

A New Approach in Measuring Cu–EMC Adhesion Strength by AFM

Cell K. Y. Wong, Hongwei Gu, Bing Xu, and Matthew M. F. Yuen

Abstract—Copper–epoxy molding compound (Cu–EMC) interface is known to be one of the weakest interfaces in an electronic package exhibiting delamination during reliability test. Thiol compound which bonds readily and forms a self-assembly monolayer (SAM) with copper is proposed to improve interfacial adhesion between copper and EMC. Conventional adhesion evaluation involves force measurement in macro-scale. However, inconclusive or even contradictive results are common in those tests because of uncontrollable surface conditions such as contamination and, in particular, roughness. To eliminate the roughness effect and reflect the true chemical bonding condition, an Si wafer was used as a substrate in the experiments. This study involves the use of an atomic force microscope (AFM) in characterizing the nanoscale adhesion force in a Cu–SAM–EMC system. Findings were used as the criteria in selecting a SAM candidate. A thiol compound having a carbonyl group is shown to be the best adhesion promoter from the measurement. The nanoscale AFM results are shown to be consistent with the result of macroscopic shear tests. It has been demonstrated, with SAM treatment on a cleaned copper surface, that the fracture force between Cu–EMC samples is improved from 119 to 195 N.

Index Terms—Atomic force microscope (AFM), copper-epoxy molding compound (Cu–EMC) adhesion, nano-force characterization, self-assembly monolayer (SAM).

I. INTRODUCTION

A prime reason for the failure in copper–epoxy molding compound (Cu–EMC) interface is lack of adhesion between copper and epoxy compound. Previous research demonstrated adhesion improvement by surface modification like ozone treatment, oxide growth, and chemical etching [1], [2], while others proposed the use of a polymeric coupling agent [3]. Oxide growth is one of the most common methods due to the inherent oxidation tendency of copper. Extensive investigation was carried out on the influence of copper oxide thickness on adhesion integrity. The findings suggested that cupric oxide (CuO) which resulted in needle-like morphology played a dominant role in copper/EMC interfacial strength and a control of oxide thickness was essential for the success of Cu–epoxy adhesion property. An optimum oxide thickness was

proposed as 20–40 nm from Tankano *et al.* [4] and 20–30 nm from Cho *et al.* [5]. However, during package assembly, the packages encountered several thermal processes. Control of copper oxide thickness is difficult if not impossible. Copper oxide growth is therefore not a practical solution for the adhesion problem. Other research work has been concentrated on the use of silane coupling agents as an adhesion promoter in electronic package interfaces. Song *et al.* [3] has applied different silane to Alloy 42 leadframes and found that high adhesion strength is obtained by the use of γ -aminopropyltrimethoxysilane. However, as chemical linking in the silane system is through Cu–O–Si–R–EMC bonds, the siloxane bonds are susceptible to hydrolytic cleavage upon moisture intrusion [6], [7]. Due to the inherent hydrophilic character of epoxy, moisture content of EMC can be as high as 3000 ppm, this causes debonding of the Cu–EMC interface during the soak test. Tong *et al.* [8] has studied adhesion improvement of Si die adhesion to underfill by silane and discovered that the adhesion degradation rate could be controlled by the mobility of absorbed water in the polymer matrix. Loss of adhesion strength due to high temperature and high humidity aging could recover to some extent by drying out the absorbed moisture. The extent of recovery depended on the polymer chain mobility, as well as the crosslinking density of polymer matrix, and was confirmed by Luo *et al.* [9]. Leung *et al.* [10] has investigated debonding phenomenon which is assisted by stress, and they observed that coupling agent chemistry, pH, temperature, and stress affected the rate of adhesion degradation. Even though bond strength could be recovered by dehumidification, the stress-assisted delamination was still critical to bond degradation during long term reliability tests. Despite being used as additives inside EMC formulation, direct application of the coupling agent onto Alloy 42 leadframe surfaces have been studied by Song *et al.* [3]. They have concluded that effective interfacial adhesion improvement has been achieved even at high humid and high temperature conditions. It is also found that the orientation of coupling agents at the interface played an important role in adhesion retention during high temperature and high humidity aging.

Muller *et al.* [11] has suggested adhesion promotion of a copper-epoxy system by SAM. Unlike conventional polymeric silane coupling agents, which are added to EMC formulation, the SAM solution develops a thin molecular layer on the copper surface. The monolayer molecules allow effective covalent linkages between copper and epoxy that can enhance interfacial integrity. One of the potent SAM candidates is thiol, an organic compound with a sulphur head and organic end. Owing to the low bonding energy in Cu–S bond, copper reacts readily with

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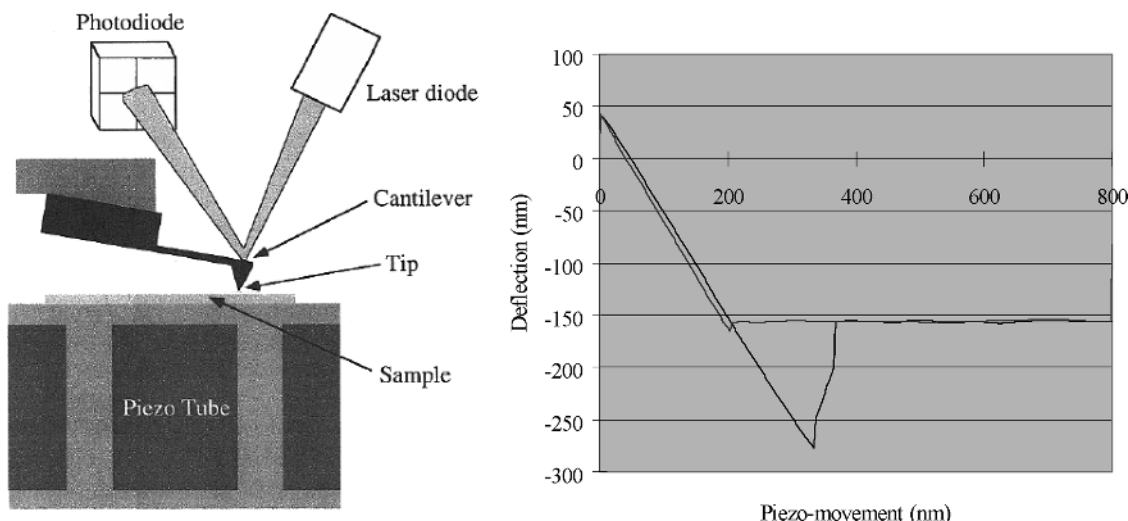


Fig. 1. (a) Schematic diagram of AFM showing the force sensing cantilever and (b) typical force plot of an AFM.

thiol. Besides, modification of the alkyl group in the thiol compound can create a wetting surface to EMC melt that can promote adhesion. Having an organic end, thiol can enhance adhesion in a Cu-EMC system through strong covalent linking C-S-Cu.

Interfacial adhesion is a complicated problem that involves fracture, dynamic interaction with the environment, surface contamination, and roughness. A simple approach is to study the fundamental adhesion mechanism between copper and epoxy, to eliminate the surface roughness effect on adhesion, and study bonding at molecular level. An atomically smooth copper substrate is used to eliminate the roughness effect so as to reflect intrinsic bonding behavior of the Cu-epoxy system. The intrinsic adhesion bond between a SAM coated copper-epoxy system is measured by AFM. AFM offers a tool to measure nano-scale forces between a probe tip and its sample as it approaches and retracts from a surface [12], [13]. The microscopic cantilever force sensor used is made of silicon nitride with a dimension of $100\ \mu\text{m}$ in length and $0.6\ \mu\text{m}$ in thickness [Fig. 1(a)]. By scanning the AFM cantilever back and forth toward the sample surface, deflection of the cantilever tip is measured. The deflection is then plotted against the displacement of cantilever. Adhesion force is recorded as force at which the adhesion between probe and sample is ruptured and the cantilever comes free from the surface. A representation of this adhesion measurement is illustrated in Fig. 1(b). Actual adhesion force is calculated by multiplying the deflection of the cantilever by the cantilever's spring constant.

In developing the Cu-SAM-epoxy bonding system, five different types of thiol SAM material have been synthesized. The adhesion force result of the Cu-SAM system has been used as a selection criteria of the SAM layer. Results of the surface characterization of SAM-coated copper in terms of chemical composition, roughness and contact angle measurement has been presented as a complimentary tool to evaluate the adhesion parameters. The adhesion of the Cu-SAM-epoxy system has been further evaluated using the standard button shear test [14].

This paper focuses on the development and characterization of the Cu-SAM-epoxy bonding system with the help of nano force measurement by AFM. The study provides a basic

TABLE I
SPECIFICATION OF THIOL SAM MATERIAL

Sample name	Thiol type
A1	acidic with amine group
A2	acidic with carboxyl group
A3	acidic with hydroxyl group
C1	with carbonyl group
C2	with polymeric carbonyl group
Uncoated Cu	N/A

evaluation of the performance of Cu-SAM-epoxy systems and will help to establish the guidelines in the selection of SAM materials so as to enhance adhesion across the copper-epoxy interface.

II. EXPERIMENTAL MEASUREMENTS

In the evaluation of thiol monolayer as an adhesion promoter in EMC-copper interface, the concentration and choice of material significantly affects the strength of the interface [15]. To enhance the adhesive strength with EMC, the tail group of the thiol molecule should be able to bond with the epoxy group. Five different thiols had been selected. As illustrated in Table I, samples are sub-divided into two groups: acidic (group A) and carbonyl thiol (group C). Unlike a conventional silane coupling agent which reacts randomly with copper surface, thiol molecules bond with copper only through the Cu-S link. Ordered film can therefore be easily obtained from SAM deposition. The adhesion force result is more accurate and representative.

A. Copper Substrate Preparation

Copper substrate was prepared by sputter $500\ \text{\AA}$ Ti/W + $5000\ \text{\AA}$ copper onto a (100) Si wafer by the sputter system.

(Explorer 14, Denton Vacuum). The atomically flat copper substrate was then degreased with organic solvent and Fry 90 flux (Fry Technology). Fry 90 cleans the metal surface by reducing surface oxide with a strong inorganic acid, and a controllable surface having a rare metal oxide can be obtained for thiol deposition. Degreasing was performed by sonicating the sample inside DI water for 10 min, then soaking in acetone and isopropanol, respectively, for 5 min. The samples were then rinsed with DI water thoroughly before being wiped in Fry 90 with a cotton bud. After they were cleaned with the inorganic acid flux, the samples were rinsed again with DI water and then dried thoroughly in nitrogen gas.

B. Tip Preparation

Silicon nitride tips for adhesion detection were purchased from Digital Instrument, Inc. The dimension of the tips are 100- μm long, 20- μm wide, and 0.6- μm thick with a spring constant reported as 0.38 N/m. To measure the adhesion force between copper and samples, the silicon nitride tip was coated with 30 Å TiW and 600 Å copper prior to nano AFM.

C. Formation of SAM

All five SAM samples were dissolved in an appropriate solvent and diluted to 0.5 mM. The pre-cleaned Cu-substrate was dipped onto the SAM solution for 15 s immediately after the cleaning process. The treated substrate was taken out of the SAM solution and rinsed thoroughly with an appropriate solvent before blow drying with nitrogen.

D. Epoxy Surface Preparation

Epon 828, a standard diglycidyl ether of bisphenol A (DGEBA) for formulation and Epikure F206, common cycloaliphatic amine curing agent from Shell Chemicals, had been chosen in the epoxy-SAM adhesion study. The epoxy resin and hardener were well mixed and coated onto a substrate by spin coating at 10 000 rpm for 1 min.

E. Button Shear Test

Three types of samples were tested in the button shear test: A1, C1, and an uncoated copper substrate. The substrate was cut into a 6 mm \times 27 mm strip, acknowledging the mold box requirement. All the substrates were cleaned and deposited according to method in part A and C.

The EMC used in the button shear test was EME G6300C (Sumikon, Sumitomo Bakelite). To avoid unexpected curing, the EMC was stored at -40°C and was taken out from the freezer 24 h before the molding process. During the defreeze process, the EMC was kept under vacuum to minimize moisture absorption.

EMC had been molded on a surface treated substrate by transfer molding. The molding and curing temperature was performed as recommended by the manufacturer.

The Dage 4000 tester was used in the shear test. The shear force was reported as the maximum load at which the sample fractures.

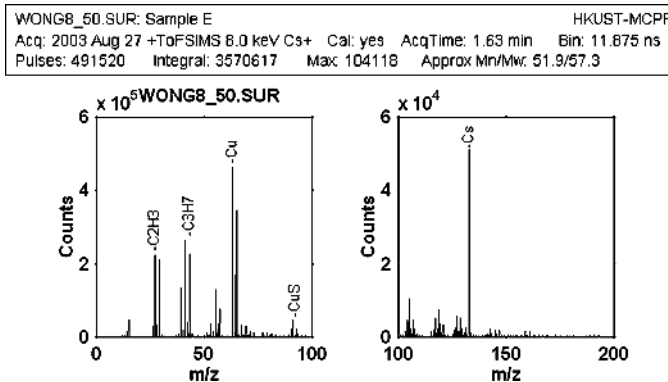


Fig. 2. Positive ToF-SIMS result of SAM film on Cu, the 96 m/z spike the Cu-S bond.

F. Surface Analysis

1) *Cu-S Bond Confirmation*: A time-of-flight secondary ion mass spectrometer (ToF-SIMS), Model PHI 7200 (physical electronics), equipped with Cs and Ga ion guns, was used to evaluate the formation of a Cu-S bond on SAM coated samples.

2) *Contact Angle*: A contact angle measurement was achieved using a Goniometer model 100 (Rame-Hart Inc.). While a sessile droplet was put to the surface, contact angles (θ_w) between samples to water were reported.

3) *Surface Roughness*: Surface roughness was determined optically by a three-dimensional (3-D) optical profiler (WYKO NT300 profiler, WYKO Corp.). The roughness was quantified by 20 \times optical lens in PSI mode with 600 μm \times 400 μm scan area.

4) *Fracture Surface Analysis*: X-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometer (ToF-SIMS) mapping was adopted to determine the fracture surfaces of samples after shear tests. Elemental content was determined from XPS and identification of molecular fragments from different regions on fracture samples was been analyzed by ToF-SIMS mapping.

G. AFM Measurement

The AFM measurement was conducted using a multimode scanning probe microscope equipped with a Nanoscope E Controller from Digital Instrument, Inc. All of the measurement was carried out in an air medium. During the test, the samples were extended and retracted from the tip in a range of 800 nm with a speed of 5 s per cycle. Fig. 5 showed a typical force plot of AFM nano-force measurement. Pull-off distance was defined as the difference between initial deflection and minimum deflection. Adhesion force can then be calculated as the pull-off distance multiple by the spring constant of the cantilever.

III. SURFACE CHARACTERIZATION RESULT

A. Cu-S Bond Confirmation

From the ToF-SIMS result shown in Fig. 2, it is confirmed that chemical bonding has been realized on the interface.

According to Ron *et al.* [16], formation of high quality thiol is not disturbed by thin surface oxide. The observation helps to confirm the chemical interaction between copper oxides and thiol. Because of the intrinsic chemical potential of these two

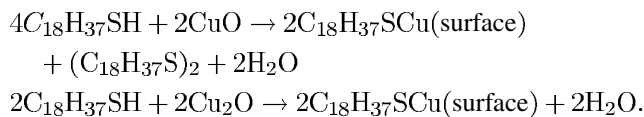


Fig. 3. SAM deposition on a rough surface.

TABLE II
CONTACT ANGLE MEASUREMENT OF DIFFERENT SAM TREATED SURFACE

Sample Type	Contact angle to water (°)
A1	58.50±5.92
A2	68.00±2.16
A3	61.25±2.06
C1	63.50±1.73
C2	66.00±2.71
Uncoated Cu	60.50±2.38

species, thiol tends to reduce to disulfide in the presence of copper oxide



From the above argument, cleaning of the substrate before SAM deposition may not be needed. However, as copper oxide can roughen a copper surface [2] and hence orients the deposited thiol in a random manner (Fig. 3), the oxide layer still needs to be removed from the sputtered surface by acid cleaning in order to get a controllable SAM film.

B. Contact Angle

Table II reviewed the wettability of water to a SAM coated copper sample. For polar polymeric material, just like the case of EMC, good wetting of the polar group to the treated surface would be expected. Given the low contact angle measured, SAM samples are ready for bonding with EMC.

C. Surface Roughness

As it has been mentioned previously, surface roughness is one of the key elements that control the chemical bonding. A rough morphology on one hand can improve adhesion through surface interlocking. On the other hand, it prevents formation of intrinsic secondary bonding for gap separation. Although a single crystal Si substrate had been used, a local roughness measurement of the samples was still needed to explain the resulting trend. A 3-D roughness plot of SAM coated copper is shown in Fig. 4.

The roughness for SAM films is around 2 nm, which is marginally rougher than the uncoated copper substrate without SAM treatment. Detailed roughness data is reported in Table III.

From surface characterization by ToF-SIMS, it is confirmed that SAM has been introduced to copper substrate through intrinsic Cu-S bonding. A low contact angle in the goniometer

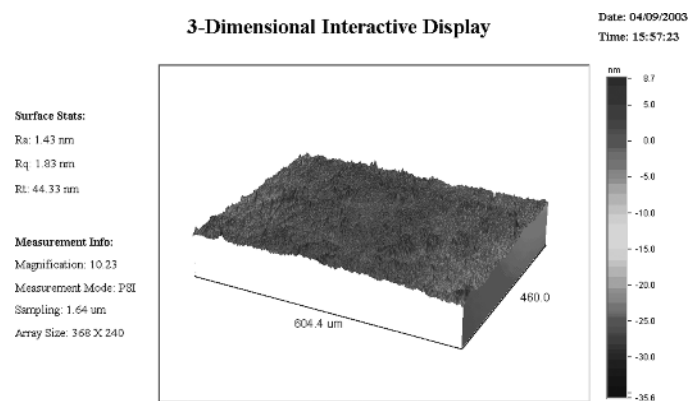


Fig. 4. Surface roughness obtained from optical profiler.

TABLE III
SURFACE ROUGHNESS OF THE SAMPLES

Sample Type	Roughness Ra(nm)
A1	2.24±0.07
A2	1.97±0.11
A3	1.99±0.04
C1	1.93±0.04
C2	2.35±0.00
Uncoated Cu	1.66±0.41

measurement reveals that the prepared SAM surface is ready to wet with polar groups. Surface roughness data shows the formation of SAM on a copper substrate has only a marginal adverse effect on the roughness depending on the type of SAM deposited. This illustrates the appropriateness of the specified treatment procedure.

IV. AFM MEASUREMENT RESULT

A. Adhesion Force Between Cu-SAM

Five SAM coated samples have been prepared according to the above procedure. The adhesion strength between these freshly prepared samples and the copper tip was evaluated by AFM. The measured adhesion force between the copper tip and substrate forms the selection criteria of SAM material. Fig. 5 shows a typical force plot for adhesion force measurement (see Fig. 6).

High adhesion force has been found from SAM samples in group C (C1 and C2). The adhesion force value for SAM C1 doubles that of SAM A1. It can be attributed to the presence of the carbonyl group in the thiol tail. Being an electron withdrawing group, carbonyl can induce a partial electrostatic attraction upon an electron drawing from the copper substrate. This can enhance tip-substrate adhesion. For those acidic SAMs (Group A), instead of electron withdrawal from a copper surface, the conjugate base of the acid group tends to behave as a nucleophile and donates an electron. This hinders the formation of strong secondary bonding to a copper substrate.

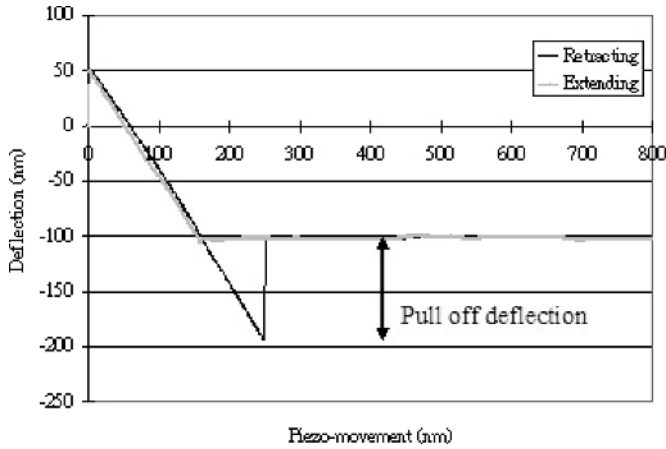


Fig. 5. Force plot of Cu-SAMC1 adhesion (tip deflection versus z movement of the piezoelectrical stage).

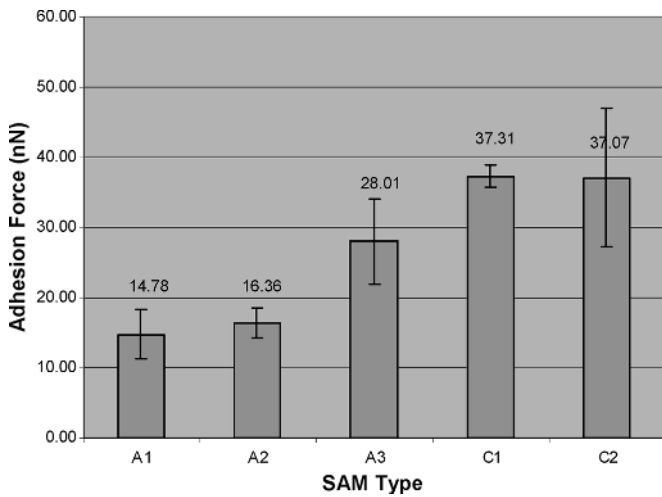


Fig. 6. Adhesion force of SAM samples to copper tip.

B. Adhesion Force Between Cu-SAM-Epoxy

To confirm the adhesion enhancement between a copper and epoxy interface by SAM, AFM measurement with a SAM coated tip and epoxy substrate was conducted.

Fig. 7 demonstrates stronger interaction between epoxy and a SAM coated copper interface. This can be explained by the interaction between organic tails of thiol molecules with the polar groups found in epoxy. H-bonding is usually involved in this case (see Fig. 8).

V. BUTTON SHEAR TEST RESULT

A benchmarking experiment was conducted to apply the findings in macro scale for electronic packaging applications. In order to justify the nano adhesion result, fracture force between EMC on SAM treated samples was evaluated using the Dage 4000 Tester.

Button shear parameters were given as

- shear height: 50 μm ;
- shear speed: 85 $\mu\text{m/s}$;
- over travel: 100 μm .

Fig. 9 reveals that significant enhancement in adhesion between Cu-epoxy interfaces has been achieved by SAM deposi-

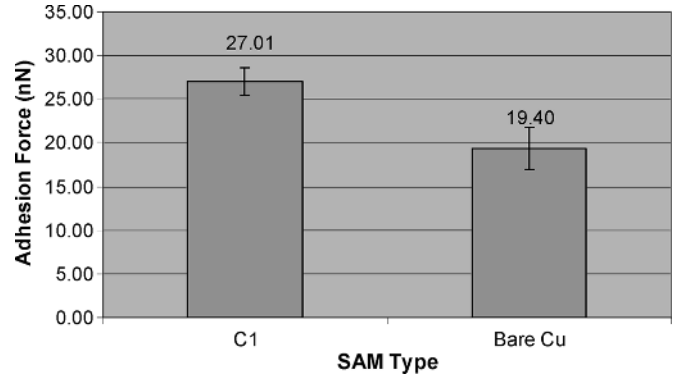


Fig. 7. Adhesion force comparison: SAM coated versus uncoated copper sample.

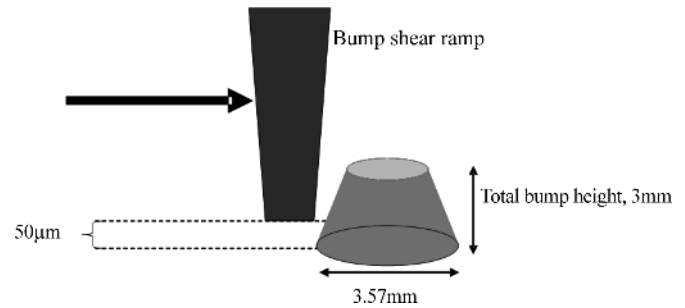


Fig. 8. Schematic diagram for button shear test.

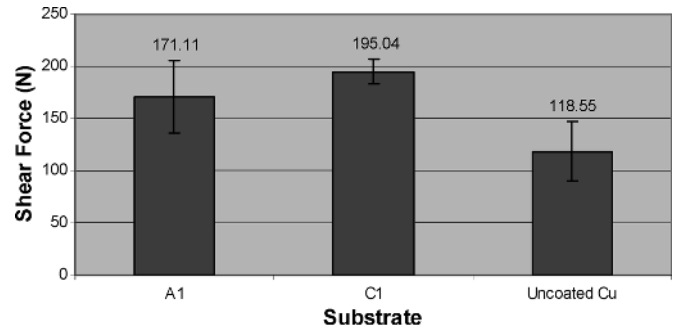


Fig. 9. Shear test result for SAM treated substrate.

tion. Adhesion force has been increased by 60% after introduction of SAM C1 to the interface.

Optical image of the sheared surface is included in Fig. 10 to explain the shear results. From all the sheared substrates, two distinguished areas could be observed: the shiny region (R1) and opaque region (R2). To examine the fracture interfaces, surface analysis by XPS and ToF-SIM were conducted.

VI. FRACTURE SURFACE ANALYSIS

A. XPS

Table IV summarized the elemental analysis for fracture surface R1 and R2 on different sample SAM A1, SAM C1, and bare copper.

XPS elemental analysis was conducted to investigate the fracture interface despite the fact that the XPS test was performed right after the shear test (note that Cu is very reactive to oxygen and it might become oxidized and affect the sample before its placement into the XPS chamber). The quantitative values stated

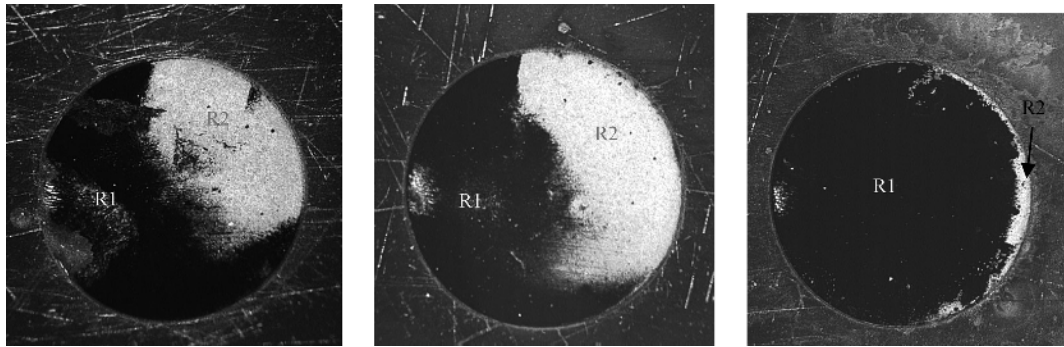


Fig. 10. Optical microscope of after-sheared surface of Cu samples, 20 \times , SAM A1 (left), SAM C1 (center), and uncoated Cu (right).

TABLE IV
ELEMENTAL ANALYSIS ON FRACTURE SURFACES ON R1 AND R2 ON SAMPLE SAM A1, SAM C1 AND BARE COPPER

	A1 (R1)	A1 (R2)	C1 (R1)	C1 (R2)	Bare Cu (R1)	Bare Cu (R2)
C1s	39.95	71.60	39.52	76.19	38.74	76.76
O1s	34.84	19.94	33.91	17.39	33.69	17.22
Si2s	0.16	1.50	0.84	1.91	1.90	3.25
S2p	0.00	0.00	0.00	0.00	0.00	0.00
Cu2p3	25.05	6.96	25.74	4.51	25.68	2.78
Possible fracture surface	Cu-EMC (interfacial)	EMC (cohesive)	Cu-EMC (interfacial)	EMC (cohesive)	Cu-EMC (interfacial)	EMC (cohesive)

in Table IV may be marginally distorted for this reason. However, the test is vital in providing information on relative elemental content on the fracture surface. It showed that carbon content in R2 was significantly higher than in R1. Besides, there was less copper found on R2 than R1. An explanation for the content derivation in the two regions is that a different fracture mode happened in these regions. Cohesive failure inside EMC lead to high carbon content being detected in R2. A relatively large amount of copper located in R1 was due to adhesive fracture where interfacial failure occurred at the copper and EMC interface. Besides, a complete lack of sulphur detected from XPS proved that strong bonding was established between copper and thiol.

B. ToF-SIM

To further justify the previous argument, ToF-SIM mapping has been conducted. Fig. 11 illustrated the composition on R1 and R2 of the three samples. Consistent results as XPS have been demonstrated. The major component in R1, the shiny region for all samples is copper. The mapping indicated that composition in R2 for SAM A1 and SAM C1 samples are organic material which is believed to be EMC. On the fracture surface of the bare copper samples, copper has been the major component with few spots of silane being detected on the surface.

From the ToF-SIM observation, it is deduced that interfacial fracture occurs in R1 while cohesive failure occurred in R2 as

EMC residue remained on the region. From Fig. 10, the intact region (R2) in SAM coated samples is much larger than that found in uncoated samples. The percentage of area for R1 and R2 has been calculated and reported in Table V. Enhancement of fracture load for SAM coated samples (C1 and A1) can be explained by a larger intact area which contributed a higher degree of cohesive influence. The findings imply a shift of fracture locus from interfacial failure in bare copper to cohesive failure inside EMC as SAM deposition.

VII. CONCLUSION

In this study, AFM has been used in quantifying the nano-adhesion force between Cu-SAM-EMC materials. Adhesion data from different SAM coated substrates presents a distinct trend showing the carbonyl group SAM as a better adhesion promoter. In the AFM measurement, higher adhesion strength has been reported for SAM C1. The same trend has been observed in the button shear test. Over 60% of improvement in fracture load has been achieved by treating the precleaned copper substrate with thiol-SAM material. Thiol has been proven to be a strong adhesion promoter in the fracture surface analysis, it shifts fracture locus from an interfacial failure to a cohesive one. In this paper, AFM was demonstrated to be a sensitive and powerful tool in the verification of the SAM material as an adhesion promoter in Cu-EMC systems.

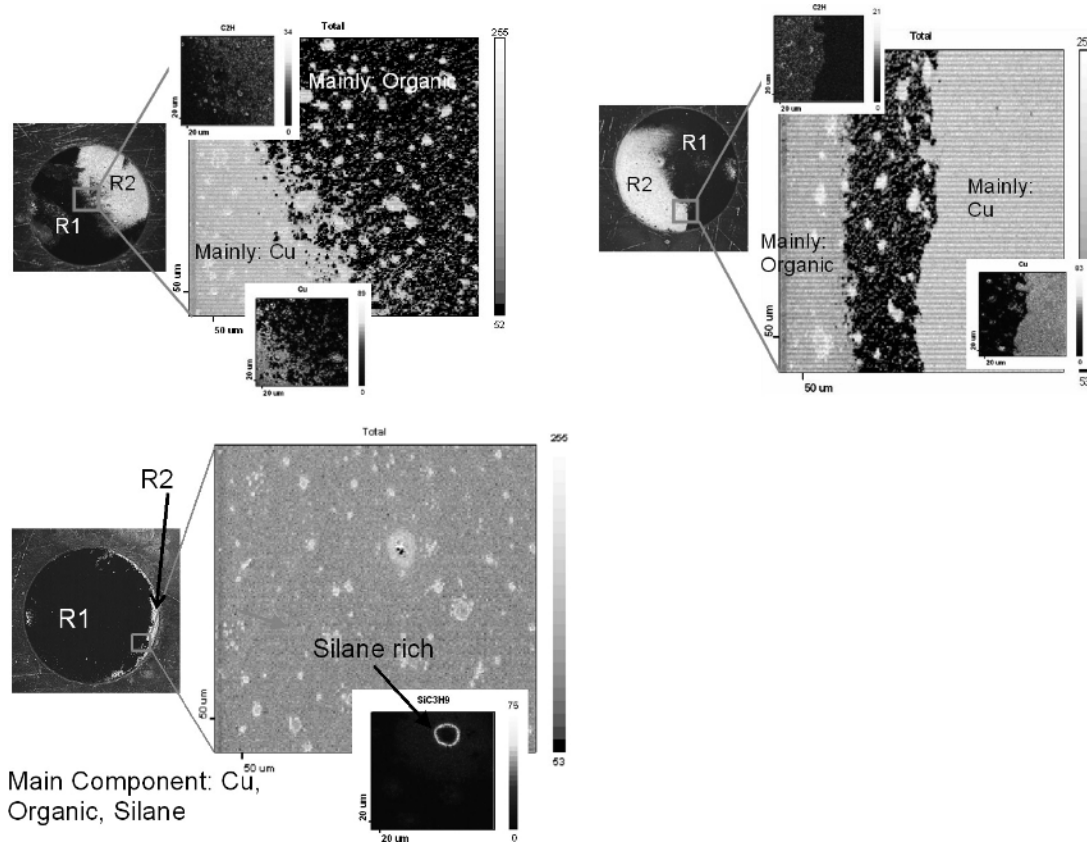


Fig. 11. ToF-SIM mapping on the fractured surface of the samples SAM A1 (top left), SAM C1 (top right), and bare Cu (bottom).

TABLE V
SUMMARY OF TOF-SIM RESULTS, DEGREE OF COHESIVE INFLUENCE AND FRACTURE STRENGTH ON DIFFERENT REGIONS IN THE THREE SAMPLES

	A1 (R1)	A1 (R2)	C1 (R1)	C1 (R2)	Bare Cu (R1)	Bare Cu (R2)
Major Fragment	Cu	Organic	Cu	Organic	Cu	Few spot of organic
Fracture surface	Cu-EMC (Interfacial)	EMC (Cohesive)	Cu-EMC (Interfacial)	EMC (Cohesive)	Cu-EMC (Interfacial)	EMC (Cohesive)
% Area	65.49	34.51	58.99	41.01	94.13	5.87
R2/ R1 ratio (degree of cohesive influence)	0.527		0.695		0.062	
Fracture Strength (N)	171.11		195.04		118.55	

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