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Electrically Conductive Adhesives: Basic Understanding and Fundamental Problems.

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Abstract

The development of isotropic electrically conductive adhesives has reached the point where the principal technical problems of the past few years have been solved. In particular, process (cure) schedules can be defined as parameter windows, rather than by single target parameters, contact resistance drift has been identified as due to corrosion and solved by compatible material selection, resistivities have been brought down by the use of bi-modal silver flake as filler, adhesion has been improved by surface treatments, and the source of impact resistance failure has been identified. The time is right, therefore, to turn to some of the more fundamental aspects of what is really quite a complex system, and to develop a better understanding of the physical mechanisms of both electrical and mechanical properties, in order to continue the improvement of these composite materials.

Introduction

There has been steadily increasing interest over the past decade in the potential replacement of solder for electronic component interconnect by electrically conductive adhesives (ECAs) in both the isotropic and anisotropic material forms (ICA and ACA). ACAs are available as both paste and film (ACF), and there is also a related non-conductive adhesive (NCA) technology. Due to space limitations, this paper will be limited to the discussion of ICAs, which are metal-polymer composites, with conductivity provided by a continuous metal path through the polymer matrix, and adhesion provided by the polymer [1].

There has been considerable progress in both the development and understanding of ICAs, but work has been concentrated on the solution of a specific principal problem at each stage of the material's development: first on the reduction of electrical resistivity, then on the improvement of adhesive strength for materials with satisfactory electrical performance, and most recently on the solution to poor drop test performance. The research has been focused almost exclusively on commercial products and funded by industry, albeit often by consortia. With the solution to the last of these problems in sight [2], and with the materials' wide-spread adoption more likely, it is now more important to fill in some of the gaps in our understanding of the materials' properties.

The driving motivations for ICA research have been primarily environmental, the continual drive towards finer pitch interconnect, and the advantages of lower process temperatures. The expectation of legislated limits on the Pb content of electronic products is an issue which comes and goes with national and international political climates. It is a stronger driving force in Europe than in the U.S.A., but in a global market it will be the most demanding standard in any of the major markets which will determine the technology for all. Pb-free solders are still the subject of intense research, but the serious candidate materials generally all exacerbate the existing problems of high temperature processing. It is the low process temperature which accounts for most current niche ICA applications, usually involving thermally sensitive components or process sequencing.

A few years ago, the superior ICA fine pitch properties which derived from smaller particulate sizes and a lesser tendency to "slump" looked like the technical advantage which would drive the materials' continued development, but the general switch from peripherally leaded

devices to area arrays has eased this particular pressure for the time being. At first, it was predicted that ICAs would be unsuitable for these applications, not possessing solder paste's capacity for self alignment. However, the intense effort made to improve manufacturing alignments for fine pitch peripheral lead devices means that the technology is already sufficiently advanced to guarantee adequate manufacturing placement accuracy without need for the self-alignment feature. Nevertheless, the drive toward higher I/O lead-count will continue, and will catch up with current capabilities, at which point either further placement accuracy advances or self-alignment will be necessary. It should be realized that the lack of a self-alignment capability is not inherent in the ICA concept, but depends solely upon the choice of polymer adhesive and its surface tension [3]. The introduction of a self-aligning ICA would be a most interesting development.

It has also been stated that the traditional motivations listed above are not the most critical at all, and that the much more important reason to replace solder is the continuing and inevitable drive towards smaller and smaller joints, whereby the time required for joints to fail from the formations of voids, brittle inter-metallics, etc., in solder alloys and contact interfaces will decrease as the square of the dimensional shrinkage [4]. Pb-free solders would not solve this problem.

By-and-large, the problem with solder replacement by ICAs has been seen to be a lack of adhesive strength in materials with quite adequate electrical properties, due to the trade-off in properties as the metal-polymer mix is varied. With progress achieved on the adhesion problem, attention turned to drop-test failures, and the well-known sobering demonstrations of components springing off boards dropped from very modest heights [5]. As materials evolved, new applications have been developed, e.g. the application of ICA materials to micro-via fill [6,7]. The only critical property here is electrical, and both adhesion and impact resistance requirements are much more easily satisfied.

ICA Structure

As the proportion of metal (typically silver) in the polymer matrix is increased, the resistance drops only slightly until the "percolation threshold" is reached, when the first continuous metal path is established through the composite material [8]. The resistance continues to drop more slowly as multiple parallel paths are developed with the continued addition of more metal filler. Ideally one would like to use the minimum quantity of filler necessary to pass the threshold, but in practice manufacturing tolerances require a design target composition significantly beyond the threshold. Very small contact volumes increase the statistical spread of resistivities already inherent in a percolation structure. Minimal filler content is a requirement for both economic reasons (since silver is expensive) and to maximize the proportion of polymer adhesive. Both issues can be addressed by the use of metal flakes (or rods) instead of spheres as filler. The benefits can be readily understood by considering the extreme case of flakes of zero thickness, which would clearly establish a percolation threshold at zero metal content, i.e. at 100% adhesive with zero filler cost. Practical commercial materials achieve substantial reduction in the threshold composition by the use of flakes, by virtue of the increased connectivity which accompanies the increased surface-to-volume ratio, compounded by bi-modal particle size distributions. The efficacy of bi-modal particle distributions has been demonstrated [9] and both flakes and powders are used for the smaller particles [10].

There has been substantial progress in the understanding of the fluid mechanics of liquids containing solid particles, building on earlier work on coal slurry transport, etc. This work has been aimed at the modeling of capillary underfills [11] or ACA flow [12] (which would be directly applicable to "no-flow" underfills [13].) These models are still highly idealized, and accurate modeling clearly becomes progressively more difficult as the solid particle content increases. Practical ICA flow modeling would be further complicated by the particle shapes, and by the variations in surface and edge roughness. It may

be reasonable to ignore this last complication, given the documented use of surface lubricants in commercial materials, but it is essential to begin the task of extending flow modeling to the syringe dispensation and screen or stencil printing of ICAs if one is improve the manufacturing process on the basis of real understanding. In particular, the origins of surface layering of the flakes must be understood. This effect, where the flakes at the surface of the material seem to be aligned parallel to the surface to a depth of a few flake thicknesses, seems to be universal, and could be due to squeegee drag, but its observation with syringe dispensation and inside air bubbles argues for a surface tension interpretation. The particulate detail at the surface is important for predictions of both adhesive strength and electrical resistance.

There are various novel approaches to the improvement of electrical connectivity at a given metal content, including magnetic alignment of nickel filler rods [14], the use of polymer particles to force alignment of flakes [15], etc. Anything that increases the packing density/efficiency of the particles also increases the connectivity and reproducibility of the structure, permitting the design composition to move closer to the percolation threshold, and a reduction in metal filler. Vibration of the print head or dispenser may achieve this goal.

Modeling of the material structure has been directed to date at the problem of accurate prediction of the electrical properties, but needs to be generalized in future to the modeling of the full range of structure-dependent properties. The sequence of computer models has progressed so far from uniformly sized spheres [16], to 2D [17] then 3D [18] rectangular particles with limited (but randomly distributed) size variations randomly oriented in the x, y, and z directions, and most recently to the extension of the rectangular particle model to 360° rotations about all three axes in 1° increments [19]. The next extensions must be to flake representations, and bimodal distributions, but this is the step that requires a dramatic increase in computing power. In all cases, the difficult part is the development of an efficient algorithm for the random placement of the particle, which of course must not impinge upon the space occupied by another. As the structure fills up, this process becomes more and more time consuming, given the constraint that the resultant structure cannot display any artificial effects of simplifying algorithms that do not reflect an actual physical process.

ICA Electrical Properties

The electrical resistance of the ICA has four distinct components [8]. Three of these are obvious; they are the metal "intra-particle" resistance, the "inter-particle" contact resistance, and the "contact" resistance between the surface particles and the lead or contact pad. The fourth component comes from the meandering "percolation" path of the continuous metallic connection(s) through the material.

Percolation theory is well developed for the elementary system of uniform conducting spheres (or cubes) in a perfectly insulating medium [20]. There are no analytical solutions, and the theoretical results are deduced by the averaging of multiple Monte Carlo randomized simulations. Conducting particles are randomly assigned to sites on a specified regular array. For any realistic random system, the site separations must be much less than the particle sizes, and the problem becomes how to fit new particles into the structure at high particle concentrations (as required here) with reasonable efficiency. The percolation modeling literature commonly includes finite intra-particle resistivities, but inter-particle resistances are seen less often. This is partly because the most common systems of interest would assume electron tunneling between particles, and the exponential dependence of the tunneling probability on separation introduces a strong parametric dependence on a poorly characterized variable. Percolation models of the electrical resistance of ICA systems have included both intra-particle and inter-particle resistances, but with both grossly approximated so far, e.g. with the simplifying assumption of uniform tunneling thickness [16-19]. (To accommodate 1nm variations in a 1-10nm tunneling separation range between particles requires an underlying

simulation grid with a 1nm pitch in a brute force approach, i.e. a substantial increase in resolution over that otherwise required for micron sized particles. It would be more efficient, however, and just as valid to superimpose the tunnel gap distribution on contacting islands distributed on a coarser grid.) The structural modeling requirements have been outlined above. Electrical modeling requires the addition of the conduction processes discussed below to each of the elements: intra-particle, inter-particle, and contact, with the structural model itself providing the percolation component. Existing models confirm the effects of surface layering at a qualitative level [18], and size effects [16, 21], i.e. the increase and decrease in effective conductivity respectively for limited ICA sample dimensions parallel and perpendicular to current flow. As models improve, one expects eventual quantitative agreement. It is probably satisfactory to continue to ignore the effect of finite polymer conductivity, but this assumption should be checked for new materials. One prediction of percolation theory which has never been validated in these systems is the frequency dependence of the conductivity in terms of the coherence length [22], which appears to be masked by the particle skin effect (see below.)

Resistivity measurements must be made on genuinely isotropic samples of suitable size, unless size or layering effects are the actual object of the measurement. On the other hand, the interconnect application will actually include both. So will measurements along a long thin sample, but with the opposite effects [8]. The measurement of small resistances with sufficient sensitivity to detect early corrosion, etc., is difficult and usually accomplished by depositing the long specimen just mentioned or by daisy-chaining multiple interconnect samples. In future, the impedance transformer should see increased application to solve this problem [8].

It is noted here that there is evidence of percolation chains dropping in and out of the conduction paths during thermal cycling. This effect is demonstrated by reproducible hysteresis in resistance versus temperature plots during thermal cycling of ICA structures where only a limited number of percolation paths are expected to exist, i.e. for very small contact areas. [23]

Inter-particle conduction is generally assumed to take place directly from metal to metal, or by tunneling through an insulating layer, whether of intervening polymer or of surface contaminants, or by conduction through a surface oxide film, which for silver would be a conductive degenerate semiconductor. There is a range of tools available to characterize conduction mechanisms, including frequency effects, non-ohmic high field behavior, temperature dependencies, etc., and the absence of any observation of negative temperature coefficient of resistance (TCR) or non-ohmic behavior is sufficient to eliminate most other conduction mechanisms from contention. Silver is typically tarnished, and presumably would oxidize within the polymer, even if initially "clean," but it is not clear what effect the surface lubricants identified on flake surfaces [24] would have on this process. An XPS surface analysis of the flake surfaces should be carried out to distinguish between the presence of silver oxide and the oxygen content of the lubricant and/or polymer residue.

No matter which of the mechanisms apply at the gap itself, the contact area is accepted as being typically small, of diameter, d, 10nm or less [25]. Clearly there will be some constriction of current flow between particles at the contact, and the Holm theory specifies this contact resistance to be $\rho/2d$ [8]. With the constriction resistance proportional to d, and the tunneling or oxide resistances proportional to d^2, one should be able to identify the dominant contribution from a pressure dependence, which has been theoretically matched to experimental data for a d^2 dependence, but not for an ICA [26]. The internal pressure exerted by the curing process will be discussed further below. Returning to the differences between the contact conduction mechanisms, the TCR will be zero (or slightly negative) for tunneling and positive for the oxide. Unfortunately, it is difficult to find data on the exact electrical properties of the oxide, which are subject to local formation conditions, so it is not known whether the TCR would be less or greater

than the metal particle TCR. It would appear from the formula that the constriction resistance would have the same TCR as the metal particles', but the derivation does not include the mean free path reduction which will be associated with contact dimensions less than the bulk value in the particles; this extension to the theory is necessary.

The inter-particle conduction mechanism remains undetermined, and the points made above, which reflect those in the literature, are very general. What is needed is a comprehensive basic study of the metal-polymer interface, to investigate charge transfer and band effects in the polymer(s), time and temperature effects, and how the process proceeds during curing. In addition, actual contact points need to be located and isolated, and the potential distribution plotted across the boundary from one particle to the other. Electrical noise measurements are also often a useful diagnostic tool, and there is noise data in the literature [27], but as is often the case, the interpretation is ambiguous.

Frequency dependencies are easily determined and can be definitive in the identification of some conduction mechanisms. In the ICA case, a.c. measurements were expected to short circuit the tunnel gaps between particles, with the corner frequencies providing the means to separate out the particle resistance from the inter-particle gap contribution. The method was validated by a.c. measurements before cure. Resistivities below the percolation threshold decreased with frequency to limiting values similar to those above it [28]. (This experiment suggests the use of impedance spectroscopy as a manufacturing quality test for ICAs, as for solder paste.) When applied to cured ICAs, however, no such effect was observed (see below), so either there is no tunneling gap (i.e. conducting oxide or metal-metal contact) or the tunnel resistance is just much less than the particle resistance [8, 10].

Intra-particle resistance accounts for a substantial proportion of the measured ICA resistance, and in some cases essentially all of it. This conclusion is based on TCR measurements on a variety of commercial ICAs, where the TCR values range downwards from the bulk metallic value, but are always positive [8, 10, 23]. Within the limits of experimental accuracy, the data are consistent with a model of the intra-particle metallic resistance in series with a zero TCR contact resistance, but cannot be totally conclusive. Thermal testing needs to be extended to much lower temperatures to resolve this point.

For ten micron diameter flakes one micron thick, and micron-sized smaller particles, the electron mean free path is not going to be reduced significantly from the bulk value, and no accounting is needed for size effects in the particles. (Note that this would not apply to the nano-particle ICA variant [29].) But the nature of the surface (i.e. rough or smooth for diffuse or specular scattering) could be important for the assessment of mean free path limitation for constriction resistance.

The a.c. measurements mentioned above were run on the same ICA materials which gave TCR s identical to the bulk value for silver, and so it is not surprising that the a.c. characteristics were in total accordance with the predictions of skin effect resistance and inductance for silver [10]. These experiments should be duplicated for materials with greater inter-particle resistances.

Contact resistance can be isolated from the bulk composite resistivity by the combination of three-terminal measurement with the more common four-terminal [30]. It is the contact resistance which has been shown to be the source of electrical reliability problems [31-33]. The oxidation/corrosion of copper or the tin in Sn/Pb contact pad or lead coatings has been demonstrated, and explains the greater long term stability of noble metal contacts (gold or palladium) under thermal cycling and 85/85 stress testing. More recently galvanic corrosion has been identified between dissimilar metals [34]. One would fully expect to see the effects of interfacial diffusion in the longer term (albeit limited by low process temperatures), and the formation of brittle inter-metallics; although these would probably have no discernible effect on mechanical reliability, the electrical impact could be significant, given the limited number of low diameter contact points to the percolation paths.

The solution to these problems would be to eliminate the dissimilar materials, and the use of silvered contacts with silver-based ICAs would seem logical. However, silver on copper introduces much the same problems, and requires a barrier layer (of nickel for example.)

ICA Mechanical Properties

The vast majority of the published ICA data is comprised of electrical resistance and adhesive strength measurements, both presented in the context of reliability testing. But while there has been some parallel effort to interpret the electrical properties in terms of both structure related and physical failure models, there has been little similar effort to understand the mechanisms of adhesion at a comparably fundamental level. Obviously, such a study requires a systematic approach to the measurement of adhesive strengths for a matrix of metal surface and polymer combinations. It is clear that surface cleanliness plays a crucial role in effective adhesion [35] (and one which may be overlooked in the desire to apply ICAs as drop-in replacements for solder), so surface treatments must be included as a secondary variable, with consideration of roughening effects also included. While this could be regarded as an empirical study, the fundamental goal of determining the relative contributions of various adhesion mechanisms (e.g. chemical, mechanical, electrical) should not be lost sight of.

Published ICA adhesive and shear strengths are on the same order as those of solder, usually a little less [36], occasionally higher [35], but anyway adequate. The problem has been the drop test failure rate, leading to the widespread adoption of the NCMS criteria [37] as a de facto standard. In general, it is the larger devices which are most at risk, and indeed current commercial materials seem adequate for smaller devices such as SMT passives, which have been in mass production with ICA attachment for some years [38]. Improved understanding of this particular phenomenon is currently leading to the development of ICA materials specifically designed to address the drop test problem. The key lies in the imaginary component of the complex Young's modulus, which represents energy dissipation in the material, as opposed to the energy storage of the simple deformation represented by the conventional form of Hooke's Law. The complex modulus is therefore directly analogous to the complex dielectric constant, and dynamic stress-strain relationship actually includes a phase shift. When one examines drop test survival data, the success rate correlates with the (imaginary) dissipation modulus rather than with adhesive strength [2]. One way to design materials with high dissipation modulus is to select polymers with glass transition temperature T_g below the operating range, i.e. below room temperature, in general. However, operating polymers above T_g carries its own penalties, e.g. higher temperature coefficients of expansion, so the next step is to develop adhesive polymer blends with high mechanical absorption without those disadvantages.

With the primary goals of satisfactory electrical performance, adhesion, and drop test survival all apparently within reach, attention will undoubtedly turn to the same testing regimen which dominates solder research, i.e. the coefficient of thermal expansion, mechanical compliance, fracture modes, and fatigue failure, all with the objectives of reliability lifetime prediction and extension. The expectation is that the compliance characteristics derived from the polymer base will be much better than solder's, and carry through to superior thermo-mechanical stress fatigue performance. Naturally the properties of the composite should be modeled in terms of the structure to demonstrate that their physical origins are understood.

ICA Materials

The polymers in common use in ICAs are thermoset epoxies, with sufficient thermoplastic mixed in to allow for softening and release for rework under moderate heat. Up until now, the polymers used in ICAs have been adapted from those developed for other purposes. The results of research programs specifically intended to develop polymer adhesives tailored to ICA applications

have yet to appear on the market, but in the meantime the identification of the need for mechanical energy dissipative materials may have already made them obsolete. Certainly the search for new materials must now focus on this property, but without compromising on others. Polymer adhesion is a fundamental property which must be understood, with fundamental contact angle wetting experiments for ICA base polymers a first step.

The cure process has been modeled successfully by remarkably simple mathematical expressions [30, 39], but which require accurately determined experimental data. The success is demonstrated by the observation of the sudden decrease in resistance, interpreted as indicative of complete cure, at the predicted point of 100% cure [25, 39]. The assumption is that the resistance drop is due to physical shrinkage of the polymer matrix with complete cure, but this particular property, i.e. physical shrinkage and the development of an internal pressure to squeeze filler particles together, has not been measured, and does not appear to have been modeled. The measurement of a dimensional shrinkage with cure should not be a major problem, nor should the measurement of internal pressures.

The metal of choice as filler has been silver, but the warning that silver is accompanied by electromigration problems keeps on coming up. Certainly the existence of the electromigration problem has been documented [40], but it does not seem to be a problem in practice. It is evident in un-cured material [41], and it has been suggested that commercial additives to the polymer seal the silver surface, defeating migration tendencies. There also appears to be a field threshold, and moisture is requisite, but systematic study is required to establish the boundaries to the effect. In addition, the diffusion and clustering of metals in polymers is well established [42, 43], and should be investigated for silver in appropriate polymers as the limiting zero-field phenomenon.

The approach of current research to establish a processing methodology based on process boundaries for underfill [44] should be immediately applicable to ICAs

Conclusion

ICA research reported in the literature has overwhelmingly been conducted on commercial product. There are three problems with the utility of the data:

1. With few exceptions, the materials are not identified, and other researchers cannot compare their data with the published results of others.
2. In addition, the commercial products contain elements which the manufacturers consider confidential, so the researcher may not have all the pertinent information about the product under study, leading to misinterpretation of the data. Both of these problems come from the confidentiality agreements which accompany the supply of test samples of current technology (often pre-release) to research groups.
3. The commercial product is probably too complex to isolate a single variable for study anyway. The standard research approach would be better, i.e. to begin with a fully known simple system, which can be accurately modeled, and add successive complications one at a time, so the effect of each is well understood, i.e. idealize, then add real elements.

This paper has been limited to the ICA field, but there are some variations on the common form which should also be mentioned. In particular, the fusible link materials must be mentioned. The fundamental point here is that the metallic particles consist of or are coated with a low melting-point metal (e.g. tin) which fuses the particles together and bonds them to a like contact pad at moderate temperature. The fused structure adds adhesive strength to the joint, there are no dissimilar metals involved, and the conduction process is totally metallic.

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