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Electrodeposition of cobalt from aqueous sulphate solutions in the presence of tetra ethyl ammonium bromide

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Abstract: The effect of tetra ethyl ammonium bromide (TEAB) as an additive on the structural, morphological characteristics of the cobalt metal produced from aqueous sulphate solutions was investigated. The concentration of TEAB was varied in a range of 1−50 mg/L to evaluate its effect on current efficiency, energy consumption and quality of electrodeposited cobalt metal. Smooth and bright deposits of cobalt were obtained at low concentration of TEAB (10 mg/L) maintaining a current efficiency of 99.6%, with a low energy consumption of 2.38 kW·h/kg. X-ray diffraction studies reveal that (100) plane is the most preferred plane of crystal growth during cobalt electrodeposition. Scanning electron micrographs indicate that smooth and uniform deposit of cobalt is obtained at 10 mg/L beyond which the deposit quality deteriorates. Cyclic voltammetric studies indicate that the presence of TEAB in the electrolytic bath polarizes the cathode and decreases the cathodic current considerably. XPS results confirm the electrodeposition of high pure cobalt with no sign of chemical bonding with TEAB as evident from the FTIR spectra.

Key words: cobalt; tetra ethyl ammonium bromide (TEAB); electrodeposition; polarisation study; current efficiency; deposit morphology

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1 Introduction

Cobalt is a strategic and critical metal considering its numerous industrial applications. Cobalt finds use in rechargeable batteries and in the manufacture of corrosion and wear resistant alloys. Other major uses of the metal include the manufacture of catalysts for the petroleum and chemical industries, as a drying agent for paint varnishes, ground coats for porcelain enamels, magnetic recording media and steel belted radial tires [1]. Cobalt comprises about 0.02% in the earth crust hence considerable research is being carried out for the commercial production of the metal [2]. Over the last three decades, 70% of the world's production of cobalt is carried out using hydrometallurgical techniques in which the final step is electrodeposition [3]. The most popular electrodeposition recovery process for cobalt metal involves a sulphate medium [4−6].

The crucial aspect in such processes is the production of superior quality cobalt metal having bright texture with uniform and smooth deposits. The effect of additives during the electrodeposition is usually important owing to their effects on the growth and structure of the deposits. The potential benefits of additives include brightening of the deposit, grain size reduction, reducing the tendency of tree formation, increasing the current density, promoting leveling and reducing stress and pitting [7,8].

The presence of impurities or additives in the electrolytic bath and their effects on cobalt electrodeposition have been reported by many researchers [9−13]. DAS and SUBBAIAH [10] have examined the role of boric acid and sodium fluoride as additives in the electrodeposition of cobalt from cobalt sulphate solutions. It was observed that additives,

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individually or in combination, yielded metal deposits with better surface morphology and higher current efficiencies. TRIPATHY et al [11] have examined the role of boric acid and manganese (Mn) in cobalt electrowinning. They observed that the presence of Mn(II) or boric acid alone increased the current efficiency by 2% to 3% and also brought a decrease in energy consumption. The effect of the organic additives on surface roughness was studied for the Co−thiourea and Co−saccharin systems [12]. TRIPATHY and KOTZE [13] have investigated the effect of thiourea and saccharin on the electrodeposition of cobalt. The saccharin and thiourea act as smoothing agents and produce a compact, smooth and high quality deposit. However, systematic studies based on the influence of organic additives on deposit morphology are few in the case of cobalt electrodeposition. FRANKLIN et al [14] have demonstrated the effectiveness of quaternary ammonium salts as additives in the electrodeposition of metals like copper and tin. The use of quaternary ammonium bromide as an additive in zinc electrodeposition has been reported [15]. CISZEWSKI et al [16] have investigated the effect of quaternary ammonium chlorides on the electrodeposition of nickel. Tetra alkyl ammonium salts have also been used in the electrodeposition of MnO_2 [17]. In the present study, the role of tetra ethyl ammonium bromide (TEAB) as an additive on the current efficiency, energy consumption and deposit morphology of cobalt during electrodeposition was investigated. FTIR and XPS studies were carried out to evaluate the interaction of the additive with cobalt and to know its purity.

2 Experimental

2.1 Experimental setup

Cobalt electrodeposition was carried out in a rectangular cell made from Perspex, consisting of independent cathodic and anodic chambers separated by a polypropylene diaphragm. Stainless steel (316 grades) and Pb−Sb (1%) sheet were used as cathode and anode, respectively. Equal volumes of electrolyte (200 mL) were used in both the cathodic and anodic compartments. The cell was placed in a thermostatic water bath (Julabo, Germany) to maintain the temperature of the electrolytic cell. All the electrodeposition experiments were carried out under galvanostatic conditions. A regulated power supply system (0−32 V, 10 A, DC, Aplab Ltd., India) was used for providing constant current. The schematic diagram of the experimental setup is shown in Fig. 1.

2.2 Reagents

The electrolytic solutions were prepared using doubly distilled water. The cobalt electrolyte was prepared from analytical grade cobalt sulphate ($CoSO_4·7H_2O$), sodium sulphate (Na_2SO_4) and boric acid (H_3BO_3). The pH of the electrolyte was adjusted to 4 by using dilute sodium hydroxide and sulphuric acid. Solutions referred to as “blank” contained 60 g/L cobalt, 15 g/L Na_2SO_4 and 9 g/L H_3BO_3. Calculated amount of TEAB was added to the electrolytic bath from the freshly prepared stock solution of 10 g/L. All chemicals were from Merck Chem. Ltd., India.

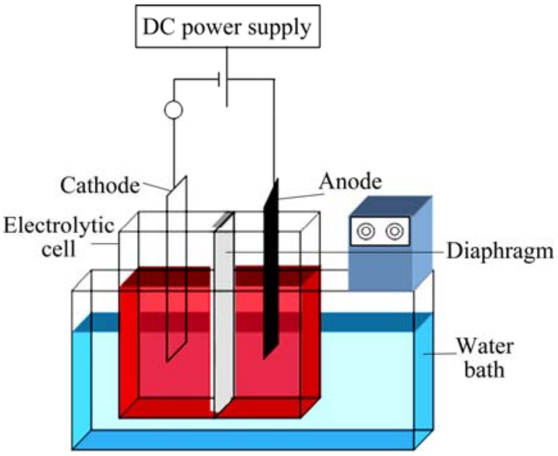

Fig. 1 Schematic diagram of arrangement of electrolytic cell with diaphragm for electrodeposition of cobalt

2.3 Electrode preparation

The cathode was first polished by 320, then by 600 grade silicon carbide papers to have a mirror-like finished surface. The cathode was then rinsed with 1 mol/L HCl followed by its washing with distilled water. It was then allowed to air dry. The dried cathodes were then weighed prior to electrodeposition.

2.4 Electrolysis

The entire sets of electrodeposition experiments were conducted at a temperature of 333 K (60 °C) and at a current density of 200 A/m^2 for 3 h. The distance between the electrodes was kept at about 2.5 cm. Cell voltage was recorded using a multimeter connected across the anode and cathode at various time intervals and finally the average cell voltage was reported. After electrolysis, the cathode was removed from the cell and washed thoroughly with water and acetone and finally dried in an oven at 383 K (110 °C). Current efficiencies were calculated from the mass gained by the cathode during electrolysis.

2.5 Polarization studies

The polarization behavior of the cathode in the presence and absence of any additive during the electro reduction of cobalt was carried out by cyclic voltammetric technique using a three electrode glass cell. A stainless steel electrode and platinum wire were used as the working and counter electrode, respectively. Standard silver−silver chloride (Ag/AgCl) was used as the reference electrode. The polarization measurements were carried out using Metrohm potentiostat/galvanostat model 128A and the potentials were reported as such. Voltammetric scans were performed in the potential range from −0.6 to −1.0 V (vs Ag/AgCl) at a scan rate of 10 mV/s. High purity nitrogen was used to sparge out the dissolved oxygen so as to make the system free from aerial or any dissolved oxygen which may affect the redox reactions.

2.6 Structural characterization

Sections of cobalt electrodeposits were analyzed by a X-ray diffractometer (PAN ANALYTICAL PW 1830; Philips, Japan) with Cu K_α radiation, λ=1.5404 Å to determine the preferred crystal orientations. The surface morphologies of the deposits were examined by scanning electron microscope (SEM, JEOL JSM 6510, Japan) to investigate the structural and morphological changes in the electrodeposited metal under various deposition conditions. Fourier transform infrared (FT-IR) spectrographs were recorded on a Perkin Elmer spectrophotometer in the mid infrared region of frequency range 400−4500 cm^{-1}. X-ray photoelectron spectrometer (S/N: 10001, Prevac, Poland) was employed to carry out XPS studies. The spectra were recorded using Al K_α radiation ($h\nu$=1486.6 eV) and a hemispherical energy analyzer. An instrument base pressure of 5×10^{-10} mbar was maintained during data acquisition.

3 Results and discussion

3.1 Cathodic current efficiency

The consequential effects of the presence of the additive tetra ethyl ammonium bromide in the electrolytic bath on current efficiencies and energy consumption during cobalt electrodeposition are shown in Fig. 2. The absence of TEAB electrodeposition of the cobalt from the blank solution resulted in current efficiency (CE) of 90.29 % and energy consumption (EC) of 3.03 kW·h/kg. Introduction of 1 mg/L of TEAB in the electrolytic bath increased the CE by 2%−3%. Further increase in the concentration of TEAB in the electrolytic bath increased the CE, and at 10 mg/L the highest CE of 99.6 % and the lowest EC of 2.38 kW·h/kg were observed. Increase in current efficiency during electrolysis is always associated with a decrease in energy consumption which is also evident here (Table 1). It should also be noted here that at this condition, very smooth and bright cobalt deposit was obtained, indicating it as the optimum condition for electrodeposition of cobalt pertaining to this system. The increase in CE can be attributed to inhibition of H_2 evolution by the additive. Such instance regarding the suppression of hydrogen evolution has already been reported in the case of zinc electrodeposition where glue was used as an additive [18]. The usual decrease in CE was observed with increase in the concentration of TEAB in the electrolytic bath. Thus, increasing the concentration of TEAB beyond 10 mg/L resulted in a decrease in CE and hence increases the EC during electrodeposition (Table 1). This decrease in the CE at high additive concentrations can be ascribed to the blocking of the active nucleation sites on the cathode surface by adsorption of the additive, inhibiting electro reduction of cobalt.

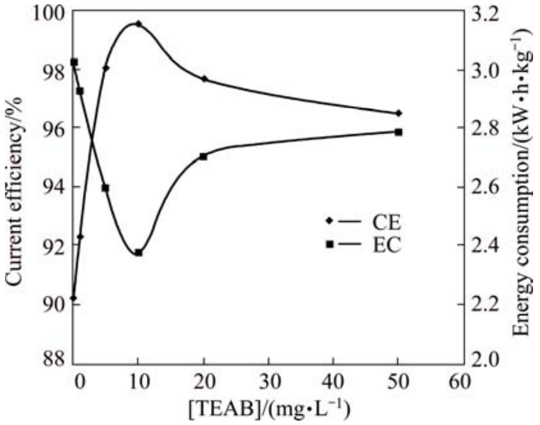

Fig. 2 Effect of additive concentration on current efficiency and energy consumption during cobalt electrodeposition ([Co] in feed solution=60 g/L, pH of solution = 4, electrolysis time=3 h, current density=200 A/m^2, temperature=60 °C)

Table 1 Effect of TEAB concentration on CE, EC, E_n, NOP, ΔNOP and crystallographic orientations during electrodeposition of cobalt from acidic sulphate bath

[TEAB]/ (mg·L^{-1})	CE/%	EC/ (kW·h·kg^{-1})	E_n/ mV	NOP/ mV	ΔNOP/ mV	Relative peak intensity (I/I_0)/%			
						(100)	(101)	(110)	(111)
0	90.29	3.029	−858	−157	0	100	43.17	23.39	−
1	92.34	2.929	−862	−161	−4	100	40.25	21.47	−
5	98.06	2.597	−869	−168	−11	100	53.39	27.22	−
10	99.56	2.377	−875	−174	−17	100	77.22	4.99	−
20	97.69	2.701	−884	−183	−26	100	58.70	45.27	52.81
50	96.51	2.785	−906	−205	−48	100	6.47	5.90	61.64

3.2 Polarisation studies

Cyclic voltammetric studies are essential to investigate the behavior of the cathode towards cobalt electrodeposition from cobalt sulphate solutions in the presence and absence of TEAB. The polarization behaviors of the cathode during electro reduction of cobalt from sulphate solutions in the absence and presence of TEAB are shown in Fig. 3. The cycle started at −0.6 V (vs AgCl) and the scan swept in the cathodic direction. The nucleation potential (E_n) and the crossover potentials (E_{co}) were then determined using the procedures reported earlier [19]. The nucleation overpotential (NOP) which is equal to the difference between E_n and E_{co} was calculated and recorded in Table 1. The NOP value in the absence of TEAB was −157 mV. The effect of TEAB with varying concentrations can be seen by comparing the shift of NOP (ΔNOP) from the baseline value of −157 mV (Table 1). Increase in the additive concentration resulted in shift of NOP to more negative values indicating polarisation of cathode with simultaneous decrease in cathodic current density. It was found that ΔNOP was higher with the increase of concentration of TEAB. With TEAB concentration of 10 mg/L, the ΔNOP was −17 mV, indicating polarisation of the cathode. In the present study, the highest polarisation was observed with 50 mg/L TEAB, where ΔNOP was the highest (−48 mV). The higher the ΔNOP is, the lower the cathodic current is. This decrease in current with increase in additive concentrations was due to the increased additive adsorption on the cathode. Similar observations have been reported by TRIPATHY et al [15]. This behavior was also seen and was also reflected with the decrease in current efficiency with increasing additive concentration in the solution (Table 1).

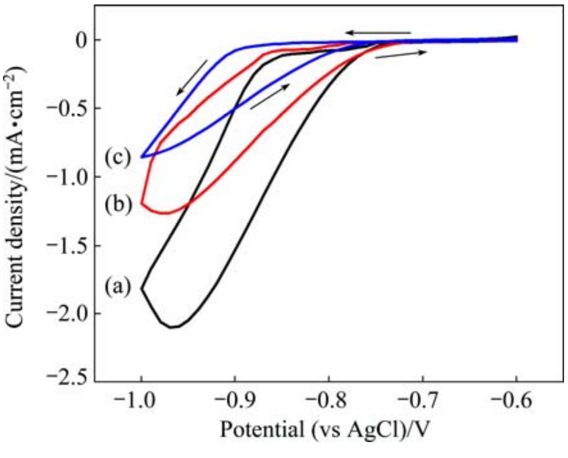

Fig. 3 Effect of TEAB concentration on polarization behavior of cathode during cobalt electrodeposition: (a) Blank; (b) 10 mg /L TEAB; (c) 50 mg/L TEAB

3.3 Crystal orientations

Figure 4 shows the X-ray diffraction patterns of the cathodic cobalt deposits obtained at various concentrations of TEAB. The crystal growth in the absence of the additive follows the order of preferred orientations (100), (101) and (110). There is practically no change on the preferred orientations with addition of TEAB. It was also observed that at higher concentration of the additive (≥10 mg/L), growth of an extra plane (111) appeared leading the preferred order of orientation to (100), (101), (110) and (111). It is also important to note that the presence of TEAB in the electrolytic bath affects the order of preferred orientation as well as the degree of crystallinity. It was observed that at higher concentrations of the additive (≥10 mg/L), the intensity of the XRD peaks decreased and broadened, resulting in non uniform, poorly crystalline growth (Figs. 4(d) and (e)).

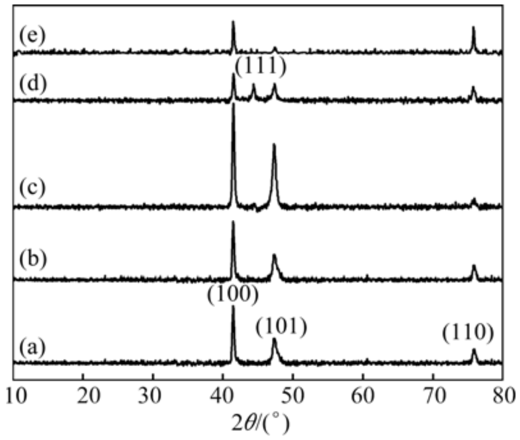

Fig. 4 X-ray diffraction patterns of cobalt metal deposits at various concentrations of TEAB: (a) Blank; (b) 1 mg/L; (c) 10 mg/L; (d) 20 mg/L; (e) 50 mg/L ([Co] in feed solution= 60 g/L, pH of solution=4, electrolysis time=3 h, current density=200 A/m^2, temperature=60 °C)

3.4 Surface morphology

Effects of TEAB on the current efficiencies and energy consumption during cobalt electrodeposition were also reflected on its surface morphologies. Figure 5 shows the SEM images of the cobalt metal deposited from acidic sulphate solutions. Figure 5(a) shows that in the absence of any additive bright and smooth deposit with randomly oriented crystallites of cobalt was obtained. Introduction of 10 mg/L TEAB in the electrolytic bath produced uniform bright and smooth deposits with increasing grain size (Fig. 5(b)). It was observed that at higher concentrations of the additive (20 and 50 mg/L), the quality of the deposits deteriorated, resulting in poorly crystalline and non uniform deposits with fibrous growth on the cobalt deposits (Figs. 5(c) and (d)). This deterioration in the deposit quality has been reflected in the current efficiency giving lower values. It could also be seen that there are some holes on the deposit indicating centers of hydrogen evolution, a

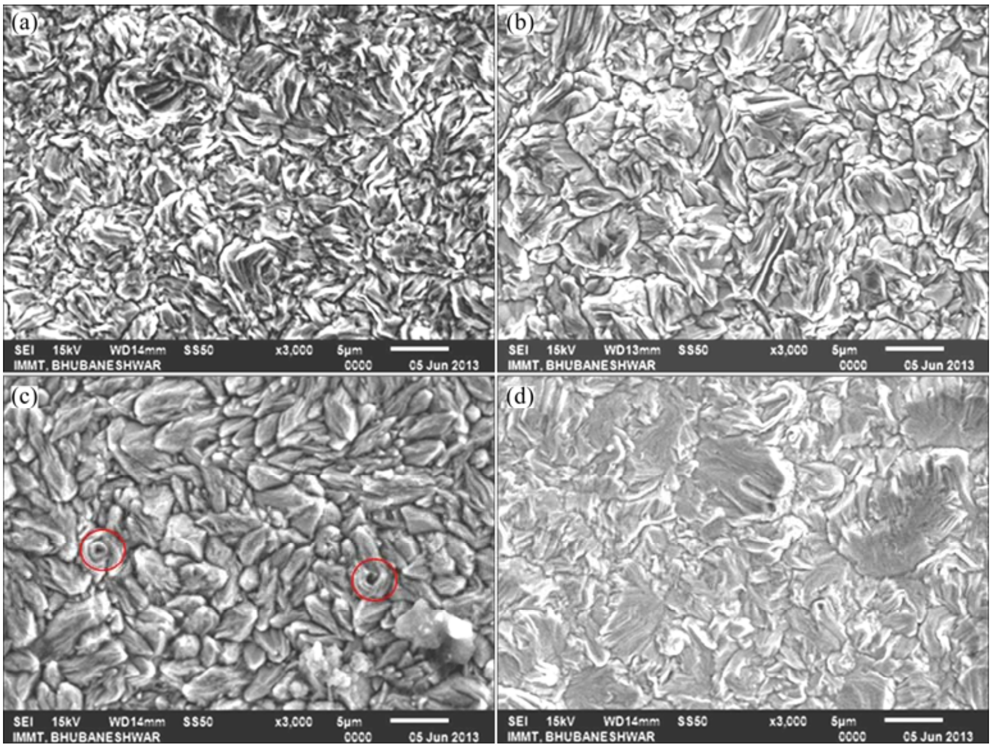

Fig. 5 SEM images of electrodeposited cobalt metal at different concentrations of additives (TEAB): (a) Blank; (b) 10 mg/L; (c) 20 mg/L; (d) 50 mg/L ([Co] in feed solution=60 g/L, pH of solution=4, electrolysis time=3 h, current density=200 A/m^2, temperature= 60 °C)

competing reaction for cobalt ion reduction. This substantiates the lower CE values during cobalt electrodeposition.

3.5 Fourier transform infra red (FTIR) studies

It is an important tool to determine the existence of any kind of association between TEAB and the cobalt ion during electrodeposition. After the addition of TEAB in the electrolytic bath, FTIR spectra were recorded before and after the electrolysis. Figure 6 showed that no chemical bonding or complex formed between the additive and the metal ion. The two peak positions at 2843 and 2913 cm^{-1} in Figs 6(b) and (c) are due to the symmetric stretching of CH$_2$ bonds of the additive. Figures 6(b) and (c) are similar, which indicates that the additive even after electrodeposition does not dissociate and keeps its identity. This also suggests that the effects observed might be only due to the physical adsorption of the additive on the cathode surface during cobalt electrodeposition. There was no sign of any bonding between the oxygen of cobalt sulphate and carbon atom of TEAB.

3.6 X-ray photoelectron spectroscopy (XPS) studies

X-ray photoelectron spectroscopy is an important characterization technique to determine the purity of the metal. Binding energies databases such as NIST database [20] generally provide sufficient data for the chemical state determination of any element, the possible state and the spin with which it is associated. Figures 7(a) and (b) show the XPS spectra of cobalt deposit obtained in the absence and presence of 10 mg/L TEAB, respectively. The full survey spectra of the deposits indicated the presence of cobalt, carbon, and oxygen elements only. The presence of oxygen may be due to the exposure of the deposits in air. This is thus an indication that the metal obtained is highly pure. Figures 8(a) and (b)

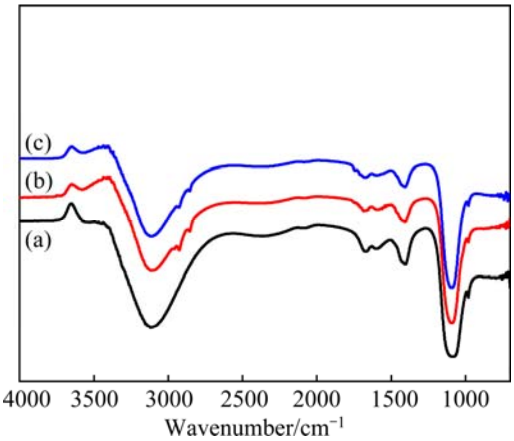

Fig. 6 FTIR spectra of cobalt sulphate solutions: (a) Blank; (b) Electrolyte with 10 mg/L TEAB before electrodeposition; (c) Spent electrolyte after electrodeposition

represent the binding energies of cobalt metal obtained from the XPS spectra of the deposits in the absence and presence of 10 mg/L TEAB, respectively. The spectral data of cobalt metal with binding energies of 778.3 and 793.3 eV corresponding to Co $2p_{3/2}$ and Co $2p_{1/2}$ states indicated the purity of the electrodeposited cobalt metal. This observation is also supported by other researchers [20,21].

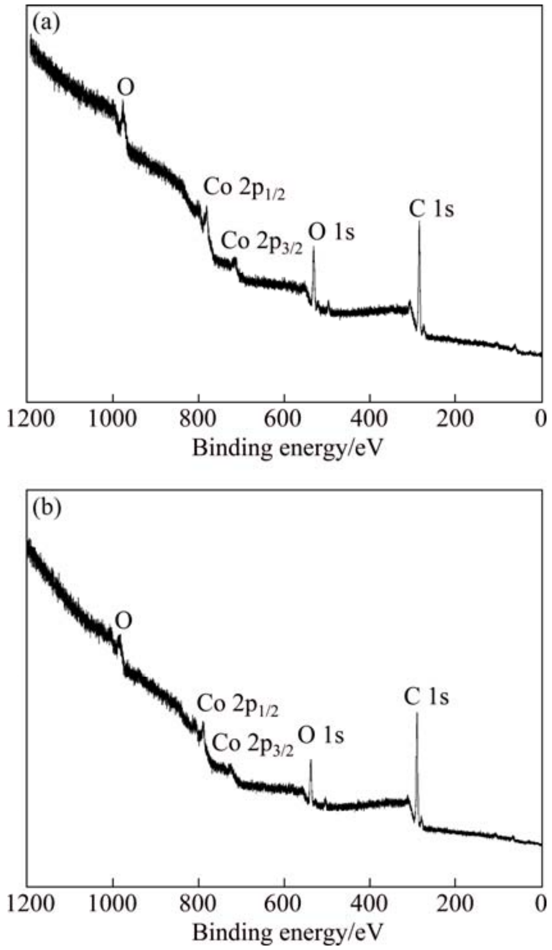

Fig. 7 XPS survey spectra of electrodeposited cobalt metal showing elements detected on surface: (a) Blank; (b) 10 mg/L TEAB

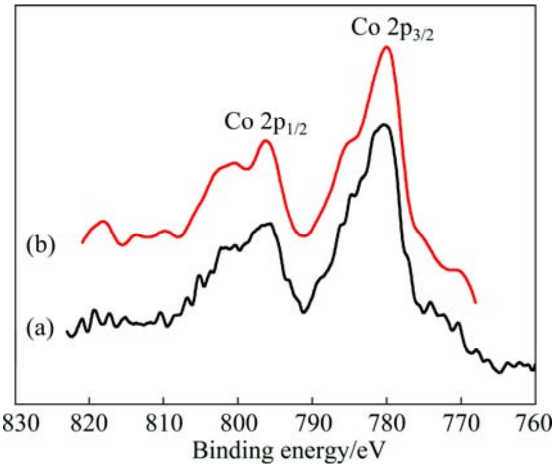

Fig. 8 XPS survey spectra of electrodeposited cobalt metal: (a) Blank; (b) 10 mg/L TEAB

4 Conclusions

The additive tetra ethyl ammonium bromide when added to the cobalt sulphate electrolytic bath at a concentration of 10 mg/L increased the cathodic current efficiency to 99.6% and decreased the energy consumption by ~0.6 kW·h/kg. Its presence in the bath resulted in bright, smooth and compact deposits with (100), (101) and (110) as preferred plane of orientations. Higher concentrations of additives not only decreased the degree of crystallinity but also resulted in deterioration in the deposit quality. Cyclic voltammetric studies indicated a shift in NOP towards more negative value indicating polarization of the cathode. XPS results confirmed the electrodeposition of high pure cobalt with no sign of chemical bonding with TEAB as evident from the FTIR spectra.

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四乙基溴化铵硫酸盐水溶液中钴的电沉积

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摘 要：在硫酸盐水溶液中制备钴金属，研究四乙基溴化铵作为添加剂对其结构和形貌特征的影响。根据四乙基溴化铵的浓度变化(范围为 1~50 mg/L)来评价其对电沉积金属钴的电流效率、能量消耗和电沉积质量的影响。当四乙基溴化铵的浓度为 10 mg/L，且电流效率为 99.6%、能量消耗为 2.38 kW·h/kg 时，可以得到平整且光亮的钴沉淀。X 射线衍射分析表明，在钴电沉淀过程中，(100)面是晶体生长的择优生长面。扫描电镜结果表明，当四乙基溴化铵的浓度为 10 mg/L 时，可获得平整均匀的钴沉淀，而浓度超过 10 mg/L 时，沉淀质量恶化。循环伏安法研究表明，在电解槽中加入四乙基溴化铵时，阴极产生极化且阴极电流大大降低。XPS 结果证实电沉积可获得高纯钴，且 FTIR 谱证明沉淀中没有与四乙基溴化铵结合的成分。

关键词：钴；四乙基溴化铵；电沉淀；极化研究；电流效率；沉淀形貌

(Edited by Xiang-qun LI)