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Isotropic conductive adhesives in electronics

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Abstract: Isotropic conductive adhesives are metal/epoxy composites with high loading beyond the percolation threshold for electrical conduction. The primary reliability issues are impact resistance and galvanic corrosion of the contacts, among others. Nanoparticles and carbon nanotubes are beginning to be incorporated into the materials, for improved performance.

Key words: isotropic conductive adhesive (ICA), drop test, corrosion, immersion silver.

4.1 Introduction

There are two primary categories of electrically conductive adhesives (ECAs): isotropic conductive adhesive (ICA) and anisotropic conductive adhesive (ACA), with the latter dividing further into paste (ACP) and film (ACF) types. Tin–lead (Sn-Pb) solder toxicity and environmental issues triggered initial ECA interest, with the advantages of lower processing temperature, no-flux, no-clean, and simple processing (Zwolinski, 1996; Detert and Herzog, 1999), but it has been other advantages which continue to drive research for flip-chip (Rusanen *et al*., 1997; Lohokare *et al*., 2006), surface mount technology (SMT), optoelectronics, and MEMS packaging. ICAs are also used extensively in die-attach, for small passive chip attachment in automotive electronics, and in RFID tags for both antenna and chip connections. More recently, high-density SMT board via-fill has emerged as a major commercial application (Das *et al*., 2006; Kisiel *et al*., 2005a, b), and novel uses continue to be proposed, e.g. as conformal interconnect for a 3D IC stack (Robinson *et al*., 2008). One disadvantage of ICAs is that the polymer cure time is inherently longer than solder reflow times, but it can be shortened by 'snap-cure' polymers (Moscicki *et al*., 2005) which use fast catalysts, and fast variable frequency microwave curing (Wang *et al*., 2001; Fu *et al*., 2003).

Much of the research focus in the literature is on reliability failure modes, e.g. resistance stability and adhesion shear tests (Liu *et al*., 1995), humidity effects (Rorgren and Liu, 1995) and other thermal testing (Gaynes *et al*., 1995; Rusanen and Lenkkeri, 1995), usually with comparisons to solder's properties (Hvims, 1995).

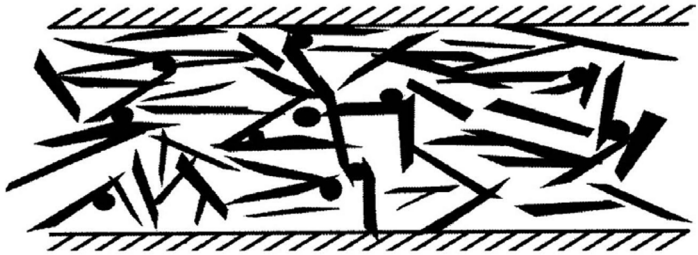
The ICA consists of a two-phase mixture of metal, typically a bimodal

distribution of silver (Ag) flakes and powder (Figs 4.1 and 4.2), in a polymer (epoxy) adhesive. ICA resistivity drops dramatically (Fig. 4.3) when the metallic content exceeds the 'percolation threshold'. Ag is popular because it is less expensive than gold (Au) with superior conductivity and chemical stability, and, unlike nickel (Ni), Ag oxides show high conductivity (Lu *et al.*, 1999c; Markley *et al.*, 1999; Shimada *et al.*, 2000; Kotthaus *et al.*, 1997; Lu and Wong, 2000f). Ag-coated copper (Cu) also shows promise (Nishikawa *et al.*, 2008), and low melting point alloys, Sn-coated Ag particles, etc. have been reported (Moon *et al.*, 2003; Lu and Wong, 2000c; Suzuki *et al.*, 2004; Yamashita and Suganuma, 2006b).

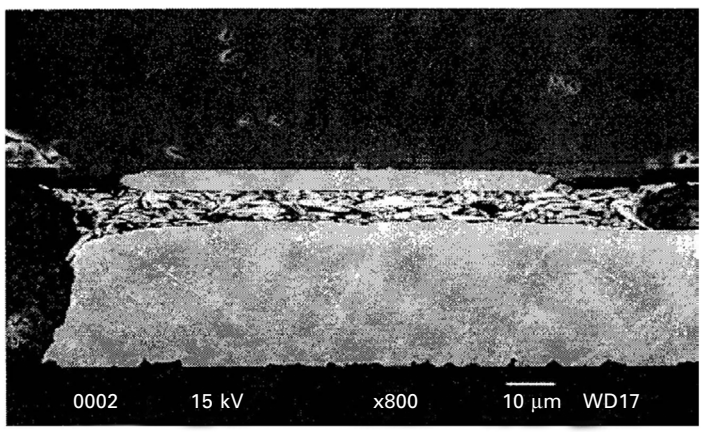
Regardless of the filler metal, the flakes require lubrication to resist the tendency to 'clump' together during processing, e.g. with stearic acid (soap). Lu *et al.*, reported on various lubricants, first on the chemistry of the lubricant layers and their interaction with the Ag flakes, and on their thermal behavior during heating (Lu and Wong, 2000b), and then on their thermal decomposition (Lu and Wong, 2000a). Wong has achieved reduction of overall resistance by replacing the traditional stearic acid with shorter chain alternatives (Wong and Li, 2004; Li *et al.*, 2004a,b, 2006a,b). Miragliotta *et al.* (2002) has shown that the lubricant breaks down during cure, and leaves a carbon residue on the flake surface, which is expected to contribute to inter-particle conductance.

Intuitively, the higher the metal content, the higher the conductivity, traded off against weaker adhesion. There have been several attempts to improve electrical conductivity at low metal concentrations, e.g. by magnetic alignment

4.1 ICA bi-modal filler distribution (Ag flakes and powder), with surface layering evident (Li, 1996).



(a)



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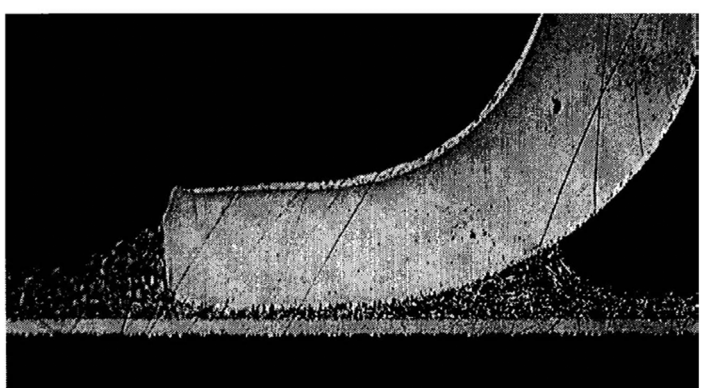


(c)

4.2 ICA contact joints: (a) schematic, (b) flip-chip on FR-4, (c) SMT on FR-4 (Kudtarkar and Morris, 2002; Morris, 2005).

of Ni filler rods (Sancaktar and Dilsiz, 1997) or particles (Ramakumar and Srihari, 2008), or by the use of polymer particles (Inada and Wong, 1998) or electric fields (Morris *et al.*, 1999) to force z-axis alignment of flakes. The

coating of contacts by intrinsically conductive polymers has been investigated, but turns out to increase resistance (Lam *et al.*, 2006).

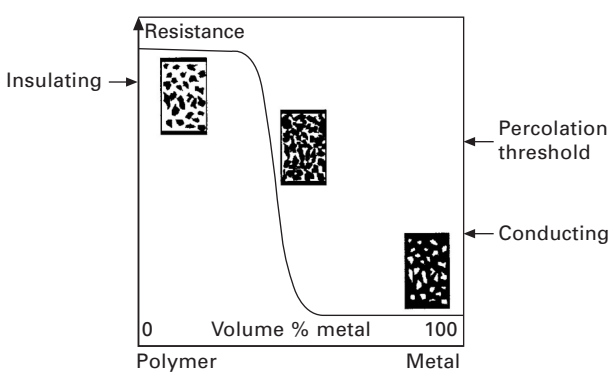
ICAs are typically dispensed by syringe, but may also be stencil- or screen-printed. Formulations are usually based on thermoset epoxy resins for the polymeric matrix, typically with thermoplastic added for rework capability. Although epoxies have excellent strength and adhesion capabilities, they tend to absorb moisture.

Self-alignment is a critical component to the success and reliability of solder attachment of area-array flip chips, and the absence of a similar surface-tension driven property in ICAs is widely seen as an impediment to their adoption for this role. Wu *et al.* (Wu *et al.*, 2001; Moon *et al.*, 2001) coaxed a minimal ICA self-alignment effect from LMPA content, but Kim *et al.* have shown that self-alignment is viable when driven by resin surface tension (Kim *et al.*, 2003).

4.2 General isotropic conductive adhesive (ICA) properties

4.2.1 Structure

The ICA resistivity only drops slightly as the metal content is increased until the 'percolation threshold' is reached (Fig. 4.3), when the first continuous metal path is established through the composite material. The primary concepts of percolation theory are well developed, especially for the elementary system of uniform conducting spheres (or cubes) in a perfectly insulating medium (Smilauer, 1991). Bi-modal particle distributions have been shown to reduce the percolation threshold (Kusy, 1977) with either flakes or powders used for the smaller particles. The electrical path includes the metallic resistance of the filler and the contact resistances between particles and at the contacts.



4.3 Percolation threshold (Morris, 1999).

Metallic resistance has been shown to dominate (Li *et al.*, 1993), in the absence of environmental effects.

At low thicknesses, the flakes are all layered, parallel to the substrate contact, but become more randomly distributed as thickness increases (Morris *et al.*, 2003), unless the application of pressure during or prior to cure forces all the flakes into alignment (Constable *et al.*, 1999). The effects of surface layering have been confirmed at a qualitative level (Li and Morris, 1997), and the consequential size effects demonstrated (Sancaktar and Dilsiz, 1998; Ruschau *et al.*, 1992), i.e. the increased/decreased conductance parallel/perpendicular to current flow. Morris *et al.* (2001) show the resistance of a z-axis contact as it is mechanically thinned, with a sharp drop in resistivity when the aligned surface layer is removed.

4.2.2 Electrical properties

It is necessary to separate contact resistance and the bulk composite resistivity in order to interpret physical effects on each, independently (see below), by the subtraction of the common four-terminal measurement, which removes test lead and contact effects, from a three-terminal measurement which includes one contact in the test current path (Morris *et al.*, 2001; Klosterman *et al.*, 1998).

Finite geometries can lead to errors if care is not taken. The ICA resistance under test is typically of the same order as PWB track resistances (Morris *et al.*, 2001; Kulkarni and Morris, 2003), and one should make some simple estimates of all resistances in the test set-up to determine contact thicknesses to avoid current crowding. Because ICA z-axis resistances are small, many experimental results in the literature have been obtained by x-axis measurements, i.e. by measuring ICA resistance along a long, thin printed track. Such x-axis measurements tend to give higher resistivities than the ideal random structures of the true bulk unless the thickness is much greater than the characteristic meandering percolation path length, and lower resistivities due to layering effects. The z-axis application of actual interest displays two opposite trends.

Li *et al.* (1993) showed that the ICA high-frequency behavior is fully attributable to skin effect in the metal filler, with no observation of the capacitive effects expected of tunneling contacts between filler particles. Their high-frequency ICA data have been extended by Wu *et al.* (1998), and into the GHz range by Dernevik *et al.* (1997), Sihlbom *et al.* (1997), and Otsuka and Akiyama (2007).

At frequencies where skin effect is dominant, the lower resistance advantage enjoyed by solder at DC disappears, as the effective cross-sectional area shrinks with the skin depth for both solder and ICA alike (Morris *et al.*, 1999), as supported by the observations of Hashimoto *et al.* (2008). For

practical purposes, there is negligible high-frequency performance difference between ICAs and solder (Liong *et al.*, 2001).

4.2.3 Mechanical properties

ICA adhesive and shear strengths are similar to solder's, occasionally higher (Herzog *et al.*, 2004) although usually a little less (Suzuki *et al.*, 1998), but generally adequate (Luchs, 1996). Plasma cleaning of the adherent surfaces would seem to be a logical step to improve adhesion. Herzog *et al.* (2004) and Paproth *et al.* (2001) have shown that plasma treatments increase the polar component of surface energy, but preliminary data show no adhesion improvement with either argon or oxygen plasmas, despite removal of organic contaminants and oxides (Dernevik *et al.*, 1997; Morris *et al.*, 1999; Morris and Probsthain, 2000). Experimental studies consistently show that the mechanical component of adhesion dominates (Liong *et al.*, 2002; Chow *et al.*, 2002), with best results from surface roughening, (which may be accomplished by high-energy plasmas.) A conducting polymer interface layer can also promote ICA adhesion (Kuechenmeister and Meusel, 1997), and Keil *et al.* (2001) improved it by structuring the contact pad so that a proportion of the ICA contacts the FR-4 epoxy surface rather than the metal contact.

Wu *et al.* (1996) studied viscoelastic ICA properties, and concluded that stability requires a low-temperature cure, followed by a stabilization ramp to higher levels. Post-cure annealing decreases resistance further, even at less than the cure temperature (Inoue and Suganuma, 2005, 2006). At full cure conditions, however, the electrical resistance and the mechanical strength of conductive adhesives can be guaranteed (Liu *et al.*, 1997).

4.2.4 Thermal properties

The thermal performance of an adhesively assembled chip is of vital interest as power dissipation in the chip increases, and for die-attach or heat-sink bonding (Inoue and Suganuma, 2006). Sihlbom *et al.* (1998) have simulated power dissipations, and Kimura *et al.* (2003) and Inoue *et al.* (2006) have performed experimental studies, but this is an area that deserves much more attention.

4.2.5 Environmental properties

The environmental impact of ECAs has been studied by several research groups. Segerberg *et al.* (1997) compared the use of conductive adhesive joining with soldering for SMT applications and concluded that the relative environmental load of the conductive adhesives is dependent on the mining

condition of Ag. Westphal (1998) concluded that conductive adhesives are generally better in terms of environmental loading compared to solder.

More work is needed to clarify environmental pros and cons, especially since environmental concerns have been an ICA technology driver, and Ag is not wholly environmentally benign. Curing agent toxicity, for example, is an overlooked issue (Yi *et al.*, 2006), and nano-Ag's anti-bacterial properties may become an environmental problem if concentrations rise (Luoma, 2008), threatening the bottom of the aquatic food chain.

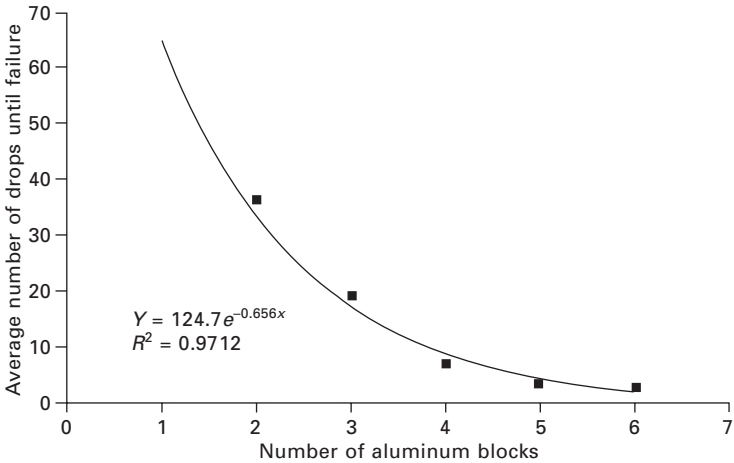
4.3 Reliability

4.3.1 Impact resistance

Impact resistance is the primary impediment to more widespread ICA technology adoption. Early identification of drop-test failure as a significant ICA problem (Rusanen and Laitenen, 2004) led to the widespread adoption of the NCMS (National Center for Manufacturing Science) criterion (survival of six drops from 5 feet) as a *de facto* standard (Zwolinski, 1996). Survival rate correlates with the (imaginary) dissipation modulus instead of adhesive strength (Tong *et al.*, 1998a, b; Xu and Dillard, 2003), but the use of polymers with glass transition temperature T_g below room temperature (Luo and Wong, 2002; Yi *et al.*, 2008) would lead to other, greater problems. The addition of carbon fibers to the ICA seems to be helpful (Keil *et al.*, 2001), but the result is ambiguous without modulus measurements, because of a simultaneous change in the contact geometry to improve adhesion. Although the loss modulus is accepted to be the most significant parameter in impact resistance, the failure mode is adhesive rather than cohesive as expected. So shear adhesive strength should play a secondary role. Several sequences of drop test experiments, which have included low T_g and 'snap-cure' ICAs, are described by Morris and Lee (2008) with the conclusions outlined below.

It is well known that drop-test survival depends on the inertial mass of the component in question, as demonstrated by the variation in drop-test survival rate with component mass shown in Fig. 4.4 for dummy aluminum component blocks (Morris *et al.*, 2003). (The larger contact area available with the dummy blocks increases the number of drops to failure in comparison with 'real' components, and improves the statistical consistency of the results.) As a consequence, commercial products may use ICA attachment for small SMT passive devices, but solder or other attachment for larger processor chips, etc. In experiments with PWBs stocked with SMT devices of varied sizes and pin configurations, the larger components clearly fall off first (Kudtarkar and Morris, 2002).

A general trend was identified, that (within limits) under-cured samples survive more drops than those cured to specification, which in turn survive more than over-cured samples, in agreement with the loss modulus effect,



4.4 Drop-test survival using aluminum blocks as dummy chips (5-foot drops; 2.5cm × 2.5cm × 3mm aluminum blocks; 1cm^2 adhesive area; blocks stacked with cyanoacrylate 'super-glue'; single block survived 120 drops without failure).

T_g increasing with the degree of cure. An experimental ICA with low T_g (below room temperature) was also shown to be much more impact-resistant than others tested, and in this case the fully-cured low T_g adhesive performed better than the under-cured.

Dummy Al components also survive better than the PQFP components used, despite the higher mass, presumably due to greater adhesive strength to the rougher Al surfaces, a postulate supported by the fact that the point of failure for the Al samples was mainly at the substrate surface, whereas the PQFP failures usually occurred at the lead interfaces. It was also observed that PQFP leads bend under impact, and sometimes fail (fracture) before the ICA bond.

Figures 4.5 to 4.8 (Morris and Lee, 2008) summarize the key results for drop tests for a single ICA and two components of different sizes:

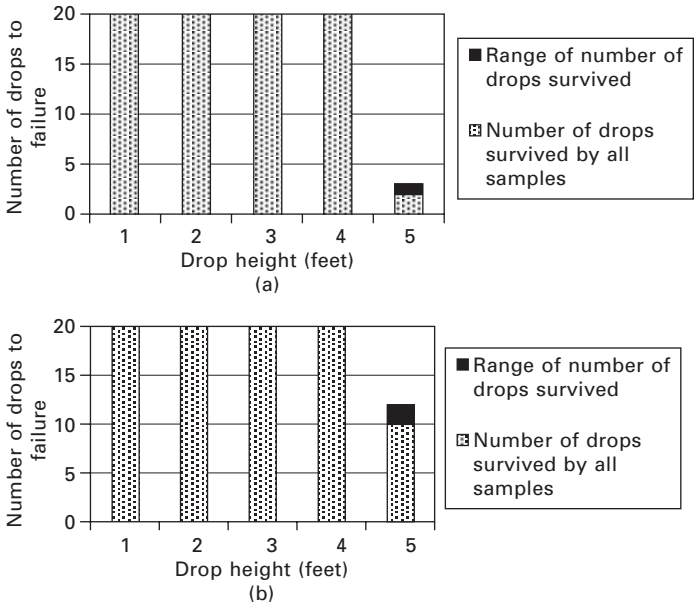
- Figs 4.5 and 4.6: small SO20GT devices
- Figs 4.7 and 4.8: larger 68-lead PLCCs

with two ideally equivalent ICA cure temperatures:

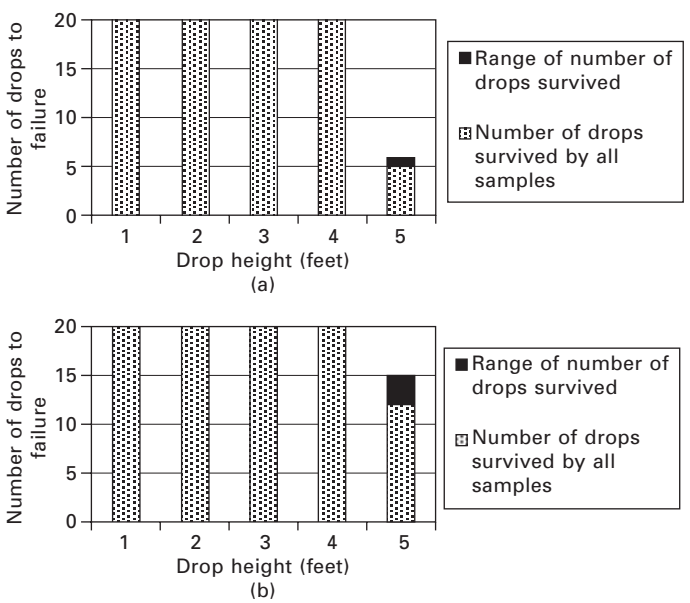
- Figs 4.5 and 4.7: 60 minutes at 150 °C
- Figs 4.6 and 4.8: 30 minutes at 175 °C

according to two profiles:

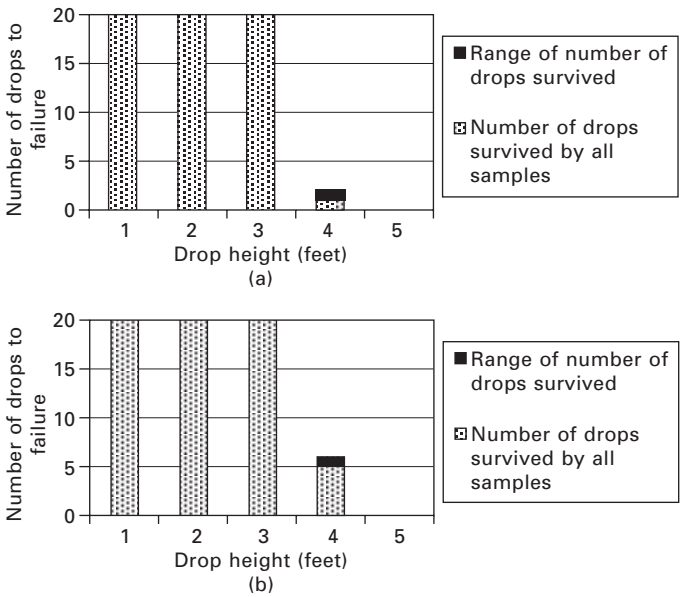
(i) simple ramp-up/hold/ramp-down specified by the manufacturer
(ii) with the addition of pre-heat/post-cool hold periods to the ramp up/down stages.



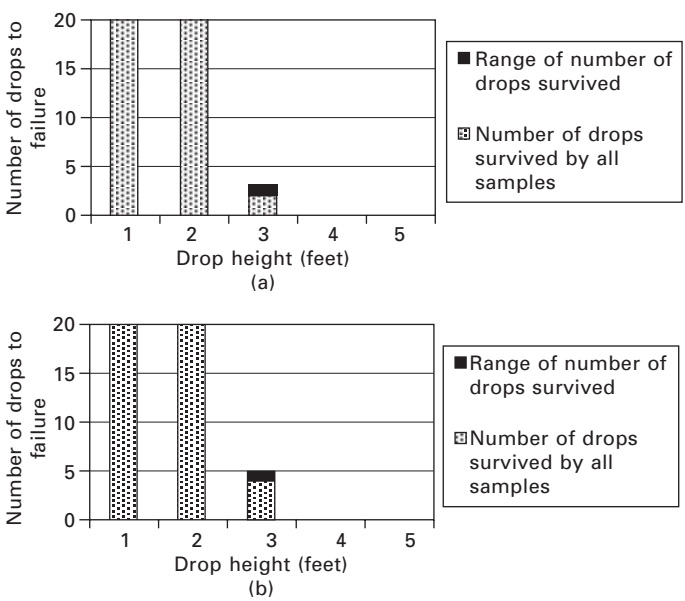
4.5 SO20GT cured at 150 °C: (a) ramp and hold, (b) pre-heat/post-cool.



4.6 SO20GT cured at 175 °C: (a) ramp and hold, (b) pre-heat/post-cool.



4.7 PLCC68 cured at 150 °C: (a) ramp and hold, (b) pre-heat/post-cool.



4.8 PLCC68 cured at 175 °C: (a) ramp and hold (b) pre-heat/post-cool.

Each sample, with one package per board, was dropped successively twenty times from each of 1, 2, 3, 4, and 5 feet heights, until at least one package lead detached from the board (which usually meant when all detached and the

device fell off the board). Therefore, where the SO20GT samples fail after some variable number of drops from 5 feet in Figs 4.5 and 4.6, they have all previously survived twenty drops from each of 1, 2, 3, and 4 feet. The variation in the number of drops survived by multiple samples is indicated in each figure by the relatively small 'range.'

As expected, the larger PLCC68 components fall off sooner than the smaller SO20GTs. There is a small improvement in SO20GT impact resistance with the change in cure schedule from 60 minutes at 150 °C to 30 minutes at 175 °C, but a significant reduction in PLCC68 performance for both the simple ramp-and-hold and pre-heat/post-cool profiles. A possible explanation is that 175 °C results in an over-cure, to which the J-lead PLCC68 samples are more susceptible than the SO20GT gull wing configuration, due to the smaller contact area.

The primary result is that samples with pre-heating and post-cooling have better impact resistance than those without. From other experiments, it was previously determined that impact damage was cumulative, i.e. that samples which survived multiple small drops would then fail after fewer 5-foot drops than those that had not undergone the prior sequence of small drops.

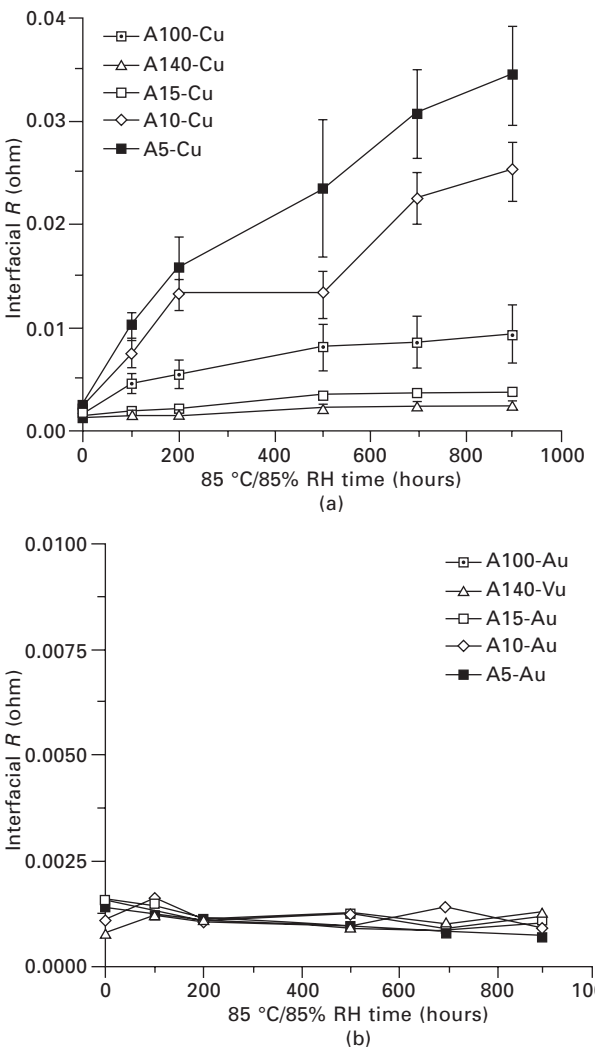
When investigating plasma surface treatments, it was discovered that vacuum exposure of the ICA prior to cure, improved contact adhesion significantly, by elimination of bubbles from the material (Morris *et al.*, 2001). If cure proceeds too quickly, the organic solvents used to control viscosity for printing or dispensing cannot escape, and become entrapped in the epoxy as bubbles, and bubbles at the contact interface have also been correlated with weak ACF adhesion (Kim *et al.*, 2006). This could explain why impact failure is almost always interfacial rather than cohesive, despite the governing property being the bulk loss modulus, and is consistent with a cumulative damage model, with crack initiation at the bubbles. Bubble content can also be reduced or eliminated by a pre-cure heat soak at a temperature sufficiently high to drive out volatile components, but low enough for a negligibly small cure rate (Perichaud *et al.*, 1998).

4.3.2 Galvanic corrosion

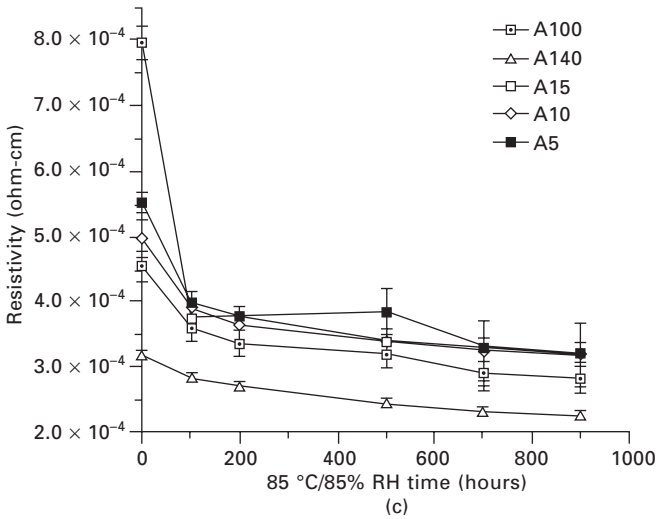
Contact resistance reliability problems (Li *et al.*, 1995; Liu *et al.*, 1996; Botter *et al.*, 1998) are primarily due to galvanic corrosion between dissimilar metals at the interfaces (Lu *et al.*, 1999a,b; Lu and Wong, 1999b) in the presence of water, with the resulting oxide leading to micro-crack failure (Kim *et al.*, 2008). The resistance drift can be reduced or slowed by the addition of corrosion inhibitors, oxygen scavengers, and/or sacrificial anode material to the polymer matrix (Tong *et al.*, 1999; Lu and Wong, 1999a, 2000e; Takezawa *et al.*, 2002), and moisture can be minimized with anhydride-cured epoxies (Lu and Wong, 2000d). However, these techniques can only

delay or reduce the effect, which requires the selection of compatible filler and contact materials.

Examples of resistance changes with time under 85/85 conditions (i.e. at 85 °C in 85% relative humidity) are shown in Fig. 4.9 (Li, 1996; Klosterman *et al.*, 1998), for Ag-filled ICA on Cu and Au contacts, where the contact and bulk resistances have been separated by combined three- and four-terminal measurements. The markedly different responses on Cu and Au correspond to their positions in the electrochemical series, relative to Ag.



4.9 Ag ICA contact resistance changes at 85/85: (a) on Cu contacts, (b) on Au contacts, with (c) corresponding bulk resistivity variations.



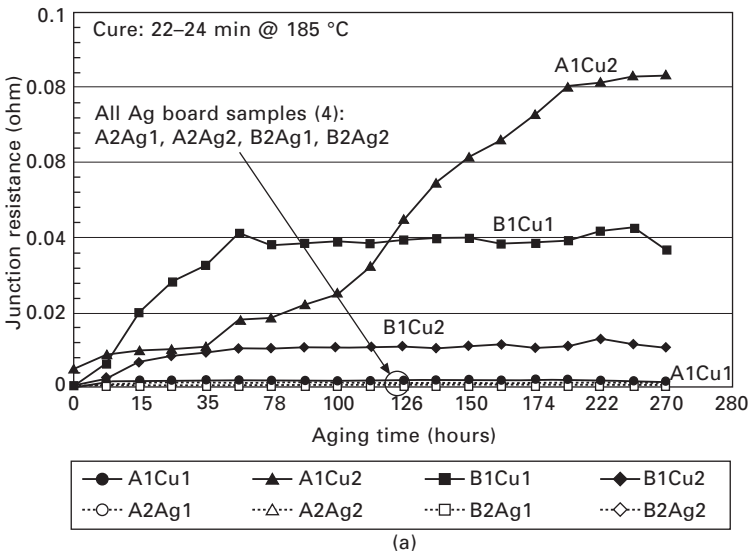
4.9 Continued.

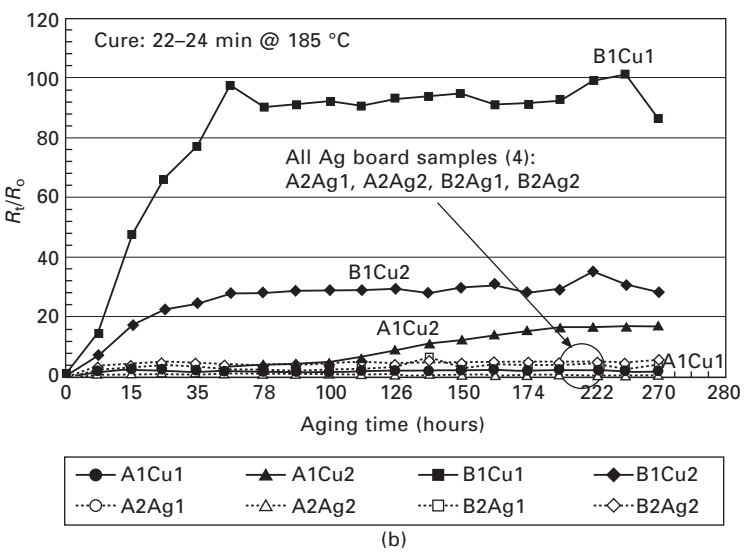
The Cu/Ag contact resistance increases significantly (Fig. 4.9a), while there is essentially no change in the Au/Ag contact (Fig. 4.9b). The reduction in the bulk resistivity (Fig. 4.9c) is attributed to continued curing at 85 °C.

As part of the elimination of Pb from electronics, PWB metallic finishes must also change from hot-air-leveled SnPb solder to alternatives such as immersion-Ag, immersion-Sn, electroless-Ni/immersion-Au, or organic solderability preservative (OSP) (Pas, 2005). Immersion-Ag is especially becoming the Pb-free final finish choice for many OEMs in the telecommunications, computer, automotive, and consumer electronics industries. For Ag-filled ICAs, the use of an immersion-Ag PWB surface finish would completely eliminate the galvanic corrosion potential (Lee *et al.*, 2009) at the interface, leaving only the slower Ag oxidation and no insulating by-product.

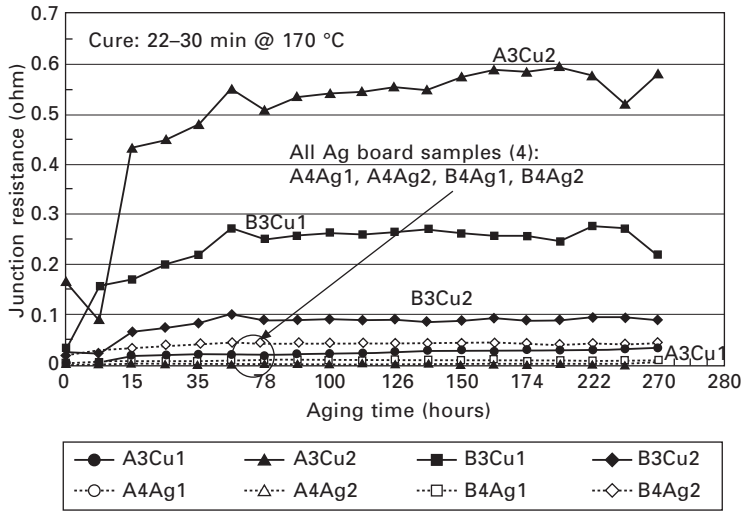
The contact resistances of two different Ag-based ICAs, designated A and B in Fig. 4.10, were studied under 85/85 conditions as functions of time, with the data for contacts to Cu and immersion-Ag PWB tracks being shown as both absolute resistances and their ratios to initial values. The total measured resistance includes both the bulk and interfacial contact resistances, with the changes assumed to be dominated by the ICA/pad interface since the bulk resistivity has been shown to be relatively stable during aging, provided it is adequately cured. Two curing schedules were used: 27–30 minutes at 185 °C, and 22–24 minutes at 170 °C, with the initial degree of cure expected to be significantly greater for the former. The degree of cure does not seem to affect ICA-A resistance values, but ICA-B appears to be under-cured at 170 °C, with initial resistances about two orders higher than the 185 °C values.

The contact resistances are smaller for the immersion-Ag PWBs, which are also more stable, as expected. Nevertheless, there are small resistance increases observed for the Ag/Ag system, consistent with simple oxidation. Since the resistances of the 170 °C ICA-B samples also increase appropriately for the Cu or Ag contacts, the contact resistance is apparently increased by





4.10 (a) and (c) 85/85 junction resistance shifts; (b) and (d) ratio of resistances to initial values (ICAs A and B, curing at 170 °C and 185 °C, Cu and immersion-Ag pad surfaces).



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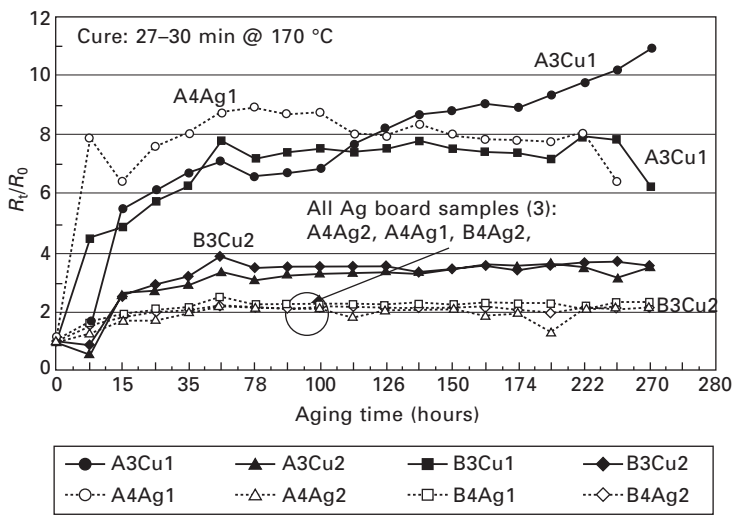

(d)

4.10 Continued.

under-curing, as well as the bulk resistivity. (Note that the resistance of sample A4Ag1 is anomalously low, which leads to the exaggerated plot for small changes in Fig. 4.10d. By contrast, the contact resistance of sample A3Cu2 is much larger than for any others, for unknown reasons.)

4.3.3 Other reliability problems

Apart from galvanic corrosion issues, the choice of surface contact metallization is also important for interdiffusion and intermetallic formation, e.g. Sn

from Sn-Pb finishes diffuses into the Ag filler at 150° C (Yamashita and Suganuma, 2006a,c).

Interfacial and bulk fracture mechanisms have been studied by Gupta *et al*. (1999), but in SMT applications the thermoplastic properties of the polymer lead to the accumulation of plastic strain, which initiates cracking (Perichaud *et al*., 1998). To understand the degradation mechanisms, Mo *et al*. (2002) focused on the electrical performance of a commercial ICA joint under mechanical loading. To gain insight into the electrical degradation mechanism, finite-element modeling (FEM) was executed, and the effects of mechanical loading on the initial intimate interaction among Ag fillers were analyzed.

Polymer creep coefficients are much higher than those of solders, and modeling predicts that ICAs would out-perform solder on room-temperature mechanical cycling tests by an order of magnitude (Rusanen, 2000). However, thermal cycling results do not show similar benefits (Nysaether *et al*., 2000), presumably due to exceeding T_g. Note also that Ag/epoxy interfacial fracture is suggested by initial wear effects (Constable *et al*., 1999), and has been directly observed (Chen *et al*., 2006).

The effect of moisture on ICA polymer degradation has been studied by Khoo and Liu (1996), with moisture distribution within the joint being modeled by Dudek *et al*. (2005) for viscoelastic modeling of thermal cycling failure. Hygroscopic strain due to moisture absorption can be found from measured strain by subtracting the calculated thermomechanical component (Low *et al*., 2007).

There has been past concern about the possible field-driven surface migration of Ag ions in the presence of water, which can lead to short circuits between adjacent ICA contacts (Suzuki *et al*., 2004). Sancaktar *et al*. (2004) have recently correlated electromigration with Ag surface pitting, and there also appears to be a field threshold (Mo, 2005). Systematic study is required to establish the boundaries to the effect (Manepallis *et al*., 1999), which is minimized by the use of high-purity Ag (with <10ppm Cu (Detert and Herzog, 1999), or Sn-Ag alloys (Suzuki *et al*., 2004; Toida *et al*., 2005) as Sn appears to inhibit migration. Ag migration is evident in un-cured material (Morris *et al*., 1999), but it is not a problem in commercial products where it has been suggested that additives to the polymer seal the silver surface, defeating migration tendencies. Recent work (Yi and Wong, 2006a,b) has shown that a molecular self-assembled monolayer (SAM) of short chain di-carboxylic acid, for example, does just that. The addition of an appropriate SAM to particle surfaces also enables metal loading beyond the practical limit normally set by material viscosity (Jiang *et al*., 2005).

At high current densities, electron momentum transfer drives mass electromigration within ultra-small flip-chip solder connections, and one might expect to see the same phenomenon in ICAs at the small contact points

between Ag filler particles. Joule heating at these points of high resistance, however, causes localized polymer degradation, and this is the high current failure mode (Kotthaus *et al.*, 1998). The effect has been demonstrated for three commercial ICAs (Morris *et al.*, 1999), where surface temperature rises with the square of current. The sample temperature coefficient of resistance can be used to determine the internal temperature from the resistance increase with current, and failure is observed when the internal temperature reaches the polymer degradation temperature.

4.4 Modeling

4.4.1 Electrical modeling

While there have been some superficial efforts at structural modeling of ICAs, comparisons of electrical models with experiment are either strictly qualitative or fitted by parameter adjustment. For flakes of 10μm diameter by 1μm thick, and μm-sized smaller particles, the electron mean free path (mfp) is essentially bulk value, with no accounting required for size effects, other than for the constriction resistance at inter-particle contacts. (Note that the mfp is limited by particle dimensions in nano-particle ICAs (Kotthaus *et al.*, 1996).

Li and Morris (1997) developed electrical conduction models for Ag-filled ICAs, combining the microscopic resistance of the bulk Ag particles and the contact between Ag flakes with the macroscale resistor network calculation by percolation theory, confirming the effects of surface layering at a qualitative level, and bimodal size effects (Li and Morris, 1996; Sancaktar and Dilsiz, 1998). The model predicts the resistivity change as stress develops during the cure process, and variations with particle size distributions (Li and Morris, 1996). A dynamic model of compression effects demonstrates flake alignment quite dramatically (Mundlein and Nicolics, 2004), but as the structure fills up, the modeling process becomes more and more time-consuming. A potential energy technique has proved effective (McCluskey *et al.*, 1998) in reducing computation time, and compression algorithms can be applied to initially well-separated particles (Mustoe *et al.*, 1999; Mundlein *et al.*, 2002; Mundlein and Nicolics, 2004, 2005). Su and Qu (2004) extended this compression concept by modeling the curing process itself. The extension of structural modeling to electrical properties requires the assumption of intra-particle, inter-particle, and contact conduction processes, with the structural model itself providing the percolation component.

4.4.2 Cure modeling

Klosterman *et al.* (1998) focused on the influence of cure on resistivity, joint resistance and reliability, with novel analytical methods developed to

define the cure conditions for optimum electrical properties and stability. The cure process seemed to have been modeled successfully by very simple mathematical expressions (Li and Morris, 1999), on the basis of a rapid resistance drop as the model 100%-cure point was approached. That success has been questioned, however, by the correlation of such resistance drops at ~20% degrees of cure (Inoue and Suganuma, 2006).

A review of polymer cure models used in microelectronics packaging applications reveals no clear consensus of the chemical rate constants for the cure reactions, or even of an effective model. The problem lies in the contrast between the actual cure process, which involves a sequence of distinct chemical reactions, and the models, which typically assume only one reaction (or two, with some restrictions on the independence of their characteristic constants). The standard techniques to determine the model parameters are based on differential scanning calorimetry (DSC), which cannot distinguish between the reactions (Morris *et al.*, 2009a), and hence yield results useful only under the same conditions, which completely misses the point of modeling. The obvious solution is for manufacturers to provide the modeling parameters, but failing that, an alternative experimental technique is required to determine individual reaction parameters, e.g. Fourier transform infra-red spectroscopy (FTIR).

Thermally cured epoxies and other polymers are extensively used in electronics packaging, as encapsulants, underfills, and adhesives, etc. The project which prompted this study was the microwave cure of a carbon-loaded epoxy encapsulant (Tilford *et al.*, 2007). The temperature rises more rapidly than in conventional isothermal or reflow oven curing systems, and the cure proceeds more uniformly within the material. Optimization of the microwave power level and application time cannot be readily accomplished experimentally, especially given the speed of the cure, so simulation is seen as the tool to sensible planning of the process development. A literature review was the obvious first step to establish the model, including the thermal dependences of the chemical reaction parameters. Prior experience suggests that the simplest first order model has proved to be effective in ICA applications, with the model parameters relatively easily obtained by DSC measurements (Klosterman *et al.*, 1998).

For α = degree of cure, the basic assumption of all models is that the reaction rate can be expressed as a function of reactant concentration, $f(\alpha)$, by

$$d\alpha/dt = K f(\alpha), \qquad [4.1]$$

with the temperature-dependent chemical rate constant $K = A \exp(-E/RT)$, for rate parameters A and E, and $R = 8.31$ J/K.mole. The models vary in the assumed form of $f(\alpha)$, as listed below.

(a) nth order model: $f(\alpha) = (1 - \alpha)^n$ [4.2]

- in this case, we can find α analytically (for constant T, i.e. isothermal cure) as:
  - 1st order: $d\alpha/dt = K(1 - \alpha)$, $\therefore \alpha = 1 - \exp(-Kt)$ [4.3]
  - 2nd order: $d\alpha/dt = K(1 - \alpha)^2$, $\therefore \alpha = 1 - [1 + Kt]^{-1}$ [4.4]
  - nth order: $d\alpha/dt = K(1 - \alpha)^n$, $\therefore \alpha = 1 - [1 + (n - 1)Kt]^{-1/(n-1)}$ [4.5]

(b) Auto-catalyzed model:

- single-step: $d\alpha/dt = K f(\alpha) = K \alpha^m (1 - \alpha)^n \ldots$

  but note $d\alpha/dt = 0$ for $\alpha = 0$ [4.6]
- double step (linear combination): $d\alpha/dt = (K_1 + K_2 \alpha^m)(1 - \alpha)^n$ [4.7]
- modified double step: $d\alpha/dt = K f(\alpha) = K(y_1 + y_2 \alpha^m)(1 - \alpha)^n$

  where $y_1 + y_2 = 1$ [4.8]

Note that only the double step auto-catalyzed model has more than a single chemical rate constant in the model, i.e. all others implicitly assume a single cure reaction, or at least a single rate-controlling reaction across the full temperature range of interest. In practice, for example, the cure of bisphenol-A diglycidyl ether (BADGE), a commonly employed epoxy in packaging applications and ICAs, requires two steps (with a third reaction occurring at high cure temperatures). So the double-step auto-catalyzed model is the *only* one with a realistic physical basis that can be expected to apply outside the measurement conditions used to determine A and E. This view is supported by published model data for a variety of polymers where the single-rate constant parameters, A and E, vary with temperature and/or degree of cure, making them applicable only within the range of measurement conditions, i.e. not useful as predictive tools. There are mathematical techniques available to extract the chemical rate parameters from isothermal and/or dynamic DSC data, but only the isothermal DSC scan can yield K_1, K_2, m, and n, (i) by plotting $d\alpha/dt$ versus α and varying the four parameters for best fit, or (ii) by plotting $d\alpha/dt$ versus $(1 - \alpha)^2$ with the assumption of $m = 1$, $n = 2$, (which seems to be supported by some data), or (iii) finding K_1 and K_2 independently from DSC peak values, but with m, n assumptions still required. Arhennius plots of K_1, K_2, then yield A_1, A_2, E_1, and E_2. Ko *et al*. (1994) have extracted consistent A_1 and E_1 values from DSC BADGE data, but m turns out to be temperature dependent, calling the A_2 and E_2 values derived from m into question.

The two BADGE reactions can be written as:

(i) primary amine + epoxide → secondary amine
(ii) secondary amine + epoxide → tertiary amine

neither of which totally dominates the rate control. The double step auto-catalyzed model could conceivably be re-written as:

$$d\alpha/dt = [K_1(1 - \alpha_1)^{m1} + K_2(\alpha_1 - \alpha_2)^{m2}](1 - \alpha)^n \quad [4.9]$$

where α is the fraction of reacted epoxides, so $(1 - \alpha)$ is the fraction remaining, and the primary, tertiary, and secondary amine concentrations are $(1 - \alpha_1)$, α_2, and $(\alpha_1 - \alpha_2)$ respectively.

The obvious solution is for manufacturers to provide full and accurate modeling parameters in their material data sheets. However, FTIR studies of the cure process have been shown to be capable of distinguishing between the successive reactions. It is proposed that FTIR replace DSC in the determination of cure model parameters (Morris *et al.*, 2009a,b).

4.4.3 Flow modeling

There is a real need for a better understanding of the ICA dispensation process, whether by stencil or screen printing, or by syringe. Flow modeling of these processes would enable a scientific approach to the problem of flake layering, for example. As for all modeling, one must first have the real material parameters, and so the first step has now been taken with the work of Zhou and Sancaktar (2008a), who measured and modeled (fitted) the rheological properties of highly filled Ni/epoxy ICAs as functions of loading, shear rate, temperature, and time (degree of cure), providing input parameters for process flow modeling. In a separate study (Zhou and Sancaktar, 2008b), they identified stratification zones within a dispensation syringe, with Ni particles concentrating in a Ni-rich region at the tube circumference and the center/axis of the tube being epoxy-rich. As dispensation proceeds, pressure-induced 'filtration' occurs, with epoxy-rich material dispensed from the center, enriching the Ni content of the residue, which then requires increased syringe pressure to maintain the mass flow. Under certain circumstances, the pressure can oscillate as it increases under flow rate control. Process modeling can lead to greater understanding of these phenomena and their minimization.

4.5 Nanotechnologies in isotropic conductive adhesives

4.5.1 Nanoparticles

Kotthaus *et al.* (1997) reported on an ICA filled with porous aggregates of nano-size Ag particles, with the goal of decreasing the metal loading to

improve adhesion. Total metal loading can be decreased with good electrical conductivity using a bimodal filler distribution (Fu *et al.*, 1999; Mach *et al.*, 2008), but the nanoparticles increase resistivity for given total filler content, due to mean free path limits and increased numbers of contacts (Wong *et al.*, 2005), although small decreases have been observed (Zhang *et al.*, 2008).

Moon *et al.* (2005) show the thermal behavior of silver nanoparticles with respect to the sintering reaction, which is critical to the effective use of nanoparticles to improve ICA performance (Bai *et al.*, 2005; Jiang *et al.*, 2005, 2006). Surface changes of the particles during sintering and crystal structure variation are also addressed in Moon *et al.* (2005). Nanoparticle shapes can be controlled during synthesis by inclusion of $AgNO_3$ into the epoxy resin (Pothukuchi *et al.*, 2004).

Ye *et al.* (1999) observed apparently sintered contacts of ~50nm diameter between Ag nanoparticles in an ICA, and similar contacts have been observed between micron-scaled ICA particles (Li and Morris, unpublished data, 1994). Sintering is clearly the key to the lower ICA resistivities appearing in the literature, and may explain the results of Yang *et al.* (2008) by a 'room temperature sintering' process (Wakuda *et al.*, 2008) of enhanced surface diffusion following removal of the protective surface layer.

Surface treatment of the flakes by a 0.2% 'reactive' solution in ethanol, following an ethanol rinse, dramatically lowers resistivity from 100 $\mu\Omega \cdot$cm to 6 $\mu\Omega \cdot$cm, presumably also by sintering, and doubles adhesion from 2.5MPa to 5MPa (Yang *et al.*, 2008).

4.5.2 Carbon nanotubes (CNTs)

The addition of carbon nanotubes (CNTs) to the Ag-flake/epoxy mix (Lin and Lin, 2004) lowers the percolation threshold, as expected, and may be more effective than Ag nano-particles. Ag nanoparticle/nanowire mixtures have also been demonstrated (Wu *et al.*, 2006a,b), as have CNT/epoxy composites (Li and Lumpp, 2006; Zhang *et al.*, 2008). However, the high resistance contacts between CNTs limit the conductivities that can be achieved with random-structure composites (Yan *et al.*, 2007). Nevertheless, CNTs will be used in the future as supplementary filler, between the Ag flakes, to both reduce resistivity and improve impact resistance (Heimann *et al.*, 2007, 2008; Wirts-Ruetters *et al.*, 2008).

4.6 Conclusions

Fundamental materials research has yielded greatly improved understanding of ICA reliability test results. However, the conventional wisdom remains that ICAs will continue as a niche technology where new no-Pb solders cannot be used. Impact strength remains the primary liability for more widespread

adoption. Since the failure mode is usually adhesive rather than cohesive, even though failure correlates with the polymer loss modulus, greater attention to crack initiation at the contact interface should yield dividends. The other long-standing fundamental issue in the field is a detailed understanding of the inter-particulate interface, and its electrical properties (Morris, 2005, 2007).

Newcomers to the ICA field are encouraged to seek out other reviews. Li and Morris (1998) cover basic background principles, while Liu and Morris (1999) offer a more comprehensive technology review, updated in Morris and Liu (2006) and Morris *et al*. (2010). The most complete sources of information are dedicated books (Liu, 1999; Gomatan and Mitttal, 2008; Li *et al*., 2010), with the last of these focused on nanotechnologies, and an on-line course (Morris and Liu, 2000 at www.cpmt.org). A new book (Morris, 2011) is in current development.

4.7 References

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