## SHORT COMMUNICATION

# Improvement of current efficiency in the cathodic formation of metal powders

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#### 1. Introduction

The manufacture of metallic powders by electrodeposition is a well-documented process [1-4] which is often accompanied by hydrogen evolution, reducing the efficiency. Previous work [5] has indicated that, in some cases at least, the evolution of hydrogen occurs not at the powderelectrolyte interface but at the solid electrode surface on which the powder layer is being deposited. This suggests that it may be possible to increase the efficiency by the use of a cathode material having a greater hydrogen overvoltage than traditional materials such as the austenitic stainless steels, the hydrogen evolution characteristics of which have been reported [6-8]. Nickeltin alloy was chosen as a promising alternative material [9–12] which might be of more practical interest than the liquid mercury electrode which appears to be the only one of this type already tried [13].

## 2. Experimental details

The rotating cylinder electrode has been previously described [5]. It was supplied with a controlled current by using a 100 A potentiostat in the galvanostatic mode, the potential being measured using a saturated calomel electrode with the Luggin capillary bridge and a non-current carrying carbon brush contact to the cylinder spindle. Electrode cylinders were made of Type 304 stainless steel or of copper, plated with 20  $\mu$ m of 67–33% Sn–Ni alloy (kindly prepared by the International Tin Research Association). All

measurements were made at a cathode rotation speed of 200 r.p.m. and at ambient temperature.

The deposition rate was not measured by weighing the powder formed, since its collection was difficult, but by analysing the plating solution for metal depletion. This was done by electrogravimetric methods, deposition to constant weight being used with platinum gauze electrodes. The electrolytes were as follows:

(a) For deposition of copper powder

 $CuSO_4 \cdot 5H_2O$  at  $80 g l^{-1}$ 

 $NH_4OH$  (0.880) at  $100 \text{ ml } 1^{-1}$ 

This electrolyte was known to deposit copper with significant accompanying hydrogen evolution.

(b) For measuring hydrogen overvoltage on the electrodes

 $(NH_4)_2SO_4 38 g 1^{-1}$ 

NH<sub>4</sub>OH (0.880) 67 ml l<sup>-1</sup>

The pH of each solution was 11.5 and they had similar sulphate ion concentrations.

## 3. Results

Fig. 1. shows the polarization curves for hydrogen evolution in Electrolyte (b) on stainless steel (SS) and tin-nickel. These curves represent the mean values for anodic-going and cathodic-going polarization, these showing a distinct hysteresis [14]. It is apparent that at all currents the hydrogen evolution reaction is more strongly polarized on the Sn-Ni electrode.

Fig. 1. also shows the current efficiencies as

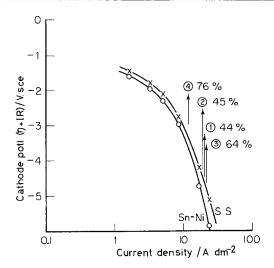


Fig. 1. Polarization data and current efficiencies for hydrogen evolution and copper powder formation.

percentage values for copper deposition from Electrolyte (a), Runs 1 and 2 being on stainless steel and Runs 3 and 4 on Sn-Ni electrodes. The vertical arrows show the current values for each run and also the range of electrode potentials during the run. In each case as a deposit of powder forms the polarization reduces and so the potential becomes more positive.

#### 4. Discussion

The results of this preliminary investigation appear to support the view that hydrogen evolution occurs mainly at the solid electrode surface and not at the powder surface. It seems practicable to improve current efficiencies in powder manufacture by searching for cathode materials of higher hydrogen overvoltage. A principle for approaching this problem has thus been demonstrated, though it should be emphasized that the question of the stability of the electrode material in service, which is of equal practical significance, has not been touched upon.

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