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Corrosion failure analysis of printed circuit boards exposed to H_2S-containing humid environments

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Abstract

The corrosion failure of a printed circuit board (PCB) with electroless nickel/immersion gold (ENIG) surface finish in a hydrogen sulfide-containing humid environment was analyzed in this work. To establish a comprehensive mechanism for the damage, the exposed surfaces were characterized by visual inspection, scanning electron microscopy/energy-dispersive X-ray spectroscopy and X-ray photoelectron spectroscopy. It was realized that merely copper traces under the edge of soldermasks (on electrical junctions) suffer a galvanic-type corrosion reaction with hydrogen sulfide and moisture adsorbed, forming dominantly copper sulfides and a small amount of copper sulfate and oxide. The creep of the corrosion products on the surfaces of ENIG-plated layers, tin-based solders and adjacent soldermasked areas was also found to be responsible for creating short circuits on the outer layers of the miniaturized PCB.

Keywords: Failure analysis; Corrosion; Copper sulfide; X-ray photoelectron spectroscopy (XPS)

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1. Introduction

A printed circuit board (PCB) is a collective array of electrical circuits assembled on generally a fiberglass-reinforced epoxy-polymer panel. PCBs contain various electrical components including resistors, capacitors, transistors and diodes which are connected to each other by copper traces with tin-based soldered junctions. In multilayered PCBs, the electrical connection is established by copper vias (through holes). To ensure corrosion protection, the copper traces lying on the PCBs are covered by a thin lacquer-like layer of epoxy (named as soldermask), whereas there are different surface finishing processes to protect the copper vias [1]. Moreover, the surface finish coatings facilitate subsequent soldering processes, through preventing the formation of surface layers, even passive films (copper oxides). The most commonly used surface finishing coatings include organic solderability preservative, immersion silver, immersion tin, electroless nickel/immersion gold, electroless nickel/electroless palladium/immersion gold and hot air solder level [2, 3]. Among them, electroless nickel/immersion gold (ENIG) surface finish coatings comprise a multi-micron layer of Ni-P and a thin layer of Au on copper connections, which are developed to improve the junction's conduction, solderability and, partly, corrosion resistance.

Corrosion, defined as the environmental degradation of materials, is currently a challenging issue in electronics. From this perspective, the tendency to miniaturization, the variety of the materials used and, less importantly, the presence of electrical potential gradients all accelerate the corrosion damage, leading to the early failure of PCBs via creating short and open circuits [4]. The corrosion rate in electronics is contorted by the PCB's and service-environment's characteristics, as categorized into process- and service-

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related types, respectively. Anyway, the electronics corrosion is atmospheric-type in the forms of gas-phase corrosion, cathodic corrosion, galvanic corrosion, fretting corrosion and stray current corrosion [2, 5-10]. In this work, the corrosion failure of a PCB with ENIG surface finish was analyzed by visual, microscopic and spectroscopic evaluations.

2. Experimental procedure

2.1. Environmental conditions

An ENIG-plated PCB containing copper traces covered by an epoxy-based soldermask and tin-based solders (Figure 1) was serving in a H_2S-containing humid environment at 15-25 °C. The concentration of the H_2S gas in the atmosphere was in the range of 30-50 ppb, which is ranked as a moderately corrosive atmosphere of G2 according to ISA Standard: 71.04 [11]. Also, the environmental humidity was in the range of 50-70 % with a chloride concentration below 4 ppb.

2.2. Characterization of corrosion products

After about 6 months of service in the above-described atmosphere, the PCB failed with some typical presentations of dark gray corrosion products on different regions, including in the vicinity of ENIG-plated through holes (Figure 2a) and Sn-based solders (Figure 2b), as shown by red arrows.

The structure of the aforementioned corroded zones was analyzed by scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) and X-ray photoelectron spectroscopy (XPS). The former was used to study the morphological and compositional characteristics of the corrosion products, whereas the latter was employed to identify the bonds and compounds formed in the corrosion products. Afterwards, considering

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the PCB configuration and the characterized corrosion products, a macro/micro mechanism was established for the occurring corrosion reaction.

3. Results and discussion

3.1. Corrosion analysis around the through holes

The SEM micrographs and EDS pattern taken of the corrosion products accumulating around a through hole (via), like that shown in Figure 2a, are presented in Figure 3. According to the low-magnification micrograph (Figure 3a), the corrosion products have covered a significant area of the ENIG-plated surface and the adjacent soldermasked regions, albeit with a biased centralization towards the junction of the via/copper traces. Typically, there are projective accumulations on the edge of the soldermask (in other words, on the junction), as indicated by arrows in this micrograph. The non-dense appearance of the corrosion products comprising nanometer-to-micron sized particles (Figure 3b) suggests that the formed corrosion film is not a passive film which generally presents an integrated feature, although the nature of the metals involved in this zone (including Cu, Au and Ni) is active-passive albeit in theory. The absence of cracks in the corrosion products denies considerable fluctuations in humidity and temperature during service [12], as noted in the experimental section.

The EDS pattern of the corrosion products around the via is depicted in Figure 3c, showing the existence of Cu, S, O and C, which is indicative of the corrosion reaction of the environment constitutes and copper traces. The related quantitative results are also listed in Table 1. On the one hand, the absence of Au and Ni as the principal components of the ENIG coating in the corrosion products, and on the other hand, the coverage of the ENIG coating by the corrosion products infer the characteristic creeping of the corrosion products on the

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surfaces of the ENIG-plated through holes and epoxy-based soldermask. Furthermore, considering the projections created by the fountain of the Cu-based corrosion products (the arrows in Figure 3a), the degradation reaction is attributed to a galvanic-type corrosion between the active copper traces at the bottom of the soldermask edge as the anode and the noble ENIG coating as the cathode. This assignment is justified on the fact that galvanic attacks generally are maximum around junctions [13], creating the projections. Also, this is in good agreement with the fact that Au is more noble than Cu in different environments.

Because of the limitations of EDS on both quantitative and qualitative chemical analyses, a corroded area similar to Figure 2a was also analyzed by XPS to identify the bonds and compounds constituting the corrosion products. Note that they can be copper sulfide, sulfate, carbonate and/or oxide, based on the EDS analysis (Figure 3c). Figure 4 shows the total survey of the XPS spectra and the de-convoluted XPS spectra of the Cu 2p, S 2p, O 1s, Au 4f, C 1s and Cl 2p orbitals of the exposed surface. In the Cu 2p spectrum, two peaks at 932.5 and 952.5 eV are recognized, whereas the asymmetric characteristic of the first peak is indicative of the possible existence of weak peaks at 935.5, 934.5 and/or 933.5 eV. A strong peak at 161.8 eV is also detected in the S 2p spectrum. Considering the XPS spectra of the Cu 2p and S 2p orbitals together, the peak of S 2p at 161.8 eV is attributed to Cu_2S or CuS (or Cu_xS in general) with a dominant level. In parallel, the peaks of Cu 2p 1/2 at 932.5 and 935.5 eV belong to cooper sulfide and sulfate, respectively. In addition, the peak of Cu 2p 3/2 at 952.5 eV is attributed to Cu^{2+} in sulfate. The above-mentioned assignments are in good agreement with Refs. [12, 14, 15]. In the O 1s and C 1s spectra, the peaks of 532 and 284.8 eV are detected, which are assigned to the C-C and C-O bonds of contamination adsorbed, respectively. The peak at 530.4 eV in the O 1s spectrum is also attributed to the lattice oxygen component (O^{2-}) in Cu_2O [16, 17]. Also, in the Au 4f spectrum, two peaks detected at

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84 and 87 eV belong to elemental Au, suggesting the lack of corrosion for this noble element. This is compatible with the EDS analysis done on the related corrosion products. The absence of any peaks in the energy range of chloride verifies the resistance of copper against this corrosive species. Accordingly, the possible presence of peaks at 933.5 and 934.5 eV in the Cu 2p spectrum, which are related to CuO and $CuCl_2$, is denied by the absence of the related peaks in the O 1s and Cl 2p, respectively. In conclusion, the XPS analysis (Figure 4) indicated that the corrosion products are merely composed of a considerable amount of copper sulfide and a low level of copper sulfate and oxide, which is agreement with the EDS analysis (Figure 3c).

3.2. Corrosion analysis around the solders

Figure 5 demonstrates the SEM micrographs and EDS pattern of the corrosion products around a solder like Figure 2b. As can be seen in Figure 5a, the corrosion products have creepingly covered the soldermasked copper trace connecting the soldered components of A and B. A similar non-corroded copper trace is depicted by a yellow arrow in Figure 2b. Concerning the solder, the accumulation of the corrosion products near the junction is evident, as shown by an arrow in Figure 5a. Also, the corrosion products present a non-passive particulate feature (Figure 5b), like Figure 3b. The EDS analysis (Figure 5c and Table 2) also shows the corrosion products contain C, S, O and C, similar to the attacked region illustrated in Figure 3. The absence of Sn in the corrosion products and the localization of corrosion attack on the junction (the arrow of Figure 5a) point out the occurrence of a galvanic corrosion between copper and tin with the creeping corrosion products which spout from the edge of the soldermask on the junction.

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Despite the fact that the electrochemical activity of Sn is higher than Cu based on the electromotive force (EMF) series, Cu played the role of the anode in this study. This apparent antithesis is justified by the fact that Sn, in contrast to Cu, nearly experiences an activation polarization event in sulfur-containing environments [18, 19], thereby lowering its corrosion activity and making it act as the cathode when connected to Cu. Indeed, metals in the EMF series have been ranked based on their reversible potential in the standard half-cell circumstance, which is not necessarily valid for other environments including sulfur-containing ones. Similarly, the XPS analysis confirmed that the corrosion products accumulating near the solder mainly consist of copper sulfide, sulfate and oxide, so that the presence of tin, chlorides and carbonates is denied, verifying the EDS analysis.

3.3. Establishment of the corrosion failure mechanism

According to the EDS and XPS analyses conducted on the corrosion products, the corrosion attack was merely assigned to Cu, not to the other metals involved in the PCB (Au, Ni and Sn). Albeit the galvanic couples of Cu with these noble metals were critical to the corrosion event. Also, the creeping corrosion products were composed of dominantly copper sulfide (CuS, Cu_2S or/and Cu_xS) and a little amount of copper sulfate ($CuSO_4$) and copper oxide (Cu_2O). To establish a comprehensive mechanism for the damage of the PCB, the below items are followed:

3.3.1. Formation of the corrosion products

Due to the humidity of the service atmosphere, which was in the range of 50-70 % (as noted in the experimental section), a water electrolytic layer is deposited on the PCB's

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surface. A number of environmental H_2S molecules are dissolved in the adsorbed water film and ionized in this neutral medium, based on Eq. 1:

$$H_2S \rightarrow H^+ + HS^- \quad (1)$$

The release of H^+ and thereby the acidification of the solution encourage the ionization of HS^-, as follows:

$$HS^- \rightarrow H^+ + S^{2-} \quad (2)$$

By further increasing the concentration of H^+ and thereby decreasing pH, the electrochemical corrosion of Cu happens through the below anodic and cathode reactions:

$$Cu \rightarrow Cu^{2+} + 2e^- \quad (3)$$

$$2H^+ + 2e^- \rightarrow H_2 \quad (4)$$

The anodic reaction occurs on the Cu surface, while the cathodic reaction happens on the Au, Ni or Sn surfaces. The overall redox reaction is obtained by adding Eqs. 3 and 4:

$$Cu + 2H^+ \rightarrow Cu^{2+} + H_2 \quad (5)$$

Subsequently, the Cu^{2+} ions produced by Eq. 5 and S^{2-} created by Eq. 2 react to form copper sulfide as the major corrosion product, as follows:

$$Cu^{2+} + S^{2-} \rightarrow CuS \quad (6)$$

It is noteworthy that at the low concentrations of H_2S (typically below a few ppm), Cu_2S or generally Cu_xS are formed instead of CuS [5]. Because of the porous nature of the Cu_xS film (as demonstrated in Figures 3b and 5b), the bottom layers of Cu are continuously corroded via the above mechanism.

The formation of copper sulfate can be attributed to the possible presence of SO_2 traces in the environment. In this regard, an amount of sulfuric acid can be created in the adsorbed layer via the below reaction:

$$SO_2 + O_2 + H_2O \rightarrow H_2SO_4 \quad (7)$$

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The produced sulfuric acid is dissolved and ionized in water, as follows:

$$H_2SO_4 \rightarrow 2H^+ + SO_4^{2-} \quad (8)$$

The anodic, cathodic and total reactions are again established based on Eqs. 3, 4 and 5, respectively. Finally, Cu^{2+} produced by the anodic reaction reacts with SO_4^{2-} created by Eq. 8, forming copper sulfate as the minor corrosion product:

$$Cu^{2+} + SO_4^{2-} \rightarrow CuSO_4 \quad (9)$$

It is noteworthy that in the absence of sulfur-containing gases, the below cathodic reaction occurs instead of Eq. 3:

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \quad (10)$$

For this case, the overall reaction is written as below:

$$2Cu + O_2 + 2H_2O \rightarrow 2Cu(OH)_2 \quad (11)$$

The produced copper hydroxide species is unstable and decomposed into Cu_2O:

$$2Cu(OH)_2 \rightarrow Cu_2O + H_2O \quad (12)$$

The produced Cu_2O layer, in contrast to the above-described porous copper sulfide and sulfate films, has a passive nature and protects the bottom layers of Cu against corrosion in sulfur-free environments [20]. Eq. 12 also explains the formation of copper oxide detected in the XPS analysis. The passive oxide layer would be reduced followed by the continuation of the corrosion damage of the underlying layers, after a possible next exposure to a sulfur-containing environment [7].

3.3.2. Role of the environmental conditions

Considering the formation reactions of the corrosion products described above, it is straightforwardly found that water and H_2S existing in the service atmosphere are responsible for the corrosion attack, without any contribution of chloride (Cl^-) based on XPS. In regard to

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water, it is known that a humidity level of at least 40 % is enough to form a water layer adsorbed on soldermasked PCBs [7], which was satisfied for the PCB studied in this work with an exposure of 50-70 % humidity. On the other hand, PCBs are designed to present a corrosion resistance against at most 4 ppb of H_2S [9], which is in good agreement with the fact that the H_2S value in this work was enough to proceed a corrosion damage and failure.

3.3.3. Role of the PCB's configuration

The corrosion products localized on the junctions originated from a galvanic-type corrosion, where the electrical connection of Cu with Au, Ni and Sn had a critical contribution to the event. As schematically illustrated in Figure 6, the non-overlapped edge of the soldermask allows for the access of the adsorbed electrolytic layer to the underlying Cu traces, forming galvanic cells (Figure 6). It is regarded as one of the main challenges of the soldermask technology from the corrosion viewpoint [7]. Also, the low area of the exposed copper at the soldermask edge provides a high ratio of the cathodic to anodic areas and thereby a great electrochemical current density crossing the anode, which accelerates the bimetallic corrosion attack. Moreover, the dominant tendency of the corrosion products towards creeping on the surface of the miniaturized PCB creates short circuits like Figure 2a, which is responsible for the PCB failure, along with open circuits formed due to the corrosion loss of the conductive species. The semi-conductive properties of these corrosion products disadvantageously offer the potential to create both short and open circuits. In this regard, it is known that a loss of 10^{-12} grams is enough to disturb the performance of PCBs [2]. Several approaches, such as using suitable conformal coatings and vapor-phase inhibitors, are suggested to control this type of corrosion.

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4. Conclusion

1) Only the copper components of the PCB suffered from the corrosion damage with a tendency towards creeping on the PCB surface, while no evidence for the degradation of Au, Ni, and Sn was found.

2) The corrosion products presented a biased accumulation on the electrical junctions of the corroded/noncorroded species, suggesting a galvanic-type corrosion attack.

3) Dominantly copper sulfides and a little copper sulfate and oxide were the principal constitutes of the corrosion products.

4) The non-overlapped edges of the soldermask coating provided a path for the access of the adsorbed electrolyte to naked copper, giving rise to the electrochemical corrosion.

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Figures

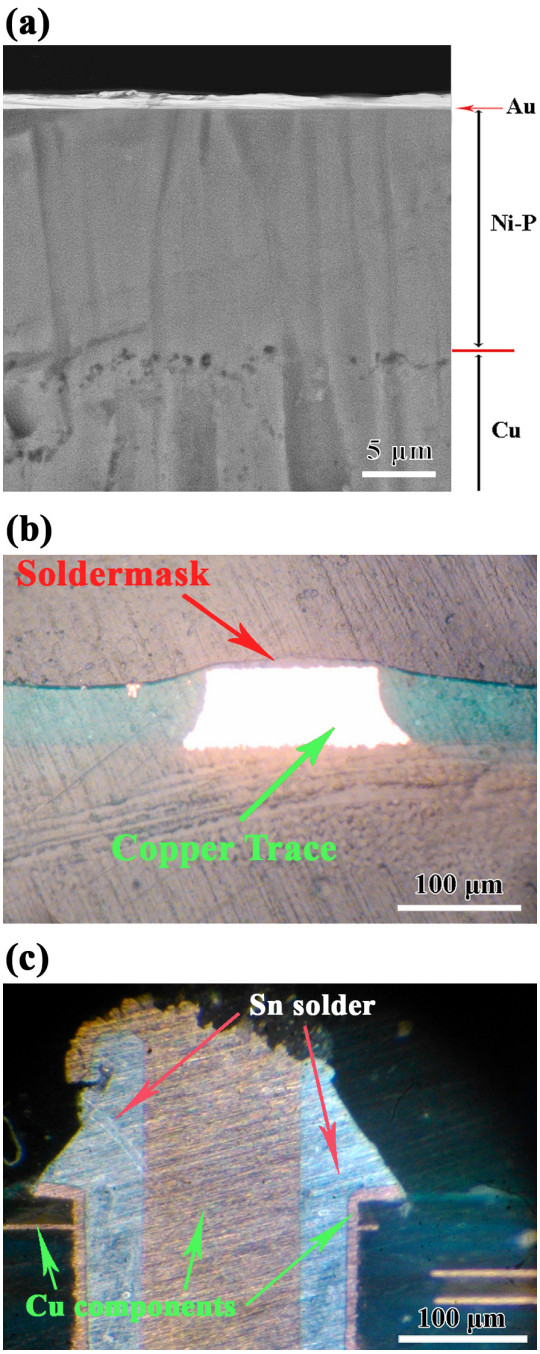

Figure 1. Cross-sectional micrographs of the ENIG-coated copper component (a), a copper trace with a soldermask coating (b) and a solder area connected to a copper trace (c).

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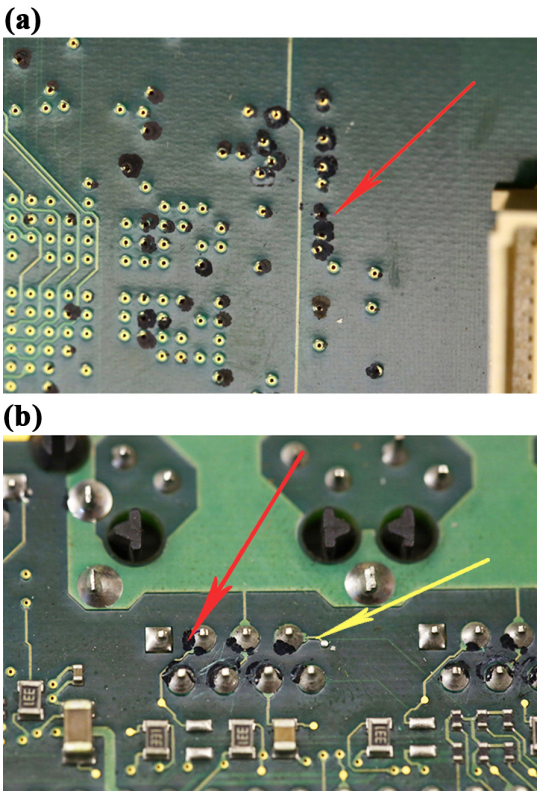

Figure 2. Macrographs of the PCB's surface after corrosion exposure, in the vicinity of ENIG-plated vias (a) and solders (b).

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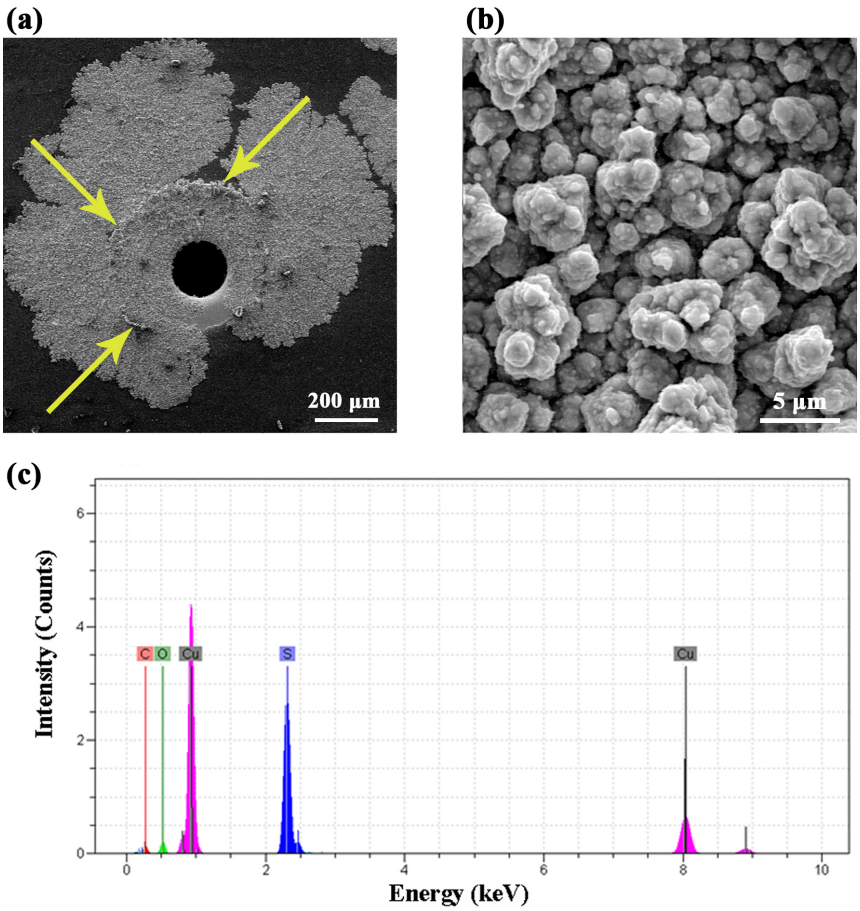

Figure 3. SEM micrographs in two magnifications (a, b) and EDS spectrum (c) of the corrosion products on a via.

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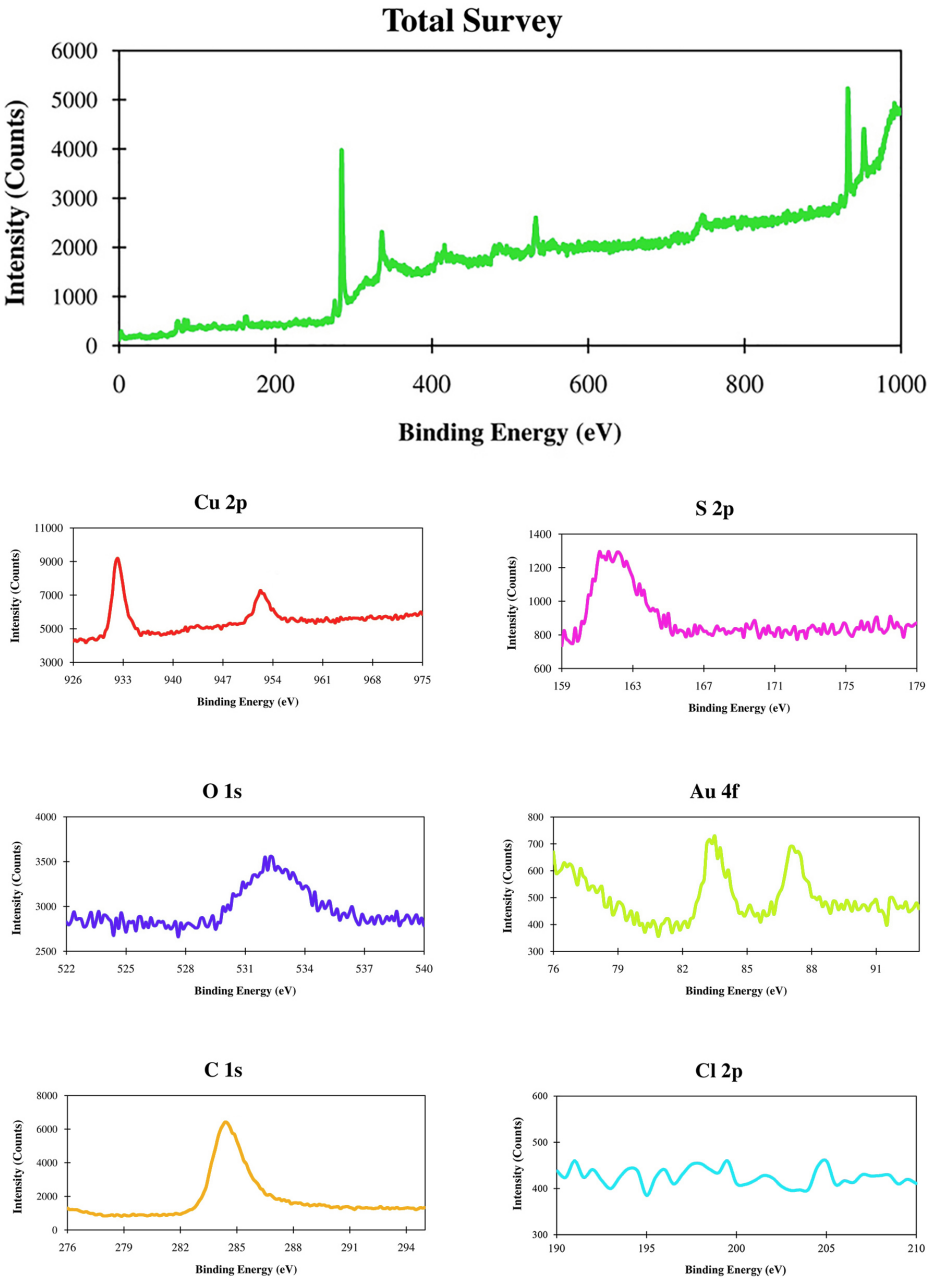

Figure 4. XPS spectra taken of an exposure via, corresponding to Figure 2a and Figure 3.

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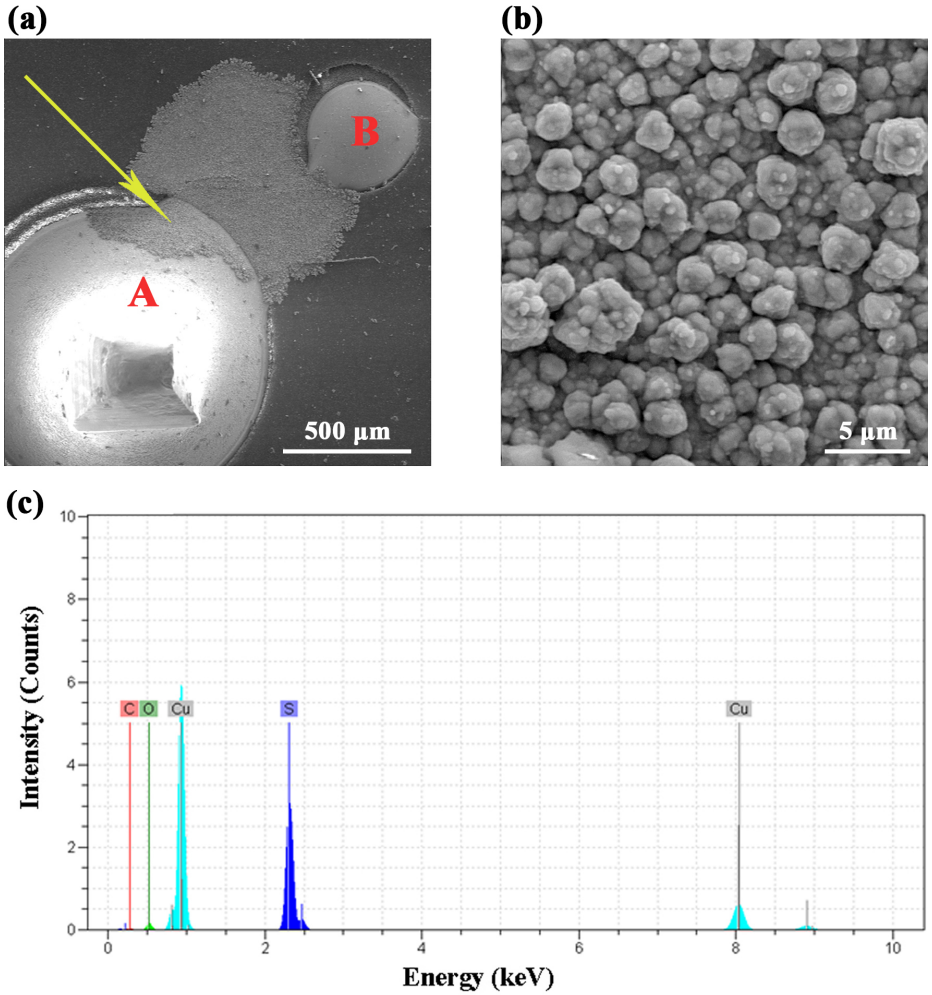

Figure 5. SEM micrographs in two magnifications (a, b) and EDS spectrum (c) of the corrosion products around a solder.

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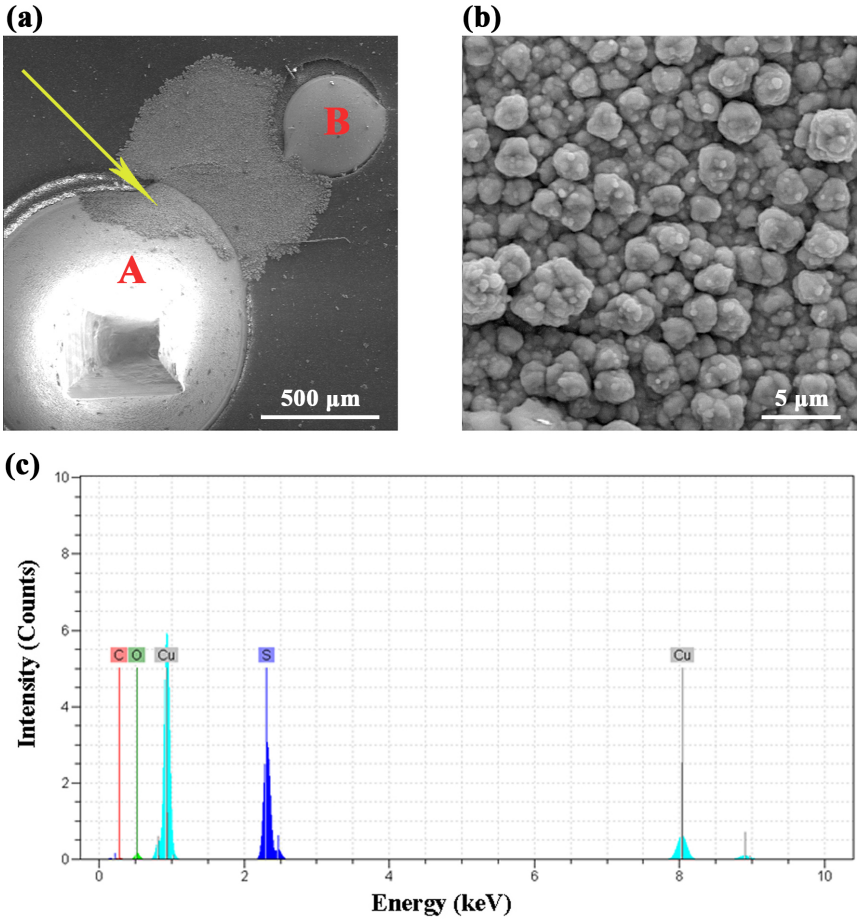

Figure 6. Schematic presentation of the corrosion mechanism of the PCB, adapted from Ref. [9].

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Tables

Table 1. Concentration of the elements existing in the corrosion products around a via, based on the EDS analysis of Figure 3.

	Elements			
	Cu	**S**	**O**	**C**
Concentration (at%)	50.0	34.4	7.2	8.4

Table 2. Concentration of the elements existing in the corrosion products around a solder, based on the EDS analysis of Figure 5.

	Elements			
	Cu	**S**	**O**	**C**
Concentration (at%)	55.1	36.3	6.3	2.3