

Research Article

Effects of Added Chloride Ion on Electrodeposition of Copper from a Simulated Acidic Sulfate Bath Containing Cobalt Ions

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The effects of added chloride ion on copper electrodeposition was studied using Pb-Sb anode and a stainless steel cathode in an acidic sulfate bath containing added Co^{2+} ion. The presence of added chloride ion in the electrolyte solution containing 150 ppm of Co^{2+} ion was found to increase the anode and the cell potentials and decrease the cathode potential. Linear sweep voltammetry (LSV) was used to study the effects of added chloride ion on the anodic process during the electrodeposition of copper in the presence of added $\text{Co}^{2+} \approx 150$ ppm; the oxygen evolution potential is polarised by adding 10 ppm chloride ion at current densities ($\geq 150 \text{ A/m}^2$), and further increase in chloride ion concentration increases the polarisation of oxygen evolution reaction more at higher current densities. X-ray diffraction (XRD) showed that added chloride ion and added Co^{2+} ion changed the preferred crystal orientations of the copper deposits differently. Scanning electron microscopy (SEM) indicated that the surface morphology of the copper deposited in the presence of added chloride ion and added Co^{2+} ion has well-defined grains.

1. Introduction

Copper is generally extracted through pyrometallurgical processes [1]. However several important factors such as non-availability/depletion of high-grade ores, increasing world demand, increasing process cost like labour cost, energy cost, and so forth, and emission of highly toxic and strongly acidic sulfur-oxide gases from smelter plants creating severe environmental pollution demanded an alternative technology to overcome these problems towards the end of nineteenth century. Thus in mid of 1980, hydrometallurgical processes involving leaching, solvent extraction, and electrowinning (L/SX/EW) were widely adopted for extraction of copper from secondary sources such as oxide ores, mixed sulfide and oxide ores, low grade sulfide ores, industrial wastes from metal plating, metal finishing, wastes from metallurgical industries, scrap copper, and alloys [1]. Although copper leaching and solvent extraction have achieved a state of advanced development, the commercial success of the process is dependent upon the ability to produce high-quality final product through the electrowinning process. The high power consumption associated with this process has been the

subject of many investigations in the last few years [2–6]. The attempts made so far are [7]

- (a) improvement in mass transport for cell operation at higher-current density without significant increase in energy requirement,
- (b) selection of different routes for production of the copper metal,
- (c) adoption of an alternative anode reaction,
- (d) use of inorganic and organic depolarisers for decreasing the overpotential of oxygen evolution reaction at the anode and copper deposition reaction at the cathode,
- (e) replacement of the Pb-Sb anode by a catalytic anode for decreasing the oxygen overpotential and many similar aspects.

In recent years, a more challenging problem is the efficient recovery of copper through electrodeposition process from the direct acidic leach solution and dilute industrial effluents with low power consumption [8, 9] eliminating SX process

that involves a lot of chemicals. The attempts described in (c)–(e) above are of particular interest as these that will be amenable to industrial implementation without any significant change in the standard plant practice. These attempts could bring significant decrease in cell voltage and power consumption through lessening of the anode potential or anode overpotential. Bivalent cobalt ion is a very useful addendum in copper electrodeposition [10–12] due to the following reasons.

- (a) It considerably decreases the overpotential of oxygen evolution reaction.
- (b) It significantly controls the lead corrosion of the lead-antimony anode and subsequently improves the cathode quality by reducing lead contamination.
- (c) It can be used in conventional copper electrodeposition process without any modification to the existing plant cell.

Our earlier investigations [5, 12] reported the effects of cobalt ion and/or H_2SO_3 on copper electrodeposition from simulated acidic sulfate bath using Pb-Sb and/or graphite anode with significant decrease in power consumption.

In the present investigation, an attempt is made to see the effects of added Cl^- ion on the electrodeposition of copper from a simulated acidic copper bath containing added Co^{2+} ion. Small amounts of chloride ion alone are known to have an accelerating effect on the deposition of copper and reduces anode polarization [13]. Besides, Cl^- ion arises in the copper electrolyte from the makeup water. Several studies were undertaken to observe the effect of Cl^- ion on electrodeposition of copper [13–19] including the interaction of Cl^- ion with some entrained extractant residuals [20]. However, no literature appears to be available so far to our knowledge on the effect of Cl^- ion on electrodeposition of copper containing Co^{2+} ion. Pd/Sb is used as an anode material. A comparison of cell potential, anode potential, cathode potential, anode polarization characteristics, current efficiency, power consumption, deposit quality, deposit morphology, and the crystal orientation is reported in the absence and the presence of Cl^- during electrodeposition of copper in the presence of Co^{2+} ion.

2. Experimental Methods

2.1. Materials. Stock solutions of 40 g/L Cu^{2+} , 10 g/L Co^{2+} and 60 g/L sulfuric acid, and 10 g/L HCl were prepared separately using AnalaR grade reagents in doubly distilled water. The concentration of Cu^{2+} was measured by iodometric method, while H_2SO_4 was determined by acid-base titration, Co^{2+} by atomic absorption spectroscopy (Model 3100, PerkinElmer) and Cl^- by EDT (England ion meter).

2.2. Electrodeposition Experiments. The electrolysis cell consisted of a lidded 200 cm^3 double wall beaker. A stainless steel cathode ($8.0 \times 5.0 \times 0.2$ cm) and a lead-antimony (Sb = 6%) anode of the same dimensions were used. The interelectrode space was maintained at 3.0 cm for all the experiments.

All electrodeposition experiments were carried out for 2 hours at room temperature ($30 \pm 1^\circ\text{C}$) using an electrolyte solution containing 20 g/L Cu^{2+} and 30 g/L H_2SO_4 , and 150 ppm Co^{2+} ion. After electrolysis, the cathode was washed thoroughly with water followed by acetone and dried. The current efficiency ($\pm 0.3\%$) was calculated from the weight of copper gained by the cathode. The electrodeposited copper was analysed as $>99.9\%$ pure.

2.3. Polarisation Measurements. Linear sweep voltammetry (LSV) was used to examine the anodic polarisation behaviour, during copper electrodeposition containing added Cl^- and/or added Co^{2+} . A Pb-Sb (0.70 cm^2) electrode was used as the working anodic electrode. A platinum wire and a saturated calomel electrode (SCE) were used as the counter electrode and the reference electrode, respectively. The surface of the working electrode was freshly prepared before each experiment, initially rinsed with 1M HCL followed by doubly distilled water. A scanning potentiostat (Model 362, EG&G Princeton Applied Research) was used for carrying out polarisation experiments between +1.2 V and +2.0 V. The linear voltammograms ($V \sim I$) were recorded by using an X-Y recorder (PAR Model RE0091, EG&G Princeton Applied Research) at a scan rate of 20 mV/sec during polarisation experiments.

2.4. Deposit Examination. An X-ray diffractometer (PW 1050, Philips) was used to determine the crystallographic orientations of the cathode copper deposits. Reproducible results were obtained using cathode-deposited sections and powders scraped from the cathode surface. The data matched those for copper powders reported in the literature (JCPDS, 1984). The deposit morphology of the electrodeposited copper samples was examined by SEM (SE 101B model, Philips).

3. Results and Discussion

3.1. Anode Potential. Effect of Added Chloride Ion Variation in the Presence of Added Cobalt Ion. The influence of $[\text{Cl}^-]_0$ on the anode voltage in the presence of $[\text{Co}^{2+}]_0 \cong 150$ ppm in the electrolyte solution is shown in Figure 1. The anode voltage in the absence of Cl^- ion in the electrolyte containing only Co^{2+} (aq) ($\cong 150$ ppm) is found to be 1.58 V. Addition of 5 ppm of Cl^- ion to the same electrolyte increases the anode voltage to 1.63 V. No significant increase in the anode potentials is observed with further increase in $[\text{Cl}^-]_0$ up to 100 ppm.

3.2. Cathode Potential. Effect of Added Chloride Ion Variation in the Presence of Cobalt Ion. The cathode potential at zero concentration of Cl^- in the electrolyte containing Co^{2+} (aq) $\cong 150$ ppm was found to be 0.25 V. The addition of $[\text{Cl}^-]_0 \cong 5$ ppm decreases the cathode potential from 0.25 to 0.19 V. Further increase in $[\text{Cl}^-]_0 \cong 100$ ppm brings about no significant change in the cathode voltage in the presence of $[\text{Co}^{2+}]_0 \cong 150$ ppm as observed in Figure 2.

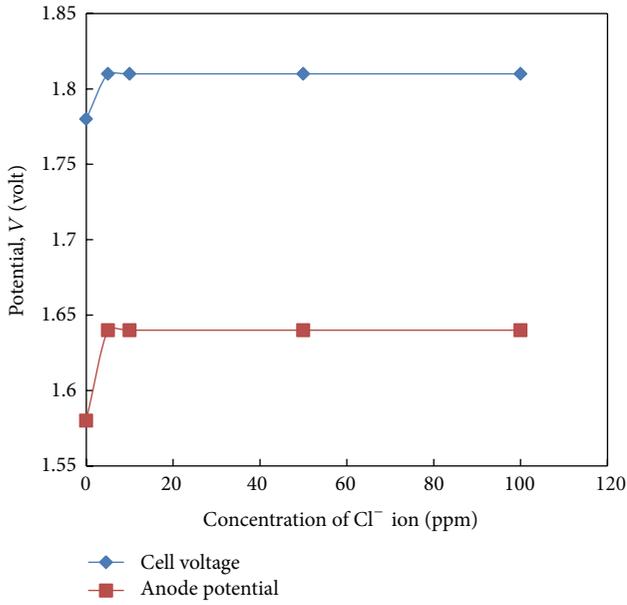


FIGURE 1: Effect of added $[\text{Cl}^-]_0$ on cell voltage and anode potential in the presence of added cobalt ion 150 ppm ($\text{Cu}^{2+} = 20 \text{ g/L}$, $\text{H}_2\text{SO}_4 = 30 \text{ g/L}$, $T = 30 \pm 1^\circ\text{C}$, $\text{CD} = 150 \text{ A/m}^2$, $t_d = 2 \text{ hr}$).

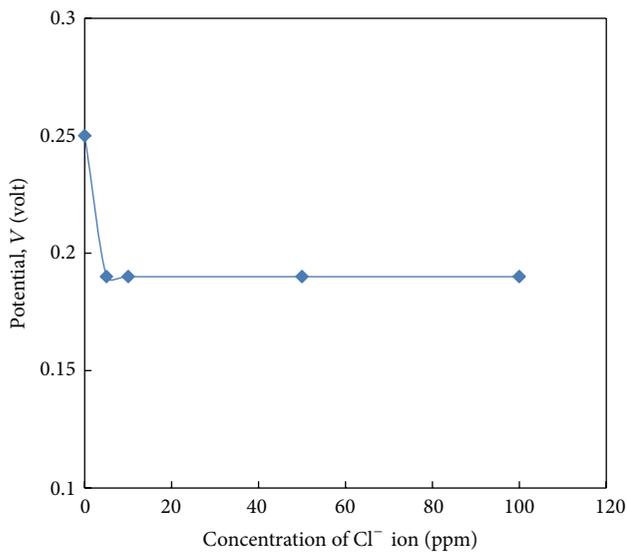


FIGURE 2: Effect of $[\text{Cl}^-]_0$ on cathode potential in the presence of added cobalt ion 150 ppm ($\text{Cu}^{2+} = 20 \text{ g/L}$, $\text{H}_2\text{SO}_4 = 30 \text{ g/L}$, $T = 30 \pm 1^\circ\text{C}$, $\text{CD} = 150 \text{ A/m}^2$, $t_d = 2 \text{ hr}$).

3.3. Cell Voltage. Effect of Added Chloride Ion Variation in the Presence of Cobalt Ion. Figure 1 shows the influence of $[\text{Cl}^-]_0$ on cell voltage in the presence of added Co^{2+} (aq) during electrodeposition of copper. The nature of the curve appears to be similar to that observed in the case of the anode potential. Addition of 5 ppm of Cl^- to the same electrolyte increases the cell voltage to 1.81 V. Further increase in $[\text{Cl}^-]_0$ in the range of 5–100 ppm brings about no significant increase in the cell voltage.

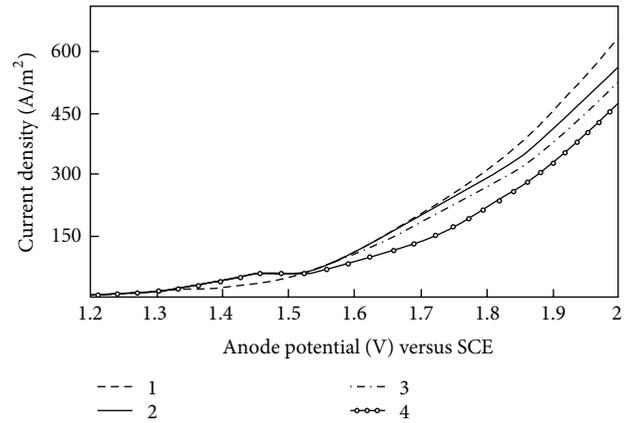


FIGURE 3: Anodic potentiodynamic curves in the presence of added chloride ion. Key: added $[\text{Cl}^-]_0$ (1) nil; (2) 5 ppm; (3) 50 ppm; (4) 100 ppm ($\text{Cu}^{2+} = 20 \text{ g/L}$, $\text{H}_2\text{SO}_4 = 30 \text{ g/L}$, $[\text{Co}^{2+}]_0 = 150 \text{ ppm}$, $T = 30 \pm 1^\circ\text{C}$, scan rate 20 mV/Sec).

3.4. Anode Polarisation. The anodic potentiodynamic experiments conducted by varying the chloride ion concentration in the presence of $[\text{Co}^{2+}]_0 \cong 150 \text{ ppm}$ in the copper electrolyte solution were studied by LSV method, and the curves are shown in Figure 3. Figure 3 curve 1 shows current density versus anode potential curve in the presence of $[\text{Co}^{2+}]_0 \cong 150 \text{ ppm}$. It was observed that the addition of $[\text{Cl}^-]_0 \cong 5 \text{ ppm}$ polarises significantly the anode potential of oxygen evolution reaction at higher current densities (Figure 3 curve 2); the region between 1.3 V and 1.5 V shows the probable occurrence of chlorine evolution that is not observed in curve 1. Further increase in $[\text{Cl}^-]_0$ to 50 ppm and 100 ppm (Figure 3 curves 3 and 4) increases the polarisation of oxygen evolution reaction.

3.5. Current Efficiency and Power Consumption. The effect of added chloride ion in the absence and the presence of $[\text{Co}^{2+}]_0$ on current efficiency and the corresponding power consumption are examined. It was found that the current efficiency remained $\sim 98\%$ for all additions of chloride ion and/or cobalt ion throughout the investigation, and the cathode remains smooth, bright, and compact. It is seen from Table 1 that the addition of $[\text{Cl}^-]_0$ in the range of 5–100 ppm in the presence of Co^{2+} (aq) shows increases in the power consumption by $\leq 35 \text{ kWh/ton}$ of Cu than that observed in the presence of only $[\text{Co}^{2+}]_0 \cong 150 \text{ ppm}$ in the electrolyte solution.

3.6. Crystallographic Orientations. The electrodeposited copper samples produced from acidic copper bath electrolyte solution on stainless steel cathode in the presence of added Co^{2+} (aq) and/or Cl^- (aq) are examined by XRD to determine the preferred crystal orientations and the relative growth of copper on the preferred planes. Representative XRD traces are redrawn and shown in Figure 4. The XRD of all the copper deposits showed an fcc structure, with the 2θ positions of the peaks remaining constant. The XRD trace for the original

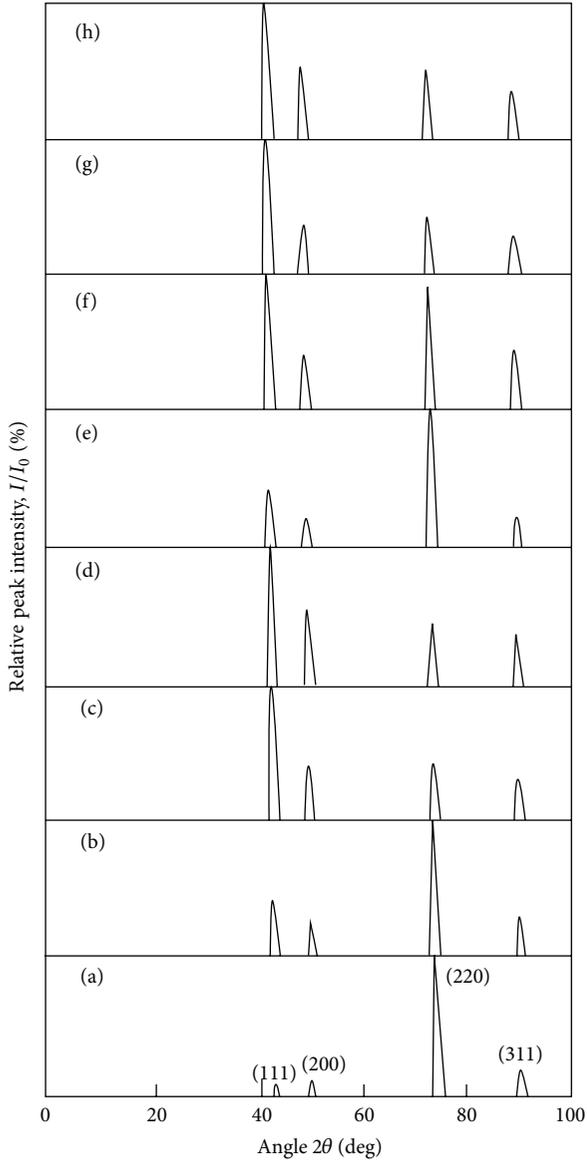


FIGURE 4: XRD patterns of electrodeposited copper samples. Key: added $[\text{Co}^{2+}]_o$ X ppm + $[\text{Cl}^-]_o$ Y ppm (a) Bl; (b) 0 + 5 ppm; (c) 0 + 100 ppm; (d) 150 ppm + 0; (e) 150 ppm + 5 ppm; (f) 150 ppm + 10 ppm; (g) 150 ppm + 50 ppm; (h) 150 ppm + 100 ppm. ($\text{Cu}^{2+} = 20$ g/L, $\text{H}_2\text{SO}_4 = 30$ g/L, $T = 30 \pm 1^\circ\text{C}$, $\text{CD} = 150$ A/m², $t_d = 2$ hr).

electrolyte (Figure 4(a)) in the absence of added Cl^- and Co^{2+} (aq) and in the presence of $[\text{Co}^{2+}]_o \cong 150$ ppm (Figure 4(d)) has been given here for comparison. The addition of only $[\text{Cl}^-]_o \cong 5$ ppm or 100 ppm changes the XRD pattern of copper deposit differently; while the presence of the former did not alter the most preferred (220) plane (in comparison to Figure 4(a)), the presence of the latter changes the most preferred (220) plane to (111) plane, and the growth along (200), (220), and (311) planes is more or less equally maintained (Figure 4(c)) and is similar in nature with that observed in the presence of only $[\text{Co}^{2+}]_o \cong 150$ ppm (Figure 4(d)). The

TABLE 1: Effect of chloride ion on power consumption in the presence of cobalt ion.

$[\text{Co}^{2+}]_o$, ppm	$[\text{Cl}^-]_o$, ppm	E , kWh/ton of Cu
150	—	1564
150	5	1599
150	10	1599
150	50	1599
150	100	1599

(—) refers to nil.

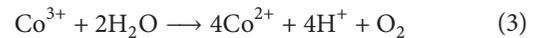
addition of small $[\text{Cl}^-]_o \cong 5$ ppm to the electrolyte containing $[\text{Co}^{2+}]_o \cong 150$ ppm again retains the most preferred (220) plane (Figure 4(e)); the increase in $[\text{Cl}^-]_o$ to 10 ppm in the same electrolyte increases equal growth along (111) and (220) planes (Figure 4(f), further increase in the $[\text{Cl}^-]_o \cong 50$ ppm and 100 ppm in the presence of $[\text{Co}^{2+}]_o \cong 150$ ppm retained the (111) plane as the most preferred plane, and the growth along (200), (220), and (311) planes is more or less equally maintained (Figures 4(g) and 4(h)).

3.7. Surface Morphology. The effect of chloride ion on the surface morphology of the deposited copper samples in the absence and the presence of Co^{2+} (aq) is shown in Figures 5(a)–5(f). It is observed that polycrystalline pyramidal deposits are obtained in the presence of only $[\text{Cl}^-]_o \cong 5$ ppm and $\cong 100$ ppm in the copper electrolyte solution (Figures 5(a) and 5(b)). The addition of $[\text{Cl}^-]_o$, 5 ppm to the electrolyte solution containing $[\text{Co}^{2+}]_o$, 150 ppm results in very small grains of copper deposits (Figure 5(c)). Increasing the $[\text{Cl}^-]_o$ to 100 ppm in the same electrolyte solution forms small size nodules and mosaic copper deposits (Figures 5(d)–5(f)).

4. Interpretation

It would be interesting to interpret the effects of Cl^- on electrodeposition of copper in the presence of Co^{2+} ion. The addition of only Co^{2+} ion to the copper electrolyte decreases the anode potential significantly but has no effect on the cathode potential as was found in our earlier investigation [12].

The decrease in anode potential in the presence of only Co^{2+} ion can be best explained by the following reactions. In the absence of Co^{2+} ion, oxygen is evolved from the electrolysis of water (1) taking place on PbO_2 surface on Pb-Sb anode which exhibits a high overpotential of ≥ 600 mV [1]:



While in the presence of only Co^{2+} ion, oxidation of Co^{2+} ion to Co^{3+} ion (2) allows the facile oxidation of H_2O in accordance with (3) and leads to lower oxygen overpotential [10, 21].

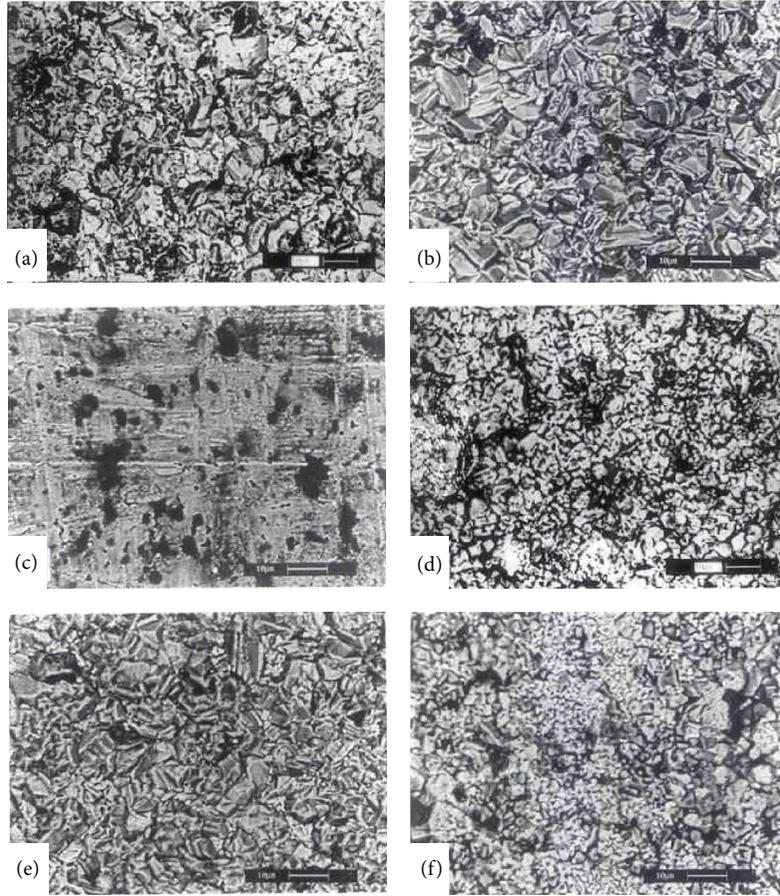


FIGURE 5: SEM photomicrographs of electrodeposited copper samples. Key: added $[\text{Co}^{2+}]_0$, X ppm + $[\text{Cl}^-]_0$, Y ppm (a) 0 + 5 ppm; (b) 0 + 100 ppm; (c) 150 ppm + 5 ppm; (d) 150 ppm + 10 ppm; (e) 150 ppm + 50 ppm; (f) 150 ppm + 100 ppm ($\text{Cu}^{2+} = 20 \text{ g/L}$, $\text{H}_2\text{SO}_4 = 30 \text{ g/L}$, $T = 30 \pm 1^\circ\text{C}$, $\text{CD} = 150 \text{ A/m}^2$, $t_d = 2 \text{ hr}$).

An important result found in the present investigation is the increase in the anode potential when a small enough $[\text{Cl}^-]_0 \cong 5\text{--}100 \text{ ppm}$ is added to the electrolyte containing 150 ppm Co^{2+} (aq); nearly 50 mV increase in the anode potential is observed. The same is also indicated in the anodic potentiodynamic study; the addition of Cl^- ion to the copper electrolyte is found to polarise the facile oxidation of H_2O molecules in the presence of Co^{2+} ion. Thus, the increase in anode voltage due to the presence of Cl^- in the copper electrolyte containing Co^{2+} ion is most probably due to the partial discharge of Cl^- ion at the anode as given in (4) [21, 22] as well as the polarisation of the facile oxidation of H_2O molecules as described in (2) and (3) due to the Cl^- ions. Indication for this interpretation is the observed evolution of Cl_2 (g) at the anode during the experimental work:



The decrease in the cathode potential in the presence of Cl^- ion may be due to the ready discharge of the $\text{Cu}^{2+}\text{-Cl}^-$ complex at the cathode [23].

5. Conclusions

The results observed in the present investigation can be summarised as follows.

- (1) The addition of 5 ppm of chloride ion to the copper electrolyte solution in the presence of 150 ppm Co^{2+} ion increased the anode potential by $\cong 50 \text{ mV}$ which is more or less maintained with further increase in Cl^- ion concentration up to 100 ppm. The increase in the chloride ion concentration (in the range of 0–100 ppm) in the electrolyte solution decreases the cathode potential in the presence of $\text{Co}^{2+} \cong 150 \text{ ppm}$ from 0.25 V to 0.19 V.
- (2) The addition of $\cong 0\text{--}100 \text{ ppm}$ of Cl^- to the electrolyte solution containing 150 ppm Co^{2+} increases the power consumption by 35 kWh/ton Cu.
- (3) The increase in the chloride ion concentration in the absence and the presence of cobaltous ion changes the most preferred (220) hkl plane to (111) hkl plane.

- (4) No significant change in the surface morphology of the copper deposits is observed. Smooth, bright deposits are obtained throughout the investigation.

Abbreviations

E_a :	Anode potential
E_{cell} :	Cell potential
E (kWh/ton Cu):	Energy consumption
$[Cl]_o$:	Initial concentration of Cl^- ion
$[Co^{2+}]_o$:	Initial concentration of Co^{2+} ion
CD:	Current density
T_d :	Electrolysis duration
T :	Temperature.

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