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Reliability Performance of Conductive Ink Subjected to Hygrothermal Aging

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Abstract:** In the era of rapid technological development, the popularity in printed technologies and electronic packaging have resulted in a tremendous increase in the use of carbon-based conductive ink due to their advantageous features such as being environmental-friendly, low cost and lower assembly temperature. From the literature, it has been highlighted that the interconnect material are exposed to some degree of humidity and elevated temperature during the service life in an actual application. To-date, there is not yet a great length of literature reporting on the reliability performance of such materials when exposed to hygrothermal aging. Therefore, the objective of this research work is to investigate the reliability performance of the conductive ink when subjected to hygrothermal aging. In this study, the samples were exposed to either the room temperature condition with temperature of 20°C and humidity of 60% RH and secondly when subjected to hygrothermal aging in an environmental chamber with humidity of 85% RH and a temperature of 85°C up to 24 hours. Following these, the samples were tested in terms of electrical conductivity using a four-point-probe (ASTM F390) and lap shear test (ASTM D1002) via tensile loading to evaluate the bonding strength at the interface between the adhesive and the substrate. Next, morphological study was done using Scanning Electron Microscopy (SEM). With the presence of water molecule in the conductive ink, the molecule of carbon black and epoxy become unstructured and traces of riverlines are evident. In addition, there is a dramatic decrease in the sheet resistance following hygrothermal aging relative to the samples conditioned at room temperature, possibly due to enhancement in the conductivity of the ink. In contrast, as for the mechanical shear stress, the lap shear stress following hygrothermal aging process becomes weaken compared to those conditioned at room temperature, which could be associated with weak surface energy, brittle and weak bonding between carbon black molecules and the aluminium substrate interface.

***Keywords:** Reliability; Performance; Conductive Ink; Hygrothermal Aging*

I.INTRODUCTION

Over the past decades, the use of electrically conductive adhesive (ECA) has expanded rapidly into the microelectronics industry. The application of ECA can be seen in liquid crystal display (LCD), die attachment, and surface-mounted for assembly component on printed wiring board (PWB).

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Besides, the ECA can be used as a glue that hole the electronic component in the correct place while passing the electric between them. Furthermore, ECA can be regarded as a replacement for conversional solder, sealing, electrical shielding, interconnection between components, fastening, component bonding and brazing [1].

ECA consists of polymer matrix and conductive metallic fillers, in which typically, epoxy resin is employed as the matrix of choice and silver as a filler, since these materials are environmental-friendly properties and does not cause health hazard to human being as compared to other types of materials.

However, when exposed to thermal and humid environment, it was argued that corrosion formed on the contact interface caused by the water gain in the surrounding, stress concentration by the surrounding and the failure connection between devices etc. Could affect the electrical resistance and reduce the bonding strength between them [2]. To-date, the trend for miniaturization that is aiming for minimizing the size of electronic device has led to rapid heat generation or energy while operating the device. Therefore, it is crucial to remove the effective heat or unwanted energy from the origin to increase the performance and the reliability of the electronic devices in this era [3].

Studies on the behaviour and properties of the material provides more understanding about these electrically conductive materials, or conductive ink when they are used in the extreme environments such as temperature, sunlight, moisture, ice, radiation, chemical, mechanical stress and combinations of these factors [4]–[6]. In the literature, degradation process is reported to be influenced by several factors, such as the aging temperature and time, [4].

Meanwhile, hygrothermal diffusion behaviour can be affected by the nature and hydrogen bonding of the network due to an increased polarity of composite and the water uptake which can cause chemical degradation. Besides that, understanding of hygrothermal ageing effect on the degradation of the material or adhesive joint is essentially important before they can be applied for any engineering applications [7].

In addition, such interconnect material offers greater convenience for the application and development of lightweight vehicles, submarines and also for the aviation industry. Furthermore, selection of proper adhesive joint will allow absorption of the impact energy therefore reducing noise and vibrations of a structure or component [8].

Hence this research aims to investigate the reliability performance of conductive ink when exposed to hygrothermal aging process for 24 hours with the humidity of 85% RH and a temperature of 85°C. Then the electrical conductivity and lap shear test will be testing.

II.EXPERIMENTAL

The experimental work begins with sample preparation using the carbon black-filled epoxy conductive ink. Bare Conductive Electric Paint is used as a conductive ink, a type of adhesive which is a non-toxic, water based and electrically conductive adhesive as Table 1.

Sample preparation for the study begin with the screen printing for electrical resistivity by using a four-point probe in accordance with the ASTM F390 as Fig 1. Then the sample was place into the curing oven and heated with the temperature of 100°C for 30 minutes and was taken out to cure for 24 hours at room temperature. Then ready for the four-point probe test to measure the conducive ink sheet resistance for room temperature condition.

Meanwhile for lap shear testing as per ASTM D1002 as Fig 2, the conductive ink sample should have a dimension 1.62 ± 0.125 mm thickness and have overlap length of 12.7 ± 0.25mm on the substrate. Next the sample were gripped with the metal clip to ensure that the thickness of conductive ink is homogenous between the aluminium substrates as show in the Fig 3. Then the sample were palce in the curing oven and heated at a 100°C for 30 minutes and were drown out from the oven and underwent for the curing process at the ambient temperature for 24 hours.

Table. 1 Properties of Bare Paint [9]

Color	Black
Viscosity	Highly viscous and shear sensitive
Density	1.16 g/ml
Surface resistivity	55 Ω/sq @50 microns
Composition	Water, conductive ink natural risin

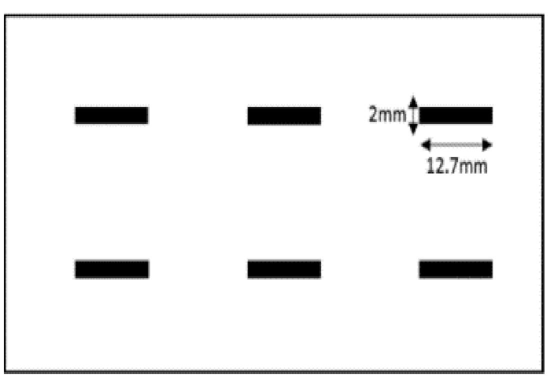

Fig. 1 6 printed inks on a polycarbonate (PC) substrate

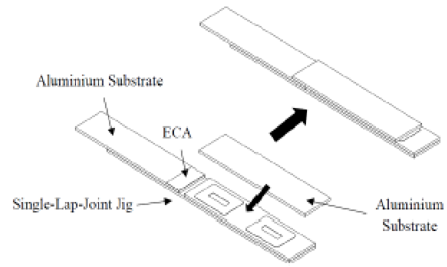

Fig. 2 Process to fabricate aluminium single –lap-joint adhesively bonded[10]

Fig. 3 Single-lap-joint adhesively bounded sample are ready put on the oven for curing process

For the hygrothermal aging test, the samples were placed at either room temperature condition (20°C/60% RH) or in an environmental chamber at the specified temperature and humidity (85% RH/85°C) for 24 hours.

The electrical characterization in term of the electrical conductivity were be measured by the resistivity test. By using the Eq. 1, sheet resistance can be calculated as follow: -

$$R_s = G\frac{V}{I} \quad (1)$$

Where R_s is sheet resistance($\frac{\Omega}{sq}$), G is correction factor = 1.9475, V is voltage and I is current. The test was done using JANDEL In-Line Four-Probe following the ASTM F390.

The purpose for mechanical test is to evaluate the shear strength of the conductive ink where the high shear strength reflects a good bonding strength and strong mechanical interlocking of conductive ini.

The lap shear test can be calculated using Eq. 2 as follows: -

$$\tau_{LST} = \frac{F_{Max}}{A} \quad (2)$$

Where τ_{LST} is lap shear test in MPa, F_{Max} is maximum tensile force in N and A is adhesive overlap area in m^2. The overlap area is calculated using the formula in Eq. 3 as follow:

$$A_{overlap} = L \times W \quad (3)$$

Where $A_{overlap}$ is adhesive area in m^2. L is length of overlap area in m and W is width of overlap area in m. From the Eq. 3 area for overlap was 322.58mm^2.

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The test grip area was joint with the small aluminium plate to ensure that the load is subjected at the center during the lap shear test as Fig 4. By using the universal Material Testing (AG-10kNX) under tensile mode the lap shear test was done accordance with the procedure ASTM D1002.

In addition, morphological study using a Scanning Electron Microscopy (SEM) is conducted to evaluate the hygrothermal aging effect on the conductive ink as well as to understand the surface profile with and without hygrothermal aging effect.

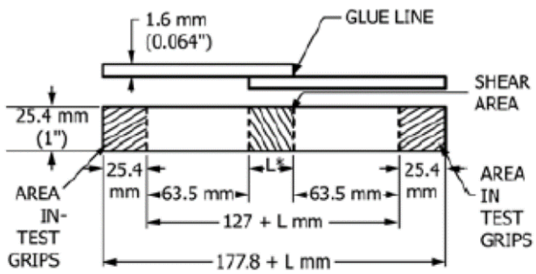

Fig. 4 Dimension of single-lap-joint Bonded[11]

The specimen was cutting in the small pieces and was placed inside the sample holder. The specimen then sputtered by the conductive material to create the negative cathode using the auto-fine coater machine as Fig 5. Then the specimen is placed inside the SEM as Fig 6.

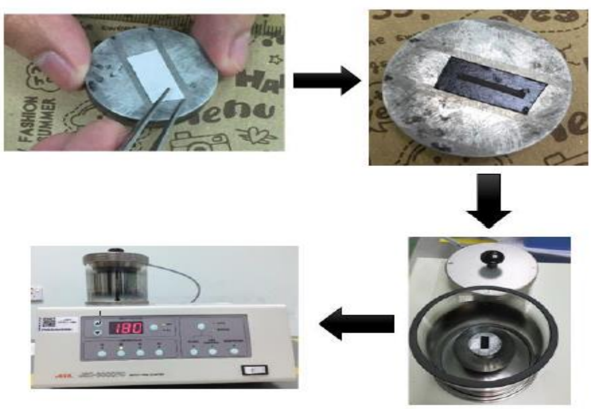

Fig. 5 Coating process before enter SEM machine

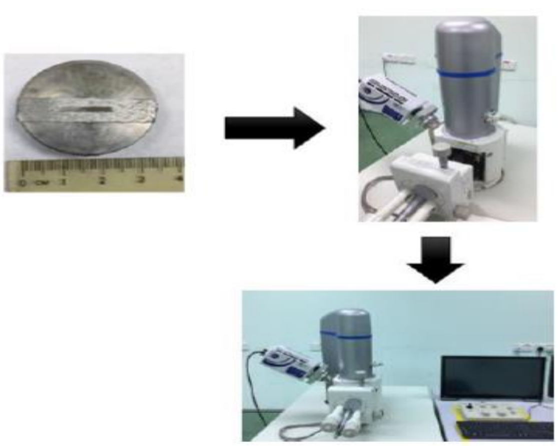

Fig. 6 Specimen placed in SEM

III.RESULTS AND DISCUSSION

3.1 Electrical Properties

Electric performance of conductive ink is discussed in terms of the sheet resistance measurement with and without hygrothermal aging up to 24 hours at temperature and humidity 85°C and 85% RH respectively which reflects the reliability performance when subjected to hygrothermal aging during service as interconnect material for electrical component.

From the study, based on Fig 7, the average sheet resistance for room temperature is 137.9351 ± 7.6424 Ω/sq. and for hygrothermal aging is 17.0737 ± 0.5091 Ω/sq. these observations suggest that the electrical resistivity of carbon black are strongly affected by structure of material change, surface area beside several other factor [12]–[14].

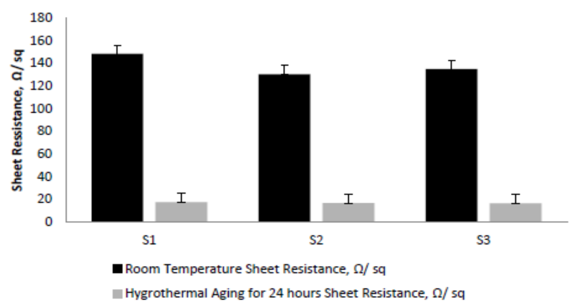

Fig. 7 Differentiation of sheet resistance between room temperature and hygrothermal aging for 24 hours

Besides the qualitative analysis of the sample was also considered in which selected sample were analyzed using Scanning Electron Microscope (SEM) to clarify the surface condition for room temperature electrical characterization as well as following hygrothermal aging of the conductive ink. The SEM are show in Fig 8 to Fig 11 with varying magnification during an analysis.

Based on the figure, there is a different structure of the conductive ink surface when subjected to electrical characterization at room temperature and hygrothermal aging. The image suggests that there is a relatively an even surface for the conductive ink composite which is blend of the carbon and epoxy with smooth surface condition referring to Fig 8 and Fig 9.

However, when subjected to hygrothermal aging for 24 hours at 85°C and 85% RH, the presence of moisture which is water molecule attach in the surface of the conductive ink both the carbon black and epoxy become unstructured and trace of river line can see clearly as in Fig 10 and Fig 11.

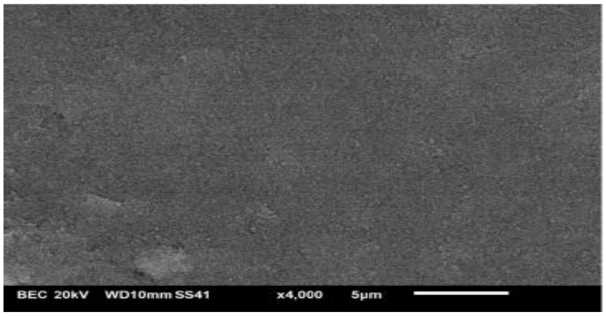

Fig. 8 SEM micrograph showing top view of the conductive ink following electrical resistivity test at room temperature condition at 4000x (backscattered-electron image)

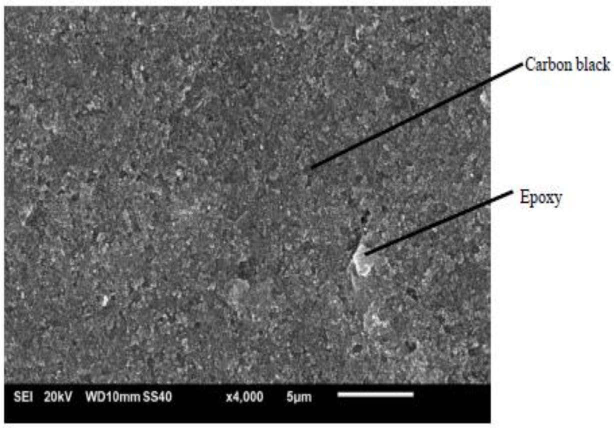

Fig. 9 SEM micrograph showing top view of the conductive ink following electrical resistivity test at room temperature condition at 4000x (secondary electron image)

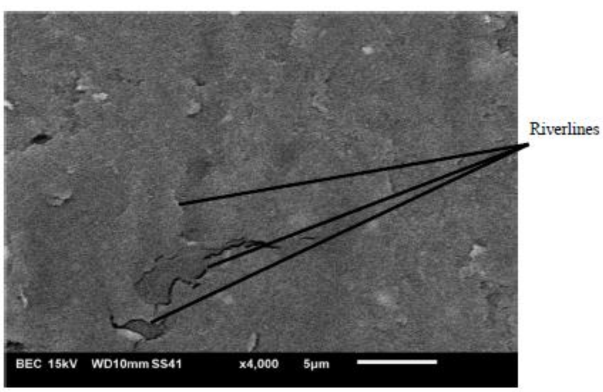

Fig. 10 SEM micrograph showing top view of the conductive ink following electrical resistivity test at humidity chamber condition at 4000x (backscattered-electron image)

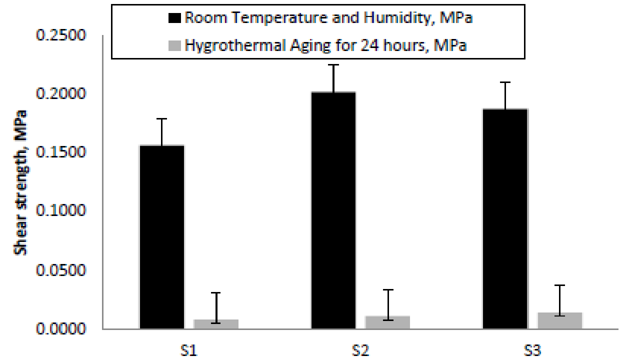

Fig. 11 SEM micrograph showing top view of the conductive ink following electrical resistivity test at humidity chamber condition at 4000x (backscattered-electron image)

3.2 Mechanical Characterization

Referring to the Fig 12, the data shows there is different between lap shear strength of conductive ink sample when subjected to room temperature and hygrothermal aging condition. The shear strength shows a decrease from 0.1816 ± 0.0230 MPa for room temperature to 0.0108 ± 0.0032 MPa for hygrothermal aging, that is almost 8 fold due to moisture attact when exposed to hygrothermal aging which humidity and temperature is extreme high [15]. Otherwise, the decrease of the this due to the week surface energy, brittle behavior and the week bonding between the black carbon filled conductive ink molecules and the aluminium substrate at the joint [16]–[18].

Beyond that, the strength and reliability of the adhesive become worse and change of the microstructure led to lower performance.

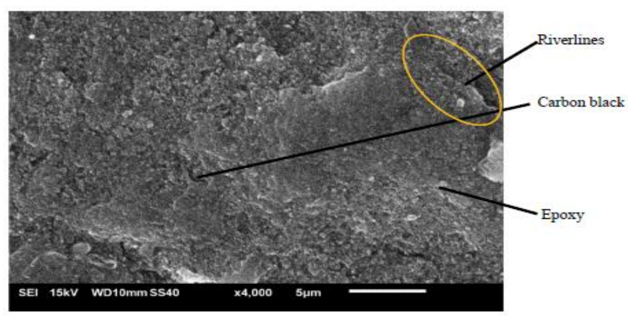

Fig. 12 Shear strength of the conductive ink when conditioned at room temperature and hygrothermal aging for 24 hours

From the observation is supported by qualitative analysis on the selected sample morphology, when subjected to lap shear test following room temperature and hygrothermal aging condition for 24 hours respectively. Fig 13 and Fig 14 shows the sample structure at room temperature. Meanwhile Fig 15 and Fig 16 for hygrothermal aging condition.

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Similar to the electrical characterization test, the presence of riverlines are evident in the sample following to hygrothermal aging, with the presence of moisture or water attach when compared without any aging defect.

The shear strength for the black carbon decreased from room temperature to the hygrothermal aging condition due to wettability of the liquid occurring during the high humidity environment. Plus, the brittle bonding between black carbon and aluminium[18].

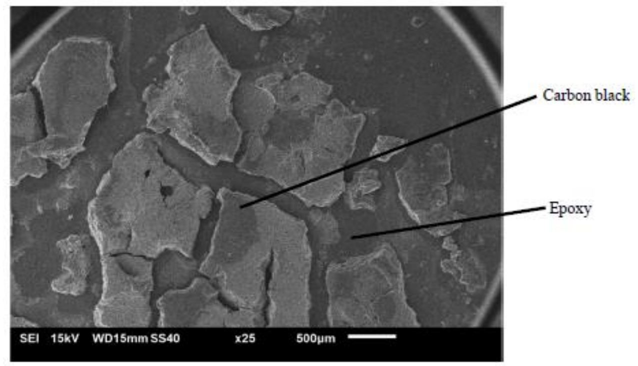

Fig. 13 SEM micrograph showing top view of the conductive ink following lap shear test at room temperature condition at 25x (secondary electron image)

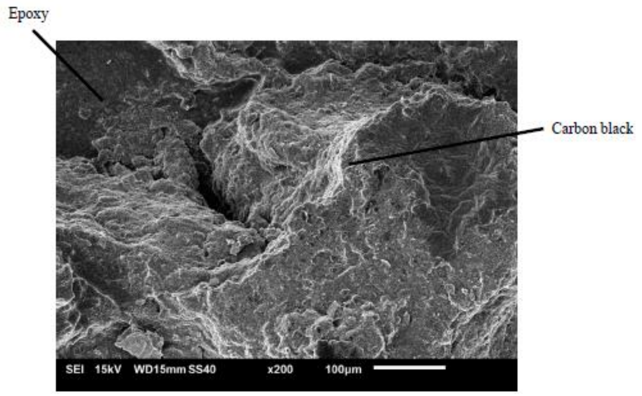

Fig. 14 SEM micrograph showing top view of the conductive ink following lap shear test at room temperature condition at 200x (secondary electron image)

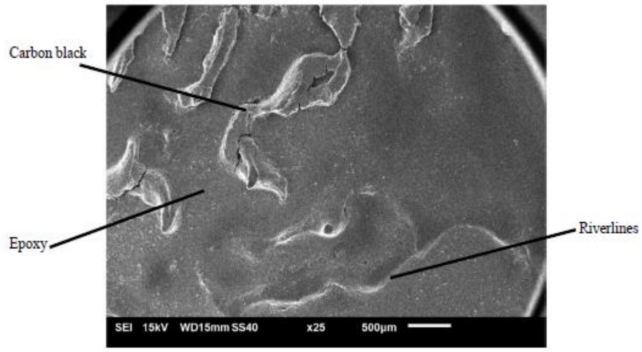

Fig. 15 SEM micrograph showing top view of the conductive ink following lap shear test at humidity chamber condition at 25x (secondary electron image)

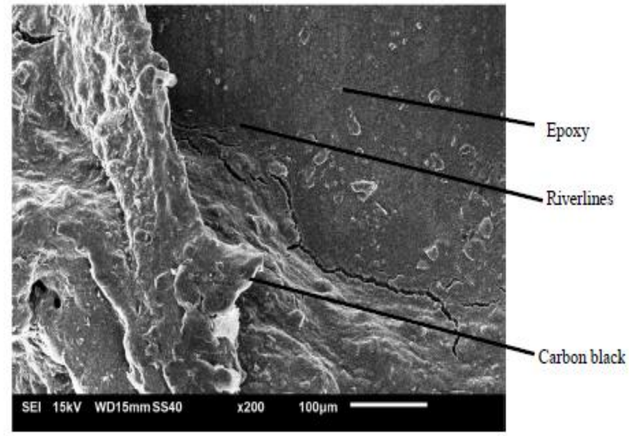

Fig. 16 SEM micrograph showing top view of the conductive ink following lap shear test at humidity chamber condition at 200x (secondary electron image)

IV.CONCLUSION

The electrical sheet resistance and mechanical shear strength are both affected by the environmental condition for the conductive ink. It has been found that an increase in water intake during the hygrothermal process due to high humidity condition results in a decrease in the electrical sheet resistance and mechanical shear strength. SEM analysis have revealed that with the presence of molecule water have caused the conductive ink to become unstructured or breaking the conductive path of the carbon black conductive filler, as evident in the SEM micrographs showing traces of the riverlines formed [19]. This results in an improved electrical conductivity, as compared to those of the room temperature conditioned conductive ink's electrical conductivity. Despite this, the shear strength of the conductive ink becomes weaker following the hygrothermal aging process, relative to those conditioned in room temperature due to weak mechanical interlocking and poor bonding strength of the epoxy polymer matrix and the carbon black filler, due to the water attack in the conductive ink system.

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