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Low-Tech Studies of Isotropic Electrically Conductive Adhesives

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Abstract

The experiments described here can provide a laboratory component to a course on isotropic conductive adhesives (ICAs), with minimal specialized equipment. Furthermore, some of the techniques can be used in ICA research to provide more controllable, reproducible results than are typically obtained with commercial devices.

1. INTRODUCTION

Over the past few years, the Components, Packaging, and Manufacturing Technology (CPMT) Society of the IEEE has cooperated with the Packaging Research Center of Georgia Institute of Technology and the National Science Foundation to fund the development of a series of Internet courses in microelectronics packaging. Most of these courses are now resident on the CPMT website at www.cpmt.org. One of these courses introduces the field of electrically conductive adhesives, covering isotropic and anisotropic materials and z-axis films. The course includes a number of experiments, with sample data, split into "high-tech" and "low-tech" classifications. The former group includes the use of typical research laboratory equipment used in the field, and is intended to support university faculty using the website course for graduate studies. The second group is aimed at the on-line student at home, who may not have access to university research facilities. The assumption is that such a student will have access to common household items (or those readily purchased at a hardware store), and possibly the level of equipment which might be found in a high-school lab, e.g. a digital multimeter with thermocouple input.. Materials can be obtained from manufacturers, but there are ICA epoxies available from general-purpose hardware stores. These ICA experiments are described here, with some sample data. More complete details of experimental procedures may be found on the website.

2. TEST OVEN

A belt oven or furnace is preferred, but any small box oven with temperature control around 150°C will suffice, even an unused kitchen stove. One must become familiar with the heating behavior of the curing oven, to make sure the manufacturer's curing specifications are fulfilled, and to assess the effect of the box oven temperature control hysteresis (and/or sample insertion transient) on cure reproducibility, especially for snap-cure adhesives. Measurements of turn-on and turn-off thermal characteristics of a small bench oven (Fig. 1) show hysteresis and a time constant clearly too long for effective snap-cure control, so such materials must be inserted and withdrawn with the oven at set-point. Fig. 2 illustrates a problematic door opening transient.

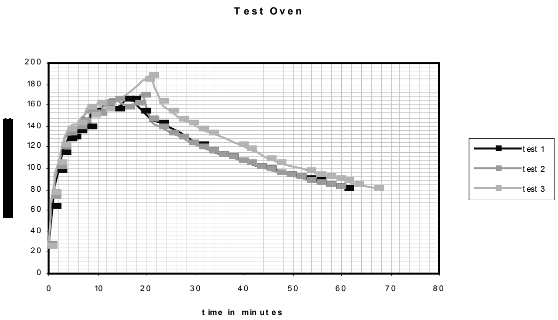

Fig. 1 Oven turn-on/off transients.

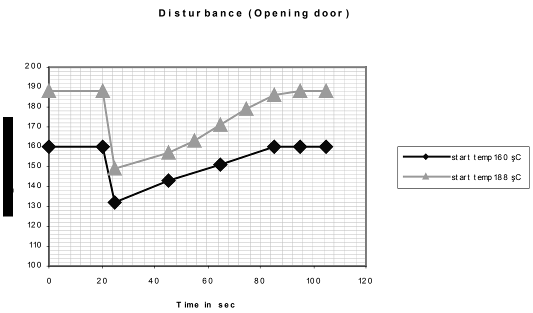

Fig. 2 Box oven transient with sample insertion

3. SCOTCH TAPE MASKING

Tracks of conductive adhesive are required with defined width, thickness and length on smooth non-conductive surface, stable up to the cure temperature, e.g. FR-4 or glass. In the absence of stencil, screen printing, or dispensing equipment, 100µm thick tracks can be defined approximately by 5 layers of scotch tape, or one of masking tape. (The actual thickness of the tape used should be verified with a micrometer.) The ICA can be printed with a razor blade as a squeegee. Wire contacts can be attached in a 4-point configuration (Fig. 3) using excess ICA as glue, and the whole assembly cured. Fig. 4 shows an example contact.

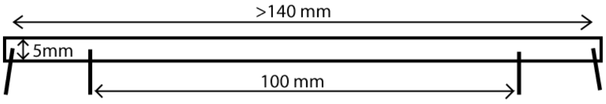

Fig. 3 Configuration for 4-point resistance measurements

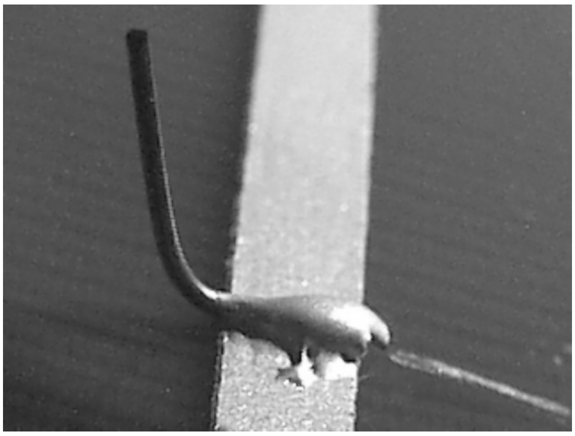

Fig. 4 Contact wire glued to track.

4. 2, 3 & 4 TERMINAL MEASUREMENTS

4-point measurements (Fig. 5) eliminate contact resistances to give the bulk resistivity; 3-point measurements, with one of the "sense" terminals used as a current "source" input or output, include one contact resistance. 2-point measurements, combining "source" and "sense" functions, include both contacts. The results (Table 1) show good consistency.

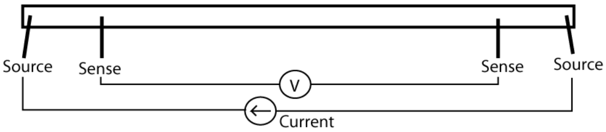

Fig. 5 Traditional 4-point resistance measurement

Method	*R (mΩ)*	*Resistances (mΩ)*
4-point	123.44	Bulk R = 123.44
3-point (end #1)	134.50	Left contact = 11.06
3-point (end #2)	140.10	Right contact = 16.66
2-point	151.40	L & R contacts = 27.96

Tab. 1 Bulk and contact resistances

5. THICKNESS VARIATION

The conductance of a printed ICA track should increase proportionally with increase in thickness, but alignment of the silver flakes on the surface yields a lower effective resistivity at low thicknesses, as illustrated in Fig. 6. Scotch tape can be used to define thicknesses in approximately 20µm steps.

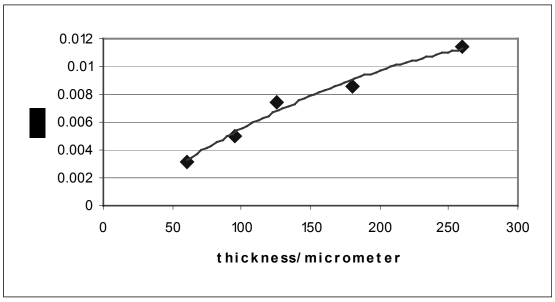

Fig. 6 Thickness vs. 1/Resistance

6. TEMPERATURE COEFFICIENT OF RESISTANCE

4-point resistances are measured at 5ºC intervals (Fig. 7), establishing steady state temperatures for each value on heating, and with the thermocouple in contact with the ICA surface. (Since cooling with the oven off is much slower than heating, thermal equilibrium should be adequate, but Fig. 7 clearly suggests that the internal ICA temperature still exceeded the measured oven value during cooling.) Hysteresis is not uncommon in thermal cycling data,, but is often accompanied by abrupt changes and parallel plots, indicative of internal "make-and-break" events amongst parallel conduction paths within the ICA. The measured TCR here of ~$2.8x10^{-3}$/K, suggests that the contribution of inter-particle contact resistances R_x is ~$0.35R_{Ag}$, where R_{Ag} represents the particle contributions. This calculation assumes that the TCR of the Ag particles (flakes) is $3.8 \cdot 10^{-3}$/K, and that the TCR of the inter-particle conduction mechanism is approximately zero.

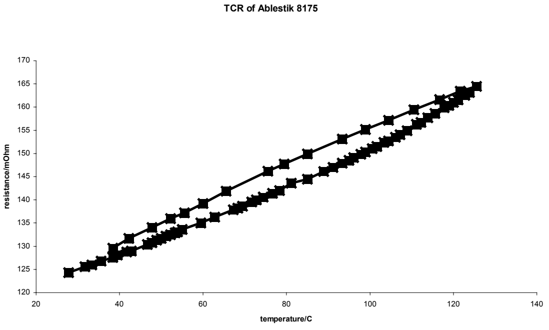

Fig. 7 Sample TCR data for Ablestik 8175

7. THERMO-MECHANICAL CYCLING

Fig. 8 shows a configuration for testing ICA thermo-mechanical reliability without the need for components. Thermal cycling applies cyclic shear stress on the ICA joints, which will eventually fail due to the mismatch between the thermal coefficients of expansion (TCE.) The FR4 board is patterned such that one can arrange multiple Cu strips to establish a series path through multiple ICA joints. The samples are placed on a hot plate with proper thermal contact for 3 minutes, followed by removal to permit cooling to room temperature. (One must monitor the thermal time constants to verify the temperature extremes actually reached, but 3 min. on and 3 min. off seems satisfactory for the set-up shown, with 3cm long 5 mm wide Cu strips. The ICA contacts reached about 125°C with the hot-plate surface at 135-140°C.) The chain resistance is recorded at each cycle until an open circuit is recorded. This failed joint is removed from the series chain (perhaps by soldering in a jumper wire,) and the experiment is continued with the remaining samples. In fact, there is no adequate sample result to provide at this time. This particular experiment series is continuing.

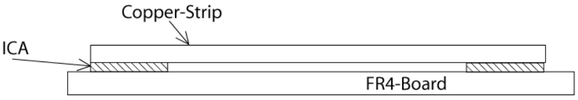

Fig. 8 Sample configuration

8. 100/100 RELIABILITY EXPERIMENT

The combination of 85°C high temperatures and 85% relative humidity (RH) is used as a standard environment to test accelerated aging and corrosion of ICAs. 85/85 testing requires specialized equipment, but can be easily replaced by 100/100 testing, (i.e. at 100°C and 100% RH,) in boiling water, which provides excellent control and stability of the test conditions. Four-point and three-point measurements isolate bulk and contact resistances, as described above. The sample is placed in a pot of sufficient size filled with boiling water. (Deionized water is recommended for ultimate accuracy, but the sample resistance is low enough that this is clearly not critical.) The bulk resistivity increases suddenly during the first two hours (Fig. 9), presumably due to the polymer absorbing water, and then stabilizes. Epoxies absorb water readily, especially if incompletely cured. Similar tests in a drier environment often yield a small initial decrease in resistance, attributable to cure completion. The steady increase of the contact resistance (Fig. 10) is indicative of galvanic corrosion of the contacts, due to the dissimilar metals, (i.e. Ag in the ICA, and Sn-plated Cu wire.).

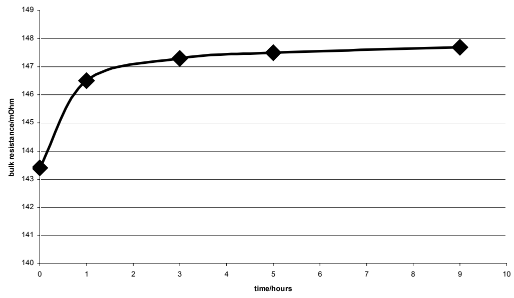

Fig. 9 Bulk resistance under 100/100 test

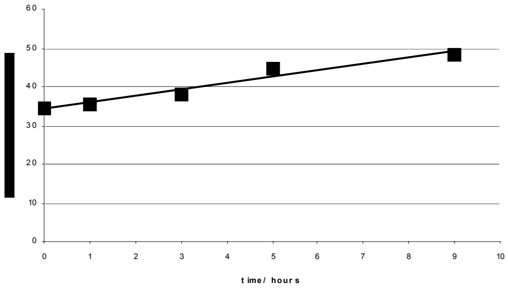

Fig. 10 Contact resistance under 100/100 test

9. NON-OHMIC/HIGH CURRENT LIMIT

The objective is to assess the behavior of ICAs at high current densities and to find the point of failure. The ICA is driven by a constant current source, comprised of a voltage source and series resistor much greater than (at least 100 times) the ICA resistance, in a 4-point configuration. A thermocouple on the track reads temperature. The current through the sample is increased in 1A steps, and resistance and temperature are recorded after reaching equilibrium. The current is increased until the sample shows clear signs of failure (specifically bubbles, smoke or discharge of black oily pulp.) At each step, the theoretical sample resistance is also calculated from the room temperature value and the previously measured TCR, and compared to the measured values on the same plot (Fig. 11.) A clear deviation from ohmic behavior occurs at around 200°C, where the sample started to deteriorate visibly with smoke and bubbles. The separation of the two curves indicates that the measured surface temperature is lower than the internal temperature. Note that the surface temperature is approximately proportional to the internal ICA power dissipation (Fig. 12.) It is clear that the failure is due to polymer degradation. (Note that the data presented are for bulk resistivities as determined by 4-pont measurement. It would be interesting to see how the contact resistances might behave).

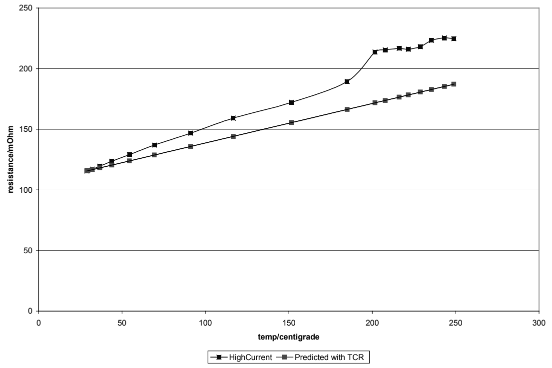

Fig. 11 Theoretical/experimental resistances vs temperature

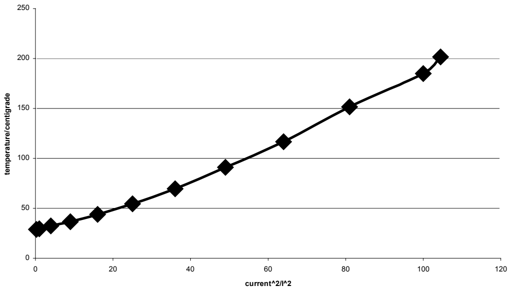

Fig. 12 Current^2-Temperature relationship

10. DROP TEST

The failure sensitivity of ICAs to impact is dependent on inertial mass, and to maintain mass as the sole variable, 2.5x2.5cm^2 3mm thick Al weights are added to the components with cyanacrylate-based superglue to increase the inertial mass. Surplus SMT components can be used, or 5x5cm^2 Al samples (0.5 to 3mm thick) can simulate components, with 1x1cm^2 ICA attachment areas. The results of 5ft drop tests are shown in Table 2 and Fig. 13, with apparatus in Figs. 14-16.

Blocks	Drops till failure
1	More than 120 (did not come off)
2	30, 43
3	17, 21
4	7, 4,11
5	4, 4
6	3

Tab. 2 Drop test data for 1-6 added weights

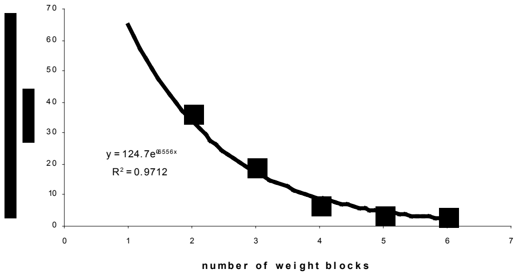

Fig. 13 Drop test survival

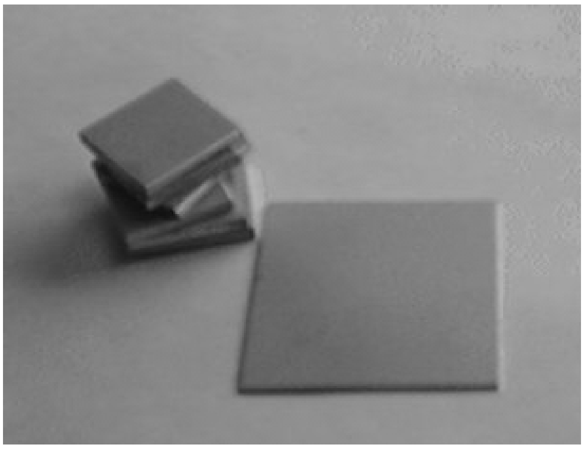

Fig. 14 Al "component" and inertial weights

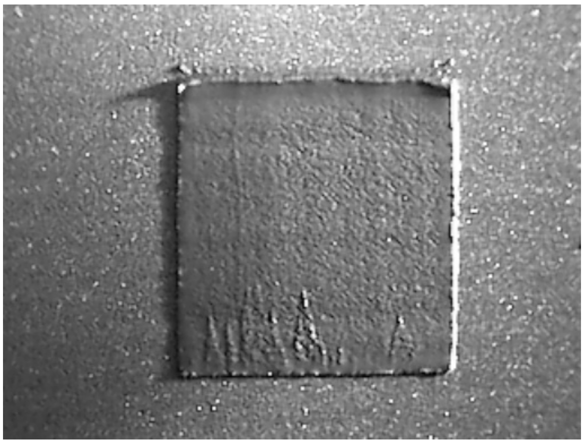

Fig. 15 1cm x 1cm ICA contact area

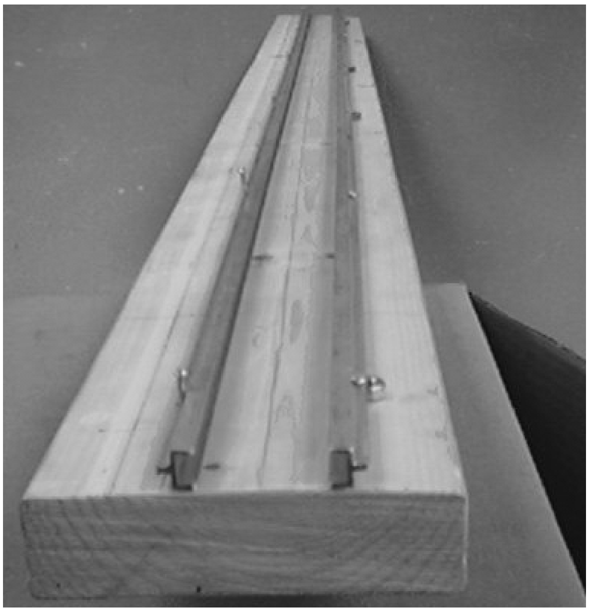

Fig. 16 Drop test track

11. NON-CONDUCTIVE ADHESIVE

Any kind of cyanacrylate-based superglue can form a conductive connection between electronic components if the film thickness of the adhesive is small enough and the surface roughness of the contact areas of the components is sufficient. The resistance and reliability of this sort of microelectronic interconnect is illustrated in Fig. 17. In other tests, without surface roughening, steadily increasing resistance has been noted.

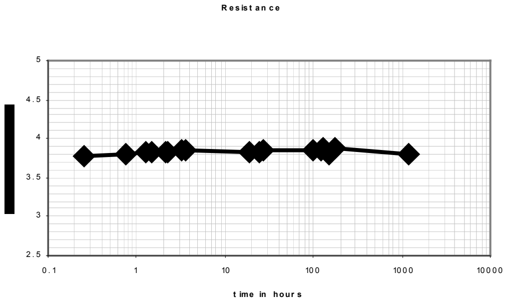

Fig. 17 NCA contact stability

12. SUMMARY

This paper has presented some sample data for ICA experiments conducted with very basic equipment, as prototypes for laboratory courses in the field at either the high school or college levels, and potentially for students at home taking on-line distance education courses. The data presented here is also available on the website, where more complete procedural details are also provided. The ICA used for these experiments was Ablestik 8175, (except of course for the standard, commercial superglue for the NCA,) but more limited experiments using a commercial conducting epoxy from an automotive hardware store worked well too. The oven used was a small laboratory box oven, but a kitchen oven would be as good in principle, but perhaps with longer time constants. Note, however, that curing may drive off organic vapors which may be hazardous to health, so do not use such an oven for subsequent food preparation, and adequate venting is required. While the use of commercial components provides additional practical information of interest to ICA manufacturers and users, (e.g. circuit assemblers,) the substitution of Cu or Al blocks provides just as useful information about the ICA itself, and possibly more reproducible due to larger sample scales. (Note, however, that surface interaction information, e.g. adhesion to lead surface materials, is not obtained.)