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Amino acids as activators for wave solder flux systems: Investigation of solderability and humidity effects

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Abstract- The role of flux activators in flux formulation is to remove oxides from metal surface, hence allow the formation of metallurgical bond between metal substrate and solder alloy. However, flux activator in the no-clean flux residue on printed circuit board assemblies (PCBAs) affects the climatic reliability due to its hygroscopic and ionic behavior under humidity exposure. Four amino acids-based wave flux were investigated using wetting balance test and microscopy inspection, and the humidity effects of four amino acids was analyzed using electrochemical impedance spectroscopy (EIS) and chronoamperometry under various testing conditions. Water absorption and thermal degradation were analyzed using gravimetric method and Fourier-transform infrared spectroscopy (FT-IR). Combined results indicate glutamine based model flux obtained superior solderability for 96.5Sn-

3Ag-0.5Cu (SAC 305) alloy due to suitable melting and activation temperature of amino group for the oxides removal. The robust humidity reliability of amino acids was dependent upon relative high deliquescence point and low moisture absorption under harsh climatic conditions.

Index Terms— amino acid; solder flux; electronic corrosion; humidity; reliability; solderability.

I. Introduction

No-clean flux residue is one of the most representative intrinsic contamination source on the printed circuit board assembly (PCBA) surface since 70-80% production of electronics today use no-clean flux chemistry [1]. It was designed with the idea that during the soldering process, content of the flux system evaporate or degrade, therefore leave minimal residues [2][3] to save cleaning steps in the manufacturing

process [1]. However, due to sluggish kinetics of evaporation and decomposition depending on the thermal conditions on the PCBA surface, considerable amount of flux residue will be presented after the soldering process [1][4]. Such flux residue mainly contains 2.5-5 wt/vol.% weak organic acids (WOAs) activators and the rosin/resin component of the flux system [2][5][6][7]. On the component side of selective wave soldered PCBAs, residue levels of WOAs up to $\sim 687\mu\text{g}/\text{in}^2$ has been reported [1]. Particularly, the ionic and hygroscopic contaminants on PCBAs reduce the humidity boundary for water condensation [8]. The most frequently used WOAs in the flux formulation today include glutaric acid, succinic acid and adipic acid etc. [3][9][10], which possess the deliquescence relative humidity (RH) level of WOAs in a range of 78-99% RH [2]. Therefore, WOAs with lower level humidity boundaries developed the risk of water condensation on PCBAs. The dissolution of WOAs to the condensed water film will reduce the pH of the electrolyte layer on the PCBA surface, and hence increases the conductivity of the condensed water layer and accelerates the corrosion of the metallic

terminals under biased conditions on the PCBA surface [11][12][13]. The electrochemical process on the biased PCBA surface, and dissolved metallic ions from anodic terminals further induce high leakage current and possibly end up with the electrochemical migration (ECM) related short circuit [14]. To sum up, no-clean flux activator played a vital role for the humidity robustness of the electronic devices.

The selection criteria of no-clean flux activators used in the flux systems is based on the removal ability of the oxide layer and evaporation temperature [9][15], while partially on corrosion reliability [1][9]. Carboxyl groups in WOAs act as functional group for the oxides removal by disproportionation reaction [16]. At meantime, the effective carboxyl group for oxides removal also possessed lower acidic dissociation constant (pKa), which reduced the corrosion reliability of the flux system [6]. Consequently, many flux manufacturers use binary or ternary blended WOAs in flux chemistry to balance the solderability and corrosion reliability[3][17], although one WOA type can dominate. However, Piotrowska et al. reported the binary blended

WOAs thermodynamically reduce the deliquescence RH compared to the individual WOA [18][19], which resulted water absorption at lower RH and accelerated corrosion of electrical terminals.

To obtain good humidity robustness of no-clean flux residue on PCBAs, a number of investigation focused on using organic amines as effective additive to enhance the corrosion reliability of the flux systems though leaving flux residue on the PCBAs after soldering process [16][20][21][22]. Organic amines behave as weak alkaline in water solution due to hydroxyl group formed by the protonation of the amino group [23], which can neutralize the acidic pH induced by the WOA residues. Additionally, investigations also shows that the growth of ECM dendrite was inhibited by forming stable Cu complex compounds with amines [21]. Compared to the mixture of WOA and organic amine, amino acids possess both carboxyl group and amino group in the chemical structure, which may provide superior solderability while maintain the robust corrosion reliability as no-clean flux activators. Moreover, amino acids are generally eco-friendly, water-

soluble, mild and biodegradable [24]. As result, the amino acid attracted the attention of the flux manufacturers as the potential alternative for commercial used WOA activators [25].

Recently, anonymous amino acid candidates were tested in the formulation of wave solder fluxes [26] and reflow solder pastes [27]. Reported results indicate amino acid contained flux formulation obtained better assembly with minimum defects compared to WOA contained solder paste in low temperature soldering application [27]. These investigations on anonymous amino acid activators were mainly focused on the printability and solderability aspects, whereas the interpretation of the corrosion reliability assessment is limited [26][27]. Kiani et al. reported the corrosion inhibition performance of cysteine, methionine, and alanine on Pb-bearing solder alloy [28]; while Yang et al. reported the good solderability of glutamic acid in halogen containing fluxes [29]. However, the available literature specifically focused on amino acid as additives to the halogen containing flux systems and Pb-based solder alloys instead of the presently employed no-clean

flux systems [30][31] and lead free solder alloys [32]. Previously, the solderability, humidity robustness and corrosion reliability of amino acid activators have not been investigated from research point view, only limited information can be found in patents which suggested isoleucine, glycine, alanine, serine, arginine, glutamine etc. can be used flux activators in both wave solder flux and solder paste [25][33]. Therefore, it is important to assess the possibility of using these amino acids as flux activators to obtain good humidity robustness. Due to the strong alkalinity of arginine solution and high cost of analytical-grade isoleucine, the other four amino acids with near-neutral pH and reasonable prices were considered as potential activator candidates. To optimize the flux chemistry with better activator for solderability and humidity robustness, it is important to understand the performance of amino acid activator with comparison of the commercial used WOA flux activators.

The present investigation focused on understanding the corrosion reliability of four types of amino acids when used as flux activators and present as flux residues, while the

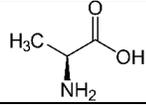
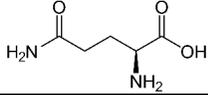
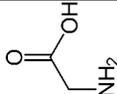
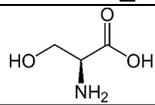
solderability of these activators are investigated together with commercial used WOA activators. An interdigitated SIR pattern was used as the test vehicle for investigations. Electrochemical impedance spectroscopy (EIS) was used to understand the humidity interaction with amino acid residues. ECM susceptibility of amino acids contaminated SIR pattern was evaluated using chronoamperometry (CA). Wetting balance was used to investigate the solderability of amino acid model fluxes. The degradation production of amino acid residue was analyzed using fourier-transform infrared spectroscopy (FT-IR).

II. Materials and methods

A. Amino acids used for the investigation

Table 1 shows the physical and chemical properties of four amino acids with relatively neutral pH value. As the table shows, the molecular weight of the amino acids vary from approximately 75 – 146 g/mol, while the melting point varies from low melting point of 185 to 314 °C. Melting point of flux activator is an important parameter corresponding to the

Table 1. Properties of the amino acids used in the investigation.

	Molecular Structure	Molecular Weight (g/mol)	Melting Point (°C)	pKa1 (carboxyl)	pKa2 (amino)	pI	Volume fraction in model flux (vol.%)
Alanine		89.09	314	2.34	9.69	6.00	2.85
Glutamine		146.14	185	2.17	9.13	5.65	2.76
Glycine		75.07	233	2.34	9.6	5.97	2.52
Serine		105.09	246	2.21	9.15	5.68	2.54

solderability. Isoelectric point (pI) indicates the pH that amino acid does not carry electrical charge. It can be expressed by Equation 1, where pK_{a1} is the dissociation constant of carboxyl group, pK_{a2} is the dissociation constant of amino group.

$$pI = \frac{pK_{a1} + pK_{a2}}{2} \quad \text{Equation 1}$$

For investigations, model fluxes were mixed using 4 wt.% of amino acid and 96 wt.% ultra pure water in an ultrasonic bath for 30 mins, where the volume fraction of flux activator was in a range of 2.52-2.85% as shown in Table 1. Ultra pure water source was adapted from Milipore water purification system (Synergy UV, Germany).

B. Amino acids used for the investigation

Cu coupons in a size of 6 mm × 25 mm × 0.5 mm were prepared using electrical discharge machining based on the requirements from IPC-TM-650 no.2.4.14.2 for solderability testing. Cu coupons were cleaned using ISO 9455-16 soft soldering fluxes standard before each test. The solderability test was performed in an air-static condition. Molten bath of SAC 305 solder alloy was controlled at 270°C using a Must 3 wetting balance (GEN 3, UK). Model flux was applied on the Cu coupons by dipping for 2 sec. After 20 sec preheating at the position of 1 cm above the molten solder bath, Cu coupons were immersed in the solder bath for 10 sec to a depth of 5 mm. The speed of immersion and withdraw of Cu

coupons was 20 mm/s. As reference, model WOA fluxes with 4 wt.% adipic acid and succinic acid in isopropanol alcohol was used. To obtain better statistics, 3 repetitions were performed for each type of acid.

C. Evaluation of humidity effects of amino acids using EIS and CA methods

Humidity exposure experiments were conducted in a climatic chamber (Espec PL-3KPH, Japan) with the accuracy of $\pm 0.3^\circ\text{C}$ and $\pm 2.5\%$ relative humidity (RH) using a five-channel potentiostat (Biologic VSP, France). Issacs et al. reported the localized contamination level of WOA on wave soldered PCBA can be as high as $687\mu\text{g}/\text{in}^2$ ($106.5\mu\text{g}/\text{cm}^2$) [1]. Therefore, the contamination level of the amino acid on the SIR testing area was determined at $100\mu\text{g}/\text{cm}^2$. To imitate the soldering process, some of the SIR test board was thermally activated at 240°C for 45 sec in a Techno HA-06 furnace. For three electrode electrochemical experiments, one electrode of the SIR interdigitated pattern was used as reference and counter, the other was used as working electrode. The aim of this three electrode system

is to imitate positive and negative bias points on a PCBA surface.

EIS technique was used to investigate the deliquescence relative humidity (DRH) and efflorescence relative humidity (ERH) of amino acid candidates. DRH indicates the humidity level triggering water absorption by amino acids, and ERH indicates the lower humidity level required for the release of water from the amino acids. In previous work, scanning frequency of 1-10 KHz in EIS was confirmed to provide the information of the transformation from capacitive dominant behavior to resistive dominant behavior of SIR components during water condensation process [18][34]. Therefore, scanning frequency was set at 10 KHz in the amplitude of 25 mV in order to record the impedance response of amino acid contaminated SIR interdigitated pattern during the 24 hours under linear humidity ramping from 30% RH to 99% RH, and then ramped back to 30% RH. To understand the influence of the service temperature on the water absorption and desorption behavior, tests were conducted at 25°C , 40°C , and 60°C respectively. All test boards were dried in the climatic chamber

at 30% RH for 6 hours before the EIS measurement started.

CA technique was used to understand the leakage current and ECM induced by amino acid residue on the SIR interdigitated pattern under humid conditions. For this purpose, a 5 V DC bias was applied on the SIR test board during the test. Prior to the data recording, test boards were stabilized at the testing climate for 4 hours. Leakage current value of the test boards was recorded at relative humidity level of 80%, 90%, and 98% over 24 hours at testing temperature of 25°C, 40°C, and 60°C respectively.

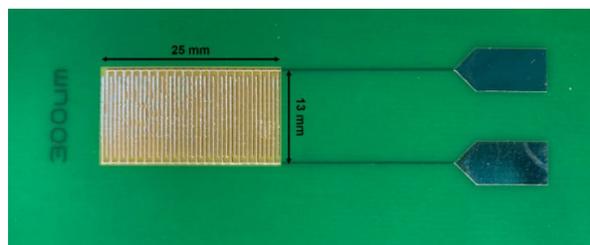


Fig. 1. Test PCB with SIR interdigitated pattern used for investigations.

Test PCB with SIR interdigitated patterns shown in Fig. 1 was used as test vehicle in the present work. The surface finish of the PCB was hot air solder leveling using SN100C alloy on the 35 μm thick Cu trace. The width of the conductive lines

and the pitch distance are 0.3 mm. Testing area of the SIR pattern was 13 mm \times 25 mm and the overlapping length of electrodes was 442.8 mm in 41 sets. Test PCBs were initially cleaned with isopropanol alcohol and dried under compressing air before each test.

D. Quantification of water absorption and test objects characterization

Water absorption behavior of amino acids and two commercial used activators as reference (adipic acid, succinic acids) were evaluated using a Vsorp gravimetric analyzer (ProUmid, Germany) under the RH stages of 85%, 90%, and 95% respectively at room temperature until the water absorption reaches the equilibrium condition. The tested samples of amino acids and WOAs were stabilized at 25°C, 30% RH for 620 hours before water absorption test. The gravimetric measurement was performed 60 times in every hour.

The surface morphology of Cu coupons used for solderability testing was inspected using a light optical microscope (LOM) (Keyence VHX, Japan). The cross section of soldered

Cu coupons and corrosion product built on SIR pattern was further evaluated using scanning electron microscopy (SEM) (FEI Quanta 200 FEG, USA) under secondary electron mode at 15 kV. The chemistry of the corrosion product on the SIR pattern was analyzed using a 50 mm² X-Max EDS detector (Oxford Instrument, UK). The thermal degradation product of tested amino acids were analyzed using a Nicolet iN10 MX FT-IR (Thermo Fisher Scientific, USA) after thermal activation under 240°C for 100 sec in a Techno HA-06 oven.

III. Results

A. Solderability assessment of amino acid candidate

Fig. 2 shows the wetting force of six types of model fluxes including four individual amino acids and two WOAs as reference. The summary of wetting balance test was shown in Table 2. The highest maximum wetting force was obtained by succinic acid at 3.97 ± 0.04 mN, followed by glutamine at 3.78 ± 0.02 mN and adipic acid at 3.69 ± 0.03 mN. Wetting force for glutamine is slightly

higher than the commercially used adipic acid. However, the time reach to zero wetting force and the time reach two third wetting force of glutamine is slightly higher than adipic acid and succinic acid. Serine also shows positive maximum wetting force at 2.83 ± 0.05 mN, while the maximum wetting force is lower than for adipic acid and the wetting took 2.37 ± 0.05 sec to reach to zero wetting force, which is much higher than 0.94 ± 0.09 sec for adipic acid and 1.00 ± 0.06 sec for succinic acid. The wetting force of alanine and glycine were in negative value, which indicate the fluxes were not able to etch the surface of Cu coupons during soldering process. The rank of the time to zero wetting force and time reach two third wetting force are the same, which were : serine > glutamine > succinic acid > adipic acid > glycine/alanine. The rank of the maximum wetting force were: succinic acid > glutamine > adipic acid > serine > glycine > alanine.

Fig. 3 shows the surface appearance and defects of the soldered Cu coupons using four amino acid model fluxes. LOM images and SEM images in Fig. 3 (a) and (c) shows the poor wetting

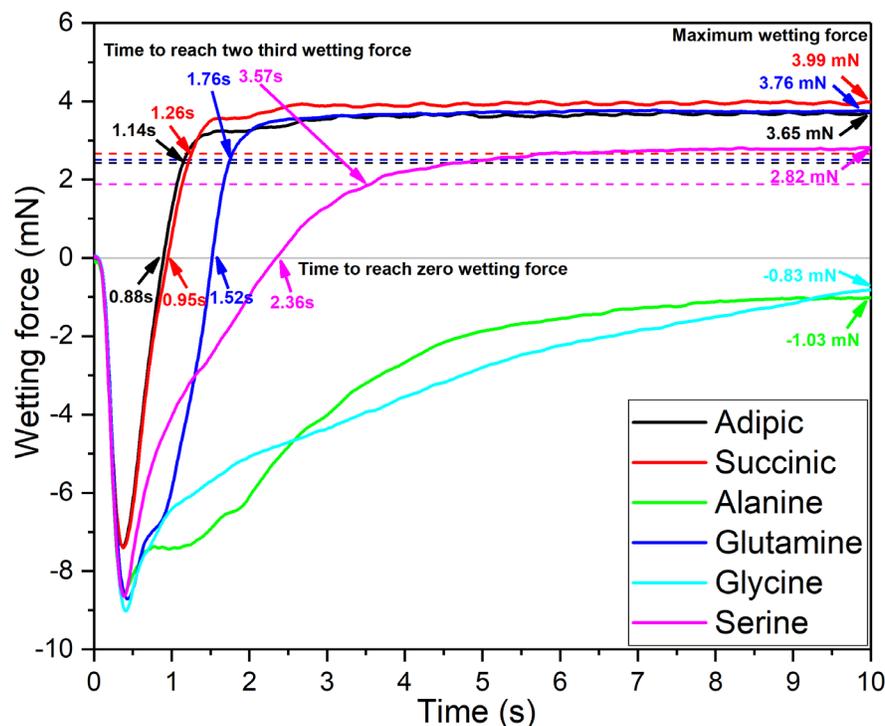


Fig. 2. Wetting balance results of amino acids and reference WOA model fluxes.

Table 2 Summary of the solderability test for amino acids and reference WOA model fluxes.

	Time to reach Zero Wetting force (sec)	Time to two thirds maximum wetting force (sec)	Maximum wetting force (mN)
adipic acid	0.94 ± 0.09	1.32 ± 0.23	3.69 ± 0.03
succinic acid	1.00 ± 0.06	1.34 ± 0.08	3.97 ± 0.04
Alanine	-	-	-1.03 ± 0.06
Glutamine	1.54 ± 0.03	1.73 ± 0.08	3.78 ± 0.02
Glycine	-	-	-1.00 ± 0.15
Serine	2.37 ± 0.05	3.61 ± 0.04	2.83 ± 0.01

properties of alanine and glycine fluxes. Interface defects were observed in the cross-section of the exposed Cu area without the coverage of solder alloy. The wetting appearance of serine flux shown in Fig. 3 (d) was better than alanine and glycine; however, the cross section image also

shows many interface defects and solder material dripping at the tip of the Cu coupon. Fig. 3 (b) shows the good wetting appearance using glutamine model flux, and the solder alloy homogeneously covered the Cu substrate without any defects.

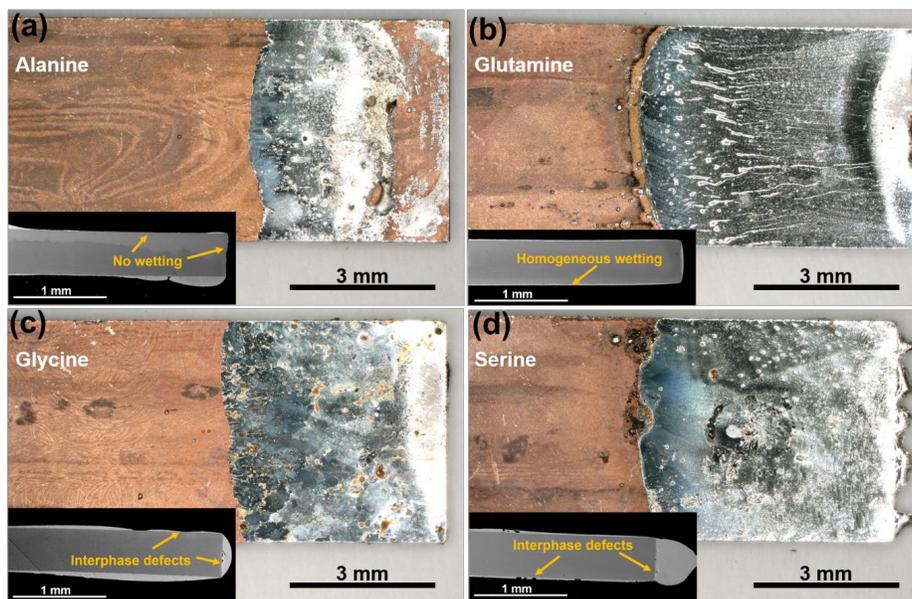


Fig. 3. LOM images of solder-wetted Cu coupons after testing using amino acids model fluxes (cross-sectional image is shown in inset): (a) alanine, (b) glutamine, (c) glycine, and (d) serine.

B. Humidity behavior of amino acids as activators

1) Water absorption behavior using gravimetric method

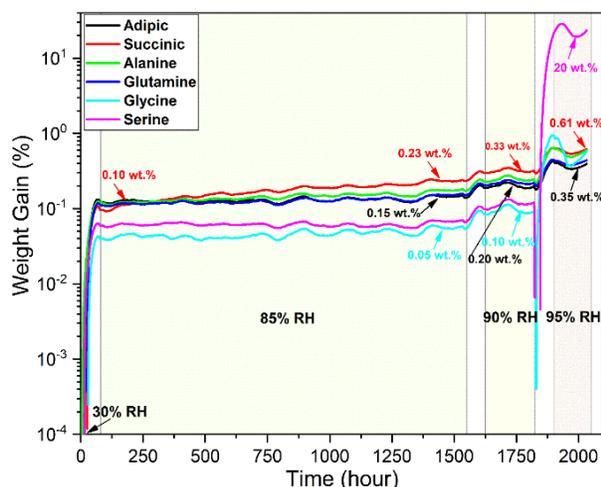


Fig. 4. Water absorption of the amino acids and WOAs under humidity exposure.

Fig. 4 shows the water weight gain of amino acids and WOAs under three stages of humidity exposure. A higher increase of weight gain was shown by succinic acid and alanine close to 85% RH, which indicates the lower DRH of succinic acid and alanine. The weight gain of glycine and serine maintained at lowest level of 0.1% under 90% RH exposure; however, the weight gain of serine suddenly increased to the highest level over 20.0% among all the tested samples when the humidity level increased to 95% RH. Water weight gain of adipic acid and glutamine increased from 0.15% to 0.35% as increasing RH level from 85% to 95%; however, the water absorption induced weight gain of adipic acid and

glutamine still maintained the lowest level among all test samples under humidity exposure of 95% RH.

- 2) Deliquescence behavior of amino acids candidate using single frequency EIS measurement

Fig. 5 shows the impedance response of the amino acid contaminated SIR test boards under various testing conditions. During the humidity ramping from 30% RH to 99% RH, the RH value

induced sudden drop of the impedance represents the DRH or water absorption behavior of amino acids, whereas RH triggered the abrupt increase of impedance over the ramping from 99% RH to 30 % RH represents ERH or water drying. Impedance response at high humidity exposure indicate the ionization behavior of amino acids. Generally, moisture hysteresis behavior of tested amino acids was observed, which indicates the lower ERH value compared to DRH value for all

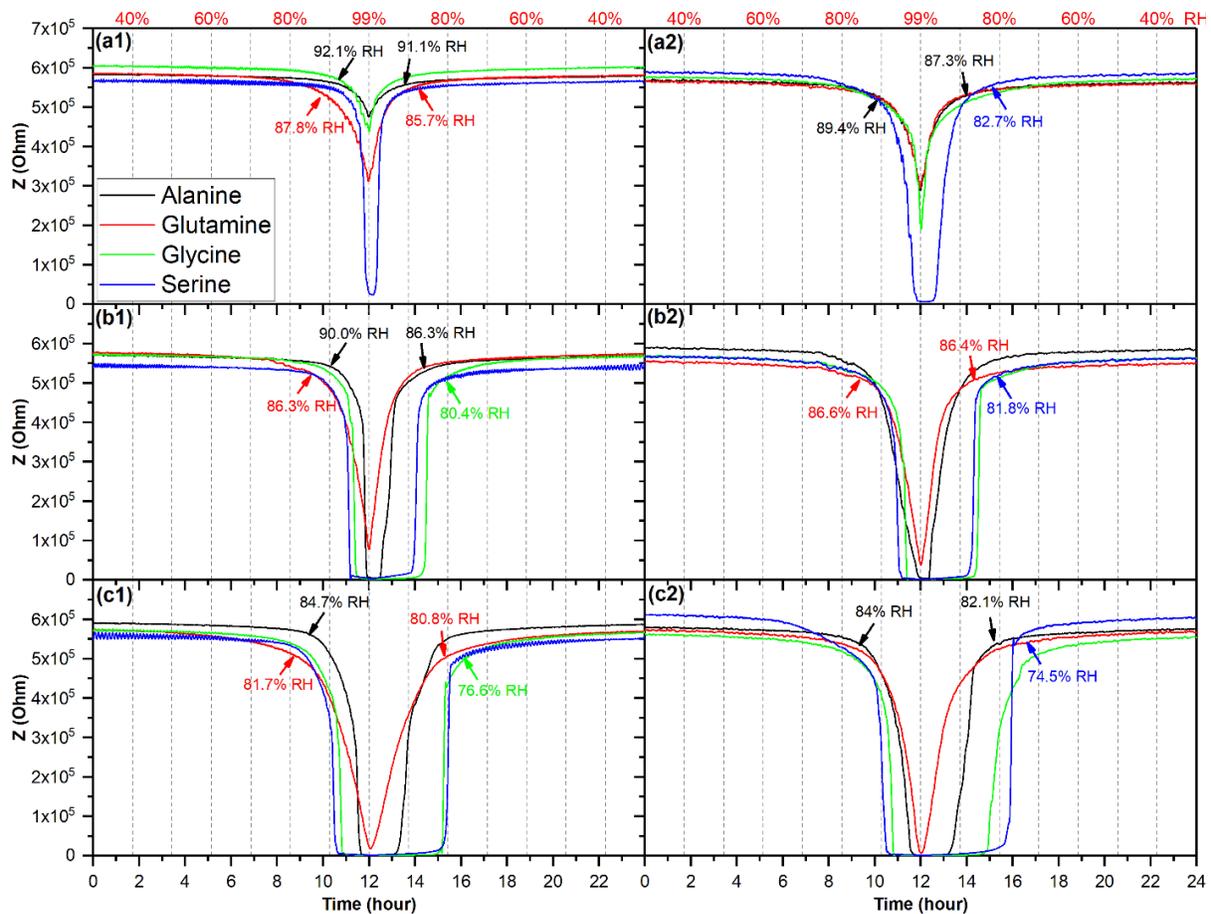


Fig. 5. Impedance response at 10 KHz of (1) non-thermal activated, (2) 240°C activated, amino acids contaminated SIR test boards under various testing temperature: (a) 25°C, (b) 40°C, (c) 60°C.

tested amino acids. The increased testing temperature triggered the decrease of the DRH and ERH level of amino acids, which is the highest for the 60°C. The reduction of the impedance response of amino acids is pronounced with increase in testing temperature. Regarding the influence of the thermal activation of the SIR prior to testing, the DRH and ERH level of thermal activated glutamine contaminated SIR pattern was slightly higher than non-thermal activated samples under all testing temperature, whereas the thermal

activation reduced the DRH and ERH levels for the other three amino acids.

Table 3 summarized the DRH value and ERH value from the EIS response at 10 kHz. In general, thermal activation at 240°C induced slight reduction of DRH and ERH value for alanine, glycine and serine samples, whereas the DRH and ERH value of glutamine slight increased after thermal activation. Under testing temperature of 25°C, the reduction of impedance response was observed serine contaminated SIR pattern at 92.1% RH as shown in Fig. 5 (a1), while the impedance

Table 3 Summary of DRH and ERH value for tested amino acid and representative WOA activators [6] at various testing conditions.

		DRH (% RH)					
Testing temperature		25°C		40°C		60°C	
Thermal activation		Non-heat	240°C	Non-heat	240°C	Non-heat	240°C
Alanine		92.1 ± 1.5	89.4 ± 1.2	90.0 ± 1.5	81.9 ± 1.5	84.7 ± 1.1	84.0 ± 1.8
Glutamine		87.8 ± 1.4	90.6 ± 1.5	86.3 ± 0.9	86.6 ± 0.7	81.7 ± 2.1	84.9 ± 3.5
Glycine		91.3 ± 1.8	91.1 ± 0.6	90.6 ± 0.9	88.0 ± 1.6	82.9 ± 1.3	81.7 ± 1.8
Serine		92.0 ± 0.6	88.7 ± 1.3	84.5 ± 2.9	83.1 ± 1.1	81.9 ± 1.1	76.3 ± 4.5
Adipic acid		97.4 ± 1.1		91.5 ± 0.8		83.2 ± 0.7	
Succinic acid		94.2 ± 0.4		91.3 ± 1.2		82.2 ± 1.5	
		ERH (% RH)					
Testing temperature		25°C		40°C		60°C	
Thermal activation		Non-heat	240°C	Non-heat	240°C	Non-heat	240°C
Alanine		91.1 ± 0.9	87.3 ± 1.4	86.3 ± 1.5	82.3 ± 0.5	78.7 ± 1.5	82.1 ± 1.6
Glutamine		85.7 ± 1.4	88.0 ± 0.8	87.8 ± 0.8	86.4 ± 0.7	80.8 ± 0.4	81.1 ± 2.4
Glycine		91.0 ± 0.4	89.8 ± 1.9	80.4 ± 1.9	82.6 ± 0.6	76.6 ± 0.8	70.9 ± 1.5
Serine		91.3 ± 1.2	82.7 ± 1.0	83.3 ± 1.3	81.8 ± 0.9	77.7 ± 1.5	74.5 ± 1.2
Adipic acid				81.1 ± 3.4		60.9 ± 1.4	
Succinic acid		91.3 ± 0.6		81.6 ± 2.6		61.7 ± 2.5	

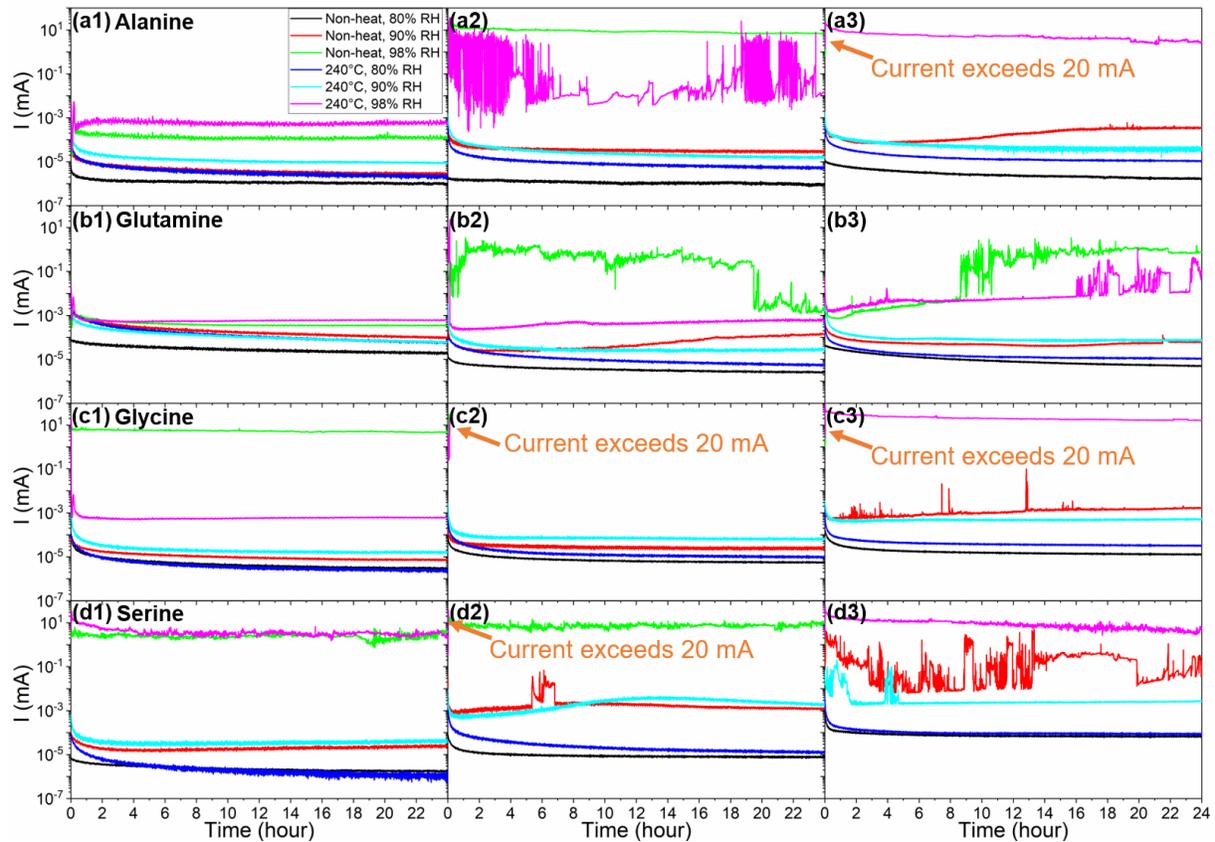


Fig. 6. Leakage current response of (a) alanine, (b) glutamine, (c) glycine, (d) serine contaminated SIR testing board at testing temperature of : (1) 25°C, (2) 40°C, (3) 60°C.

response of alanine, glycine and glutamine was maintained at relative high level under 99% RH exposure as shown in Fig. 5 (a1)(a2). Under the testing temperature of 40°C, DRH level of alanine dramatically decreased from 90% RH to 81.9% RH after thermal activation (Table 3), whereas less than 3% reduction of DRH values were obtained by glycine and serine. When the testing temperature increased to 60°C as shown in Fig. 5 (c1)(c2), the increase of the DRH level of glutamine was observed from 81.7% RH to

approximately 85% RH after thermal activation , which obtained highest DRH value after thermal activation. According to Fig. 5 and Table 3, the ranking of the corrosion reliability from the deliquescence and ionization point of view is: glutamine > alanine > glycine > serine.

3) Leakage currents for amino acids under humidity exposure

Fig. 6 shows the leakage current response of the activated and non-activated amino acids at three

different humidity levels near their DRH and at three testing temperature levels. An increase of leakage current was obtained by increasing the testing temperature for amino acids. At 25°C, the leakage current of alanine and glutamine contaminated SIR test board maintained below 1 μA at 98% RH (Fig. 6 (a1)(b1)), whereas high leakage current was obtained from the glycine

and serine contaminated SIR test boards at 98% RH (Fig. 6 (c1)(d1)). At 40°C, critical RH of serine decreased from 98% RH to 90% RH (Fig. 6 (d2)), whereas alanine, glutamine and glycine contaminated SIR testing board still maintained low leakage current below 1 μA at 90% RH (Fig. 6(a2)(b2)(c2)). At testing temperature of 60°C, the critical RH value of glycine and serine

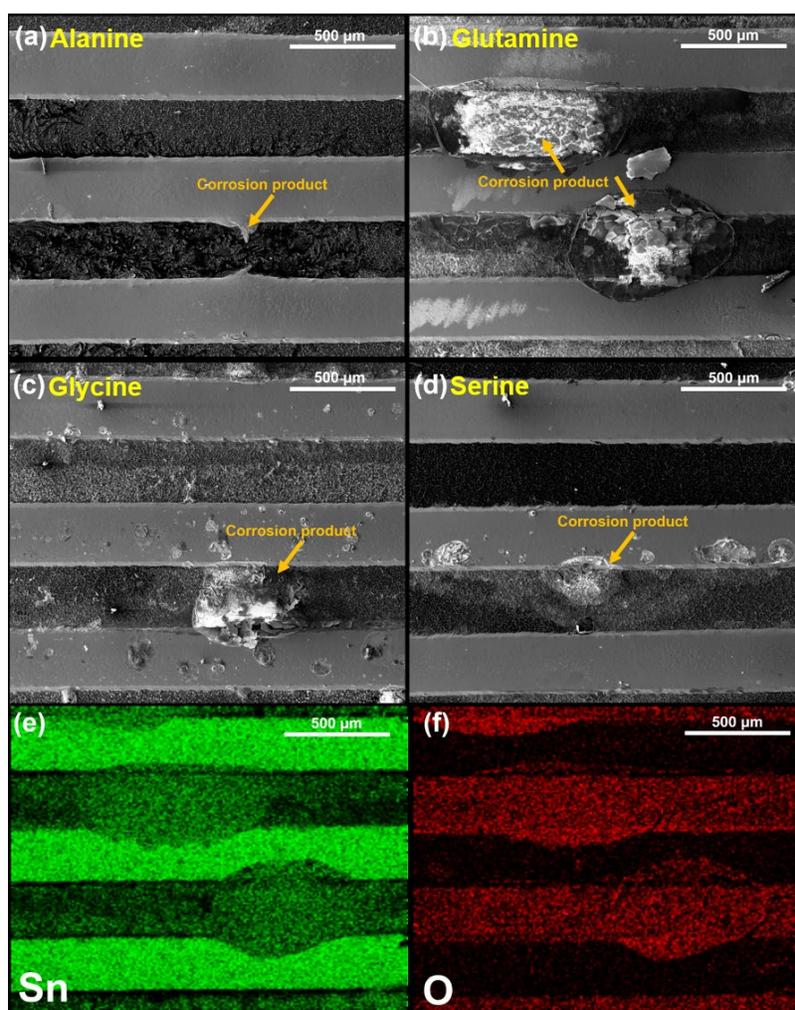


Fig.7. SEM images of non-thermal activated amino acids contaminated the SIR interdigitated pattern after CA testing at 40°C, 98% RH: (a) alanine, (b) glutamine, (c) glycine, (d) serine, and EDS mapping result of glutamine

declined to 90% RH as shown in Fig. 6 (c3)(d3). In many cases as shown in Fig. 6 (a3)(c2)(c3)(d2), leakage current exceeded 20 mA at the beginning of the bias loading and induced short circuit failures.

Fig. 7 presents the appearance of the amino acids contaminated SIR interdigitated pattern after leakage current measurement at 40°C, 98% RH. The plate like corrosion product was observed on glutamine, glycine and serine contaminated SIR pattern and deposited between two electrodes (Fig. 7 (b)(c)(d)). After CA testing at 40°C, 98% RH, the dendritic corrosion product was observed on the alanine contaminated SIR pattern as shown Fig. 7 (a). The EDS mapping result in Fig. 7 (e)(f) indicates the corrosion product was in consist of Sn and O element.

C. Evaluation of structural degradation of amino acids during thermal activation

Fig. 8 shows the influence of thermal activation temperature of 240°C on the degradation behavior of four amino acids. Fig. 8 (b) shows that the O-H peak at 3402 cm⁻¹ and four N-H stretch peaks in a range between 3170-3308 cm⁻¹ for glutamine disappeared after thermal

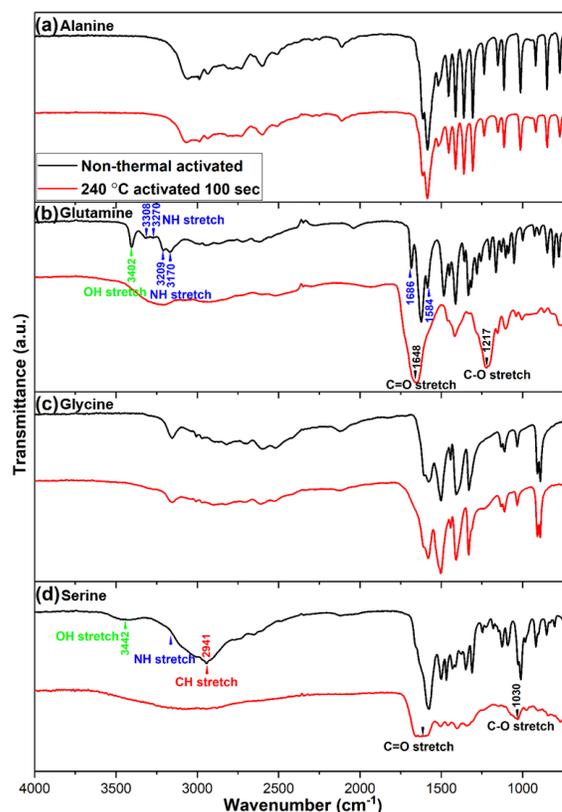


Fig. 8. FT-IR analysis of 4 tested amino acids after thermal activation at 240°C: (a) alanine, (b) glutamine, (c) glycine, (d) serine.

activation at 240°C, while the C=O peak at 1648 cm⁻¹ and C-O stretch peak at 1217 cm⁻¹ are still remained in the degradation product. Fig. 8(d) shows that O-H stretch peak, N-H stretch peaks, and C-H stretch peaks disappeared in serine after thermal activation, while presented C=O and C-O stretch peaks in the degradation product. The thermal activation did not influence the structure of alanine, glycine, as shown in Fig. 8 (a)(c).

IV. Discussion

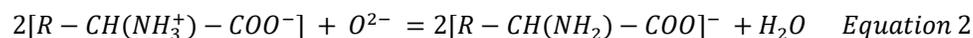
Present study investigated the solderability and corrosion reliability of four amino acid as candidates for solder flux activators. When dissolved in water, all of them has pH close to neutral value. The solderability of flux depends upon the activator type [35], melting temperature of flux activator [36], and flux formulation temperature [35]. The interesting character of amino acid as activator is that the oxide removal function of amino acids on copper can be obtained by both carboxyl group and amino group. The oxide removal action of carboxyl group is through complexation and disproportionation [16], whereas the oxide removal action of amino group is through redox reaction [23].

However, on the other hand, the corrosion reliability of the activator residue under humid conditions is dependent upon the hygroscopicity and ionization behavior when water layer is present. The hygroscopicity indicate the water absorption ability, which is determined by the DRH [6][37], while the water retention ability is indicated by ERH. The ionization behavior is related to the dissociation constant of the compound, which in terms of corrosion reliability

changes the conductivity of the water layer formed on the PCBA surface [14][37]. The results obtained in this work suggest good solderability and corrosion reliability using glutamine as flux activator. Even though the corrosion robustness of glycine and alanine was confirmed by single frequency EIS and CA techniques, the solderability performance of these two candidates is inferior to the commercial used WOAs. The differences between DRH and ERH level of amino acid candidates indicate the hysteresis from water uptake and release, which is similar to commercially used WOAs [6]. After thermal activation, the reduction of the DRH and ERH value for alanine, glycine and serine could be due to the degradation of the carboxyl group, which induced higher hygroscopic anhydride species [5].

A. Solderability assessment of amino acids and the commercially used WOA flux activators

The solderability of amino acid depends on the activity of the carboxyl group and amino group at soldering temperature unlike for WOAs, which contain only carboxyl groups. It is known that the



pK_{a1} value cannot interpret the activities of carboxyl group in amino acids and WOAs under soldering condition since the pK_{a1} value only represent the activities of carboxyl group in water. However, it is noticed that the wetting force of succinic acid is higher than adipic acid as shown in Fig. 2, which indicate the activity of two carboxyl groups in succinic acid is higher than adipic acid under soldering temperature of 270°C. This agreed with the lower pK_{a1} value for succinic acid (4.21) compared to pK_{a1} value for adipic acid (4.44) [6]. Amino acids used in the present investigation contain one carboxyl group and one amino group. The ranking of wetting force for amino acid agreed with the ranking of pK_{a1} value as shown in Table 1, where the pK_{a1} value of glutamine is the highest, followed by serine, glycine and alanine from high to low. Based on this finding, the pK_{a1} value might be used as a screening factor for identifying the solderability for the selection of flux activators from the same chemical category.

The effect of melting temperature also influence the wetting force of flux activators used the in

present work. Flux activators with low melting temperature provide a liquid barrier between metal and atmosphere to avoid re-oxidation. For instance, the melting temperatures of glutamine, succinic acid, and adipic acid are 185°C, 185°C, and 151.5°C respectively [2][38], which provide relative good solderability compared to the other three tested amino acids with higher melting temperatures as shown in Fig. 3(a)(c)(d). From the results of wetting force in Fig. 2, it can be seen that the flux activators with melting temperature below 185°C is able to protect the Cu surface from re-oxidation and maintain wettability at the soldering temperature of 270°C, whereas the glycine flux activators with melting temperature beyond 233°C cannot maintain the solderability. Additionally, the wetting force obtained by serine flux is higher than the glycine although the melting temperature of serine (246°C) is higher than glycine (233°C) as shown in Fig. 2. However, the pK_{a1} value of serine (2.21) is lower than glycine (2.34), which indicate the stronger acidity of serine as agreement with the result of higher maximum wetting force. It indicates that the melting temperature is not the only the important

factor affecting the solderability, but also the activity of the carboxyl group. Amino group in glutamine and glycine degrade under temperature of 240°C shown in Fig. 8 (b)(d), which resulted in redox reaction with Cu oxide [23]. The higher maximum wetting force obtained by glutamine could be attributed to the higher amount of zwitterions since glutamine is the only tested candidate which possesses two amino group in chemical structure. The zwitterions resulted in a more reactive positive charge group of $-NH_3^+$ when applied on metal oxides, which is more effective in oxide removal and ready for the oxides reaction in most of time during soldering process [26], as shown in the Equation 2.

B. Humidity interaction assessment of amino acids and the commercial used WOA flux activators

Humidity interaction with flux activators plays the key role in the corrosion related failure of electronics when they presented as flux residues. Water absorption behavior of various WOA activators were investigated using single frequency EIS method [6][3] and gravimetric method [18]. In Table 3, results from previous

EIS investigations reported the DRH values of succinic acid are 94.2% RH, 91.3% RH, 82.2% RH at 25°C, 40°C, 60°C respectively [6]. While for adipic acid, the DRH values are 97.4% RH, 91.5% RH, and 83.2% RH at 25°C, 40°C, 60°C respectively [6]. Compared to the summarized DRH value in Table 3, the DRH level of tested amino acids was lower compared to adipic acid and succinic acid, whereas the ERH levels of tested amino acids were slightly higher than succinic acid under all testing temperatures. Even though the DRH level of glutamine (86.3% RH at 25°C, 86.6 at 40°C) is lower than adipic acid and succinic acid, the glutamine obtained higher DRH value compared to adipic acid and succinic under testing temperature of 60°C.

The thermal activation effect on serine shows the reduction of DRH and ERH level, which is similar to the thermal activation effect on commercial used WOA flux activators. For commercially used WOA activators, the thermal effect on the reduction of the DRH and ERH value is due to the formation of the anhydride-like degradation products with higher affinity to water absorption [5]. While for serine, the influence of

the thermal degradation product could be the reason induced higher hygroscopicity. Fig. 8 (d) shows a wide peak of C=O stretch in a range of 1600-1700 cm^{-1} and C-O stretch peak, which could be from the anhydride. Yablokov et al. reported thermal decomposition of serine took place at 30-50°C below melting temperature and induced degradation product such as short-chain WOAs and short-chain amides [39], which obtained the deliquescent properties [40]. In contrast, the thermal degradation product of glutamine possessed lower hygroscopic behavior than glutamine, this could be due to the relative larger size and length of the side chains in the degradation product [6][41]. For alanine and glycine, stable molecule structure as shown in Fig. 8 (a)(c) resulted the minimal change of DRH and ERH after thermal activation.

In the present study, the gravimetric results of water absorption for tested WOAs and amino acids shown in Fig. 4 agreed with the results of single frequency EIS testing as shown in Table 3. The highest water gain rate was shown by succinic acid at 85-90% RH, whereas the water gain rate of serine surpassed the succinic acid at

95% RH. Nonetheless, EIS testing and gravimetric testing of flux activators revealed humidity interaction from different perspectives. For instance, DRH level obtained from EIS testing shows the sudden effect of the water absorption under humidity exposure, which is 94.2% RH for succinic acid at 25°C [6]. However, gravimetric result in Fig. 4 demonstrates the water absorption behavior of succinic acid started at a much lower RH range ($\leq 85\%$ RH) than DRH with a low water absorption rate. This gravimetric testing confirmed the effect of the flux residue on water condensation layer, but the amount of water also depends on the kinetics under constant humidity exposure. Therefore, adipic acid and glutamine obtained the best humidity robustness due to their lowest water gain rate among all tested activators based on constant humidity exposure.

C. Influence of amino acid activators on leakage current under DC load

The threshold value of leakage current in this study was determined as 1 μA according to the previous DC investigation on WOA based wave flux contaminated SIR interdigital pattern [3][42].

It is noticed that the leakage current value of glutamine contaminated SIR pattern under 98% RH reduced after thermal activation as shown in Fig. 6 (b2)(b3), while the thermal activation of 240°C did not influence the leakage current of alanine, glycine and serine contaminated SIR pattern under humidity exposure. Weiss et al. reported that the degradation of glutamine took place at 185°C and form H₂O and NH₃ [43], while the thermal degradation temperature of alanine, glycine, and serine was 309°C, 240°C, and 222°C respectively [39]. Therefore, the reduction of the leakage current response of glutamine could be due to less ionic residue presented on the SIR pattern. However, the effect of testing temperature on the leakage current values is more pronounced for alanine, glycine and serine. This could be due to the increased temperature, which enhances of the mobility of the water molecules and induces the formation of thicker water layer on PCBA surface [3]. As result, the thicker water layer raises the solubility of the amino acid residue on the SIR pattern and triggers higher leakage current [6].

The hygroscopic and ionic nature of flux activator candidates played a key role on the leakage current values of SIR pattern under DC load [6][42]. The leakage current measurements comply with the water absorption results as shown in Fig. 4. Water absorption of serine is the highest (> 1.0 wt. %) in all the tested candidates under 95% RH, 25°C. The highest water absorption triggered the ionization of the serine on the SIR pattern and lead to the highest reduction of the impedance as shown in Fig. 5 (a1)(a2) and highest leakage current (Fig. 6 (d1)) under same climatic condition. The critical RH level indicates the RH level induced leakage current exceeding the threshold leakage current of 1 μA. Piotrowska et al. reported the critical RH values of adipic acid and succinic acid are 98% RH and 95% RH under 60°C [6]. In the present work, the crucial RH of serine induced higher leakage current decreased from 98% to 90% RH as the testing temperature increased from 25°C to 60°C. Coinciding with this, the water weight gain values of serine and glycine are much higher compared to succinic acid and adipic acid obtained under high humidity exposure as shown in Fig. 4. On the contrary, the water weight gain

of glutamine is the lowest (0.35 wt.%) in all tested flux activators under 25°C as shown in Fig. 4, and possessed the lowest leakage current as shown in Fig. 6 (b1). Water absorption results confirmed that the glutamine absorbed similar water level as adipic acid, and alanine absorbed similar water level as succinic acid (Fig. 4). This is in agreement the low leakage current values of glutamine and alanine under exposure of 98% RH at 25°C and 40°C (Fig. 6 (b1-3)(a1-3)). Piotrowska et al. [6] reported the leakage current values of 100 $\mu\text{g}/\text{cm}^2$ adipic acid and succinic acid contaminated SIR pattern were below 1 μA at 99% RH under testing temperature of 25°C, whereas exceeded 1 μA at 98% RH under testing temperature of 40°C and 60°C. The alanine or glutamine contaminated SIR pattern tested in present investigation also demonstrates the occurrence of high leakage current failure at 98% RH under 40°C and 60°C, as shown in Fig. 6 (a2-3)(b2-3). However, Fig. 6 (b2) shows the leakage current level of 240°C activated glutamine contaminated SIR pattern was below 1 μA at 98% RH/40°C, which indicates the better humidity robustness of glutamine as flux activator in comparison with adipic acid and succinic acid.

The occurrence of deteriorative leakage current was due to the conductive corrosion product formation, which bridged the adjacent oppositely biased electrodes under DC load [44]. The morphology of the corrosion product in Fig. 7(b) confirmed that the corrosion product induced by glutamine is not like the ECM dendrites with structures of main-branches and side-branches [45][46]. Additionally, ECM dendrites are in the form of metal or in combination with hydroxide [44][46] agreed with the EDS mapping result as shown in Fig. 7 (e)(f).

V. Conclusions

1. Solderability testing results shows that the wetting force of glutamine model flux is located between the wetting forces of commercial used adipic acid and succinic acid model fluxes, and homogeneous wetting appearance was obtained after using glutamine model flux under wave soldering temperature of 270°C. However, the wetting forces of alanine, glycine and serine model fluxes are much lower than adipic acid model flux.

2. Water absorption test shows that the water weight gain rate of glutamine and adipic acid are the lowest in all 6 tested flux activator candidates under constant humidity exposure at 25°C, 95% RH, whereas water weight gain rate of serine and glycine are much higher than succinic acid. However, the water weight gain rate of serine and glycine are the lowest when exposed under humidity level below 90% RH at 25°C.

3. EIS results indicates the DRH and ERH levels of 240°C activated glutamine are the highest in all tested amino acid candidates under same testing temperature, and glutamine contaminated SIR pattern possessed highest impedance response under 99% RH humidity exposure. Thermal activation decreased the DRH and ERH level of alanine, glycine and serine in a rate of 1-5%, whereas an improvement of DRH and ERH level has been found on glutamine after thermal activation. Increased testing temperature decreased the DRH and ERH levels of all tested amino acids.

4. A significant reduction of the leakage current has been observed on the glutamine contaminated after 240°C thermal activation when exposed

under 98% RH at 40°C and 60°C. Critical RH value of serine contaminated SIR pattern decreased from 99% RH to 90% RH after increasing test temperature from 25°C to 60°C. Higher baseline of leakage current of serine and glycine was obtained as increasing test temperature, whereas the leakage current induced by glutamine and glycine remained at the same level under exposed humidity level below 90% RH.

5. Compared to adipic acid and succinic acid used in current commercial flux, the higher DRH value at 60°C and higher wetting force of glutamine potentially suggested better reliability performance of surface mounting device in higher temperature environment.

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