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A highly stabilized–inhibited nitric acid/ferric nitrate-based solder stripping solution

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Abstract

The effect of ammonium sulfamate and benzotriazole on the prevention of exothermic conditions, the evolution of toxic NO_x gases and the reduction of copper attack during solder stripping were studied. The effect of these additives on the rate of temperature rise for solutions treating 63Sn37Pb solder alloy and pure copper were investigated. Weight loss analysis was also performed in order to evaluate the rate of copper etching in various compositions. Scanning electron microscopy was employed to investigate the nature of the attack on the copper surface. In order to identify the role of ammonium sulfamate and benzotriazole on the stripping process, anodic polarization measurements were performed for the copper electrodes. The results revealed that ammonium sulfamate is capable of eliminating the reduction of nitric acid and the evolution of toxic NO_x gases, can prevent excessive temperature rise, and can inhibit the rate of copper etching. On the other hand, benzotriazole is capable of providing a mirror bright copper surface after the solder stripping. Simultaneous addition of ammonium sulfamate and benzotriazole to the stripping solution led to a unique solder stripping composition which is substantially secure in terms of the temperature rise, evolution of toxic NO_x gases and copper attack.

1. Introduction

Conventionally, a layer of an etch resist metal or alloy such as various compositions of tin–lead alloys, or substantially pure tin, is plated on circuit patterns to protect the underlying copper against the ammonical cupric chloride etching solutions used to etch away the undesired copper and form the circuit patterns [1]. After performing its task of protecting the copper patterns, the metal etch-resist layer must be removed from the copper substrate so that the latter is exposed and finished with the final desired finishing methods, i.e. electroless tin, Ag, Ni/Au [2] or Hot Air Solder Leveling (HASL).

Numerous acidic compositions have been patented for use in selectively stripping tin and tin/lead alloys from copper substrates. These acidic solutions can be categorized as fluoride containing compounds and peroxide solutions [3, 4], solutions employing nitro-substituted aromatic compounds as a principal ingredient, often in conjunction with an inorganic acid [5, 6], an organic acid [7], or both of them [8], alkane sulfonic acid-based methods [7–10], and compositions based on nitric acid and ferric ions [11–16].

The most recently developed solder strippers involve nitric acid, to dissolve and maintain tin or tin/lead in the solution, and ferric nitrate, to remove intermetallic tin/copper film. Not withstanding the fact that nitric acid-based formulations have advantages such as short stripping time and high metal load capacity, some shortcomings are associated with employing such compositions. These problems include the potential for the occurrence of exothermic conditions and the evolution of toxic NO_x gases from nitric acid, as well as the negative effects on the appearance of the underlying copper and high rates of copper etching. To remove these shortcomings, various additives have been added to the nitric acid/ferric nitrate stripping solution, in spite of the fact that the price of the added chemical often increases the cost of the stripping process. In some cases the added ingredients are so expensive that the resultant stripping solution is impractical to use [12].

The Kawanabe patent [17] describes stripping solutions consisting of an inorganic and/or organic acid, an oxidizing agent, and a heterocyclic compound free of sulfur but containing a nitrogen atom as a ring forming member, e.g. imidazoles and triazoles. According to the patent, these types of additive inhibit copper attack. Campbell [12] claimed that most of such solutions, using only imidazoles and triazoles as copper corrosion inhibitor, release approximately 100–1000 ppm of NO_x gases during the stripping process. Campbell also claimed that the emission of toxic NO_x gases can be avoided through stabilization of the nitric acid with ammonium sulfamate. However, the copper surface appearance is unacceptable when only ammonium sulfamate is used as the nitric acid stabilizing agent.

In this research, a nitric acid/ferric nitrate-based method of stripping tin or tin/lead from the copper substrate of printed circuit boards is described. The main objective of this research was to eliminate the major problems encountered with the application of nitric acid/ferric nitrate formulations including the evolution of NO_x gases, the potential for exothermic conditions, and high degree of copper surface attack. These objectives were achieved through addition of ammonium sulfamate and benzotriazole to a simple nitric acid-based stripping solution. The mechanisms of the improvements achieved are described in detail.

2. Experimental

The chemicals used were chosen from the extra pure grade of Merck Company. The composition and operating conditions of the stripping solutions are listed in Table 1. Four different solutions of various constituents were employed for the experiments. All these solutions contain nitric acid in order to dissolve tin and lead, and ferric nitrate, to remove the tin–copper intermetallic diffusion layer.

The substrates used for the experiments were chosen from a conventional FR-4 copper clad laminates with a copper thickness of 18 μm on both sides. The dimensions of these specimens were 100 × 100 × 1.6 mm. The substrates were electroplated with copper and 63Sn37Pb solder alloy to a thickness of approximately 25 and 15 μm, respectively. Cupracid from Atotech was employed for copper electroplating. The method used for tin/lead plating was "Tin–Lead Bath La", which is a fluoborate-based tin/lead plating solution purchased from Schlötter Co., Germany.

In order to evaluate the effect of ammonium sulfamate and benzotriazole on the occurrence of exothermic conditions, a total surface area of 25 cm^2 of 67Sn37Pb ingots and 25 cm^2 of 35 μm copper foil were immersed in 50 ml of each of the solutions described in Table 1. The profiles of the temperature rise vs. immersion time were measured for the mentioned solutions. Also, to evaluate the effect of additives on the degree of copper attack, the rate of copper etching was measured through the weight loss method as well as scanning electron microscope observations.

Table 1. Composition of the solder stripping solutions

Constituents	Solution			
	1/M	2/M	3/M	4/M
Nitric acid	3.2	3.2	3.2	3.2
$Fe(NO_3)_3$	0.4	0.4	0.4	0.4
Hydrochloric acid	0.023	0.023	0.023	0.023
Ammonium sulfamate	–	0.5	–	0.5
1,2,3-Benzotriazole	–	–	0.007	0.007

The scanning electron microscope (SEM) (CamScan MV2300) was used to study the surface structure of the underlying copper before solder plating and after stripping of the tin/lead layer in various solder stripping solutions.

In order to analyze the effects of ammonium sulfamate and benzotriazole on the copper attack reduction, anodic polarization measurements were made at 25 °C in a cell equipped with a platinum counter electrode and Ag/AgCl reference electrode (663 VA Stand, made by Metrohm Company, Switzerland). The working electrode was a copper clad laminate with a total effective area of 1 cm^2. These measurements were made with an instrument made by μAutolab (Type II) Co., the Netherlands. For anodic polarization measurements, the potential of the working electrode was scanned in the positive direction at the speed of 10 mV s^{-1}.

3. Results

3.1. *Temperature rise of the stripping solutions*

Figure 1 represents the temperature rise profiles vs. immersion time during which 50 ml of the solutions described in Table 1 were stagnantly in contact with the total surface area of 25 cm^2 of 63Sn37Pb ingots. The numbers assigned to the curves are the numbers indicative to the compositions listed in Table 1. As shown in Figure 1, the existence of two completely different groups of curves is obvious. For the first group of curves, comprising curves 1 and 3, the slope of the temperature rise is very high, i.e. 0.92 °C min^{-1} for solution No. 1 and 0.86 °C min^{-1} for solution No. 3. However, for the second group of curves, comprising curves 2 and 4, the rate of temperature rise is in the vicinity of 0.42 °C min^{-1}. The initial conditions for both groups were similar.

Since the only difference between the chemical composition of the two different groups of solutions is the absence or presence of ammonium sulfamate (Table 1), it can be concluded that ammonium sulfamate is the only constituent that inhibits the temperature rise of the stripping solution effectively.

In practical conditions, after complete stripping of the tin–lead layer, the exposed copper is in contact with the stripping solution for a few seconds. Therefore, the temperature rise profiles for 50 ml of the solutions loaded with 25 cm^2 of pure copper foil were examined. The results of such experiments are shown in Figure 2. The rate of temperature rise obtained for solution No. 1 is 4.8 °C min^{-1}, those for solutions No. 2 and No. 4 are nil, and that for solution No. 3 is 2 °C min^{-1}.

Referring to Table 1, solutions No. 2 and No. 4 contain the same concentration of ammonium sulfamate while solution No. 3 contains only benzotriazole as additive. It is obvious that temperature rise in the

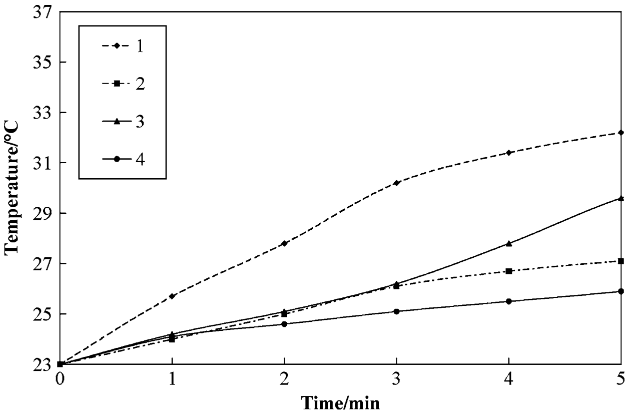

Fig. 1. Temperature rise profiles vs. immersion time for the stripping solutions No. 1–4 with a total load of 25 cm^2 of 53Sn37Pb. For compositions see Table 1.

solutions containing ammonium sulfamate is essentially avoided. The noticeable difference between curves 1 and 3 indicates that benzotriazole is also a relatively effective reagent in inhibiting temperature rise in the stripping solution in the presence of pure copper.

Moreover, no NO_x fume evolution was observed during the immersion of both copper and solder into the stripping solutions containing ammonium sulfamate, i.e. solutions No. 2 and No. 4. However, for the other two solutions, the evolution of orange fumes of NO_x gases was visually evident.

3.2. *Copper attack*

The rate of copper etching achieved with various stripping solutions listed in Table 1, are shown in Figure 3. For this set of experiments, solder coating was not applied on the copper electroplated specimens.

Solution No. 1 exhibits the highest rate of copper etching, i.e. about 3.67 μm min^{-1}. This solution contains neither stabilizing nor copper corrosion inhibitor reagent. In contrast, the etching profile of solution No. 2 reveals that the addition of ammonium sulfamate to solution No. 1 achieves a copper etch rate of 0.21 μm min^{-1}. This rate is significantly lower than that for solution No. 1. The copper etch rate achieved with solution No. 3, containing benzotriazole, is 3.23 μm min^{-1}, which is slightly lower than that for solution No. 1. The copper etch rate achieved with solution No. 4 is similar to that obtained with solution No. 2. Incorporation of benzotriazole, as a copper anti-corrosion reagent, results in a slightly lower rate of copper etch with respect to that of solution No. 2. In conclusion, ammonium sulfamate is the only ingredient that can significantly decrease the rate of copper etching.

In order to investigate the surface morphology of copper after solder stripping, four copper and solder plated test coupons were immersed into stripping solutions listed in Table 1. After approximately 1 min, the entire solder coating was removed from the copper surface for all solutions. After thorough rinsing with distilled water and drying, SEM images for these coupons were obtained. The results are shown in Figure 4(a–d). In order to compare the results with the as-plated copper surface, an SEM image of such a surface is represented in Figure 4(e).

As shown in Figure 4(a), in comparison with Figure 4(e), the copper surface is non-uniformly etched and evenly distributed small corroded grains of copper are visible. The footprints of parallel lines formed by pre-plating preparation using a brushing machine, as clearly seen in Figure 4(d) and (e), are not present. Furthermore, the color of this test coupon was brownish pink, revealing the occurrence of corrosion.

In Figure 4(b), it is obvious that the surface of the underlying copper has been roughened to some extent by solution No. 2. The color of this test coupon appeared yellowish pink. These observations indicate that solution No. 2 has attacked the copper and spoiled its appearance. However, the slightly visible brushing footprints show that the severity of copper attack is less than that of solution No. 1. Also, it is obvious that the corrosion of copper is limited to grain boundaries. This indicates that copper attack has been reduced with respect to that in solution No. 1. The latter observation is in good agreement with results of etching rate experiments shown in Figure 3. Although

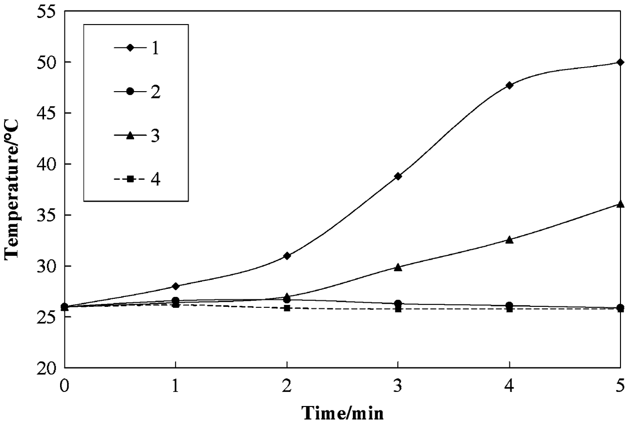

Fig. 2. Temperature rise profiles vs. immersion time for the 50 ml of stripping solutions No. 1–4 with a total load of 25 cm^2 of 35 μm thick copper foil. For compositions see Table 1.

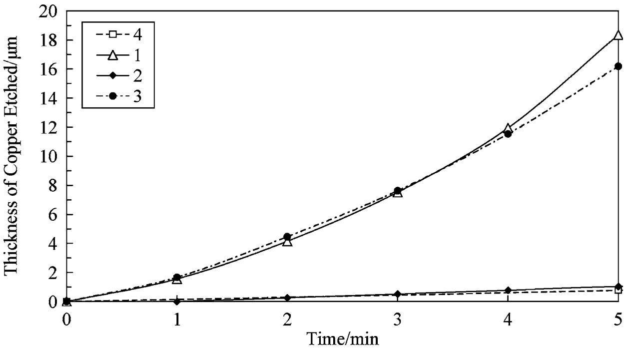

Fig. 3. Thickness of copper etched by various stripping solutions vs. time. For compositions, see Table 1.

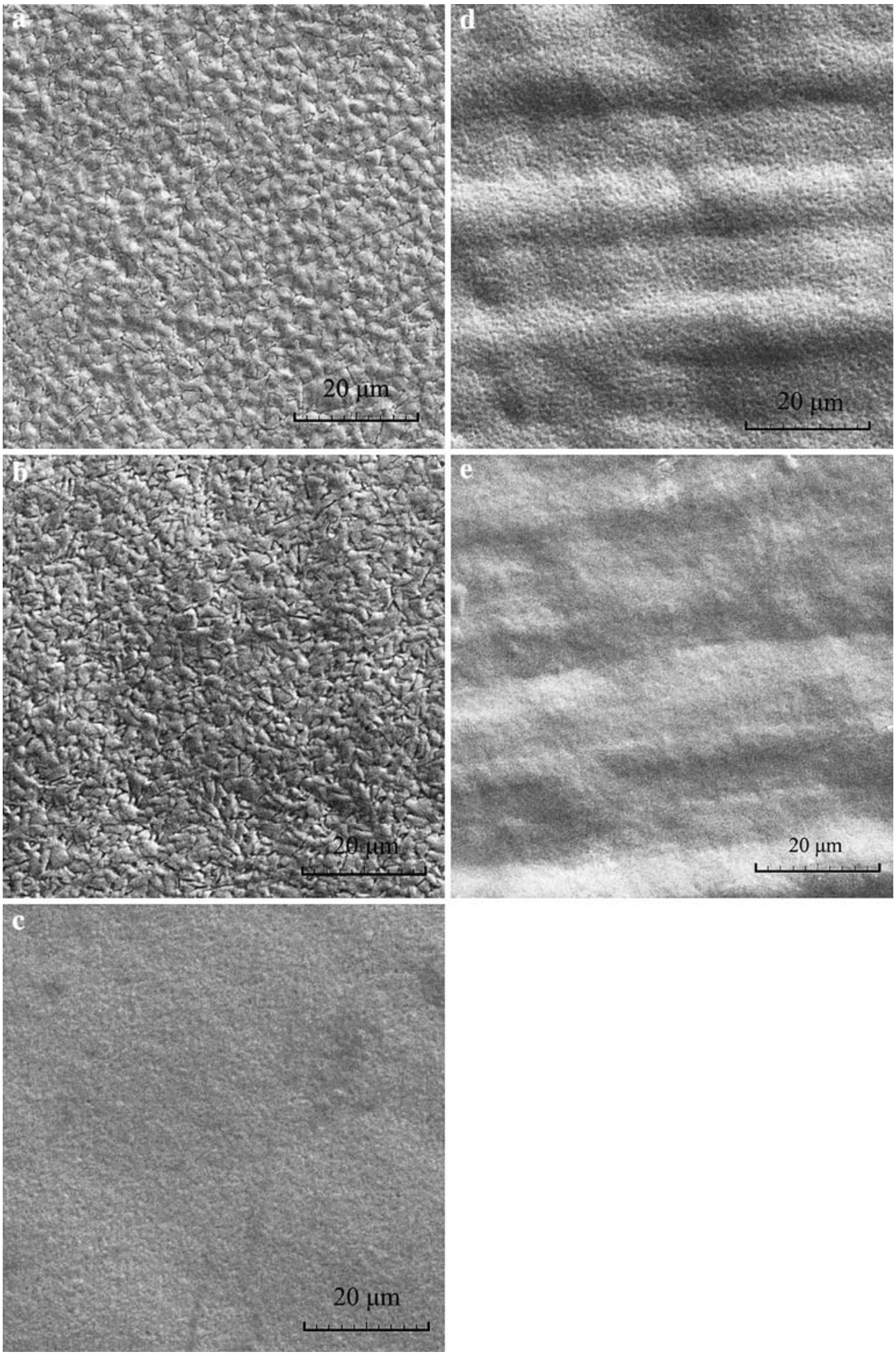

Fig. 4. SEM images of the copper surfaces after 2 min of stripping time in: (a) solution No. 1, (b) solution No. 2, (c) solution No. 3, (d) solution No. 4, and (e) as-plated copper. For compositions, see Table 1.

the rate of copper etching in this stripping solution is industrially acceptable, the appearance of the copper surface is not aesthetic and the small extent of attack, which is occurring at grain boundaries, will lead to further copper corrosion.

Not withstanding the fact that solution No. 2 is highly stabilized, the lack of a copper corrosion inhibitor or brightening reagent results in a non-uniform and visibly etched copper surface, which is not industrially desirable. The improvement achieved through incorporation of ammonium sulfamate in a typical nitric acid/ferric nitrate stripping solution can only be expressed in terms of significant reduction in the rate of copper dissolution, but not the quality of the copper surface appearance.

The copper surface topography represented in Figure 4(c) is highly uniform and free of small features stemming from uneven dissolution of the underlying copper surface. No brushing footprint is visible in this picture. This is in good agreement with the visible appearance of the surface, which was mirror bright. The high rate of copper etching indicated in Figure 3 and the absence of brushing footprints both show that the rate of copper dissolution is high but very uniform. Despite the acceptable surface appearance achieved by this solution, the high rate of copper etching restricts its application.

The mirror bright surface of the copper after stripping of the solder coating is attributable to the brightening effect of benzotriazole in combination with chloride ions [1] which gives rise to a highly uniform but severely dissolved copper surface. As a result, the only improvement achieved through incorporation of benzotriazole is the acceptable appearance of the copper surface.

As shown in Figure 4(d), the copper surface has been roughened only very slightly with respect to that of Figure 4(e), i.e. as-plated copper. However, the footprints of the brushing pre-preparation are graphically visible in this image. The color of this specimen also appeared uniformly bright pink. Thus the improvement in copper surface appearance along with the negligible rate of copper dissolution has been achieved through simultaneous incorporation of ammonium sulfamate and benzotriazole in the base solution, i.e. solution No. 1. No further copper corrosion was visibly detected for the specimens prepared with this solution even after 1 month of storage.

4. Discussions

4.1. *Effect of ammonium sulfamate*

Reduction of nitric acid by metallic species occurs during solder stripping in the non-stabilized nitric acid-based solutions according to the following reaction:

$$n\mathrm{NO_3^-} + 3n\mathrm{H^+} + 2\mathrm{M} \rightarrow n\mathrm{HNO_2} + 2\mathrm{M}^{n+} + n\mathrm{H_2O} \qquad (1)$$

Nitrous acid is produced as a result of reduction of nitric acid. Gladyshev et al. [18] stated that HNO_2 first accumulates in solutions containing metal ions and nitric acid and then decomposes to nitrogen oxides. The reactions showing the possible products of decomposition of nitrous acid are presented elsewhere [19, 20].

Therefore, evolution of toxic NO_x fumes accompanied by the release of a large amount of heat, due to the high rate of metal dissolution and decomposition of nitric acid, occurs as a result of nitric acid reduction by metallic species during the solder stripping process. However, reduction of nitric acid by metals can be essentially avoided through incorporation of sulfamic acid and its salts [18, 19, 21]. Sulfamate ions are capable of reacting with nitrous acid and convert it to sulfuric acid [21, 23] according to Equation (2):

$$\mathrm{H_2NSO_3H} + \mathrm{HNO_2} \rightarrow \mathrm{N_2} + \mathrm{H_2SO_4} + \mathrm{H_2O} \qquad (2)$$

Gladyshev et al. [18] found that this reaction is of first-order with respect to both N_2HSO_3H and HNO_2. The following rate equation has been suggested by these authors [18]:

$$\upsilon = k_{\mathrm{ap}}[\mathrm{H_2NSO_3H}][\mathrm{HNO_2}] \qquad (3)$$

where, υ is reaction rate and k_{ap} stands for apparent rate constant ($k_{\mathrm{ap}} = 0.93\ \mathrm{l\ mol^{-1}\ s^{-1}}$ at $[\mathrm{H^+}] > 0.04\ \mathrm{M}$).

Consequently, ammonium sulfamate, as a source of sulfamate ions, can eliminate the formation of nitrous acid. Therefore, formation of toxic NO_x gases and the simultaneous temperature rise of the stripping solution can be avoided through incorporation of sufficient amounts of sulfamate ions. The latter conclusion is in good agreement with the results obtained in this research.

4.2. *Effect of benzotriazole*

Figure 5 represents the anodic polarization curves for various stripping solutions listed in Table 1. These curves are obtained in stagnant conditions at room temperature (25 °C). The reference electrode used was Ag/AgCl.

Solutions No. 3 and No. 4 exhibit similar profiles which are completely different from those obtained with solutions No. 1 and No. 2. A limiting current density is obvious for solution No. 3 and No. 4 in the voltage range 0.05–0.12 V. These solutions both contain benzotriazole.

Benzotriazole is an efficient corrosion inhibitor for copper and copper-based alloys. The copper surface reacts with physically adsorbed benzotriazole molecules to precipitate insoluble Cu–BTA (Copper–Benzotriazole Complex) complexes on the surface [22–27]. Two mechanisms have been proposed to account for the high inhibiting efficiency of benzotriazole. The first mechanism is attributed to the adsorption of benzotriazole on the copper surface [27]. The second

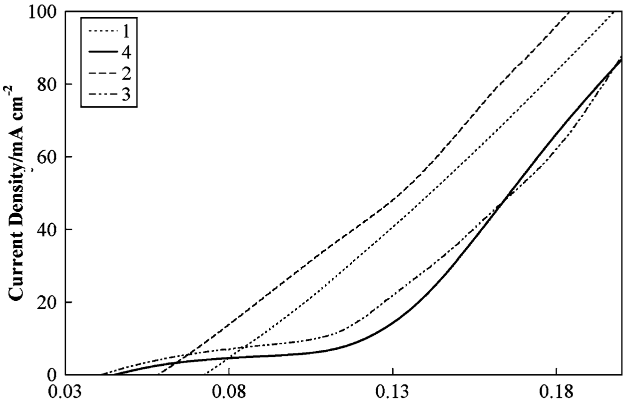

Fig. 5. Anodic polarization curves for dissolution of copper in various stripping solutions. For compositions, see Table 1.

mechanism postulates the formation of a polymeric complex of Cu(I)BTA using Cu–N bonds [27–31]. Mansikkama and co-workers [31] believe that the latter polymeric film acts both as a physical barrier to ions as well as hindering copper dissolution through binding copper atoms, i.e. BTA (Benzotriazole) inhibits anodic dissolution sites.

As previously shown, benzotriazole is capable of retarding the temperature rise in the stripping solution in contact with pure copper (Figure 2), slightly decreasing the rate of copper etching (Figure 3) as well as inhibiting the corrosion at copper grain boundaries (Figure 4d). These observations, along with the presence of a limiting current density observed with stripping solutions containing benzotriazole, indicate the fact that in nitric acid-based stripping solutions, benzotriazole can form a protective film. However, as shown in Figure 3, regarding the high rate of copper etching, the above mentioned film cannot perform its protective function in the absence of ammonium sulfamate. Benzotriazole therefore significantly reduces the copper surface attack only in the presence of ammonium sulfamate and in the absence of the latter acts only as a brightener for copper.

Also, to the knowledge of the authors, there is no work indicating that ammonium sulfamate is a copper corrosion inhibitor. Furthermore, as described earlier, ammonium sulfamate is capable of eliminating the reduction of nitric acid by metals. In conclusion, none of these additives can prevent copper attack by nitric acid-based stripping solutions independently. On the other hand, combination of ammonium sulfamate, as a stabilizing agent, with benzotriazole, as a corrosion inhibiting agent, has a synergetic effect on the reduction of copper attack.

The prepared stripping solution has been used successfully in a horizontal spray machine for more than a year and no shortcoming has been reported [32]. This means that the application of additives does not adversely affect the spray properties of the solution. On the other hand, ammonium ions in the solution along with the chloride ions can reduce the amount of sludge formation. However, formation of sludge (mainly SnO_2) is inevitable. Almost all commercial solutions produce a certain amount of sludge that should be clarified out once a while. The produced sludge is, however, not sticky and does not clog the pipes.

5. Conclusions

It was shown that ammonium sulfamate serves as an effective stabilizer that eliminates the evolution of toxic NO_x gases, hinders temperature rise and profoundly reduces the rate of copper etching in nitric acid-based solder stripping solutions. However, by incorporation of ammonium sulfamate alone copper surface attack cannot be avoided. Although having a slight effect on reducing the temperature rise, evolution of toxic NO_x gases, and copper etching rate, addition of benzotriazole to the stripping solution containing ammonium sulfamate leads to a highly stabilized–inhibited solution with no adverse effect on the underlying copper surface.

Employing such a highly stabilized–inhibited nitric acid/ferric nitrate-based solder stripping solution resulted in a fast and complete removal of solder alloy from the copper surface, elimination of the evolution of toxic NO_x gases, achievement of a copper etching rate in the vicinity of 0.2 μm min^{-1} and a uniformly bright copper surface.

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