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**CHAPTER - 2**

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**EXPERIMENTAL**

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**2.1 EXPERIMENTAL SET UP**

The experimental set up used for single metal and alloy electro-deposition is illustrated in fig.2.1. It consists of an electrolytic cell (a pyrex glass beaker of 150 ml capacity), containing bath solution. It was provided with a precision type ammeter, electrodes with holders, a copper coulometer, an adjustable rehostat, a D.C. power supply unit all connected in series but voltmeter was connected in parallel as shown in the diagram. The thermostat, with stirring arrangement was used to maintain the required temperature of the bath solution.

**2.2 EXPERIMENTAL PROCEDURE FOR THE PREPARATION OF BATH SOLUTION**

For the preparation of the stock solution the double distilled water was used. The chemical bath as detailed under the respective tables 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 200 ml.

**2.3 COPPER COULOMETER & PREPARATION OF OTTEL'S SOLUTION**

In order to measure the cathode current efficiency it is necessary to measure accurately the quantity of electricity flowing through the bath. The most accurate and commonly used technique is to place a coulometer in series with the bath.

A coulometer is a small electroplating cell in which the deposition process is carefully chosen so that all the current flowing through the cathode is used in one electrode process only, such as metal depo-

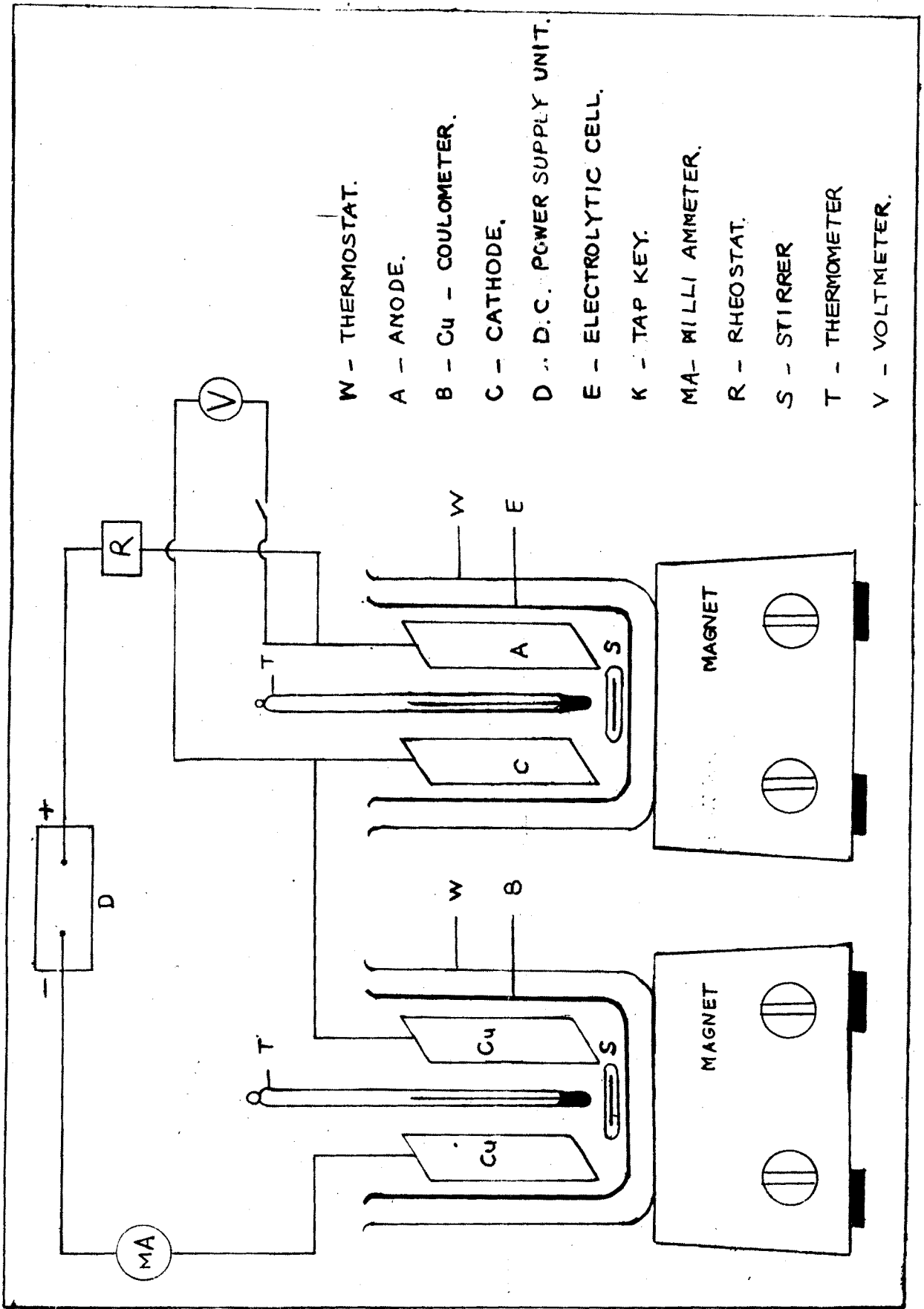


FIG. 2-1- EXPERIMENTAL SET UP FOR ELECTRO-DEPOSITION.

sition, i.e. a process with cathode efficiency of 100%. A common type of coulometer is a copper coulometer which is found to be more convenient and relatively more accurate than others. It consists a copper cathode and anode of same size and solution of composition,

Copper Sulphate hydrated	..	150 g/L
Sulphuric acid	..	50 g/L
Ethyl alcohol	..	80 ml

This composition was as per Ottel's recommendations (7).

Thus Ottel's solution was prepared by adding 150 g of recrystallised copper sulphate, 50 g of sulphuric acid (sp. gr. 1.84) and 80 ml of redistilled ethyl alcohol were dissolved in a ~~litre~~ of double distilled water and diluted to one litre.

The coulometric cell containing the Ottel's solution, 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 ample 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 MEASUREMENT OF pH

Usually, high accuracy is not required in measuring pH for plating baths. Most practical plating processes work well in a range of pH units at least 0.5 unit wide, and an accuracy of 0.2 pH units is sufficient for control.

pH of the bath solution was measured by electrometric method. A standard commercial instruments, equipped with a glass electrode and a reference calomel electrode, both contained in the bulb, was used in the present work.

## 2.5 ANODE AND CATHODE MATERIALS

In most cases the positive electrodes or anodes consists of plates or bars of the metal that was to be plated, i.e. soluble anodes. In some cases insoluble anodes are also used such as platinum electrodes, steel electrodes etc.

Anodes used in the work were of pure metals such nickel sheet (5 x 2.5 cm) or platinum electrodes (5 x 2.5 cm).

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

## 2.6 CLEANING OF THE CATHODE MATERIAL

In all types of electroplating, it was important that the base metal to be plated was absolutely clean. The common foreign materials likely to be present on metal surfaces are of the type such as grease or oil, scale etc. In general, it was essential to remove all foreign substances from a metal surface prior to plating. In fact, it was considered that adherent, impervious coatings could be applied only to metal surfaces that were perfectly clean.

Even a trace of grease on the cathode spoils the deposition. The cleaning was carried as given below.

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 soda solution, for 20 min. The grease, fats & oils gets converted into soluble soaps by the saponification reaction. Then it was washed with water. Then the plate was immersed

in a moderately strong nitric acid solution, containing ~~five ml~~ concentrated sulphuric acid and copper nitrate. This treatment 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 was done because acids are easier to wash off from copper plate than alkali which adhere 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.

## 2.7 WORKING PROCESS OF METAL & ALLOY DEPOSITION

The experimental set up was done as shown in the fig. 2.1. The electrical connections were made by using wires. Then 100 ml of the chemical bath prepared according to 2.2 was taken in the electrolytic cell. The cell was kept in the thermostat to attain the desired temperature ( $\pm 0.1^\circ\text{C}$ ) of the bath solution. To avoid deposition by immersion, the current was first switched on and then well cleaned rectangular cathode plate (5 cm x 2.5 cm) was placed in front of the anode and the electrolysis was carried out under a definite set of experimental conditions specified under the respective 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 metal or alloy deposited, during electrolysis.

The ammeter measures the current in amperes passing through the cell and the voltmeter recorded the voltage. 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 comparatively harder deposit, the effect of the following parameters was studied in detailed in each of the system :-

- 1] Concentration of the electrolyte
- 2] Current density
- 3] Hydrogen ion concentration
- 4] Temperature
- 5] Duration of electrolysis
- 6] Electrode distance
- 7] Addition agents.

## 2.8 ANALYSIS OF DEPOSITS

For the analysis of the deposits of metals and alloys of various metals, various physico-chemical methods were used. Firstly for the detection of the metal ions the qualitative tests were applied.

The accurately weighed deposit was carefully stripped from the cathode in the minimum amount of the stripping solution, i.e. solution containing definite proportion of conc. sulphuric acid and fuming nitric acid and then diluted with double distilled water accurately to the known volume, from which qualitative and quantitative analysis were carried out.

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

After ascertaining the presence of particular ion in the test solution, the quantitative analysis of the metal was carried out by either chemical or by physical method.

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

Analysis of the deposits, whether of single metal or alloy, was made by Atomic Absorption spectrophotometry (AAS).

## 2.9 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 of electrolysis.

The following formula was used -

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

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

## 2.10 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 optimum condition were taken by using uniform magnification (x40) in all the photographs.