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V. K. Nagesh
(M. S. thesis)

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ELECTRICAL CONDUCTION IN METAL-Glass COMPOSITE MATERIALS

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ELECTRICAL CONDUCTION
IN METAL-GLASS COMPOSITE MATERIALS

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ABSTRACT

The importance of the surface characteristics of the glass phase used in the development of conducting thick film systems has been investigated. The system studied was the lead borosilicate glass-silver system. By surface activation of the glass particles, a conducting thick film microstructure has been developed using a reduced amount of silver compared to commercial compositions. Thick films with resistivities of the order of 75 milliohms/square (mΩ/□) with a silver content 30 wt.% (w/o) have been obtained. This resistance is comparable to the 50 milliohms/square obtained with 80 wt.% Pt in Pt-Pd commercial systems.
I. INTRODUCTION

Hybrid integrated circuits are becoming increasingly important as a result of the miniaturization of electronic components, circuits, and systems. Thick films are extensively used as capacitors, conductors, and resistors in these hybrid circuits. They are also used in high power T. V. circuits, microwave devices and other integrated circuit applications. Thick films have been used not only because they are reliable but also because they are economical.

Conductor thick films are essentially films of a composite of glass and noble metals on a ceramic substrates, having a thickness of the order of a few mils. The Glass Content is small (~15 weight %) in conventional films, and its purpose is to provide good bonding between metal and substrate. Thick films are differentiated from thin films both in mode of preparation and in their thickness. Thin films are essentially of a thickness ranging from a few thousand Å to a few microns and are normally vacuum evaporated, sputtered or chemically deposited.

Even though a considerable amount of development work has been done, little research to obtain a basic understanding of the conducting mechanisms is reported. A brief review of the basic research done in this field is given below.

The mechanism of conduction has been studied in thick film resistor systems (RuO₂ crystal, Ru resinates Pd-Ag system) under the application of high electromagnetic stress by Polinski. It is predicted that interfacial energies between glass and the conductive phase cause the glass
particles to sinter together and allow the conductive phases to form a channel. The resistance would then depend on the amount of discontinuity of this channel. Increase of electromagnetic stress is found to increase the conductivity.

Sintering studies have been conducted in the silver-palladium-glass system by Cole. These have led to certain important conclusions. Selective solution and reprecipitation is found to be the reason for the necessity of close control of both peak furnace temperature and time of firing. Heating rates and particle size are also found to be very important in determining the final properties of the film. Knowledge of intrinsic properties of the glass have been thought to be important because changes in silver and palladium solubility and changes in contact angles will have profound affects on the film properties.

The interrelationships of surface oxidation and solderabilities on silver-palladium thick film conductors and the variations of conductivity characteristics with the metal content have been studied by Kubota and Shinmura. With the increase of the palladium content silver migration rates are found to increase whereas the cohesive strength of the solder joints are found to decrease.

Adhesion, phase morphology, and bondability of reactively bonded and frit bonded gold and silver thick film conductors as a function of firing conditions and material types have been studied by Hitch. From SEM studies of the interfaces of the binding phases in the reactively bonded silver and gold inks, it was concluded that the main force of adhesion between the metal and the substrate was due to chemical bonding rather than mechanical interlocking. He did not, however, discuss the affect
of the glass phase in his 96 w/o alumina substrates.

The development and study of the microstructure of the conducting thick films are very important. Studying the microstructure would give an insight of the processes at the microlevel and a knowledge of how to develop new thick film systems with more reliable properties and if possible of making it more economical.

The present investigation was undertaken with an objective to develop a microstructure for a conducting thick film using the glass-metal composites, lead borosilicate glass - silver-metal system.

Conventionally, conducting thick films having a mixture of glass and metal particles in a suitable proportion in an organic vehicle with a suitable viscosity are printed onto a substrate. (Fig. 1) The glass usually a low softening glass, with a composition giving an expansion coefficient less than that of the substrate (96 w/o $\text{Al}_2\text{O}_3$) is powdered to a micron size* and mixed with the micron size* metallic powder (which is normally a noble metal). Usually the metal content varies from 70 to 90 w/o of the solids. This mixture is then suspended in an organic vehicle. The organic consists of a resin binder, solvent and additives to give a suitable viscosity. The liquid ratio is normally ~75 w/o depending on the viscosity. The liquid-solid mixture is screen printed onto an alumina substrate. The system is dried and fired for a specific time at a certain temperature to give a conducting thick film.

Conventionally, the purpose of the glass is to provide good bonding between the substrate and the metallic film. The microstructure of the fired system would have metallic particles in contact with one another.

---

*1 $\mu$m - 10 $\mu$m range
and the glass phase would be interspersed (Fig. 2a). It is obvious that the same degree of conductivity could be attained with a reduced metal content and the proper microstructure. If the glass particles could be coated with a precious metal like Ag, uniformly and with sufficient bonding, it is possible to get a microstructure necessary for a conducting film (Fig. 2b). This would reduce the metal content to a large extent and glass phase would be the major phase in the composite system. Surprisingly, nothing has been mentioned in the literature about getting this kind of a microstructure or making the glass particles surface active for this purpose. To achieve good glass to metal bonding, it would be advantageous to have the glass particles made surface active. The metal (here silver) can then be deposited chemically giving a uniform and strong coating of the metal around the glass particles. With these points in mind, the present investigation of developing a thick conducting film with as small a metal content as possible, was undertaken.
II. EXPERIMENTAL DETAILS

The general block diagram for the new process of obtaining conducting thick film, is given in Fig. 3. This process uses considerably less metal than the conventional thick film conductors. Details of the process are given below.

A. Glass Preparation

The glass used was a lead borosilicate glass, whose softening temperature is quite low (approximately 450°C). The matching of thermal expansion coefficient of the substrate and the glass that is used, is very essential to avoid any thermal stresses that may develop after firing. The contributions of the different component oxides to the thermal expansion coefficient of the glass was determined, by finding the expansion coefficient for glasses of different compositions using a dialatometer. The composition of the glass used for the thick film system was chosen to give an expansion coefficient equal to that of alumina (~8 X 10^-6/°C). The composition was 70 wt% PbO, 20 wt% B_2O_3, 10 wt% SiO_2.

The glass was powdered using alumina balls in an organic lined mill with isopropyl alcohol as the liquid medium. After milling the glass was size separated and a particle size fraction between 20 and 44 μm was used for all experiments.

B. Surface Activation

The surface treatment essentially involves surface reactions with dil. HCl (.01N HCl) for a given time. A known weight of glass particles was etched for different times and the percentage weight loss was recorded. The weight of powder used was nearly the same (~3gms) for each
treatment and the amount of acid used was 50c.c.

A plot of the percentage weight loss vs. time is shown in Fig. 4.

C. Thick Film Conductor Formulation and Application

The surface active glass powder was mixed with a silver resinate [15 w/o Ag, in a solvent (xylene)] manufactured by Western Gold & Platinum Co. In the usual run 3-5 gms. of glass were used. The metal content of the glass-metal mixture was controlled by controlling the amount of silver resinate used (10 gms. of solution gives ~.15 gms. of Ag). The mixture was heated to 80°C to evaporate the solvent. After this appropriate amount (~1.2 gms. ) of the organic vehicle (manufactured by Drakenfield colors, Oil 1014) was added to form a paste with the proper viscosity for screen printing.

A stainless steel screen (mesh size 200) and squeegee were used to apply thick films of a definite pattern (Fig. 5) onto alumina substrates (96% alumina, 1" X 1" X 0.030").

D. Firing

After printing, the printed substrates were dried in air for 15 minutes and then fired in air. The temperature program consisted of raising the temperature at 16°C/min to the firing temperature, holding and allowing the substrate to furnace cool. Films were fired at different temperatures for different times.

E. Electrical Resistivity

The electrical sheet resistivity was measured using four point probe method. The circuit diagram is shown in Fig. 6. The voltage drop across the specimen was determined at currents from 4.53 to
15 mA. Typical data (Fig. 7) indicate a linear variation of current with voltage. The voltage (in μV), at a current 4.53 mA is numerically equal to the sheet resistivity in mΩ/□. For thick films it is customary to define a quantity called "sheet resistance", $R_s$, which is equal to $\rho/d$, where $\rho$ is the resistivity and $d$ is the thickness of the film. As the film is essentially 2-dimensional, $R_s$ can be considered to be a material property. Sheet resistivity is conveniently measured using the four point probe method, wherein four contacts are made to the film and current is injected and taken out through the end contacts. Voltage drop is measured between the two middle contacts. The sheet resistance is proportional to the ratio of the voltage, $V$ to the current, $I$.

That is,

$$R_s = C(V/I)$$

where $C$ is a constant of proportionality that depends on the configuration, position and orientation of the probes, shape and size of the sample. For an infinite film $C = \frac{\pi}{\ln^2} = 4.5324$. If the ratio of the length of the film to the distance between the probes $\sim 100$, then $C$ would be equal to 4.53 and when the ratio is of the order of 10, $C$ would be 4.36.

Hence, when the current is made equal to 4.53 A, the measured volts would directly give the sheet resistance in ohms per square.

G. Microstructural Analysis

For microstructural analysis, optical microscopy and scanning electron microscopy were used. Standard techniques for preparing the specimens for microstructural observation were followed. For obtaining the scanning electron micrograph of the etched glass surface, the surface coated with $\sim 200$ Å of gold to eliminate charging affects.
In optical micrographs, a better phase contrast was obtained relative to scanning electron micrographs.
III. RESULTS AND DISCUSSION

A. Glass Composition

Table I gives the thermal expansion data for various glasses compounded at the start of this study. From this data, contributions to the thermal expansion coefficient of the glass by the constituent oxides was calculated.

\[ \alpha_{\text{glass}} = p_{\text{PbO}} \alpha_{\text{PbO}} + p_{\text{B}_2\text{O}_3} \alpha_{\text{B}_2\text{O}_3} + p_{\text{SiO}_2} \alpha_{\text{SiO}_2} \]

where \( p \)'s are the percentage by weights of the component oxides of the glass and \( \alpha \) is the contribution of each individual component. From this it was possible to select a glass composition giving a glass of \( \alpha \) less than that of alumina.

B. Etching Characteristics

Figure 4 shows a plot of percentage weight loss vs. the time of etching. Initially, the plot is linear and with increase of time of etching the rate of wt. loss decreases. This is understandable because the amount of acid is a constant volume (50 cc) and the activity of the acid reduces with time during reaction. 2.8 wt/o loss corresponds to an etched depth of 0.25\( \mu \)m for an average particle size of 30 \( \mu \)m. The surface of an etched glass particle as seen by scanning electron microscopy is shown in Fig. 8. The surface is shown to contain many micropores. These micropores are generated during etching of HCl. It has been found\(^{12} \) that high lead glasses containing \( \text{SiO}_2 \) do not exhibit a homogeneous structure, but are essentially two phase. There will be a droplike microdispersed phase in a silica rich phase. Etching with the acid dissolves the low silica phase leaving micropores in a high \( \text{SiO}_2 \) skeleton.
C. Film Firing

Films were fired in an air atmosphere, as previously described. From thermogravimetric analysis the resinate was found to decompose at 210°C giving metallic silver. During air firing, silver resinate decomposes to give silver, which coats the glass particles uniformly. At higher temperatures, the particles become relatively fluid and deform to develop an interconnecting metallic film surrounding the glass particles. Firing at different temperatures and times is found to have an important effect on the final conductivity. Hence, different temperatures ranging from 500°C to 800°C and times from 8 to 30 minutes were used. Thick films formulated with the untreated glass particles were also made following similar procedures of printing, drying and firing.

The microstructures obtained from optical microscopy, after the specimens were polished are shown in Fig. 9. For the surface treated glass, Fig. 9(a), and the untreated glass, Fig. 9(b), the firing was the same (600°C, 8 minutes). The silver content was 25 w/o in both films. Film (a) gave good conduction and (b) did not. This is understandable from the microstructure, as there is a good continuity of the metal in the treated glass thick film conductor. The untreated glass thick film had a high resistance and as shown in Fig. 9(b), the microstructure indicates agglomerated silver metal in a glass matrix. The sheet resistivity of thick film shown in Fig. 9(a) was 160 mΩ/□. This value compares favorably with a commercial Pt-Pd thick film which has about 80 wt/o Pt and a sheet sensitivity of 50 mΩ/□.

Hence, there is a good indication that by surface treating the
glass particles and making them surface active, the microstructure intended is being developed.

D. Analysis of Surface Reactivity

Silver metal is bound to the surface of the treated glass particles and during firing, these particles viscously flow together without appreciably affecting the metal configuration in the microstructure.

When the glass particles are etched, as mentioned earlier there will be micropores developed. In addition, the activity of the surface is increased by an ion exchange reaction. The ion exchange reaction proposed is between the H⁺ ion in the acid and Pb²⁺ ion in solution in the glass;

\[ \text{Pb}^{2+} \text{(in glass)} + 2 \text{H}^+(\text{soln}) \rightleftharpoons \text{Pb}^{2+} \text{(soln)} + 2\text{H}^+(\text{in glass}) \]

The hydrogen ions will make the surface of the glass particles active as they can enter into another ion exchange reaction. When silver resinate is added, an ion exchange between Ag⁺ ions from the resinate and H⁺ ions in the glass takes place. Elevated temperatures (80°C) increase the reaction rate. The kinetics of the ion exchange of resins indicate that ion exchange would not be favorable if the ion that has to be exchanged has a higher valence than the ion in the fluid medium. This indicates a reduced probability of ion exchange between Pb²⁺ in the glass and Ag⁺ in solution.

As a result of the previous ion exchange and formation of a surface active glass the following reaction can occur

\[ \text{Ag - resinate} \text{(in solution)} + \text{H}^+(\text{in glass}) \rightleftharpoons \text{H - resinate} + \text{Ag}^+(\text{in glass}) \]

The surface of the glass powders would have a high concentration of Ag⁺ ions and the glass would be saturated with Ag⁺ at the surface. When
the residual resinate is decomposed (at 210°C, as determined by TGA), during firing, the silver metal formed can form a good bond to the glass particles. According to the theory developed by Pask and Fulrath, the essential condition for good bonding between glass and a metal is that there should be continuity of chemical bonding at the interface. For this, saturation of the glass by the metal ion is a necessary condition. In the surface inactive powder, there is insufficient silver implanted and hence glass-metal bonding and probably wetting characteristics are adversely affected. Therefore, agglomeration and discontinuous Ag islands will be promoted as seen in the microstructure (Fig. 9(b)) giving rise to high resistivity.

To find experimental evidence for the proposed ion exchange mechanism, the following experiment was conducted. Two glass powders, surface active and passive (20-44μm), were reacted with silver resinate at 80°C for 90 minutes. The mixture was filtered and the residual glass particles were washed well with isopropyl alcohol to remove unreacted silver resinate. After drying the powders, an X-ray fluorescence spectra was obtained (Fig. 10). The active powder gave peaks corresponding to silver Kα and Kβ lines. Spectra of the surface passive powder did not show any peaks for silver. This result strongly supports the proposed ion exchange mechanism.

Table II gives the sheet resistivities of the different thick films obtained with surface active glass powder, and with varying metal contents. The sheet resistivities vary from 40mΩ/ to 230Ω/ where as commercial thick films have sheet resistivities varying from 10mΩ/ to 50mΩ/ for
silver-palladium systems with silver content varying from 70 w/o to 90 w/o. When the metal content is increased, the resistivity decreases fairly linearly. Thick films using surface passive powders gave were essentially non-conductors ($\Omega/\square > 10^6$).

As mentioned by several authors, firing parameters have an important affect on the final resistivities. In this study there is little change in resistivity with increase in temperature of firing from 500°C to 700°C. Firing at higher temperatures changes the resistivity drastically. Films fired at 800°C, show no conduction. At this temperature the microstructure developed for conducting films breaks down, due to a tendency for the silver to agglomerate (Fig. 11), and also the possibility of the increase in the solubility of silver as $\text{Ag}^{2+}$ in the glass. Firing for longer times at lower temperatures will increase the resistivity for reasons similar to short times at higher temperatures. Using the scanning electron microscope hot stage the temperature at which the viscous flow of the glass particles become effective was found to be 600°C. Hence, by developing a suitable microstructure, making use of the technique of surface activation of the glass resistivities comparable to those of commercially available conducting thick films are obtained with reduced precious metal content.
CONCLUSIONS

It has been shown that the surface characteristics of the glass particles are extremely important in developing a thick film microstructure conducive for electrical conduction. Conductivities of the order of $\approx 50 \, \text{m\Omega/\square}$ with metal content as low as 30 w/o have been obtained. Commercial thick film conductors use as much as 80 w/o of precious metals to achieve conduction in the same range. Hence, the conducting thick films have been made very economical by the process developed.
ACKNOWLEDGMENTS

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Finally, I am grateful to my parents for their help and encouragement.

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REFERENCES


Table I. Thermal Expansion Data of Lead Borosilicate Glasses

<table>
<thead>
<tr>
<th>No.</th>
<th>SiO₂</th>
<th>PbO</th>
<th>B₂O₃</th>
<th>α_{glass} (in./in./°C ×10⁶)</th>
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</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>80</td>
<td>15</td>
<td>9.8</td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>80</td>
<td>5</td>
<td>10.0</td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>70</td>
<td>20</td>
<td>8.3</td>
</tr>
<tr>
<td></td>
<td>-0.33×10⁻⁷</td>
<td>1.33×10⁻⁷</td>
<td>-0.33×10⁻⁷</td>
<td></td>
</tr>
</tbody>
</table>

α_{glass} = α_{PbO}PbO + α_{SiO₂}SiO₂ + α_{B₂O₃}B₂O₃
Table II. Sheet Resistivity of Silver Glass Thick Film Systems

<table>
<thead>
<tr>
<th>Silver Metal Content W/o</th>
<th>Surface Treatment (45 sec.)</th>
<th>Firing Temp.</th>
<th>Firing Time Min.</th>
<th>Resistivity mΩ/□</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>0.25 N HCL</td>
<td>550</td>
<td>8</td>
<td>44</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>575</td>
<td>14</td>
<td>32</td>
</tr>
<tr>
<td></td>
<td>0.01 N HCL</td>
<td>500</td>
<td>20</td>
<td>80</td>
</tr>
<tr>
<td></td>
<td>&quot;</td>
<td>500</td>
<td>30</td>
<td>120</td>
</tr>
<tr>
<td>30</td>
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<td>600</td>
<td>8</td>
<td>85</td>
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<td></td>
<td>&quot;</td>
<td>640</td>
<td>8</td>
<td>70</td>
</tr>
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<td></td>
<td>&quot;</td>
<td>700</td>
<td>8</td>
<td>80</td>
</tr>
<tr>
<td>25</td>
<td>0.01 N HCL</td>
<td>500</td>
<td>8</td>
<td>160</td>
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<tr>
<td></td>
<td>&quot;</td>
<td>600</td>
<td>8</td>
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<td>700</td>
<td>8</td>
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</tr>
<tr>
<td>20</td>
<td>0.01 N HCL</td>
<td>500</td>
<td>30</td>
<td>230</td>
</tr>
</tbody>
</table>
FIGURE CAPTIONS

1. Flow diagram for conventional thick film conductor processing.

2. Schematic diagram showing conventional and intended microstructure for conducting thick films.


4. Weight loss characteristics of the glass particles (20-44μm) treated with 0.1N HCl for varying times.

5. Silk screen and squeegee used for printing thick films on alumina substrates.


7. Voltage - current characteristics of the conductive thick film.

8. Surface of an etched lead borosilicate glass as seen by SEM (radius of the pores <1μm).

9. Transmission optical micrographs of the thick films with 25 w/o Ag (Firing: 600°C for 8 minutes).
   (a) With surface active glass powder (20-44μm)-Here interconnected metallic phase (dark phase) gives rise to electrical continuity.
   (b) With surface passive glass powder-Discontinuous metallic phase (dark phase) gives rise to high resistance.

10. X-ray fluorescence spectra of silver resinate treated glass powders, both surface active and passive.

11. Transmission optical micrograph of a high temperature fired thick film using surface active glass powder (25 w/o Ag, 800°C 8 minutes).
PROCESSING OF CONVENTIONAL THICK FILM CONDUCTORS

(precious) METAL POWDER 70\%, MICRON SIZE

GLASS POWDER (PbO·SiO₂·B₂O₃·Al₂O₃, etc.) 30\%, MICRON SIZE

MIXING AND MILLING

ORGANIC VEHICLE 80\% SOLID, 20\% LIQUID

BY SCREEN PRINTING, PAINTING, ETC.

APPLIED ONTO A SUBSTRATE (96\% Al₂O₃)

FIRED

THICK FILM CONDUCTOR

XBL 759-7372

Fig. 1
CONVENTIONAL MICROSTRUCTURE
METAL PARTICLES IN CONTACT WITH EACH OTHER. GLASSY PHASE BOUND IN BETWEEN.

INTENDED MICROSTRUCTURE
GLASS PARTICLES WITH A METALLIC COAT IN CONTACT WITH EACH OTHER.

XBL 759-7368

Fig. 2
NEW PROCESS FOR INTENDED MICROSTRUCTURE

GLASS POWDER
70PbO-10 SiO₂-20 B₂O₃

SURFACE TREATMENT

SURFACE ACTIVE GLASS POWDER

SILVER METAL RESINATE

MIX AND HEAT
GLASS 75%, METAL 25%

MIX WITH ORGANIC VEHICLE

APPLY ONTO A SUBSTRATE

FIRE

THICK FILM CONDUCTOR

XBL 759-7369

Fig. 3
GLASS 70 PbO-10 SiO$_2$-20 B$_2$O$_3$ w/o (20-44 μm) 0.01 N HCl

Fig. 4
Fig. 5
CIRCUIT DIAGRAM

Standard Resistance \approx 10 \, \Omega

Voltage Supply

Fig. 6
V-I CHARACTERISTICS OF CONDUCTIVE THICK FILM

FIRING
600°C - 8 min.

30 \text{%} Ag

25 \text{%} Ag

I (mA)

V (\mu V)

XBL 765-6866

Fig. 7
THICK FILM OPTICAL TRANSMISSION MICROGRAPHS
(25\% Ag, FIRING 600°C, 8 MIN.)

(a) SURFACE ACTIVE GLASS POWDER

Fig. 9(a)

XBB766-4725
THICK FILM OPTICAL TRANSMISSION MICROGRAPHS
(25% Ag, FIRING 600°C, 8 MIN.)

(b) SURFACE PASSIVE GLASS POWDER

62 μm

XBB766-4726

Fig. 9(b)
X-RAY FLUORESCENCE OF SILVER RESINATE TREATED GLASS POWDER
(20–44μ)

1. SURFACE ACTIVE GLASS POWDER

2. SURFACE PASSIVE GLASS POWDER

Fig. 10

XBL 759-7370
THICK FILM OPTICAL TRANSMISSION MICROGRAPH
(25% Ag, FIRING 800°C, 8 MIN.)

SURFACE ACTIVE GLASS POWDER

XBB766-4724

Fig. 11
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