

## Chapter 5

# Determining the Impact Resistance of Electrically Conductive Adhesives Using a Falling Wedge Test

### Abstract

This study was conducted to determine the impact resistance of electrically conductive adhesives (ECAs). A novel falling wedge test that was used to quantitatively characterize the impact resistance of conductive adhesives at a material level has been described and some important findings obtained from the falling wedge test are presented. This unique impact resistance testing method is not only a substitution of the conventional drop test which has several severe drawbacks, but also provides some useful information for screening adhesives at the materials level and helping formulate new conductive adhesives with improved impact performance. Three model conductive adhesives were studied in this work and the impact fracture energies of the adhesive materials were measured utilizing the falling wedge test. The effect of test temperature on the fracture behavior of ECAs was examined and the correlation between the impact resistance and damping property of the conductive adhesive was also investigated. This study suggests that (1) the falling wedge test is able to discriminate between adhesives and this technique is capable of screening adhesives for bonding purposes; (2) the viscoelastic energy has played an important role in the fracture behavior of the conductive adhesives. As a measure of the internal friction, the loss factor  $\tan d$  is found to be a good indicator of a conductive adhesive's ability to withstand impact loading.

**Keywords:** Electrically conductive adhesives, falling wedge test, double cantilever beam, impact resistance, fracture energy, loss factor, viscoelastic energy

### 5.1 Introduction

Electrically conductive adhesives (ECAs) are gaining increased interest as possible replacements to environmentally harmful solders in electronic applications. ECAs can provide an environmentally friendly solution for interconnections in electronic applications. Moreover, ECAs also offer many potential advantages over conventional solder interconnection technology including finer pitch printing, lower temperature processing, and

more flexible and simpler processing [1, 2]. However, efforts to replace lead-based solders for microelectronic chip attachment by electrically conductive adhesives (ECA) have found limited applicability due to two major drawbacks: unstable contact resistance under environmental aging conditions and limited impact resistance of the adhesive interconnections. Numerous studies [1-5] have been conducted to investigate the unstable contact resistance phenomenon. Jagt [6] has suggested several general failure causes to explain the resistance increase of some ECA joints upon exposure to environmental aging, including oxidation of non-noble metallizations, crack formation/delamination, formation of a silver depleted surface layer in the adhesive, creep of the adhesive and the formation of an intermetallic layer and processing defects such as undercure, etc. Xu et al [7,8], have studied environmental aging effects on bulk conductive adhesives and their joints and investigated the possible degradation mechanisms of conductive adhesives upon exposure to environmental aging conditions.

The drop test, which was established by the National Center for Manufacturing Sciences (NCMS) [9], has been widely used to evaluate the impact performance of conductive adhesives in the conductive adhesive industry. The drop test involves dropping mounted chip carrier and circuit board assemblies onto hard surfaces from a height of 1.5 m (60 inches) and passing six drops is a pre-requisite for the application of conductive adhesives. The drop test is easy to conduct. However, as the drop test can only qualitatively distinguish the impact performance of conductive adhesives, not much optimal information could be obtained from the drop results. To improve the impact performance of conductive adhesives, adhesive manufacturers must first accurately characterize the impact behavior of a conductive adhesive and investigate factors that may affect the impact performance of the adhesive. Therefore, it is desirable to develop a test technique that can quantitatively characterize the impact resistance of conductive adhesives at a material level and yield useful information in screening adhesives and helping formulate new conductive adhesives with more favorable impact performance. As a cooperative project with Emerson & Cumming and Motorola, one of the main objectives of this study has been to develop a test method for quantitative characterization of impact performance of conductive adhesives.

As adhesively bonded joints have been increasingly used in demanding engineering applications, interest has arisen in the failure behavior of such materials and bonded joints

under high rates of test including impact loading conditions. A variety of impact tests have been devised to characterize the impact performance of various adhesive joints, including the ASTM block impact test [10], the impact wedge-peel (IWP) test [11], and the impact wedge test [12]. These impact tests have been adopted for particular applications where the assessment of impact performance is needed. However, certain limitations have prevented these tests from being adopted in the evaluation of conductive adhesive joints. The ASTM block impact test has been shown to only give a qualitative comparison of the ability of different adhesives to withstand impact loading. Moreover, this test technique is found to be “machine” sensitive and the actual stress condition experienced by the adhesive layer may be significantly affected by the way the loading is applied to the specimen [13]. Both the impact wedge-peel (IWP) test and the impact wedge test have been designed for the automotive industry to characterize fracture behavior of the adhesive joint under impact loading conditions and are intended for metallic adherends that plastically deforms. Although the impact wedge test [12] was found to be able to determine the toughness of an adhesive, the extensive deformation of the adherends during testing would have complicated exact calculations of the fracture energy of the adhesive studied. On the other hand, the IWP test was only designed to characterize the fracture behavior of an adhesive joint “system”, instead of the behavior of the adhesive itself, and the mechanical properties of the adherends can greatly influence the results recorded from such a test.

To characterize the impact performance of conductive adhesives at the material level, a novel falling wedge fracture test [14] was adopted in this research. This test technique is capable of quantitatively determining the impact resistance of conductive adhesives and screening adhesives for bonding purposes, as well as providing useful information for conductive adhesive development. A modified double cantilever beam (DCB) geometry was successfully developed for the purposes of this study. Based on the concepts of linear-elastic fracture mechanics (LEFM), the fracture energies of three different conductive adhesives were measured using this new test method. The effect of test temperature on the fracture behavior of ECAs was studied in this research. The correlation between the impact resistance and damping property of the conductive adhesive was also investigated using this test technique.

## 5.2 Experimental

### 5.2.1 Materials

Three silver-filled epoxy-based adhesive systems, which were designated as ECA1, ECA2 and ECA3, respectively, were supplied by Emerson & Cuming. They are not commercial products, but rather are model systems formulated for this research. Thermogravimetric analysis results showed that ECA1, ECA2 and ECA3 had silver loadings of 83%, 82% and 86% by weight, respectively, while the epoxy resin, thermoplastic epoxy copolymer, curing agents and other additives accounted for the rest of the adhesive formations [7].

FR-4 printed circuit boards (PCB) with gold metallization strips were used as substrates for the DCB specimens. The PCB adherends had dimensions of 200 x 25 x 1.2 mm, and the gold metallization strip was plated only along the center of the PCB boards with a width of 6.5 mm, where the adhesive was applied to bond two substrates together. The gold plated PCB substrates were made by electroless plating and manufactured by Triad Circuit Inc. located in Round Lake, Illinois. As a conventional plating process in the industry, a copper layer was first plated on the PCB board as the base metal and then a nickel layer with a thickness of 4~6  $\mu\text{m}$  was made on the copper layer as a diffusion barrier to hinder the copper from diffusing into the gold and vice versa. Finally, a thin layer of gold with an approximate thickness of 0.08~0.2  $\mu\text{m}$  was plated on the top of the nickel layer to prevent oxidation.

There were several considerations for the selection of the substrate material and geometry in this study. First, FR-4 boards selected as substrates are very representative of actual applications. Second, in contrast with traditional DCB samples in which the adhesive is spread across the entire substrate width, the DCB samples used in this study were bonded by placing the conductive adhesive only in a central strip along the substrate length. With reduced bonding area of the adhesive joint, the amount of adhesive as well as the area of gold plating was greatly reduced for each DCB sample. Hence, the material cost (including adhesive and gold plating) for each specimen was also reduced. Moreover, the reduced adhesive width allowed voids trapped in the adhesive escape more easily in the width direction during cure. As a result, porosity problems, which were encountered in the joints

with wider adhesive bondlines, were greatly reduced. Another advantage of using the bonded width as some fraction of the specimen width is that the stresses in the substrates are significantly lowered during testing and as a result, the possibility of damage or nonlinear behavior in the FR-4 boards can be greatly reduced.

### **5.2.2 Sample Preparation**

Conventional DCB samples are generally made with the adhesive spread across the entire substrate width. However, based on several considerations stated above, DCB samples in this study were only bonded along a central strip and the ensuing bondline width of the samples accounted for only a quarter of the total substrate width. To have good control of bondline width and thickness of the DCB samples, a sample fabrication method was developed in this study and is reported herein.

The first step of the sample fabrication was to fabricate a spacer used to control adhesive thickness and width, as shown schematically in Figure 5.1. A piece of Teflon film which was 0.1 mm thick and 25mm wide was first fit into an aluminum mold and then an aluminum strip with two narrow slits was placed on the top of the Teflon film. The width of aluminum strip was the same as the width of Teflon film and FR-4 substrates, and the distance between the two slits was 6.5 mm. By sliding a razor blade along the two slits in the aluminum strip, two new slits were made in the Teflon film with a distance of 6.5 mm apart. A Teflon spacer, similar to a gasket, was then created by cutting off the Teflon between the two slits, as illustrated in Figure 5.1.

The second step of the sample fabrication was to bond a DCB sample, as illustrated in Figure 5.2. First, a gold plated FR-4 substrate was placed in an aluminum mold. Then a Teflon spacer was used to cover the substrate, and as a result, only the center area with gold plating was exposed. The adhesive was then placed on the exposed area along substrate length. Following this step, the other substrate was placed on the top of the adhesive and the whole sample with the mold was clamped together with binder clips on both sides of the sample along the length direction. The clamped sample was finally placed in an oven and cured at 150°C for 1h before the power of the oven was turned off. After the sample cooled down in the oven, the sample was then taken out from the oven, and the Teflon spacer in the

specimen was carefully removed from the sides of the specimen. All cured samples were stored in a desiccator at room temperature prior to testing.

### 5.2.3 Test Procedure

In this study, the falling wedge test was adopted to study the properties of adhesive joints under high speed or impact loading. The experimental setup for this test technique is shown schematically in Figure 5.3. A Dynatup, model 730-I drop tower with a maximum drop height of 1.25 meters and a basic drop mass of 4.5 kg, which could be increased with attachments, was used to impact DCB specimens. Two 30° polycarbonate wedges were mounted on the drop weight to split the DCB specimens apart during testing. Polycarbonate wedges were selected because their transparency would not block the view of the specimen and because of their resistance to damage under impact situations. As will be discussed later, load signals were not used to calculate the fracture energy of the conductive adhesive joints. Therefore, no load cells or load transducers were installed in the experimental setup for acquisition of load values during impact testing. During testing, the polycarbonate wedges dropped down to impact the hinge pins, which were bonded to one end of the DCB substrates, forcing the specimen secured vertically at the base of the drop tower apart. The impact speed can be adjusted through altering the height of the drop tower before testing. In this study, the drop tower weight was released at a height of 450 mm and the associated opening displacement rate was about 1.6 m/s. Specimens were tested at several temperatures including,  $-70^{\circ}\text{C}$ , room temperature,  $60^{\circ}\text{C}$ , and  $90^{\circ}\text{C}$ . When the specimens were tested at temperatures other than room temperature, they were conditioned at the corresponding temperatures in an oven for 20 minutes and then quickly mounted in the experimental setup with a two-piece of insulated jacket surrounding the specimens. The insulated jacket was removed from the specimen just before the test was conducted to maintain the specimen at the desired temperature. The exposure time of the specimen in the atmosphere was less than 15 second after the samples were out of the oven and before testing. The bondline temperature was examined with a small thermocouple inserted on the fractured samples immediately after testing, and it was found that samples were maintained at the desired test temperatures.

A Kodak EktaPro Motion Analyzer was used to monitor the testing and film the fracture process of test specimens. The high-speed camera allowed for recording images at

rates as high as 12,000 frames per second (fps). In this study, a framing rate of 2000 fps was used. A focused tungsten spotlight was utilized to illuminate the specimen and was turned on just before testing to minimize heating the specimen. An electric trigger associated with the Kodak EktaPro high-speed camera [15] was connected to the drop tower. When the drop tower was released, the electric trigger was switched on automatically and the fracture sequence was recorded. The recorded images were then transferred to a computer through an image acquisition board and projected frame by frame onto a screen, allowing measurements to be made from the magnified image. To accelerate data analysis, a National Instruments IMAQ Vision Builder software was used to measure opening displacements along the specimen length for a single image frame and to further obtain the crack length and the opening displacement at the loading points, as will be discussed in the following section. A typical photographic frame recorded using the high-speed camera is shown in Figure 5.4.

#### 5.2.4 Calculation of the Fracture Energy

The double cantilever beam (DCB) test is a very popular fracture-mechanics test and has been used extensively study the effects of static, fatigue, and environmental loads on adhesive bonds, and thereby the durability of the bonded joints [16,17,18,19]. This test has been incorporated into a testing standard as ASTM D 3433-93 [20]. Based on simple beam theory, the fracture energy under static, or quasi-static loading conditions can be determined by using either a load based or a displacement based formulation, as given in the following equations:

$$G_I = \frac{P^2 a^2}{BEI} \quad (1)$$

or

$$G_I = \frac{9d_0^2(EI)}{4Ba^4} \quad (2)$$

where  $P$  is the applied load,  $d_0$  is the opening displacement at the load points,  $a$  is the crack length,  $B$  is the bond width,  $E$  is the elastic modulus of the adherends, and  $I$  is the second moment of area of the adherends.

Under impact loading, however, the loading is quite different from quasi-static loading. Research by Blackman *et al* [21] showed that at high loading rates, the load signal was greatly influenced by dynamic effects, precluding sensible and reliable values. Therefore, significant errors are produced if the load-based equation (1) is used to calculate the fracture energy at high loading rates. Meanwhile, Blackman *et al* also pointed out that if the displacement based equation (2), in which the load-point displacement and the crack length could be correctly obtained with the aid of high-speed photography, is utilized for the calculation of fracture energy under high-speed loading conditions, errors induced in the fracture energy results are small.

Based on the observations made by Blackman *et al*, the displacement based equation (2) was used in this research. As noted in equation (2), both the crack length and the corresponding load-point displacement while the crack propagates along the crack length need to be known to calculate the fracture energy of the adhesive joints. The crack length and the opening displacement at load point could be obtained by observing them from the recorded frames. However, due to the limited resolution of the camera and the recessed adhesive layer, it was difficult to accurately locate the crack tip. Consequently, misleading estimates of the value of the fracture energy could result, especially since the crack length is raised to the fourth power in Equation (2). To solve this problem, we used curve fitting to accurately determine the crack length, as well as the opening displacement at the point of load application, based on simple beam theory. First, a series of displacement values along the specimen length were taken for a single image frame. By assuming the two substrates act as two single cantilever beams subjected to concentrated forces applied at the ends, the load-point displacement and the crack length were calculated by mathematically fitting the displacement values using the following deflection equation of a cantilever beam loaded at its free end by a concentrated force:

$$\mathbf{d}(x) = \frac{P}{6EI} [3a(a-x)^2 - (a-x)^3] \quad (3)$$

Where  $P$  is the load applied at the end of the substrate,  $\delta$  is the opening displacement along the specimen length,  $a$  is the crack length,  $x$  is the distance from a point on a specimen

to the point of load application,  $E$  is the elastic modulus of the adherends, and  $I$  is the second moment of area of the adherends.

All the curve fittings in this study were made using a MATHEMATICA® [22] routine. As can be seen from Figure 5.5, which is a typical fitting curve, the experimental results show very good agreement with simple beam theory. This suggests that the results obtained based on beam theory are reliable and the calculation of fracture energy using the beam-based equation 2 should be valid. This procedure insures accurate location of the crack tip even when not physically observable. After the load-point displacement and the crack length were obtained from the fitting curve, in which the y-intercept and x-intercept are the load-point displacement and the crack length, respectively, they were then substituted into Equation (2) to calculate the fracture energy of the adhesive.

## 5.3 Results and Discussions

### 5.3.1 Falling Wedge Test Results

Figure 5.6 is a typical plot showing the variation of crack velocity with time. Note that the crack growth accelerates and decelerates in an irregular way. This behavior probably can be attributed to the dynamic effects, and is typical of high rate tests. Furthermore, Figure 5.6 clearly illustrates that the crack growth in the impact testing was of stick-slip type, as also observed by Blackman et al [21,23]. The crack initiates and then propagates at a crack speed of as high as 8 m/s, followed by crack arrest. As the crack continues to move forward along the length of the specimen, the process of crack initiation, propagation and arrest is repeated. Figure 5.7 is a typical plot showing the  $G$  values calculated for a test. Again, evident stick-slip behavior can be observed from the figure, and correspondingly, the fracture energy  $G$  in the figure can be divided into initiation, arrest and propagation values of  $G$ , as shown in the figure. Typical behavior of the fracture energy of conductive adhesives as a function of crack velocity is shown in Figure 5.8. The general trend exhibited in the figure is that the fracture energy tends to decrease with increasing crack velocity, although data points shown in the figure are somewhat scattered.

Figure 5.9 summarizes the falling wedge test results for ECA1, ECA2 and ECA3 joints as a function of test temperature. It should be mentioned that all the adhesive joints failed cohesively through the adhesive layer, based on visual observation. The error bars in the figure are one standard deviations based on 24 fracture energy values from four specimens. To make the figure clear, only the upper half part of each error bar is added. The relatively large errors appearing in the figure are not unexpected, as the fracture energy value shown in the figure is the average value of the initiation, arrest and propagation values of  $G$ , which were calculated by randomly selecting a series of frames. As can be observed from the figure, the three conductive adhesives exhibited different temperature-dependent behavior. ECA1 joints show no obvious difference in the fracture energy for samples tested at different temperatures from as low as  $-70^{\circ}\text{C}$  up to  $90^{\circ}\text{C}$ . For ECA2 joints, the fracture energy is low and similar when tested at room temperature and  $-70^{\circ}\text{C}$ . As the test temperature continues to go up, however, the fracture energy begins to increase and continues to go up at  $90^{\circ}\text{C}$ . For ECA3 joints, we can observe that the fracture energy obtained at  $-70^{\circ}\text{C}$  and  $-20^{\circ}\text{C}$  is low and is basically at the same level as the values obtained on ECA1 and ECA2 joints tested at the low temperature. When the test temperature continues to go up to  $60^{\circ}\text{C}$ , the fracture energy of ECA3 shows an increasing tendency. As the test temperature was further increased from  $60^{\circ}\text{C}$  to  $90^{\circ}\text{C}$ , however, the fracture energy of ECA3 shows a decreasing tendency, as can be observed from Figure 5.9. The different temperature-dependent behavior of the three conductive adhesives may be explained by the viscoelastic nature of the adhesives. As will be found in the following section, the three conductive adhesives ECA1, ECA2 and ECA3 show glass transition temperatures at around  $125^{\circ}\text{C}$ ,  $85^{\circ}\text{C}$  and  $50^{\circ}\text{C}$  (peak of  $\tan \delta$ ), respectively. This means that the glass transition temperature of ECA1 is well above all the test temperature and ECA1 remains glassy during testing. As a result, viscoelastic energy dissipation, which is a result of internal friction due to the motion of polymer chains in the material, may have played little role in joints made with this adhesive. On the other hand, ECA2 and ECA3 exhibit glass transition temperatures at  $90^{\circ}\text{C}$  and  $50^{\circ}\text{C}$ , respectively. When the materials were loaded in their viscoelastic regions, part of the applied mechanical energy was dissipated as heat through molecular friction. Therefore, the total energy required to fracture the adhesives are higher in contrast with the adhesives tested in their glassy state.

As observed above, the conductive adhesives showed different fracture behavior at the temperatures that are far below the glass transition temperatures of the ECAs and the ones that are around the glass transition temperatures. To better understand this phenomenon, scanning electron microscopy (SEM) was performed on the failure surfaces of the conductive adhesive joints. Figure 5.10 shows fracture surfaces of the ECA3 joints tested at  $-70^{\circ}\text{C}$ , and  $60^{\circ}\text{C}$ . The SEM pictures reveal evident differences in the fracture behavior of the adhesive at different test temperatures. When the ECA3 joint was tested at  $60^{\circ}\text{C}$ , where the adhesive shows the greater value of fracture energy, the failure occurred close to the interface and extensive polymer drawing can be observed from the figure. On the other hand, at the test temperature of  $-70^{\circ}\text{C}$ , where the adhesive showed relatively low fracture energy, no obvious polymer drawing can be observed on the fracture surface of the adhesive. Instead, the adhesive actually exhibits brittle failure features in Figure 5.10 (b). Similar observations were also seen on ECA1 and ECA2 samples, where high fracture energy is always related to ductile deformation of the adhesive on the fractured surfaces, while low fracture energy corresponds to brittle fracture features. These observations further suggest that viscoelastic energy and probably plastic deformation of the adhesive have contributed to the increase in the value of fracture energy.

### **5.3.2 Relationship between Damping Properties and Impact Fracture Energy of the ECAs**

As shown earlier, the fracture behavior of the conductive adhesives exhibited temperature-dependent behavior. Further observations of Figure 5.9 suggest that the temperature dependency of the adhesive fracture behavior was small when the test temperatures were far away from the glass transition temperatures of the adhesives and was apparent when the test temperature was around the glass temperatures. As also observed by Bitner, et al [24] in their study on an unmodified epoxy, the fracture behavior of the conductive adhesives is qualitatively similar to the linear viscoelastic behavior as shown in Figure 5.11 in that both exhibit similar temperature dependence over the temperature range of interest. This similarity may suggest that the change in the fracture energy with test temperature is mainly due to the ability of the adhesives to dissipate viscoelastic energy at different temperatures. It has been demonstrated that the viscoelastic energy dissipated during

loading essentially is a result of internal friction, or viscosity mechanism [25], which is created by relative motions of segmental chains in the adhesive material. Through molecular friction, some of the applied mechanical energy can be converted to heat, which would affect the adhesive fracture energy consisting of the adhesive intrinsic energy and other energy dissipations including the viscoelastic energy. As the loss factor ( $\tan \delta$ ), which is defined as the ratio of the loss modulus  $E''$  and the storage modulus  $E'$  and represents the damping ability of a material, is a measure of the internal friction in the adhesive material [26], the loss factor would be a good indicator of the ability of the material to dissipate the mechanical energy through heat. Some studies [26,27] have qualitatively demonstrated that the impact performance of an adhesive is closely related to the loss factor value of the material at the impact test conditions. In this section, the relationship of the loss factor and the impact fracture energy of the conductive adhesives will be further investigated.

To obtain the damping characteristics of the three conductive adhesives, experiments were performed on the three conductive adhesives using a TA Instruments 2980 dynamic mechanical analyzer (DMA). Neat cured samples with dimensions of 37 x 6.5 x 1.2 mm were made from the uncured conductive adhesives [7]. Figure 5.11 shows changes of the loss factor  $\tan \delta$  with the test temperature. The  $\tan \delta$  traces were obtained by testing the DMA samples at 1°C/min and a fixed frequency of 1Hz between the temperatures of -80°C and 160°C in single cantilever beam mode. As can be seen from the figure, the three adhesives exhibit very different damping behavior. ECA3 shows high  $\tan \delta$  values at a lower and broader temperature range and reaches its peak value at around 50°C. On the other hand, ECA1 and ECA2 have higher  $\tan \delta$  peaks that are centered near 85°C and 125°C, respectively.

The  $\tan \delta$  traces shown in Figure 5.11 were obtained with oscillation frequency of 1 Hz. The corresponding strain rate under this oscillation frequency was about  $1 \times 10^{-7} \text{ s}^{-1}$ . Under impact loading, however, the rate of deformation of the adhesives is much higher than that obtained at 1Hz, which would shift the viscoelastic region of the ECA to higher temperatures. Following the analytical solutions of the beam deflection  $w(x)$  for DCB specimens, which were obtained by Kanninen [28], as well as Arakawa and Takahashi [29],

based on the theory of beam on elastic foundation, the beam deflection  $w(0)$  at the crack tip may be expressed in the following way:

$$w(0) = \frac{3d_0}{2\sqrt{6}} \left[ \left(\frac{h}{a}\right)^2 + \frac{1}{\sqrt[4]{6}} \left(\frac{h}{a}\right)^3 \right] \quad (4)$$

where  $d_0$  is the opening displacement at load point,  $h$  is the thickness of the substrate and  $a$  is the crack length. In the general case,  $a \gg h$ . Thus, the above equation may be simplified into the following expression

$$w(0) = \frac{3}{2\sqrt{6}} \left(\frac{h}{a}\right)^2 d_0 \quad (5)$$

and the deformation rate of the adhesive  $\dot{\epsilon}$  at the crack tip then may be expressed as follows

$$\dot{\epsilon} = \frac{3}{2\sqrt{6}} \left(\frac{h}{a}\right)^2 \frac{1}{h_a} \dot{d}_0 \quad (6)$$

where  $\dot{d}_0$  is the opening displacement rate at the load point, which is approximately 1.6 m/s for the impact tests conducted in this work, and  $h_a$  is the adhesive bondline thickness, which is 0.1mm for the DCB specimens tested. Using Equation (6), it was estimated that the strain rate in the impact DCB tests was around  $2.5 \text{ s}^{-1}$ , which is around 7 orders higher than the deformation rate of the adhesives in the DMA tests obtained with oscillation frequency of 1 Hz. To investigate the relationship of the damping properties and the fracture toughness of the conductive adhesives, the loss factor values of the adhesives at the deformation rate under impact DCB testing were calculated by applying time-temperature superposition principle (TTSP). DMA tests were first performed on ECA1, ECA2 and ECA3 adhesives by isothermally sweeping frequencies of 1, 2, 5, 10, and 20 Hz at a series of temperature points with an interval of  $3^\circ\text{C}$  from  $-100^\circ\text{C}$  to  $150^\circ\text{C}$  and results obtained on each adhesive were then utilized to generate master curves of the loss factor,  $\tan \delta$ . After a master curve was obtained at a reference temperature, master curves for other temperatures of interest could then be obtained by referring to the shift factor-temperature plot. For detailed procedure of application of the time-temperature superposition, one can be referred to [30]. The equivalent loss factor data under the impact condition are listed in Table 5.1.

Figure 5.12 shows a correlation of the fracture energy and the  $\tan \delta$  obtained at 1 Hz at the temperatures corresponding to the impact test temperature, and Figure 5.13 shows the relationship of the fracture energy and the equivalent  $\tan \delta$  under the impact test conditions. As can be seen from the figures, the fracture energy of the conductive adhesives shows reasonably good agreement with the equivalent loss factor,  $\tan \delta$ , in contrast with the loss factor value obtained at 1 Hz, as the fracture energy increases with the higher loss factor value. By drawing a least squares fit along the data points plotted in the figure, we can observe that there is an approximately logarithmic relationship between the impact fracture energy and the corresponding loss factor. This analysis may suggest the following two points: First, the viscoelastic energy may be the main energy dissipation mechanism in the fracture tests conducted on the conductive adhesives and play an important effect on the impact performance of the adhesives. Second, the loss factor is still a good indicator of the ability of the conductive adhesives to absorb the impact energy, even though these conductive adhesives were filled with a significant amount of silver flake, and the ECA materials should be designed such that the damping property is maximized at the conditions under which the impact tests are conducted, in order to make the ECA materials have enhanced impact resistance.

### **5.3.3 Correlation between Falling Wedge Test Results and Drop Test Results**

The drop test has been widely accepted in the electronic industry to distinguish the impact performance of conductive adhesives. As a possible alternative to the drop test to screen conductive adhesives, the falling wedge test should be able to provide comparable results to the drop test, as well as be able to offer more meaningful guidance to adhesive manufacturers to formulate new conductive adhesives with favorable impact performance. In this section, the falling wedge test results were compared with results obtained from the drop test, which was conducted in our lab.

The drop samples were provided by Emerson and Cuming with one chip package bonded to the FR-4 board using a kind of conductive adhesive for each specimen. The drop tests were conducted using a newly designed drop test method at Virginia Tech [31]. This new drop test method was developed as an improvement on the free drop test method. Based on an inverted design, a new drop tower was designed, built, and validated. Instead of

dropping the test specimen on a hard floor, this new drop test involves dropping an appropriate mass guided by linear bearings on the test specimen that was held in place by a small drop of wax. This inverted drop test has been verified to be equivalent with the NCMS free drop test [9], but could provide more precise control of drop heights, release, and impact angles, resulting in more repeatable results by reducing human factors, in contrast to the free drop test. The specimens in this study were impacted by the drop tower from a height of 1.5 m (60 inches) at room temperature. Five samples were tested for each of the three conductive adhesives tested in the falling wedge test. Specimen failure was defined as any observable gap between the lead and the conductive adhesive. The drop test results, along with the falling wedge results obtained at room temperature are summarized in Table 5.2. As noted, ECA3 apparently exhibits the best impact performance in both the drop test and the falling wedge test among the three conductive adhesive. On the other hand, ECA1 and ECA2 show similar performance in both the drop test and the falling wedge test and both adhesives display relatively inferior impact performance in contrast to ECA3 under the current test conditions. These results demonstrate that the falling wedge test and the drop test can give consistent results for adhesive screening purposes, notwithstanding the falling wedge test may be able to yield more information than the drop test.

## 5.4 Summary and Conclusions

In this study, we utilized a novel falling wedge test to quantitatively characterize the impact resistance of electrically conductive adhesives. A special DCB geometry was developed by utilizing gold plated FR-4 as substrates and placing the conductive adhesive in a narrow strip down the center of the substrate length. The results obtained in this study show that the falling wedge test is able to discriminate between the impact performance of adhesives and this technique is capable of screening conductive adhesives for bonding purposes. This test technique could enable the microelectronics industry to use an *adhesive specification* rather than the current *drop test specification* to evaluate and determine acceptance of candidate adhesive systems.

The viscoelastic energy, which is a result of the internal friction created by chain motions within the adhesive material, has played an important role in the fracture behavior of

the conductive adhesives. As a measure of the internal friction, the loss factor  $\tan \delta$  is found to be an indicator of an adhesive's ability to dissipate the mechanical energy through heat. By quantitatively relating the impact fracture energy of the adhesive joints to the loss factors of the ECAs, we have demonstrated that the impact fracture energy tends to exhibit a logarithmic relationship with the corresponding loss factor, as the increased loss factor at the test conditions consistently results in the improved impact performance. This finding suggests conductive adhesive manufacturers that the impact performance of a conductive adhesive may be improved by formulating the adhesive with good damping ability under impact conditions that may be encountered in the service.

## 5.5 References

- 1 J. C. Jagt, P. J. M. Beris, and G. F. C. M. Lijten, "Electrically Conductive Adhesive: A prospective Alternative for SMD Soldering? ", *IEEE Transaction on Components, Packaging, and Manufacturing Technology*, Part B, **18** (2), 292-298, 1995.
- 2 Q. K. Tong, D. L. Markley, G. Fedrickson, R. Kuder, and D. Lu, "Conductive adhesives with superior impact resistance and stable contact resistance", *Proc. 49<sup>th</sup> Electron. Comp. Technol. Conf*, 347-352, 1999.
- 3 J. Liu, K. Gustafsson, Z. Lai and C. Li, "Surface Characteristics, Reliability, and Failure Mechanisms of Tin/Lead, Copper, and Gold Metallizations", *IEEE Transactions on Components, Packaging, and Manufacturing Technology*, Part A, **20**, 21-30, 1997.
- 4 D. Lu and C. P. Wong, "Development of Conductive Adhesives Filler with Low-melting-point Alloy Fillers", *International Symposium on Advanced Packaging Materials*, Port Erin, Isle of Man, British Isles, 7-13, 2000.
- 5 D. Lu, Q. K. Tong, and C. P. Wong, "Mechanisms Underlying the Unstable Contact Resistance of Conductive Adhesives", *IEEE Transaction on Components, Packaging, and Manufacturing Technology*, Part C, **22**, 228-232, 1999

- 6 J. C. Jagt, "Reliability of Electrically Conductive Adhesive Joints for Surface Mount Applications: A Summary of the State of the Art", *IEEE Transactions on Components, Packaging, and Manufacturing Technology, Part A*, **21** (2), 215-225, 1998.
- 7 S. Xu and D. A. Dillard, "Environmental Aging Effects on Thermal and Mechanical Properties of Electrically Conductive Adhesives", To be submitted to the *Journal of Adhesion*.
- 8 S. Xu and D. A. Dillard, Environmental Aging Effects on the Durability of Conductive Adhesive Joints, To be submitted to the *International Journal of Adhesion and Adhesives*.
- 9 M. Zwolinski, J. Hickman, H. Rubin, Y. Zaks, S. McCarthy, T. Hanlon, P. Arrowsmith, A. Chaudhuri, R. Hermansen, S. Lau, and D. Napp, "Electrically Conductive Adhesives for Surface Mount Solder Replacement," *IEEE Transactions on Components, Packaging, and Manufacturing Technology, Part C*, **19**, 241-250. 1996.
- 10 ASTM, "Standard Test Method for Impact Strength of Adhesive Bonds", *Standard D950-94*, 40-44, 1995.
- 11 B. R. K. Blackman, A. J. Kinloch, A. C. Taylor and Y. Wang, "The Impact Wedge-peel Performance of Structural Adhesives", *Journal of Materials Science*, **35**, 1867-1884, 2000.
- 12 M. D. Thouless, J. L. Adams, M. S. Kafkalidis, S. M. Ward, R. A. Dickie, G. L. Westerbeek, "Determining the Toughness of Plastically Deforming Joints", *Journal of Materials Science*, **33**, 189-197, 1998.
- 13 R. D. Adams and J. A. Harris, "A Critical Assessment of the Block Impact Test for Measuring the Impact strength of Adhesive Bonds", *Int. J. Adhesion and Adhesives*, **16**, 61-71, 1996.
- 14 S. Xu, B. Boggess, and David A. Dillard, "Development of a Falling Wedge Test for Adhesive Bonds", *Proceedings of the 23<sup>rd</sup> annual meeting of the Adhesion Society*, 205-207, 2000.

- 15 Kodak EKTAPRO EM Motion Analyzer, User's Manual Model 1012, Motion Analysis Systems Division, Kodak.
- 16 S. Hashemi, A. J. Kinloch and J. G. Williams, "The Analysis of Interlaminar Fracture in Uniaxial Fiber-Polymer Composites", *Proc. R. Soc. Lond.* **A427**, 173-199, 1990.
- 17 M. D. Rakestraw, M. W. Taylor and D. A. Dillard and T. Chang, "Time Dependent Crack Growth and Loading Rate Effects on Interfacial and Cohesive Fracture of Adhesive Joints", *Journal of Adhesion*, **55**, 123-149, 1995.
- 18 D. R. Lefebvre, D. A. Dillard, and H. F. Brinson, "The Development of a Modified Double-Cantilever-Beam Specimen for Measuring the Fracture Energy of Rubber to Metal Bonds", *Experimental Mechanics*, **28**, 38-44, 1988.
- 19 H. Parvatareddy, J. G. Dillard, J. E. McGrath and D. A. Dillard, "Environmental Aging of the Ti-6Al-4V/FM-5 Polyimide Adhesive Bonded System: Implication of Physical and Chemical Aging on Durability", *Journal of Adhesion Science and Technology*, **12** (6), 615-637, 1998.
- 20 American Society for Testing and Materials, "Standard Test Method for Fracture Strength in Cleavage of Adhesives in Bonded Metal Joints", D3433-93, 212-218, 1993.
- 21 B. Blackman, J. Dear, A. J. Kinloch, H. Macgillivray, Y. Wang, J.G. Williams, and P. Yayla, "The Failure of Fiber Composites and Adhesively Bonded Fiber Composites Under High Rates of Test-Part I Mode I loading-Experimental Studies", *Journal of Materials Science*, **30**, 5885-5900, 1995.
- 22 MATHEMATICA, Version 4.0.2.0, Wolfram Research, Inc.
- 23 B. Blackman, A. J. Kinloch, Y. Wang, and J.G. Williams, "The Failure of Fiber Composites and Adhesively Bonded Fiber Composites Under High Rates of Test-Part II Mode I loading-Dynamic Effects", *Journal of Materials Science*, **31**, 4451-4466, 1996.
- 24 J. L. Bitner, J. L. Rushford, A. S. Rose, D. L. Hunston and C. K. Riew, "Viscoelastic Fracture of Structural Adhesives", *Journal of Adhesion*, **13**, 3-28, 1981.

- 25 A. J. Kinloch, *Adhesion and Adhesives, Science and Technology*, Chapman and Hall, London, New York, 314, 1987
- 26 J. M. G. Cowie, *Polymers: Chemistry & Physics of Modern Materials*, 2<sup>nd</sup> edition, Blackie Academic & Professional, 1991.
- 27 S. A. Vona, Jr. Q. K. Tong, R. Kuder and D. Shenfield, “Surface Mount Conductive Adhesives with Superior Impact Resistance”, *International Symposium on Advanced Packaging Materials*, 261-267, 1998
- 28 M. F. Kanninen, “An Augmented Double Cantilever Beam Model for Studying Crack Propagation and Arrest”, *International Journal of Fracture*, **9**, 83-92, 1973.
- 29 K. Arakawa and K. Takahashi, “Interlaminar Fracture Analysis of Composite DCB Specimens”, *International Journal of Fracture*, **74**, 277-287, 1995.
- 30 Aklonis, J. J. and Macknight, W. J., *Introduction to Polymer Viscoelasticity*, 44-52, second edition, 1982.
- 31 D. C. Ohanehi, J.-H. Yu, S. Xu, D. A. Dillard, W. Shaw, D. Lefebvre, R. A. Schultz, and G. Fredrickson “Impact Drop Testing and Finite Element Modeling of Printed-Circuit Board Joints”, *Proceedings of the 24<sup>th</sup> Annual Meeting of the Adhesion Society*, 92-94, 2001.