2.1 Substrate for the electrodeposition of Nickel

Initially copper strips of dimension 2 cm x 5 cm x 0.1 cm were used as substrates for nickel electrodeposition studies but in most of the subsequent experiments the copper strip of dimensions 2 cm x 1 cm x 0.1 cm were used. The strips were mechanically polished with emery papers 1/0, 2/0, 3/0 & 4/0, degreased in soap (Idipol) solution for some time, washed thoroughly, dipped in acetone and dried under the folds of a warm filter paper prior to putting in the dessicator. The copper strip was weighed at this stage and then electrocleaned cathodically without sodium hydroxide for 4 min as recommended by Leiner. The electrocleaning solution was maintained at a temperature around 50-55°C, as in hotter solution the drag-out dried on the
part resulting in inferior quality of deposits\textsuperscript{210}. Immediately after electrocleaning, the strip was pickled in a light picking solution for 4 min and then made the cathode in the experimental cell. In between each operation the copper strip was carefully washed with double distilled water. The electrocleaned specimen (the cathode) for nickel plating was placed in between two parallel pieces of high-purity nickel anodes. The interelectrode distance in all the experiments was maintained at 2.0 cm. The different solutions used for the electrodeposition of nickel were purified by electrolysis at low current density\textsuperscript{211}. All experiments were carried out without agitation. Current was measured by a copper coulometer. The cathode current efficiency was calculated after measuring the weight of the nickel deposits.

A glass container (150 ml capacity) was used as an electrolysis cell for nickel electrodeposition. It was covered with a wooden block that did not seal airtight and contained holes for connecting the electrodes. The electrolysis cell was placed in a thermostate to maintain a uniform temperature inside the cell (Fig1).
Experimental setup

a - Nickel anodes, c - Copper strip cathode
V - Voltmeter A - Ammeter
Cu - Copper Coulometer B - Battery
T - Thermostat

Fig.1
2.2 Purification of the solvents

2.2.1. N, N-Dimethylformamide

N, N-Dimethylformamide was purified as suggested by Sears and coworkers. N, N-Dimethylformamide (E. Merck) was allowed to stand in contact with solid potassium hydroxide (A.R.) for a day. After decanting, the liquid was fractionated at atmospheric pressure. The large middle fraction, which was obtained, was again distilled.

2.2.2 Formamide

Formamide was purified as suggested by Dawson and coworkers. Formamide (IDPL, India) was purified by several distillations over calcium oxide at a pressure less than 0.1 mm where the temperature remained between 51 and 58°.

Amide-water mixture of different compositions were prepared by weight using double distilled water. Water was always added to the amides and the mixture

[54]
was allowed to stand for sufficient time to attain room temperature before the addition of nickel salts or boric acid or other additive.

2.3 Physical properties of the electrodeposits

(i) Microhardness

Microhardness was measured on the surface of the nickel deposits using a Tuken Wilson microhardness tester and a $136^\circ$ diamond pyramid indenter. The diamond pyramid (Vickers) hardness number expressed in Kg/mm$^2$ is a function of the load ($P$) applied to the indenter and the surface area of the indentation:

$$DPH = \frac{2P \sin \theta}{d^2}$$

where $\theta$ is the angle between opposite faces of the diamond ($136^\circ$). The diagonal of the indentation ($d$) was obtained in mm by multiplying the actual mean filar reading with the objective calibration factor, which was 0.85 for an objective lens magnifying 10 times and 0.212 for an objective lens magnifying 40 times. A load
of 10 g was used for all indentations. A number of indentation measurements were taken and the average, along with the statistical variation, has been reported in the results.

(ii) Micro structure

The surface of the electrodeposits and the micro indentations on the surface of nickel were scanned on JEOL SM-840 A at B.H.U. Varanasi Metallurgy Department. The specimen for scanning electron micrography were mounted on studs with the help of silver paste.

(iii) Cathode current efficiency (CCE)

CCE was computed as the ratio of the weight of the nickel actually electrodeposited to the maximum that could be deposited by that quantity of the current in accordance with Faraday's law. The cathode current efficiency of the deposited nickel was calculated using the formula

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\text{CCE} = \frac{\text{Wt. of nickel deposited on cathode in electrolytic cell \times 108}}{\text{Wt. of copper deposited on the cathode in Cu Coulometer}}
\]