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# HARD NICKEL ELECTROFORMING FOR PRODUCING HOLOGRAMS

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**Holograms gives a three dimensional perception by including information on the depth. Electro forming technique has been utilized for duplicating the holographic designs produced on the optical glass. For the reproduction of the 3D images on the aluminized polyester film hard electroformed nickel stampers are produced from sulphamate bath. Nickel can be deposited either as a soft coating characterized by excellent ductility or as a hard coating to resist wear and tear depending on the applications. This could be achieved by proper conditioning of the sulphamate bath and also by the use of suitable addition agents. In this study 5-sulpho salicylic acid has been used as an additive to improve the hardness of the deposit for use in the production of holograms.**

**Keywords: Nickel electroforming, halograms, electroplating and soft coating**

## INTRODUCTION

Electroforming is a specialised application of electroplating process [1,2]. Electroplating is concerned with taking an existing article and applying metallic coating which then becomes a permanent part of the article to provide an attractive, decorative surface, a corrosion resistant coating or both [3,4]. Electroforming is however a production or reproduction of the article by electrodeposition upon a mandrel or mold that is subsequently separated from the deposit.

Electroforming is special form of electroplating in which the thickness of the deposit is much greater than that used in plating and the surface on which the deposit is formed can be separated from it at the end of the process, leaving a finished metal item which reproduces the initial surface down to the finest detail. In electroforming the deposition conditions are often altered to reduce the time necessary to build-up the much thicker deposit.

Apart from the watts nickel bath the most important is the nickel sulphamate solution which has gained a good deal of ground in recent times [5,6]. The position has been challenged from time to time by various other baths often developed for special purposes but the only one to be adopted on a really substantial scale has been the sulphamate bath. This bath sometimes needs addition agents to have some improved deposit properties like hardness, reducing internal stress, pitting, etc [7-12]. In this work 5-sulpho salicylic acid is added to the nickel sulphamate bath for improving the hardness of the deposit.

## EXPERIMENTAL

The nickel sulphamate electrolyte was prepared by adding sulphamic acid solution slowly to the nickel carbonate slurry with continuous stirring. Activated charcoal treatment was given at 328 K for removing any organic impurities present in the solution. High *p*H treatment, dummying and conditioning of the sulphamate bath were carried out in the usual manner. The concentrated solution is diluted with deionized water for getting a nickel sulphamate bath of the desired composition.

Hull cell experiments were conducted to optimise the concentration of 5-sulpho salicylic acid at which the desired deposits could be obtained. A 267 ml Hull cell was used at a plating current of 1A for a duration of 10 min. using an electrolyte of the following composition:

Nickel sulphamate 240 g/l, Boric acid 40 g/l, Hydro chloric acid 3 g/l,Sodium lauryl sulphate 1 g/l, Sodium saccharin 1.5g/l, 5-Sulpho salicylic acid 1-10 g/l *p*H 2-3.5, Temperature 303 K

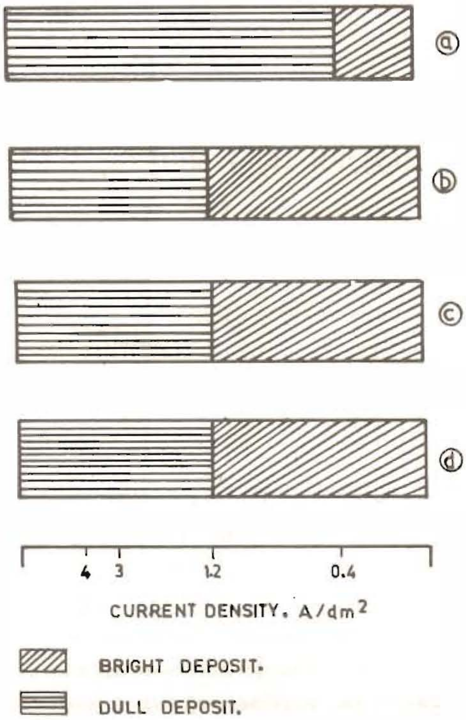


*Fig. 1: Influence of 5-sulpho salicylic acid concentration on Hull Cell pattern*

Deposition was carried out by varying the 5-Sulpho salicylic acid concentration.

The throwing power of the electrolyte was measured, by using the Haring-Blum cell at a current density of 2A/dm$^2$. The percentage throwing power was calculated using Field's formula. Experiments were conducted at various Sulpho salicylic acid concentration and also the influence of current density and temperature for a bath containing 1g/l Sulpho salicylic acid.

Current efficiency studies were carried out from the above bath containing various concentrations of Sulpho salicylic acid by passing a fixed quantity of electricity with the help of a coulometer and a regulated power supply. The influence of current density and temperature of the bath on current efficiency was also carried out. From the gain in weight the efficiency was calculated.

Linear sweep voltammetry experiment was carried out using Bio Analytical System (BAS) 100A USA and employing a conventional three electrode cell. Copper foil of one square centimeter area was used as the working electrode. Nickel foil and saturated calomel electrodes were used as auxiliary and reference electrodes respectively. The experiment is carried out using the above bath with and without Sulpho salicylic acid and at a sweep rate of 1mV/second from 0 mV to -200 mV. The resultant E-I curves were recorded.

**TABLE I: Influence of sulpho salicylic acid concentration on the current efficiency**

| Conc. S.S.A g/l | Current efficiency % |
|---|---|
| 0 | 95 |
| 1 | 92 |
| 5 | 90 |
| 10 | 85 |

The hardness of the deposit was measured using the low hardness tester M400 MS2 LECO Micro Hardness tester (LECO Corporation, USA) applying a load of 100 g. The specimens produced at various current densities (2-4A/dm$^2$), temperatures (303 - 323 K) in presence of different concentration of Sulpho salicylic acid (0-10 g/l) were tested for the hardness value of the deposits.

## RESULTS AND DISCUSSION

The Hull Cell pattern obtained from the nickel sulphamate bath containing various concentrations of Sulpho salicylic acid is shown in Fig. 1. Fig. 1(a) is the Hull cell pattern of the plain nickel sulphamate bath. It is observed that the bright deposit is obtained at a current density greater than 0.5 A/dm$^2$. With the addition of Sulpho salicylic acid the onset of bright deposit is shifted to 1.2 A/dm$^2$. Further increase of sulpho salicylic acid there is no marked change in the Hull cell pattern.

### Current efficiency

The current efficiency of the nickel deposit obtained from the bath without Sulpho salicylic acid is 95%. This efficiency is decreased gradually to 85% as the Sulpho salicylic acid concentration is increased from 1 g/l to 10 g/l. This is shown in the Table I.

### Throwing power

From the Table II it is observed that there is no appreciable change in throwing power whether the bath contains Sulpho salicylic acid or not.

**TABLE II: Influence of sulpho salicylic acid concentration on the throwing power of the bath**

| Conc. S.S.A g/l | Throwing power % |
|---|---|
| 0 | 33 |
| 1 | 33 |
| 5 | 34 |
| 10 | 34 |

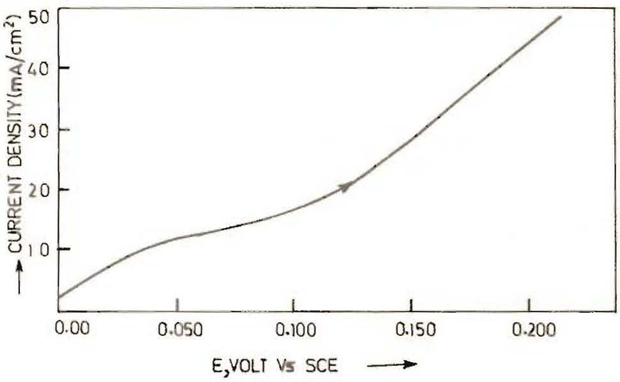


*Fig. 2: Linear polarisation curve for the plain nickel sulphamate electrolyte*

**Polarisation study**

Fig. 2 shows the linear polarisation curves for the plain nickel bath. Fig. 3 shows the bath containing 1 g/l of Sulpho salicylic acid. Addition of Sulpho salicylic acid leads to a strong polarisation effect due to electrolytic reduction and its subsequent incorporation in the deposit. This factor may lead to the achievement of higher hardness of the deposit.

**Hardness**

It is seen from the Table III that the hardness of the deposit is increased from 200 HV to 450 HV, when Sulpho salicylic acid (1 g/l) was added. With a further raise of 5-Sulpho salicylic acid to 5-10 g/l the hardness value decreased to 350 HV. From the above results it was concluded that 1 g/l of

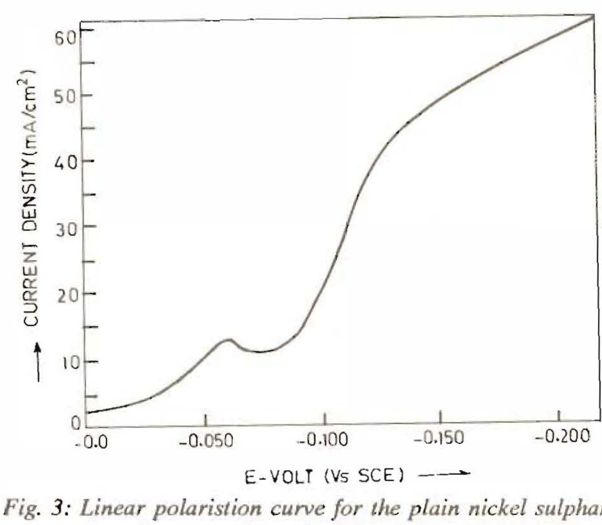


*Fig. 3: Linear polaristion curve for the plain nickel sulphamate electrolyte containing 1 g/l 5-sulpho salicylic acid*

**TABLE III: Influence of sulpho salicylic acid concentration on the hardness of nickel deposit**

| Conc. of S.S.A g/l | Hardness HV |
|---|---|
| 0 | 200 |
| 1 | 450 |
| 2 & 3 | 430 |
| 5 | 360 |
| 10 | 360 |

**TABLE IV: Influence of Current density and temperature on the current efficiency of the optimised bath**

| Current density | Current efficiency % at Temperature K | | |
|---|---|---|---|
| A/Sq.dm | 303 | 313 | 323 |
| 2 | 92 | 92 | 93 |
| 3 | 92 | 92 | 93 |
| 4 | 93 | 93 | 95 |

**TABLE V: Influence of Current density and temperature on the throwing power of the optimised bath**

| Current density | Throwing power % at Temperature K | | |
|---|---|---|---|
| A/Sq.dm | 303 | 313 | 323 |
| 2 | 32 | 32 | 33 |
| 3 | 34 | 34 | 34 |
| 4 | 34 | 35 | 36 |

5-Sulpho salicylic acid is the optimum concentration chosen for further studies.

**Influence of the optimum concentration of 5-sulpho salicylic acid**

**Current efficiency**

From Table IV it is observed that the current efficiency is affected neither by temperature of the solution nor by the operating current density.

**Throwing power**

It is seen from Table V that the throwing power of the bath is not changed appreciably as the deposition current density and the temperature of the electrolyte are increased.

**TABLE VI : Influence of Current density and temperature on the hardness of the optimised bath**

| Current density A/Sq.dm | Hardness (HV) at Temperature K 303 | 313 | 323 |
|---|---|---|---|
| 2 | 420 | 450 | 470 |
| 3 | 425 | 455 | 480 |
| 4 | 440 | 455 | 490 |

**Hardness**

The hardness of deposit is not changed much with increase of current density from 2 to 4 A/dm$^2$, whereas the hardness of the deposit is increased from 420 HV to 490 HV as the temperature of the bath is increased from 303 K to 323 K, as shown in Table VI.

## CONCLUSION

Utilization of 5-Sulpho salicylic acid as additive in the nickel sulphamate electroforming bath improves the hardness of the nickel electroform from 200 HV - 490 HV, which is suitable for holographic applications.

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