Effect of chemical treatment on substrate surface for MWCNT-filled epoxy electrically conductive adhesives

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ABSTRACT – Surface treatment of aluminium substrate is one of the alternative method to enhance the strength of the composite ECA adhesive bonding. This paper investigates the effect of chemical surface treatment on aluminium substrate on the mechanical performance of electrically conductive adhesive (ECA). The experimental results revealed that the chemically etched aluminium substrate exhibit higher surface roughness as compared to the as-received aluminium substrate, which is due to high concentration of voids with large opening structure on the surface of former the as-received substrate. The result from lap-shear test reveal that the ECA bonded to chemically etched aluminium substrate exhibits higher shear strength than ECA bonded to as-received aluminium substrate. Such observation is possibly due to high surface roughness and surface wettability that enhance the effective bond area between ECA/substrate interface, which results in enhanced mechanical interlocking of the ECA onto the substrate.

1. INTRODUCTION

The use of lead-based solder for electronic components interconnection in printed circuit board (PCB) are widely used in microelectronic industry. As the awareness to environment increase, the use of lead material for component interconnection is not recommended; hence a substitute material, that is lead-free electrically conductive adhesive (ECA) is introduced [1], [2]. Other alternative besides the ECA is lead-free solder alloys; nonetheless one of the main concern is on its melting temperature, which exceeds the design temperature of various types of circuit board. Moreover, compared to lead and lead-free solder, the processing temperature of ECA is the lowest and below the design temperature of many circuit board [3].

The critical aspect in fine-pitch interconnection field is the adhesion strength of the ECA. This is because ECA is detrimental to shock encountered during handling, assembly and lifetime which require excellent adhesion bond between ECA/substrate interface. Basically, the overall adhesion strength of ECA is from two types of adhesion mechanisms; these being the chemical and physical bonding [4].

This paper investigates the effect of chemical treatment on mechanical strength of ECA bonded joint since there is only limited research found in the literature.

2. RESEARCH METHODOLOGY

The ECA were prepared in dry and clean plastic container. The epoxy and hardener with a ratio of 100:30 were mixed and stirred manually for 1 minute. Then, MWCNT with loading in the range of 5 wt.%, 6 wt.%, and 7 wt.% were added to the mixture for 5 minutes to form the uncured ECA. The uncured ECA were then applied onto the substrates and cured in an oven at 100 ℃ for 30 minutes.

Here, aluminum alloy was used as the substrate. Two types of chemicals were used for chemical surface treatment on aluminium substrate, these being hydrochloric acid (HCl) and sodium hydroxide (NaOH). HCl solution used was QReC Hydrochloric Acid Solution 1 mol/1 (1N) with molecular weight of 36.46 g/mol. Details on the preparation of the MWCNT-filled epoxy electrically conductive adhesives are described in the previous paper by Rahman et al. [5].

3. RESULTS AND DISCUSSION

3.1 Sheet resistance

Figure 3.1 depicts that the average sheet resistance decrease with increasing MWCNT filler loading, from 5 wt.% to 7 wt.%, due to an improved contact between MWCNT particles and enhancement formation of three-dimensional network [4].

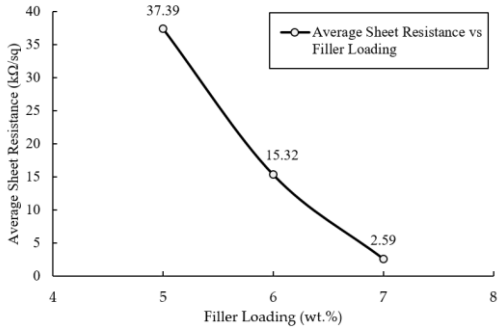

Figure 3.1 Graph of average sheet resistance per unit area against percentage of MWCNT filler loading

3.2 Surface microstructure and roughness

The results from surface roughness measurement by stylus profilometer on as-received and surface treated aluminium substrates are given in Table 3.1. It is apparent that the chemically etched substrate exhibits the highest surface roughness up to 2.60 ± 0.30 μm which is attributed to deep valleys and high peaks distribution throughout the substrate surface observed in 3D profile image.

Table 3.1 Result of single-lap-joint, contact angle and surface roughness for different substrate surface conditions.

Surface Treatment	Average Surface Roughness, Ra (µm)	Average Contact angle, Θ	Filler loading w.t. %	Average Shear Strength, MPa
As-received	0.36 ± 0.02	74.47	5.00	12.98±0.30
			6.00	14.68±0.63
			7.00	10.92±0.94
Chemical Etching	2.60 ± 0.30	16.78	5.00	13.47±0.94
			6.00	16.52±1.51
			7.00	12.02±1.00

The as-received aluminium substrate exhibits the lowest surface roughness due to no surface treatment applied and with the oxide layer presence on the substrate surface provide uniform shallow valleys and low peaks on the entire surface. Based on the 2-D microscopic image in Figure 3.2 (a) & (b) at 200x magnification, the as-received aluminium substrate surface reveal formation of aluminium oxide crack with small voids while chemically etched aluminium substrate has high concentration of voids with large opening structure. Chemically etched substrate has deeper voids on its entire surface as compared to as-received substrate, as depicted in Figure 3.2 (c) & (d).

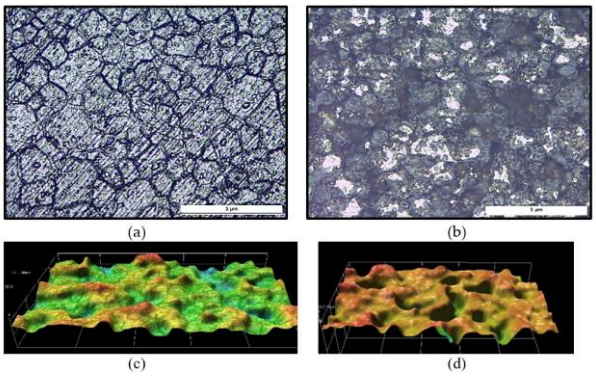

Figure 3.2 2-D and 3-D microscopic images of (a) & (c) as-received and (b) & (d) chemically etched aluminium substrate respectively.

3.3 Shear strength

The ECA with MWCNT filler loading of 5 wt.%, 6 wt.% and 7 wt.% were subjected to lap shear test and the result obtained is shown in Table 3.1. Based on the result obtained, the ECA bonded to chemically etched substrates have higher shear strength as compared to ECA bonded to as-received substrates for filler loading of 5 wt.%, 6 wt.%, and 7 wt.%. This is due to the relation between liquid contact angle and ECA shear strength which chemical etching treatment on aluminium surface yield to excellent wettability since the contact angle is low which result to high shear strength.

Besides, chemically etched substrate provides the highest surface roughness which introduce to large interfacial area between aluminium and ECA that enhance the mechanical interlocking of the interface. The combination of high surface roughness and good wettability of adhesive toward aluminium surface yield to an excellent bond strength as the enhancement of effective bond area [5].

Other factors that may contribute to good adhesion are the chemical etching process activated and cleaned aluminium surface, and dissolved natural alumina layer or weakly bound oxide layer which the natural alumina can reduce the ability of adhesive to spread [6].

4. SUMMARY

This study reported on the effect of chemical treatment on aluminium substrate towards ECA mechanical properties through examination on surface properties and roughness and their correlation with the mechanical properties, that is the shear strength of the ECA. From the experimental work, it was observed that the chemically etched substrate yield in the highest ECA shear strength via lap shear test which is attributed by highest surface roughness and highest wettability, which is an indication of a large effective bond area. In summary, high surface roughness of substrate, which is an indication of high surface area contributes to an excellent adhesion strength between the ECA and the substrate, hence yield in high shear strength.

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REFERENCES

[1] K. Mantena, "Electrical and Mechanical Properties of MWCNT Filled Conductive Adhesives on Lead Free Surface Finishd PCB's.," 2009.

[2] C. C. Sean, S. Pamornpol, O. H. Keat, and R. Durairaj, "Mechanical and Electrical Properties of Carbon Nanotubes Based Isotropic Conductive Adhesives," vol. 857, pp. 93–97, 2016.

[3] C. P. Wong, R. Zhang, and J. C. Agar, "Conductive polymer composites," *Encycl. Polym. Sci. Technol.*, 2011.

[4] L. Yi, L. Daniel, and W. C.P., *Electrical Conductive Adhesives with Nanotechnologies*. New York: Springer Science+Business Media, 2010.

[5] Rahman, W. A. W. A., et al. "The effect of substrate surface conditions on mechanical performance of electrically conductive adhesive." 1st Colloquium Paper: Advanced Materials and Mechanical Engineering Research (CAMMER'18). Vol. 1. Penerbit Universiti, Universiti Teknikal Malaysia Melaka, 2018.

[6] K. Leena, K. K. Athira, S. Bhuvaneswari, S. Suraj, and V. L. Rao, "Effect of surface pre-treatment on surface characteristics and adhesive bond strength of aluminium alloy," *Int. J. Adhes. Adhes.*, vol. 70, pp. 265–270, 2016.