

CHAPTER - II

EXPERIMENTAL

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The experimental part of the present work is discussed here, in brief, with respect to following points.

2.1 EXPERIMENTAL SET UP :

The typical experimental set up used for electro deposition of single metals and alloy, is as shown in fig. 2.1. Mainly it consists of two electrodes (cathode and anode) dipped in a bath solution of an electrolyte. It is provided with a stirring arrangement, precision type ammeter, a coulometer and D.C. power supply unit (Aplab - 7113) are all connected in series. The voltage applied across the cell is measured by connecting two electrodes to a voltmeter.

2.2 PREPARATION OF BATH SOLUTION :

The standard stock solution of the required compounds (A.R.grade) were prepared in double distilled water. The actual chemical bath was then prepared by adding together, the calculated amounts of the required constituents as detailed under the respective tables and then making up the total volume with the distilled water accurately to 100 ml.

2.3 ANODE AND CATHODE MATERIAL:

For all the experiments anode used was platinum in the form of thick (usually 2 mm) plates with surface area approximately 10 sq. cm. (4 cm x 2.5 cm.).

Copper plates of desired size i. e. same as above (10 sq. cm. area), were used as cathode for electrodeposition of metal. Copper is preferred as a substrate or base metal because it is easier to clean and much smoother surface can be obtained for deposition, than any other metal.

2.4 CLEANING OF THE BASE METAL :

In electrodeposition work cleaning of the base metal (cathode) is of supreme importance. A practical definition of the word clean is “containing no contaminants that would interfere with satisfactory deposition of an adherent finish”.

A particular cleaning cycle adopted here to remove surface contaminants and oxides if any, is as follow:

- i) Degreasing of the copper plate was carried out by dipping it in a hot, twenty percent caustic soda solution for five minutes and then it was rinsed with water.
- ii) The plate was then dipped in moderately strong nitric acid, containing a few drops of conc. sulphuric acid and some copper nitrate. It removes and surface scales or oxide film. The presence of copper nitrate checks the violent action of acid on the plate.
- iii) The place was then washed with sodium carbonate and again dipped in a dilute acid solution (acetic acid) to remove the alkali, adhering to the plate. This was done, because acids are easier to wash out from copper plate than alkalies which adhere to the plate and have pronounced chemical action on the plate.
- iv) Finally the plate was washed thoroughly, with tap water, distilled water and alcohol successively and dried well. It was then accurately weighed and used as cathode.

Before drying, the cleanliness of the plate was tested by a very simple, so called, water break test. This test depends on the fact that clean metal surfaces are hydrophilic and water sheds on such as surface in an unbroken sheet. If traces of grease remain on the surface the water will run off, leaving unwetted areas, which can be easily observed. So, simple visual examination was sufficient to give an idea of whether the surface was suitable for deposition or not. The cleaned plates are kept in vacuum-desiccation before their use.

2.5 COPPER COULOMETER :

The quantity of electricity passed through the bath solution during electrolysis was measured by using copper coulometer. The stock solution for coulometer was prepared as per Ottel's recommendation. (7) Thus 150 gms. of recrystallised copper sulphate, 50 gms. of conc. Sulphuric acid (sp. Gravity 1.84) and 80 ml. of ethyl alcohol were dissolved in one liter of double distilled water. During electrolysis, the coulometer cell containing 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 cleaned copper cathode in the coulometer was weighted accurately before and after deposition. It was washed with distilled water and alcohol before weighting. The amount of copper deposited in the coulometer during electrolysis gave a measure of current from which cathode efficiency for metal deposition was calculated.

2.6 MEASUREMENT OF pH :

Usually, high accuracy is not required to 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 instrument, equipped with a glass electrode and a reference calomel electrode both contained in the bulb, was used for the present work.

2.7 ELECTRODEPOSITION- WORKING PROCESS :

The apparatus was arranged as illustrated in fig. 2.1 and 100 ml. of the bath solution were taken in electrolytic cell. The bath solution was stirred mechanically using magnetic stirrer. The cell was kept at the desired constant temperature in thermostat. To avoid deposition by immersion. Current was first switched on and then the cleaned copper plate which served as the cathode, was dipped in the bath. Electrolysis was carried out under a definite set of experimental conditions, specified under the corresponding tables. The ammeter measured the current in amperes passing through the cell and the voltmeter recorded the voltage. Copper coulometer gave the quantity of electricity passing through the circuit during electrolysis.

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 desiccators and finally weighed accurately. Increase in the weight gave the amount of the metal or alloy deposited during electrolysis.

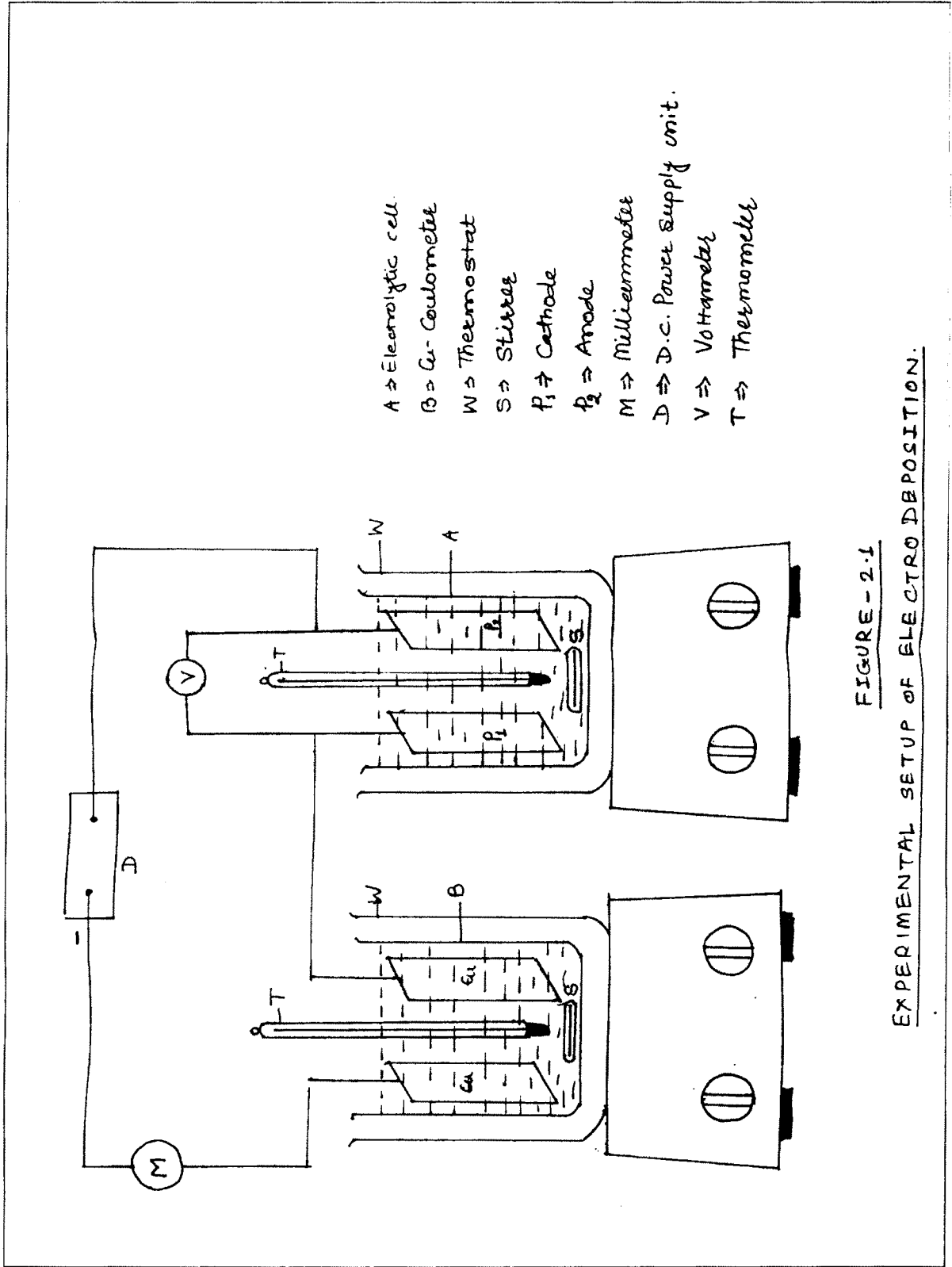


FIGURE - 2.1
EXPERIMENTAL SETUP OF ELECTRO DEPOSITION.

2.8 MEASUREMENT OF OVERVOLTAGE(POLARISATION) :

Direct measurements of over voltage (polarisation) accompanying the electrodeposition of metal at various electrodes were made by observing the potential of the electrodes, when current was passing through the solution.

The electrical set up used for this purpose is illustrated in figure 2.2. It consists of electrolytic cell C (150ml.pyrex beaker) containing electrodes of known cross sectional area(10 sq. cm.) and stirrer S .Electrodes are connected to D.C. supply unit through a milliammeter M.. The electrode A under study is dipped in a bath solution and combined with another electrode B .The potential of electrode A is determined by coupling it with a saturated calomel electrode F, which acts as a reference ,through a salt –bridge E. In order to avoid inclusion of any appreciable IR drop through the solution in measuring the potential of A against the reference electrode ,the arm of the salt-bridge is drawn out to a fine tip and placed pressed against the electrode A (Luggin capillary) .

To make measurement the stirrer is started and the current through the electrolyzing circuit is adjusted to small values (50 mA) as indicated by M. After the current has become steady, the emf of the cell containing electrode A and F was measured by means of potentiometer P. The current is then readjusted to next higher value and the whole operation was repeated .In this manner a series of emf readings corresponding to various values of the current passing through the cell C were obtained.

On dividing the current strength at each point by the area of the electrode A, the current density was calculated and electrode potential of electrode A was obtained by subtracting from the measured emf, the potential of the reference electrode.

Finally, in order to arrive at the overvoltage the reversible potential of A was subtracted from the observed electrode potentials. The reversible potential of A was measured by taking the potential of A against the reference electrode when no current was flowing through the cell.

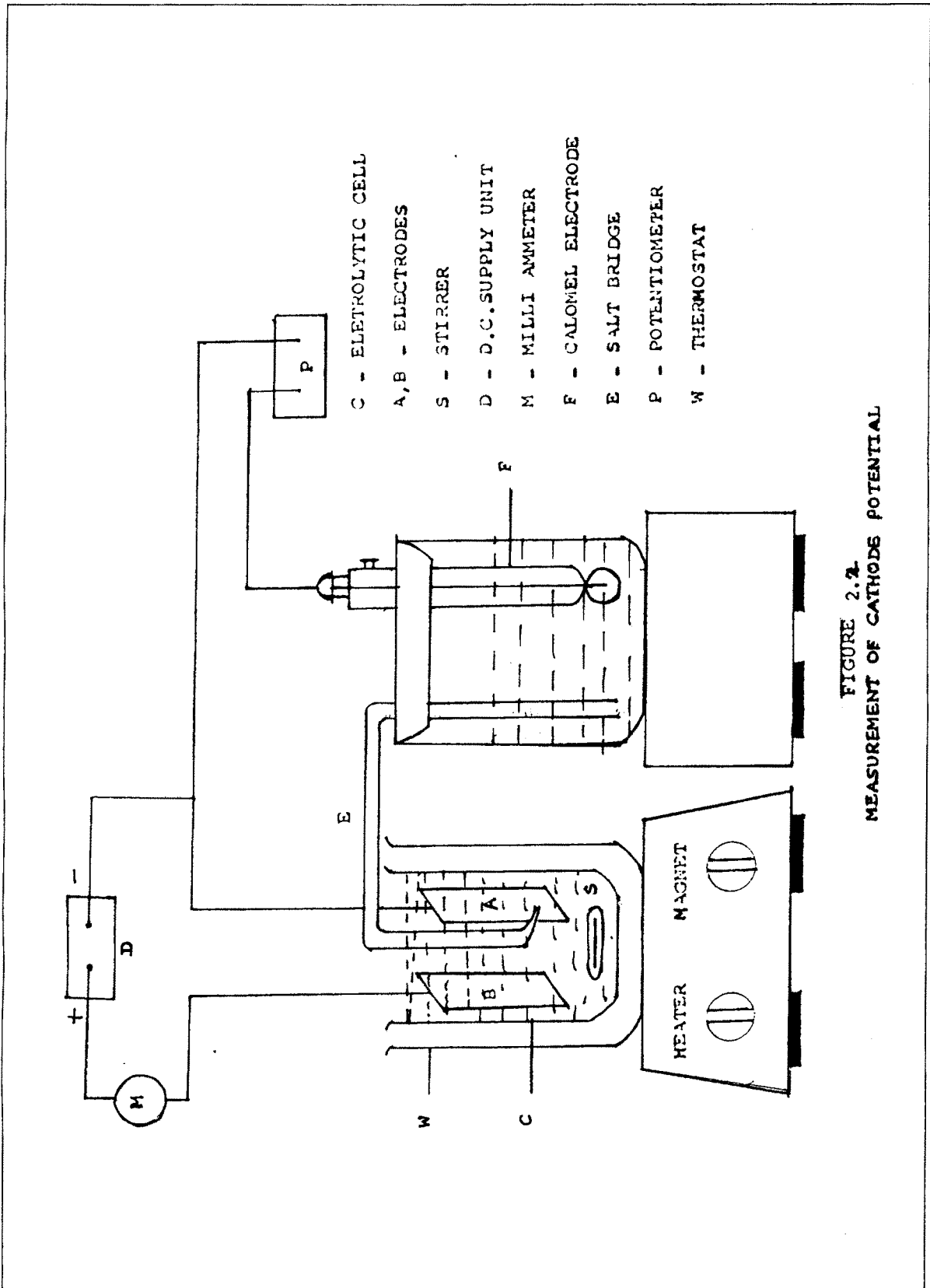


FIGURE 2.2
MEASUREMENT OF CATHODE POTENTIAL

2.9. ADHESION TEST :

The usual meaning of the term adhesion is the extent or degree to which one thing sticks to another. The adhesion of a coating to the basic metal is, of course, of considerable importance to the quality of a plated item. Electrodeposits obtained on a clean metallic substrate usually have much more adhesion.

A very complex bibliography and review of the methods of measuring adhesion has been recently published.

The adhesion, of deposits obtained by us for optimum conditions was tested by carrying out the following testes.

- a) **Bent Test:** The coated copper plate was bent through an angle of ninety degree or more and the deformed part was examined for peeling or flaking of the coating from the substrate.
- b) **Heat-quench Test:** The coated copper plate was heated in an oven to a temperature of 250° C and then the plate was quenched in water at room temperature and was examined for peeling of the coating.
- c) **Adhesion – tape Test:** Adhesion tape was applied to the coated surface and then pulled off to test the adherency of the deposit.

2.10 TARNISH AND CORROSION RESISTANCE TEST :

One of the most important purpose for which electroplated coatings are used is to improve the appearance and particularly to maintain it for a longer period, without getting stained or tarnished.

The properly that determines the behaviour of a plated coating is its susceptibility to chemical action. especially in the atmosphere, which always contains oxygen, moisture and carbon dioxide; in industrial locations, sulphur dioxide, and in marine locations. sodium chloride.

So in order to test the resistance offered by the deposits obtained under the optimum conditions for corrosion and tarnish, plates were exposed to different atmospheres for desired period.

Since the electrodeposition work carried out by us is mainly for decorative purpose, the optimum conditions are selected in such a manner as to obtain bright, smooth and adherent deposits.

2.10 MEASUREMENT OF THROWING POWER :

The ability of a bath to produce deposits of more or less uniform thickness on cathode having macroscopic irregularities is known as throwing power. In recent years, the term throwing power has been used principally as a measure of the distribution of the deposits i.e., of their relative thickness on different parts of an article. More recently, the distinctions between plating into minute recesses on one hand, and producing uniform deposits on large scale profiles on the other, have been elucidated. The term macro throwing power refers to the latter. Sometimes a different but related property named as covering power of an article is also used. In practice the two terms are closely related, since solutions with poor throwing power than a covering power.

Being the most simple and probably the most useful measurement of the throwing power studied by us was done, qualitatively, by the bent cathode method. In this method, a 25 x 100 mm. copper plate was bent a right angles 25 mm. from the lower end and used as a cathode. This cathode was then placed at 2 cm. distance from a flat anode and plated in a 1000 ml. beaker at an appropriate current density (fig. 2.3). The cathode provided an area of low current density, a shelf area and areas of high and medium current density. Poor or good throwing power was then estimated by observing the extent (distance) to which a given metal or alloy was deposited into the base of the right angle. The test was carried out for optimum conditions only.

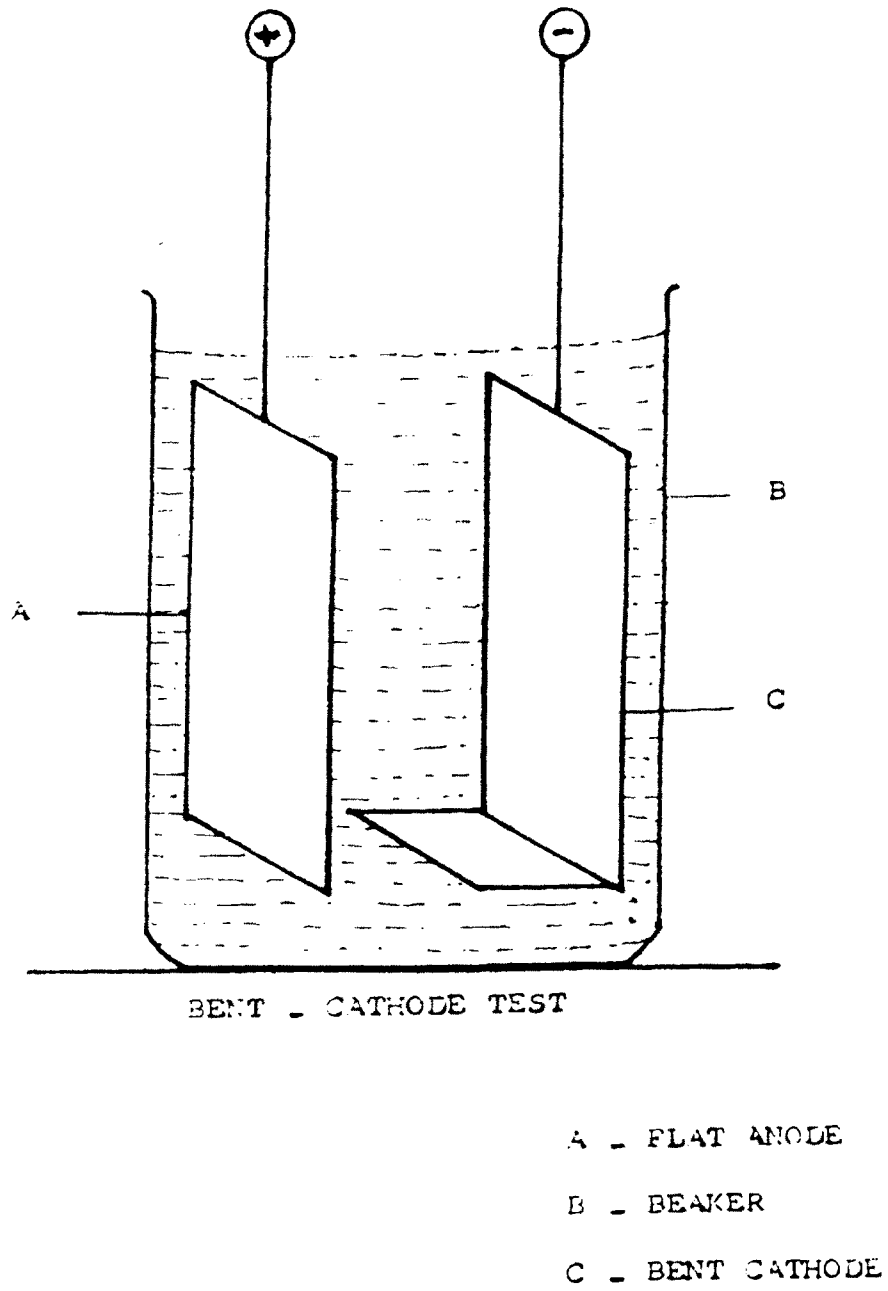


FIG. NO. 2.3

MEASUREMENT OF THROWING POWER