Chemical stripping of gold deposits from different substrates

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The present paper presents the results of a study on the development and optimisation of a chemical stripping solution for the removal of gold deposits from different substrates. This stripping solution essentially contains potassium hydroxide, potassium cyanide, sodium citrate and m-nitro benzoic acid. Rate of stripping has been studied at varying concentrations of constituents; temperatures and an optimum composition derived.

Keywords: Electrodeposition, Stripping, Gold plating, Nickel plating, Electronics

Introduction

Owing to the attractive electrical and chemical properties of gold, plating of gold plays an important role in the electronics industry. The relatively high price of this metal restricts its use to those applications where its unique properties make its use essential. Gold electrodeposits find a large number of applications both decorative and functional.¹ These applications are mostly based upon its aesthetic appeal, outstanding electrical conductivity, nobility, resistance to different environments and solderability. Gold is electrodeposited on copper, stainless steel, nickel, aluminium, brass, palladium and silver substrates depending on its end use.

With increasing applications of the metal, it becomes essential to remove defectively plated gold such as poorly adherent gold deposits resulting from poor pretreatment of base metal or passivation of undercoat, poor coverage gold deposits, etc. A few solution compositions are mentioned in the literature for the removal of gold deposits. Ludwig² after reviewing theoretical aspects of stripping concludes that an ideal stripping solution should have the properties of being rapid in its removal action, no or low basis metal attack and the possibility of recovering the dissolved gold. In a recent contribution on stripping of precious metals, Jones³ discusses chemical stripping of gold using acid mixtures or hydrogen peroxide solutions both of which require fume exhaust systems, and anodic stripping using cyanide solution, an electrolytic process. Discussion on the anodic dissolution behaviour of gold in acid solutions,⁴ alkaline solutions⁵ and thiourea⁶ solutions are in the literature, and a very recent short review of stripping processes by Gabe also includes gold strippers.⁷ But there are no data available on stripping rate from any of these solutions. The present work is undertaken to investigate the rate of removal of gold deposits from different substrates by chemical immersion stripping using a suitably formulated solution.

Experimental

Cold rolled copper sheets $(2.5 \times 7.5 \times 0.1 \text{ cm})$ were mechanically polished and degreased with trichloroethylene and electrocleaned cathodically for 2 min at in alkaline solution composed of sodium 4 A dm^{-2} 20 g dm^{-3} , trisodium carbonate orthophosphate 9 g dm⁻³ and sodium hydroxide 7 g dm⁻³ at 343 K using stainless steel as anode. The specimens were then washed in running water, followed by a 10 s dip in 5%v/v H₂SO₄ solution, with subsequent thorough washing and a final rinse in distilled water. Nickel was electrodeposited (6 µm thickness) on the copper plates using an APLAB regulated dc power supply (Model L-3230) unit. The bath composition and the operating parameters are given in Table 1. The nickel plated samples were activated in 5%v/v H₂SO₄ for 10 s and gold electrodeposited on them in an alkaline gold plating solution. The gold plating bath composition and the operating parameters are also given in Table 1. A number of preliminary experiments were carried out to arrive at a stripping solution containing oxidising agent, stabiliser, buffer and inhibitor to strip gold at a sufficiently fast rate. Different components and their effects are described in the 'Results' section below.

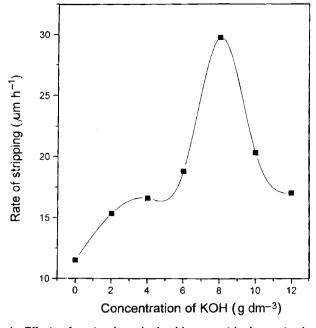
The stripping rate was measured with variation of concentration of the ingredients of the stripping solution and temperature. For study of stripping rate, the following procedure was adopted. Gold was deposited over Ni plated copper panels suitably masked to expose 10 cm^2 area. They were introduced in 100 cm^{-3} of stripping solution maintained at a specific temperature using a REMI: 1 MLH heater and the stripping carried out for a duration of 10 min The mass of gold dissolved was determined by weighing the panels before and after stripping and the stripping rate calculated using the formula

Thickness stripped ($\mu m h^{-1}$) = $\frac{w \times 10000 \times 60}{dAt}$

where w is the weight of the gold deposit, d is the density of the gold, A is the area of the gold plated specimen and t is the stripping time in minutes. The usability of the stripping solution for removal of gold plated on different substrates and undercoats was assessed by immersing different metals such as brass, copper, nickel,

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1 Effect of potassium hydroxide on stripping rate in solution containing potassium cyanide 25.0 g dm^{-3} , potassium citrate 15.0 g dm^{-3} and m-nitro benzoic 30.0 g dm^{-3} at 323 K

silver, platinum, palladium and stainless steel in the stripping solution for known duration and determining the weight loss.

The bath was stored for a period of 3 years and the stability assessed visually after that time.

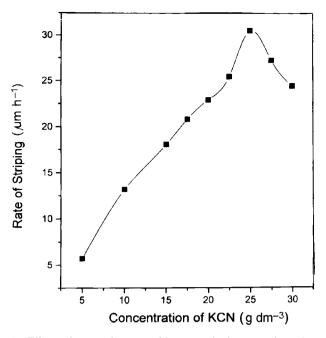
Results and discussion

Results of experiments carried out to optimise the concentration of different components in the stripping solution are discussed below.

Generally, potassium hydroxide was beneficial in maintaining the alkalinity of the electrolyte and preventing cyanide decomposition. Potassium hydroxide concentration was varied from 0 to 12 g dm⁻³ in order to fix the concentration in the stripping solution. From Fig. 1 it may be seen that with increase in potassium hydroxide concentration up to 8.0 g cm⁻³ stripping rate increases to a maximum of ~ 30 μ m h⁻¹, and thereafter decreases.

Potassium cyanide addition has been tested as cyanide is a very good complexant to keep the dissolved gold in complex form and facilitate further dissolution. By keeping the concentration of potassium hydroxide at 8.0 g dm^{-3} , the concentration of potassium cyanide was varied. From Fig. 2 it may be seen that the stripping rate increases with increasing concentration of potassium cyanide up to 25.0 g dm^{-3} .

The addition of potassium citrate as an additive complexant and buffering agent is shown in Fig. 3 which presents the variation of stripping rate with concentration of potassium



2 Effect of potassium cyanide on stripping rate in solution containing potassium hydroxide 8⋅0 g dm⁻³, potassium citrate 15⋅0 g dm⁻³, m-nitro benzoic acid 30⋅0 g dm⁻³ at 323 K

citrate. Keeping the concentration of potassium hydroxide at 8.0 g cm^{-3} and potassium cyanide 25.0 g dm^{-3} , the concentration of potassium citrate was varied and the stripping rate studied at 323 K. It may be seen that the stripping rate increases until the potassium citrate concentration reaches 15.0 g dm^{-3} and thereafter decreases showing that a concentration of 15.0 g dm^{-3} potassium citrate may be optimum to obtain high stripping rate.

Nitro aromatic compounds are good oxidising agents to facilitate chemical dissolution of metals and m-nitro benzoic acid is the oxidant assessed in the present stripping solution. Figure 4 shows the rate of stripping action for various concentrations of m-nitro benzoic acid along with potassium hydroxide at 8.0 g dm^{-3} , potassium cyanide at 25.0 g dm^{-3} and potassium citrate at 15.0 g dm^{-3} . It may be seen that at the m-nitro benzoic acid concentration of 30.0 g dm^{-3} the stripping rate is maximum. Figure 5 gives the effect of temperature on the stripping action; it may be noted that the stripping rate increases with temperature up to 323 K, but decreases with further rise in temperature.

Table 2 presents the chemical attack of the optimised stripping solution composition on different basis metals. It can be seen that metals such as nickel, stainless steel, palladium and platinum are not attacked even after 10 min immersion in the stripping composition at 323 K whereas soft metals such as brass, copper and silver are attacked initially but passivate after longer durations. These soft metals can be once again buffed and replated.

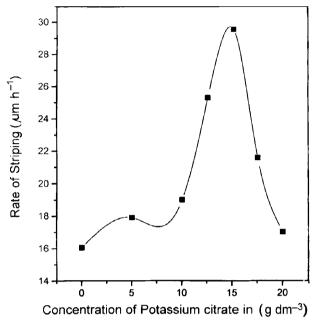
Table 1 Bath composition and operating parameters for nickel and gold plating

Bath composition, $g dm^{-3}$	Nickel	Gold
	Nickel sulphate: 240	Gold potassium cyanide: 5
	Nickel chloride: 40	Potassium cyanide: 45-75
	Boric acid: 30	Potassium hydroxide: 40-60
	Saccharin sodium salt: 1.0	
Temperature, K	323	333–343
Anode	Nickel	Platinum
c.d*, A dm ⁻²	2	0.4

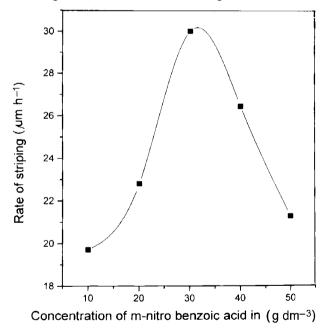
* Current density.

Table 2 Effect of stripping solution on different base metals

Series no.	Base metal	Etching loss in 5 min μ m	Base metal appearance after 10 min immersion
#1	Nickel	0.00	No change
#2	Stainless steel	0.00	No change
#3	Platinum	0.00	No change
#4	Palladium	0.00	No change
#5	Brass	0.53	Passivated
#6	Copper	0.33	Passivated
 #7	Silver	0.22	Passivated



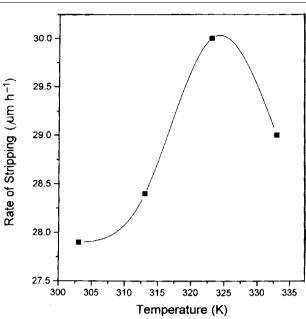
3 Effect of potassium citrate on striping rate in solution containing potassium hydroxide 8.0 g dm⁻³, potassium cyanide 25.0 g dm⁻³, m-nitro benzoic acid 30.0 g dm⁻³ at 323 K



4 Effect of m-nitro benzoic acid on stripping rate in solution containing potassium hydroxide 8⋅0 g dm⁻³, potassium cyanide 25⋅0 g dm⁻³ and potassium citrate 15⋅0 g dm⁻³ at 323 K

In each set of experiments carried out, the maximum stripping rate achieved was $\sim 30 \ \mu m \ h^{-1}$.

The stripping solution was a highly stable and clear solution with no visual degradation and precipitation



5 Effect of temperature on stripping rate in solution containing potassium hydroxide 8.0 g dm⁻³, potassium cyanide 25.0 g dm⁻³, potassium citrate 15.0 g dm⁻³, m-nitro benzoic acid 30.0 g dm⁻³

observed, even after storage for prolonged period of three years.

Conclusions

From the present studies carried out on the removal of gold deposits from defective substrates by a chemical immersion method, the following conclusions may be drawn.

1. Gold electrodeposits may be stripped at a fast rate of 28–30 μm $h^{-1}.$

2. Substrate metals such as nickel, palladium, stainless steel and platinum are not attacked. Slight attack is observed in the case of brass, copper and silver which may be replated after a mild buff.

3. The optimised stripping solution composition is: potassium hydroxide -8.0 g dm⁻³, potassium cyanide -25.0 g dm⁻³, potassium citrate -15.0 g dm⁻³ and m-nitro benzoic acid -30.0 g dm⁻³ at the temperature of 323 K.

4. The stripping solution appears to be very stable, even after storage for a period of up to three years.

References

- 1. F. H. Reid and W. Goldie: 'Gold plating technology', 1974, Ayr, Electrochemical Publications Ltd.
- 2. R. Ludwig: Oberflache (Berlin), 1972, 2, 84.
- 3. T. Jones: Met. Finish., 2004, 102, 15.
- 4. M. J. Nicol: Gold Bull., 1980, 13, 46.
- 5. M. J. Nicol: Gold Bull., 1980, 13, 105.
- 6. J. C. Lin and J. J. Huang: J. Appl. Electrochem., 1994, 24, 157.
- 7. D. R. Gabe: Trans. IMF, 2007, 85, (2), 72-74.