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**A RAPID ELECTROCHEMICAL TECHNIQUE FOR DETERMINING THE CURRENT EFFICIENCY FOR CADMIUM PLATING**

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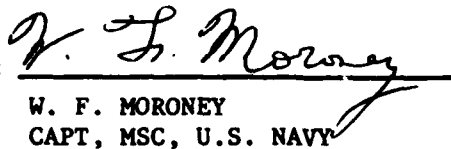
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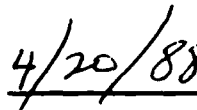
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<p>A rapid electrochemical technique has been developed to determine the current efficiency for cadmium plating from dull and bright cadmium-cyanide plating baths. Cyclic voltammetry experiments are described which were performed to develop the proposed method. The technique was validated by comparing the results obtained with weight loss and gain measurements to those measured electrochemically. Current efficiency for cadmium plating performed over a range of applied current densities and total hydrogen analysis tests were performed to show the usefulness of the technique.</p> <p>A rapid electrochemical technique has been described for determining the current efficiency for Cd plating using standard Cd-cyanide baths. Current efficiency measurements have been made over a range of applied current densities and have been statistically compared with weight gain and loss measurements. Total hydrogen analysis tests have been used in conjunction with this technique to correlate current efficiency for Cd plating with</p>			
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hydrogen uptake. It was shown that a 2% decrease in current efficiency for Cd plating increased hydrogen uptake by approximately 50%. This method can be used as a sensor to monitor the effects of impurities, bath composition or part geometry on the current efficiency for Cd plating. In addition, it can be used as a research tool to quickly and accurately measure the effects of various plating parameters or bath additions on the current efficiency for Cd plating.

*Keywords →*

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A RAPID ELECTROCHEMICAL TECHNIQUE FOR DETERMINING  
THE CURRENT EFFICIENCY FOR CADMIUM PLATING

INTRODUCTION

Cadmium (Cd) is commonly used as a plate material to protect steels against corrosion where it serves as a sacrificial coating. Usually components are electroplated with Cd using standard Cd-cyanide plating baths. An unavoidable side reaction, occurring simultaneously with the plating of Cd, is hydrogen codeposition. Dittle failures of Cd plated structural components, such as high strength steel aircraft landing gear under stress (1), are due to this hydrogen absorbed during the Cd plating process. Thus, all Cd plated high strength steel components are required to be baked in order to lower the hydrogen concentration in the component and reduce the possibility of a brittle failure. One method to minimize the amount of hydrogen absorbed by the component during the electroplating process is to monitor the plating bath to observe any decrease in the current efficiency for the Cd plating. Current efficiency, expressed as a percent, is that fraction of the total current flow which is used in the deposition of the Cd. The partial current used for hydrogen codeposition with Cd is reduced to zero when the current efficiency for plating reaches 100%. If the current efficiency is observed to be lower than normal during Cd plating, the plating bath can be further analyzed and/or corrected to achieve the maximum current efficiency. This report describes a rapid electrochemical method to determine the current efficiency for Cd plating from both bright and dull Cd plating baths.

Due to the effects of impurities and other factors, precise measurements and control of the plating bath chemistry is critical in obtaining consistent results in production. The constituents of a bath play an important role in keeping the current efficiency for plating at a maximum. There are many sensors which are used to monitor plating baths. Sensors are used to monitor temperature, pH and metal ion concentration (2). Voltammetric stripping has recently been included in continuous plating baths to monitor metal ion concentrations (2). Polarographic techniques are useful for the determination of materials in low and high concentrations (3). Standard analytical procedures also are available for the analysis of Cd plating baths (4). Although current analytical techniques provide useful information, they are time consuming and not immediately available. A direct measurement of the current efficiency for Cd plating is not available.

A simple electrochemical method, which is suitable for the rapid determination of the current efficiency for Cd plating and the quantity of hydrogen produced during Cd plating from standard Cd-cyanide plating baths, is described in this paper. The amount of Cd deposited during plating is measured via an anodic stripping process. The number of coulombs used for the stripping process is directly related to the mass of Cd stripped as described by Faraday's law. Current efficiency measurements at various applied current densities and, also, total hydrogen analysis of Cd plated specimens were performed to show the usefulness of the technique.

## EXPERIMENTAL PROCEDURES

## Electrochemical Apparatus

All bench scale Cd plating and stripping experiments were performed using a typical glass beaker cell shown in Figure 1. The working electrode was placed in the center of the cylindrical counter electrode for proper current distribution. A saturated calomel reference electrode (SCE) was used to monitor the potential at the working electrode. A 1018 steel cylindrical working electrode was prepared by using standard metallographic polishing techniques for final finish to 1000 grit SiC paper. The counter electrode was a one inch wide Cd strip. The solution was agitated with a magnetic stirrer. The test solutions were the standard bright and dull Cd plating solutions. Table I shows the composition of each test solution. A Princeton Applied Research (PAR) Model 273 galvanostat/potentiostat was used to supply the applied plating currents and stripping potential, and for the cyclic voltammetry experiments. The total cathodic and anodic current flow was measured using a coulometer (part of PAR Model 273). The schematic for cyclic voltammetry, and Cd plating and stripping experiments is shown in Figure 2.

## Cyclic Voltammetry

Cyclic voltammetry tests were performed to determine if it would be possible to selectively oxidize Cd after plating onto steel in the Cd-cyanide plating baths. It was used to monitor current-voltage relationships upon the application of a constantly changing potential to the working electrode in both bright and dull Cd-cyanide plating baths. In general, a triangular voltage/time waveform is applied (at a fixed sweep rate (millivolts/second), to the working electrode, and the resulting current response is plotted as a function of the applied potential. This is called a cyclic voltammogram. The basic feature of the voltammogram is the appearance of a current peak at a potential characteristic of the electrode reaction taking place. The currents may result from a number of different types of reactions occurring at the working electrode. Using this technique, the oxidation and reduction potentials of the Cd and the steel substrate were determined in both bright and dull Cd-cyanide plating baths. A potential range was found to study only the cadmium and hydrogen oxidation and reduction reactions.

## Cd Plating and Stripping Technique

After specimen preparation and cell assembly, the 1018 steel working electrode was Cd plated. The applied plating current densities were 20 and 48 mA/cm<sup>2</sup>. Two current densities were used to study the effectiveness of this technique at different current efficiency levels. The current efficiency for Cd plating does change value with current density (5) due to the codeposition of hydrogen and the changes in kinetics of Cd deposition. The total coulombs passed during the plating process ranged from 10 to 120 (a coulomb is equal to the applied current times the time, i.e. amps\*seconds).

Different amounts of Cd were deposited on the steel specimen to investigate the sensitivity of the technique. After Cd plating, the Cd plated cylindrical steel specimen was then weighed to the nearest 0.1 mg. After weighing, the Cd was electrochemically stripped off of the steel working electrode potentiostatically. The stripping (Cd oxidation) coulombs were measured. The amount (mass) of Cd stripped was then calculated by multiplying the stripping (Cd oxidation) coulombs by 0.5824 mg Cd/coulomb (electrochemical equivalent for Cd). The mass of cadmium deposited was calculated and compared to the mass of Cd weighed. Theoretically, these two values should be equal. The data when plotted should give a linear relationship between the Cd plating coulombs determined from the weight of Cd (weight of Cd, mg/0.5824 mg/coulombs) and the stripping coulombs measured during the anodic

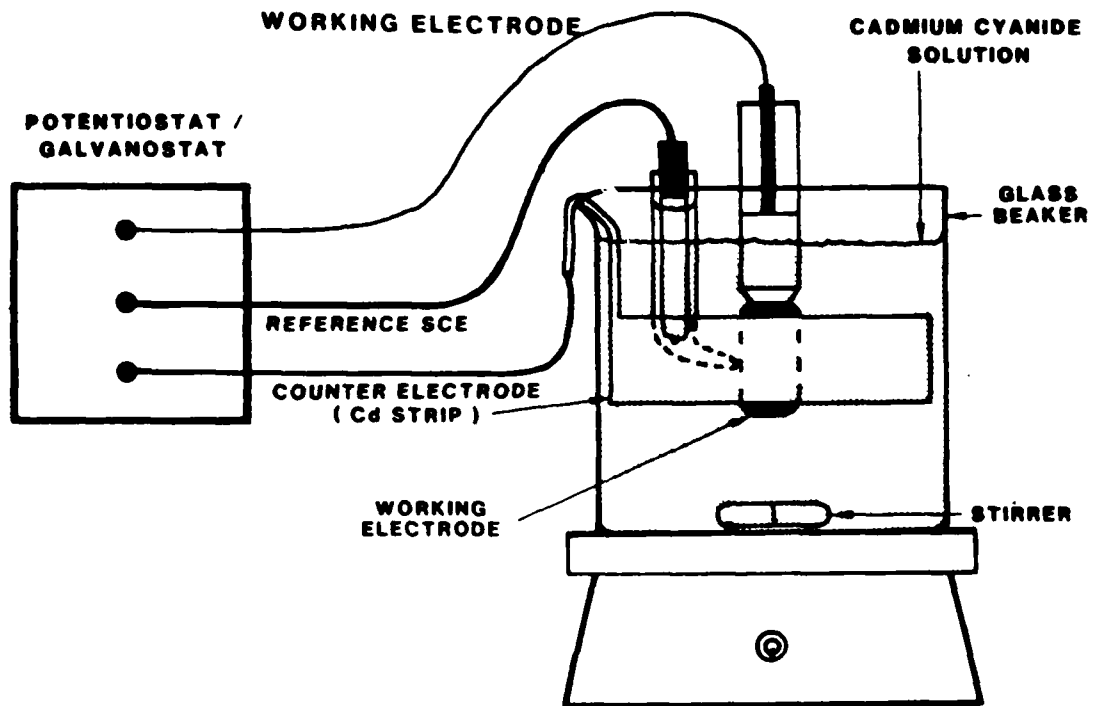


Figure 1 Electromechanical cell used for Cadmium plating and stripping experiments

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**TABLE 1**

**Composition of Standard Bright and Dull  
Cadmium-Cyanide Plating Baths**

	<i>Bright Cd-Cyanide</i>	<i>Dull Cd-Cyanide</i>
CdO	3.0 oz/gal.	3.0 oz/gal.
Cd	2.6 oz/gal.	2.6 oz/gal.
NaCN	18.4 oz/gal.	18.4 oz/gal.
Udylite 153*	1 volume %	-----

\*Udylite 153 is a propriety brightener manufactured by Udylite Inc.

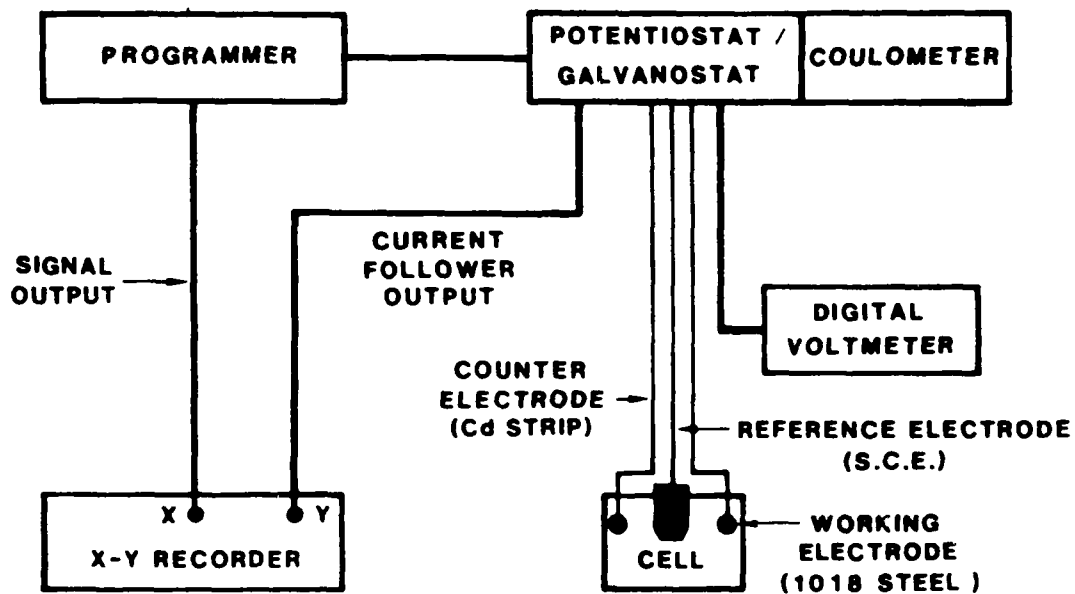


Figure 2 Schematic for cyclic voltammetry, constant current Cadmium plating and potential stripping experiments

stripping. The linear relationship should follow the expression of  $Y = MX + B$  with the intercept  $B$  equal to zero. The sample correlation coefficient  $r(1)$  was calculated to determine whether there was a linear relationship. A value of  $r(1)$  equal to 1 indicates a perfect linear relationship.

The Cd plating and stripping technique was used for the rapid determination of the current efficiency for Cd plating at a number of current densities. The applied current density values ranged from 3 to 50 mA/cm<sup>2</sup>. Three runs were performed for each current density.

#### Hydrogen Analysis of Cd Plated Parts

In addition to determining the current efficiency the usefulness of the technique was used to study the effect of plating bath on the amount of hydrogen absorbed during the Cd plating process. The total hydrogen in the Cd plated steel was measured using a LECO RH2 Hydrogen Determinator. Three runs for each specimen were made for both bright and dull Cd plated specimens. The applied plating current density was 20 mA/cm<sup>2</sup> for a total of 100 coulombs. The total bright and dull Cd plated was approximately 72 and 71 milligrams, respectively. The amount of hydrogen in the Cd plated steel was compared to the current efficiency or the amount of hydrogen produced in each solution. The same cell was used for each test, as shown in Figure 1.

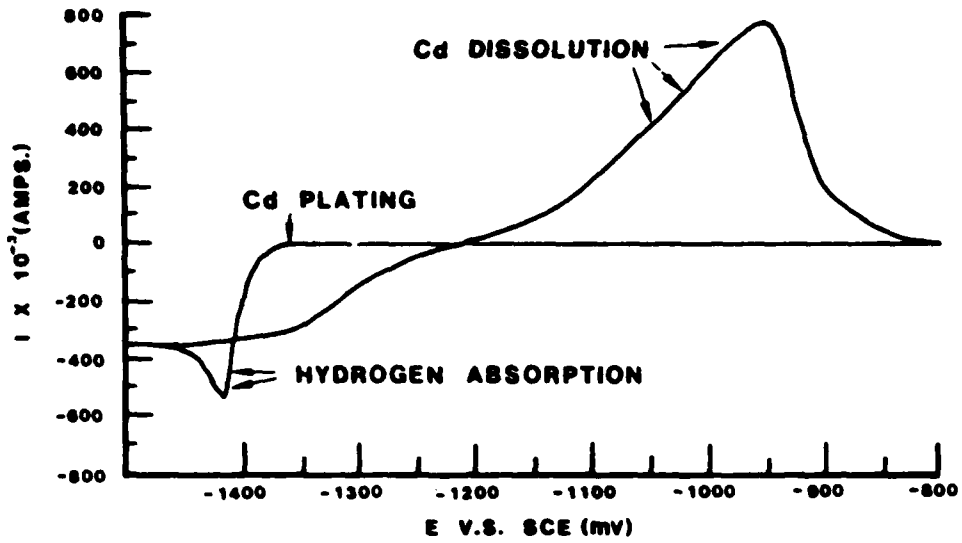
## RESULTS AND DISCUSSION

#### Cyclic Voltammetry

Figure 3 shows the cyclic voltammogram for a bright plating bath. Shown are regions of Cd plating, Cd dissolution and hydrogen absorption. These were the only significant reactions occurring during the scanning of the potential between -1.5 and -0.8 volts versus SCE at a sweep rate of 150 mV/second. There is a larger cathodic peak at -1.43 volts, as a result of the brightener in the solution. The labeled Cd dissolution peak was due only to the dissolution of the Cd that was plated during the time the potential was in the cathodic region. The current-voltage curve for the dull Cd plating bath is similar to the bright Cd plating bath except the peak at -1.43 volts was not observed. From this analysis, it was determined that Cd could be electrochemically stripped (oxidized) from the 1018 steel substrate, without the oxidation of the steel. If the potential were directed to more positive potentials than -800 millivolts, the steel substrate would oxidize. Thus, the potential used for the Cd stripping potential was more negative than the potential for iron oxidation. As a result, the only coulombs recorded during a Cd stripping process would be only from the oxidation of Cd that was previously deposited. The anodic stripping potential for Cd used was -1.0 volts versus SCE. (Note: The amount of hydrogen oxidized and oxygen reduced at the working electrode during the stripping (oxidation) of Cd is minimal as compared to the Cd oxidation at these current levels).

From these results, a rapid technique for measuring the plating efficiency for Cd plating was developed. The total amount (mass) of cadmium which was plated at any applied current density or plating bath (bright or dull) could be determined by applying an anodic stripping potential (more negative than -0.8 volts vs. SCE) and measuring the resultant stripping coulombs. The current efficiency could then be calculated by dividing the Cd stripping coulombs by the total coulombs used for the plating process. It could be used in-situ or a specimen could be plated and stripped in a bright or dull Cd plating solution with an experimental setup as shown in Figure 1.

BRIGHT CADMIUM SOLUTION



EXPERIMENT TYPE: CYCLIC VOLTAMMOGRAM  
INITIAL E (MV) -900  
VERTEX E (MV) -1500  
FINAL E (MV) -900  
SCAN RATE (mV/S) 150

Figure 3 Cyclic voltammogram of a 1018 steel in a bright Cd-cyanide plating solution

### Cd Plating and Stripping Technique

Figure 4 shows a graph of the coulombs consumed for Cd plating, calculated from the weighing measurements versus those measured by the anodic stripping potential technique. The brightening agent had no affect the technique. Different amounts (mass or thickness) of Cd were plated to observe if there was a "chunk effect" for Cd dissolution. The "chunk effect" is due to the undercutting and falling off of the Cd metal during the dissolution process. If this happened, the results would show less Cd being oxidized than actually plated. The sample correlation factor,  $r(1)$ , for dull and bright plating result were +0.9995 and +0.9991, respectively. This shows that weight measurements can be omitted for determining current efficiency for Cd plating. Current efficiency for Cd plating studies could be performed without removing specimens from the plating bath. Since the plating bath conditions and part geometry are known to affect the current efficiency for Cd plating, any changes in the amount of hydrogen produced during plating could be detected. Once the current efficiency for Cd plating is determined, the amount of hydrogen produced can be calculated by subtracting the current used for Cd plating from the total applied current.

### Current Efficiency for Cd Plating

The current efficiency of a plating reaction is 100% if all the coulombs were used for Cd plating. If the amount is less than the expected amount calculated from Faraday's law, the efficiency of the process is lessened due to the hydrogen evolution reaction. The effect of applied current density on the current efficiency for Cd plating for both the bright and dull Cd plating is shown in Figure 5. For the constant current plating, the lowest current efficiency over the range of current densities used was observed for the bright Cd plating bath. This represents a greater chance for hydrogen uptake. Typically, the standard deviation for all the data shown in Figure 5 is approximately  $\pm 0.5\%$ . This data was determined by the described technique in order to correlate the current efficiency values to the amount of hydrogen absorbed.

### Total Hydrogen Analysis

Figure 6 shows the amount of hydrogen in the Cd plated specimens expressed in ppm H per gram of Cd plated. The result showed a standard deviation of  $\pm 15\%$ . By comparing the current efficiency for Cd plating at  $20 \text{ mA/cm}^2$  in Figure 5 and the amount of hydrogen absorbed, it shows that the approximate 2% increase in current efficiency decreased the amount of hydrogen absorbed by 50%. Of course, this result was expected because the current efficiency for bright Cd plating was lower compared to dull Cd plating at the applied current density of  $20 \text{ mA/cm}^2$ . This lower current efficiency means that there was a larger amount of current used for hydrogen codeposition during bright Cd as compared to dull Cd plating. Thus, more hydrogen was available to enter the substrate during bright Cd plating.

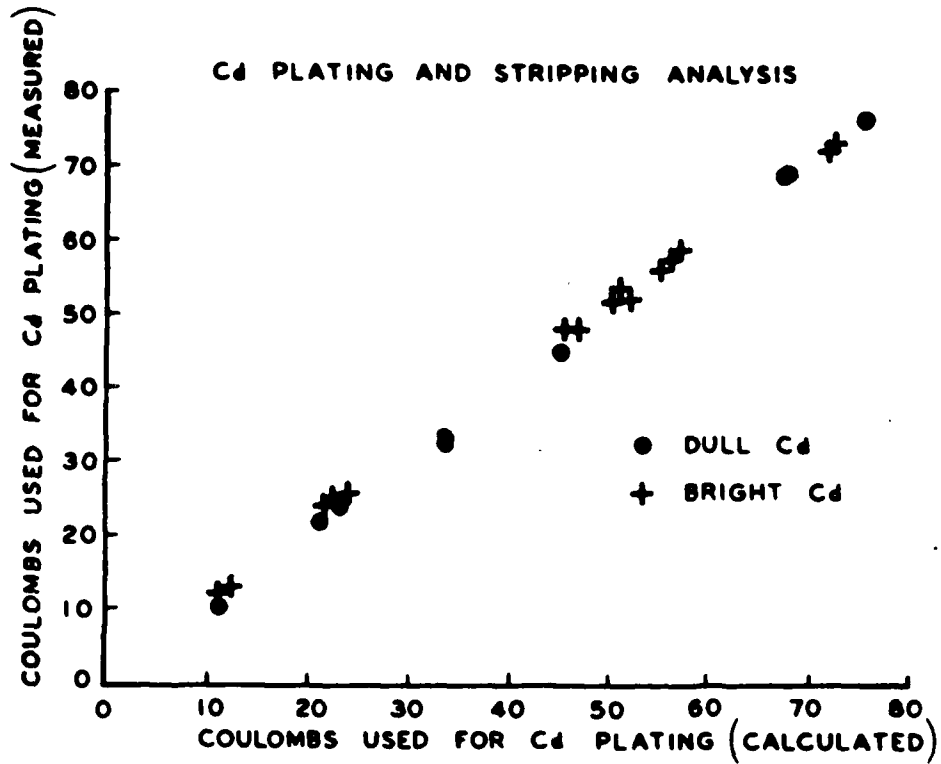


Figure 4 Relationship between the amount of coulombs used for Cd plating (calculated) and the coulombs used for cadmium stripping off of steel substrate (measured)

CONCLUSIONS

A rapid electrochemical technique has been described for determining the current efficiency for Cd plating using standard Cd-cyanide baths. Current efficiency measurements have been made over a range of applied current densities and have been statistically compared with weight gain and loss measurements. Total hydrogen analysis tests have been used in conjunction with this technique to correlate current efficiency for Cd plating with hydrogen uptake. It was shown that a 2% decrease in current efficiency for Cd plating increased hydrogen uptake by approximately 50%. This method can be used as a sensor to monitor the effects of impurities, bath composition or part geometry on the current efficiency for Cd plating. In addition, it can be used as a research tool to quickly and accurately measure the effects of various plating parameters or bath additions on the current efficiency for Cd plating.

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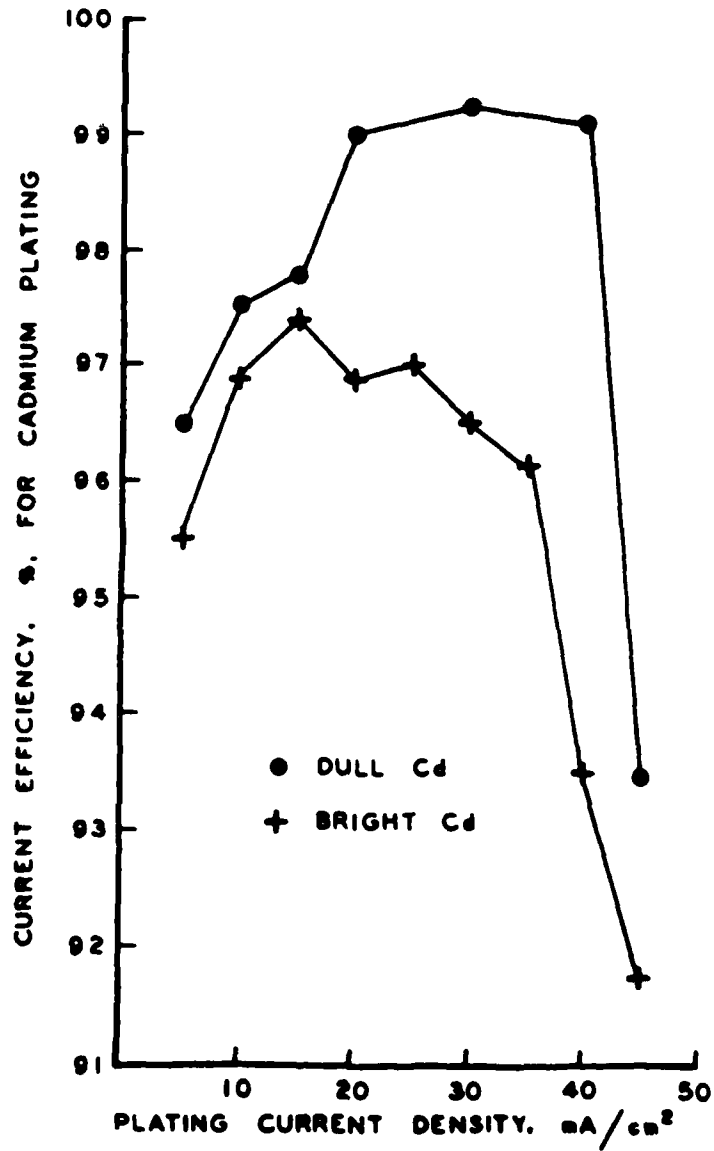


Figure 5 The effect of constant plating current density on the current efficiency for bright and dull Cadmium plating

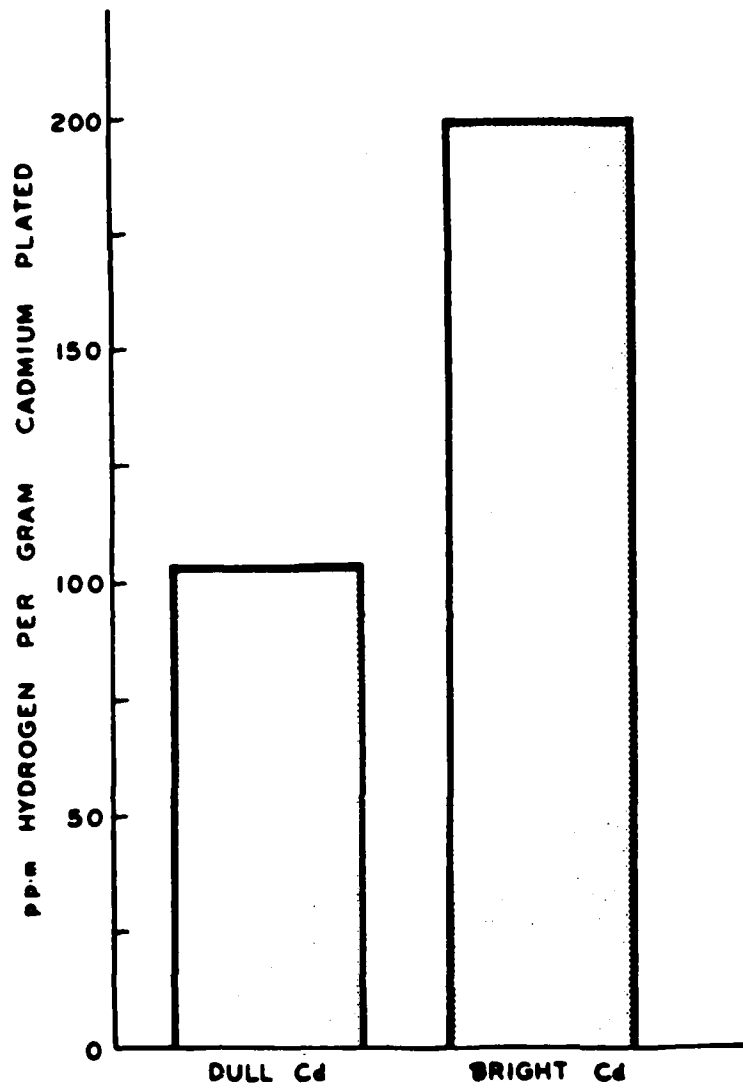


Figure 6 Total hydrogen uptake during bright and dull Cadmium plating

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