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Selective Desoldering Separation of Tin–Lead Alloy for Dismantling of Electronic Components from Printed Circuit Boards

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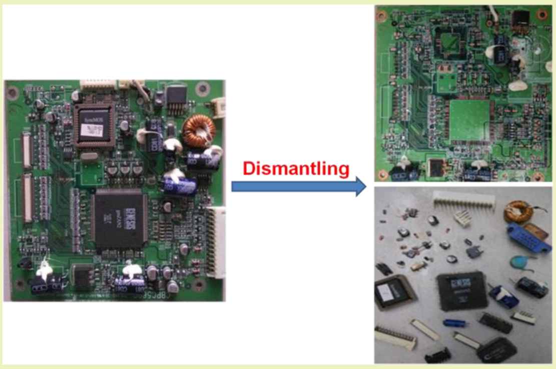
ABSTRACT: Desoldering separation of solder from waste printed circuit boards (PCBs) is a key step to recycle or dispose the electronic waste effectively. In the present work, a novel way for selective desoldering separation was achieved by dissolving lead–tin solder in fluoroboric acid containing H_2O_2. This leachant was observed possible to take the solder into solution with negligible dissolution of copper or other metals within 35 min. After leaching, the electronic components (ECs) could be easily removed from the boards under good experiment conditions. The effects of oxidants, dosage of H_2O_2, and acid concentration were explored to obtain the suitable experimental conditions. The results showed that 2.5 mol/L fluoroboric acid containing 0.4 mol/L added oxidant H_2O_2 within the reaction of 35 min could be regarded as the optimum conditions for the selective desoldering separation from the waste PCBs. Both theoretical analysis using HSC Chemistry software and actual waste PCBs examination verified that the solder could be dissolved selectively by this novel process via excess solder added to inhibit the effect of copper dissolution if exists. This safe, fast, economic, and environmentally friendly dismantling PCBs process would be of great importance in recycling waste electrical and electronic equipment (EEE) effectively.



KEYWORDS: *Solder recycling, Selective separation, Leachant, Electronic wastes, Thermodynamic analysis, Desoldering, Printed circuit boards*

INTRODUCTION

Recently, the replacement of electrical and electronic equipments (EEEs) is increasing rapidly with the swift development of the economy and electronic manufacturing industry. As such, large amounts of electronic wastes (e-wastes) are generated worldwide. It is estimated that 20–25 million tons of e-wastes are generated per year.[1] Hence, the recycle of e-wastes all over the world is being drawn more and more attention.[2,3] As a part of the typical e-wastes, printed circuit boards (PCBs), an essential part of almost all EEEs, are becoming a hot research topic in the field of recycling e-wastes.[4] Generally, approximately 70% nonmetals and 30% metals are contained in waste PCBs.[5] The waste PCBs are regarded as an "urban mineral resource" owing to 10-fold higher purity of precious metals in waste PCBs than that of the rich-content minerals.[6,7] Therefore, recycling of waste PCBs is an important subject both for the protection of environment and the recovery of valuable materials such as metals.

Currently, the pyrometallurgical, hydrometallurgical, mechanicalphysical, vacuum pyrolysis, and supercritical methods are widely employed to recycle the waste PCBs.[4,8−12] However, the aforementioned processes are usually used to recover the bare PCBs and the attention for the dismantling and recycling of PCB assemblies is much less drawn. A PCB assembly is a PCB mounted with electronic components (ECs), connected by solder.[13] Therefore, the first key step is to remove solder for dismantling PCB assemblies. For dismantling ECs from PCBs, an infrared heater or electric heating tube,[14,15] a liquid heating medium,[16] and solder baths heating medium[17,18] are usually used to remove solder. However, these technologies are needed to heat PCB assemblies to the temperature above the melting point of solder, and add external forces, such as impact, shearing and vibration, etc.[19−21] As such, the aforementioned methods suffers from some unexpected problems that high temperature will make the performance of the ECs to be adversely affected and the value to be lost for further recycled usage.[22−24] Furthermore, more thermal energy is consumed and a secondary pollution might be caused.[25−27] Therefore, it is necessary and indispensable to dismantle PCB assemblies environmentally friendly, with focus on removing solder from

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PCB assemblies without additional energy input, and leaving the bare board for further recycled treatment.

In the present study, a novel chemical leaching method was explored to dissolve selectively solder for dismantling of ECs from PCBs. As for this method, the commercial electrolyte of fluoroboric acid was taken as the leachant. This method has two significant advantages: (i) tin and lead could be selectively dissolved without serious corrosion of other metals and thus avoiding the ECs to suffer damage; (ii) tin and lead in the leachant may be directly electrolyzed to reclaim the solder rather than undergone a purification process.[28,29] As such, the dissolution of the solder would allow the dismantling of the ECs, which could be separated for subsequent treatments based on the value or toxicity. Hence, a novel selective desoldering separation method for dismantling of ECs from PCBs based on the commercial electrolyte of fluoroboric acid was examined in the present research. The effects of the added oxidants, H_2O_2 dosage, and acid concentration were evaluated to justify the process feasibility. The analysis of the chemical thermodynamics established by HSC software also gives insights into the mechanism of the new method. At last, the actual waste PCBs was examined to verify the behavior of the novel process.

EXPERIMENTAL SECTION

Materials. Fluoroboric acid (HBF_4, 48%), nitric acid (HNO_3, AR), ferric chloride ($FeCl_3$, AR), titanium tetrachloride ($TiCl_4$, AR), and hydrogen peroxide (H_2O_2, 30%) were all obtained from Sinopharm Chemical Reagent Co., Ltd., China and used without any further treatment. Ultrapure water (18.2 MΩ·cm) was used for all the experiments. The PCB samples were customer-made to make sure the negligible difference among each other (Figure 1). For the purposes of representative, all the examined PCB samples underwent the basic process of copper and tin plating, and ECs were mounted by solder (lead (40%)/tin(60%), (wt %)) connection.

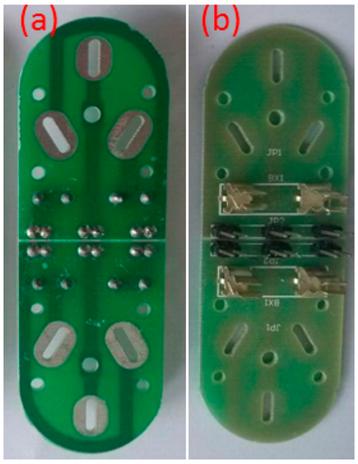

Figure 1. Examined PCB samples: (a) front side and (b) back side.

Experimental Procedures. All the batch leaching experiments were performed in 100 mL of mixed solution containing certain concentrations of HBF_4 and oxidants equipped in a beaker with a 250 mL capacity immersed in a constant temperature bath at 20 ± 2 °C. Prior to each experiment, the PCB samples and certain aliquots of chemicals were transferred to the reactor vessel to obtain the desired concentrations. 1 mL of leaching samples was withdrawn at various time intervals, followed by addition into 50 mL colorimetric tubes filled to the capacity with 2% HNO_3 solution. After 30 min, the sample solutions were detected with an iCAP 6300 inductively coupled plasma-atomic emission spectrometry (ICP-AES) instrument, provided by Thermo Scientific. Most of the batch experiments were conducted in duplicate, and the relative error was less than 2%.

Thermodynamic Analysis. Thermodynamic calculations of all the leaching reactions were carried out using the HSC Chemistry, Version 5.0 software, a chemical reaction and equilibrium software with an extensive thermochemical database.

RESULTS AND DISCUSSION

Effect of Different Oxidants. Without any oxidant, the solder was hardly dissolved into the leachant of fluoroboric acid (Figure 2a). Therefore, to obtain the dissolution of solder, perhaps some oxidizing agents would be needed to oxidize solder to generate metal ions. The commonly used oxidants, such as H_2O_2, Fe^{3+}, Ti^{4+}, etc., were evaluated. It was observed in Figure 2 that the welded ECs fell off the PCBs by the oxidation of 0.5 mol/L of H_2O_2, whereas Fe^{3+} and Ti^{4+} hardly had influences on the dissolution of solder even the dosages of the oxidizing agents and fluoroboric acid, reaction time, and temperature were adjusted. Furthermore, H_2O_2, proceeding the dissolution of solder, was relatively stable under acidic medium, the final product of which was environmental-friendly H_2O hardly affecting the recycling of the leachant. Consequently, H_2O_2 was chosen as the oxidizing agent in this work.

Effect of H_2O_2 Concentration. To ensure the satisfying performance of oxidant, the concentrations of H_2O_2 ranging from 0 to 0.8 mol/L were examined. It is shown in Figure 3 that with increasing the concentration of H_2O_2 and prolonging the reaction time, the dissolution rates of tin and lead increased rapidly when the concentration of H_2O_2 was less than 0.4 mol/L. When 0.4−0.6 mol/L H_2O_2 was added, almost 100% of solder was dissolved. However, when the concentration of H_2O_2 was over 0.6 mol/L, the leaching rate of solder was decreased with increasing the used dosage of H_2O_2. A decrease for the leaching of solder was largely caused by the fact that excessive amounts of H_2O_2 promoted autogenous decomposition to generate more O_2, which made the easy formation of SnO, subsequently further oxidized to form highly insoluble $Sn(OH)_4$ precipitated on the surface of solder giving rise to a passive film. The stability of the passive film increases with its irreversible dehydration to SnO_2, which prevented metals from dissolving into leachant mechanically.[30,31] Moreover, the leaching of copper was unexpected observed from Figure 3c. However, the dissolution rate of copper was only less than 6% when no more than 0.4 mol/L H_2O_2 was added, which could be negligible relative to much more leaching of the solder. Therefore, 0.4 mol/L H_2O_2 can be regarded as the optimum condition.

Effect of Acid Concentration. The influences of the fluoroboric acid concentration were examined, Figure 4. With the extension of the reaction time, the higher dissolution rate of metals is observed to be achieved. Furthermore, the leaching concentrations of metals increased gradually with increasing the fluoroboric acid concentration from 1.5 to 2.5 mol/L. When the concentration of fluoroboric acid was over 2.5 mol/L with the reaction time of 35 min, almost all the tin and lead included in the solder were removed from the PCBs (Figure 4a,b). Moreover, with the extension of the reaction time, the leaching of copper was also unexpected increased (Figure 4c). However, the total dissolution rate of copper was less than 15% when 2.5 mol/L fluoroboric acid was added after the reaction of 35 min, which can be regarded as a negligible effect on the dismantling of ECs from PCBs. Therefore, the PCB samples can be

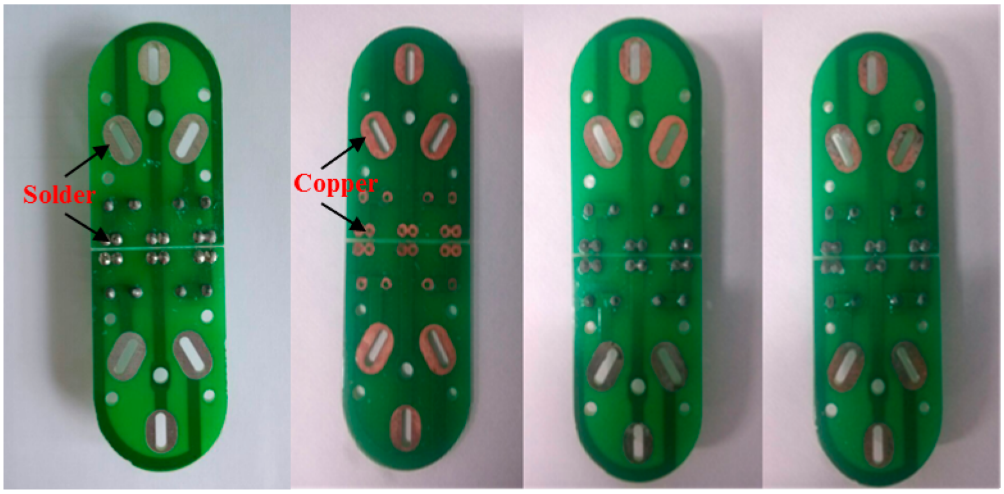

Figure 2. Influence of oxidants: (a) control, (b) H_2O_2, (c) Fe^{3+}, (d) Ti^{4+}. Conditions: $[HBF_4] = 3.0$ mol/L, $[H_2O_2] = [Fe^{3+}] = [Ti^{4+}] = 0.5$ mol/L, $t = 35$ min, $T = (20 \pm 2)$ °C.

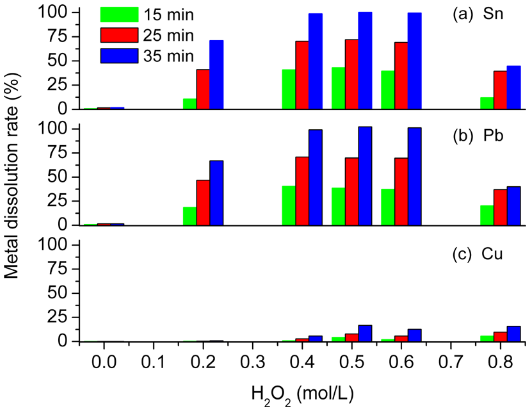

Figure 3. Effect of the H_2O_2 concentration on the dissolution of Sn (a), Pb (b), and Cu (c). Conditions: $[HBF_4] = 3.0$ mol/L, $T = 20 \pm 2$ °C.

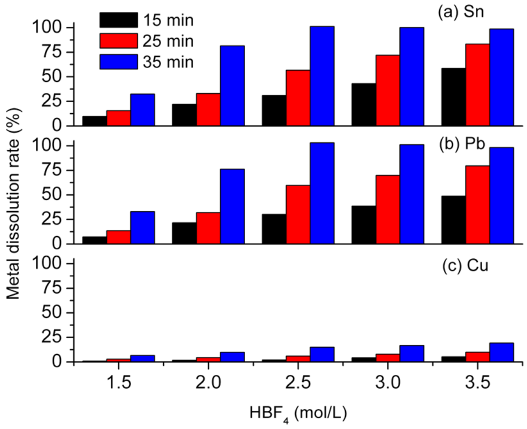

Figure 4. Effect of the concentration of HBF_4 on the dissolution of Sn (a), Pb (b), and Cu (c). Conditions: $[H_2O_2] = 0.5$ mol/L, $T = 20 \pm 2$ °C.

dismantled over 35 min in fluoroboric acid with an optimized concentration of 2.5 mol/L.

Mechanism Discussion. $H^+$and BF_4^- are ionized by fluorboric acid in an aqueous solution. In the HBF_4–H_2O_2 system, the differences of the standard electrode potential between hydrogen peroxide and tin, lead are 1.912 and 1.902 V, respectively (eqs 1−3). Therefore, the dissolution reactions of tin and lead can proceed easily at room temperature. When immersed in the fluoroboric acid leachant containing hydrogen peroxide, the oxidation−reduction reactions between solder and hydrogen peroxide occur very quickly, which could be presented by eq 4. Furthermore, the electrode potentials for tin and lead are so close (eqs 1 and 2) that they were soon stripped down from the PCBs with almost the same ratio.[28]

$$Sn^{2+} + 2e^- = Sn, \qquad \varphi_1{}^0 = -0.136\ V \tag{1}$$

$$Pb^{2+} + 2e^- = Pb, \qquad \varphi_2{}^0 = -0.126\ V \tag{2}$$

$$H_2O_2 + 2H^+ + 2e^- = 2H_2O, \quad \varphi_3{}^0 = +1.776\ V \tag{3}$$

$$2H_2O_2 + Sn + Pb + 4H^+ = Sn^{2+} + Pb^{2+} + 4H_2O \tag{4}$$

Nevertheless, with the dissolution of lead and tin into the leachant, the leaching of the copper substrate layer also occurs partly, because the difference of standard electrode potential between hydrogen peroxide and copper is 1.439 V (eqs 3 and 5). Therefore, the reactions between copper and hydrogen peroxide in the fluoroboric acid leachant could be expressed by eq 6.

$$Cu^{2+} + 2e^- = Cu, \qquad \varphi_5{}^0 = +0.337\ V \tag{5}$$

$$H_2O_2 + Cu + 2H^+ = Cu^{2+} + 2H_2O \tag{6}$$

Generally, for a chemical reaction: $aA + bB +... = cC + dD +....$

The change of Gibbs energy for the reaction is expressed as eq 7:

$$\Delta G_r = \sum v_iG_i(\text{products}) - \sum \nu_iG_i(\text{reactants}) = (cG_C + dG_D + ...) - (aG_A + bG_B + ...) \tag{7}$$

And the equilibrium constant K is expressed as eq 8:

$$K = \frac{[C]^c[D]^d...}{[A]^a[B]^b...} = e^{-\Delta G_r/RT} \tag{8}$$

where [X] is the activity of species A, B, C, D, and so on; x is the stoichiometric coefficient of species X in the reactions (a, b, c, d, ...); R is the gas constant, 1.987 cal/(K·mol) = 8.314 J/(K·mol); T is the reaction temperature, K.

The thermodynamic Gibbs energy functions for a chemical reaction is calculated as the difference between the products and reactants via eq 7. The equilibrium constant K can be calculated via eq 8. The analysis results of reactions 4 and 6 at the temperature range from 0 to 50 °C by the HSC Chemistry, Version 5.0 software are shown in Table 1 and 2, respectively.

Table 1. Analysis Results of Chemical Reaction 4

T (°C)	ΔG (kcal/mol)	K
0.000	−181.755	2.730×10^{145}
10.000	−181.640	1.625×10^{140}
20.000	−181.515	2.161×10^{135}
30.000	−181.385	5.976×10^{130}
40.000	−181.251	3.215×10^{126}
50.000	−181.116	3.169×10^{122}

Table 2. Analysis Results of Chemical Reaction 6

T (°C)	ΔG (kcal/mol)	K
0.000	−69.510	4.167×10^{55}
10.000	−69.269	2.948×10^{53}
20.000	−69.028	2.926×10^{51}
30.000	−68.789	3.947×10^{49}
40.000	−68.553	7.039×10^{47}
50.000	−68.319	1.617×10^{46}

Within the temperature range from 0 to 50 °C, the change of Gibbs free energy of reaction 4 is much less than zero. Generally, the change of Gibbs free energy of −10.1 kcal/mol was set as a boundary,[32] when $\Delta G < -10.1$ kcal/mol, the reaction in eq 4 can be thought to occur spontaneously to the right. In addition, the equilibrium constant K is relatively big enough, indicating that the reaction in eq 4 can react more thoroughly.

Compared the Gibbs free energy change and equilibrium constant K value of eq 6 with eq 4, the reaction in eq 4 can also be thought to occur spontaneously to the right. However, the reaction degree of eq 6 is much lower than that of eq 4, namely only little copper is dissolved into the leachant, which is correspondent to the above results (Figures 3 and 4). Therefore, according to the foregoing analysis, to eliminate the influence of copper in the leachant, excess solder can be added to displace the dissolved copper ion (eq 9) for the reason that the standard electrode potential of Cu is far less than that of lead and tin (eqs 1, 2, and 5).

$$Sn + Pb + 2Cu^{2+} = 2Cu + Pb^{2+} + Sn^{2+} \qquad (9)$$

Examination of Waste PCBs. To verify the effectiveness of novel selective desoldering separation for dismantling of ECs from the PCBs, the actual waste PCBs samples disassembled from liquid crystal display (LCD) of a waste computer were also examined (Figure 5). After the whole PCB was leached under the suitable condition for 35 min, the ECs were collected from the bottom of the leaching vessel, consequently washed, and dried. The results showed that almost all the soldered "legs" were freed from the ECs (Figure 5b). In addition, the released ECs were unaffected by examining the external feature, such as color, symbols, characters and letters, etc. outside the ECs. Furthermore, the leaching behaviors of the solder and copper were also investigated (Figure 6). A large amount of tin and lead (total accounting for 6% in PCBs[33]) were dissolved with prolonging the reaction time, and only less copper (accounting for 20% in PCBs[33]), was leached out. The results indicate that tin and lead could be dissolved selectively by this novel process, which had little corrosion to other metals.

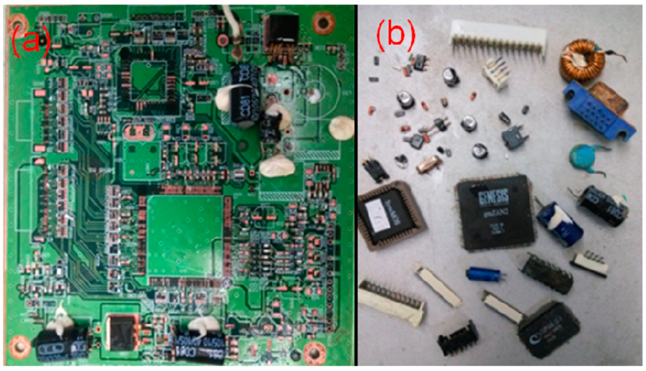

Figure 5. Copper-coated laminate (a), and dismantled ECs (b). Conditions: [HBF_4] = 2.5 mol/L, [H_2O_2] = 0.4 mol/L, t = 35 min, T = 20 ± 2 °C.

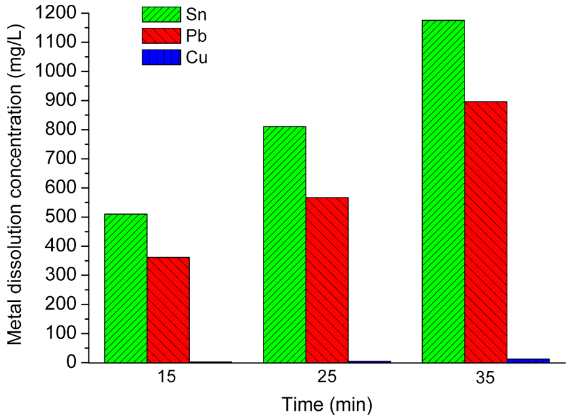

Figure 6. Leaching behavior of solder and copper in the actual waste PCBs. Conditions: [HBF_4] = 2.5 mol/L, [H_2O_2] = 0.4 mol/L, T = (20 ± 2) °C.

CONCLUSION

A novel process for selectively desoldering separation of tin–lead alloy for dismantling of the ECs from PCBs was reported. The influences of added oxidants, hydrogen peroxide dosage, acid concentration, and reaction time on the leaching behavior of tin, lead, and copper were examined. Both thermodynamic analysis and actual waste PCBs experimental research have proved this method feasible. The solder had a good leaching behavior in the leachant of fluoroboric acid containing H_2O_2. The concentration of H_2O_2 had distinct effects on the leaching of metals. When the concentration of H_2O_2 was less than 0.4 mol/L, the leaching concentration of tin and lead was increased with increasing the H_2O_2 dosage. When 0.4–0.6 mol/L H_2O_2 was added, almost all the solder were dissolved. When H_2O_2 was more than 0.6 mol/L, the leaching rates of tin and lead decreased with increasing H_2O_2. Therefore, 0.4 mol/L of H_2O_2 could be regarded as the optimum condition. The leaching concentration of metals increased gradually with the increased concentration of HBF_4 from 1.5 to 3.5 mol/L, while the components on PCB samples are all disassembled within 35 min when the acid concentration exceeded 2.5 mol/L. The optimized concentration of fluoroboric acid was found to be 2.5 mol/L. The thermodynamic analysis and actual waste PCBs experimental examination verified that the solder could be dissolved selectively by this novel process via excess solder added to inhibit the effect of copper dissolution if necessary.

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Notes

The authors declare no competing financial interest.

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