

## TECHNICAL NOTE

**A simple and versatile rotating disk electrode design for use in studies of electroless plating**

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**1. Introduction**

An important property of electroless plating baths is their plating rate. This rate is normally determined by suspending a planar substrate in the bath and determining its weight gain after an appropriate period of time [1, 2].

In recent years there has been a growing interest in applying electrochemical methods to the study of electroless plating bath characteristics. For example, measurements have been made of the plating potential [3, 4], Tafel regimes [3], polarization resistance [5–7] and polarization curves over a wider potential range [3, 8], in efforts to find useful correlations with the gravimetric plating rates.

The advantages of the rotating disk electrode (RDE) in electrochemistry are well known, yet its use in electroless plating studies has been very limited. One obvious reason for this is the problem of making numerous quick and accurate gravimetric plating rate measurements on such electrodes, which tend to be either too small in area or too massive for accurate measurements of weight gain to be made in a reasonably short time. We have now developed a simple and effective design, described below, which is suitable for both gravimetric and simultaneous electrochemical measurements.

**2. Experimental details**

The electrode assembly is shown in Fig. 1, and closely approximates to the shape 'd' recommended by Riddiford [9]. An electrode of this shape, and of similar size, has been shown to give limiting currents which are in excellent agreement with theory over a wide range of conditions [10]. The actual disks are machined to a diameter of 25 mm (area = 4.91 cm<sup>2</sup>) from a rod of mild steel, and weigh about 4 g. The edge and upper surface of the disk, excluding the threaded section, are coated with several layers of insulating lacquer ('Lacomit', W. Canning Ltd). A soft rubber O-ring prevents access of the electrolyte solution to the interior of the assembly. Disks of other metals can be obtained by soft soldering thin sheets to the steel base; in this way we have prepared disks of platinum, palladium, silver and copper. The assembly can be rapidly dismantled by loosening the stainless steel screw, allowing disks to be changed in a minute or two with no handling of the disks themselves.

In order to test the utility of this RDE, we have carried out studies on the low temperature alkaline nickel bath described by Feldstein [2]. Solutions were prepared using 'chemically pure' or better grade reagents and deionized water. The plating bath contained NiSO<sub>4</sub> · 6H<sub>2</sub>O (25 g l<sup>-1</sup>), Na<sub>4</sub>P<sub>2</sub>O<sub>7</sub> · 10H<sub>2</sub>O (50 g l<sup>-1</sup>), 25% aqueous NH<sub>3</sub> (25.2 ml l<sup>-1</sup>) and NaH<sub>2</sub>PO<sub>2</sub> · H<sub>2</sub>O (25 g l<sup>-1</sup>), and operated at pH ~ 10.95. A one-compartment, water-jacketed Pyrex cell of volume 200 ml was used for all measurements.

The RDE was driven through a shaft powered by a d.c. electric motor with a speed controller unit [11]. A saturated calomel electrode (SCE) was used as a reference electrode, and a sheet of nickel of area ~ 15 cm<sup>2</sup> as the counter electrode when required. The polarization resistance measurements were obtained in linear potential sweeps at 0.2 mV s<sup>-1</sup>, using a home made potentiostat.

Plating on the iron disks was initiated by brief polarization to -950 mV vs SCE. All measurements were carried out without purging oxygen from the solutions. Temperature control was ± 0.1°C, using a circulator-regulator.

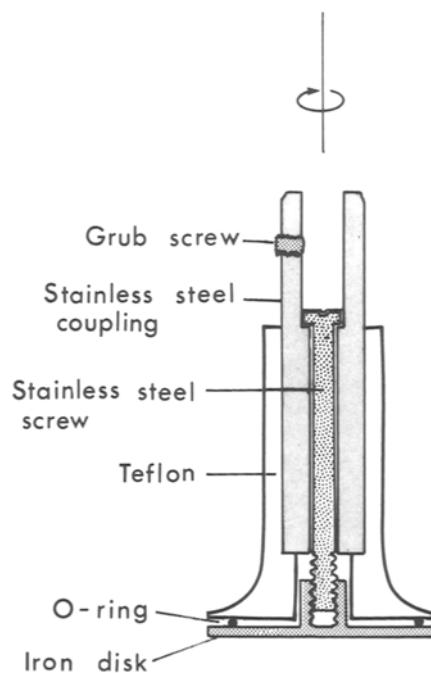


Fig. 1. Vertical cross section through rotating disk electrode assembly.

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Table 1. Variation of gravimetric plating rate (R) with rotation speed ( $\omega$ ) (temperature = 35.0°C)

$\omega$ (r.p.m.)	R ( $\text{mg cm}^{-2} \text{h}^{-1}$ )
0	2.13
500	2.13
1000	2.11
1500	1.88
2000	1.82

### 3. Results and discussion

Even with the relatively slow plating, low temperature alkaline nickel bath which we have used for investigating the utility of this RDE assembly, 30-min plating periods, giving about 5 mg of plate, were sufficient to obtain reproducibility of within a few per cent. Thus five successive plating runs at 35.0°C and 500 r.p.m., varying between 30 and 62 min in length, yielded an average rate of  $2.09 \text{ mg cm}^{-2} \text{h}^{-1}$  with a standard deviation of only  $0.08 \text{ mg cm}^{-2} \text{h}^{-1}$ . This level of reproducibility compares very favourably with gravimetric results obtained on much larger stationary substrates [2].

We have also measured the gravimetric plating rate as a function of the electrode rotation speed. These results are given in Table 1. The effect of rotation rate  $\omega$  on the plating rate is slight, and is qualitatively similar to that reported for the same type of bath by Feldstein and Amodio who used a stationary electrode and an independent stirrer [12].

We have also successfully used this electrode assembly to measure the dependence of the plating potential and the polarization resistance on a variety of experimental conditions, demonstrating its suit-

ability for electrochemical measurements in addition to gravimetric work.

### 4. Conclusions

The RDE assembly described is well suited to both gravimetric and electrochemical measurements of electroless plating. In this respect it appears to be an improvement on most previously reported substrate designs for electroless plating rate measurements. It is presently being used in further studies of the electrochemical fundamentals of electroless plating baths. Similar electrodes could also prove to be useful in studying electroplating or electrochemical corrosion.

### Acknowledgement

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