# **Recent Results of ICA Testing**

James E. Morris & Christina Cook, Department of Electrical Engineering, T. J. Watson School of Engineering & Applied Science, State University of New York at Binghamton, NY 13902-6000, U. S. A. Markus Armann, Electrical Engineering Department, Chemnitz University of Technology, Germany Albrecht Kleye & Peter Fruehauf, Electrical Engineering Department, Dresden University of Technology, Germany

#### Abstract

The paper includes data from recent ICA testing at Binghamton University of the effects of plasma cleaning of ICA adhesion to copper and plated surfaces, of high current densities, and of the application of electric fields before and during cure. In addition, drop tests were performed on conventional and new impact resistant materials. It is emphasized that all results are preliminary, and some clearly point to deficiencies in the cure schedules used. The work described is continuing.

### Introduction

The reliability of isotropic electrically conductive adhesives (ICAs) has progressed steadily over the past several years since they became the focus of intensified research as a potential lead-free replacement for solder in surface mount applications. In particular, recent results have demonstrated that the electrolytic corrosion process at the ICA-contact interface can be slowed by the addition of moisture and corrosion inhibitors [1, 2] and that impact resistance can be improved by the use of high loss modulus polymers, eg. above the glass transition temperature, T<sub>g</sub> [2, 3]. Nevertheless, there is still a great deal understood about not vet the materials, and much room for further improvement in the technology [4].

# **High Current Effects**

The capability of the ICA materials to operate at high current densities will

be especially important for applications in power electronics. Figure 1 shows the changes in ICA resistance for three different commercial materials subjected to successively higher current stress. The ICA samples were 1mm long, with a cross-sectional area of 2.55mm<sup>2</sup>. Each current was applied for 30 minutes, with the resistance shown being measured at the end of that period, with 60 minutes between the end of each test period and the start of the next. The ICA surface temperature was measured during the test, with the results at 10A (the second highest value) tabulated in Table 1. There is a clear correlation with resistance and power dissipation, and the surface temperature increase for Ablebond 8175A with the current increase to 15A is negligible, since the resistance drops. At 15A, the samples began to give off strong fumes as the polymer broke down for both Ablebond 84-1 LM1 and Epo-tek 3116-5, but the Ablebond 8175A remained stable.

Initial low current resistance reductions are attributed to continued curing effects, with mid-range increases assumed to be due to thermally induced fractures of percolation paths, (which may be reversible.)

# Electric Field Effects

In previous experiments, it had been observed that the application of an electric field to the uncured ICA paste led to a sudden decrease in resistance (increase in current.) It was not determined whether this effect was actually due to ordering of the Ag flakes in the material, or to an atomic Ag electromigration effect. In either case, the question was whether the lower initial paste resistance persisted into the cured state and a lower resistance cured material. A series of experiments were run to determine this point, with simultaneous curing of three sets of samples. One set had fields of 50V/mm applied to 200µm thick stenciled samples for 15 minutes before cure. For the second set, the field was also applied during curing itself, and the third was the control samples where no field was applied.

The results are presented in Figure 2. In general, sample resistances of the samples with fields applied were lower than the control values by 30-50%, but there was no consistent, significant difference between those with the field applied prior to cure and those where the field continued to be applied

# Plasma Cleaning

It is expected that ICA adhesion would be improved if the adherent surfaces were clean, and especially kept clean of oxides, and organic contamination. A series of experiments were run to verify the hypothesis, using both bare copper and copper coated with immersion deposited gold. The results of Table 2 were obtained for adhesion areas of approximately 121mm<sup>2</sup>. Both pull and shear tests were performed. A mixture of Oxygen and Argon was used as the sputter gas, in the  $Ar/O_2$ ratio of 19/2 by flow rate. The plasma pressure was 30mTorr with net 195 W power, and plasma cleaning was applied for six minutes. The resistance values of all samples compared favorably with the manufacturer's data sheet, but adhesion values were typically an The primary result order less. demonstrated in the tables is that there is negligible difference in adhesive strengths with and without plasma treatments. This is the same result as determined elsewhere [5], but it is counter-intuitive, and demands further studv. Surface analysis showed substantially reduced organic contaminants and surface oxide following the plasma treatment.

# **Drop Tests**

There were three distinct series of drop test experiments. In all cases, four samples of each adhesive were split, two each, between the 3-foot drop test and the 5-foot drop, the de facto standard test developed by the Manufacturing National Sciences Consortium. The materials tested and the cure schedules used ate tabulated in Table 3. The 136H3 is a low  $T_{\alpha}$ T<sub>g</sub> is below room material, i.e. temperature, so it has a significant mechanical loss modulus [3]. In both Series A and B, the cure schedule is based on the data sheet recommendations (3 minutes at 150°C for 8175A,) but there is a thermal

transient when the samples are inserted into the oven. Typically the temperature falls to 140°C, recovering to 150°C within one minute, but nevertheless with significant adverse effect on the 5.5-minute cure. (Hence the increase to 7 minutes in Series B, which still did not seem to be sufficient.) The 7-minute cure for 136H3 was as per data sheet recommendation for Series B, with the 60-minute samples supplied by the manufacturer. The chips used in each test are listed Table 4, and the drop test results in Tables 5 and 6. Figure 3 shows the component distribution on the board for Series A.

Clearly, the larger components drop off first, only one chip-capacitor detaching for example. In addition the new 136H3 formulation is much more impact resistant than the other two. But the observations of note here relate to the degree of cure, and its effect on the results. It has been noted already that the 8175A appeared to be incompletely cured in Series A, (in particular for samples #1 and #2,) but sample 8175A #5 in Series B also appeared to be tacky and under-cured. It is the dramatic improvement in the results for the longer curing time, however, which is the main feature of interest.

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# References

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Figure 1(c)



Figure 1 Resistance variation with current load.

ICA	Resistance #1	Resistance #2	Temperature
Ablebond 8175A	53.0 m $\Omega$	52.4 mΩ	52°C
Ablebond 84-1 LM1	163.4 m $\Omega$	223 m $\Omega$	144°C
Epo-tek E3116-5	59.7 m $\Omega$	$57.8~{ m m}\Omega$	58°C

Table 1. ICA surface temperatures at 10A.

Figure 2(a) (Column 5: Average values)



Figure 2(b) (Column 6: Average values)



Figure 2(c) (Column 6: Average values)



Figure 2 Effect of applied field before and during curing on ICA resistances.

Sample	Resistivity: $\Omega$ -cm	Pull Test: N/mm <sup>2</sup>	Shear Test:	psi
			N/mm <sup>2</sup>	
AUI 1	0.00075		1.4423	209.24
AUI 2	0.0007	0.3884		56.42
AUI 3	0.00055		1.4190	205.71
AUI 4	0.0008		1.2562	182.15
AUI 5	0.00065		1.4678	212.83
AUI 6	0.00105		2.1074	305.57
AUI 7	0.00065	0.5190		75.26
ACI 1	0.0006	0.4620		67.00
AU 1	0.0003	0.5595		81.13
AU 2	0.00035		3.2835	476.11
AC 1	0.000225	0.4463		64.71
AC 2	0.00025		2.5537	370.29
BUI 1	0.00085		1.7430	252.74
BCI 1	0.0006	0.4380		63.51
BCI 2	0.0005		1.8645	270.35
BU 1	0.00035	0.4702		68.18
BU 2	0.0004		3.0810	446.75
BC 1	0.000225	0.4537		65.79
BC 2	0.000225		2.7967	405.52
MUI 1	0.0009	0.1215		17.62
MU 1	0.0003	10 ft drop: failed		
MU 2	0.000225		0.6893	99.95
MC 1	0.00025	10 ft drop: failed		
MC 2	0.00035	0.3041		44.09

Table 2(a) Bare copper substrate

Key:

A = Ablebond 8175 A B = Ablebond 84-1LMI

U = Uncleaned

C = Cleaned (Plasma)

M = Methode Development Company 1210 A

I = Initial Test

Table 2(b) Immersion gold plated copper

Sample	Resistivity: Ohm-cm	Pull Test: N/mm <sup>2</sup>	Shear Test:	psi
			N/mm <sup>2</sup>	
AU 1	0.00025		3.3240	481.98
AU 2	0.0003		2.2298	323.32
AU 3	0.00025	0.5471		79.33
AC 1	0.000225		3.0405	440.87
AC 2	0.00025		2.2702	329.18
AC 3	0.0002	0.4942		71.66
BU 1	0.00045	0.4298		62.32
BU 2	0.000325		2.5537	370.29
BU 3	0.000475		2.7157	393.78
BC 1	0.0005	0.3967		57.52
BC 2	0.00035		2.6347	382.03
BC 3	0.00055		2.3512	340.92

Table 2..Mechanical testing of ICA adhesion to (a) copper and (b) gold substrate surfaces, with and without plasma treatments.

	Seri	es A	Seri	es B	Series C
	8175A	84-1LM	8175A	136H3	136H3
Cure temp (°C)	150	150	150	150	150
Cure time (min)	5.5	60	7	7	60
<b>T</b>     0 0					

Table 3 Cure schedules for drop test samples

Component	Comments	Series A	Series B	Series C
Dummy pkg	Large QFP	1		1
CF62613FN	Medium QFP	1		
731XF	Small QFP	3		
TI610XT	Large DIL	2		
H9506	Large DIL		1	
74ACT244	Medium DIL	2	4	
M912FMM	"Square" DIL	3	2	
74F109	Small DIL	5		
74F20	Small DIL		3	
TI749A	Smallest DIL		5	
Chip Capacitor	Std. small SMT pkg	2		

Table 4 Numbers of components on drop test boards

(a) 3-foot		Series A														Series B																								
drop tests	5	817	75A	\ #'	1	5	817	75A	× #2	2	84-1LMI #1 84-1LMI #2										817	75A	\#	5	8175A #6						136	5H3	3 #	136H3 #2						
Drops→	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r
Large QFP	1	1			0	1	1			0	1	1			0	1	1			0																				
Med. QFP	1		1		0	1			1	0	1				1	1	1			0																				
Small QFP	3		3		0	3	2	1		0	3	3			0	3	3			0																				
Large DIL	2		2		0	2			2	0	2	2			0	2	2			0	1	1			0	1		1		0	1		1		0	1		1		0
Medium DIL	2		2		0	2			2	0	2	2			0	2	2			0	4	1	1	1	1	4		2		2	4			1	3	4			1	3
Square DIL	3				3	3			1	2	3		3		0	3	2	1		0	2				2	2			1	1	2				2	2	1			1
Small DIL	5				5	5		1		4	5				5	5		1	1	3	3			1	2	3				3	3			1	2	3				3
Smallest																					5		2		3	5	1	1		3	5			2	3	5	1		2	2
Chip capac	2				2	2				2	2				2	2		1		1																				

(b) 5-foot		Series A														Series B																								
drop tests	8	817	75A	\ #(	3	5	317	75A	\ #4	1	8	4-1	LN	11 #	ŧ3	8	4-1	ILN	/II #	<b>‡4</b>	8175A #7					8175A #8						136	3H3	3 #3	3		136	SH3	<del>,</del> #2	4
Drops→	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r	s	1	2	3	r
Large QFP	1	1			0	1	1			0	1	1			0	1	1																							
Med. QFP	1	1			0	1	1			0	1	1			0	1	1																							
Small QFP	3	3			0	3	2	1		0	3	3			0	3	3																							
Large DIL	2	2			0	2	2			0	2	2			0	2	2				1	1			0	1	1			0	1			1	0	1				1
Medium DIL	2	1	1		0	2	2			0	2	2			0	2	2				4	1	1	1	1	4		1	2	1	4		1		3	4		1		3
Square DIL	3		3		0	3	3			0	3		1		2	3	1	2			2			1	1	2		1		1	2				2	2				2
Small DIL	5		1		4	5			1	4	5				5	5			1		3		1		2	3		1	1	1	3		1	1	1	3				3
Smallest																					5	1		2	2	5	1	1	1	2	5	1		1	3	5			1	4
Chip capac	2				2	2				2	2				2	2																								

Table 5 Drop test results (Series A & B): [s=number of components at start; r=number remaining after 3 drops.]

	3-foot	t drop	5-foot	t drop
	136-H3 #5	136-H3 #6	136-H3 #7	136-H3 #8
Number of drops until component moves	27		6	
Number of drops until component falls off	29	13	7	10

Table 6 Series C drop test results: Number of drops to failure. (Testing consisted of single, large QFP and 136H3 adhesive.)





Figure 3 3"x3" Copper Circuit Board Layout