# Effect of Boric Acid, $Cl^-$ and $K^+$ on the Physical Properties of Nickel Electrodeposited from DMF/Water Mixture Bath

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Addition of boric acid, nickel chloride and potassium acetate to a nickel acetate-5 mol percent DMF/water bath produced softer deposits with a reduced number of cracks. Micrographs of electrodeposits obtained from the nickel acetate bath without addition had multiple cracks near side edges, microhardness of these deposits being greatest. Addition seems to help relieve the internal stress, reducing the number of cracks.

Preliminary studies of nickel electrodeposition in the dimethylformamide/water mixture baths(1) showed that good hard deposits of nickel can be obtained at a moderate cathode current efficiency. Electrodeposition of nickel using nickel acetate was found to produce harder and brighter deposits as compared to the use of nickel sulphate. The acetate salt along with boric acid was more effective in keeping the solution buffered. The effect of pH on the physical properties of nickel electrodeposited from sulphate salt in a particular composition of 5 mol percent DMF-water mixture bath was quite marked(2).

There exists a relationship between pH and hardness(3,4) and also between the oxygen content and the hardness of the deposit(4). The stabilization of the basic colloidal hydroxides formed during the electrodeposition process by the positively charged potassium ions and destabilization by the negatively charged chloride ions has also been reported(3).

Previous studies on electrodeposition of nickel from acetate salt in 5 mol percent DMF-water mixture gave hard deposits but scanning electron micrographs (SEM) of the surface of the deposit revealed multiple cracks near the edges (5). These cracks were attributed to the high internal stress produced during electrodeposition because of fine-grained deposition-especially at the edge due to high current density.

The present investigation relates to the study of the effect of the addition of boric acid, nickel chloride and potassium acetate on the physical properties viz. microhardness and microstructure of the electrodeposits obtained from a nickel acetate-5 mol percent DMF/Water mixture bath.

## EXPERIMENTAL

The experimental set-up was the same as in earlier work(1). For electrodepositing nickel, mechanically polished (4/0 emery paper) copper electrodes of 2.5 cm x 2.0 cm size were used, after thorough cleaning and electrocleaning as described in detail earlier(5). Composition of the different baths and the characteristics of the electrodeposits obtained from them are given in Table 1.

Microhardness tests of the surface of the electrodeposits were carried out on a Tukon Wilson Microhardness tester as described earlier(1). A load of 50 gm was used for all indentations. The average values of the microhardness (DPH) of nickel electrodeposits obtained from different baths are also included in Table 1.

## RESULTS AND DISCUSSIONS

Table 1 gives the bath compositions and the quality of deposits obtained at respective cathode current efficiencies (CCE). It is maximum from bath A, (containing only nickel acetate) but changes only slightly in the addition of other salts. The variation in the pH value of the different bath solutions is also low, pH of bath A being maximum (5.1). The quality of electrodeposited nickel improved from dull to gray (with bright edges) by the addition of boric acid (Bath B; Table 1). The deposits obtained from bath C and D were gray (with whitish streaks).

The deposit obtained from Bath A has a maximum average microhardness value (475). The addition of boric acid (Bath B) softens the deposit; microhardness decreases. The increase in the concentration of both $Cl^-$ and $K^+$ softens the deposit.

The scanning electron micrograph (SEM) of a typical portion of the surface at the side edge of the electrodeposited nickel from Bath A shows multiple cracks. These may have developed due to the increased stress there(5). Addition of boric acid reduces the number of cracks. The side edge of the deposit has a single crack while the central portion of the deposit is completely free

**TABLE 1**

BATH COMPOSITIONS AND CHARACTERISTICS OF ELECTRO-DEPOSITS OBTAINED IN 5 MOL PERCENT DMF/WATER

Anodes high purity nickel;
Interelectrode distance, 2.0 cm ;
Copper strip used as substrates, 2.5 cm x 2.0 cm;
Current density (CD), 1.5 A/dm$^2$
Temperature, 35°C
Duration of electrolysis, 30 mins;
Agitation, nil. For microhardness testing (DPH) a load of 50 g was used. Load for microhardness testing (DPA), 50 g

| Bath | Content | | Conc (M) | pH | (CCE %) | Quality of electro deposits | DPH |
|---|---|---|---|---|---|---|---|
| A | Nickel acetate | | 0.5 | 5.1 | 79 | Dull | 475 |
| B | Nickel acetate | | 0.5 | | | | |
| | + Boric acid | 1. | 0.05 | 5.1 | 76 | Gray,edges bright | 391 |
| | | 2. | 0.1 | 4.9 | 76 | - do - | 371 |
| | | 3. | 0.2 | 4.8 | 70 | - do - | 320 |
| C | Nickel acetate | | 0.5 | | | | |
| | +Nickel Chloride | 1. | 0.01 | 5.0 | 72 | Gray,whitish streaks | 371 |
| | | 2. | 0.05 | 4.9 | 75 | - do - | 356 |
| D | Nickel acetate | | 0.5 | | | | |
| | +Potassium acetate | 1. | 0.01 | 5.0 | 76 | Gray,whitish Streaks | 401 |
| | | 2. | 0.05 | 5.0 | 71 | - do - | 328 |

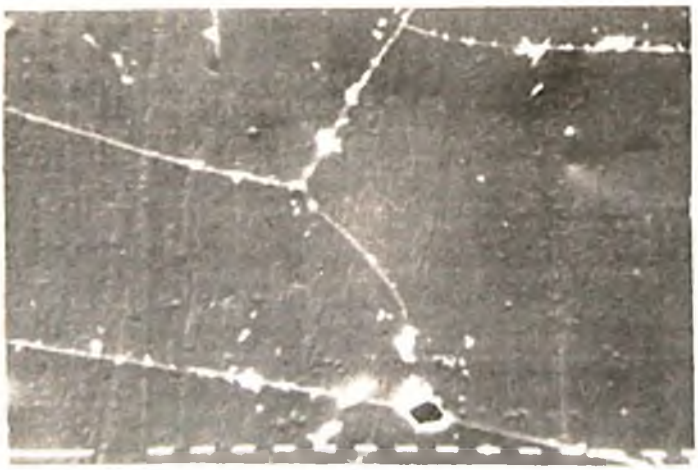


Fig. 1 Bath A 288 x, side edge

of cracks. The deposits obtained from Bath C and D also had a reduced number of cracks at the side edge while the central portion was free of cracks. It seems that the addition of boric acid, nickel chloride and potassium acetate to the nickel acetate bath helps in relieving the stress in the deposit to some extent. The decrease in the microhardness value with the increase in the concentration of the additives corborates this finding.



Fig. 2a Bath B, 1 1500 x, side edge



Fig. 2b Bath B, 1 2750 x, centre

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