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Synergistic galvanic-pitting corrosion of copper electrical pads treated with electroless nickel-phosphorus/immersion gold surface finish

E. Salahinejad [a,*], R. Eslami Farsani [a], L. Tayebi [b,c]

[a] Faculty of Materials Science and Engineering, K.N. Toosi University of Technology, Tehran, Iran

[b] Department of Developmental Sciences, Marquette University School of Dentistry, Milwaukee, WI 53201, USA

[c] Department of Engineering Science, University of Oxford, Oxford OX1 3PJ, UK

Abstract

This paper reports a study on the corrosion damage of a copper pad coated with electroless nickel-phosphorus/immersion gold in a sulfur-containing atmosphere. For this purpose, the surface and cross section of the pad showing localized corrosion products were analyzed by optical microscopy, scanning electron microscopy/energy-dispersive X-ray spectroscopy and X-ray photoelectron spectroscopy. According to the results, slightly nickel and predominantly copper suffered from corrosion attacks, accompanied by the formation of mainly sulfide with a small fraction of sulfate. Considering the characterized configurations of the pad and corrosion products, a sequential mechanism was established to interpret the galvanic-pitting corrosion attack of both species. In this regard, the nanoporous immersion gold surface acted as the cathode in the bimetallic degradation of nickel, whereas the environmental attack of copper was

* Corresponding Author: Email Addresses: <salahinejad@kntu.ac.ir>, <erfan.salahinejad@gmail.com>

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accelerated because of its electrical contact with cathodic nickel and gold via the formation of a trimetallic galvanic microcell.

Keywords: Corrosion; X-ray photoelectron spectroscopy (XPS); Microelectronics

1. Introduction

There are various metals currently used in microelectronics, such as Cu, Al, Au, Ag, Ni, Sn, Fe, Pd and so on, because of their appropriate electrical conductivity and solderability. Concerning corrosion, most of these metals belong to the group of active-passive metals which present suitable corrosion resistances in most circumstances, via the formation of passive films which protect the underlying metal [1, 2]. However, they may suffer from serious corrosion damages under especial atmospheres, including chloride and sulfur-containing environments. For instance, Ni-based alloys present corrosion-cracking in hydrogen sulfide- and chloride-containing environments at room temperature [3, 4]. Copper is also known to be susceptible to sulfur-related environmental damages, particularly in H_2S and SO_2 humid atmospheres [5-7].

The corrosion problem in microelectronics is remarkable due to the following reasons [6, 8]. First, the current tendency toward the miniaturization of electrical components, regarding both dimension and spacing, leads to provoking a disruption in operation even with an insignificant damage. Second, the variety of materials used in this industry and their electrical contacts, when exposed to a corrosive atmosphere, establish galvanic cells which enhance the corrosion rate of the anodic or more-active species. Third, the existence of an electrical potential difference between the connecting components of the circuit creates an electrochemical corrosion potential difference, accelerating the corrosion attack in line with the current direction.

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On the other hand, most corrosion products having a creeping behavior are conductive enough to form undesirable short circuits between the components. Furthermore, the loss of materials due to corrosion damage in a conductor can create open circuits, where a loss of 10^{-12} grams is sufficient to disturb the function of an microelectronic device [6].

Depending on the device and service environmental characteristics, the different forms of corrosion can dominate, including galvanic corrosion, gas-phase corrosion, cathodic corrosion, stray current corrosion, and fretting corrosion [6, 7, 9-12]. In this study, a sequential galvanic-pitting corrosion event was recognized in a copper-based pad coated with electroless nickel-phosphorus/immersion gold surface finish exposed to in a sulfur-containing atmosphere.

2. Experimental procedure

An electrical board was working in an atmosphere with the specifications summarized in Table 1. The board's function was disturbed after almost 3 years of service in this environment, accompanied by a reduction in the electrical conductivity of golden pads assembled on the board. Typically, several black spots appeared on the pad's surface, even visible to the naked eye (Fig. 1), due to environmental corrosion attack.

To analyze the corrosion failure, the cross section of the pad after mounting and grinding by sandpapers to #3000 was assessed by optical microscopy (OM) and scanning electron microscopy (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS). Moreover, the surface of the exposed pad was characterized SEM/EDS and X-ray photoelectron spectroscopy (XPS), especially on the spots. For SEM imaging, both backscatter electron (BSE) and secondary electron (SE) modes were used to provide composition and roughness contrasts, respectively.

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Eventually, considering the results of the above-mentioned evaluations, a mechanism was established to describe the damage.

3. Results and discussion

The cross-sectional OM micrograph of the mounted pad is displayed in Fig. 2, inferring a thickness of about 55 μm for the pad assembled on an epoxy-based substrate. To characterize the pad's structure in more detail, the SEM/EDS study was also conducted on the related cross section, as presented in Fig. 3. An overall EDS scan on the cross section yields the presence of Au, Ni, Cu and P in the investigated area (Fig. 3c). Considering both the BSE-SEM micrograph (Fig. 3a) and the horizontally-related linear EDS scans together for the detected elements of Au, Ni, Cu and P (Fig. 3b), it is concluded that a layer of Ni-P with the mean thickness of almost 8 μm and then an Au film of about 1 μm in thickness have been deposited on the Cu-substrate pad, respectively. The individual compositions of each layer constituting the pad are also confirmed by the corresponding point EDS analyses (Figs. 3d, 3e and 3f). Hence, it is concluded that the pad is essentially composed of a Cu foil coated with electroless nickel-phosphorus/immersion gold which is one of the most-commonly used surface-finish methods to improve solderability and somewhat corrosion resistance in microelectronics [6, 13]. According to Fig. 4, the top layer of the pad (Au) contains nanopores, which is typical for gold coatings obtained by immersion processes [8, 12, 14], whereas the Ni-P interlayer is not porous.

Fig. 5 indicates the SEM micrographs and EDS spectra of the exposed surface. The appearance of a dual contrast in the low-magnification BSE micrograph (Fig. 5a) infers a typical difference in the chemical compositions of the spots (as the corrosion damage) and intact

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surface. Based on this micrograph, the spots present a wide range of size from submicron to 300 μm, with a relatively rounded shape and uniform distribution on the surface. There is also another contrast although at a smaller level on the spot's its surface magnified by a BSE micrograph (Fig. 5b) which can be attributed to a small compositional and/or topographic difference. The related SE micrograph (Fig. 5c) indicates that the spot is a progression of a pit filled and overflowed with corrosion products. The non-dense particulate nature of the corrosion products is also evident in the high-magnification SE micrograph (Fig. 5d) which is in contrast to the packed and integrated feature of protective passive corrosion films. The porous and oxide/salt nature, in contrast to a packed metallic structure, of the corrosion products is responsible for a lower density and thereby more volume for them, justifying their overflow from the pit's edge. The absence of obvious cracks in the corrosion scale also reveals a relatively constant humidity level and temperature of the environment [15]. The EDS analysis (Fig. 5e) demonstrates the presence of Cu, Ni, S, O and C in the corrosion products, which can be attributed to the possible formation of the S, O and/or C-containing compounds of copper and nickel. According to the related quantitative analysis listed in Table 2, the significant amounts of Cu and S are noteworthy, which suggests the domination of copper sulfides in the corrosion product. On the other hand, the EDS analysis of the intact golden surface (Fig. 5f) indicates the existence of Au, Cu and Ni, due to the multilayer feature of the pad.

The XPS analysis was also employed in order to further characterize the exposed surface (Fig. 6). The de-convoluted spectrum of the Cu 2p orbital represents two typical peaks at 932.5 eV and 952.5 eV, which is attributed to the existence of Cu^{2+} ions bonded to the sulfide and sulfate functional groups, respectively [15-17]. The relative intensity of these peaks suggests the

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domination of sulfide to sulfate. In the S 2p spectrum, the strong peak of 161/8 eV and the weak peak of 168.8 eV are indicative of a major level of sulfide and a minor content of sulfate, respectively, which is in good agreement with the Cu 2p spectrum in both the type and amount of the existing bonds. The Ni 2p orbital spectrum also shows two weak peaks at 853.5 eV and 872 eV belonging to the Ni^{2+} state [18, 19]. Considering the Ni 2p and S 2p spectra together, it is speculated that a small amount of oxidized Ni in the form of sulfide/sulfate also exists in the corrosion products, verifying the EDS analyses. In the Au 4f orbital spectrum, the two typical peaks of 84 eV and 87 eV are related to elemental Au, suggesting the lack of corrosion for this noble element, in agreement with EDS analysis done on the corrosion products. In this regard, the absence of any XPS peaks related to nonionic Cu and Ni infers that the surface has been covered by a film of Au, whereas the Cu and Ni species originate from the underlying layers to form the corrosion products. In other words, the XPS and EDS analyses on the exposed surface together show that the above-described cross-sectional studies, where the pad is a multilayer composite with an intact upper film of Au and the bottom layers of Cu and Ni exposed to the corrosion attack. Also, the corrosion products comprising Cu and Ni sulfide/sulfate reach the surface through the porosity of the Au layer, as pointed out in Fig. 4. The XPS peaks of 532 eV and 284.8 eV detected in the O 1s and C 1s spectra are also related to contamination adsorbed, respectively. The absence of any XPS peak corresponding to chlorine verifies that Ni and Cu are not susceptible to this species. In conclusion, the XPS and EDS analyses infer that predominantly Cu and insignificantly Ni have experienced the environmental corrosion attack in the form of principally sulfide with a small fraction of sulfate, whereas the gold species of the pad has remained intact.

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Considering the characterizations conducted on both the pad and the corrosion products, a sequential mechanism was established to describe the corrosion damage (Fig. 7), as follows:

Step 1: The environmental humidity adsorbed on the board and forms an electrolytic layer of water on the pad's surface. For this purpose, at least 40 % humidity is needed [10], whereas the board was exposed to the environment of 50 % to 70 % humidity (Table 1).

Step 2: Regarding the considerable concentration of hydrogen sulfide in the environment (Table 1), several protons as a corrosive species are produced by the consecutive ionization reactions of hydrogen sulfide ($H_2S \rightarrow H^+ + HS^- \rightarrow 2H^+ + S^{2-}$). In addition, a small amount of sulfuric acid is formed probably via the reaction $SO_2 + O_2 + H_2O \rightarrow H_2SO_4$, where the existence of SO_2 traces is possible in the environment. As a result of the ionization of sulfuric acid ($H_2SO_4 \rightarrow 2H^+ + SO_4^{2-}$), some sulfate is also created in the electrolyte.

Step 3: According to the electromotive force (EMF) series, Au is noble with respect to the hydrogen reduction reaction. This means that the Au top layer of the pad is not corroded in this environment, which is compatible with the XPS and EDS analyses done on the corrosion film. In the ideal state, the Au top layer should be dense (like Fig. 7a), but it is nanoporous in practice according to Fig. 4. The open pores of the Au layer, as schematically shown in Fig. 7b, allows the corrosive electrolytic adsorbed layer to diffuse toward the Ni-P intermediate layer.

Step 4: A galvanic cell is formed at the bottom of the open pores inside the Au layer, where Au acts as the cathode and Ni acts as the anode, based on the EMF series. The exposure of both cathode and anode to the electrolyte and their electrical contact lead to the localized corrosion attack of Ni (Fig. 7c). The above-mentioned concentration cell also assists the fast propagation of corrosion pits in the vertical direction, whereas a minor growth along the horizontal occurs. The

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sensitivity of Ni to sulfur attacks has been also pointed out in the literature [3, 4]. No contribution of chloride to this pitting corrosion event was verified above by the XPS analysis (Fig. 6), where the shielded porosity of the Au layer provides the requirements of this reaction. The above-mentioned mechanism for pitting corrosion is in good agreement with studies conducted on substrates coated with porous gold layers [8, 12, 14].

Step 5: By progression of pitting in the Ni layer, the electrolyte reaches the Cu substrate and the corrosion damage of Cu becomes dominant (Fig. 7d). This hypothesis is supported by the facts that, first, the unexposed Ni-P interlayer is nonporous and dense (Fig. 4) and, second, the corrosion products accumulated on the Au layer surface are composed of both Ni and Cu (Figs. 5 and 6). Note that in the EMF series, Cu is more noble than Ni; however, Cu is more susceptible to sulfur attacks. It means that in this studied sulfur-containing environment, Cu is corroded in this trimetallic layer galvanic cell in which Ni and Au act as the cathode with a larger surface area, dictating a faster corrosion for anodic Cu.

Step 6: Considering the lower electrical conductivity of the Cu and Ni sulfide/sulfates in comparison to the related metals, the pad's operation is disturbed by progression of the corrosion event because the loss of the metals in the pits reduces the electrical conductivity in the own pads. Moreover, the overflow and projections created by the corrosion products on the edge and in the vicinity of the pits disadvantageously affect the electrical connection between the pads' surface and the components of adjacent boards.

The domination of Cu to Ni in the corrosion attacks and products is due to the following reasons. First, Ni is corroded in a bimetallic cell with Au, while the anodic reaction of Cu is accompanied by a large cathodic surface. Second, upon the arrival of the pits to the Ni/Cu

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interface, both Au and Ni are cathodically protected by Cu which acts as a sacrificial anode. Third, the thickness of the Ni layer is much smaller than that of the Cu substrate. This means that the pits travel to the Ni layer fast, so that the propagation of the pits in Cu is assigned to most of the exposure period. The domination of sulfides to sulfates in the corrosion products is also attributed to the above-described concentration cell, where the oxygen required to form sulfate is depleted in the shielded pores.

4. Conclusion

An electrical board containing copper-based pads with electroless nickel-phosphorus/immersion gold surface finish was exposed to a sulfur-containing humid atmosphere. The average thickness of the upper Au layer of the pads was about 1 μm, whereas that of the Ni-P interlayers was 8 μm. The environmental exposure of the board created several pits filled with corrosion products on the pad. According to the EDS and XPS analyses on the corrosion products, it was found that mainly the Cu substrate and slightly the Ni interlayer experienced the corrosion attack. The corrosion products also contained predominantly sulfide with a small fraction of sulfate. Based on the characterization done on the pad and corrosion products, a synergistic galvanic pitting corrosion event was realized to be responsible for the attack. The nanoporous nature of the Au layer played a critical role in this regard.

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Tables

Table 1. Specifications of the service environment.

Temperature (°C)	H_2S (ppm)	Humidity (%)	Cl (ppb)
15-25	30-50	50-70	< 4

Table 2. Quantitative EDS analysis on the corrosion products located on the pad's surface.

Concentration	Elements				
	Cu	Ni	S	O	C
at%	37.4	4.0	35.6	12.8	10.2
wt%	58.2	5.7	28.0	5.1	3.0

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Figures

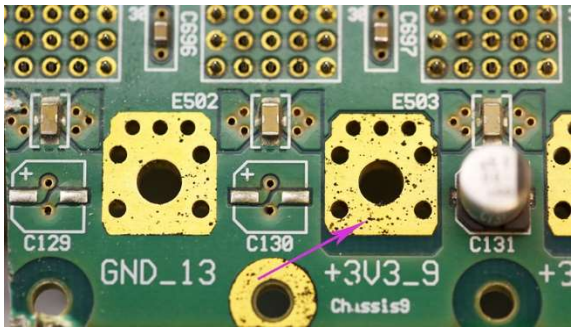

Fig. 1. Macrograph of a part of the exposed board with typical spots on golden pads, as indicated by an arrow.

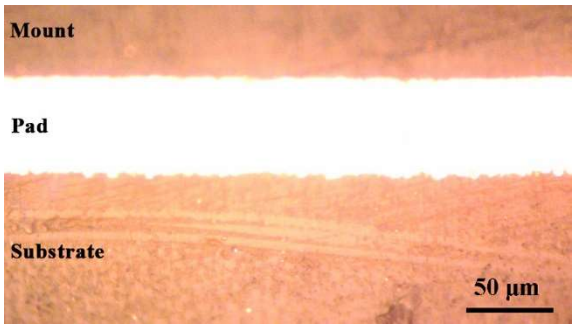

Fig. 2. Cross-sectional OM micrograph of the unexposed pad.

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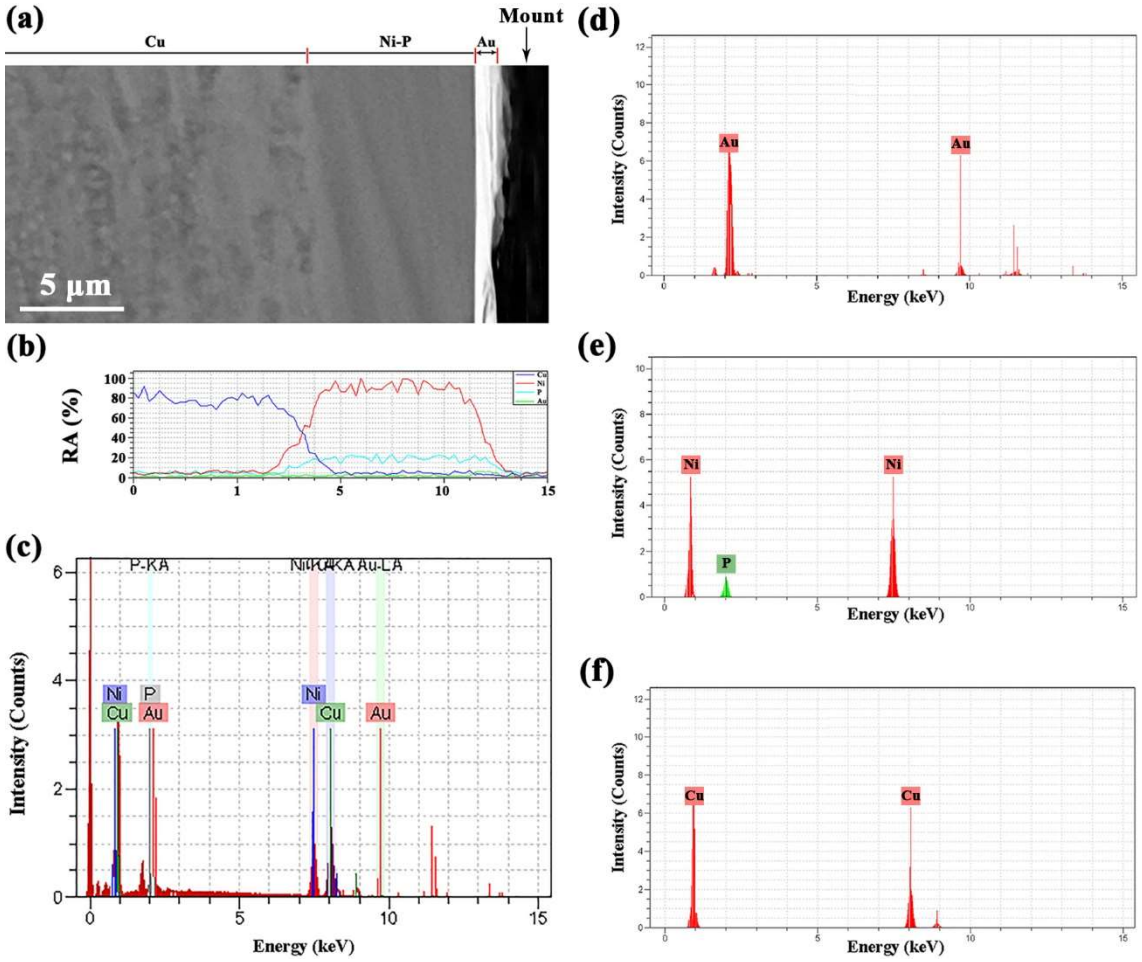

Fig. 3. Cross-sectional BSE-SEM micrograph of the unexposed pad (a) with the corresponding linear EDS scans for Cu, Ni, Au and P, where RA refers to Relative Abundance in the vertical axis (b), overall EDS pattern of the cross section, (c) and point EDS patterns of the different layers of the pad: the top layer (d), middle layer (e) and bottom layer (f).

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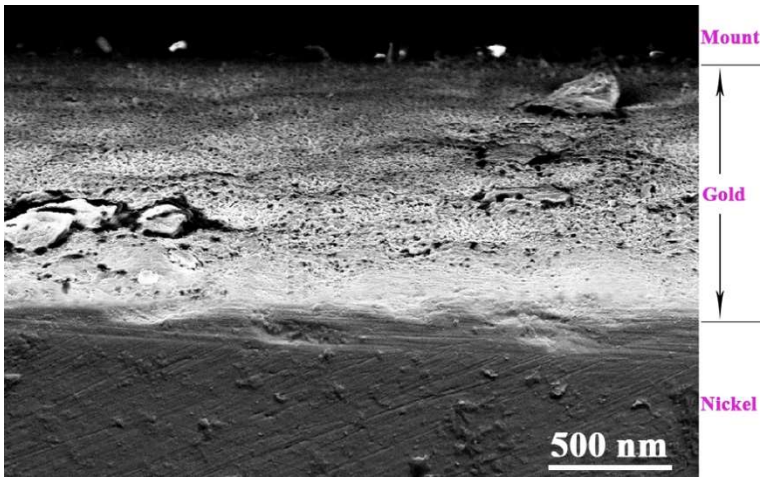

Fig. 4. Cross-sectional SE-SEM micrograph of the layers of the pad before the environmental exposure.

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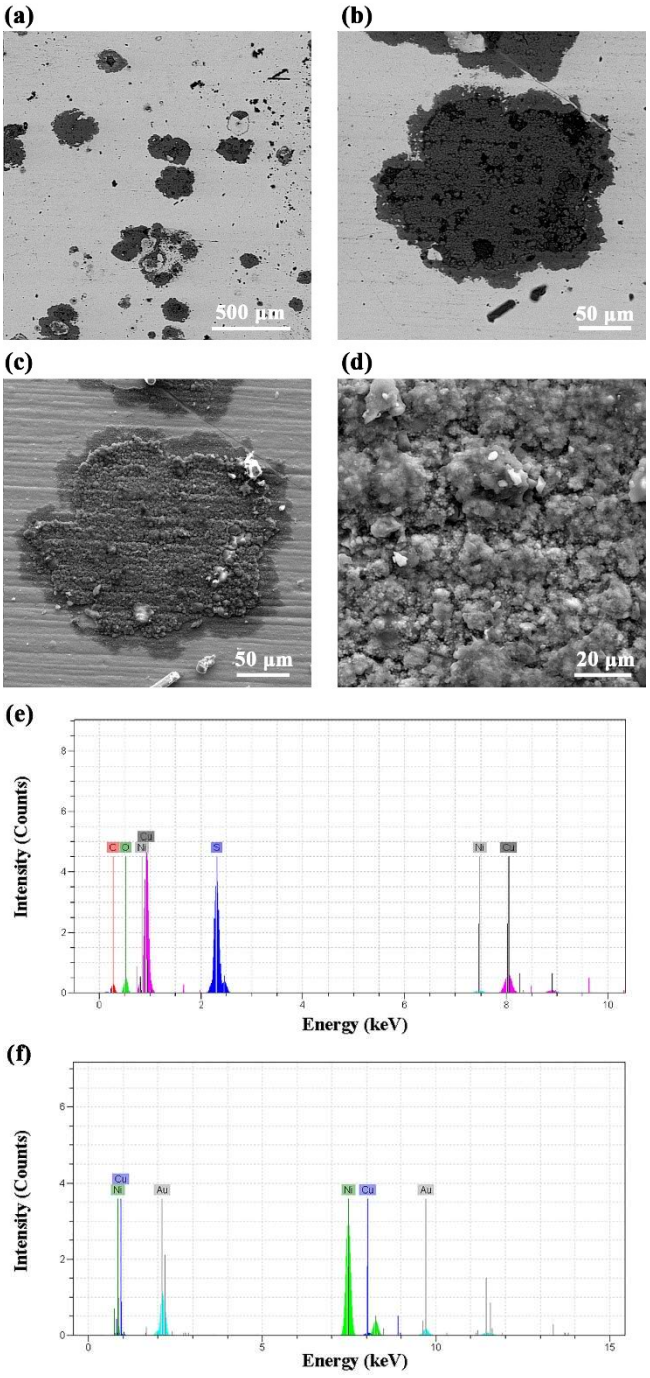

Fig. 5. BSE-SEM micrographs of the exposed pad's surface in two magnifications (a, b), SE-SEM micrographs of the pad's surface in two magnifications (c, d) and EDS patterns taken of a spot on the pad's surface (e) and the intact pad's surface (f).

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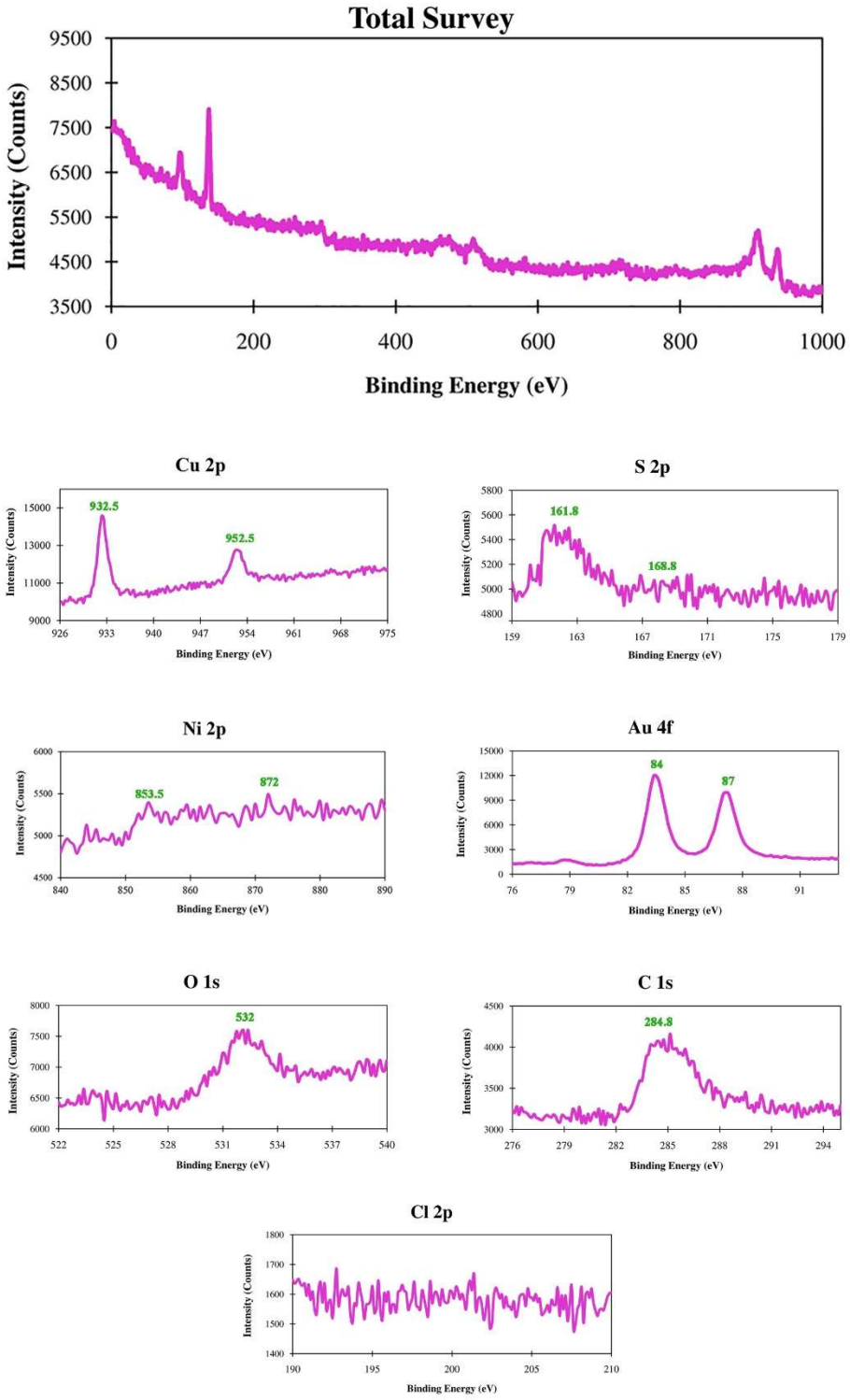

Fig. 6. Total and de-convoluted XPS spectra taken of an exposed surface.

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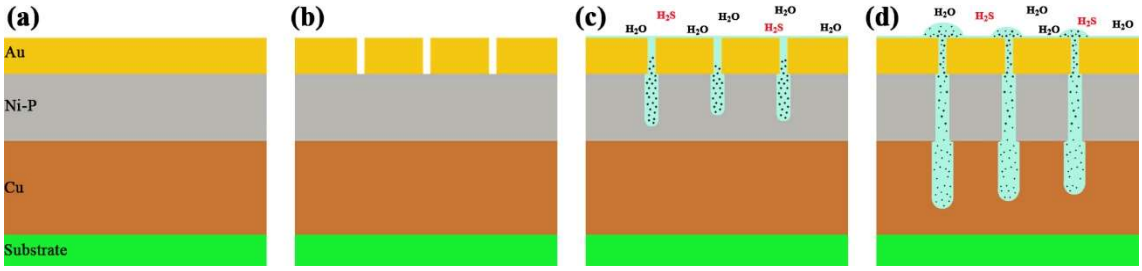

Fig. 7. Schematic illustration of the corrosion attack mechanism involved: ideal configuration of the pad (a), real configuration of the pad before the environmental exposure (b), the pad at the initial stages of the corrosion attack (c) and the pad after the developed corrosion damage (d).