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CHAPTER-2

EXPERIMENTAL SET UP

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### EXPERIMENTAL

#### 2.1 Experimental Set Up :

The experimental set up used for single metal and alloy deposition is illustrated in fig.2.1. It consists of an electrolytic cell (Glass beaker of 250 ml) containing bath solution. It is provided with a precision type ammeter, electrodes with holders, a copper coulometer, an adjustable resistance and D.C. power supply unit connected in series.

Electroplating or Electrodeposition is the process by which the coating metal is deposited on the base metal by passing a direct current through an electrolytic solution, containing the soluble salt of the coating metal.

The base metal to be plated, is used as cathode of the electrolytic cell, whereas the anode is either the coating metal itself or an inert material of good electrical conductivity.

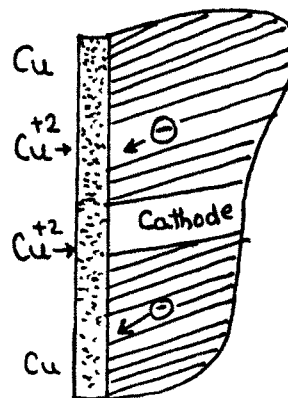


Fig.: 2.2. In metal plating which occurs at cathode a forced supply of electrons causes a reversal of the corrosion reaction.

Corrosion and electroplating are opposite. Corrosion can be defined as destruction and loss of a solid metallic through an unintentional chemical or electrochemical attack, by their environment starting at its surface, e.g. Rusting of Iron.

During electrolysis electrons are introduced from an external source as in fig. 2.3. Thus in principle, electroplating is the reverse of corrosion i.e. in electroplating metal is deposited from solution, while in corrosion metal is dissolved in solution. Corrosion always occurs at the anode and electroplating always occurs at the cathode.

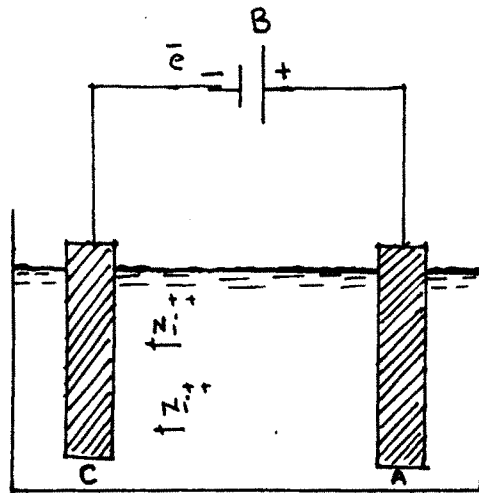


Fig.: 2.3 Electroplating of Ni. Electrons are forced to be the cathode by external source like battery.

2.2 : EXPERIMENTAL PROCEDURE FOR THE PREPARATION OF  
BATH SOLUTION :

The standard stock solution of the required compounds were prepared in double distilled water. The chemical bath was prepared by adding together the calculated amounts of the required constituents and then making up the total volume with double distilled water accurately to 100 ml.

2.3 : COPPER COULOMETER AND PREPARATION OF OTTEL'S  
SOLUTION. :

A copper coulometer was found to be more convenient and relatively more accurate than others to measure the quantity of electricity passing through the circuit, during electrolysis. The coulometer solution was prepared as per Ottel's recommendations.

Thus it was prepared by adding 150 gms of recrystallised copper sulphate, 50 gms of sulphuric acid ( sp. gr. 1.84 ) and 80 ml of ethyl alcohol in a litre of double distilled water. During electrolysis, the coulometer cell containing the Ottel's solution and provided with two copper plates of the same size to serve as anode and cathode, were connected in series with the experimental cell. The copper

cathode in the coulometer was weighed accurately before and after deposition. It was washed with distilled water and alcohol, before weighing. The amount of copper deposited in the coulometer during electrolysis gave the measure of current from which cathode efficiency for metal deposition was calculated.

#### 2.4 ; ANODE AND CATHODE MATERIALS :

Anodes used in the work of pure metals such as nickel sheet ( 5 cms X 2.5 cms ) or platinum electrodes ( 5 cms X 2.5 cms ) etc. depending on the nature of the deposit.

Copper plates of desired size were used as substrate for deposition. Copper is preferred as cathode to any other base metal, because it is easier to clean and much smoother surface can be obtained for deposition than any other metal. <sup>F</sup>for qualitative and quantitative work cathode of copper or of platinum of the same dimensions were employed.

Even a trace of grease on the cathode spoils the deposition. The cathode should be as clean and greaseless as possible. Hence its cleaning is very important during deposition. The cleaning was carried out as follows :

A thin rectangular copper plate cut according to the desired size was first rendered free from grease by dipping it in a hot and strong 20% alcoholic caustic soad solution for a while, and then washed with water.

It was then immersed in moderately strong nitric acid solution, containing very little concentrated sulphuric acid and copper nitrate. This removes the surface scales. The copper nitrate checks the violent action of the acid on the plate. It was then washed with sodium carbonate and again dipped in a slightly acidic solution to remove the alkali adhering to the plate. This is done because acids are easier to wash off from copper plate than alkalies which adheres to the plate and have pronounced chemical action on the metal. Then the copper plate was thoroughly washed with tap water, distilled water and finally with alcohol or acetone, and then dried well. It was then accurately weighed and used as cathode.

#### 2.5 WORKING PROCESS OF ALLOY DEPOSITION ;

The experimental set up of the apparatus described in fig. 2.1 is first arranged and then 200ml of the chemical bath prepared according to 2.2 were taken in the - electrolytic cell provide with stirring arrangement. To avoid deposition by immersion, the current was first switched on and then a well cleaned rectangular cathode plate

(5cm x 2.5 cm ) was placed between two parallel anodes and the electrolysis carried out under a definite set of experimental conditions specified under the corresponding tables.

In almost all the experiments, duplicate runs were taken from the same bath solution which was discarded after the work and the metals were recovered from the waste.

After electrolysis, the cathode which was previously weighed, was withdrawn from the cell and washed thoroughly under tap water, which was followed by distilled water and alcohol wash. It was then dried, cooled in the vacuum desiccator and finally weighed accurately. The difference in weight before and after deposition gave the amount of alloy deposited during electrolysis.

The ammeter measured the current in amperes passing through the cell. Copper coulometer gave the quantity of electricity accurately passing through the circuit during electrolysis.

In order to establish the optimum conditions for the production of smooth, fine grained, bright, uniform, adherent and comparatively harder deposit, the effect of the following parameters was studied in details in each of the systems.

1. Concentration of the electrolyte
2. Current density.
3. Temperature
4. Duration of electrolysis
5. Electrode distance
6. Addition agents.

#### 2.6 ANALYSIS OF DEPOSITS :

The accurately weighed deposit was carefully dissolved from the cathode in the minimum amount of conc. HCl/HNO<sub>3</sub> and then diluted with double distilled water accurately to a known volume, from which qualitative and quantitative analysis was done.

First of all the presence of metal ions in the test solution of the pertinent deposit was detected by applying the usual tests in qualitative analysis.

After ascertaining the presence of particular ions in the qualitative analysis of the metal was carried out by either chemical or physical method. Various physical methods were employed for analysis of alloy deposit as well as for bath solutions by different workers in the past. For example, colorimetric and spectrophotometric methods were used for analysis by Claubaugh W.S. and Bayley Spectrographic analysis was followed by Garden H.E.Z. and Rober, E.R. while polarographic methods were used by Kolthoff, I.M. and Lingane, J.J., Sing V.B. and Tikoo P.R.



However, on comparison of various methods of analysis, it has been found that colorometric and chemical methods suitable for our work.

Employing these methods, the estimations of single metal content or two metal contents ( binary system ) at a time were carried out.

Principles and relevant procedures for quantitative estimation of  $Ni^{+2}$  ,  $Fe^{+2}$  individually or in presence of each other have been adopted from standard text books.

Calculations :

From the knowledge of the weights of the alloy deposit and the codeposited metals, the metal content in the alloy deposit was calculated. Similarly knowing the weight of the copper deposited in the coulometer, cathode current efficiency (%) for each metal was determined on the basis of Faraday's laws.

The following formula was used -

$$C C E \% = \frac{100 \times \text{Eq. Wt. of Copper} \times \text{Wt. of metal deposited.}}{\text{Wt. of copper deposited in the coulometer} \times \text{Eq. wt. of metal}}$$

The results of metal content and CCE% for each parameter are shown under respective tables and graphically under corresponding figures.

2.7 : MICROPHOTOGRAPHS :

The nature especially the grain size and the structure of the deposits was visually examined with a microscope having high magnification. Microphotographs of the deposits were taken wherever necessary, using uniform magnification in all the photographs.