimeters
Applied voltage	450 dc
Deposition current	4.3-4.9 amp
Deposition time	50 sec

A crawling type of defect was present in certain areas of the fired coatings regardless of their thickness. Some areas were defect-free. Possible causes of the defect were fine particle size of glass, formation of microcracks in dry coatings imparted by grid stresses, and relatively low firing temperature which would retard healing of crawled areas due to high viscosity of the glass. Another type of defect was produced when small areas of coating were spalled from the front face of the grid during removal of the coating from the back face. The amount of spalling appeared to be proportional to the pressure applied in removing the coating from the back of the grid. These defects were not a problem in a previous study using Code 7052 glass. The difference was attributed mainly to differences in dry-coating strength, and glass fluidity at the temperatures to which the coatings were fired.

Providing defects can be eliminated, the electrodeposition process is suited for applying, in a single step, uniform coatings of appropriate thickness.

INTRODUCTION

The function of the glass-coated Kaufman thruster grid and the feasibility of preparing such a composite grid have been described previously. The basic requirement of the grid is that one face and the hole walls of the molybdenum grid be coated with an electrically insulating glass layer around 0.050 centimeter thick. Banks[1, 2]* has presented details of the grid system and of a process for fabricating a glass-coated grid. Following Banks' procedures except for the coating application method (which was by wet spraying), Lennon[3] has demonstrated the feasibility of the electrodeposition process for applying the glass coating. Advantages of the electrodeposition technique include the possibility of applying the total coating thickness in a single processing treatment and the uniformity of coating thickness.

In previous studies, several 7.5- and 30-centimeter-diameter grids were coated with Corning Code 7052 glass. Although some of the parameters associated with the successful electrodeposition were documented, others were not. In the present program, Corning Code 1723 glass was used because of its higher electrical resistivity. The object of this study was to characterize completely, the processing parameters resulting in the successful application of this glass.

EXPERIMENTAL PROGRAM

It is convenient to consider the application of the glass coating to the molybdenum-substrate grid in two steps. The first includes the deposition of the glass powder on the grid and the other preparations for firing; the second step is the firing process. The major effort of the program was on the deposition process, largely because of difficulties encountered because of the switch from the Code 7052 glass to the Code 1723 glass.

Electrodeposition of Coatings

Discussion of Process

The application of a glass coating to a metal substrate by the electrodeposition process consists of immersing two electrodes into a glass-water suspension and applying a d-c potential across them. Because the particles of glass assume a negative charge in a water suspension, they will move to the anode under the influence of the applied field. Under appropriate conditions, the glass particles adhere to the metal anode and a coating is formed. While the process is simple mechanically, many parameters can relate to the successful deposition of an adherent coating of a specific thickness. It is convenient to consider, separately, the electrodeposition process, the characteristics of the glass-water suspension, and the coating-process parameters.

*References are listed on page 28.

The Electrodeposition Process

The electrodeposition phenomenon requires that the glass particles be charged electrically, so that they move in the applied field. The common silicate glasses are made up of a framework of SiO_4 tetrahedra with alkali- and/or alkaline-earth ions relatively loosely held within this framework. A particle of glass in water presents a surface with a net negative charge due primarily to the predominance, at the surface, of the large negatively charged oxygen ions. Positive ions in the water, such as hydrogen ions or alkali- or akaline-earth ions (leached from the glass, or otherwise present) are adsorbed on the surface of the glass particle and partially neutralize the negative charge of the oxygen ions. So long as there remains a net negative charge, the glass particle will move to the anode. Further, the higher the net charge on the particle, the greater is its mobility under the influence of the applied field.

The ions in the water-glass suspension also act as current carriers. There are at least three sources of ions in the suspension – electrolytes added to the suspension, ions leached from the glass, and ions resulting from oxidation of the electrodes. Under influence of the applied field, these ions, as well as ions of the water itself, move to the appropriate electrodes, i.e., positive ions to the cathode and negative ions to the anode.

The electrochemical reactions occurring at the anode and cathode are not well established. For the system of interest here, previous observations indicate that the following reactions take place:

$$\text{At the cathode} - H^+ + e \rightarrow 1/2\ H_2\ (\text{gas})$$

$$\text{At the anode} - Mo \rightarrow Mo^{4+} + 4e$$

$$Mo^{4+} + 2\ O^{2-} \rightarrow MoO_2.$$

It is thought that an interaction occurs between the Mo^{4+} ions formed at the anode and the depositing negatively charged glass particles. This interaction is instrumental in developing adherence of the glass coating to the molybdenum substrate, and in deflocculating and densifying the glass coating itself.

Experience has shown that the successful electrodeposition of a glass coating involves, not only the deposition of negatively charged glass particles on the anode, but in addition, the development of adherence of the deposited coating to the anode. A properly coated anode can be removed from the glass-water suspension and immersed in water to wash away extraneous glass particles – such as those which settle on the piece because of gravitational forces – without affecting the electrodeposited coating.

The Glass-Water Suspension

In the present study, the glass was the only solid addition to the water. No suspending agents, such as clay, could be tolerated because they would contribute to the formation of bubbles in the glass during the firing process, and thus lower the electrical resistivity of the coating. While this system is relatively simple, a number of characteristics must be considered in studying the electrodeposition process. These include

Glass-to-water ratio

Glass particle size

Electrical charge on the glass

Electrical conductivity of suspension

pH of suspension

Temperature of suspension.

With the exception of the electrical charge on the glass particles, these characteristics are readily measurable.

Coating-Process Parameters

When a voltage is applied across two electrodes in the glass-water suspension, an electric current results. To the extent that the system is an electrical circuit, the following parameters are measurable and can be varied:

Applied voltage

Current

Distance between electrodes

Area of electrodes

Time of voltage application.

Coating Trials

The ultimate objective of the various studies was to determine the parameters involved in coating 30-centimeter-diameter grids. It was planned to study most of these parameters using 7.5-centimeter-diameter grids. However, prior to this, some studies were conducted using molybdenum sheet. The simpler form of the sheet (relative to that of the grid with its holes and hole walls) permitted more convenient measurement and control of area being coated.

The electrodeposition trials, when coating the sheet or 7.5-centimeter grids, were conducted using the suspension tank shown in Figure 1. A suspension based on 11 kilograms of glass powder was used. Electrical conductivity of the glass-water suspension was measured using the conductivity bridge shown in Figure 2. The bridge was calibrated, using KCl solutions of known concentration, to permit determining the specific conductance of the electrodeposition suspension. Other facilities used included a pH meter, d-c power source, appropriate volt meters and ammeters, and a timer.

Throughout the program unless noted otherwise, glass-distilled water suspensions of 1.2 specific gravity were used. The distilled water had a specific conductance of 1.5×10^{-12} ohm^{-1} cm^{-1}. The minus 200-mesh Code 1723 glass was calcined at 537 C for 1 hour to burn out any contaminating organic material before using in coating trials. Suspension temperatures were monitored and found not to vary from room temperature, i.e., 20 C.

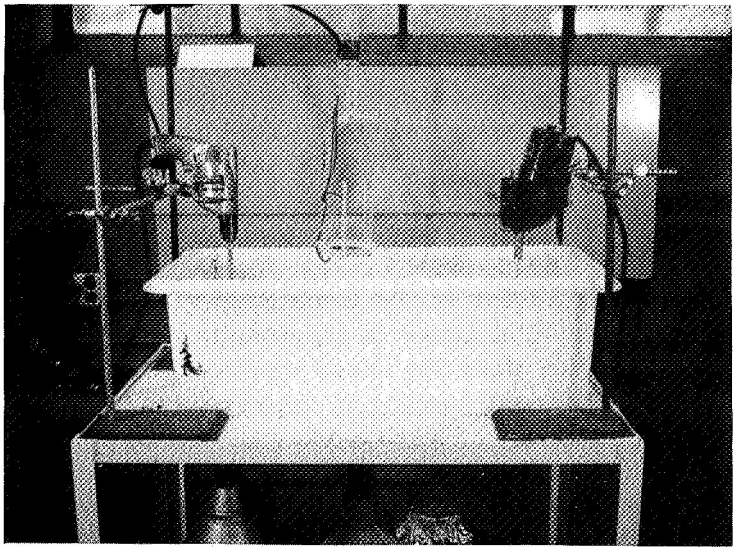


FIGURE 1. ELECTRODEPOSITION TANK AND STIRRERS FOR COATING TRIALS OF SHEETS AND GRIDS

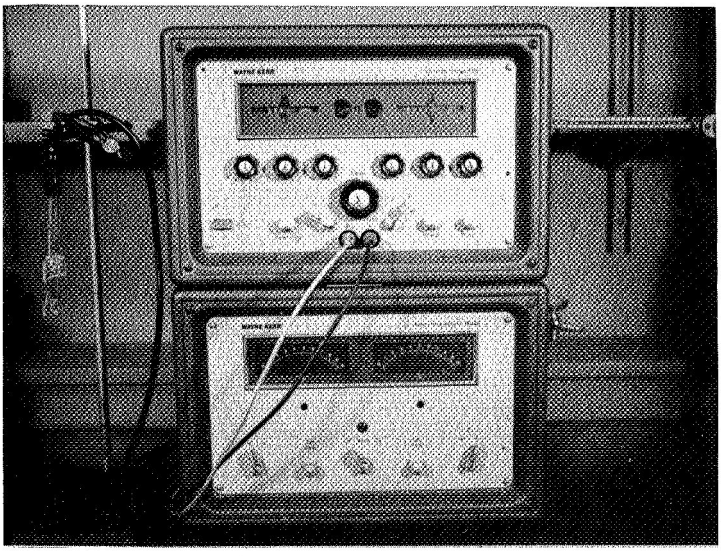


FIGURE 2. CONDUCTIVITY BRIDGE FOR MEASURING AND CONTROLLING CONDUCTIVITY OF GLASS-WATER SUSPENSIONS

Initially, the molybdenum metal substrates were cleaned in various organic solvents, a NaOH solution, and water. However, these agents did not satisfactorily clean a piece once it had been subjected to the electrodeposition coating process. To remove the oxide film formed during coating (so that the piece could be reused), it was found necessary and satisfactory to scour the piece with a mixture of 200-mesh quartz and an alkaline commercial detergent in a water slurry. Thorough rinsing in water followed.

Coating Molybdenum Sheet

An initial trial with a suspension of the Code 1723 glass was made under conditions paralleling those using the Code 7052 glass in a previous program.[3] For this first trial a 7.5-centimeter grid rather than the sheet was used. With the Code 7052 glass, it was possible to deposit an adherent coating of appropriate thickness by applying potential of 100 volts for 60 seconds. These same conditions resulted in virtually no coating being deposited using the suspension made with the Code 1723 glass. It was found that when the applied voltage was increased to 300 to 350 volts, coatings of appropriate thickness could be deposited, but adherence of the coating to the substrate metal was poor. Also, the blue molybdenum oxide discoloration appearing as the coating dried indicated that the initial suspension characteristics of the Code 1723 glass-water system were similar to a deteriorated Code 7052 glass-water system, i.e., one that was too electrically conductive after numerous deposition trials. Because the electrodeposition characteristics of these two glasses differed, some of the characteristics[4] of the glasses themselves are presented below for comparison.

Composition

Oxide, percent	Code 7052	Code 1723
SiO_2	65	57
Al_2O_3	7	15
B_2O_3	18	5
Li_2O	1	--
Na_2O	2	--
K_2O	3	--
BaO	3	6
MgO	--	7
CaO	--	10
F	1	--

Other

Thermal Expansion, 10^{-6}/C (0-300 C)	4.6	4.6
Temperature at Which Viscosity is 10^4 Poise, C	1115	1175
Log Volume Resistivity at 350 C, ohm-cm	7.4	11.3

It was of interest also to compare characteristics of water suspensions of Code 7052 and Code 1723 glass powders. Their pH and conductivity are shown below:

Property	Code 7052	Code 1723
pH	8.4	10.3
Electrical Conductivity, $ohm^{-1}\ cm^{-1}$		
5 Min After Mixing	1.8×10^{-4}	9.2×10^{-5}
75 Hr After Mixing	5.7×10^{-4}	1.2×10^{-4}

On the basis of the chemical compositions of the two glasses, the Code 7052 can be described as an alkali (Li, Na, K) glass, and the Code 1723 glass as an alkaline-earth (Mg, Ca, Ba) glass. The lower pH of the Code 7052 glass-water suspension is attributed to the higher B_2O_3 content and lower content of basic oxides of this glass relative to those of the Code 1723 glass. The former component, when leached from the glass, results in a boric acid addition to the water.

The major difference in the electrodeposition behavior of the two glasses was thought to be associated with the alkali versus alkaline-earth contents of the glasses. These components exist in the glass structure as relatively loosely held and readily leachable ions. They can exist in the glass interior, adsorbed on the surface of the glass, and in the suspension water. The alkali ions in the Code 7052 glass are monovalent (Li^{1+}, Na^{1+}, K^{1+}). The alkaline-earth ions in the Code 1723 glass are divalent (Mg^{2+}, Ca^{2+}, Ba^{2+}). It is thought that the positive divalent alkaline-earth ions more effectively neutralize the negative charge at the surface of the glass particle. Not only do the alkaline-earth ions carry a 2+ charge, but they are smaller than their counterpart alkali ions in that the alkali ions are considered to carry a water layer which prevents their packing as densely as the alkaline-earth ions over the surface of the glass particles.

This situation would result in the Code 1723 glass particle being less negatively charged than an equivalent Code 7052 glass particle, and would explain the need for a 300-volt potential to deposit the Code 1723 glass in contrast to the 100-volt potential used for the Code 7052 glass. Also, the 300-volt potential would be expected to develop a higher ionic current in the water suspension of the Code 1723 glass, even though the conductivity of this glass-water suspension was lower than that of the suspension of the Code 7052 glass. Though the ratio of ionic current to glass-particle current was not known, it was thought that for the Code 1723 glass-water suspension used in these initial trials, it was too high for satisfactory deposition and adherence.

With the aim of improving the electrodeposition of Code 1723 glass, several suspensions were prepared, which after mixing had conductivities of around 1.1×10^{-1} $ohm^{-1}\ cm^{-1}$. After a 24-hour period the water above the settled glass was decanted and replaced with distilled water. The suspensions then had conductivities of around $4 \times 10^{-5}\ ohm^{-1}\ cm^{-1}$.

To these lower conductivity suspensions were added, individually, distilled water solutions of KOH, NaOH, LiOH, or H_3BO_3. The hydroxides were added to cause monovalent ions to adsorb on the glass particles – replacing divalent ions. The H_3BO_3

(boric acid) was added to determine whether a lower pH suspension, similar to that of Code 7052 glass suspensions, would influence deposition.

Regardless of the addition made to the suspensions, coatings applied to a 5 x 5-centimeter sheet at a 350-volt potential for 15 seconds had borderline adherence. At a 100-volt potential for 60 seconds, the adherence appeared to be good. The suspension conductivity for depositing the more adherent coatings was centered around 5×10^{-5} $ohm^{-1}cm^{-1}$.

Next, a study was undertaken of the coating thickness and quality as affected by the deposition parameters of applied voltage, electrode spacing, and electrode area. A Code 1723 glass-water suspension containing 11 kilograms of glass was used. Its specific gravity was 1.2.

The suspension was prepared for coating trials by mixing the glass and water for 5 minutes, allowing the glass to settle for about 45 hours, decanting the water, and replacing it with distilled water. At this point the conductivity of the suspension was 3×10^{-5} ohm^{-1} cm^{-1}. About 8 ml of a 10 percent aqueous KOH solution was added to the suspension to raise the conductivity to 7.6×10^{-5} ohm^{-1} cm^{-1}. The pH of the suspension decreased gradually from a value of 10.3, at the start, to 9.8.

The specimens coated were square molybdenum sheets with areas of 4, 16, or 64 cm^2. These specimens were covered with electrical tape on one face to prevent deposition on this face. Stainless steel specimens of matching areas were used as cathodes; they were spaced opposite the anodes at distances of 2.54, 5.08, or 10.16 centimeters during the deposition trials. Applied voltages were 50, 100, or 200 volts, and deposition time was always 60 seconds. The specimens were coated with their plane in the vertical position and were rinsed in distilled water after removal from the suspension while maintaining the applied voltage.

After the specimens were dried, coating thickness was measured and the specimens were photographed. The current during deposition, current density (anode area/current), and coating thickness for the various trials are shown in Table 1. The corresponding photographs are shown in Figures 3, 4, and 5.

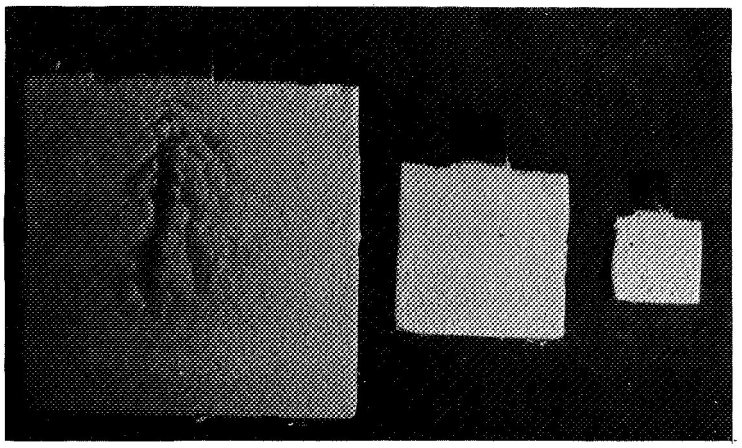
In attempting to derive a relationship between various parameters and coating thickness or uniformity, it should be noted that the value for deposition current in Table 1 is not so precise nor meaningful as are the values for electrode spacing or area, or deposition voltage or time. At the instant the voltage is applied, the current-indicating needle moves from zero to some maximum value, then immediately starts to indicate decreasing current. As an example of an extreme case, when the two 64-cm^2 electrodes (spaced 2.54 centimeters apart) were coated using an applied voltage of 200 volts, the maximum current attained immediately after applying the voltage was the 640-ma value shown in Table 1. At the end of the 60-second coating time, the current was 350 ma. Some inaccuracy occurs in reading the maximum current because no sooner does the needle indicate this value than it starts to return to lower values. In the case cited, a relatively thick coating was building up on the anode and the decrease in current with time is attributed to the deposition of increasingly thicker layers of the electrically insulating glass.

The results presented in Table 1 indicate that deposition current, current density, and coating thickness are directly proportional to applied voltage. This assumes that

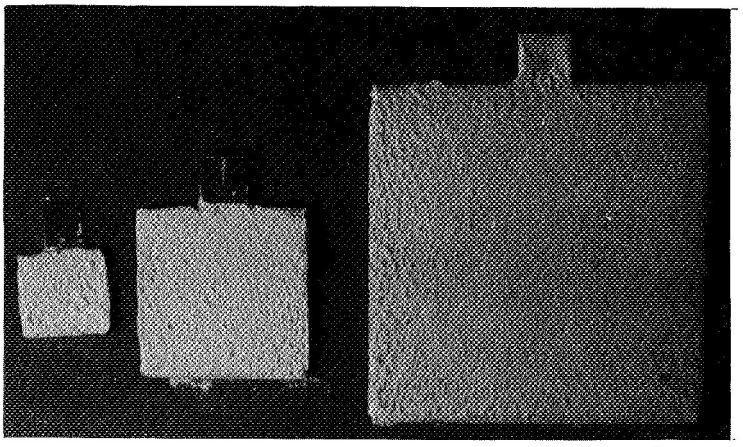
TABLE 1. EFFECTS OF APPLIED VOLTAGE, ELECTRODE SPACING, AND ELECTRODE AREA ON DEPOSITION CURRENT AND COATING THICKNESS

	Electrode Spacing, cm								
	2.54			5.08			10.16		
	Electrode Area, cm^2			Electrode Area, cm^2			Electrode Area, cm^2		
	4	16	64	4	16	64	4	16	64
Applied Voltage, 50 volts									
Current, ma	30	55	150	25	50	110	20	50	85
Current Density, ma/cm^2	7.5	3.4	2.3	6.3	3.1	1.7	5.0	3.1	1.3
Coating Thickness, cm	0.053	0.048	0.043[a]	0.056	0.048[a]	0.051[a]	0.046	0.046[a]	0.056[a]
Applied Voltage, 100 volts									
Current, ma	40	120	300	45	100	160	40	80	160
Current Density, ma/cm^2	10.0	7.5	4.7	11.3	6.3	2.5	10.0	5.0	2.5
Coating Thickness, cm	0.097	0.089	0.089	0.086	0.071	0.071[a]	0.086	0.064	0.056[a]
Applied Voltage, 200 volts									
Current, ma	90	220	640	80	190	420	75	170	310
Current Density, ma/cm^2	22.5	13.8	10.0	20.0	11.9	6.6	118.8	10.6	4.8
Coating Thickness, cm	0.196	0.165	0.152	0.175	0.130	0.104	0.170	0.117	0.081

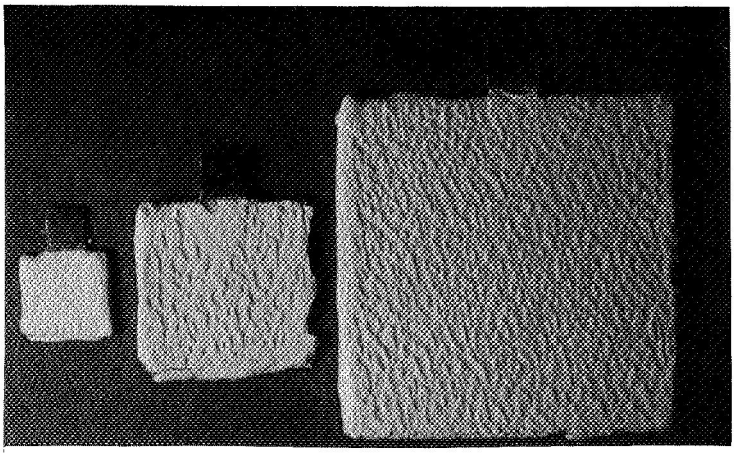
(a) Thickness near edge; center area thinner or uncoated (see photographs).



Applied at 50 volts

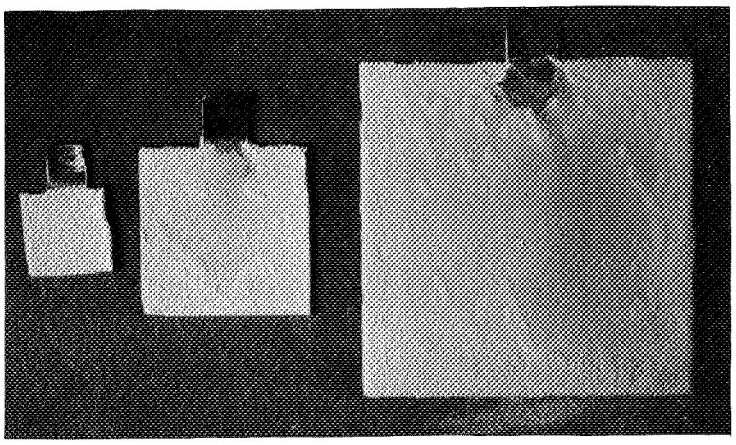


Applied at 100 volts

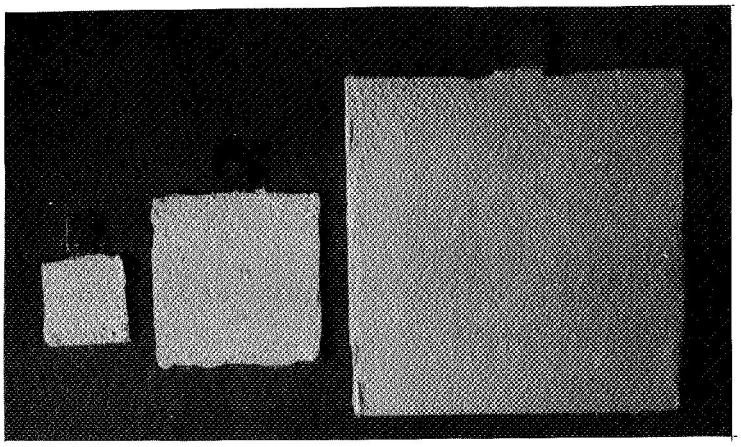


Applied at 200 volts

FIGURE 3. COATINGS APPLIED ON 4-, 16-, AND 64-CM^2 PLATES SPACED 2.54 CENTIMETERS APART



Applied at 50 volts



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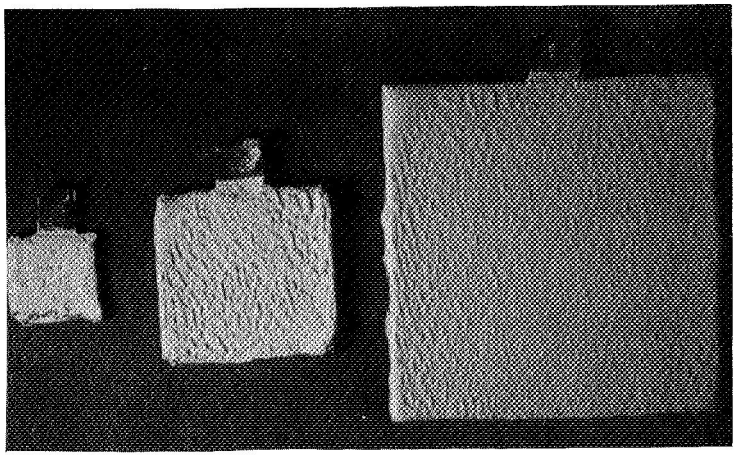
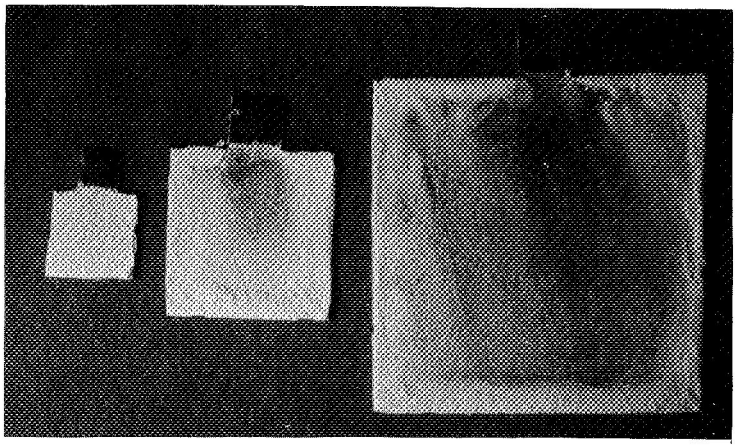
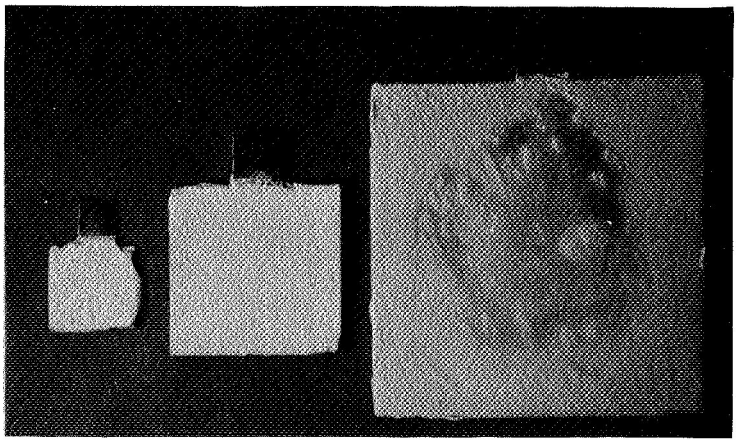


Applied at 200 volts

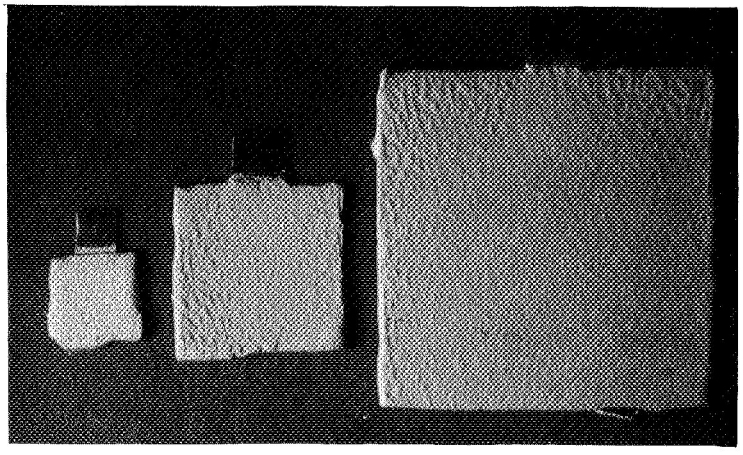
FIGURE 4. COATINGS APPLIED ON 4-, 16-, AND 64-CM^2 PLATES SPACED 5.08 CENTIMETERS APART



Applied at 50 volts



Applied at 100 volts



Applied at 200 volts

FIGURE 5. COATINGS APPLIED ON 4-, 16-, AND 64-CM^2 PLATES SPACED 10.16 CENTIMETERS APART

an adherent coating of uniform thickness is produced. Coating thickness decreased with increase in spacing between electrodes, but not proportionately. Rates of coating-thickness decrease were greater at higher applied voltages and at higher electrode-area-to-spacing ratios. A minimum curent density – in this study, around 3 to 4 ma/cm^2 – was required to achieve adherent, uniform coatings.

Additional coating trials were made using the suspension described above but with a conductivity of 7.7×10^{-5} $ohm^{-1}cm^{-1}$ (only slightly higher than previously). The 64-cm^2 electrodes spaced 10.16 cm apart were used in these coating trials in which deposition time was varied. Applied voltage was 200 volts. The deposition time-coating thickness relationship was as follows:

Time, seconds	Coating Thickness, cm
30	0.056
60	0.091
90	0.137

Another trial was made using a 64-cm^2 cathode (stainless steel) and a 16-cm^2 anode (molybdenum). The coating was applied at 200 volts for 60 seconds at an electrode spacing of 10.16 centimeters. A comparison of current and coating thickness for this trial and for the trials (Table 1) using both 64-cm^2 electrodes or both 16-cm^2 electrodes follows:

Electrode Area, cm^2			Current Density,	Coating Thickness,
Cathode	Anode	Current, ma	ma/cm^2	cm
64	64	310	4.8	0.081
64	16	220	13.7	0.160
16	16	170	10.6	0.117

It is noteworthy that the current density at the anode can be increased by increasing the cathode area, rather than by increasing applied voltage or decreasing electrode spacing.

Using the same suspension after decanting, replacing with distilled water, and adjusting the conductivity to a value of 4.3×10^{-5} ohm^{-1} cm^{-1}, a trial was made to study the effect of the lower conductance. The 64-cm^2 electrodes were spaced 10.16 centimeters apart and coated for 60 seconds at an applied voltage of 200 volts. The results of this trial are compared with those of the same trial (Table 1) using the suspension with a conductance of 7.6×10^{-5} ohm^{-1} cm^{-1}.

Suspension Conductance, ohm^{-1} cm-1	Current, ma	Current Density, ma/cm^2	Coating Thickness, cm
7.6×10^{-5}	310	4.8	0.081
4.3×10^{-5}	200	3.1	0.15 near edge and 0.069 near center

Using this same suspension (conductivity – 4.3×10^{-5} $ohm^{-1}cm^{-1}$), a coating trial was made in which the two 64-cm^2 electrodes were spaced 25.40 centimeters

apart. At an applied voltage of 200 volts, the current was 130 ma, and a poorly adherent coating on the one side resulted. Next, a 64-cm^2 anode was coated on both sides while positioned midway between two 64-cm^2 electrodes; the electrodes were 27.94 centimeters apart. At an applied voltage of 200 volts, the current was 280 ma, and the coating adhered poorly. That the coatings did not adhere was not unexpected – the trial was made to confirm that similar current densities at the anode face(s) would result with the two configurations. This was confirmed; in the first case, the current density was 2.0 ma/cm^2 and in the second it was 2.2 ma/cm^2.

Coating 7.5-Centimeter-Diameter Grids

The grids to be coated were 7.5 centimeters in diameter, 0.038 centimeter thick, with 0.190-centimeter-diameter holes spaced 0.254 centimeters center-to-center. The holes in these grids were formed by chemical etching. The grids were mounted in the stainless steel ring used in a previous program and were positioned 6.4 centimeters from a stainless steel screen cathode. This assembly was immersed in the glass-water suspension with the grid in the horizontal plane, and above the cathode.

In the first coating studies made, the glass-water suspension used for coating was the same as that used to coat sheets. Whereas it was possible to coat the molybdenum sheet described previously, no conditions of voltage, current density, electrode spacing, etc., were found for depositing adherent coatings on the grids. Although the grids coated satisfactorily, immediately after they were rinsed in clear water the coating on the bottom side of the grid began to drain and pull the coating from the top surface. The inadequate bonding of the glass to the molybdenum was attributed, basically, to insufficient electrical charge on the glass particles.

In the attempt to increase the charge on the glass, two treatments of the as-received Code 1723 glass were studied. One was an ion-exchange treatment, and the other was wet ball milling.

In the ion-exchange treatment, the glass particles were heated in molten KNO_3. The K^+ ions diffuse into the surface of the glass to replace the alkaline-earth ions in the Code 1723 glass. In the present case, the glass-molten KNO_3 was heated for 1 hour at 593 C and briefly to 705 C. The treatment at 705 C was stopped when the KNO_3 began to boil violently. After cooling to room temperature, the batch was leached repeatedly with distilled water to remove the soluble KNO_3. When the glass-water suspension reached a conductivity of 4×10^{-5} ohm^{-1} cm^{-1}, coating trials were made.

Coatings applied at 100 volts on the 5-cm^2 plates were 0.14 cm thick, which is about four times as thick as coatings applied from previously described suspensions. Using a suspension with a conductivity of 6×10^{-5} ohm^{-1} cm^{-1} (attained by adding KOH solution), the coating thickness was 0.102 centimeter, or about twice that obtained using the suspensions described previously. In all of these trials, the deposition time was 60 seconds.

Another difference was noted in the coating behavior of the "ion-exchanged" glass-water suspension and the other suspensions described. Very thin, uniform coatings could be applied from the ion-exchanged glass suspension, whereas coatings deposited from the other suspensions started to build up, first at the corners and edges of the plate, then in the center. In the latter case, the coating ultimately attained uniformity in thickness across the surface, but in the early stage the coating near the edges was

thicker. Using the ion-exchanged glass, the coating appeared to build up uniformity over the surface from the start of deposition. This was confirmed in a trial in which the coating plate was withdrawn and examined after 5 seconds' deposition time.

While it appeared that the ion-exchange treatment significantly benefitted the deposition characteristics of the Code 1723 glass, it adds an involved processing operation in the preparation of the glass suspension. For this reason, and because of the success with the ball-milling treatment (described below), no further trials were made using ion-exchanged glass.

Concurrent with the ion-exchange processing, the effect on electrode position due to decreasing glass particle size by ball milling was studied. Reducing particle size results in particles with higher area-to-volume ratio. This in turn results in a higher electrical charge-to-volume ratio which increases the mobility of the charged particle in the applied field.

Two batches of the Code 1723 glass were milled for 12 hours in preparation for coating trials. Both batches comprised 500 grams of glass and 1,000 milliliters of water. One batch contained, in addition, 1 milliliter of a 10 percent KOH solution; the other did not. The milled batches were allowed to stand for 24 hours, after which the supernatant liquid was decanted and distilled water was added to give suspensions with a specific gravity of 1.2.

Coating trials on small grids were made using the batches of milled glass (with the KOH addition) with suspension conductivities of 6.8×10^{-5} ohm^{-1} cm^{-1} and 1.1×10^{-4} ohm^{-1} cm^{-1}. In each case, good adherent coatings resulted using applied voltages of 100 volts for 3.5 minutes. Using the suspension milled without the KOH addition, a good adherent coating resulted when the suspension conductivity was 7.4×10^{-5} ohm^{-1} cm^{-1}, but only fair adherence of the coating was noted when the conductivity was increased to 1.0×10^{-4} ohm^{-1} cm^{-1}.

Next, a suspension was prepared based on 11 kilograms of glass. The glass was ball milled for 12 hours using water and KOH contents proportional to that used in the 500-gram glass batch described above. After settling, decanting, and adding distilled water, the suspension had a conductivity of 6.5×10^{-5} ohm^{-1} cm^{-1}. Five grids were coated, applying 200 volts for various periods between 30 and 90 seconds. Good adherent coatings resulted.

After the coating was removed from the back side of the grids, the grids were fired according to a temperature-atmosphere schedule discussed in a later section. The relationships between deposition time and fired-coating thickness for the five grids were:

Deposition Time, sec	Fired-Coating Thickness, cm	Deposition Rate, cm/sec
30	0.036	0.0012
45	0.058	0.0013
60	0.076	0.0013
75	0.086	0.0011
90	0.099	0.0011

Two types of defects were observed in the coatings. One was a black crust on the surface of the glass, and the other was the deep grooves in the coating at its narrowest cross section. The black crust was considered to be molybdenum oxide formed during electrodeposition. The grid shown in Figure 6 is typical of this set of coated grids.

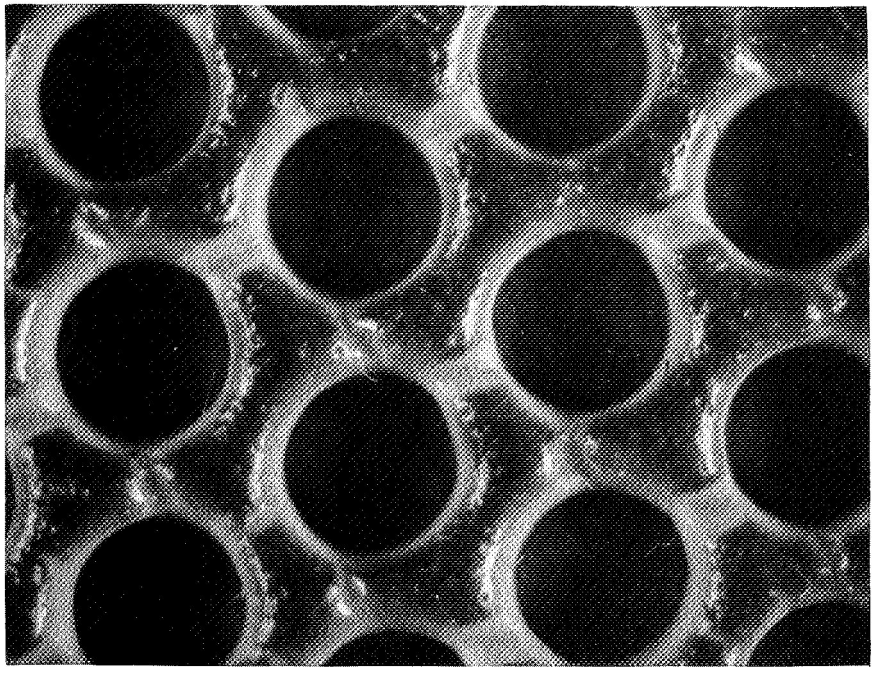


FIGURE 6. BLACK CRUST AND GROOVES ON CODE 1723 GLASS COATING FIRED IN NONOXIDIZING ATMOSPHERE

A second set of grids was coated after adjusting the conductivity of the suspension to a lower value of 4.3×10^{-5} ohm^{-1} cm^{-1}. This lower conductivity was expected to result in less molybdenum-ion formation during electrodeposition. After the grids were coated, a glass (petri) dish of appropriate diameter was placed, inverted, over the top of the coated grid. The covered grid was supported on a ring stand in such a way as to expose the bottom coated surface of the grid to the air during drying. This arrangement was devised with the aim of causing the drying to take place from the bottom coated surface which is scraped from the grid prior to firing it. It was considered that during drying the molybdenum-ion migration would be toward the bottom surface, which is the one scraped from the grid.

Four grids were coated at a potential of 200 volts. The deposition time for one was 40 seconds. To check reproducibility, the other three were coated for 50 seconds. Before firing, the grids (except one of the three that was coated for 50 seconds) were prefired by heating in air to 482 C or 537 C for 1 to 10 minutes.

The fired coatings did not exhibit the black crust (Figures 7 and 8). The relationship between deposition time and fired-coating thickness for the grids was:

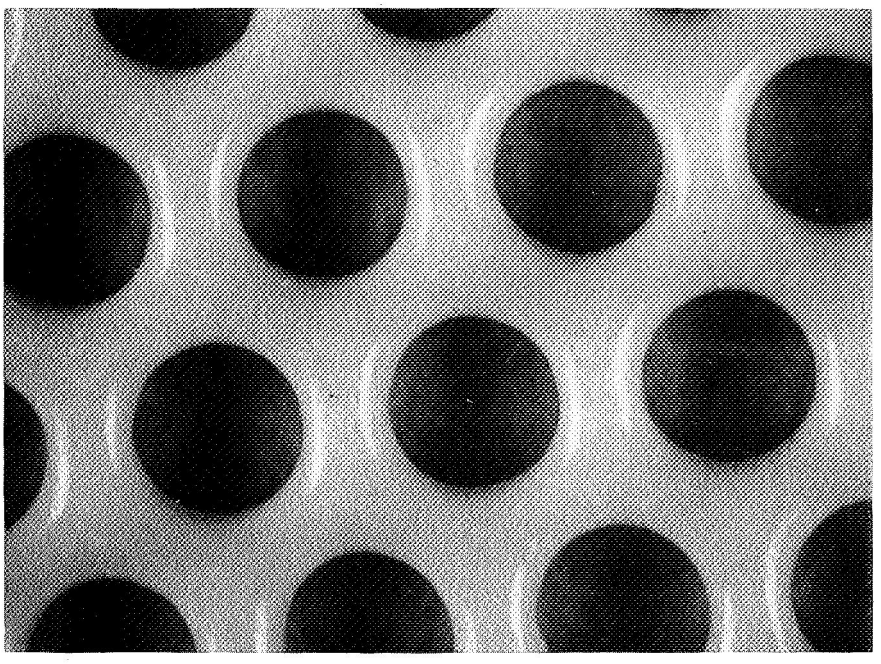


FIGURE 7. DEFECT-FREE CODE 1723 GLASS COATING, 0.041 CENTIMETERS THICK (13X)

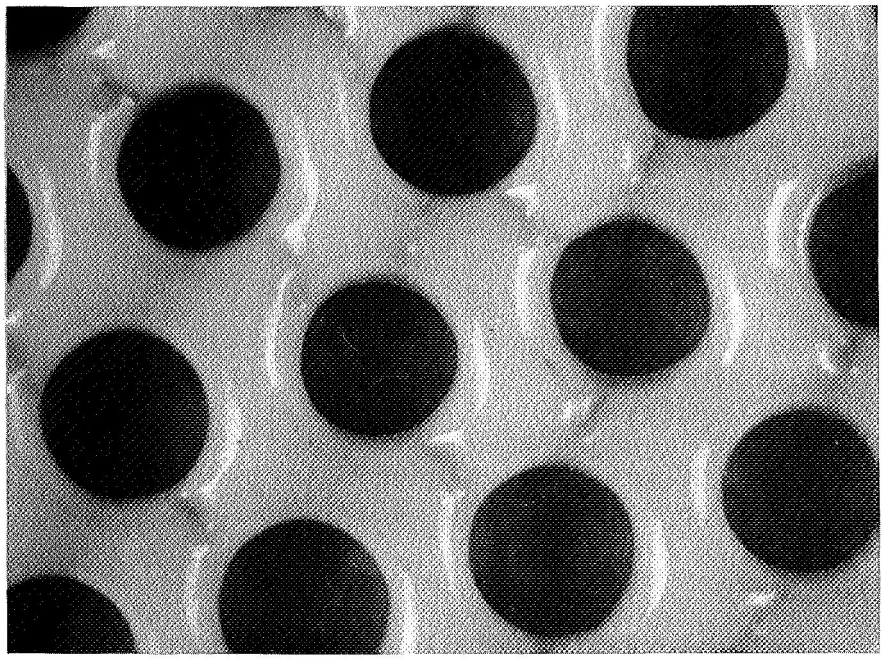


FIGURE 8. GROOVED CODE 1723 GLASS COATING, 0.053 CENTIMETERS THICK (13X)

Deposition Time, sec	Fired-Coating Thickness, cm	Deposition Rate, cm/sec
40	0.041	0.0010
50	0.053	0.0011

The three grids coated for 50 seconds had the same fired-coating thickness.

The freedom of groove defects in the 0.041-centimeter coating and their presence in the 0.053-centimeter coating were thought to be related to the combination of coating thickness and fineness of particle size of the glass. The phenomenon, known in vitreous coating technology as crawling, occurs when relatively thick, fine-grained coatings are fired. Shrinkage of such a coating, on drying and heating can produce microcracks. When the temperature becomes high enough to fuse the glass, the particles coalesce and pull away from any crack formed previously. Since the coating-thickness requirement for the 30-centimeter grids was set at between 0.041 and 0.051 centimeter, it was thought that the crawling or grooving might not occur.

Coating 30-Centimeter-Diameter Grids

On the basis of results of studies using 7.5-centimeter-diameter grids, and of experience coating 30-centimeter-diameter grids with Code 7052 glass, four of the chemically etched, 30-centimeter grids were coated with the following conditions prevailing:

Glass particle size	50 percent finer than 13 microns
Glass pretreatment	12-hour ball milling
Suspension volume	Based on 34 kilograms of glass
Suspension specific gravity	1.21
Suspension pH	10.0
Electrode spacing	12 centimeters
Suspension conductivity	$3.8\text{-}4.6 \times 10^{-5}$ ohm^{-1} cm^{-1}
Applied voltage	450 dc
Deposition current	4.3-4.9 amperes
Deposition time	Varied

Glass particle size was measured using the Coulter Counter. The as-received 200-mesh glass had a particle size distribution with 50 percent of the particles finer than 27 microns, which was reduced to 13 microns through the ball milling.

The electrode assembly used in a previous program when coating with the Code 7052 glass is shown in Figure 9. Coating on the back side of all of the grids was removed by rubbing with a facial tissue, using as little pressure as possible to abrade away the glass powder.

Before the final firing, the grids were placed in a furnace at 537 C for 5 minutes. During this treatment the coated grids were exposed to air atmosphere with the aim of volatilizing any molybdenum oxide in the glass, and to develop an oxide film on the metal grid for the purpose of promoting good adherence between the glass and metal during final firing.

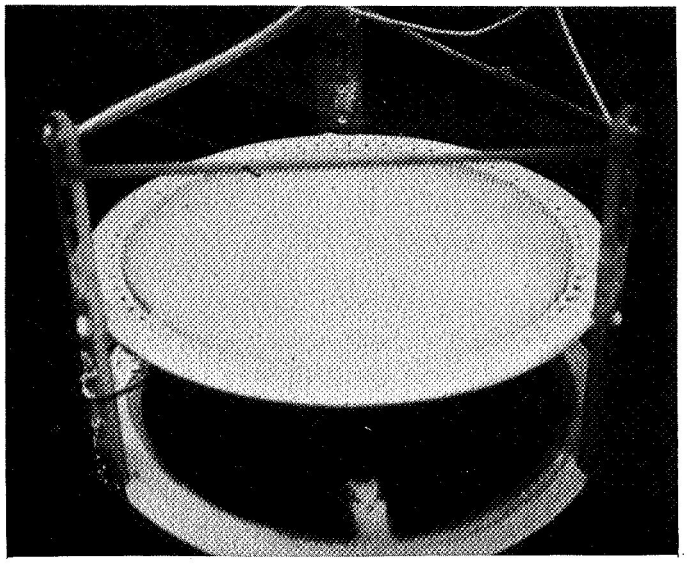


FIGURE 9. ANODE-CATHODE POSITIONER

The coated molybdenum anode is above the bronze cathode.

The deposition time and fired-coating thickness for the first and second coating trials were:

Time, sec	Fired-Coating Thickness, cm
60	0.058
40	0.030

Since the desired coating thickness was between 0.041 and 0.051 centimeter, the next two grids were coated for 50 seconds. The fired-coating thickness of each was 0.043 to 0.046 centimeter.

Firing of Coated Grids

The time-temperature-atmosphere schedule followed in firing all of the coated grids was designed to produce bubble-free glass coatings with optimum electrical resistivity. The process of firing in a helium atmosphere up to the peak temperature and then switching to a nitrogen atmosphere to cause the helium-filled bubbles to diffuse to the surface has been demonstrated.[1]

In the present work, the first set of 7.5-centimeter-diameter grids was fired without an oxidizing treatment prior to the firing process. The coated grids were placed in an Inconel box, the box was purged with helium at room temperature, and the grids were fired to 983 C. After a period at this peak temperature, the atmosphere was switched to nitrogen. Following a brief period, still at 983 C, the temperature was decreased to 260 C (cool enough to remove from the nitrogen atmosphere to air).

This treatment resulted in the formation of a black crust on these coatings. It was reasoned that molybdenum oxide present in the glass coating, as a result of the electrodeposition process, remained as a low or nonvolatile suboxide throughout the entire firing process. Thus, prior to subsequent firing trials, the coated grids were first subjected to a heat treatment in an air atmosphere. The purpose of this treatment was to oxidize the molybdenum oxide in the coating to the more volatile MoO_3 form which would permit it to volatilize, leaving a molybdenum-free coating. An oxide film on the molybdenum-grid surface would be expected also to promote good adherence of the glass to the metal.

The time-temperature-atmosphere schedule used to fire molybdenum-free coatings on the 30-centimeter-diameter grids was as follows:

(1) Room temperature purging in helium atmosphere for 30 minutes

(2) Increasing temperature to 670 C and holding at 670 C for 30 minutes – helium atmosphere.

(3) Increasing temperature to 983 C and holding for 50 minutes. Of this 50-minute period, a helium atmosphere was maintained for 35 minutes, and a nitrogen atmosphere for the remaining 15 minutes.

(4) Cooling to 260 C in nitrogen atmosphere after which specimen was removed to air atmosphere.

The grids were fired in the horizontal position and heating and cooling rates were as rapid as the furnace would allow.

Characteristics of Coated Grids

While the electrodeposition process, per se, was controlled to yield fired coatings of the desired thickness, problems encountered in other processing areas resulted in undesirable coating characteristics. The processing subsequent to depositing the coatings included drying of the coating, removal of the coating from the back side, a preheat treatment in air atmosphere, and final firing. In commenting on the problem areas related to these processes, reference is made to the photographs in Figures 10 through 17.

Figures 10, 11, and 12 show typical areas of the fired coatings. Defect-free areas and areas where the glass is grooved occur on both thin and thick coatings. It is thought that grooves result from microcracks formed in the coatings prior to their being fused. Some of the microcracks are thought to form during rubbing away of the coating on the back side of the grid. The extreme radial grooving (Figure 11) was present in the center area of the fourth grid processed. During rubbing of the coating from its back side, the grid flexed somewhat like the bottom of an oil can, with little pressure applied to it. Although no cracking of the coating was visible to the naked eye after removal of the coating on the back side, microcracks could have been present. Also, the unidirectional grooves (Figure 12) present in other areas suggest that their formation resulted from unidirectional stresses possibly occurring as the coated grid was expanding during the early stages of the final firing or during the preheating in the air atmosphere. These, in turn, could be associated with the tightness of sewing of the grids to the ring in selected areas around the circumference.

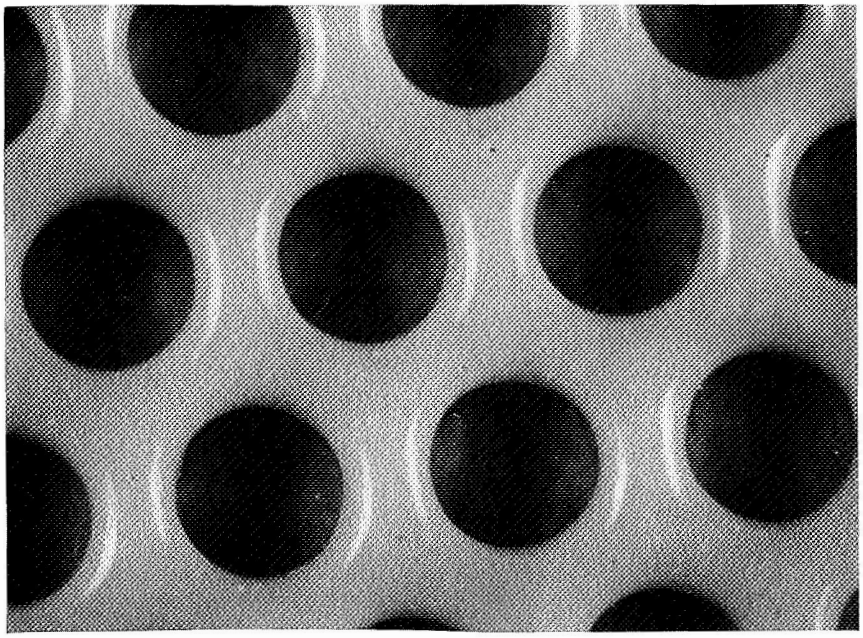


FIGURE 10. TYPICAL DEFECT-FREE AREA OF COATED GRIDS

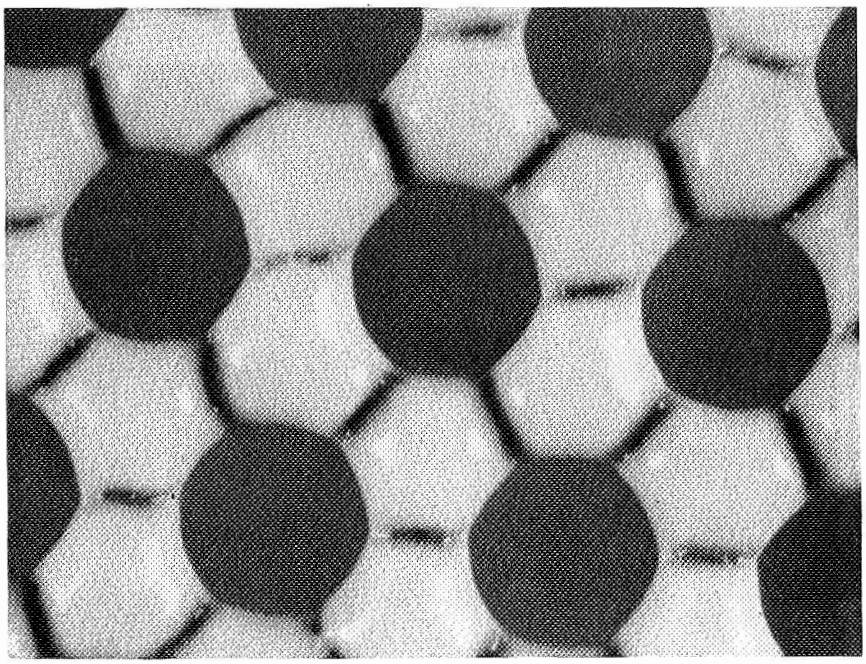


FIGURE 11. AREA OF EXTREME GROOVE FORMATION ON COATED GRIDS

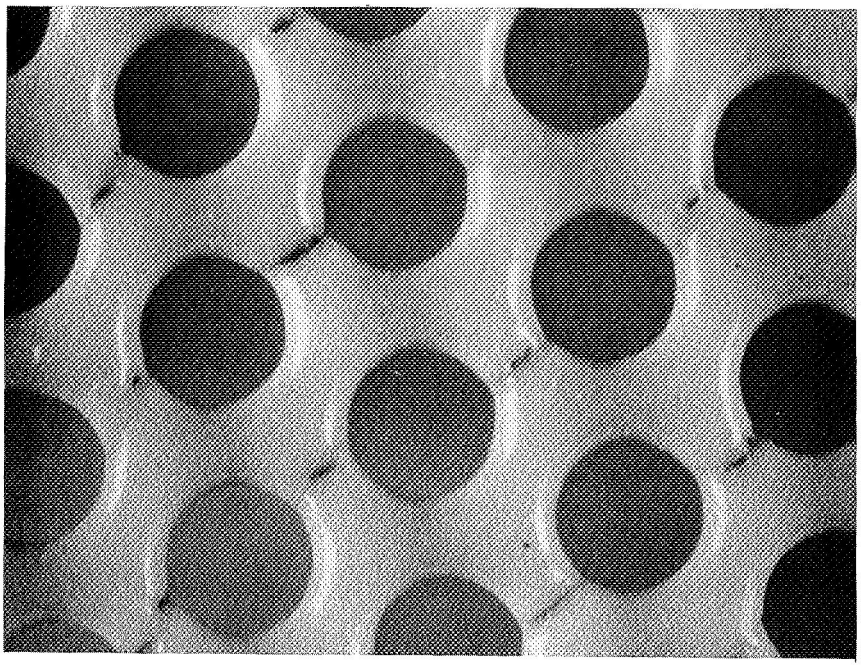


FIGURE 12. AREA OF UNIDIRECTIONAL GROOVE FORMATION ON COATED GRIDS

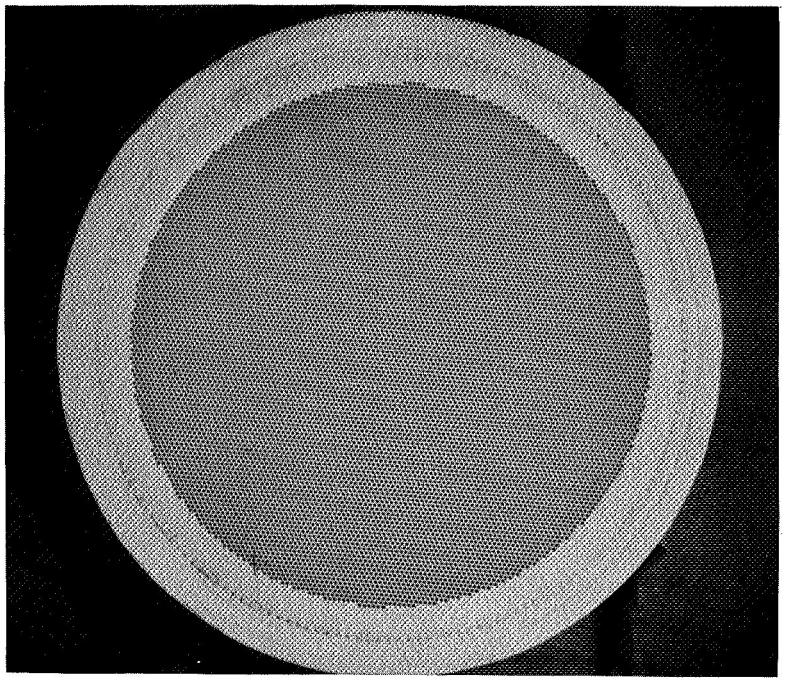


FIGURE 13. COATED GRID BEFORE FIRING (FIRED-COATING THICKNESS - 0.30 CENTIMETER)

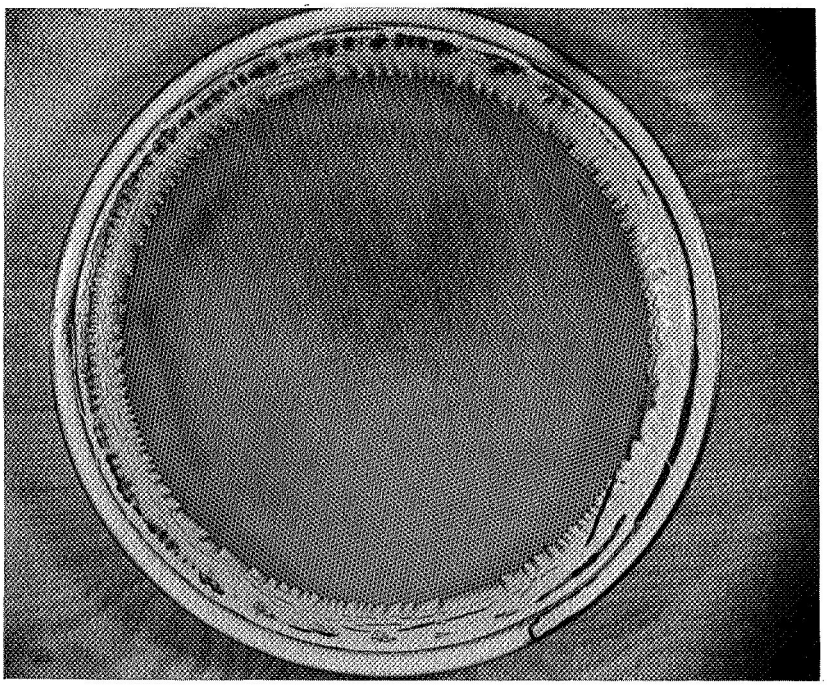


FIGURE 14. FIRST GRID FIRED, COATING THICKNESS - 0.058 CENTIMETER

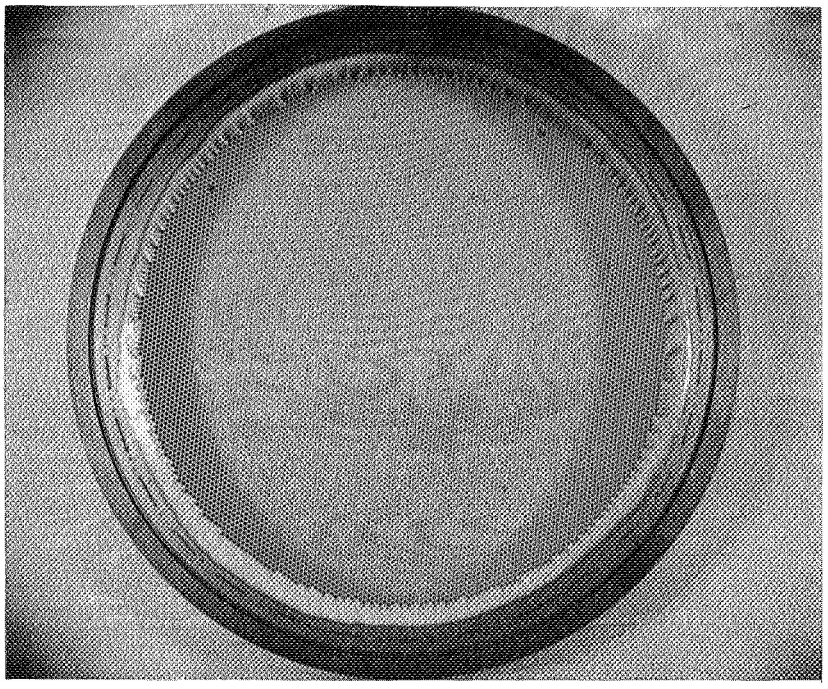


FIGURE 15. SECOND GRID FIRED, COATING THICKNESS - 0.030 CENTIMETER

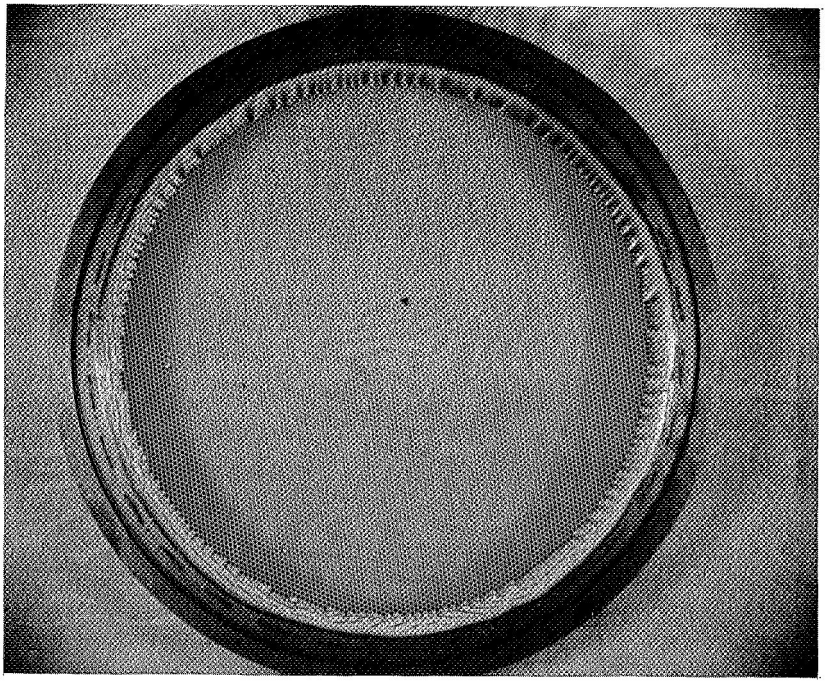


FIGURE 16. THIRD GRID FIRED, COATING THICKNESS - 0.043 TO 0.046 CENTIMETER

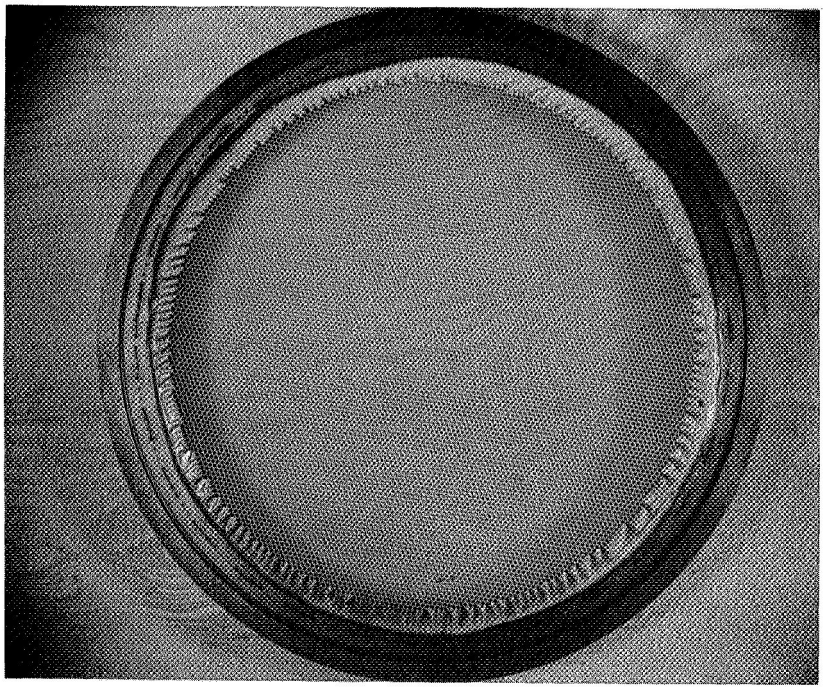


FIGURE 17. FOURTH GRID FIRED, COATING THICKNESS - 0.043 TO 0.046 CENTIMETER

It is thought that the presence of grooves in the coatings made of Code 1723 glass and the absence of these grooves in previous coatings made of the Code 7052 glass are due primarily to the difference in fluidity of the glasses at their maximum firing temperatures. The Code 1723 coating was fired to a lower temperature (938 C) than reached for the Code 7052 coating (955 C), whereas a higher temperature would be required for the Code 1723 glass to become as fluid as the Code 7052 glass. Possibly, the Code 7052 coating also formed grooves, and they filled in as the glass became more fluid.

A second source of defect was in the technique for removing the coating on the back side of the grid. As this coating was being rubbed off – and particularly as it became very thin – an excess pressure would sometimes push the coating off the grid. This did not occur on the first grid coated (Figure 14), which had the thickest coating. It occurred most frequently on the second grid, with the thinnest coating. Figure 13 shows the coated grid before firing and Figure 15 shows the fired grid. The dark spots are areas where the coating had been "popped" off during removal of the coating on the back side.

Figure 16 shows the third grid coated. The white spots are areas of more opaque glass which was placed over areas where the original coating had been "popped" off during removal of the coating on the back side of the grid. The glass in these patched areas did not fuse enough to blend with the coating and, in fact, shrank away from it – again probably because of the relatively low firing temperature.

By the time the grid in Figure 17 was coated, the technique for rubbing the coating from the back side with minimum pressure against the grid had been improved to the point that only two areas (in the 6 o'clock position) were popped off. The tendency to pop the coating from the front of the grid is thought to have been a problem with the Code 1723 glass-coated grids (whereas it was not with the Code 7052 coatings) because the coatings were deposited at low suspension conductivities and were dried under conditions favoring the migration of molybdenum oxide to the coating on the back side of the grid.

DISCUSSION OF RESULTS

A set of electrodeposition parameters for depositing a coating of a specific thickness was determined. The studies using simple sheet electrodes showed that coating thickness could be varied by varying applied voltage, current density, deposition time, or electrode spacing. Increase in all but the last parameter resulted in increased thickness; increasing the distance between electrodes decreased coating thickness.

Electrodeposition trials using grids indicated that the electric charge on the as-received minus 200-mesh Code 1723 glass particles was insufficient for depositing a satisfactory coating on this substrate configuration. Heating the glass before using it, to burn out contaminating lint or other organic matter which might be a source of bubbles in the fired coating, would be expected to further decrease the active net negative charge required on the glass to achieve good electrodeposition characteristics. The glass was known to be negatively charged because of its migration to the molybdenum anode, in the applied field. Grinding the glass to a smaller particle size resulted not only in the migration of the particles to the anode, but also in the formation of an adherent coating.

The higher surface-to-volume ratio of the finer glass, as well as its better adherence to the anode, indicated that the suspension of ground glass contained more particles of higher electrical charge. The proportion of glass-particle current carriers to ion current carriers was not measured, but the ratio increased through the grinding and adjusting of the suspension conductivity by decanting and replacing with distilled water. Good coating adherence resulted through appropriate electrochemical reactions at the anode.

The results of the present studies with the Code 1723 glass and those obtained previously with the Code 7052 glass indicate that the difference in the electrodeposition characteristics of the two glasses is due to their different chemical nature. Further, it is suggested that any given glass has its own electrodeposition characteristics – depending on its chemical constitution – but that the requirements for its sucessful deposition can be determined. Though a set of conditions was determined for successfully depositing the required coating thickness, difficulties were encountered in subsequent processing of the coated grids. Possibly, alternative deposition parameters would alleviate some of the difficulty in preparing defect-free coatings. The defects present in the finished coatings were of two types – grooves and small uncoated areas near the holes.

The grooves in the coating at its narrowest cross section suggest crawling. However, conventional crawling is considered to be a consequence of thick coatings of glass of a very fine particle size. Shrinkage of the coating on drying and in early stages of firing causes microcracks which do not heal on fusion and, in fact, widen because of surface-tension forces in the glass. The grooves are thought to result from microcracks that formed, not so much because of high shrinkage of the coating on drying but because of mechanical stresses placed on the coating either through flexing of the metal substrate or through directional stresses associated with the thermal expansion of the grid-ring assembly in the early stages of the firing cycle.

This view is supported by the fact that groove-free areas were present on all of the grids coated, including the one with the thickest coating. Also, although the 30-centimeter-diameter grid with the thinnest coating had grooved areas, a 7.5-centimeter-diameter grid with the same coating thickness was free of any grooves. Conflicting with this view is the presence of grooves on 7.5-cm grids with coatings of the same thickness as the thicker coatings on the 30-cm grids.

There are three possibilities for eliminating the groove defect.

(1) To the extent that fineness of glass particle size is a source of the defect, the as-received minus 200-mesh glass might be ball milled for a period less than the 12 hours used here. However, it would be expected that this would detract from the bonding of the electrodeposited coating to the grid.

(2) The coating might be deposited from a suspension of higher or lower conductivity. This would be expected to vary the extent of molybdenum oxide formation in the glass coating. The molybdenum oxide is thought to serve as a bond in the glass coating. The optimum bonding – which is not necessarily the strongest bonding – would have to be determined.

(3) The grooves might fill in again if a higher firing temperature were used. If the metal substrate was not exposed at the bottom of the grooves, surface tension forces at higher temperatures where the glass is more fluid might cause the grooves to heal.

The presence of grooves was never noted in coatings of the Code 7052 glass. Although no particle-size determination was made, this glass also was ball milled on several occasions. It is conceivable that at some point in firing the Code 7052 coatings, they also were grooved but the grooves disappeared as the glass became more fluid. To attain the same fluidity in the Code 1723 glass, it is estimated that a peak firing temperature of around 1065 C or higher would be required.

The cause of the other defect – the exposure of the metal grid near holes – is known. The pressure of rubbing the glass powder from the back side of the grid became excessive occasionally, and resulted in pushing the glass off of the hole wall as well as some of the coating on the front side of the grid.

This defect also might be eliminated by coating from suspensions of higher conductivity which would permit more molybdenum oxide bonding of the glass powder. Alternatively, if the as-deposited coatings on the 30-cm grids were allowed to dry in a way to cause the molybdenum oxide to form on the front of the grid, the coating would be expected to be better bonded. It is now known that this can be tolerated provided the coated grid is subjected to preheating in an air atmosphere to remove this oxide.

An alternative approach to eliminating this defect is to abrade the coating from the back side using a device which would limit the pressure that would be applied, e.g., via a spring. A third approach would be to mask the back of the grid to prevent a coating from being deposited on this face.

One other process not resolved in the present program was that of coating the mounting ring. It is thought that this ring can be coated in an independent process.

CONCLUSIONS

This investigation stressed the study of electrodeposition parameters for applying a glass coating to thruster grids. The rather unconventional chemical constitution of the Code 1723 glass used was related to its electrodeposition characteristics, and served to call attention to the importance of the role of electrical charge of the glass in the successful deposition of a coating. It was concluded that a set of deposition parameters, such as applied voltage, suspension specific gravity, electrode spacing, etc., could readily be established once the appropriate electric charge on the glass particles was developed. In this study, the charge which was initially inadequate was increased through grinding the glass to a finer particle size. When the glass was appropriately prepared, coatings of the desired thickness were readily deposited. Because of the emphasis on definition of acceptable electrodeposition parameters, a complete study was not made of subsequent processing and the relationships between the electrodeposition variables and these subsequent processing parameters. These parameters include:

Rate of water removal from electrodeposited coating

Direction of water migration to coating surface during drying (toward front or back surface of grid)

Abrading pressure for removing coating on back surface of grid

Firing time-temperature conditions for healing grooves in coating.

The most serious defect in the fired coatings was the presence of grooves in the coating at its narrowest cross section. Whether these grooves formed as a result of excessively fine grinding of the glass or stressing of the coating before the glass was fired, or both, was not determined. Further, it was not determined whether higher temperature firing of the glass would result in the grooves filling. It is thought that this defect might be eliminated by varying the firing temperature as it was not present on grids coated with Code 7052 glass which was similarly processed, with the exception of the firing treatment. In any future programs, the emphasis should and could be shifted from the electrodeposition process of coating, per se, and include the overall processing.

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