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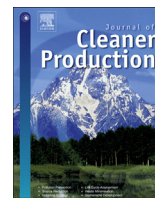Haccuria, E.; Ning, P.; Cao, H.; Venkatesan, P.; Jin, W.; Yang, Y.; Sun, Z.

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Effective treatment for electronic waste - Selective recovery of copper by combining electrochemical dissolution and deposition

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ABSTRACT

The difficulties for selective metal recovery from electronic waste are induced by the high complexity of such waste stream and complicated purification procedures. In this research, an electrochemical stimulated method is demonstrated to selective recover copper from an industrially provided electronic waste. More specific, one-step selective copper recovery was achieved by combining electrochemical dissolution and deposition in ammonia-based electrolyte. In the process, copper instead of other impure metals from the electronic waste is selectively dissolved and electrochemically enhanced at the anode. On the other side of the cell, pure copper was electrodeposited at the cathode. The current efficiency was found to be significantly influenced by the applied current density if galvanostatic process was facilitated. Both temperature and copper concentration in the electrolyte were found to have positive effects on the current efficiency. In a well-controlled process, copper of 99.56 wt% purity can be obtained which can be further improved by preventing potential oxidation on the electrodeposited layer. This research provide a possibility for selective copper recovery in one-step by substantially decreasing the purification cost and simplifying copper recovery procedures from electronic waste.

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1. Introduction

Waste electric and electronic equipment (EEE) has been increasing substantially worldwide in the last decades. It is considered to be one of the fastest growing solid waste streams and increases at a rate of around 10 million tons in the EU (Henckens et al., 2014; Nelen et al., 2014) with a world increasing rate of 20–50 million tons annually (Sun et al., 2017). Since a number of WEEE streams are classified as hazardous waste, its management and recycling is an important issue of worldwide environmental concern (Henckens et al., 2014; Nelen et al., 2014). On the other hand, significant content of valuable metals, including copper, silver, gold, makes WEEE a strategically important secondary resource. According to the new WEEE directive issued in 2012, WEEE export has been clearly forbidden (EU, 2012). However, in accordance to the current technological situation, a large number of WEEE streams are not properly treated with a low recycling rate

globally. The effective recycling rate (average) in the EU is only 37% in 2013 and 28% in China in 2015 according to the up to date reports although the rate is not in line with the management targets and some EU member countries mitigate higher recycling rates than the others (Baldé et al., 2015; Zhang, 2016). There is still significant room to be improved. With the recycling rate, a large fraction of the recycled WEEE is with relative high-quality which means small fraction of less valuable metals and high content of valuable metals (e.g. low value Fe comparing with high value Cu), insignificant compositional variation and stable material supply. When the WEEE is low-quality with low quantity of valuable metals, complex composition and phase contents, the recycling becomes technically difficult with high processing cost and potentially induces significant secondary pollution.

In literature, the processing technologies can be categorised into three types, i.e. pyrometallurgy, hydrometallurgy and biometallurgy. Prior to metallurgical processing, a variety of physical/mechanical processes need to be applied to preliminarily separate end-of-life electronic products into a number of streams (Kahhat and Williams, 2009; Nnorom and Osibanjo, 2008; Widmer et al., 2005). In line with the compositions, materials can be further

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extracted via aforementioned metallurgical processes. A typical metallurgical process of WEEE recycling is based on the principle of pyrometallurgy which needs to burn the organic materials at high temperature, smelt/reduce the targeting metal and oxides. After further being refined or physio-chemically treated, metallic or salt products can be recovered (Bigum et al., 2012; Flandinet et al., 2012; Ni et al., 2012; Pareuil et al., 2010; Tange and Drohmann, 2005; Tuncuk et al., 2012).

A hydrometallurgical process is usually near to room temperature or a high temperature under pressure and it is considered to be potentially effective if WEEE is with complex composition and minor calorific value (Fleming, 1992; Gao et al., 2017; Ha et al., 2010; Havlik et al., 2011; Jha et al., 2001; Kim et al., 2011; Shuva and Kurny, 2013; Sun et al., 2015b). In the process, shredding and leaching are commonly introduced and the targeted elements together with the impurities are usually leached into a water based solution. With further purification and separation of the impurities, products can be further recovered. In a typical hydrometallurgical process, it is portable to facilitate metal recovery even with low concentrations through a range of technologies, e.g. sedimentation, solvent extraction and recrystallisation. Although it still needs to include a step of waste water treatment and the productivity is usually lower comparing with a pyrometallurgy process (Auer et al., 1998; Cui and Zhang, 2008; Reck and Graedel, 2012; Sum, 1991), it is still considered to be a technology of flexible, high recovery selectivity of critical metals and potentials of eliminating secondary waste (e.g. waste water). On top of the technological issues of electronic waste recycling, its effective management is also of great importance. With a proper model, the criticality and recyclability of different metals in electronic waste can also be identified (Sun et al., 2016a; Zeng and Li, 2016). It can be noticed that copper as one of the main values in electronic waste constitutes the importance for an electronic waste stream being considered for effective recycling.

Copper is among the most important/valuable metals in WEEE and it is commonly found in both mono-streams or relatively 'clean' WEEE, for instance, copper wires, printed circuit boards (PCB) (Cucchiella et al., 2016), waste mobile phones (Zhang and Xu, 2016), and complex electronic waste, such as shredding residue of mixtures of different end-of-life products or waste from different streams (Sun et al., 2015b). Hydrometallurgical process to recover copper usually includes steps of leaching and subsequent electro-winning (Diaz et al., 2016). The efficiency of copper recovery are highly influenced by the implemented conditions in both steps. In the leaching step, mineral acids were frequently introduced while base metals including iron, aluminum, lead etc. were all leached into the solution. Purification or solvent extraction had to be applied in order to obtain a solution ready for electrowinning (Gao et al., 2016). Direct electro-dissolution and electrowinning was tried by using ball-milled powder from waste PCB and HCl-based electrolyte solution was used to recover copper. However, high energy consumption from ball-milling and insufficient electric contact of the PCB powder which require additional effort to prepare suitable electrode were noticed (Guimarães et al., 2014). Meanwhile, it is not suitable for processing complex electronic waste. Therefore, effective copper recovery from the complex waste is still a challenge due to high content of other metals by using a hydrometallurgical process. Recently, an ammonia-based process has been developed in accordance to the treatment of such waste where copper could be selectively extracted in the leaching step while the leach solution is subjected to electrowinning in order to obtain pure copper (Sun et al., 2016b). In this research, we demonstrate a process for one-step selective copper recovery by combining these two technologies, where direct electro-leaching and -winning is facilitated.

2. Experimental

2.1. Waste sample preparation

The complex WEEE is provided by Van Gansewinkel Groep (VGG) and was characterised in accordance to the method developed in (Sun et al., 2015a). It contains a large fraction of sand, stone, glass, ceramics and plastic particles. Except copper, it contains a range of other base metals. Most metallic items are coated with different metallic or non-metallic materials and some wires are encapsulated in plastic cables or embedded between plastic boards. These diverse conditions may highly decrease the degree of liberation and the extraction efficiency. The material was shredded into size of less than or equal to 8 mm (Sun et al., 2015a). This size is defined to be a limit since further shredding will significantly increase the processing costs. A typical view of the waste after pretreatment is presented in Fig. 1 and the average composition is given in Table 1.

2.2. Electrochemical process

The electrochemical recovery process was performed in a specially designed PMMA (Poly-methyl methacrylate) cell with a wall thickness of 5 mm and dimensions of 100 × 70 × 100 mm (L × D × H) which results in a gross volume of 0.7 L. The plates were laser-cut and melted together with chloroform. The experimental set-up is shown in Fig. 1. The complex electronic waste was pressed via a stainless steel cylinder die with a pressure of 40 MPa in order to prepare pellet samples of around 2~3 g. The sample was subsequently introduced into an anode basket (Titanium) with a mesh size of 1.5 mm × 1.5 mm (Fig. 1). As it is important that the basket does not dissolve in the electrolyte, the titanium basket was plated with platinum. As the cathode, a copper plate was used, from which one side was made inert by covering with resin materials during the process. The copper electrode had a height of 100 mm and a width of 25 mm (the emerging area is 25 mm × 25 mm). The electrodes were weighted before and after each test. The electrolyte had a composition of 12 wt% ammonia and 155 g/L ammonium-sulphate. Agitation was in some cases provided by electrolyte stirring with a magnetic stirrer to improve mass transport in the solution. Electrochemical experiments were carried out with a potentialstat/galvanostat (VersaSTAT4, Princeton Applied Research) interfaced with a PC running software (VersaStudio 2.43.3). The reference electrode in this research was a leak-free Ag/AgCl electrode ($E^0 = 0.222$ V vs. SHE, 3M KCl). The pH of the electrolyte was measured during the experiment with the WTW inolab pH 7310. Composition of the liquid samples were diluted and prepared for analyses with induction coupled plasma-optical emission spectrometry (ICP-OES, PerkinElmer Optima 3000DV). 3D optical microscope (Keyence, VHX-5000) and scanning electron microscope (SEM, JEOL JSM 6500F) with energy-dispersive spectroscopy (EDS) were used for morphology characterisation when needed.

3. Results and discussion

3.1. Electrochemical behavior of the electrodes

As aforementioned, the pellet of electronic waste was used as the anode to experience electrochemical dissolution and to directly recover copper at the cathode. It is therefore important to understand the electrochemical behavior of the anode especially when the pellet is introduced. One of the important feature is the dissolution potential with which the thermodynamics can be reflected. The anodic reactions can be given as
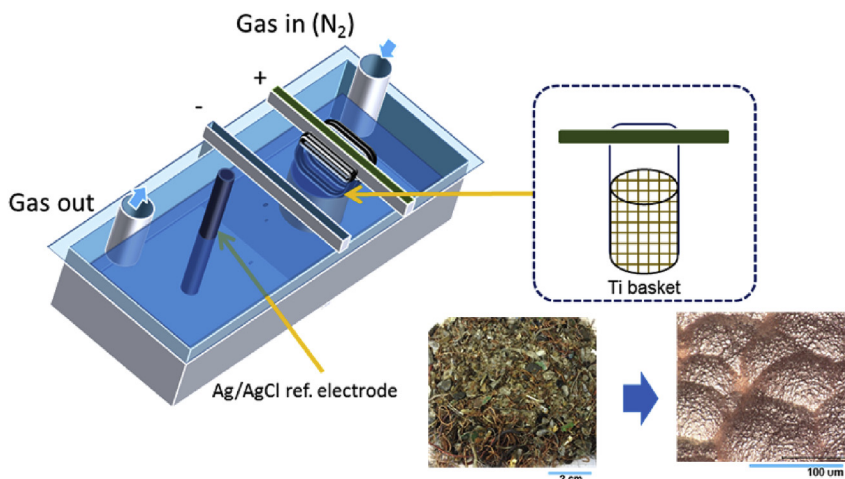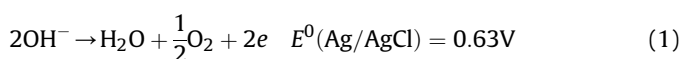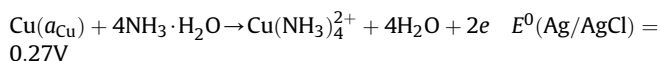
Fig. 1. Electrodissolution-electrodeposition set-up.

Table 1
Main metallic contents of the electronic waste.

Elements	Cu	Fe	Al	Sn	Pb	Zn	Ni
wt%	34.47	5.03	7.14	2.80	2.30	3.04	0.51

$$Cu(a_{Cu}) + 4NH_3 \cdot H_2O \rightarrow Cu(NH_3)_4^{2+} + 4H_2O + 2e \quad E^0(Ag/AgCl) = 0.27V$$

$$2OH^- \rightarrow H_2O + \frac{1}{2}O_2 + 2e \quad E^0(Ag/AgCl) = 0.63V \tag{1}$$

The anodic process is possibly dominated by both the dissolution of metals and the generation of oxygen, depending on the physiochemical properties of the pellet sample and electrolyte. The exact dissolution potential can be expressed by

$$E = E^0(Ag/AgCl) + \frac{RT}{nF}\ln\left(\frac{a_{Cu(NH_3)_4^{2+}}}{a_{Cu}}\right) \tag{2}$$

To understand the anode behavior, galvanostatic polarization experiments were facilitated. As given in Fig. 2, the potential range
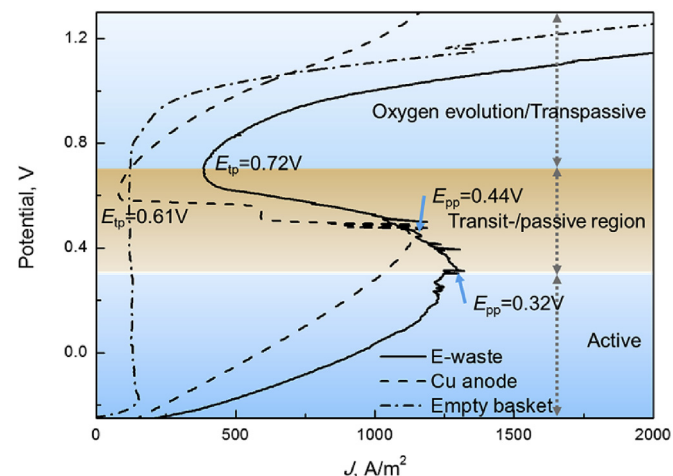

Fig. 2. Polarization curves with: a) copper anode, b) anode basket with scrap, and c) empty anode basket.

with the desired electrochemical reactions at the anode can be determined. The polarization curves were measured at 2 mV/s scanning rate in an electrolyte (copper concentration of 23 g/L) with pH equal to 9.85. The initial potential was set at −0.22 V and the final potential at 2 V vs the reference electrode. Three types of anodic materials were used for comparison in the experiments.

- A Cu rod of 120 mm diameter embedded in epoxy resin was used first, so that only the bottom section of the rod could act as active surface area
- The basket with a pellet of the complex electronic waste with the weight of 2.2 g
- The empty basket

As given in Fig. 2, there is no stable passivation region both for copper rod and the pellet of electronic waste under current experimental conditions, while the Pt coated titanium basket is clearly passive to the electrochemical leaching. Although a thin Cu_2O layer may form prior to dissolution in an ammonia leaching process without an applied electrochemical potential (Sun et al., 2015b), the absence of stable passivity region in Fig. 2 indicates that the formation of an oxide/oxy-hydroxide layer on the metal surface is less stable and dissolves once the applied potential is high. The passive layer breakdown enables further dissolution of copper, while oxygen evolution may happen simultaneously and the current efficiency becomes low. This means the dissolution of electronic waste can be enhanced by introducing an electrochemical potential with ammonia-based electrolyte while the applied potential needs to be properly regulated. When the potential is high enough, i.e. in the transpassive region, the dissolution rate of the passive layer is potential dependent similar to the active region. Concerning the metal dissolution, it can be found that the primary passive potential (peak potentials) for pure copper rod is higher in comparison to the electronic waste case, i.e. 0.44 V against 0.32 V. This indicates that the dissolution of pure copper is easier to be electrochemically enhanced. The presence of other less noble metals (e.g. Fe, Al, Ni), combining with the selective leaching characteristic of ammonia-based electrolyte, makes it easier to form a passive layer.

Fig. 3 shows the cyclic voltammetry profiles of the electrodissolution and electrowinning system. By using a small area glassy carbon as the anode (instead of the pelleted electronic waste), the electrochemical behavior of the electrolyte as well as the cathode can be clarified. On the side of positive potential, it is
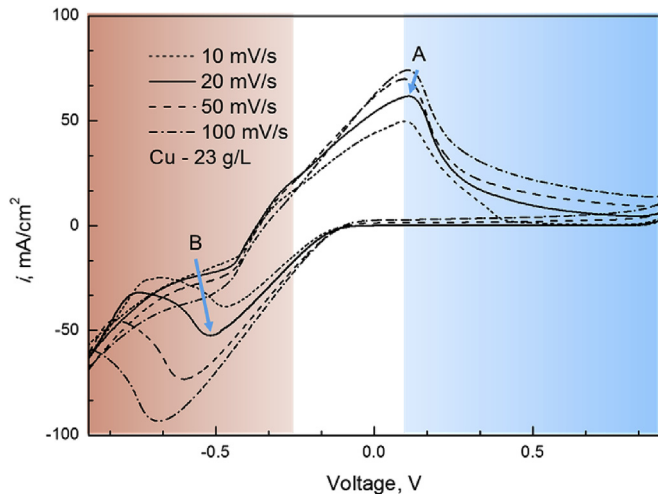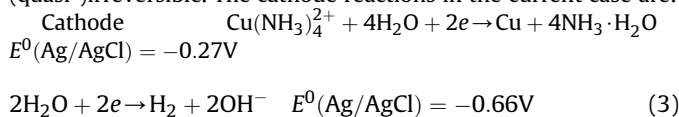
Fig. 3. Cyclic voltammetry at different scan rates.

clear that the oxidative peak (peak A) position is almost independent of the scanning rate meaning that the oxidation of copper on a glassy carbon anode is a reversible process. However, the reduction peak shifts with the scanning rate indicating that the process is (quasi-)irreversible. The cathode reactions in the current case are:

Cathode $\quad Cu(NH_3)_4^{2+} + 4H_2O + 2e \rightarrow Cu + 4NH_3 \cdot H_2O$
$E^0(Ag/AgCl) = -0.27V$

$$2H_2O + 2e \rightarrow H_2 + 2OH^- \quad E^0(Ag/AgCl) = -0.66V \quad (3)$$

The reduction of other metals are negligible due to large differences in their reduction potentials (Sun et al., 2016b). However, co-deposition of other impurity metals and hydrogen evolution is still possible caused by cathode overpotential and local concentration polarization. Hydrogen evolution is found to be the main reason for lowering the current efficiency (Oishi et al., 2007).

3.2. Direct copper recovery by combining electrodissolution and electrowinning

In order to recover copper from the electronic waste directly in the electrochemical cell of this research, the problem of conduction between the pellet of waste and anode basket needs to be tackled prior to the electrochemical recovery. According to observation, there is a large amount of metallic phase including metal wires and it is possible to improve the conduction by using the metallic phase as the structural frame and compressing with a certain pressure. Three prototypes were therefore tested for understanding the effect of loading method. In the first prototype, the electronic waste was put in the basket and loss contact of the material with the basket was assumed. To improve the contact in prototype 2, the waste was compressed with a pressure of 40 MPa in a steel die before it was put into the basket. In the third prototype, further improvement was done by loading a plastic rod to press the pellet with a constant pressure through the electrochemical processing. During electrowinning, there are usually two types of electrochemical processes, i.e. potentiostatic and galvanostatic, in order to obtain pure metal on the cathode. Although potentiostatic electrowinning is frequently used for fundamental research, galvanostatic electrowinning is mostly used in a practical case with which it is easier to determine/control the energy consumption. In this research, recovery selectivity is defined to be the key parameter to ensure the product quality or the impurity level in the electrowon copper. By using an ammonia-based electrolyte, it has been found

that selective leaching is possible, especially for copper recovery (Sun et al., 2015b) and the principle is assumed to be applied in this electrolysis process. Because of the high potential of selective copper recovery, energy consumption becomes important to optimise the electrochemical process and therefore galvanostatic method was adopted. Additionally, the loading prototypes were compared when ammonia-based electrolyte was used, it is clear that the third prototype can significantly improve the current efficiency (Fig. S1). The current efficiency is calculated by

$$\eta = \frac{m_{Cu}}{M_{Cu}} \cdot \frac{nF}{It} \quad (4)$$

where m_{Cu} is the weight the electrodeposited copper, M_{Cu} is the molar weight, n is the transferred electron number, F is the Faraday constant, I is the supplied current in the cell and t is the time (the electrodeposition is galvanostatic).

3.2.1. Effect of current density

Current density is usually a key factor that determining the electrochemical behavior of the cell in a galvanostatic process. In accordance to the electrode reactions, it requires enough electrons being transferred in order to ensure effective dissolution at the anode as well as effective electrodeposition of copper from the solution at the cathode. Based on Ohm's law, potentials at the electrodes is corresponding to the applied current density and their correlation reflects the resistance evolution during the electrochemical process. Fig. 4 gives the relationship between the applied current density against the measured starting voltage between the electrodes and the linear correlation indicates the experimental conditions being well controlled. However, the current efficiency shows clearly peak values in the range between 300 and 450 A/m² of the current density. It is directly related to the electrochemical behavior at both electrodes (Figs. 2 and 3). When the current density is too low, the supplied electrons are insufficient to ensure effective electrodissolution and electrowinning as well as overcoming resistance of the cell. The electrodissolution is not fast enough to keep a stable copper concentration in the electrode while electrowinning may dominate the process which has already been found to have a low current efficiency (Oishi et al., 2007). On the other hand, if the supplied current is too high, i.e. the potential reaching a level of dissolution passivation or even oxygen evolution at the anode and at the same time hydrogen evolution at the cathode, the supplied electrons used for selective dissolution and
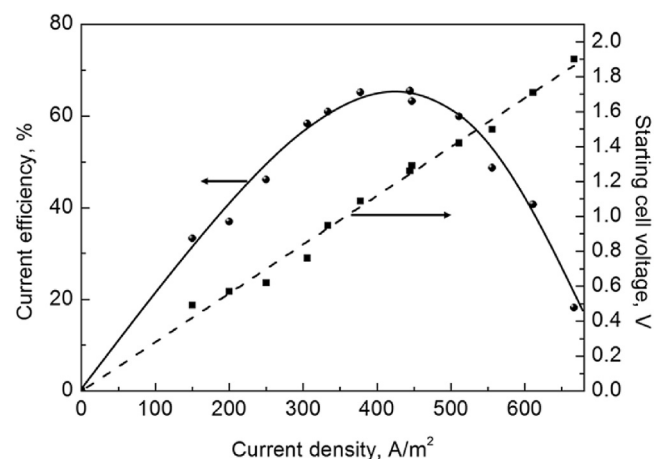

Fig. 4. Current efficiency vs current applied for electrowinning (concentration kept with 40 g/L at 40 °C).
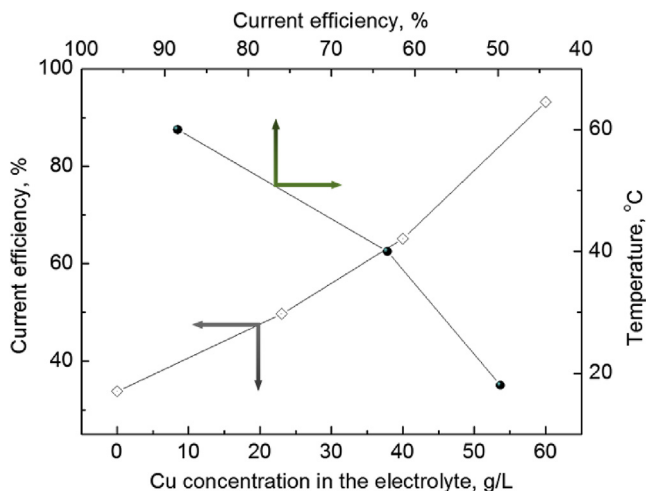
Fig. 5. Effect of temperature and copper concentration in the electrolyte on the current efficiency.

electrodeposition of copper can be severely decreased. This will deteriorate the electrochemical situation to decrease the current efficiency.

3.2.2. Effect of temperature

Temperature is believed to have a positive effect on the current efficiency by increasing the kinetic conditions including mass transfer and diffusion of ions from the solution through the stern layer and diffusion layer. However, the ammonia partial pressure increases significantly with temperature since ammonia-based electrolyte is used. As given in Fig. S2, the vapour pressure of ammonia is around 1.5 to 2 times of that for water. A proper operation temperature is then critical for ensuring the effectiveness of the process. As already realised in literature, depletion of copper ions in the solution or near to the cathode may decrease the local electrochemical efficiency which is directly relevant to the current

efficiency (Guimarães et al., 2014). The concentration of copper during the electrochemical process was inspected. As given in Fig. S3, it is noticed that the deviation of copper concentration in the electrolyte is around ± 0.1 g/L (or 0.02 g in 200 ml solution). It is therefore assumed that the copper concentration is stable with minor effect on the electrochemical process. Fig. 5 shows the effect of temperature on the current efficiency and a clear increase can be realised. In the experiments, copper concentration was kept at 40 g/L and current density of 350 A/m^2 was applied. At 60 °C, the current efficiency can be improved to be around 90%. However, a lower temperature e.g. 40 °C is suggested due to the high vapour pressure of ammonia bringing operational issues. The efficiency is possibly improved by changing other parameters, for instance, the electrolyte.

3.2.3. Effect of copper concentration

As aforementioned, copper concentration in the electrolyte can influence the current efficiency during an electrowinning process. With the same principle, copper concentration is believed to be an important factor to stimulate/improve the electrochemical conditions. In order to investigate the effect of copper concentration, the operation temperature was kept at 40 °C and the applied current density was set at 350 A/m^2. Copper ions in the electrolyte may affect both the anode and cathode processes. At the cathode, it is obvious that more Cu^{2+} ions are ready for being deposited and the slope at the interface of concentration polarization can be larger than that for a low copper concentration electrolyte. Mass transfer or the kinetics in general can be improved and subsequently charge transfer induced copper electrodeposition becomes more competitive than other reactions (e.g. hydrogen evolution) when the copper concentration is increased. On the other hand, the presence of large amount of Cu^{2+} ions in the solution may chemically enhance the dissolution of metallic copper at the anode especially at the early stage of a current being applied. The reactions can be given as
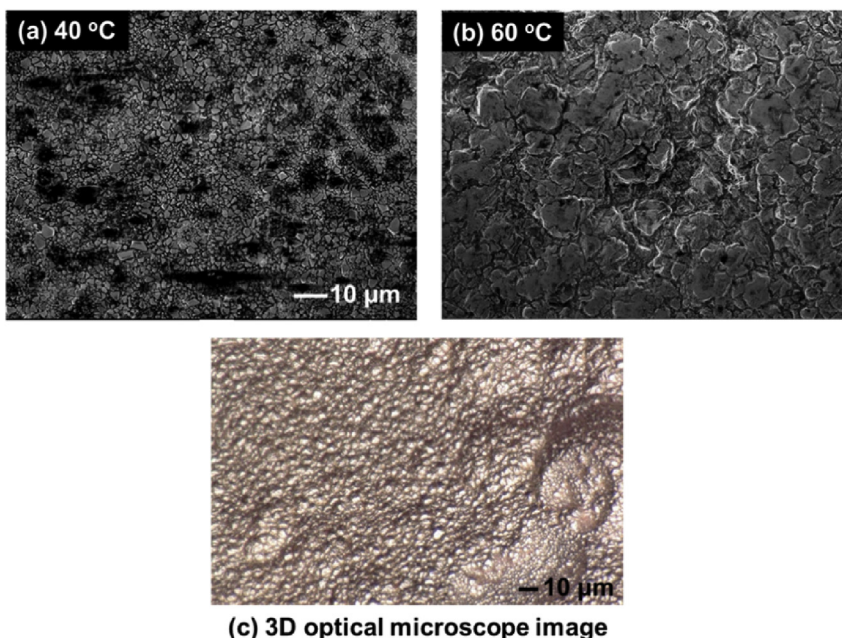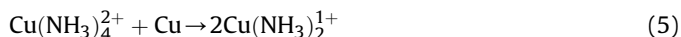
$$\mathrm{Cu(NH_3)_4^{2+} + Cu \rightarrow 2Cu(NH_3)_2^{1+}} \tag{5}$$

Fig. 6. Morphologies of the electrodeposited copper in this process at different temperatures (a) electrodeposited copper at 40 °C, (b) at 60 °C and (c) 3D optical microscope image at 40 °C (350 A/m^2, 40 g/L copper concentration, time for 1 h).

The dissolution rate at the anode can be simultaneously increased at the early stage. Therefore there are two types of effects (i.e. at both the anode and cathode) on the electrochemical process by adjusting the copper concentration. As shown in Fig. 5, significant improvement on the current efficiency is noticed, especially, that it is increased to be around 90% when the copper concentration is 60 g/L in the ammonia-based electrolyte.

3.2.4. Characterisation of the electrodeposited copper

After 1 h electro-dissolution and electrodeposition, pure copper can be obtained at the cathode. The morphology of the electrodeposited copper can be characterised. Fig. 6 provides the SEM images at different temperatures. The surface of the electrodeposited copper was found to be adequately uniform and slightly rough. A clear increase of the grain size was observed when the temperature was increased from 40 to 60 °C. Fig. 6 (c) gives the 3D optical microscope image of the deposited copper at 40 °C showing directly the surface roughness. With further electrochemical processing for totally 2 h, the composition of the post anode was analysed and given in Table S1. According to the mass balance (2.36 g pellet and 1.51 g post anode weight) and weight of the obtained pure copper (0.73 g) on the cathode, the recovery of copper can be noticed to be 85.9%. Energy consumption according to Equation S(1) can be obtained to be 2.33 kWh/kgCu (the average voltage for a fixed current density 350 A/m^2 was measured to be 1.622 V when the copper concentration in the electrolyte was kept at 40 g/L at 40 °C). The purity of the product was measured by ICP-OES after peeling off and acid dissolution of the copper layer from the cathode. As shown in Table S1, the purity is 99.56 wt% with a range of minor impurities and there is 0.245% of impurity elements not detected. "The rest" is believed to be oxygen due to oxidation of the surface after electrodeposition since the pealing process was operated in ambient condition. Practically it may be avoided by passivation (Herrasti et al., 2007). This research aims to prove the possibility of selective copper recovery by applying an electrochemical cell and detailed investigation on optimisation of the process will be provided in next stage.

4. Conclusions

Direct recovery of copper from an industrial provided complex electronic waste through a process combining electro-dissolution and electrodeposition, was achieved. In one cell/one step, copper could be effectively recovered to obtain pure copper. The effect of different parameters on the process current efficiency during copper recovery was investigated by using an ammonia-based electrolyte. The conclusions can be drawn as follows:

(1) Galvanostatic polarization experiments show that there is no stable passivation region both for copper rod and the pellet of electronic waste under current experimental conditions, while the Pt coated titanium basket is clearly passive to the electrochemical leaching. The dissolution of electronic waste can be enhanced by introducing an electrochemical potential with ammonia-based electrolyte while the applied potential needs to be properly regulated. Electrowinning of copper is a (quasi-)irreversible process while copper dissolution at the anode indicates reversible features electrochemically.

(2) Clearly peak values of current efficiency were found in the range between 300 and 450 A/m^2 of the current density. With a low current density, the supplied electrons may be insufficient to ensure effective electrochemical process and to keep a stable copper concentration in the electrode. With a high supplied current density, especially when the potential reaches a level of dissolution passivation or even oxygen

evolution at the anode and at the same time hydrogen evolution at the cathode, the supplied electrons used for selective dissolution and electrodeposition of copper can be severely decreased.

(3) Temperature has a positive effect on the current efficiency. At 60 °C, the current efficiency can reach more than 90% in current conditions. It needs to at the same time consider the operation conditions including vaporisation of ammonia since its vapour pressure increase significantly with temperature. Practically, 40 °C is suggested to ensure both requirements.

(4) Pure copper can be obtained with a purity of 99.56 wt% which can be further improved by prevent potential oxidation on the electrodeposited layer. A clear increase of the grain size was observed at elevated temperatures.

Acknowledgements

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.jclepro.2017.03.112.

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