Reworkable Anisotropic Conductive Adhesive for Assembly of Medical Devices

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Abstract

Reworkable anisotropic conductive adhesives (ACAs) are of interest when the material is used for the assembly of electronic modules with high value, such as in medical ultrasound probes. Commercially available ACAs are generally difficult or even impossible to rework due to the common use of thermosetting adhesive matrix. ACA material with competitive performance compared to conventional ACAs and with reworkability at modest conditions is developed in this work. Adhesive matrices comprising a blend of a thermosetting epoxy and a thermoplastic polymer are selected because it has shown potential to ensure good electrical and mechanical integrity whilst still allowing reworkablity for ACA interconnects. This paper presents the findings of favourable mixing ratios between an epoxy system compatible with ACA applications and a high-performance thermoplastic polymer that offer good mechanical strength combined with reworkability. Die shear strength at varying temperatures relevant for production/storage (23 °C), operation of medical ultrasound probes (50 °C) and rework of ACA bonds (190 °C) is studied. Complete removal of adhesive remaining on bonding surfaces, for successful rework, is verified. The results show a high die shear strength at both 23 °C and 50 °C for adhesive formulations comprising up to 67 wt% of thermoplastic polymer, being comparable to the shear strength obtained for common lead-free solders and conventional ACAs. The die shear strength at 190 °C drops dramatically, and agrees very well with the results from the rework evaluation.

Key words: anisotropic conductive adhesive, reworkable, repairable, medical devices

Introduction

Anisotropic conductive adhesives (ACAs) have emerged as an alternative to soldering for flip-chip interconnection of electronic devices to a variety of substrates. An ACA comprises a non-conductive adhesive matrix filled with a low concentration (below the percolation threshold) of mono-disperse conductive particles [1]. The particles are either solid metal particles or metal-coated polymer spheres. The mechanical strength of ACA interconnects comes from the adhesive matrix, while the anisotropic electrical conduction is established when the conductive particles are trapped between the mating bumps-pads on a chip and a substrate (Figure 1). ACAs can be either in paste-form or in film-form, being called anisotropic conductive paste (ACP) and anisotropic conductive film (ACF), respectively. ACF is the most popular used form of ACAs, due to the ability to better control the volume of material, the density and the distribution of conductive particles within the sample, as well as the ease of handling. Compared to solder technology, the ACAs offer major benefits such as lower processing temperature, fewer processing steps, and finer interconnection pitch.

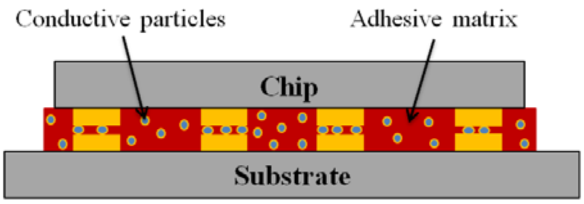


Figure 1: An illustration of an ACA assembly

The ACAs have widely been used as a standard interconnection technology in the assembly of flat-panel displays where soldering is not applicable for bonding chips to glass substrates, because of low pitch and metallization available [2]. The technology has also shown high potential for use in bonding chips to silicon/ceramic substrates, chips to organic substrates such as flexible and rigid printed circuit boards (flex and FR4), flex to flex, flex to FR4 [1, 2]. The main reason is the ACA technology can provide electrical connection, mechanical strength and sealing/underfilling in one quick bonding step. Furthermore, the technology is capable of providing high interconnect yield and reliable interconnects [3-6].

The present work is motivated through the assembly of medical ultrasound probes in which electronic modules are bonded using an ACF. Since the cost of these modules are high, it is a huge advantage if ACF bonds are reworkable. That means one is able to separate mating modules from each other, remove the residual adhesive on bonding surfaces, and reuse / re-bond functional module(s), either during production or when repairing returned products. Commercially available ACFs are generally not reworkable due to the common use of thermosetting adhesive matrix [2]. A limited number of commercially available ACFs are claimed to be reworkable/reparable. However, a successful rework is still challenging, particularly the process of removing the residual adhesive on the bonding surfaces [7]. ACA materials with competitive performance compared to conventional ACAs and with proper reworkability at modest conditions are investigated in this work. Enabling the reworkability of the ACAs demands modifications of its adhesive matrix. Adhesive matrices comprising a blend of a thermosetting epoxy and a thermoplastic polymer are of interest because it has shown potential to enable the reworkablity whilst still ensuring good electrical and mechanical performance as well as the reliability of adhesive-based interconnects [1, 8].

This paper presents the findings of favourable mixing ratios between an epoxy system compatible with ACA applications and a thermoplastic polymer that result in adhesive blends with good mechanical performance while enhancing the reworkability of the adhesive. Test samples include silicon-based dies and substrates being bonded using adhesive blends with varying ratios of the epoxy and the thermoplastic. Die shear strength at varying temperatures relevant for production/storage, operation of ultrasound probes and rework of ACA bonds is studied. The ability to fully remove the adhesive remaining on bonding surfaces, for successful rework, is also verified.

Experimental

Preparation of adhesive blends and adhesive films

The thermosetting epoxy system comprises a low-viscosity bisphenol F resin, a curing agent and a curing catalyst. The mixing ratio between the components of the epoxy system is selected to ensure the compatibility with the ACA applications in terms of viscosity, curing temperature and curing time. The thermoplastic polymer used is a polysulphone (PSU) with low molecular weight to promote resin flow during bonding. The PSU materials are amorphous polymers with excellent thermal stability (glass transition temperature T_g ranging from 180 to 280 °C), good mechanical properties and resistance to chemicals such as dilute acids, alkalis, electrolytes [9, 10]. Since PSU is supplied as pellets, 1-Methyl-2-pyrrolidinone (NMP) was used to dissolve the PSU. NMP is selected thanks to its good solvency properties to PSU. In addtition, this choice is acceptable because NMP is also used as a solvent for a wide range of polymers, and as a cleaning agent in a variety of industrial applications such as electronics, automotives, chemicals, pharmaceuticals [11]. Conductive particles were not included in the adhesive formulations in this work. This is supposed to have insigificant impact on the mechanical performance of the adhesive due to the low concentration of the particles in ACA formulations.

A blend of epoxy and PSU was prepared by adding epoxy resin, curing agent and curing catalyst to the NMP-solution of PSU. A uniform paste without air bubbles was obtained, using a SpeedMixer DAC 150.1 FVZ-K from FlackTek Inc. The paste was then coated onto a glass substrate in form of stripes by using Kapton tape (thickness of about 70 µm) as mask. The stripes were dried at room temperature for 2 days. After drying, adhesive films could be peeled off the glass substrate, and were used similarly as ACF.

In the present work, adhesive formulations comprising 25 – 67 wt% of PSU were prepared. The adhesive films after drying have a thickness in the range of 30 to 45 µm, depending on the formulations.

Preparation of samples for die shear tests

Test samples consisting of a bare silicon die bonded to a bare silicon substrate were prepared for destructive die shear tests. The dies and the substrates were washed using acetone and isopropanol, and then dried with nitrogen gas. The assembly process was similar to a typical ACA/ACF bonding process. An adhesive film was cut to an appropriate size, and was subsequently applied on the substrate. For samples being bonded using only epoxy, a drop of the epoxy was deposited on the substrate. The die was assembled on the adhesive using a flip-chip bonder FinePlacer Pico from Finetech GmbH. A bondline thickness of 20 µm between the die and the substrate was controlled by means of spacers. The bonding was performed by applying a sufficient force at 190 °C for 5 minutes. The bonding time was selected somewhat longer than needed in order to ensure a high curing degree of the adhesive.

Die shear tests at varying temperatures & visual inspection

An F&K DELVOTEC Bond Tester 5600 was used to conduct the destructive die shear tests. A custom-built sample holder, which could clamp the sample and keep it at a specified temperature, was machined in aluminum. The sample holder was mounted on the workstage of the bond tester, being compatible with the conventional shear test procedure. Figure 2 illustrates the setup for the die shear testing trials. The sample holder is heated by a Watlow Firerod cartridge heater with feedback from a thermocouple installed inside the holder. The actual temperature in the bondline during the die shear tests was checked by measuring the temperature on top of the substrate and on top of the die. The precision in temperature control of our test samples was found to be within +/- 1 – 5 °C, depending on the test temperature. The die shear tests were performed at room temperature (about 23 °C), 50 °C and 190 °C, representing temperatures relevant for production/storage, operation of medical ultrasound probes, and rework of bonded electronic modules, respectively.

Visual inspection was performed on all samples subjected to the destructive die shear tests to analyze fracture surface, and to measure bonding area. The die shear strength of each test sample was determined based on the measured maximum force and the bonding area.

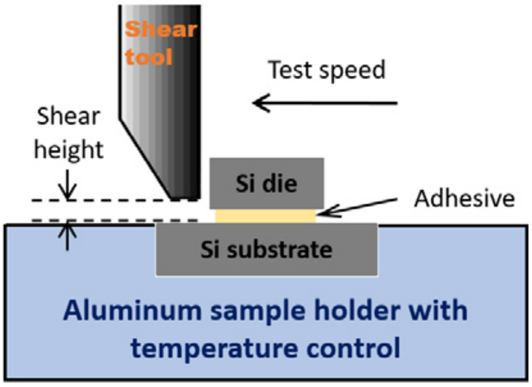


Figure 2: Illustration of a test specimen in the custom-built die shear test fixture

<u>*Evaluation of reworkability*</u>

The rework process includes two main steps; separating the die from the substrate of a bonded sample, and removing the adhesive remaining on bonding surface of the die and the substrate. In this work, test samples with a bonding area relevant for real applications (e.g. from 25 to 36 mm^2) were subjected to the rework evaluation. A sample was heated up to 190 °C on a hot plate and kept for about 1 minute. The die was then separated from the substrate by using tweezers to apply a twisting force between them. After separation, the residual adhesive on the bonding surfaces was removed by rubbing a cotton swab soaked in the solvent NMP at room temperature, followed by applying a light force on the residue at about 50 – 60 °C using a wooden spatula.

Results

The die shear strength at room temperature (23 °C) of varying adhesive formulations is shown in Figure 3. Adhesive formulations comprising 25 – 67 wt% of PSU exhibit comparable die shear strength. Compared to bare epoxy, the die shear strength of adhesive formulations with PSU is about 30 % lower. The visual inspection of samples tested at room temperature showed mainly cohesive fracture for samples based on adhesive formulations with PSU whereas cohesive-adhesive fracture was observed for bare epoxy samples.

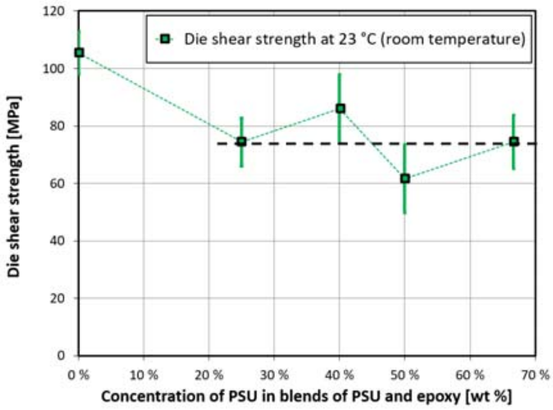


Figure 3: Die shear strength at room temperature of adhesive formulations of interest. Each datapoint is the average value of at least 6 repeated tests.

The die shear strength at 50 °C of the adhesive formulations comprising 25 wt%, 50 wt% and 67 wt% of PSU is shown in Figure 4. The die shear strength retains high values (over 50 MPa) at 50 °C. Adhesive formulations comprising 50 – 67 wt% of PSU exhibit comparable die shear strength which is somewhat lower than that of the formulation with 25 wt% of PSU. The visual inspection showed predominantly cohesive fracture for all samples tested at 50 °C.

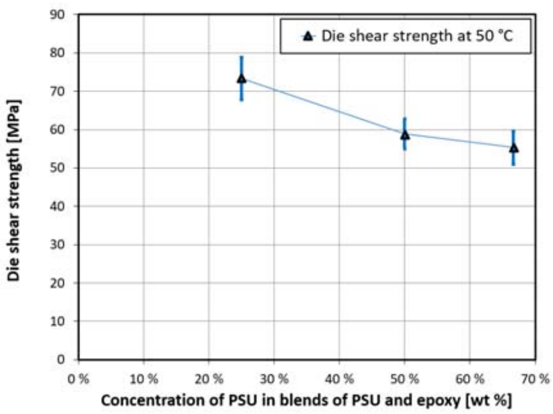


Figure 4: Die shear strength at 50 °C of adhesive formulations of interest. Each datapoint is the average value of at least 6 repeated tests.

Figure 5 shows the die shear strength at 190 °C of varying adhesive formulations. While epoxy still maintains a die shear strength over 4 MPa, all adhesive formulations with PSU exhibit a considerably lower die shear strength, being about 1 MPa and below. The higher the concentration of PSU in the adhesive formulations is, the lower the die shear strength is. The failure mechanisms of samples tested at 190 °C are similar to that of samples tested at room temperature; mainly cohesive fracture for samples based on adhesive formulations with PSU, and cohesive-adhesive fracture for bare epoxy samples. Figure 6 shows typical fracture surfaces of test samples bonded with bare epoxy and with an adhesive formulation comprising PSU. These samples were sheared at 190 °C.

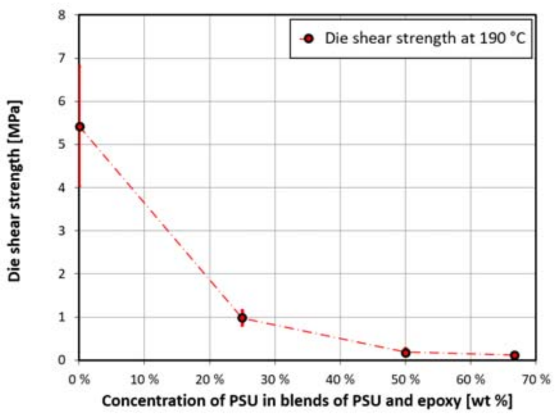
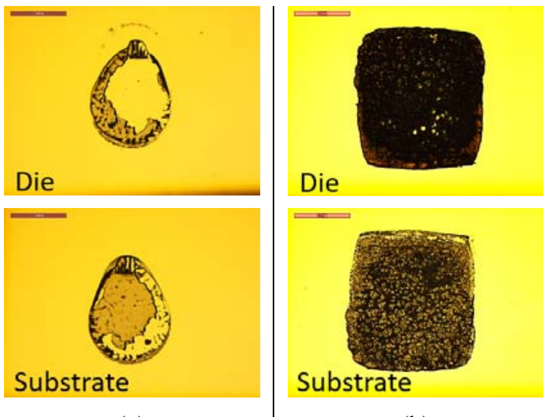


Figure 5: Die shear strength at 190 °C of adhesive formulations of interest. Each datapoint is the average value of at least 5 repeated tests.



(a) (b)

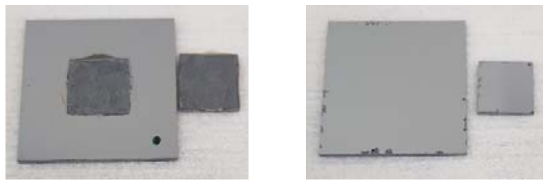
Figure 6: Typical fracture surfaces of (a) a sample bonded with bare epoxy, and (b) a sample bonded with an adhesive formulation comprising PSU. The samples were sheared at 190 °C.

Table 1 shows the results from the rework trials. The reworkability is enabled when PSU is included in formulations with a concentration from 25 wt%. However, adhesive formulations comprising more than 40 wt% of PSU allow a successful rework with less effort, compared to those with less concentration of PSU. Figure 7 shows typical result from a rework trial for test samples based on adhesive formulations comprising PSU. After separating the die from the substrate of a bonded sample, the bonding area (in the range of 25 – 36 mm^2) of both the die and the substrate is covered with adhesive (Figure 7-a). After the process of removing the residual adhesive, clean bonding surfaces are obtained (Figure 7-b), indicating a successful rework.

Table 1: Results from the evaluation of reworkability

Adhesive formulations	Possibility to separate a die from a substrate (*)	Removal of residual adhesive on bonding surface	
		Possibility	Difficulty
Bare epoxy	No	No (**)	Very hard
25 wt% PSU	No	Yes (**)	Hard
40 wt% PSU	Yes	Yes	Moderate
50 wt% PSU	Yes	Yes	Easy
67 wt% PSU	Yes	Yes	Easy

* Samples with a bonding area from 25 to 36 mm^2
** Trials using samples with a considerably smaller bonding area



(a) Before removal of residual adhesive
(b) After removal of residual adhesive

Figure 7: Typical result from a rework trial for samples based on adhesive formulations comprising PSU

Discussion

Figure 8 shows the comparison of die shear strength measured at room temperature (23 °C), 50 °C and 190 °C for adhesive formulations with varying concentrations of PSU. The temperature ranging from 23 to 50 °C has a minor effect on the mechanical die shear strength of the adhesives tested in this work. The die shear strength in such temperature range retains a high value (over 50 MPa), being comparable to the shear strength measured at room temperature for common lead-free solders (30 – 65 MPa [12-14]) and conventional ACF used in Chip-on-Glass applications (40 – 110 MPa [15, 16]). When the temperature is increased to 190 °C, the die shear strength of all adhesive formulations drops dramatically, compared to the corresponding values at room temperature and 50 °C. This implies the possibility to remove a die from a substrate of a bonded sample by applying a low force in certain conditions.

The remarkable drop in die shear strength at 190 °C of all adhesive formulations is attributed to the reduction of the modulus of polymer materials at temperatures within and above its T_g range. The test temperature of 190 °C is within the T_g range of PSU (180 – 280 °C [9, 10]) and is above the T_g of cured epoxy, which is typically lower than the curing temperature applied (190 °C in this work). At such temperature, the modulus of both PSU and epoxy is reduced considerably [9]. However, the modulus of PSU used in this work, which is an amorphous polymer and has low molecular weight, is probably reduced more than that of the cured epoxy, which is highly cross-linked. This explains the dramatic drop in die shear strength at 190 °C for all adhesive formulations, as well as the reason for considerably lower die shear strength of the formulations with PSU compared to that of the bare epoxy (Figure 5).

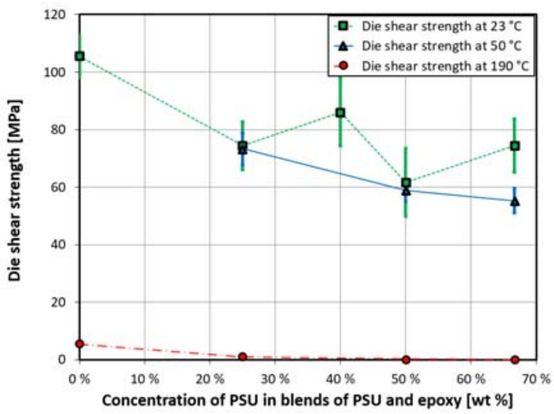


Figure 8: Die shear strength at varying temperatures of adhesive formulations with varying concentrations of PSU

The temperatures selected for die shear tests in this work are relevant for the production/storage (23 °C), the operation of medical ultrasound probes (50 °C) as well as the rework of bonded electronic modules inside the probes (190 °C). The high die shear strength in the temperature range of 23 – 50 °C as well as the remarkably low die shear strength at 190 °C indicate the high potential for using adhesive formulations comprising PSU as the matrix for the ACA under development. Such an ACA, even with a high concentration of PSU, is supposed to provide sufficient mechanical and electrical performance as well as integrity for use in medical applications whilst facilitating the reworkability.

The failure mode of all adhesive formulations comprising PSU is similar, being predominantly cohesive fracture. The failure mode is the same even with varying shear test temperatures. That means the adhesion of adhesive formulations with PSU to the surface of silicon dies and silicon substrates is comparable. This indicates a well dispersion of PSU and epoxy in the adhesive films manufactured in this work.

The results from the rework evaluation (Table 1) and from the die shear testing trials at 190 °C, the rework temperature (Figure 5), agree very well with each other. The lower the die shear strength at the rework temperature is, the easiser the rework process is. A die shear strength below 1 MPa at the rework temperature is an indication of satisfactory reworkability.

Further environmental tests relevant for medical applications such as temperature cycling, temperature aging, humidity testing are crucial to select the adhesive formulations that can provide good mechanical integrity as well as good electrical performance, whilst still allowing an easy rework. Electrical characterization for the ACA under development is also planned.

Conclusions

Adhesive formulations comprising a thermosetting bisphenol F epoxy system and a thermoplastic polysulphone (PSU) are developed for use as an adhesive matrix in a reworkable anisotropic conductive adhesive (ACA). Die shear strength of adhesive formulations comprising 0 – 67 wt% of PSU was characterized at varying temperatures. The test temperatures were 23 °C, 50 °C and 190 °C, being relevant for production/storage, operation of medical ultrasound probes (the target application for the ACA under development), and rework of ACA bonds, respectively. The reworkability of these adhesive formulations was also evaluated in practice.

High die shear strength at 23 °C and 50 °C, being over 50 MPa, was attained for adhesive formulations comprising up to 67 wt% of PSU. The die shear strength at rework temperature (190 °C) drops dramatically, and agrees very well with the results from the rework evaluation. The lower the die shear strength at the rework temperature is, the easiser the rework process is. A die shear strength below 1 MPa at the rework temperature is an indication of satisfactory reworkability. The results from the present paper confirm the feasibility of using blends of epoxy and PSU as the adhesive matrix for the reworkable ACA under development.

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References

[1] J. E. Morris and J. Liu, "Electrically Conductive Adhesives: A Research Status Review," in *Micro- and Opto-Electronic Materials and Structures: Physics, Mechanics, Design, Reliability, Packaging*, vol. 2, E. Suhir, Y. C. Lee, and C. P. Wong, Eds. U.S.A.: Springer US, 2007, pp. B527-B570.

[2] P. J. Opdahl, "Anisotropic Conductive Adhesives," in *Handbook of Visual Display Technology*, J. Chen, W. Cranton, and M. Fihn, Eds. 2 ed. Switzerland: Springer International Publishing, 2016, pp. 1533-1541.

[3] A. Larsson, F. Oldervoll, T. A. T. Seip, H.-V. Nguyen, H. Kristiansen, and Ø. Sløgedal, "Anisotropic Conductive Film for Flip-Chip Interconnection of A High I/O Silicon Based Finger Print Sensor," in *The 22nd Micromechanics and Micro systems Europe Workshop*, Tønsberg, Norway, 2011, pp. 186-189.

[4] H.-V. Nguyen, T. Eggen, B. Sten-Nilsen, K. Imenes, and K. E. Aasmundtveit, "Assembly of Multiple Chips on Flexible Substrate Using Anisotropic Conductive Film for Medical Imaging Applications," in *The 64th Electronic Components and Technology Conference*, Orlando, Florida, USA, 2014, pp. 498-503: IEEE.

[5] P. Palm, J. Maattanen, Y. De Maquille, A. Picault, J. Vanfleteren, and B. Vandecasteele, "Comparison of different flex materials in high density flip chip on flex applications," *Microelectronics Reliability,* vol. 43, no. 3, pp. 445-451, 2003.

[6] M. J. Yim *et al.*, "Highly Reliable Flip-Chip-on-Flex Package Using Multilayered Anisotropic Conductive Film," (in English), *Journal of Electronic Materials,* vol. 33, no. 1, pp. 76-82, 2004.

[7] Z. W. Zhong, "Various Adhesives for Flip Chips," *Journal of Electronic Packaging,* vol. 127, no. 1, pp. 29-32, 2005.

[8] K. Moon, C. Rockett, C. Kretz, W. F. Burgoyne, and C. P. Wong, "Improvement of adhesion and electrical properties of reworkable thermoplastic conductive adhesives," *Journal of Adhesion Science and Technology,* vol. 17, no. 13, pp. 1785-1799, 2003.

[9] J. M. G. Cowie and V. Arrighi, *Polymers: Chemistry and Physics of Modern Materials*, 3rd ed. CRC Press - Taylor and Francis Group, 2007.

[10] V. R. Sastri, *Plastics in Medical Devices - Properties, Requirements, and Applications*. William Andrew, 2010, p. 352.

[11] Mitsubishi-Chemical. N-Methyl-2-Pyrrolidone (NMP) [Online]. Available: https://www.m-chemical.co.jp/en/products/departments/mcc/c4/product/1201005_7922.html

[12] T. Siewert, S. Liu, D. R. Smith, and J. C. Madeni. Properties of Lead-Free Solders [Online]. Available: https://www.msed.nist.gov/solder/NIST_LeadfreeSolder_v4.pdf

[13] Y. A. Su, L. B. Tan, V. B. C. Tan, and T. Y. Tee, "Rate-Dependent Properties of Sn-Ag-Cu Based Lead Free Solder Joints," presented at the 11th Electronics Packaging Technology Conference, Singapore, 9-11 Dec. 2009, 2009.

[14] Y. Tian, C. Wang, S. Yang, P. Lin, and L. Liang, "Shear Fracture Behavior of Sn3.0Ag0.5Cu Solder joints on Cu Pads with Different Solder Volumes," presented at the International Conference on Electronic Packaging Technology & High Density Packaging (ICEPT-HDP), Shanghai, China, 28-31 July 2008, 2008.

[15] J. H. Zhang, Y. C. Chan, M. O. Alam, and S. Fu, "Contact Resistance and Adhesion Performance of ACF Interconnections to Aluminum Metallization," *Microelectronics Reliability,* vol. 43, no. 8, pp. 1303-1310, 2003.

[16] K.-C. Chen, H.-T. Li, C.-W. Hsu, and C.-P. Yang, "Properties and Reliability Test of Anisotropic Conductive Film in Chip on Glass Package," in *The 1st Electronics Systemintegration Technology Conference*, Dresden, Germany, 2006, vol. 1, pp. 51-55.