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PROGRESS REPORT

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ON

CONDUCTIVE PAINTS

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Submitted by

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This report covers the work performed from August 3, 1992 - September 30, 1992, under a subcontract from Sayles Enterprises entitled " A Study of Conductive Paints".

I. Paint Deposition

Electrical resistivity measurements require uniformly thick, pinhole free, smooth films deposited on insulating substrates. In order to accomplish this, a paint preparation procedure and deposition procedure must be developed.

Preliminary evaluation of paint films has yielded the following paint preparation procedure:

1. Filter through a 1000 mesh aluminum paint screen.
2. Continuously shake on a 100 rpm orbital shaker for one hour.
3. Immediately deposit after removal from shaker.

We have explored several different paint deposition methods, including spraying, rolling, and spinning. The results are summarized as follows:

1. Spraying undiluted paint- Paint dries before contacting the surface, resulting in a film that consists primarily of flakes.
2. Rolling- Employ an ink roller to apply the paint. The paint does not adhere to the rubber roller, but instead smears out over the substrate.
3. Spinning- Apply paint to the substrate, and spin at approximately 1000 rpm for 20 seconds. The major difficulty here is that near the edge of the substrate a ridge of paint forms. In the body of the substrate that is 0.5 x 0.5 inches, large thickness variations also exist, as seen in Figure 1. However, this technique is adequate for adhesion strength testing as discussed in Section II.
4. Improved Spinning- Apply a fixed amount of paint through a syringe onto a large area (1 - 2 inches in diameter) substrate, and spin. Small areas of the film, 0.5 x 0.5 inches, taken near the center of the substrate which avoid the edge effect have a uniformity variation on the order of $\pm 5\%$ as seen in Figure 2. This size area can be isolated, and used to obtain the film resistivity.

II. Adhesion Strength Testing

Virtually any application involving a coating requires that the coating adhere well to the underlying substrate. The adhesion strength of the Sayles Enterprises paints is

Figure 1. Film thickness in microns of a film spun on a 0.5 x 0.5 inch glass substrate at 1000 rpm for 20 seconds.

Figure 2. Thickness in microns of a film spun on a 2 inch diameter SiO₂ coated silicon wafer. The measurements were taken over a 0.5 x 0.5 inch area near the center of the wafer.

directly measured by the Sebastian IV Thin Film Adhesion Strength Measurement system manufactured by Quad Group Inc..

The paint is spun on the substrate as described previously, where adjustments are made for the specific substrate under study. The paint is then dried by inserting it into a preheated furnace for 30 minutes in a forming gas (80% N₂/20% H₂) atmosphere. The forming gas atmosphere minimizes oxidation. At this point we have used 100 C as the process temperature, although, in the next phase of work other temperatures will be explored.

Preparation for the adhesion test entails fastening a metal slug to the paint surface with epoxy, as seen in Figure 3. The slugs are precoated with high strength epoxy, and are supplied as part of the Quad Group system. The slug is held perpendicular to the paint surface while the epoxy is cured at 150 C. It should be pointed out that the epoxy contact area is less than 0.25 inches in diameter, and contact is made in the center of substrate 0.5 x 0.5 inches square.

Figure 3. Diagram of epoxy coated slug, paint film, and substrate.

The sample is then inserted into the adhesion strength measurement system, where a steadily increasing force is applied to the end of the metal slug. When failure occurs the system displays the force applied at the failure point. Table I shows the results obtained for Paint #3003 on SiO₂ coated silicon substrates, and glass substrates, as well as the results for Paint #2002 on glass substrates. Clearly, Paint #3003 adheres much better to the SiO₂ coated silicon, than to the glass. Further, it would appear that Paint #2002 adheres better to glass than #3003, although more tests are needed to confirm this.

SAMPLE #	ORDER OF TEST	RESULT, kg/cm ² or comment	PAINT NUMBER, # OF DROPS	SPIN SPEED, rpm	SUBSTRATE MATERIAL
8-19-1-1	1	80	3003, 3	1400	SiO ₂
8-19-1-2	10	77			
8-19-1-3	3	94 *			
8-21-1-1	8	15 *	3003, 4	1000	Glass
8-21-1-2	7	**			
8-21-1-3	2	**			
8-21-1-4	12	33			
8-21-1-5	11	**			
8-20-7-1	6	51 *	2002, 4	1000	Glass
8-20-7-2	5	52			
8-20-7-3	9	**			
8-20-7-4	4	**			

*slightly non-perpendicular

**failed before test

Table I. Adhesion strength of Paint #3003 on SiO₂ coated silicon, and glass, and Paint #2002 on glass.

III. Electrical Resistivity Measurements

The paint samples were prepared as described in Section II, however, the initial substrate was a 2 inch diameter SiO₂ coated silicon wafer where a region 0.5 x 0.5 inches near the center of the substrate was isolated to yield a relatively uniform film for electrical measurements.

The electrical resistance of the thin film was measured, where a schematic diagram of the resistance measurement system is seen in Figure 4. The electrical contact to the sample were arranged in the van der Pauw configuration, which allows the electrical resistivity of the film to be calculated from the relation:

$$\rho = [(\pi d)/\ln 2](R_1 + R_2)f/2$$

where d is the film thickness as measured by a mechanical stylus, and f is a geometrical factor. The resistance R_1 is the value of film resistance obtained from the initial measurement, while R_2 is obtained after rotating the sample by 90° with respect to its initial position in the measurement system. The resistivity of Paint #3003 was determined to be $43 \mu\Omega\cdot\text{cm}$.

Figure 4. Schematic diagram of thin film resistance measurement system.

A complicating factor in accurate resistivity measurements of this type of material is the surface roughness as seen in the mechanical stylus scan in Figure 5. The average thickness of the film is $\pm 5\%$, however, the roughness is also of this order. The effect of this on the measured value of film resistance is not entirely clear at this time. We will attack this problem by attempting to improve film roughness through polishing, examining current literature concerning electrical measurements on this type of material, and if necessary developing mathematical models that account for roughness in resistivity measurements.

Figure 5. Mechanical Stylus scan of a typical paint film showing the surface roughness.

IV. Next Phase

We will prepare samples covering Paint #'s 1001, 2002, 3003, 4004, on each of the following substrates; silicon, glass, SiO_2 , Delrin, Teflon, and Aluminum Silicate. The paints will be processed at 100, 200, and 300 C in forming gas. The adhesion strength of each sample will be measured, and compared with each other. Further, these results will be compared with samples prepared from Ernest F. Fullam Silver Paint, and G-C silver Paint.

We will also measure the electrical resistivity of these samples, and continue our efforts, as discussed above to improve the surface uniformity to provide more accurate measurements.

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