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Screen-Printing of 1720 and 7059 Glass Insulating Bonds for the Radioisotopic Thermoelectric Generator

C. W. Jennings

Sandia Laboratories

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SLA-73-0080

SCREEN-PRINTING OF 1720 AND 7059 GLASS INSULATING BONDS
FOR THE RADIOISOTOPIC THERMOELECTRIC GENERATOR

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December 1973

ABSTRACT

As part of the RTG fabrication sequence, Si/Ge and Si/Ge-glass substrates are coated with Corning 1720 and 7059 glass by a screen-printing process. A slurry of glass, pine oil, and terpinol is printed, followed by a burnout of the organic matter in oxygen at 500°C. It has been determined from overall fabrication considerations that there should be sufficient glass present to yield a fused-glass thickness of 1.2 to 1.4 mils. Glass content of the slurry, screen size, squeegee pressure, and breakaway distance are the important parameters affecting the amount of glass printed. Less variability in glass thickness is experienced when the screen contacts the substrate during printing. This occurs at higher squeegee pressures. The desired glass thickness is achieved by adjusting the glass content of the slurry to the screen mesh size, i. e., less glass is needed for an 80-mesh screen than for a 105-mesh screen. Not all of the slurry is retained on the substrate during printing; the fraction adhering to the screen is a function of the breakaway distance. Moderate slurry viscosity and temperature changes during screen printing have only minor effects on the thickness of the glass deposited. Routine printing of 1720 and 7059 glass slurries with 65- and 70-percent glass have consistently yielded glass thicknesses between 1.2 and 1.4 mils.

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SCREEN-PRINTING OF 1720 AND 7059 GLASS INSULATING BONDS FOR THE RADIOISOTOPIC THERMOELECTRIC GENERATOR

Introduction

This investigation was a part of the Radioisotopic Thermoelectric Generator (RTG) development. At the time it was carried out, attempts were being made to fabricate 2-D thermopiles. In this fabrication, Corning 1720 glass in an organic matrix was screen-printed or transferred from a tape onto Si/Ge wafers. The organic binder was burned off in oxygen at 500°C and the wafers were stacked and hot pressed at a temperature sufficient to fuse the glass and form an adherent laminated stack. This stack was sliced lengthwise normal to the wafers and the Si/Ge-glass stack slices were screen-printed with a lower melting glass, Corning 7059. An organic burnout and hot-pressing operation followed to produce the RTG laminated stack. Screen-printing of both 1720 and 7059 glass was studied.

Since that time, changes have been incorporated into the RTG processing. The 7059 glass is screen-printed for the first bond and a 7052 tape is used for the second bond. Although only the 7059 results have been directly applicable to the current RTG, the understanding of screen-printing generated in the study has been of general applicability.

The screen-printing process has many variables. In the earliest phase of the RTG development, difficulty was encountered in controlling the variables to achieve consistently a desired glass thickness. A better understanding of the printing process and interrelationship between variables was required if adequate process control was to be established. The objective of this investigation was to examine this process and determine the optimum conditions for screen-printing of glass slurries so that a desired thickness of glass could be produced consistently on an Si/Ge or laminated Si/Ge-glass substrate. Nominal bond thickness required was 0.001 inch or 1.0 mil. However, it was found desirable (to minimize the size and number of voids and unfilled areas in the bond) to introduce a slight excess of glass onto the substrates. The initial thickness range was set at 1.2 to 1.4 mils with an optimum of 1.25 mils of fused glass.

The principal variables investigated were glass composition, viscosity of the glass organic slurry, screen size, squeegee pressure, and breakaway distance used in the printing operation.

Process Description

This process used for depositing bond material differs from typical screen-printing applications in that the entire substrate surface is printed instead of a pattern. A slurry is spread through a screen that is held on or close to the substrate surface. Spreading is accomplished with a diamond-shaped polyurethane applicator, called a squeegee, which sweeps across the screen, pressing the screen and the slurry to the substrate (Figure 1). The screen behind the squeegee lifts as the pressure on it is relieved, leaving a deposit of slurry adhering to the substrate. The screen is normally

set a small distance above the substrate, causing it to be under tension as the squeegee depresses it during its sweep across the surface. This distance, which is adjustable, is called the breakaway distance. The greater this distance, the greater the shear force on the slurry in contact with the screen and substrate behind the squeegee. In general, the viscosity of a slurry under higher shear rates decreases. This tends to leave more slurry on the substrate and less clinging to the screen mesh, hence thicker prints.

Figure 1.

Contact printing occurs at zero breakaway distance, i.e., the screen is in contact over the entire surface. The screen must be lifted after the squeegee completes its sweep. At other breakaway settings, the screen is in contact or closest to the substrate only in an area directly under the squeegee as it moves across the surface.

In earlier printing, screen size, slurry viscosity, and breakaway distance were varied in an attempt to control the deposition. The required glass thickness could only be obtained with the lowest mesh screen, size 105, and the 7059 glass slurry was used directly from one container. Glass thicknesses were usually lower than the minimum 1.2 mils and did not have the desired reproducibility from day to day for the same printing conditions. With the exception of the first substrates printed in a series, the weight of slurry printed was consistent throughout a series of 10 to 12 specimens. In spite of the unexplained day-to-day variations, the good reproducibility within a series, after cleaning and reprinting of the initial substrates, made it feasible to monitor the printing control by weighing only the first, middle, and last specimen of each series. The adoption of this procedure, rather than weighing each specimen, facilitated data acquisition. The three specimens were also weighed after burning out the organic material in oxygen at 500°C for 4 hours. Using the weight of glass after burnout, specimen area, and glass density, a thickness of fused glass was calculated. The ratio of weight of glass after burnout (BOW) to weight of wet slurry (WW) was found to be consistent for a given slurry composition. This ratio was used to calculate the wet-weight limits necessary to provide a glass thickness within the required range. Using the wet slurry weight, specimen area, and density of the slurry, the thickness of slurry deposited can be calculated.

Models for the Printing Process

The thickness or volume of slurry printed from a given screen can be estimated for various assumptions regarding the distribution of slurry on the screen. Using Industrial Reproductions, Inc., data on their woven stainless-steel screens and the following assumptions, maximum slurry thicknesses for contact-printing have been calculated. These are shown in Table I.

TABLE I

$$t_m = \frac{V_m}{A_2}$$

$$A_2 = (m+d)^2$$

$$t_{m+w} = \frac{V_{m+d} + V_m - V_{\text{wire}}}{2A_2}$$

$$V_m = 2dm^2$$

$$t_{m+2w} = \frac{V_{m+d} - V_{\text{wire}}}{A_2}$$

$$V_{m+d} = 2d(m+d)^2$$

$$V_{\text{wire}} \sim 2 \frac{\pi d^2}{4} \sqrt{(m+d)^2 + d^2}$$

S.S. Screen Size	Wire Dia (d)	Mesh Opening (m)	t_m	t_{m+w} (mils)	t_{m+2w}
080	3.7	7.0	3.2	4.2	5.3
105	3.0	6.5	2.8	3.6	4.4
150	2.6	4.1	2.0	2.7	3.5
165	2.0	4.2	1.8	2.4	2.9

Model t_m	Only slurry within the mesh opening is deposited on the substrate to provide the slurry thickness.
Model t_{m+w}	Slurry within the mesh opening and under the wires is deposited for the slurry thickness.
Model t_{m+2w}	Slurry within the mesh opening and over and under the wires is deposited for the slurry thickness.

Models t_m and t_{m+w} are more probable than t_{m+2w} , especially for the more viscous slurries. Not all of the slurry is retained on the substrate. The fraction remaining on the screen is a function of the difference in adhesion between the screen and substrate, the viscosity, and the break-away distance. The slurry viscosity and adhesion to the substrate can vary with the temperature and humidity of the environment. For most of the substrates printed in this investigation, the temperature and humidity did not vary appreciably and, within modest limits, did not appear to be critical process parameters.

Slurry thicknesses necessary for a given glass thickness can be calculated from the material densities and slurry composition, i.e.,

$$\begin{aligned}\text{Slurry thickness} &= \text{glass thickness} \times \text{glass density} / \text{glass concentration} \\ \text{Glass concentration} &= \text{grams of glass/cm}^3 \text{ slurry} = \text{weight-percent glass} \times \\ &\quad \text{slurry density}\end{aligned}$$

For slurries with low glass contents, the required thickness may not be obtained with good control by contact printing using the largest available mesh screen, i.e., required $t_{\text{slurry}} > t_{m+w}$. To obtain the proper thickness, the screen must remain a fixed distance away from the substrate during printing so that some slurry flows between the screen and the substrate. To achieve the proper flow of slurry, the squeegee pressure, breakaway distance, and viscosity must be adjusted by trial and error and remain unaltered during subsequent printing. In earlier experimentation, this was found to be very difficult. More reproducible results could be obtained by placing a spacer on the fixture holding the substrate or by building up a spacer on the screen in the printing area such that the screen is always a fixed distance from the substrate during printing.

Another approach is to determine the volume of slurry deposited from a given screen under conditions similar to contact printing. A close approximation to this volume can be obtained by selecting slurry thickness between t_m and t_{m+w} . The glass content of the slurry is then adjusted to provide the necessary weight of glass in this volume to give the required glass thickness. Minor variations in thickness can be effected through other process variables. Under present circumstances, this method is believed to provide the best control over the process.

Experimental

Material Description

The earlier two-dimensional RTG required the screen-printing of two kinds of glass, 7059 and 1720. The 7059 glass softens at 842°C, the 1720 at 915°C. The nominal compositions of the glasses in weight-percent are:

<u>Glass</u>	<u>SiO₂</u>	<u>B₂O₃</u>	<u>Al₂O₃</u>	<u>BaO</u>	<u>MgO</u>	<u>CaO</u>	<u>Li₂O, Na₂O, K₂O</u>	<u>As₂O₃</u>	<u>SrO</u>
7059	48.8	12.88	11.84	22.2	0.35		< 0.2	0.15	0.2
1720	57.0	4.0	20.5		12.0	5.5	1.0		

Density of the 7059 glass is 2.75 g/cm³ and the 1720 glass, 2.53 g/cm³.

A slurry was made up of each glass at ElectroScience Laboratories in Philadelphia, Pennsylvania. Each glass was to be ground to an average particle size of 5μ prior to being suspended in their No. 401 vehicle (pine oil). The supplier reported that ethyl acetate and Borundum balls were used in ball milling. Blending was carried out on a three-roller mill. The 1720 slurry was made up to contain 65 parts by weight glass and 35 parts pine oil. Their No. 400 thinner (terpinol) was added to achieve a viscosity of 138 Kcp at 10 rpm and 25°C using a special spindle for the viscosity measurement. The 7059 slurry was made up to contain 70 parts glass and 30 parts vehicle. No thinner was reported, and the viscosity measured with their viscometer was 370 Kcp at 10 rpm at 25°C.

The glass contents of the two slurries as sampled by R. M. Merrill, 5521, from the shipping jars are tabulated below:

<u>Glass</u>	<u>Jar</u>	<u>Weight- Percent of Glass</u>	<u>Viscosity (Sandia)</u>	<u>Slurry Density</u>	
				<u>Calculated</u>	<u>Measured</u>
7059	1	71.0	215 ± 5 Kcp	1.75 g/cm ³	
	2	62.1	220 ± 5 Kcp	1.59 g/cm ³	1.61 g/cm ³
1720	1	49.9	57 ± 2 Kcp	1.36 g/cm ³	
	2	50.5	55 ± 2 Kcp	1.365 g/cm ³	1.394 g/cm ³

The viscosities were determined at 25±2°C with a Brookfield model HBT viscometer at 10 rpm with an SC 4-14 spindle. Minimum recording time was 2 minutes per measurement. The slurries are thixotropic in behavior. Any meaningful comparison between measurements with different slurries or suppliers must be carried out with the same spindle type at the same speed and recording time. Variations in viscosity with temperature and spindle speed of the 1720 slurry are shown in Figure 2.

Settling of the glass in the slurry prior to packaging may have caused the difference in glass content as shown above between the two jars of 7059 slurry. Particle size analyses of the glasses are given in Table II.

Figure 2a.

Figure 2b.

TABLE II
Particle Size Determinations of RTG Glasses

	Sedimentation ^(a) Avg Particle Size (μ)	Range for 10 to 90 Weight- Percent of Particles (μ)	SEM ^(b) Particle Size Range (μ)
1720 glass from Jar 2 slurry ^(c)	1.4	0.5-2.7	0.5 to 1.5
1720 glass, ball milled for 168 hrs ^(d)	3.4	1.0-5.8	0.5 to 4
1720 glass extracted from glass tape ^(e)	4.2	1.5-13	1 to 15
7059 glass from Jar 1 slurry ^(c)	2.9	0.9-5.0	0.5 to 4

(a) MSA Sedimentation Particle Size Analysis at Coors Spectro-Chemical Laboratory, Whitby Size Analyzer; sedimentation liquid, 0.25 percent calgon in distilled H₂O, feeding liquid 0.25 percent calgon in H₂O with acetone.

(b) Scanning Electron Microscope Analyses by C. J. Miglionico, 5522; all particles were angular in shape.

(c) Extracted with acetone.

(d) Used as makeup glass for 1720 slurries.

(e) Layers separated with acetone and carbonaceous material removed in low temperature plasma asher.

Both the 7059 and 1720 glass slurries were to be printed to provide 1.2 to 1.4 mils of fused glass. As mentioned earlier an excess of glass over the RTG design requirement of 1.0 mil was desired to permit extrusion of the glass during the hot-pressing operation. The excess minimizes void and bubble formation. Because of this bondline adjustment through extrusion, process control was needed to provide a small excess of glass and avoid any deficiency.

Because the slurries differed in glass content, each required a different slurry thickness to be printed to provide the required fused-glass thickness. This required careful adjustments of the printing process variables. An alternative as mentioned earlier was to determine the slurry thickness for optimum printing, and adjust the glass content of the slurry to provide the required fused-glass thickness for this slurry thickness. The operating region in which there is minimum sensitivity of slurry thickness to process change is considered optimum for printing. To make the adjustment in glass content, the slurry components had to be identified and their densities determined. Measured density of the 401 vehicle is 0.94 g/cm³ and the 400 thinner, 0.915 g/cm³. Approximate densities of the slurries were calculated from their glass contents, the reported ratios of glass to vehicle, and the densities of the constituents. Measured densities of two of the slurries agree within the experimental error with the calculated densities.

Because we were determining glass weight (and thickness) from deposited slurry weight, it was necessary that the volatilization between specimen deposition and weighing be as small and constant as possible. To determine some measure of the effect of evaporation on the vehicle and thinner content of the slurries, a few weight-loss experiments were run. The thinner lost 0.7 percent by weight on evacuation to 1 torr in 15 minutes at room temperature and 3 percent to 0.5

torr in 30 minutes. Under a clean bench at room temperature, a situation similar to the screen-printing, the thinner lost 0.03 percent in 15 minutes, 0.08 percent in 30 minutes, 0.17 percent in 60 minutes, and 3.1 percent in 24 hours. At 100°C, 98 percent of the thinner was volatilized in 2 hours. Under the same conditions, 27 percent of the vehicle was volatilized.

Slurry Printing

The size 105 screen was used for all printing in this investigation. Earlier results and the data in Table I indicated this screen to be the best choice for meeting the slurry thickness requirements. Size 80 screen was not available. Squeegee pressure and breakaway distance were investigated for slurries of both types of glass. These variables determine the closeness of approach of the screen to the substrate and regulate the shear force on the slurry after deposition.

7059 Slurries -- Variation in weight of slurry deposited for various squeegee pressures and constant 0.027-inch breakaway distance is shown in Figure 3. Squeegee pressure was adjusted by contacting the squeegee to the screen while the screen was on the substrate and by screwing the holder down a given fraction of a turn. This is not an exact operation but serves to show the influence of the squeegee pressure on the printing operation. A dial gage under the screen would probably be a more exact measure of this pressure.

Contact prints were made by manually lifting the screen after squeegee spreading. The slurry weight for contact printing was approximately that obtained at high squeegee pressures in the normal printing. At low squeegee pressures, the screen does not contact the substrate; hence, there are large variations in slurry thickness with small variations in squeegee pressure.

Variation in breakaway distance at a squeegee pressure of 1/16 turn produced the slurry weights shown in Figure 4. Slurry viscosity was varied by additions of terpinol thinner. As seen in Figure 5, slurry weight decreased gradually with decreasing viscosity under conditions of constant squeegee pressure and breakaway distance; thus, viscosity variations associated with moderate changes in room temperature should not cause appreciable changes in the thickness of glass deposited.

The data in Table III for the printing of 7059 glass on Si/Ge-glass stack slices show that with optimum printing conditions the required glass thicknesses can be produced consistently.

Figure 3.

Figure 4.

Figure 5. 10-RPM SC4-14 Spindle

TABLE III

Screen Printed 7059 Glass - Jar No. 1
(215 KcP Viscosity - 105 Screen)

Specimen Series	Avg Spec Wt (mg)	Break- away (inch)	Temp (°F)	Wet Wt (mg)	Calc Glass Thickness (mils)	Burned Out Wt (mg)	Calc Glass Thickness (mils)	Ratio (BOW) WW	BOW Range (mg)
100	469	0.027	81	28.2	1.35	20.4	1.35	0.723	19.9-20.9
102	466	0.027	81	27.0	1.29	21.3	1.41	0.789	17.7-25.4
103	470	0.027	80	28.1	1.35	20.5	1.36	0.729	19.0-21.7
104	471	0.030	82.5	28.9	1.38	20.7	1.37	0.716	19.7-21.4
105	471	0.030	82.5	29.3	1.40	21.2	1.40	0.724	20.6-21.8
106	468	0.030	78	28.0	1.34				
107	468	0.028	78	29.5	1.41	20.4	1.35	0.692	20.1-20.7
109	470	0.027	81	26.9	1.29	19.4	1.28	0.721	16.7-20.8
110	445	0.027	81	26.6	1.33	19.3	1.34	0.725	19.0-19.5
113	467	0.027	82	27.7	1.33	19.7	1.31	0.711	18.6-20.7
114	466	0.027	82	26.2	1.25	19.4	1.28	0.741	18.1-20.4
116	472	0.027	80	27.3	1.31	19.7	1.30	0.711	19.3-20.1
117	465	0.027	80	26.7	1.28	19.4	1.28	0.726	18.0-20.5
118	470	0.027	78	31.1	1.49	22.2	1.47	0.714	21.7-23.5
119	469	0.027	78	29.5	1.41	17.5	1.16	0.593	16.1-20.1
120	468	0.027	82.5	29.2	1.40	21.4	1.42	0.733	20.5-22.2
121	469	0.027	82.5	30.6	1.46	22.1	1.46	0.723	20.4-25.1
122	472	0.027	82	31.1	1.49	22.4	1.48	0.719	21.3-23.7
123	470	0.027	82	28.9	1.38	21.1	1.40	0.731	20.5-22.0
111	499	0.027	82	34.2	1.39	24.7	1.54	0.722	24.1-25.4
112	498	0.027	82	30.9	1.40	22.5	1.40	0.728	21.5-23.2
124	469	0.0285	82	31.6	1.51	22.9	1.52	0.725	21.8-23.8
125	468	0.0285	81	29.8	1.42	21.8	1.44	0.731	20.0-23.1
87	416	0.0285	80	26.1	1.41	18.7	1.40	0.716	18.2-19.6
133*	469	0.025	81	27.2	1.30	19.9	1.32	0.731	18.5-21.0
134	449	0.025	81	22.1	1.11				
135	448	0.025	82	24.6	1.23	17.8	1.23	0.724	17.4-18.3
136	449	0.025	81	26.0	1.30	18.9	1.31	0.729	18.3-19.7
137	451	0.025	82	26.1	1.31	18.9	1.30	0.724	18.6-19.1
138	446	0.025	81	25.2	1.26	18.3	1.27	0.726	17.4-18.8
139	446	0.025	80	26.4	1.32	19.3	1.34	0.730	19.1-19.4
140	448	0.025	81	25.1	1.26	18.2	1.26	0.726	17.7-18.6
141	444	0.025	81	25.0	1.25	18.2	1.27	0.728	16.3-19.2
142	445	0.025	80	26.0	1.30	18.9	1.32	0.729	17.3-20.1
143	445	0.025	81	26.1	1.31	18.9	1.32	0.724	18.4-19.1
144	447	0.025	81	26.7	1.34	19.5	1.36	0.730	18.3-20.7
145	447	0.025	79	26.9	1.35	19.5	1.35	0.725	18.1-20.3
146	449	0.025	81	25.1	1.26	18.5	1.28	0.737	16.4-20.2
147	449	0.025	81	26.5	1.33	19.5	1.35	0.735	18.1-20.7
1081	219	0.025	81	53.0	1.25				
1099	225	0.025	79	54.4	1.25				
1221	225	0.025	82	54.4	1.25				
1009	226	0.025	82	53.7	1.23				
1081	219	0.025	79	50.9	1.20	37.3	1.21	0.733	
1101	224	0.025	79	57.8	1.33	42.1	1.33	0.728	
1008	228	0.025	82	52.8	1.20				
1221	234	0.025	82	62.7	1.39				

* All specimens after 133 had 1/16-turn squeegee pressure.

1720 Slurries -- A similar investigation of squeegee pressure and breakaway distance was carried out with 1720 glass slurries. The glass concentration in the slurry as received from the supplier was insufficient to produce the required glass thickness under conditions approaching contact printing, as shown in Figure 6. Rough calculations indicate that this would also be true for size 80 screen. This means that the consistency in amount of glass deposited is greatly dependent on the process parameters' remaining invariant.

Increasing the glass content of the slurry permits the slurry to be deposited under conditions which minimize the effect of small variations in squeegee pressure, breakaway distance, and viscosity, as shown by the results of Figures 7 through 11. A large mesh screen such as size 80 would permit lower glass concentrations in the slurry for the same glass thickness. Table IV records the results with 1720 glass.

Figure 6.

Figure 7,

Figure 8.

Figure 9.

Figure 10.

Figure 11.

TABLE IV
Screen Printed 1720 Glass - 105 Screen

Specimen Number	Avg Wt	Break- away (inch)	Slurry Viscosity (Kcp)	Temp (°F)	Squeegee Pressure	Wet Wt (mg)	Calc Glass Thickness (mils)	Burned Out Wt (mg)	Calc Glass Thickness (mils)	Ratio (BOW) (WW)	Calc Wet Thickness (mils)
N1023	758	0.027	100		Contact	53.3	1.15	34.2	1.18	0.641	3.1
N1023	755	0.027	100		Contact	67.7	1.46	42.1	1.45	0.622	3.9
N1023	751	0.027	100		Contact	76.0	1.64	47.4	1.64	0.624	4.4
P1019	511	0.027	100		Contact	77.5	1.67	48.3	1.67	0.623	5.0
P1019	513	0.027	31	82	Contact	74.3	1.60	46.3	1.60	0.623	4.3
P1019	513	0.025	31	82	Contact	74.6	1.60	46.4	1.60	0.622	4.3
P1019	513	0.029	100		Contact	93.4	2.00	58.2	2.00	0.623	5.4
P1019	513	0.030(a)	100		Contact	34.8	0.75	21.6	0.75	0.623	2.0
N1023	755	0.027	57(b)	83	Contact	53.8	0.96	27.6	0.95	0.513	3.5
N1023	755	0.030	57(b)	82	Contact	75.4	1.34	38.4	1.33	0.509	4.9
N1023	750	0.032	57(b)	82	Contact	38.6	0.69	20.1	0.69	0.511	2.6
N1023	755	0.029	57(b)	83	Contact	57.5	1.02				3.8
N1023	750	0.031	57(b)	83	Contact	61.1	1.09				4.1
N1023	755	0.0305	57(b)	83	Contact	66.9	1.19				4.5
N1023	750	0.033	57(b)		Contact	69.6	1.24				4.6
N1012	1091	0.033	57(b)	82	Contact	61.0	1.09	31.9	1.10	0.523	4.1
P1056	513	0.032	57(b)	80	Contact	72.1	1.28	37.2	1.28	0.516	4.8
P 231	517	0.032	57(b)	80	Contact	58.7	1.04	30.5	1.05	0.519	3.9
N1077	756	0.032	57(b)	82	Contact	73.5	1.31	37.8	1.30	0.514	4.9
P1050	512	0.033	57(b)	77	Contact	67.4	1.20	34.7	1.20	0.515	4.5
P1053	511	0.032	57(b)	77	Contact	62.9	1.12	32.5	1.12	0.516	4.2
P1055	518	0.032	57(b)	77	Contact	67.3	1.20	34.6	1.20	0.514	4.5
N1076	756	0.032	57(b)	80	Contact	53.0	0.94	27.4	0.95	0.517	3.6
N1078	756	0.033	57(b)	82	Contact	75.7	1.35	38.9	1.34	0.514	5.0
N1076	753	0.022	250(c)	79	1/16T	52.4	1.24	35.9	1.24	0.685	3.0

(a) 150 screen

(b) As received from Jar No. 1, 49.9% glass

(c) Made up to be 65.5% glass

Discussion

For optimum screen printing, the desired glass thickness is produced with minimum variation in slurry weight deposited per unit adjustment of squeegee pressure and breakaway distances as shown in Figures 3, 6 and 10. This low sensitivity or plateau region is approached at high squeegee pressures or for contact printing. Breakaway distance provides a convenient parameter for small adjustments in glass thickness, as shown in Figures 4, 9, and 11. Although wet slurry weight has been plotted, this may be readily converted to equivalent glass thickness using the ratio of BOW to WW (or the glass content of the slurry), substrate area, and glass density. Average thickness of slurry printed in the plateau region was 2.9 mils for the 7059 glass and 3.0 for the 1720 glass. These values may be compared with a slurry thickness of 2.8 mils calculated for Model t_m and 3.6 mils for t_{m+w} , shown in Table II. To achieve the required glass thickness, additional glass must be added to those slurries with low glass contents. The amount of glass to be added or the required glass content of the slurry may be approximated by calculation using the available data on slurries and that from Table I. A sample calculation is shown in the appendix.

Recommendations

The recommendations made as a result of this study were:

1. The first specimen in a series is often observed to deviate from the remainder in weight of printed slurry. Clean and reprint or discard this specimen for a more consistent series.
2. Device a better method to monitor squeegee pressure.
3. Conduct a squeegee pressure breakaway distance study with size 80 screen.
4. Blend 1720 slurry with 1720 glass powder to increase the glass content to 64 to 68 percent.
5. Procure and characterize an additional supply of 1720 and 7059 glass powder.
6. On the next procurement order, obtain complete details on the makeup of the glass slurry from the supplier.
7. Investigate the small difference between the ratio of burned-out weight and wet-slurry weight and the glass content of the slurry.
8. Exercise care that particulate matter is not present between the substrate and the screen.

This report represents an initial effort in the RTG development. Subsequent to it, a substantial effort has been made to implement these recommendations and modify equipment. Future components which require screen-printed insulator films may profit from the experience generated in this development, especially with regard to slurry procurement and deposition.

Summary

Glass coatings of controlled thickness can be screen-printed reproducibly onto Si/Ge substrates. The glass can be used to bond and insulate these substrates in devices such as the radioisotopic thermoelectric generator (RTG). Two types of glass, Corning 1720 and 7059, were printed from an organic slurry. Squeegee pressure and breakaway distance were found to be critical parameters in the printing process. A minimum glass content in the slurry was needed to obtain a reproducible desired glass thickness with available printing screens. Moderate viscosity changes had only a minor effect on thickness under conditions approaching contact printing.

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APPENDIX

CALCULATION FOR APPROXIMATE ADDITION OF CORNING 1720 GLASS TO JAR NO. 1,
1720 SLURRY TO CONTACT PRINT 1.25 MILS OF GLASS EQUIVALENT

APPENDIX

CALCULATION FOR APPROXIMATE ADDITION OF CORNING 1720 GLASS TO JAR NO^o 1, 1720 SLURRY TO CONTACT PRINT 1.25 MILS OF GLASS EQUIVALENT

- A. Assume 3.4 mils of slurry printed from size 80 screen (from Table II and results of size 105 screen):

$$\text{Glass concentration} = \frac{\text{thickness glass}}{\text{thickness slurry}} \times \text{glass density}$$

$$\begin{aligned} \text{Glass concentration} &= \frac{1.25 \text{ mils}^{\text{glass}}}{3.4 \text{ mils}^{\text{slurry}}} \times 2.53 \frac{\text{g}^{\text{glass}}}{\text{cm}^3 \text{ glass}} \\ &= 0.95 \frac{\text{g glass}}{\text{cm}^3 \text{ slurry}} \end{aligned}$$

- B. For 100 grams of 1720 slurry No. 1 with X grams of 1720 glass added (ratio 35/65 pine oil to glass):

$$(X + 49.9) \text{ g} \frac{\text{glass}}{2.53 \text{ g/cm}^3} = \left(\frac{X}{2.53} + 19.7^2 \right) \text{ cm}^3 \text{ glass}$$

$$\left(49.9 \times \frac{35}{65} \right) \text{ g} \frac{\text{pine oil}}{0.94 \text{ g/cm}^3} = 28.6 \text{ cm}^3 \text{ vehicle}$$

$$\left(100 - 49.9 - \frac{49.9 \times 35}{65} \right) \text{ g} \frac{\text{terpinol}}{0.915 \text{ g/cm}^3} = 25.4 \text{ cm}^3 \text{ thinner}$$

$$\text{Total volume} = \left(\frac{X}{2.53} + 19.7 + 28.6 + 25.4 \right) \text{ cm}^3$$

$$0.93 \text{ g} \frac{\text{glass}}{\text{cm}^3 \text{ slurry}} = \frac{(49.9 + X) \text{ g}^{\text{glass}}}{\left(\frac{X}{2.53} + 73.7 \right) \text{ cm}^3 \text{ slurry}}$$

$$X = \frac{18.6}{0.632} = 294. \text{ grams glass}$$

$$\text{Glass content} = \frac{79.3}{100 + 29.4} = 61.3 \text{ percent glass}$$

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