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ADSORPTION OF CADMIUM IONS ON A PLATINUM ELECTRODE

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16. Abstract

It has been reported that the life of an experimental cadmium-oxygen battery cell is limited by degradation of the platinum-oxygen electrode. In this work, it was confirmed that dissolved cadmium causes an increase in the overvoltage for oxygen electroreduction at a smooth platinum electrode in 1 N KOH. Using linear sweep voltammetry, evidence was found that cadmium ions adsorb in a valence state lower than +2, perhaps as neutral cadmium adatoms. The adsorption becomes appreciable just below the reversible oxygen potential (1.23 V with respect to the reversible hydrogen electrode in the same electrolyte), varies from 0.2 to 0.25 of a monolayer (assuming zero-valent cadmium) at potentials from 0.7 to 0.1 V, and then rises steeply as the potential is lowered further. The observed cadmium adsorption isotherm is consistent with observed catastrophic failure of a practical oxygen electrode operating at constant current in the presence of cadmium ions.

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SUMMARY

Electrochemical determinations of hydrogen and oxygen adsorption from 1 normal KOH, on a smooth platinum microelectrode, were made under carefully controlled conditions. The results indicate that a highly reproducible ("clean") surface may be generated through anodic-cathodic pretreatment. When the electrolyte is saturated with cadmium ions, coverage of the surface with hydrogen is decreased and an anodic charge Q_{Cd} must be supplied (in the range of "oxygen adsorption" potentials) to restore the surface to the "clean" state. This is taken as evidence for adsorption of cadmium ions in a valence state of less than +2, perhaps as neutral cadmium adatoms.

Based on measurements of Q_{Cd}, it has been concluded that steady-state fractional coverages with cadmium vary slightly (from 0.25 to 0.2, assuming adatoms) over the potential range 0.1 to 0.7 volt (with respect to a reversible hydrogen electrode in 1 normal KOH). The coverage falls off more steeply to 0 over the range 0.7 to 1.2 volts, likely due to higher heats of adsorption for adsorbed oxygen or oxide precursors than for cadmium. Cadmium coverages rise steeply below 0.1 volt as the reversible potential for cadmium metal deposition is approached.

Adsorbed cadmium increases the overvoltage for electroreduction of oxygen at a smooth platinum microelectrode in 1 normal KOH. From the cadmium adsorption isotherm, it is possible to anticipate that the potential of a practical oxygen electrode operating at constant current in the presence of cadmium ions might be driven from oxygen to hydrogen potentials, as actually reported.

INTRODUCTION

Wagner (ref. 1) noted the degradation of the platinum-catalyzed oxygen electrode in an experimental cadmium-air battery cell and suggested that the effect might be due to the adsorption of cadmium ions. In this study we verified the degradation effect and made some evaluation of the nature and extent of the causative adsorption.

The work was begun at the NASA Electronics Research Center and completed at the Lewis Research Center.

EXPERIMENT APPARATUS AND PROCEDURE

Voltametric measurements were made using the electronic equipment described in reference 2. Measurements were made in either cell I or cell II. Cell I was fabricated of pyrex glass and was preferred for convenience of use and provision for controlled mechanical stirring. It comprised a main compartment holding 200 cubic centimeters of solution in which was immersed electrode A or electrode B, and a glass stirrer. The main compartment was separated from the counter electrode compartment by means of a porous glass disk and from the reference electrode compartment by means of a Luggin capillary and a closed glass stopcock. The counter and reference electrodes were platinized platinum foils, and a stream of pure hydrogen gas was allowed to impinge upon the reference electrode surface. Test electrodes A and B were prepared by annealing lengths of 0.030-inch- (0.076-cm-) diameter, chemically pure platinum wire and sealing into glass tubing. The geometric areas of electrodes A and B were 0.43 and 0.16 square centimeter, respectively.

Cell II was designed so that the electrolyte would not be contacted by any material other than platinum (Pt) and with less than 0.1 square centimeter of polytetrafluoroethylene (PTFE) insulator for the test electrode. A small, smooth, Pt crucible served as both the counter electrode and the container for the electrolyte. The test electrode was a length of 0.030-inch- (0.076-cm-) diameter, chemically pure Pt wire annealed in a hydrogen flame and encased in heat-shrinkable PTFE tubing, exposing 0.21 square centimeter of geometric area. The reference electrode was a length of 0.1-inch- (0.25-cm-) inside-diameter Pt tubing platinized at the end immersed in the electrolyte and through which was passed a slow stream of a 1-percent-hydrogen, 99-percent-argon gas mixture. Gas flow was discontinued before making a measurement if a quiescent solution was desired, or continued if stirring was desired. The reference electrode potential (as measured against a conventional hydrogen electrode) was found to be highly reproducible and stable. Calculations revealed that the charge associated with the oxidation of hydrogen from the approximately 10^{-5} molar solution of hydrogen gas (resulting from use of the dilute hydrogen reference electrode) was negligible and could contribute no significant error to the measured adsorption charges. The platinum crucible was enclosed in a gas-tight quartz vessel provided with a tapered ground quartz cap which served as the electrode support. A second interchangeable cap held a reference electrode and a large platinized "gettering" electrode (ref. 2). For cleanup purposes, the getter electrode was alternatively driven to oxygen evolution potentials, hydrogen evolution potentials, and back to oxygen evolution potentials in a portion of electrolyte, and then quickly transferred to the test vessel with the protection of the anodic film (ref. 2). The potential was then adjusted to 0.06 volt and stirring was provided by the reference gas jet.

The 1 normal KOH electrolyte was prepared using analytic-reagent grade KOH pellets and triply distilled water degassed by sparging with a stream of pure helium for over 1 hour. The solution was prepared in a Pt evaporating dish under a helium atmosphere in a glovebox. Cell II was opened (for electrolyte transfers and ''gettering'') only in the glovebox.

When required, cadmium ions were added to the KOH solutions by introducing sufficient 0.01-molar cadmium perchlorate to make the solution 10^{-4} molar and then allowing at least several hours for excess cadmium hydroxide to precipitate out.

Both cells I and II were thermostated at 30^{0} C in an air bath.

All potentials are referenced to that of a reversible hydrogen electrode in equilibrium with 1 atmosphere of hydrogen in 1 normal KOH.

RESULTS AND DISCUSSION

Polarization Curves for Oxygen Reduction

These experiments were performed in cell I using electrode A. The electrode potential was programmed as in reference 2 to regenerate a clean and reproducible surface. In the experiment of figure 1, after the anodic pretreatment at 1.8 and 1.2 volts, the surface was reduced and exposed to possible adsorption of cadmium ions at a potential U for a time τ. The polarization curves were measured by applying a positive-going potential sweep (speed, 0.1 V/sec) and measuring the resulting current. Pure oxygen was kept bubbling through the electrolyte with additional vigorous stirring by means of the glass stirrer. A single polarization curve was obtained in the absence of dissolved cadmium for values of U between 0.1 and 0.5 volt and for values of τ between 0.1 and 1000 seconds (dashed trace of fig. 1). That curve exhibits exponential dependence of current upon potential in the fractional milliampere current density range. A diffusional influence (curvature toward the voltage axis) is apparent at higher current densities. An oscillatory limiting current dependent on the rotational speed of the stirrer (the average limiting current for the conditions of figure 1 is given by the horizontal dashed lines) is assumed to be diffusion-limited. In the presence of dissolved cadmium, polarization increased with decreasing U and increasing τ. Intermediate curves exhibit an inflection at current densities well below the diffusion limit. The inflection may mark a transition between reduction of oxygen to peroxide and to water, as observed on some catalysts other than Pt (e.g., Au, ref. 3). Retardation of oxygen reduction kinetics by metal ions, notably the alkaline earth ions in acid solution, has been observed by several investigators (refs. 4 to 6).

Reference Surface State of a Platinum Electrode

The surface state of a Pt electrode in KOH solution may be probed conveniently through application of a linear potential-time sweep. In the experiment of figure 2, the potential sweep was applied after anodic treatment of the electrode followed by reduction for 1 second at 0.06 volt. Stirring was discontinued during the latter part of the pretreatment to minimize convective diffusion of impurities to the electrode. A complete discussion of the pretreatment scheme appears in reference 2. The trace obtained during the sweep to 1.5 volts is rather similar to that obtained in acid electrolytes (ref. 7). Two closely spaced maxima appear during the anodic sweep to approximately 0.4 volt. Virtually the mirror image of this portion of the trace was obtained during the cathodic sweep to complete the similarity to the "hydrogen adsorption" region observed in acid electrolytes (ref. 7). Above approximately 0.8 volt, as in acid solutions, there is a very broad oxidation wave terminating in oxygen gas evolution above approximately 1.6 volts. The corresponding reduction process occurs at markedly lower potentials (peak at 0.7 V) and, by analogy to the acid electrolyte system, may be assigned to reduction of a film (adsorbed oxygen or oxide precursor) formed during the anodic sweep. Unlike the situation in acid electrolytes, there is no "double layer" region at potentials intermediate between hydrogen and oxygen adsorption, during the positive-going sweep. Somewhat symmetrical cathodic currents (above the hydrogen adsorption region) may be obtained if the sweep is interrupted at potentials below 0.8 volt. These currents may correspond to those occurring to a much smaller extent (between approximately 0.6 and 0.8 V) in acid solutions and ascribed by Böld and Breiter (ref. 8) to hydroxyl radical adsorption.

The effect of electrode pretreatment on the anodic sweep trace was explored in the experiments of figure 3. The potential sequence used was based on the principles formulated in reference 2 for acid electrolytes. Employing that sequence, it was found that any particular trace usually could be reproduced to within a fraction of 1 percent on the current axis. From figure 3 it is evident that the traces stabilize sometime between 10 and 100 seconds. Two mechanisms could account for the time variations:

(1) Adsorption of impurities from solution

(2) Slow reduction of the surface

To test for the first possibility the experiments were repeated with stirring, and no effect was observed. This would seem to rule out a transport-limited impurity adsorption. In a second test, an attempt was made to getter impurities on a large-area platinized Pt gauze electrode held at 0.06 volt overnight. No effect of gettering was observed, arguing against the possibility of activation-controlled adsorption of impurities.

In support of the second possibility for time-dependence of the electrode surface state, an exponentially decaying reduction current could be measured, where the current remained greater than 0.01 milliampere per square centimeter for several seconds. Additional evidence that the variations of figure 3 correspond to slow reduction of the surface was obtained through measurement of the anodic and cathodic charges in the hydrogen adsorption region. The traces were measured as in figure 2, with the potential sweep extending from 0.06 to 0.5 volt. The time τ at 0.06 volt was the variable and the charges Q were obtained by integrating the total area under the traces, using a planimeter. The anodic and cathodic charges (corresponding mainly to hydrogen dissolution and deposition, respectively) are plotted against τ in figure 4. It can be seen that after approximately 10 seconds, Q reaches a value that is (within a few percent average deviation) independent of the direction of the potential sweep and of stirring. At shorter times, the anodic charge decreases and the cathodic charge increases, as would be expected if there were a concomitant reduction (of the surface) process during τ. If the time effect were due instead to adsorption of an impurity, it would be expected that both the anodic and cathodic values of Q would decrease with increasing τ. It may be noted that the time effects of figure 3 are most striking in the hydrogen region where there is a tendency for the waves to shift to more anodic potentials with increasing τ. The effect is less marked at lower values of the sweep speed, indicating that the effect on hydrogen adsorption is partly a kinetic one.

Although impurity effects are not indicated by the experiments of figure 3, such effects were encountered in some of our other experiments. Stirring-dependent decreases of currents in the hydrogen region and increases of currents in the oxygen region similar to those encountered in unpurified acid solutions (ref. 2) were observed when experiments were performed with some exposure of the electrolyte to the atmosphere. If carbon dioxide (or carbonate) does not adsorb from alkaline solutions as reported in reference 9, it appears likely that other organic substances in the air may be troublesome.

The effect of glass as a contaminant was tested by adding crushed pyrex glass to the Pt crucible of cell II. A small but noticeable effect of glass was evident after only 10 seconds.

Hydrogen Adsorption

Assuming that the anodic and cathodic waves (fig. 2) obtained below 0.5 volt correspond to dissolution and deposition of hydrogen atoms, we may estimate the charge ${}_{S}Q_{H}$ corresponding to monolayer coverage of the surface with hydrogen atoms, as in acid electrolytes (ref. 2), from the cathodic sweep trace. For electrode B, ${}_{S}Q_{H}$ equals 0.28

millicoulomb per square centimeter, suggesting a roughness factor of 1.3 if we assume (ref. 2) an ${}_{S}Q_H$ of 0.21 millicoulomb per square centimeter corresponds to a roughness factor of 1.

Adsorption of Cadmium Ions

Our solutions of cadmium ions were obtained by allowing the hydrolysis of an excess of neutral cadmium perchlorate solution to come to equilibrium in 1 normal KOH at 30° C. Under these conditions (but 5° lower in temperature) the total concentration of bivalent cadmium should be 8×10^{-6} molar according to reference 10 or 5×10^{-6} molar according to reference 11.

The potential sequence of figure 5 was selected to determine the effect of cadmium ions on the anodic sweep trace. As shown in a preceding section, a highly reproducible surface is generated during reduction of the surface at 0.06 volt, although complete reduction of the surface is not accomplished for τ less than about 10 seconds. Adsorption of cadmium occurs on the freshly (or partially reduced surface before application of the potential sweep). A high (v = 100 V/sec) value of the sweep speed was chosen so that the adsorption of cadmium ions during the sweep was negligible. The dashed traces were obtained in 1 normal KOH in the absence of cadmium and the solid traces were obtained after addition of cadmium. It is apparent that cadmium ions cause a decrease in hydrogen adsorption and an increase in the charge flowing in the "oxygen adsorption" region. Similar observations were made by Breiter (ref. 12) and by Bowles (refs. 13 and 14) for acid solutions containing copper and thallium ions, respectively.

The decrease in hydrogen adsorption may be attributed to blockage of hydrogen adsorption sites by adsorbed cadmium. The increase of charge Q_{Cd} at more anodic potentials must correspond to the oxidation of zero valent (adsorbed cadmium atoms) or monovalent (adsorbed cadmous ions) cadmium to dissolved bivalent cadmium. The present experiments cannot distinguish between these two possibilities, but there is evidence in support of the corresponding adatom adsorption for copper (ref. 12) and thallium (refs. 13 and 14). For adsorption at 0.06 volt, it is apparent from figure 5 that separation of Q_H (charge corresponding to hydrogen adsorption) from Q_{Cd} becomes difficult at large values of τ. This introduces uncertainty only for adsorption potentials less than about 0.4 volt, where hydrogen coverages are appreciable. Values of Q_{Cd} were approximated at the lower adsorption potentials by integrating from the point at which the traces intersected for the cadmium-containing and cadmium-free solutions (shaded areas in fig. 5).

Values of Q_{Cd} obtained over a wide range of adsorption potentials and apparent steady state at $\tau = 1000$ seconds, appear in figure 6. For $U > 0.45$ volt, it was necessary to pre-reduce the surface briefly at 0.3 volt, as shown. If we make the tentative assumption that cadmium adsorbs as the adatom and that one cadmium adatom occupies one hydrogen adsorption site, a monolayer of cadmium adatoms would correspond to $Q_{Cd} = 2\ {}_SQ_H = 2 \times 0.28 = 56$ millicoulombs per square centimeter. By that criterion, fractional surface coverage, θ_{Cd}, ranges from 0.25 to 0.2 over the range $U = 0.1$ to 0.7 volt. At the low potential end, the coverage rises steeply as we approach the reversible potential (approx -0.05 V, according to ref. 15) for cadmium metal deposition. At the high potential end, θ_{Cd} drops gradually to 0 as we approach 1.2 volt.

Interpretation of the Q_{Cd}-against-U plot of figure 6 is difficult because of the complexity of the equilibria between the dissolved ions and because of the interactions of the Pt electrode with the electrolyte in the absence of cadmium ions. Bivalent cadmium might be present as $Cd(OH)_2$, $Cd(OH)_3^-$ and $Cd(OH)_4^{--}$ in alkaline solutions. It has been suggested in reference 11 that $Cd(OH)_3^-$ and $Cd(OH)_4^{--}$ predominate for OH^- concentrations greater than 0.5 N, while Visco and Sonner (ref. 12) argue that $Cd(OH)_4^{--}$ is not encountered even in 15 N KOH. Adsorption of either negative ion at the original valence would be expected to start near the zero point of charge (zpc) here estimated at 0.72 volt (based on zpc = -0.11 with respect to the hydrogen electrode at pH 11.2, ref. 16) and to increase with increasing potential. The reverse dependence of Q_{Cd} upon U was actually found. Adsorption as the neutral atom might be expected to yield a semilogarithmic or linear (if heat of adsorption decreases with coverage) dependence of Q_{Cd} upon U. This may be reflected at the low and high potential ends of figure 6. The arrest at intermediate potentials may be due to a tendency to form a surface alloy of fixed composition. The decline of Q_{Cd} at high potentials may alternatively reflect a competition with anodic film formation.

Cadmium Ion Adsorption and Electroreduction of Oxygen

From figure 6 we see that $Q_{Cd} = 0$ for $U > 1.2$ volt and, hence, Cd ion adsorption ought not to affect a platinum-catalyzed oxygen electrode operating near the reversible potential (1.23 V). However, practical oxygen electrodes tend to operate at 0.9 volt or less, as in Wagner's experiment (ref. 1). Beginning at 0.9 volt, in the presence of dissolved cadmium, θ_{Cd} would tend to rise toward the intermediate value reflected by Q_{Cd} in figure 6. That coverage would cause a drop in voltage (increase in oxygen overvoltage) and an increased tendency to adsorb cadmium ions until the highest values of Q_{Cd} and the potential of the counterelectrode is reached. This was actually observed in a laboratory cadmium-oxygen battery cell (ref. 1).

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, October 29, 1970,
120-34.

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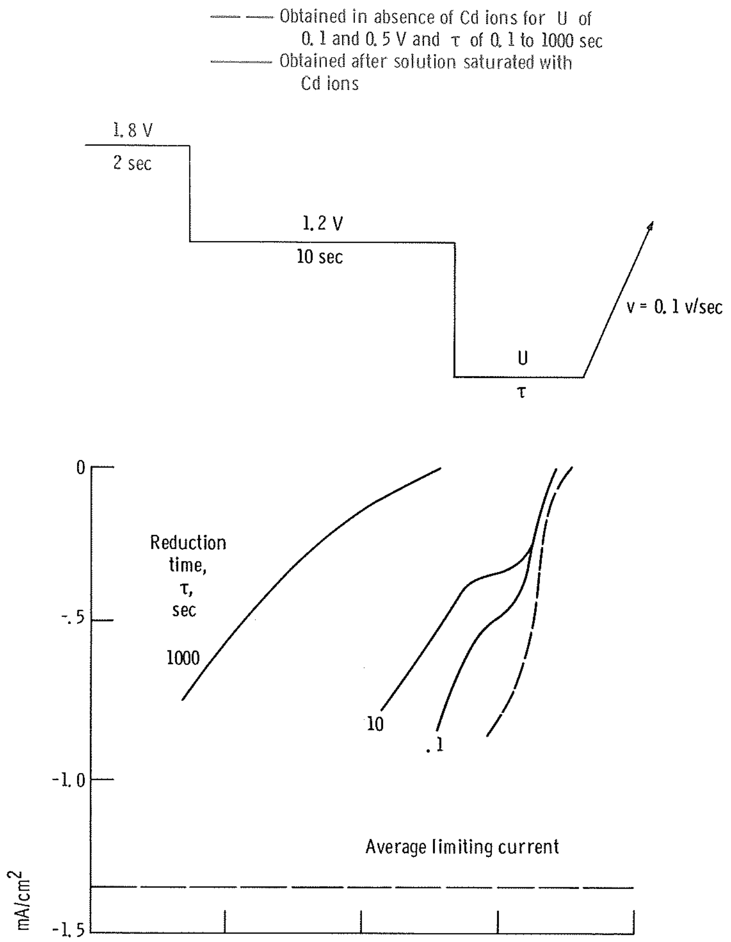
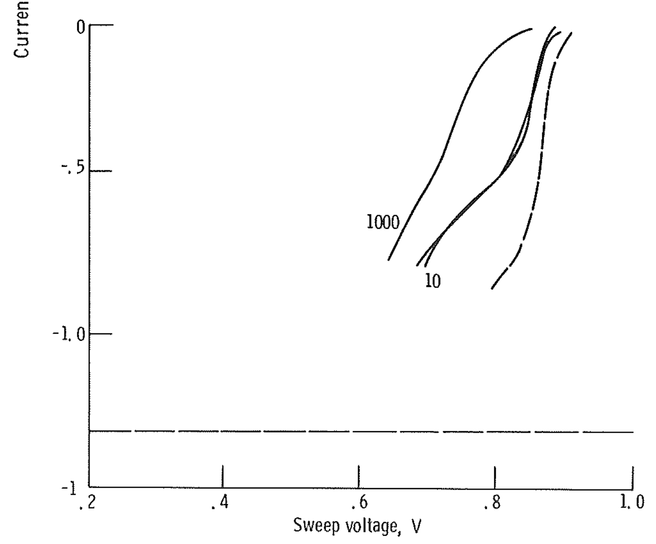


(a) Potential, U = 0.1 volt.



(b) Potential, U = 0.5 volt.

Figure 1. - Polarization curves for electroreduction of oxygen in 1 N KOH solution. Polarization curves measured by applying a linear potential-time sweep after indicated electrode pretreatment; electrode A used in cell I; oxygen-saturated solution mechanically stirred.

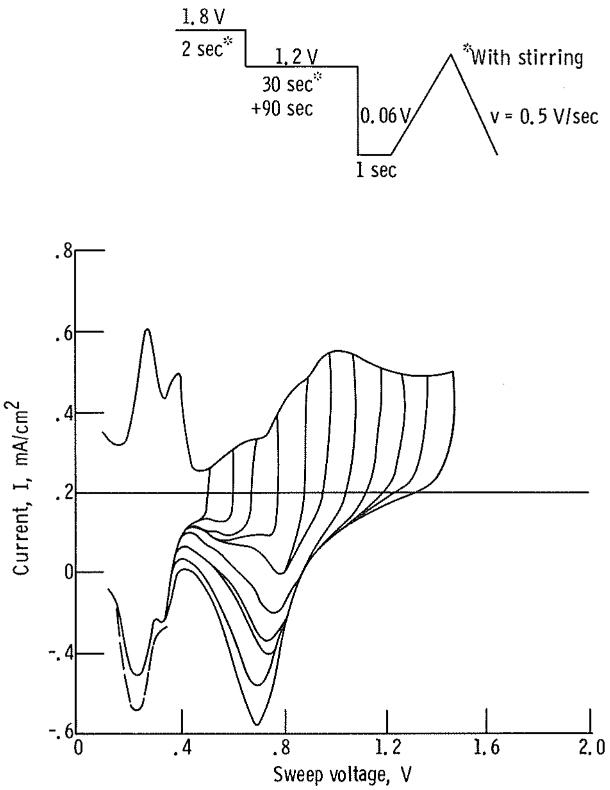


Figure 2. - Current-voltage curves measured during application of a linear potential-time sweep. Solution of 1 normal KOH degassed with argon; traces measured by applying triangular sweeps (different amplitudes) after indicated pretreatment.

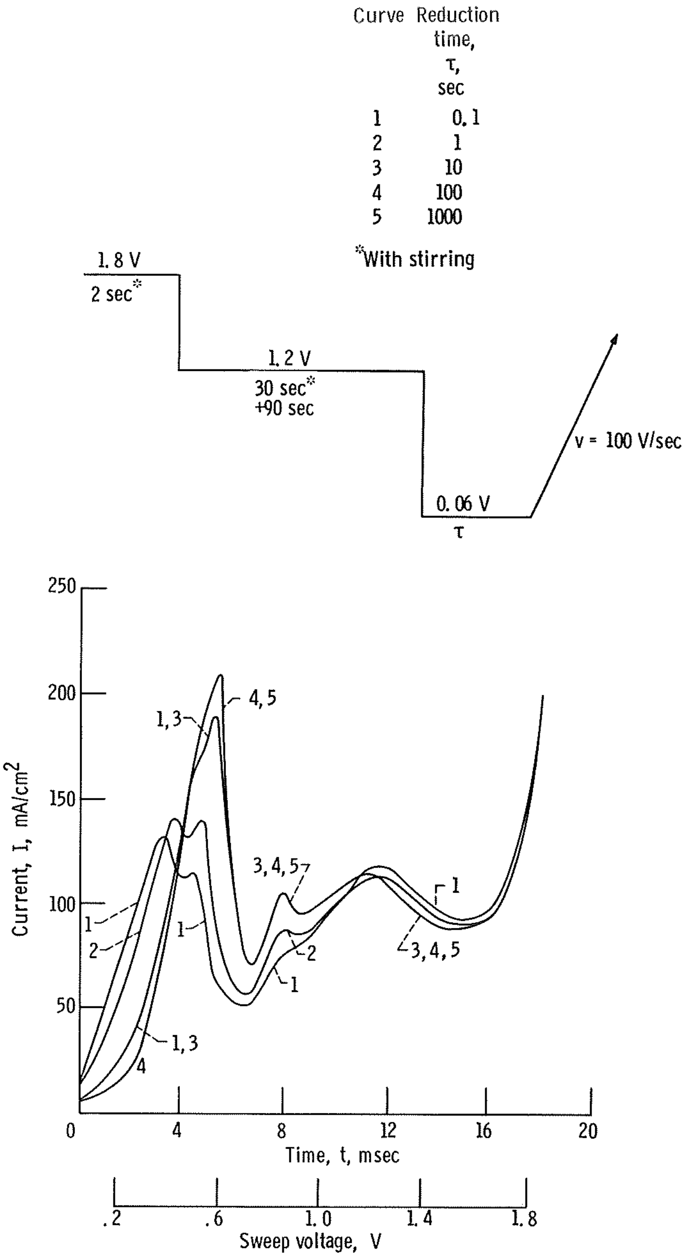


Figure 3. - Time-dependence of trace obtained during application of a linear anodic sweep. Experiment performed in cell II with solution degassed with argon-H_2 mixture.

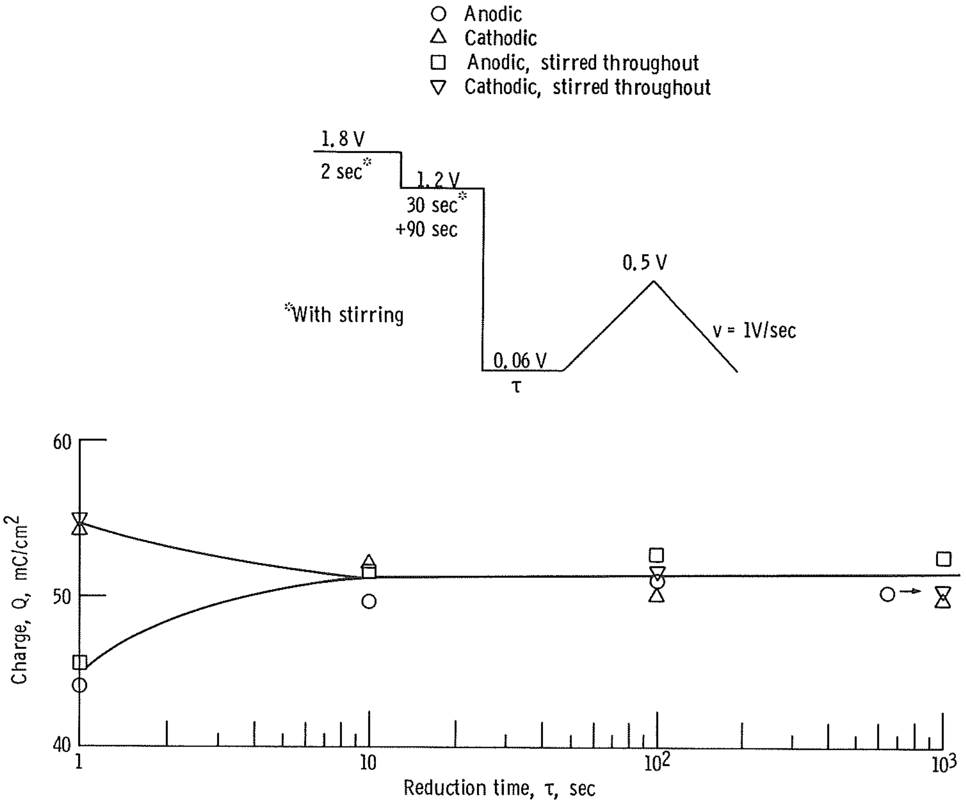
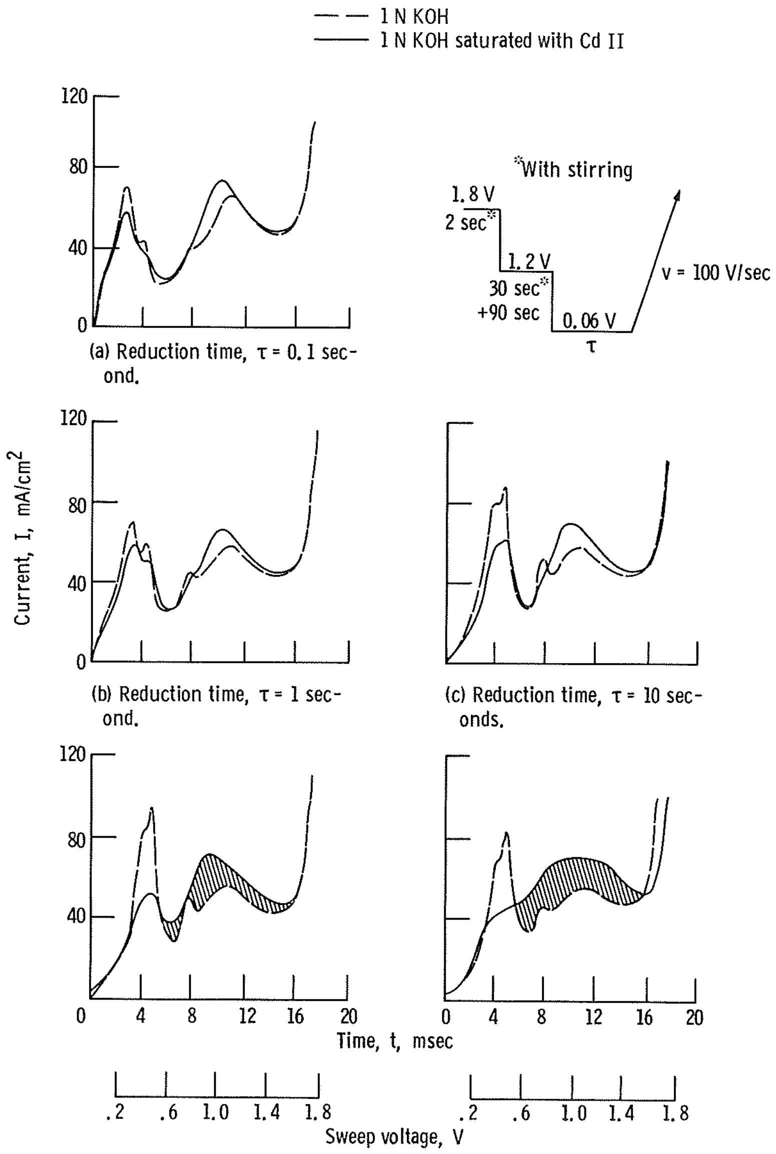


Figure 4. - Dependence of anodic and cathodic charges in hydrogen adsorption region upon reduction-time interval.



(a) Reduction time, τ = 0.1 second.

(b) Reduction time, τ = 1 second.

(c) Reduction time, τ = 10 seconds.

(d) Reduction time, τ = 100 seconds.

(e) Reduction time, τ = 500 seconds.

Figure 5. - Time-dependence of trace obtained during application of linear anodic sweep in presence and absence of dissolved cadmium ions.

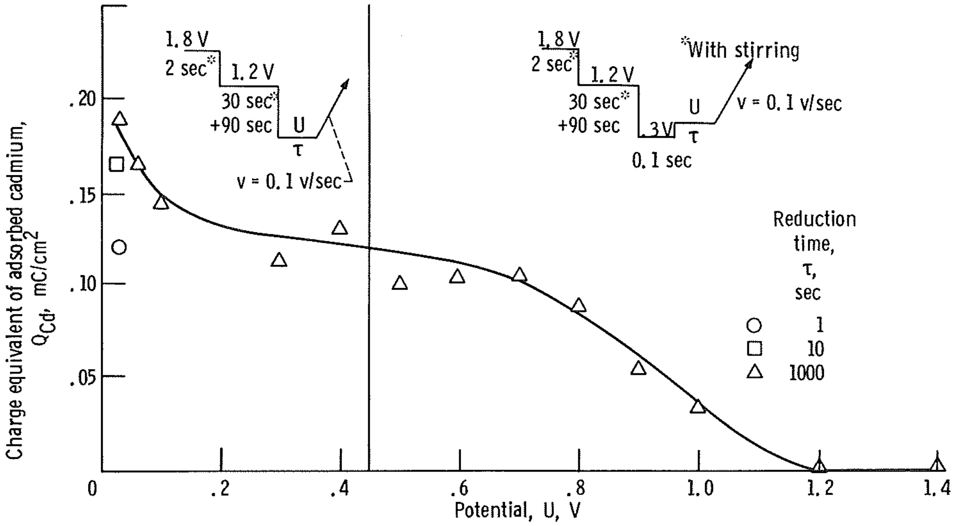


Figure 6. - Dependence of charge equivalent of adsorbed cadmium upon potential.

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