Enhanced Electrical Properties of Anisotropic Conductive Adhesive With π-Conjugated Self-Assembled Molecular Wire Junctions

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Abstract—We have investigated the electrical properties of anisotropic conductive adhesive (ACA) joint using submicrometer-sized (∼500 nm in diameter) silver (Ag) particle as conductive filler with the effect of π-conjugated self-assembled molecular wires. The ACAs with submicrometer-sized Ag particles have higher current carrying capability (∼3400 mA) than those with micro-sized Au-coated polymer particles (∼2000 mA) and Ag nanoparticles (∼2500 mA). More importantly, by construction of π-conjugated self-assembled molecular wire junctions between conductive particles and integrated circuit (IC)/substrate, the electrical conductivity has increased by one order of magnitude and the current carrying capability of ACAs has improved by 600 mA. The crucial factors that govern the improved electrical properties are discussed based on the study of alignments and thermal stability of molecules on the submicrometer-sized Ag particle surface with surface-enhanced Raman spectroscopy (SERS), providing a fundamental understanding of conduction mechanism in ACA joints and guidelines for the formulation of high-performance ACAs in electronic packaging industry.

Index Terms—Anisotropic conductive adhesive (ACA), current carrying capability, self-assembled monolayer, surface-enhanced Raman spectroscopy.

I. INTRODUCTION

TIN–LEAD solder alloy has been widely used as a joining material in the electronic packaging industry. However, there are some intrinsic problems associated with tin–lead solder, such as environmental concerns and high processing temperature limiting their future applications. Considerable advances have been made in the development of lead-free solders

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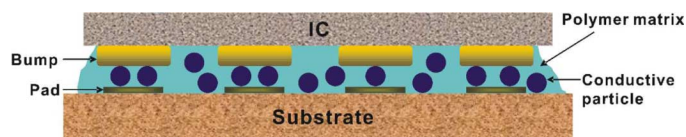D. D. Lu is with the Intel Corporation, Chandler, AZ 85226 USA.

Fig. 1. Schematic representation of the cross section of an ACA junction.

and electrically conductive adhesives (ECAs) as promising alternatives of the tin–lead solder. ECAs have the advantages of environmental friendliness, mild processing conditions, fewer processing steps, low stress on the substrates, and fine-pitch interconnect capability. These numerous advantages of ECAs over the conventional solder technology allow the wide applications of ECAs in the fields of flat panel displays such as liquid crystal displays (LCDs) and semiconductor packaging such as smart cards where low-temperature interconnects are needed. Nevertheless, some challenging problems have to be solved before the replacement of tin–lead solder with ECAs such as lower electrical conductivity, conductivity fatigue in reliability testing, limiting current-carrying capability, and poor impact strength [1]–[5]. A cross section of the joint formed by ACA between the integrated circuit (IC) and substrate is shown in Fig. 1. The unidirectional electrical conductivity is achieved through the conductive particles trapped between IC and substrate when the ACA is fully cured under pressure and heat. During the assembly process, it is very likely that a small amount of resin is left behind or trapped in between conductive particles and IC/substrate, which is an insulating layer that hinders current flow between IC and substrate after curing. The contact resistance increases as the coverage of the particles with the insulating layer increases and finite-element analysis (FEA) by Divigalpitiya [6] indicated that the solid particle cannot tolerate debris coverage of the contact spot without increase in the resistance. Moreover, the contaminants such as hydrocarbon on the conductive particle also increase the contact resistance, which has been generally neglected.

Over the past two decades, self-assembled monolayers (SAMs) have attracted significant interests due to their potential applications in corrosion prevention, lubrication, wetting control, wear prevention, oxidation retardation, adhesion, and molecular electronic devices [7]. Of particular interest are electron transport in metal-molecule(s)-metal junctions and thus applications of SAMs in molecular electronics. Significant decrease (down to two orders of magnitude) in metal-molecule contact resistance can be achieved using an alkanethiol-based

junction due to chemically bonded contacts instead of non-bonded (mechanical) contacts [8]. Generally, the Fermi level of the noble metal electrodes lies between the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of contact molecules. By tuning the work function of the electrodes with SAMs, tunneling resistance could be significantly reduced and thus improvement of electrical conduction can be obtained. Furthermore, the efficiency of charge transport through π-conjugated molecules sandwiched between two electrodes further contributes to the enhanced electrical conduction of metal-molecule contacts. Besides the improvement of electrical conduction, Kobayashi *et al.* demonstrated the ability of SAMs to control the charge-carrier density in organic field-effect transistors (FETs) [9].

Taking advantages of those unique properties of SAMs, we introduced self-assembled molecular wire junctions to improve the electrical properties of ACAs [10] and nonconductive films (NCFs) [11] for the electronic interconnects in previous work [12]. The resulting ACAs and NCFs exhibited improved electrical conduction, higher current carrying capability and better thermal stability. Crucial for successful applications of SAMs in ACAs and NCFs are interfacial properties (chemisorption or physisorption), the characteristics of the molecules (conjugated or unconjugated, thermal stability, and so on), and surface binding geometry. Current work will investigate the effects of those factors on the performance of ACAs by using a series of π-conjugated molecules with different functional groups, providing an insight into the factors that govern enhanced properties of resulting ACA interconnects.

II. EXPERIMENTAL

Three molecules with different functional groups, terephathalic acid (TPA), terephthalonitrile (TPN), and 4-cyanobenzoic acid (CBA) were used. Submicrometer-sized Ag particles ($\sim$500 nm in diameter, Degussa Corporation) were treated in the SAM solutions and then sonicated for 10 min. After the SAM treatment for 24 h, the samples were removed from the solution and rinsed with solvent used for the dissolution of the molecules in order to remove excess unadhered SAM molecules. Ag particles were separated from SAM solutions by centrifuge and then dried under vacuum before the characterization. The coating of SAM on Ag particles was characterized by Raman spectroscopy. Raman spectra were obtained using Bruker Optics FT-Raman (Equinox 55) with near infrared (NIR) lasers ($\lambda = 1064$ nm). To obtain the thermal behavior of SAM on Ag particles, the sample were heated at 150 °C and 200 °C for 0.5 h and then characterized by Raman spectroscopy.

For characterizing the electrical properties of submicrometer-sized Ag particles filled ACA, 5 wt% of Ag particles were added in a polymeric resins, in which diglycidyl ether of bisphenol-F (DGEBF), anhydride, and imidazole were used as epoxy, hardener, and catalyst, respectively. The electrical contact resistance of the ACA joints (contact area: 150×150 μm^2) was measured by a four-point probe method [10]. The applied four-point probe currents were varied by a power supplier (HP model 6553A, HP
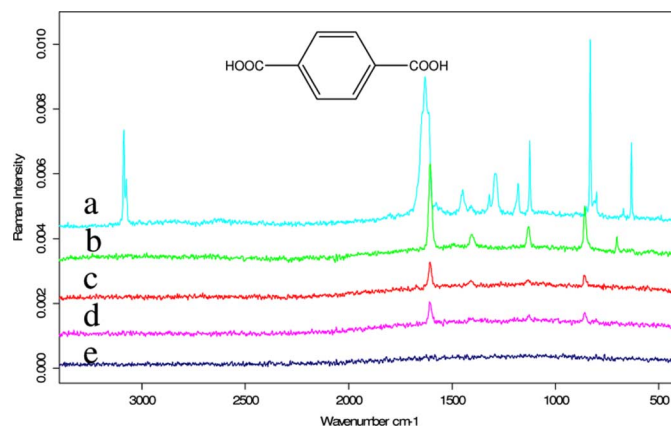

Fig. 2. Raman spectra of (a) TPA, (b) TPA adsorbed on Ag particles, TPA adsorbed on Ag particles baked at (c) 150 °C, and (d) 200 °C for 0.5 h, respectively, and (e) Ag particles.
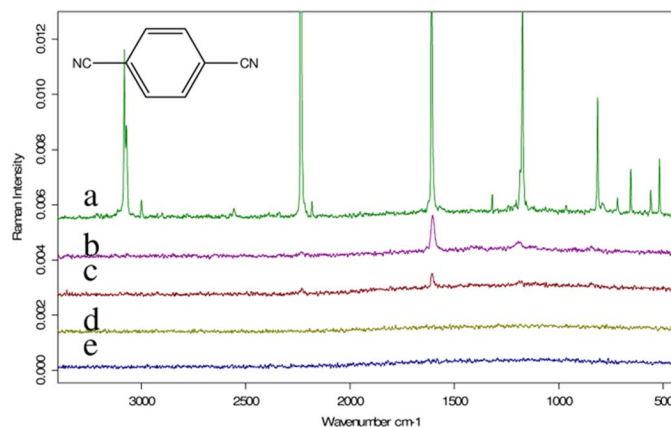

Fig. 3. Raman spectra of (a) TPN, (b) TPN adsorbed on Ag particles, TPN adsorbed on Ag particles baked at (c) 150 °C and (d) 200 °C for 0.5 h, respectively, and (e) Ag particles.

Hewlett Packard, Palo Alto, CA) and the voltage of the interconnect joints were measured by a Keithley 2000 multimeter (Cleveland, OH).

III. RESULTS AND DISCUSSION

Submicrometer-sized Ag particles with narrow size distribution were coated with terephathalic acid (TPA), terephthalonitrile (TPN), and 4-cyanobenzoic acid (CBA), and their thermal stability on Ag particle surface was investigated by surface-enhanced Raman spectroscopy (SERS). SERS is a very useful surface spectroscopic technique for the study of the orientation of various kinds of molecules adsorbed on noble metal surfaces (Au, Ag, and Cu) due to its high spectral resolution and the spectral sensitivity from monolayer adsorbates on those metal surfaces. SAM molecules, Ag particles, and SAM-treated Ag particles at room temperature, baked at 150 °C and 200 °C for 0.5 h are shown in Fig. 2 (TPA), Fig. 3 (TPN), and Fig. 4 (CBA). The assignment of peaks is based on the [13]–[16] and summarized in Table I. In Fig. 2, the C=O stretching peak at 1629 cm^{-1} of TPA disappeared in the spectrum of TPA absorbed on the Ag surface. Instead, the symmetric stretching and bending
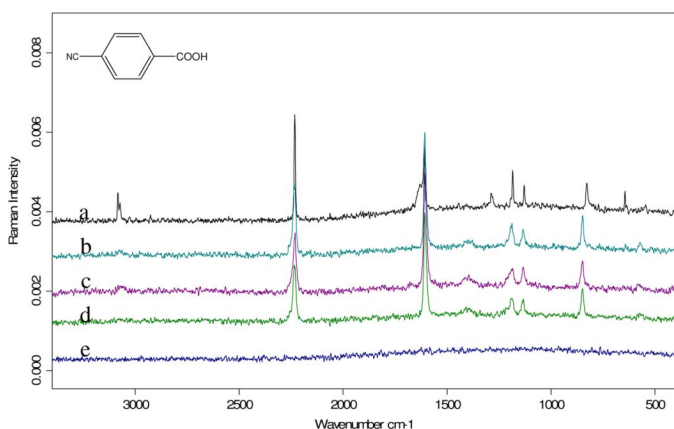
Fig. 4. Raman spectra of (a) CBA, (b) CBA adsorbed on Ag particles, CBA adsorbed on Ag particles baked at (c) 150 °C and (d) 200 °C for 0.5 h, respectively, and (e) Ag particles.

modes of COO$^-$ appeared at 1405 and 703 cm^{-1}. Hence, TPA was chemisorbed on the silver surface via two Ag-COO$^-$ bonds after deprotonation and lay flat on the Ag particle surface. Moreover, according to the electromagnetic enhancement mechanism for SERS, the absence of the peak at 3086 cm^{-1} due to the C-H stretching further indicated that TPA lay flat on the Ag surface [17]–[19].

Aromatic nitriles can coordinate with Ag by three different ways, namely the nitrogen lone-pairs, the C ≡ N π system and the π system of the benzene ring. Generally, a majority of aromatic nitriles were absorbed on the silver surface via C ≡ N π system rather than the nitrogen lone-pair electrons [14]. A slight increase in C ≡ N stretching frequency of TPN was attributed to the disruption of conjugation, as was observed when TPN adsorbed on the silver sol (silver colloidal particle) surface. However, upon surface adsorption of TPN on a submicrometer-sized Ag particle, C ≡ N stretching frequency decreased. C ≡ N stretching frequency can either decease or increase depending on the relative importance of nitrile-to-metal π donation and conjugation. More importantly, substantial broadening of C ≡ N stretching strongly indicated that C ≡ N of the adsorbed TPN interacted directly with Ag particle. The complete absence of C-H stretching band in the SERS spectra of TPN implied that the benzene ring of TPN lay flat on the Ag particle surface.
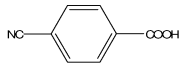
Kim *et al.* reported that the CBA molecule adsorbed onto a silver film as 4-cyanobenzoate with a perpendicular orientation with respect to the surface while it lay flat on the surface of silver sol [20]. The adsorption behavior of CBA on the flat silver surface appeared to be completely different from that on the silver sol surface. The origin of such a different adsorption mechanism was not clarified. As for the adsorption behavior of CBA on submicrometer-sized Ag particles, 1) the presence of band at 1378 cm^{-1} is due to ν (COO$^-$) and the absence of bands at 1630 cm^{-1} (ν (C=O)) and 1287 cm^{-1} (δ(OH) + ν(C − O)) indicates that CBA absorbs on Ag as 4-cyanobenzoate; 2) A slight increase (3 cm^{-1}) in the C ≡ N stretching frequency on absorption attributed to the disruption of conjugation rather than end-on coordination and the substantial band broadening indicates a direct interaction of nitrile group(s) with Ag particle surface; 3) A noticeable C-H stretching at 3078 cm^{-1} in Fig. 4

in contrast with the near-absence of C-H stretching in Figs. 2 and 3 is a very useful guideline in SERS to probe the orientation of adsorbate on the metal surface which implies that the benzene ring is oriented perpendicularly with respect to the Ag particle surface. Interestingly, the adsorption behavior of CBA on submicrometer-sized Ag surface was different from that of Ag sol. Based on the above assignments, it can be concluded that CBA adsorb perpendicularly on submicrometer-sized Ag surface with two oxygen atoms bound symmetrically to the surface instead of lying flat on the particle surface, as schematically illustrated in Fig. 5.

Thermal stability of SAM molecules on Ag particles is of great importance for their applications in ACA since the SAM coating is used to improve the interface between conductive adhesive joints after the curing of ACA. The SERS in Fig. 3 indicates that TPA was partially detached from the Ag surface after baking at 150 °C for 0.5 h and no further detachment was observed at 200 °C for 0.5 h. For TPN, it was almost detached from the Ag surface at 150 °C for 0.5 h and completely removed from the surface at 200 °C for 0.5 h (see Fig. 4). However, even after baked at 200 °C for 0.5 h, no obvious detachment of CBA on Ag particle surface was observed (see Fig. 5), which may indicate that the surface alignment of SAM molecules may have some effect on their thermal stability.

During the assembly of an ACA joint, electrically conductive fillers were trapped between the two pads, which provide electrical conduction in the Z-axis, as shown in Fig. 6. The current-voltage ($I–V$) curves of submicrometer-sized Ag filled ACAs are shown in Fig. 7(a). Compared with control sample (ACA with unmodified Ag particles), decreased contact resistance was observed for the ACAs with TPA and CBA-treated Ag particles while the ACA with TPN-treated Ag particles showed higher resistance. Previous research in our group has demonstrated that SAM molecular wires can act as electrical junctions between the conductive filler of the ACA and the contact pad of the substrate [10]. As such, the electrical path at the interface could be enhanced, which enables the improved conductivity. For TPN, the weak interaction of Ag with the molecule and flat surface alignment may weaken the interaction between Ag particle and metal bond pads, resulting in decreased conductivity. It has been well demonstrated that thin tarnish films (excluding gold) or ultrathin insulative adhesive films are present on the contact surfaces of the ACA joints, which create a high contact resistance (tunneling resistance) [6], [21]–[25]. The existence of silver oxide and surface contaminants was confirmed by X-ray photoelectron spectroscopy (XPS) (Fig. 8). The O1s peak at 533.8 eV in XPS general survey and broad Ag 3d/2 and Ag 5d/2 peaks in high resolution XPS of Ag 3d may indicate the presence of silver oxide or possibly silver carbonate. The C1s peak at 286.1 eV may result from the surface contaminant. Compared with TPN, TPA has stronger affinity to Ag particles and more importantly, the removal of silver oxide and surface contaminants on the Ag surface may play an important role in the increased conductivity as reported by our research group [26]. As a result, the conductivity of the resulting ACA joint increases, although TPA lies flat on the particle surface. The significant improved electrical properties of CBA-treated Ag particles are because the molecule

TABLE I
SPECTRAL DATA AND THE VIBRATIONAL ASSIGNMENTS OF TPA, TPN AND CBA

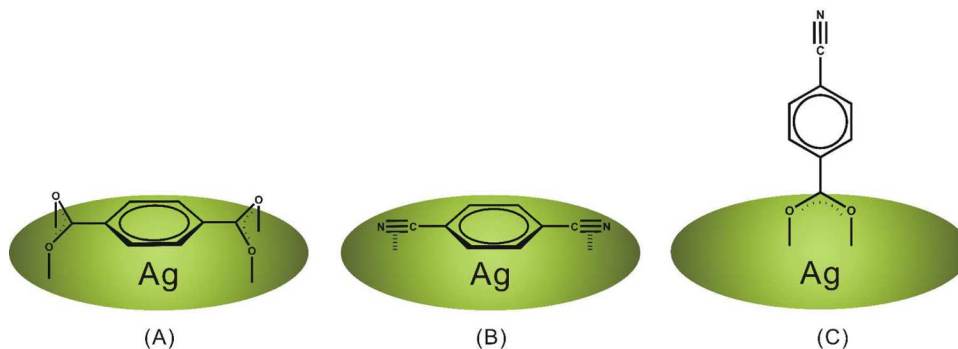| TPA | | | TPN | | | CBA | | |
| HOOC—⬡—COOH | | | NC—⬡—CN | | | NC—⬡—COOH | | |
Ordinary Raman (ν/cm^{-1})	μ-Ag SERS (ν/cm^{-1})	Assignment	Ordinary Raman (ν/cm^{-1})	μ-Ag SERS (ν/cm^{-1})	Assignment	Ordinary Raman (ν/cm^{-1})	μ-Ag SERS (ν/cm^{-1})	Assignment
631		6b	517		δ_{as}(CN)	829		δ(OCO)
670		4	559		γ_{as}(CN)		847	δ(COO$^-$)
	703	δ(COO$^-$)	655		6b	1131	1133	7a
830	858	1	813		1	1185		13+9a
1123	1129	7a	1173		7a		1188	9a
1283		ν(C-O)		1194	13	1287		δ(OH)+ ν(C-O)
	1405	ν_s(COO$^-$)	1608	1604	8a		1378	ν(COO$^-$)
1448		δ(OH)	2236	2234	ν(CN)	1610	1607	8b
1610	1606	8a	3071		ν_s(CH)	1630		ν(C=O)
1629		C=O stretching	3080		ν_{as}(CH)	2233	2236	ν(CN)
3075		ν_s(C-H)				3074		ν_s(CH)
3087		ν_{as}(C-H)				3084	3078	ν_{as}(CH)

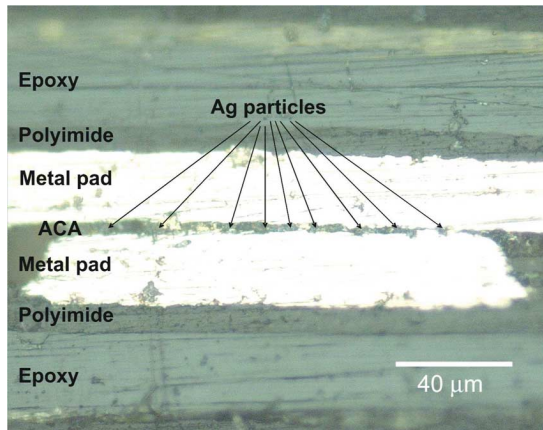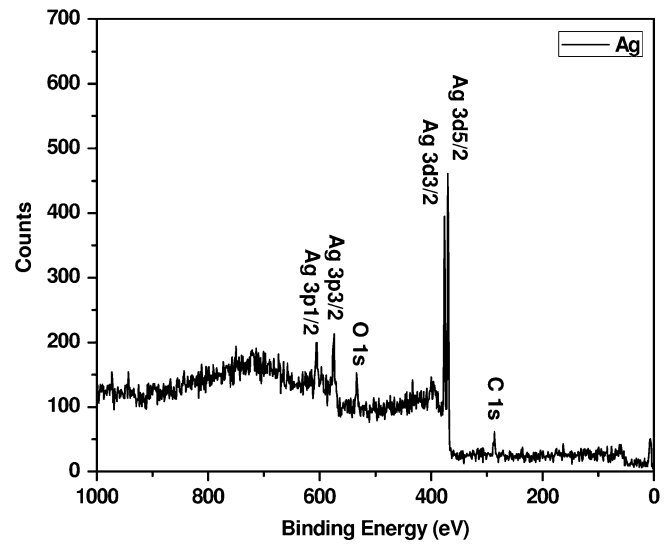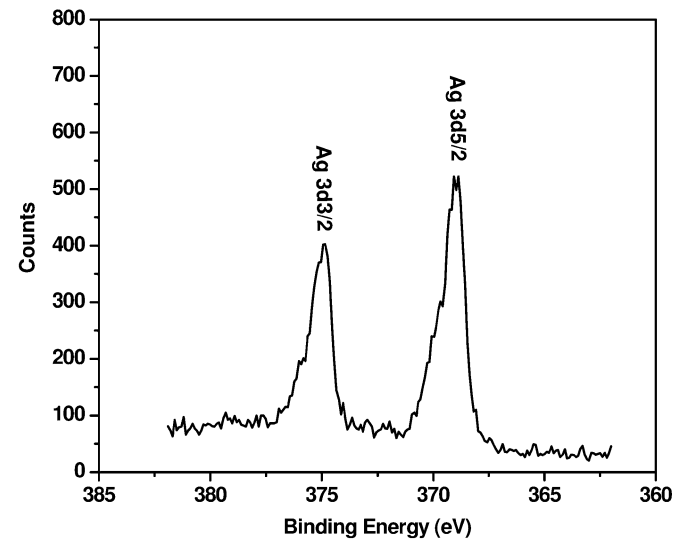Fig. 5. Alignments of TPA, TPN, and CBA on a submicrometer-sized Ag particle surface.

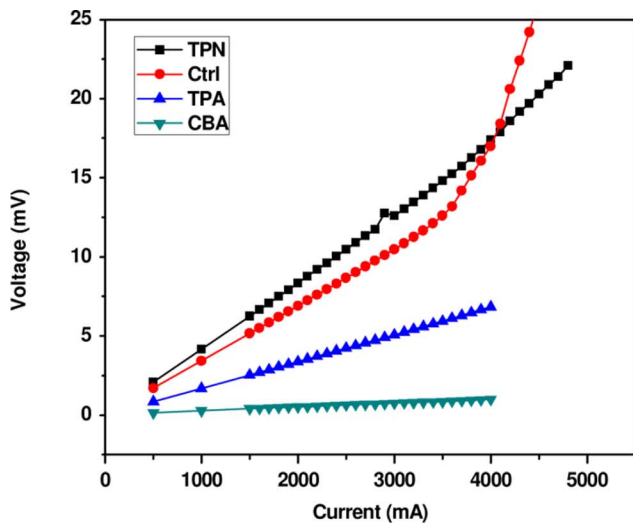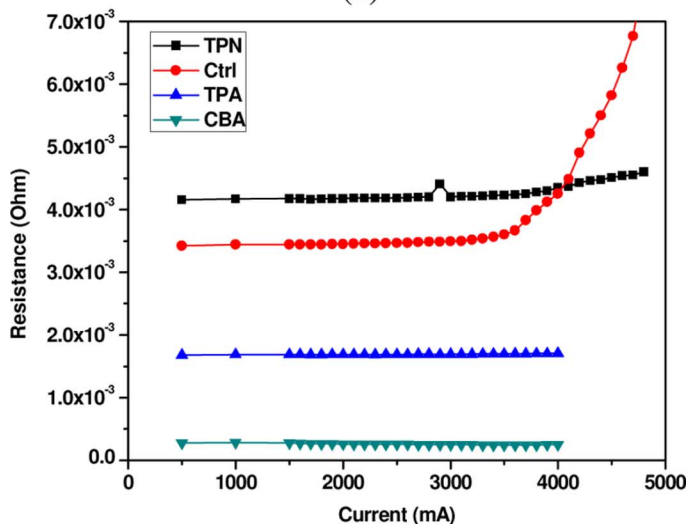Fig. 6. Optical photograph of a cross section of an ACA joint.

(a)

(b)

Fig. 7. (a) $I–V$ curves of ACA filled with Ag particles (Ctrl), TPN-treated Ag particles, TPA-treated Ag particles, and CBA-treated particles. (b) Corresponding $I–R$ curves. All $I–V$ curves were measured at room temperature.

(a)

(b)

Fig. 8. (a) XPS survey spectrum and (b) high-resolution XPS Ag 3d peaks obtained from submicrometer-sized Ag particle.

Moreover, the vulnerability of conductive adhesives with regards to mechanical deformation is commonly observed, which may result from the edge of the contact between conductive particles and IC/substrate carrying more current than the rest of the structure [27]. Thus, the construction of self-assembled molecular wire junctions between conductive particles and IC/substrate will maintain the conductive particles in place and contribute to the reliability of the ACA joint. For ACA filled with CBA-treated Ag, the resistance decreased from 10^{-3} to 10^{-4} Ω [see Fig. 7(b)]. Moreover, the submicrometer-sized Ag particles have higher current carrying capability ($\sim$3400 mA) than micro-sized Au-coated polymer particles ($\sim$2000 mA) and Ag nanoparticles ($\sim$2500 mA) [28]. More importantly, with the introduction of SAMs on an Ag particle surface, the current carrying capability increased steadily from 3400 mA for control sample without SAM-treatment to about 4000 mA with SAM-treated samples. All measurements were conducted at

can adhere to conductive fillers and substrate (-CN group can coordinate with Au substrate), forming physic-chemical bonds and allow electrons to freely flow through the interfaces.

room temperature. With increasing the applied current, the joint temperature increased. Typically, for metal, as the temperature increases, the mobility of charge carriers goes down and thus the conductivity decreases.

IV. CONCLUSION

Surface alignment of SAM molecules and their thermal stability on submicrometer-sized Ag particles were investigated by SERS. With the introduction of π-conjugated self-assembled molecular wire junctions, the electrical conductivity and current carrying capability of ACAs were significantly improved. The crucial factors that govern enhanced electrical properties of the resulting ACAs interconnect were explored based on the study on the characteristics of SAM molecules, interaction between Ag particles and SAM molecules, molecular chain orientation of SAM molecules on Ag particle surface and their thermal stability. With the improved electrical conductivity and current carrying capability, the ACA material is expected to be a promising solder replacement for high electrical performance, fine pitch, high current carrying capability interconnect applications.

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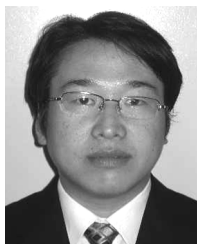
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