

Electrochemical stripping of gold from Au–Ni–Cu electronic connector scrap in an aqueous solution of thiourea

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The electrochemical behaviour of a gold-plated connector from electronic scrap in aqueous thiourea solution has been investigated. An attempt was made to recover gold from scrap in a nontoxic thiourea medium by an electrochemical method rather than the traditional cyanide process. Linear sweep voltammetry indicated that thiourea extraction of gold is more efficient in acidic solution than in neutral and alkaline. Hydrochloric acid is preferable to control the pH of the solutions; the optimum concentration of thiourea is 2.5% (0.33 M). Analysis of voltammetric data yielded a critical potential (0.40 V vs SCE), which is the upper limit for significant extraction of gold from scrap. Higher potentials should be avoided in practice to prevent decomposition of thiourea and its passivation of the scrap. Electrolysis at constant potential indicated that gold was extracted selectively in the range 0.20–0.30 V vs SCE. Electrolysis at potentials either less than 0.15 V vs SCE or greater than 0.40 V vs SCE is not recommended, because of slow dissolution in the former and severe passivation in the latter.

1. Introduction

Although a cyanide process has long been used to extract gold from both primary and secondary resources, it is uneconomical to treat directly refractory ores because of poor extraction of gold and high consumption of reagents. The cyanide materials constitute a severe pollution problem [1].

Acidic leaching of gold by thiourea is evolving as an alternative to leaching by cyanide. Dissolution of gold in thiourea solution was reported first in 1941 by Plaskin and Kozhukhova [2] and continued in 1960 [3]. Other researchers have since investigated the dissolution of gold from metallic discs [4, 5] and ores [6, 7]. Electrochemical experiments on gold electrodes have been conducted [8, 9]. The advantages of acidic thiourea solution in the presence of an oxidant over cyanide leaching are small toxicity, greater rate of dissolution, high selectivity and smaller consumption by complexation reactions with interfering ions [10, 11]. This process seems a prospective substitute that may reach commercial application before other non-conventional lixivants [12]. The hydrolysis or degradation of thiourea by oxidation remains a critical limitation.

Both natural ores and secondary resources are main sources of gold production. Many ores are successfully extracted in thiourea solution [1, 5–7, 10, 12]. Secondary resources such as electronic scrap and electrical parts are commonly leached to recover gold in aqua regia and cyanide solutions rather than in

thiourea [13, 14]. The objective of this work was to examine electrochemical dissolution of Au–Ni–Cu connector scrap in thiourea solutions, to try to discover optimum conditions for selective stripping of gold from scrap.

2. Experimental details

A gold-plated connector from electronic scrap was taken as raw material for this work. After dissolution in aqua regia and analysis by atomic absorption spectrophotometry (Perkin Elmer 5100PC), the composition was calculated in wt % to be Au 0.55, Ni 0.36, and the balance in Cu. Examination by SEM and EPMA (Fig. 1(a), (b) and (c)) showed the connector to have a thin layer ($\sim 10 \mu\text{m}$) of gold on the surface, beneath which was a thin film of nickel ($\sim 5 \mu\text{m}$) deposited on the copper substrate. Interdiffusion was evident between these elements, especially between gold and nickel. The specimen was cleaned ultrasonically in acetone and coated with epoxy resin to produce an exposed area of 60 mm^2 for linear-sweep voltammetry. Thiourea solutions at various concentrations were prepared by dissolving recrystallized thiourea into doubly distilled water at 0.1, 0.5, 2.5 and 4.0 wt %, (0.08, 0.40, 1.9 and 3.0 M, respectively). The solution pH was adjusted with NaOH and mineral acids (HCl, H_2SO_4 and HNO_3). The freshly prepared solutions were used as electrolytes for anodic stripping using a beaker (500 ml) and magnetic stirring.

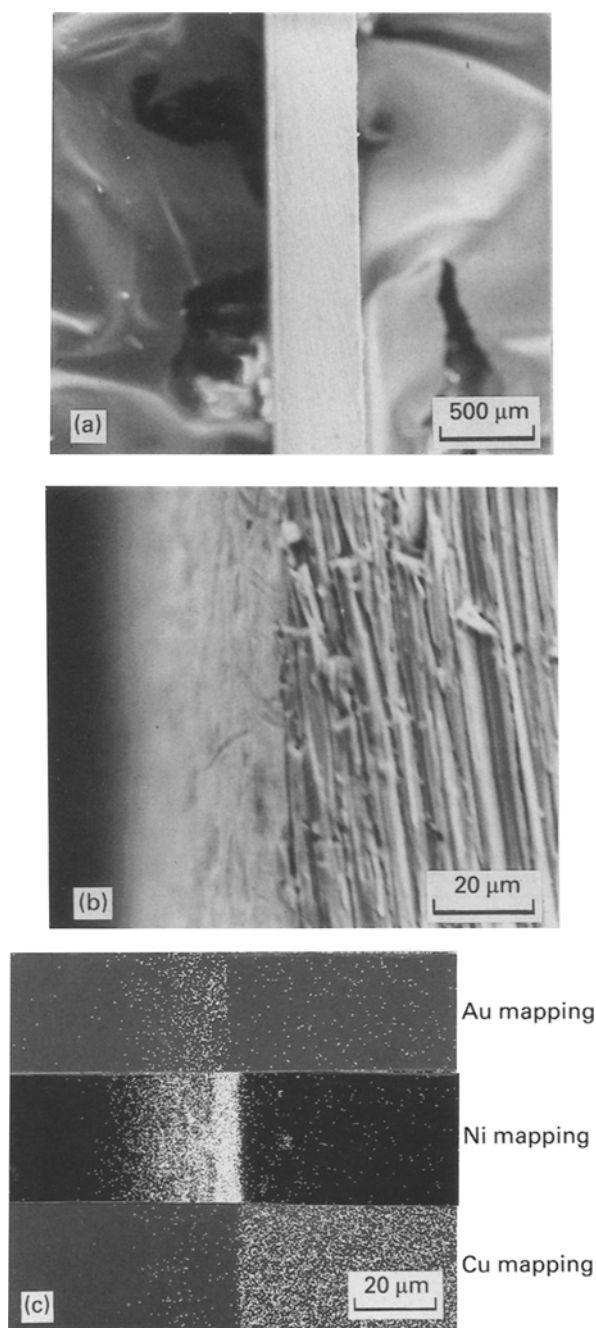


Fig. 1. (a) Lateral view of SEM micrograph of the specimen. (b) Magnification of (a). (c) EPMA mapping of Au, Ni, and Cu corresponding to (b).

Linear-sweep voltammetry of the specimen in thiourea solutions was conducted with an electrochemical analyser (BAS-100B). A graphite rod of diameter 5 mm served as counter electrode (cathode), the specimen was the working electrode (anode), and a saturated calomel electrode (SCE) with a Luggin probe was the reference electrode. Before polarization measurements, the solution was purged by purified nitrogen gas for at least 30 min; the potential was scanned from 0 to 0.700 V vs SCE at various rates to determine the anodic current.

Constant-potential electrolysis of the specimen was carried out with a potentiostat/galvanostat (Hokudo HA-320) in a cell (1 dm³) in which scrap (0.01 kg) was held in an anode basket of platinized titanium.

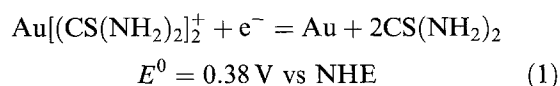
A graphite plate (40 mm × 30 mm × 2 mm³) was used as cathode. Electrolysis was conducted at controlled constant potentials 0.100, 0.200, 0.300, 0.400, and 0.500 V vs SCE, respectively. During electrolysis the current was recorded and the concentrations of gold, nickel and copper in the solution were analysed by atomic absorption spectrophotometry (Perkin-Elmer 5100PC) with respect to reaction duration. The morphology of the specimen was examined by optical microscopy and SEM.

3. Results and discussion

3.1. Effect of pH on the stripping of scrap in thiourea solution

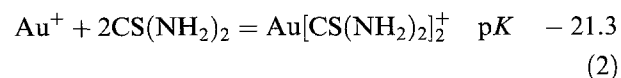
3.1.1. Effect of pH on anodic current of the linear-sweep voltammogram. From the forward scan of anodic (cyclic voltammogram) curves the variation of anodic current with potential in thiourea solutions (2.5%) at various pH is shown in Fig. 2. The pH of a freshly prepared thiourea solution (2.5%) is 7.2; this was adjusted within the range 1.4–12.0 by addition of HCl or NaOH. The anodic current in acidic solutions of thiourea was much greater than that in either neutral or alkaline solutions (Fig. 2). Hence dissolution of gold is more significant in acidic thiourea solution in an optimal range of potential. This result is illustrated by the concept of electrochemical equilibrium between gold and thiourea as follows.

The redox potential of gold (I) thiourea complex in acidic thiourea solution is [9]



Accordingly, gold is thermodynamically dissolved in acidic thiourea solutions at potentials greater than 0.38 V vs NHE (0.14 V vs SCE), the threshold potential for dissolution of gold. The potential is set either by connecting the gold anode to an external d.c. rectifier or by adding oxidants to the solutions [1–10].

The bis(thiourea) gold (I) ion indicated in Equation 1 is a stable cationic complex of which the formation constant is governed by [15]



Due to its complexing ability, thiourea is an effective extractant for gold at a potential exceeding 0.14 V vs SCE. During dissolution of gold, thiourea may suffer oxidation in successive stages to several products that fail to dissolve gold [16, 17]. The stage, and thus the dominant product, is determined by the electrochemical potential. For instance, in a solution with potentials at 0.42 V vs NHE (0.18 V vs SCE), oxidation of thiourea terminates at the first stage and forms formamidine disulphide (NH₂(NH)CSSC(NH)NH₂)

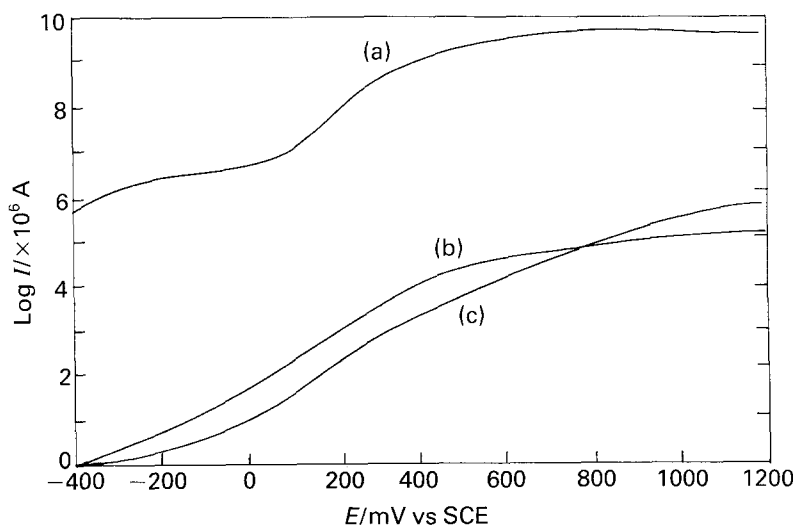
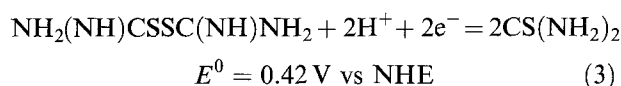
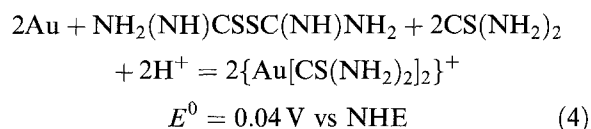


Fig. 2. Plot of current against potential for 2.5% thiourea solution with the pH at (a) 1.4, (b) 7.2 and (c) 12.

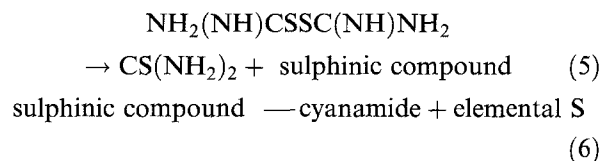
according to the reaction [18]



Presler [18] demonstrated that formamidine disulphide becomes an active oxidant on formation. The combination of Equations 1 and 3 into Equation 4 describes the dissolution/complexation of metallic gold in acidic thiourea solutions containing formamidine disulphide:



Gold dissolved readily at 0.18 V vs SCE, at which formamidine disulphide aids dissolution of gold. Hence dissolution of gold is governed by Equations 3 and 4 rather than by Equation 1. The leaching conditions should be carefully controlled in order to introduce formamidine disulphide in sufficient concentration to activate dissolution of gold instead of enabling thiourea decomposition. When the potential is much more positive than 0.18 V vs SCE (0.42 V vs NHE), there is no advantage as further irreversible oxidation of formamidine disulphide occurs. Thus degradation products such as cyanamide (NH_2CN) and elemental sulphur (S) are formed [17, 18]



Adsorption of degradation products onto the specimen makes it passive and inhibits gold dissolution. Passivity is more pronounced in both neutral and alkaline solutions as thiourea is unstable and subject to decomposition. According to Fig. 2, the anodic current in both neutral and alkaline solutions is much smaller than in acidic solution; this is consistent with the above conditions.

To extract gold efficiently, it is important to choose an appropriate range of potential in which thiourea

tends to complex with gold rather than to decompose. Degradation of thiourea can be limited to an acceptable level either by controlling the gold electrode within an optimal range of potential, or by adding a mild oxidant in the solution. Groenewald [8, 19] found satisfactory electrochemical dissolution of pure gold at overpotentials less than about 0.30 V vs SCE. Other oxidants such as Fe^{3+} and oxone are recommended to control the potential [1, 20–24].

3.1.2. Effect of pH on the rest potential. Figure 3 shows the variation with pH of the rest potential for the specimen in thiourea solution (2.5%). For pH > 10.2, the specimen was rapidly darkened and an extremely negative potential resulted. The solution became turbid for the duration of the experiment. The shift of potential to the active end resulted from the decomposition of formamidine disulphide. The suspension of the degradation product, fine sulphur powder, in solution produced the turbidity [18].

Over the range of pH from 4.3 to 10.2, the rest

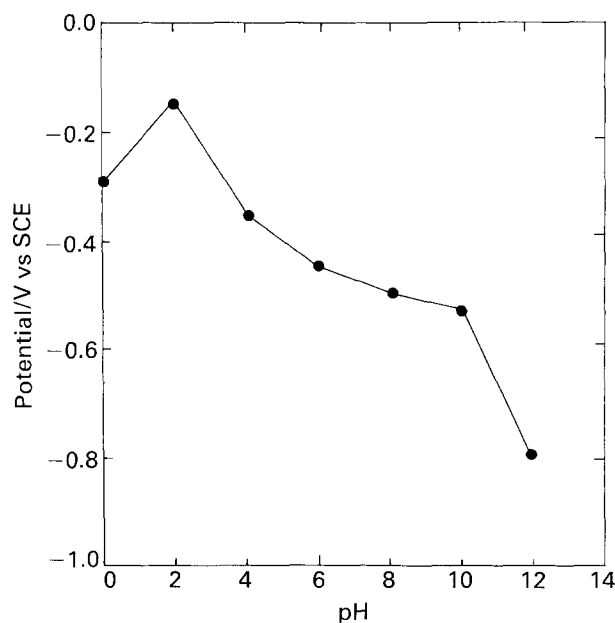


Fig. 3. Variation with pH of the rest potential of the specimen in 2.5% thiourea solution.

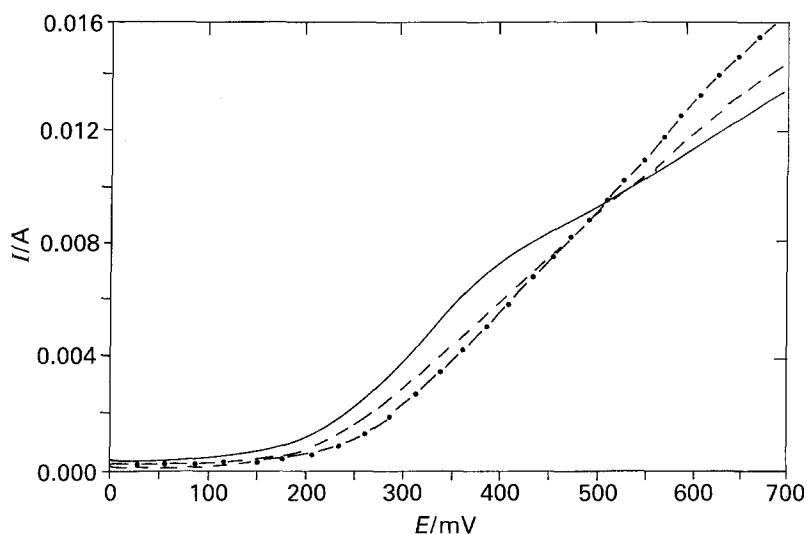


Fig. 4. The anodic linear sweep voltammograms for the scrap in 2.5% thiourea solution, where the pH has been fixed at 1.4 by HCl, H₂SO₄ and HNO₃. Key: (—) hydrochloric acid; (---) sulphuric acid; (- · - ·) nitric acid.

potential varied slightly, and only a few dark spots were observed on the specimen after several hours. Hence the rate of degradation of thiourea is slower in a neutral solution than in an alkaline one. In acidic solutions (in the pH range 0 to 4.3), the rest potential depended on the solution pH and reached a maximum at pH 2. The surface of the specimen maintained a lustrous appearance for a few days. For this reason an acidic thiourea solution is more stable and thus a better extractant for gold.

3.1.3. Choice of mineral acids. Figure 4 shows the variation of anodic current with potential for thiourea solutions (2.5%) in which the pH was controlled at 1.4 with various mineral acids. Within the potential range 0.00 to 0.50 V vs SCE (0.24–0.74 V vs NHE), the anodic current in HCl is a little greater than in both sulphuric and nitric acids. These results are consistent with those of Lodeischikov [20] and are ascribed to the aggressive character of chloride and its complexing with gold. In contrast, over the range 0.50–0.90 V vs SCE, the current was smaller in HCl than in the other acids. Presumably the passive film on the specimen was somewhat broken down and partially removed in both nitric and sulphuric acids.

Figure 4 also indicates that the anodic current was small at potentials less positive than 0.15 V vs SCE; then it increased steeply at potentials greater than 0.250 V vs SCE. Thermodynamically, in the absence of formamidine disulphide, gold dissolves in thiourea only when the potential is more positive than 0.14 V vs SCE, as indicated by Equation 1. However, in the presence of formamidine disulphide, dissolution of gold is facilitated even at potentials as small as –0.20 V vs SCE, according to Equation 4. Although the occurrence of formamidine disulphide may enhance gold dissolution, it is initiated only for potentials more positive than 0.18 V vs SCE. Consequently, potentials more positive than 0.14 V vs SCE are necessary for extraction of gold by thiourea; the process is more efficient about 0.18 V vs SCE. Kinetically, the rate of dissolution of gold is accelerated

if sufficient formamidine disulphide is present in the thiourea solution. From Fig. 4, the anodic current remains small at about 0.18–0.20 V vs SCE, as the concentration of disulphide is inadequate. This behaviour corresponds with Groenewald's result [19] that the dissolution of gold is rapid up to anodic overpotentials 0.30 V and reaches nearly the maximum rate controlled by diffusion. At greater overpotentials the possibility of decomposition of thiourea is greater and inhibits gold dissolution. Therefore large potentials should be avoided.

3.2. Influence of concentration of thiourea

Figure 5 depicts voltammograms of dissolution of gold in acidic solutions of thiourea at various concentrations. Regardless of concentration the anodic current is small (<0.001 A) at potentials less positive than 0.15 V vs SCE. At greater potentials the current varies markedly with thiourea concentration. Curves for dilute solutions of thiourea, for example at 0.1% (0.01 M) and 0.5% (0.06 M), indicate a smaller current and possible passivation in the range 0.23–0.35 V vs SCE. The curves for concentrated solutions indicate that the current increases with increasing thiourea concentration to 2.5% (0.33 M). No advantage is gained by a concentration of thiourea greater than 4.0% (0.53 M), as passivity occurs at 0.30 V vs SCE and current decreases.

This condition is delineated by Equation 3, i.e. the reversible couple of formamidine disulphide/thiourea, and the corresponding Nernst equations

$$E = E^0 + \frac{RT}{2F} \ln \frac{(\text{formamidine disulphide}) (\text{H}^+)^2}{(\text{thiourea})} \quad (7)$$

At 25°C,

$$E = 0.420 + 0.0296 \log \frac{(\text{formamidine disulphide})}{(\text{thiourea})} - 0.0592 \text{ pH} \quad (8)$$

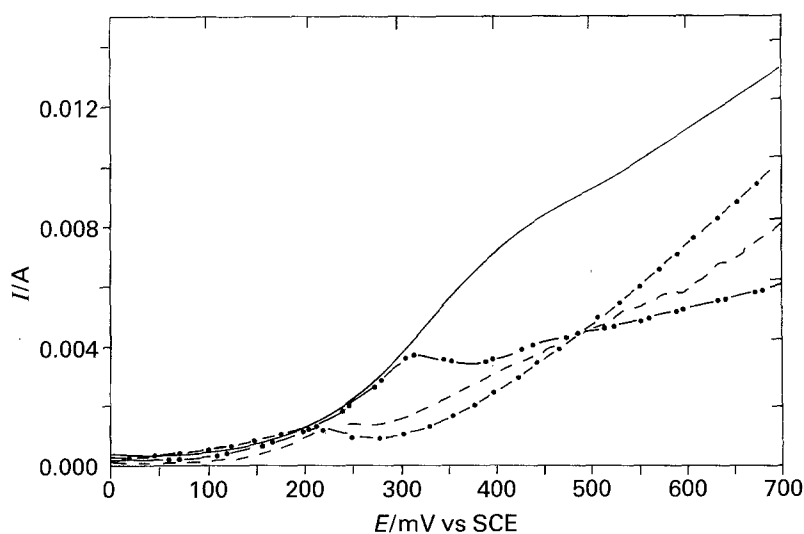


Fig. 5. The anodic linear sweep voltammograms for the scrap in the acidic solution with thiourea concentration at (— · — ·) 4.0%, (—) 2.5%, (---) 0.5% and (- · - ·) 0.1%.

For pH 1, Equation 8 becomes

$$E = 0.360 \text{ V vs NHE} + 0.0296 \log \frac{(\text{formamidine disulphide})}{(\text{thiourea})} \quad (9)$$

The magnitude of reversible potential, E , depends on the ratio of activities of formamidine disulphide and thiourea. Furthermore, the total concentration of thiourea influences the solution potential. Preisler reported that this concentration causes the potential to increase 0.03 V for each ten-fold decrease in the total concentration of any fixed ratio of concentrations of formamidine disulphide and thiourea. The conditions thus become more oxidizing on diluting the thiourea solution [18]. Therefore, passivation by sulphide is more likely in dilute solutions of thiourea, i.e. 0.1 and 0.5%.

With concentrations of thiourea greater than 4%, the rate of dissolution of gold is controlled by diffusion. Through the reverse reaction of Equation 3, thiourea becomes oxidized to formamidine disulphide, which then accumulates in the vicinity of the anode to a large concentration and is subject to

further decomposition at greater potentials. The subsequent adsorption of the decomposition products on the specimen make it passive. As a result, during the electrochemical stripping of the scrap, the concentration of thiourea is best set at 1–3% to prevent passivation.

3.3. Influence of scan rate on linear-sweep voltammogram

Figure 6 shows a linear-sweep voltammogram of scrap in solution (2.5%) of thiourea at pH 1. From Fig. 6, the anodic current was the smallest at a large rate of scan (100 mV s^{-1}) independent of potentials in the range 0.2–1.0 V vs SCE. For comparison, the anodic current increased on decreased rate of scan (10 mV s^{-1}), and eventually at a small scan rate (1 mV s^{-1}) the largest current occurred at potentials less than 0.4 V vs SCE. In another experiment in which the sweeping potential terminated at +0.20 V vs SCE, the magnitude of anodic current for various rates of scan decreased in the order: $1 > 10 > 100 \text{ mV s}^{-1}$. For this reason, at small scan

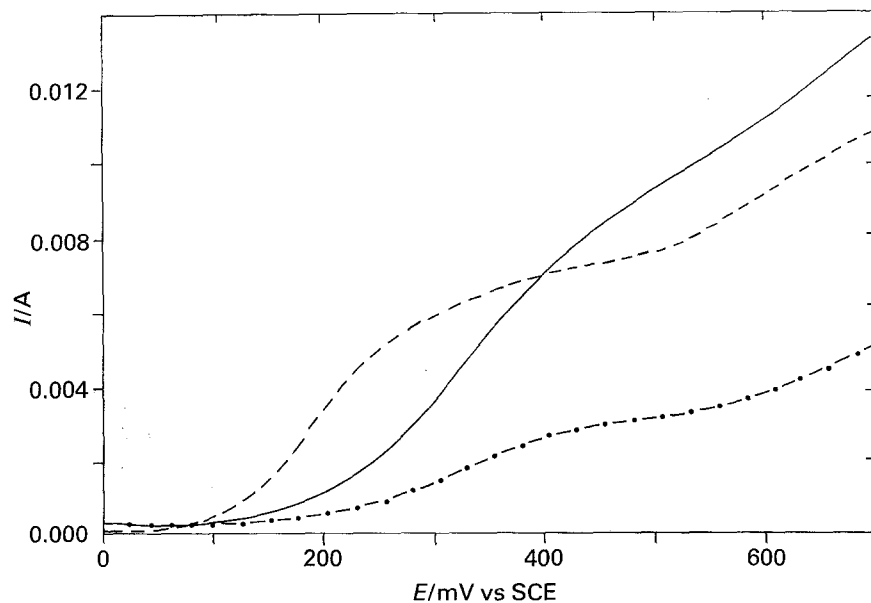


Fig. 6 The anodic linear sweep voltammograms for the scrap in 2.5% thiourea solution with the scan rates at (---) 1, (—) 10 and (- · - ·) 100 mV s^{-1} .

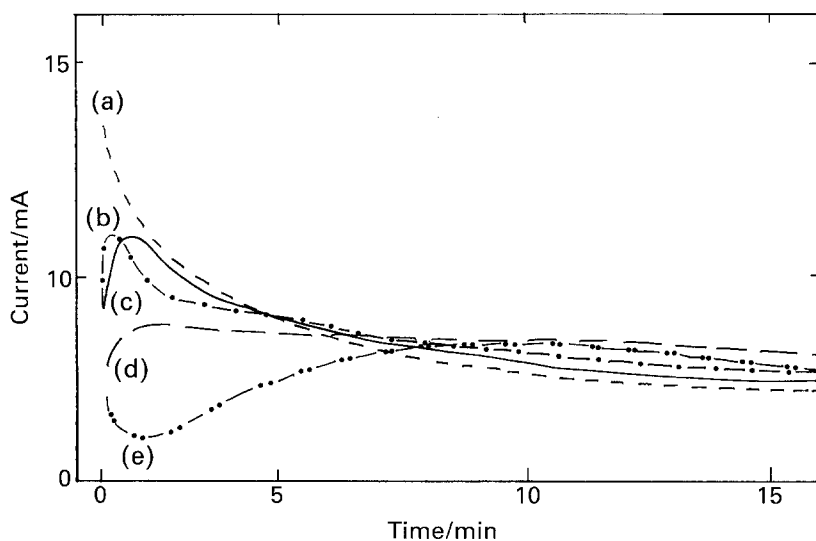


Fig. 7. Current profiles for electrolysis under a constant potential at (a) 0.50, (b) 0.40, (c) 0.30, (d) 0.20 and (e) 0.15 V vs SCE.

rates there is sufficient time for thiourea to oxidize gradually; thus sufficient formamidine disulphide forms around the specimen surfaces, and acts as both oxidant and complexing agent for dissolution of gold. In contrast, for a large scan rate, the formamidine disulphide formed on the electrode surface at small potentials immediately encounters larger oxidation potentials and is subject to decomposition. The resulting products of degradation react with the specimen to form a sulphide film that passivates the surface, and thus reduces the current.

At potentials above +0.4 V vs SCE, the anodic current for a small scan rate (1 mV s^{-1}) was smaller than that for the medium one (10 mV s^{-1}). For the case of a small scan rate the electrode experienced a greater duration of high potential during the later period of scanning. Under these conditions formamidine disulphide accumulates to facilitate further oxidation and then produces passivation.

3.4. Anodic dissolution of the scrap at constant potential

3.4.1. Variation of anodic current with time at constant potential. Electrochemical dissolution of the gold-plated connector scrap was tested at constant potentials in acidic solutions (2.5%) of thiourea. Electrolysis at constant potential was conducted at 15°C for 30 min. The anodic current was recorded against time (Fig. 7). When the electrolysis proceeded at potentials greater than +0.30 V vs SCE, the initial current was larger ($>10 \text{ mA}$) and then decreased rapidly in 5 min. For electrolysis at potentials less than 0.20 V vs SCE, the current was small ($<3 \text{ mA}$) initially, then increased gradually. For comparison, the current remained almost steady (7 mA) throughout the period during electrolysis at +0.20 V vs SCE. This result indicates that

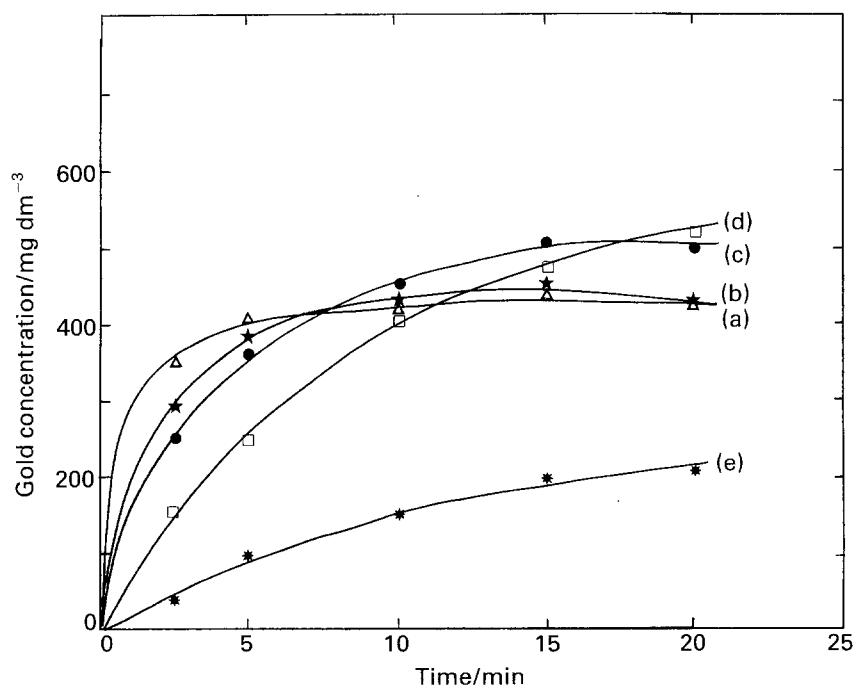


Fig. 8. Variation with time of the gold dissolution in 2.5% thiourea solution under constant-potential electrolysis with the potentials at (a) 0.50, (b) 0.40, (c) 0.30, (d) 0.20 and (e) 0.15 V vs SCE.

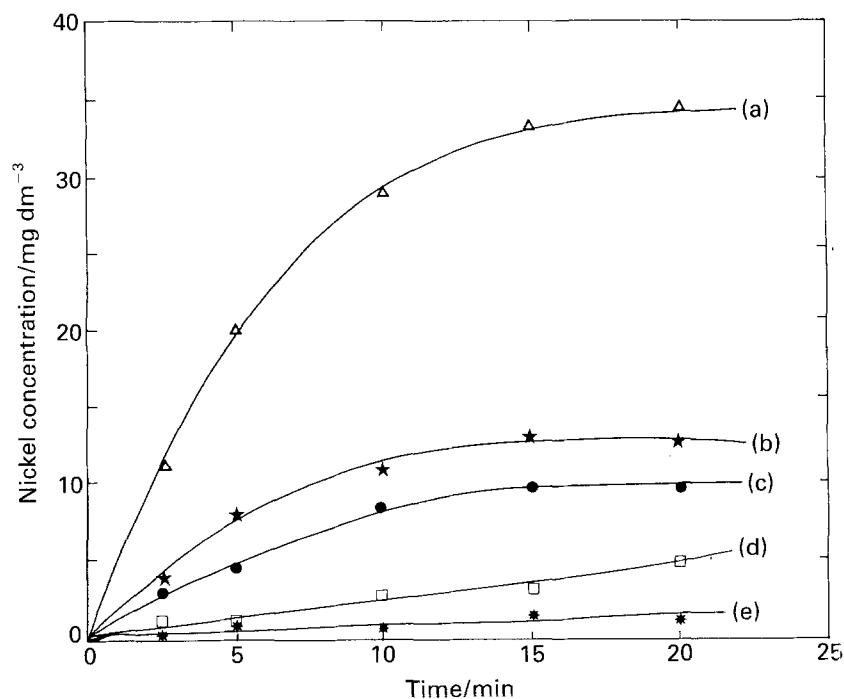


Fig. 9. Variation with time of the nickel dissolution in 2.5% thiourea solution under constant-potential electrolysis with the potentials at (a) 0.50, (b) 0.40, (c) 0.30, (d) 0.20 and (e) 0.15 V vs SCE.

electrolysis at 0.20 V vs SCE generates a steadily large rate of stripping of the scrap.

3.4.2. Analysis of extracted ions in solution. During electrolysis at constant potential, the solution was sampled periodically to monitor the concentration of the extracted ions by means of atomic absorption spectroscopy. Figure 8 shows the variation with time of the gold dissolution in thiourea (2.5%) at various potentials. The slopes of the curves indicate the rate of gold dissolution. The rate of stripping of gold is significant within 5 min and levels off rapidly for electrolysis at greater potentials, for example 0.500 and 0.400 V vs SCE. In contrast at 0.150 V vs SCE the rate is small initially but increases and then decreases gradually. At 0.200 V vs SCE, even though the initial rate of dissolution is smaller than that at 0.500 and 0.400 V vs SCE, the rate subsequently remains almost constant. This steady condition results in the most concentrated solution of gold in 20 min. Therefore, it is better to conduct the electrolysis at constant potentials of 0.200 V vs SCE, at which gold dissolution (Equations 1 and 4) dominates and degradation of thiourea (Equations 5 and 6) is negligible.

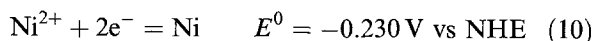
During electrolysis at potentials greater than 0.300 V vs SCE, the competition between gold dissolution and degradation of thiourea occurs. At the beginning of electrolysis, as soon as formamidine disulphide is derived from thiourea near the anode, it is readily able to react with gold [9], and no excess disulphide accumulates for degradation. This results in a large rate of leaching of gold at the beginning of tests at 0.300, 0.400, and 0.500 V vs SCE. The rapid dissolution of gold may cause local attack at the weak points of the surface layer, where the sublayer of nickel and its substrate are exposed to undergo dissolution and to form pitting. As time passes,

more disulphide is produced than is needed for gold complexation. This surplus disulphide accumulating near the anode is susceptible to further oxidation; thus a series of reactions, such as Equations 3, 5 and 6, play an important role at longer duration. Then decomposition of thiourea governs the reaction and thus decreases the rate of stripping.

In comparison of leaching morphologies, the surface layer of gold was almost completely dissolved and the nickel sublayer with its copper substrate remained in the case of electrolysis at 0.200 V vs SCE. However, in the case of 0.500 V vs SCE there are a number of pits dispersed on the surface and some film-like substances covered the unpitted regions. With the mapping of Au, Ni and Cu by microanalysis, pits are the locations where not only Au and Ni but also Cu are dissolved. The density of pits becomes greater with increasing potential from 0.200 to 0.500 V vs SCE. This is due to severe local attack of gold and nickel.

The concentration of nickel extracted into solution is shown in Fig. 9. A solution more concentrated in nickel is obtained when electrolysis is carried out at high potentials, whereas dissolution of copper is detected only at potentials greater than 0.300 V vs SCE. The concentration of copper extracted into solution is shown in Fig. 10.

The sublayer of nickel of the scrap is ready to be dissolved by extractant only when the gold surface layer has been removed. The dissolution of naked nickel is governed by



During electrolysis at a potential less than 0.150 V vs SCE, only a confined coating of gold was stripped by thiourea. Consequently, a limited sublayer of nickel was exposed to dissolution by acid, which resulted in a dilute solution of nickel.

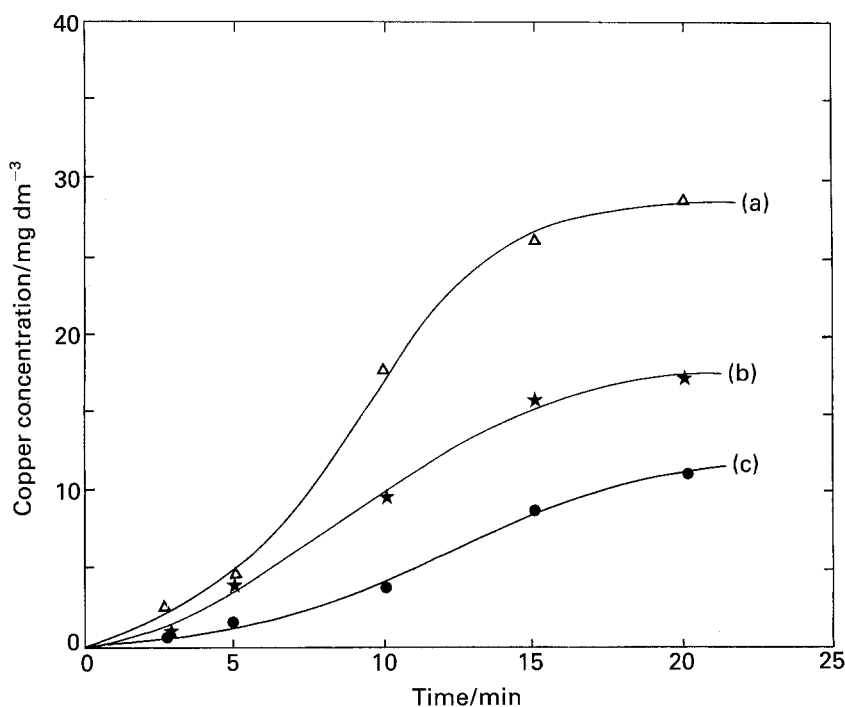
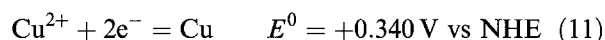
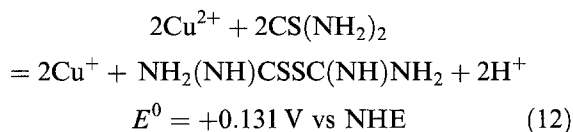


Fig. 10. Variation with time of the copper dissolution in 2.5% thiourea solution under constant-potential electrolysis with the potentials at (a) 0.50, (b) 0.40, and (c) 0.30 V vs SCE.

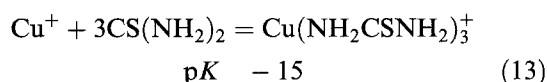
As the substrate is covered by the layers of gold and nickel, the dissolution of copper can occur only after removal of both Au and Ni. A greater possibility for copper to be exposed results when the electrolysis is carried out at high potentials. This condition facilitates exposure of copper and dissolution



As soon as copper (II) ion (Cu^{2+}) appears, it oxidizes thiourea to formamidine disulphide, which subsequently reacts with the copper (I) ion (Cu^+) to form a complex. The reactions are



and



Consequently, there is a delay between dissolution of the surface layers and copper (compare Figs 8, 9 and 10), as both the stripping of Au and Ni and the degradation of thiourea accelerate at a potential greater than 0.300 V vs SCE, and a greater concentration of copper is obtained for electrolysis at higher potentials. Nevertheless, no enhancement of gold stripping is obtained. This results from the decomposition of formamidine disulphide, formed according to Equation 12, and passivation of the substrate, as mentioned earlier. For potentials smaller than 0.200 V vs SCE, dissolution of copper is negligible for two reasons. The copper substrate is still protected by both outer layers, and copper may redeposit by dissolution of nickel.

The surface deposit of gold on the scrap metal is

selectively extracted in acidic solution of thiourea electrochemically over an optimal range of potential (0.200–0.300 V vs SCE) in 30 min. This result is in accordance with Groenewald's result [19]. If the potential is less than about 0.250 V vs SCE (0.492 V vs NHE), thiourea is oxidized mainly to formamidine disulphide in an acidic solution, rather than to degradation products.

4. Conclusions

(i) Linear-sweep voltammetry provides useful information about stripping of gold from a Au–Ni–Cu connector in thiourea solutions.

Acidic solutions of thiourea are more suitable for stripping than neutral and alkaline solutions. Hydrochloric acid is preferable to control the acidity of solutions, in which the concentration of thiourea is best fixed at about 2.5 percent (0.33 M).

The results of linear-sweep voltammetry at various scan rates indicate that efficient stripping of gold is obtained on gradual oxidation of thiourea at potentials less than 0.40 V vs SCE, the critical potential for passivation.

Formamidine disulphide, derived from oxidation of thiourea at potentials greater than 0.18 V vs SCE, enhances extraction of gold, but it is subject to decomposition and passivates the scrap at potentials greater than 0.40 V vs SCE.

(ii) Tests on electrolysis at constant potential indicate the following.

Below 0.15 V vs SCE, the rate of dissolution of gold is slow, because of formamidine disulphide is not yet formed to aid the dissolution.

At 0.20–0.30 V vs SCE, the amount of formamidine disulphide formed is almost balanced with its need for dissolution of gold; hence selective extraction of gold from scrap is obtained.

At a potential greater than 0.40 V vs SCE, excess disulphide accumulates and undergoes degradation; in this way it diminishes the rate of dissolution of gold.

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