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APPLICATIONS OF THE LAWRENCE CORROSION DETECTION GAGUE (CDG-2)

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PHASE REPORT

ANALYTICAL REWORK PROGRAM - WORK REQUEST WR-2-5145

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S U M M A R Y

INTRODUCTION

The Naval Air Development Center is actively engaged in efforts to reduce the cost of materials and processes in support of the Analytical Rework Program. In keeping with this aim, the new Lawrence hydrogen detection gauge, CDG-2, was obtained on a no-cost loan basis for possible use in existing or new application areas in Analytical Rework Programs. This work was carried out under Analytical Rework Program Work Request WR-2-5145..

In view of the short loan period, it was decided to use the instrument for tests considered immediately relevant to naval rework programs. A current interest is protective coatings for steel fasteners used on removable aircraft access panels, which are presenting corrosion problems because of their exposure to corrosive environmental conditions. The hydrogen detection gauge was evaluated as a potential tool to detect inferior coatings of steel fasteners and to determine corrosion prone tempers of aluminum alloys.

In the Naval Air Development Center's coatings evaluation program, fasteners with different coatings are installed on aircraft panels and then are examined at six month intervals (reference (a)). In addition, preliminary data have been obtained from salt spray and SWAAT* tests. It is evident that if results can be obtained in hours using the hydrogen detection gauge as compared with weeks or months, this instrument would be of great value to the quick response capability, e.g., the fastener coating problem could be interpreted in terms of quality control of coatings, corrosive properties of materials, etc.

SUMMARY OF RESULTS

Calibration test data indicated that the reproducibility of the instrument was good. Results of test measurements for fastener coatings possessing different degrees of corrosion resistance were in general agreement with the ratings obtained from salt spray and SWAAT tests; i.e., low readings (small amounts of hydrogen generation) for the corrosion resistant coating and significantly higher readings for those coatings possessing poor corrosion resistance. Results of a limited number of tests to determine the susceptibility to exfoliation of aluminum alloy 7178-T6 and -T76 tempers were in agreement with the findings obtained with EXCO (exfoliation corrosion) immersion tests.

CONCLUSIONS

The results of the limited tests performed indicate that the CDG-2 has good potential for application in analytical rework programs. The use of the instrument can very likely be expanded to tests other than those performed in this work and should be a valuable tool where a quick response capability is important.

* sea water acetic acid test

RECOMMENDATIONS

It is recommended that additional work be done at the NAVAIREWORKFAC laboratory level to determine the specific role that the CDG-2 gauge might play in the Analytical Rework Program.

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DISCUSSION AND EVALUATION

BACKGROUND

The Lawrence Hydrogen Detection Gauge (LHDG) has been previously used as a practical tool for predicting hydrogen embrittlement and for the study of the attack of cadmium plating by aggressive paint removers (reference (b)). In this instrument the window of the vacuum hydrogen detection probe or sensor (HDP-3) is 1020 steel. In a very recent development, the window of the probe (ODP-H) is made of palladium/silver alloy, which greatly increases the measurement sensitivity for hydrogen.

COMPARISON OF THE CDG-2 AND LHDG INSTRUMENTS

The CDG-2 instrument used is shown in Figure 1. The probe or sensor of this instrument introduces a new method for measuring very small amounts of hydrogen generated by corrosion or released by parts after plating. After the test is completed, the hydrogen picked up by the probe is driven into the sensor for measurement by heating in an oven. The amount of hydrogen measured is expressed either as the hydrogen peak (HP) noted on the recorder chart or as I_r, the reading displayed on the NIXIE (a glow tube which converts a combination of electrical impulses into a numerical display). Both measurements are in arbitrary units.

The probe is used as an ionization gauge and the electronic system measures currents, which are related to the change in pressure or hydrogen content of the hydrogen which has permeated the membrane. A detailed description of the instrument and the principle of measurement are given in references (b) and (c). The new CDG-2 instrument has improvements in the fundamental functional features such as data storage, read out, oven and probe.

Since the oven is one of the most critical areas affecting reproducibility, improvements in the method of heating the probe in the oven during measurements has been accomplished in the CDG-2. Up until 1972, the probe was heated in a small oven maintained at 208 deg. C with the differential electronic control system depending on the response of thermistors. However, the thermal response was affected by the techniques of measurement; viz., time of insertion of probe, stray air currents, etc. which affected accuracy. In the CDG-2 the probe is heated in a programmed manner and has reduced measurement variables affecting accuracy.

In the LHDG instrument the hydrogen window of the probe is a 1010 steel membrane one inch in diameter and an area either 0.4, 1.8 or 4.8 sq. in. (small, regular and large windows respectively) depending upon the nature of the material or process to be evaluated. The probes are shown in Figure 2, and it is to be noted that an inert paint coating is used to mask the membrane to obtain the desired window area. In the CDG-2 instrument the window is a palladium/silver alloy tube 1/16 in. diameter and approximately 1¼ in. long (Figure 2). The use of the palladium/silver alloy is to increase the sensitivity of the instrument by taking advantage of the very high absorbing power of palladium. However, the walls of the tube are very thin

(0.003 in. maximum) and are easily bent or crushed. This necessitates great care in handling, since the probe can be easily destroyed.

The aforementioned comparison has been made to show the differences in the two hydrogen detection instruments to indicate their advantages or limitations for use in solving different types of problems.

EXPERIMENTAL PROCEDURES

A. Calibration Procedure

The initial phase of the work was concerned with setting up and calibration of the instrument. Calibration consists of baking out the hydrogen, to obtain a probe blank. The GDH-P probe is then cathodically charged for three minutes at 1 milliampere. After charging, it is washed for 10 seconds by gentle rinsing in approximately 450 ml of deionized water at 65 ± 5 deg. C and dried with a Kimwipe. It is immediately placed into the oven with a nitrogen flow of 2 liters per minute. After 2 minutes the oven is turned on and the calibration reading, I_r, (integral from "oven on" to the r point) and the hydrogen pressure peak, HP, are noted.

B. Procedure for Testing Corrosion Protection of Fastener Coatings

The corrosion test procedure used for the coated steel fasteners was performed in a 250 ml beaker containing an inverted glass funnel. Most of the stem of the glass funnel was cut off and a 1 in. polyethylene sleeve was force-fitted over the stem. The fastener was placed under the funnel and the corrosive test solution (3.5% NaCl + 0.05N H_2SO_4 in distilled water) was added to just cover the top of the polyethylene sleeve. The Ag/Pd probe was placed into the solution through the sleeve opening and the beaker raised using a precision support jack so that 1 in. of the probe was immersed. This ensured exposing a constant area of the probe test surface. At the end of a 15 minute immersion, the probe was removed; rinsed for 10 seconds in hot (70 deg C) deionized water; quickly and carefully dried with a Kimwipe, and then placed in the oven and the HP or NIXIE reading determined.

C. Procedure for Detecting the Susceptibility to Exfoliation of Aluminum Alloy 7178, T6 and T76 Tempers

Work on establishing a procedure for detecting the susceptibility of aluminum alloy T6 and T76 was rather limited because of time. Therefore, the following procedure described is to be considered preliminary and exploratory in nature. The specimen used was 1 x 1 x 1/4 in. It was placed on the bottom of a 50 ml beaker and covered with 40 ml of the arbitrarily selected corrosive test solution (7% NaCl + 0.1N H_2SO_4). The specimen was not covered with a funnel as in the previous procedure. One inch of the probe was inserted into the solution near the center of the specimen. At the end of 10 minutes the probe was removed; rinsed for 10 seconds in hot (70 deg. C) deionized water; quickly and carefully dried with a Kimwipe; and then placed in the oven and the HP or NIXIE reading determined.

RESULTS

A. Calibration Tests

Data obtained for a series of consecutive calibration runs are given in Table I. It is seen that the reproducibility is good, since results vary only about 10% from the mean value of the tabulated runs. (Run #3 is excluded from the reproducibility calculation because of the large deviation which cannot be explained).

B. Corrosion Resistance of Fastener Coating Tests

Standard aircraft fasteners of low alloy steel, UTS 125 ksi, which had been coated, were used for the tests. The coatings were Ametek (plated aluminum) Sermetel W (metallic coating in a ceramic matrix), Chromalloy (magnesium and silicon applied by a pack cementation process) and plated cadmium. Results obtained with the CDG-2 and preliminary results of salt spray and sea water acid (SWAAT) tests are presented in Table II for comparison. It is evident from the data that there is a marked difference in the corrosion resistance of the four coatings as indicated by the salt spray and SWAAT tests. The cadmium and Ametek coatings showed no corrosion and slight corrosion respectively, whil. the Chromalloy and Sermetel W coated fasteners were badly rusted. The data obtained with the CDG-2 also showed the same trend, i.e., very low readings for the corrosion resistant coatings and significantly larger readings for the coatings showing poor corrosion resistance.

C. Susceptibility to Exfoliation Tests

Results of tests for detecting susceptibility to exfoliation of aluminum alloy 7178, T6 and T76 tempers are given in Table III. Although the measurements are rather limited in number they indicate, in general, a trend of higher I_r readings for the T6 temper as compared with the T76 temper. These results also show the same trend as those obtained from EXCO immersion tests, where the T6 temper was found to be more susceptible to exfoliation then the T76.

DISCUSSION

Preliminary experiments were attempted with a 3.5 per cent sodium chloride solution but no readings above background were obtained. When the probe is used to pick up hydrogen in a test, it is necessary to use a more corrosive solution. For example, the manufacturer of the CDG-2 uses 0.1N sulfuric or hydrochloric acids or 3% sodium chloride in 0.1N sodium hydroxide for aluminum corrosion studies.

The corrosive test solution used for testing the corrosion protection coating of fasteners was 0.05N sulfuric acid + 3.5 per cent sodium chloride. For the preliminary work on detecting the susceptibility of aluminum alloy 7178 T6 and T76 tempers a 0.1N sulfuric acid was used and found to be unsatisfactory. When using the CDG-2 for a specific problem, it is necessary to determine empirically the concentration and length of exposure time of the probe.

Extreme caution must be exercised because the probes are extraordinarily sensitive to hydrogen and pressures in excess of a NIXIE reading of 3000 can destroy the probe. It is evident from the above examples that the CDG-2 instrument has made possible the measurements of hydrogen in corrosive environments, which would not have been possible with the previous LHDG model.

It is interesting to note that the instrument manufacturer has been experimenting with two new techniques. In one, a carrier gas is used and the probe is placed in the hot oven and nitrogen is passed over the heated sample (in an oxygen free chamber) before entering the heating coils of the CDG oven. The disadvantage of this technique is that the hydrogen/palladium reaction time is limited. In the second technique the probe and sample are placed in a closed cell filled with nitrogen, where heat can be applied to the probe tip and sample independently. Palladium bars can be immersed in the corroding test solution/metal environments and analyzed by the above techniques. This overcomes the problem of extreme care in handling the probe and in addition increases the versatility of the instrument (reference (d)). Other applications, for example, the determination of hydrogen in steel, zirconium and titanium are given in reference (e).

In view of the above mentioned applications based on our work and that of the manufacturer, the CDG-2 offers promise for application in the naval rework program.

TABLE I

CALIBRATION DATA

Run No.	I_r (NIXIE READING)*
1	235.2
2	223.3
3	146.3
4	259 1
5	251.3
6	276.8
7	256.8

* Integral from "oven on" to the r point, the hydroge pressure peak, HP.

TABLE II

COMPARATIVE CDG-2 AND SALT SPRAY TEST DATA FOR CORROSION PROTECTION COATINGS ON AIRCRAFT FASTENERS

COATING	SALT SPRAY TEST 14 DAY RATING	COMMENTS	SEA WATER ACID TEST (SWAAT)	CDG-2 READING I_r
Cadmium	1	No corrosion	2	.000 .003
Ametek	4	Slight corrosion	1	.001 .001
Chromalloy	10	Badly rusted	4	7.61 14.2 15.0
Sermetel W	12	Badly rusted	3	0.157 0.469 2.18

TABLE III

CDG-2 TEST DATA FOR DETECTING EXFOLIATION SUSCEPTIBILITY OF ALUMINUM ALLOY 7178 T6 and T76 TEMPERS

	T 6	T 76
TEST NO.	I_r READING	I_r READING
1	0.380	0.207
2	0.285	0.147
3	0 486	0.327
MEAN	0.384	0.227

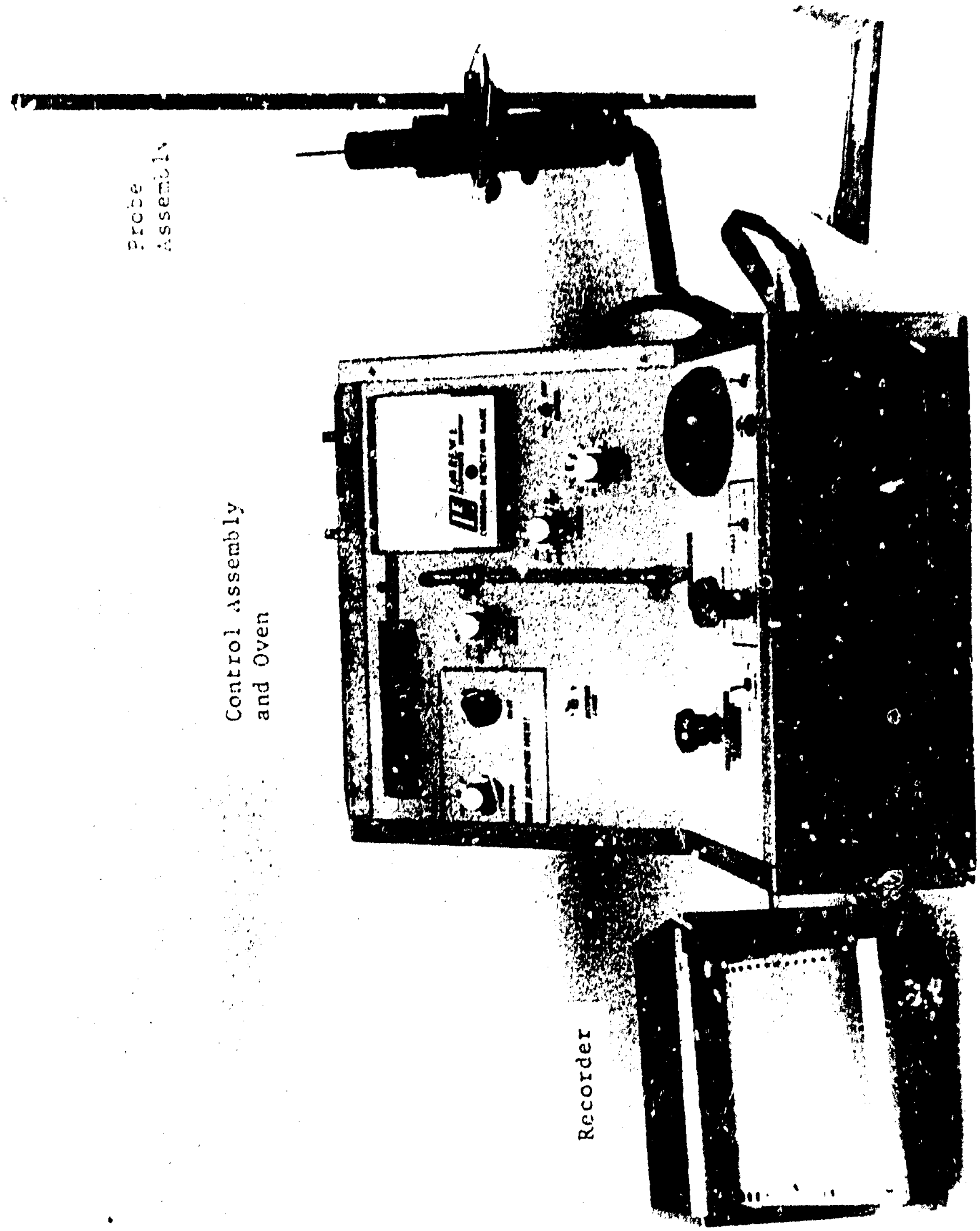

Figure 1. Lawrence Corrosion Detection Gauge (CDG-2)

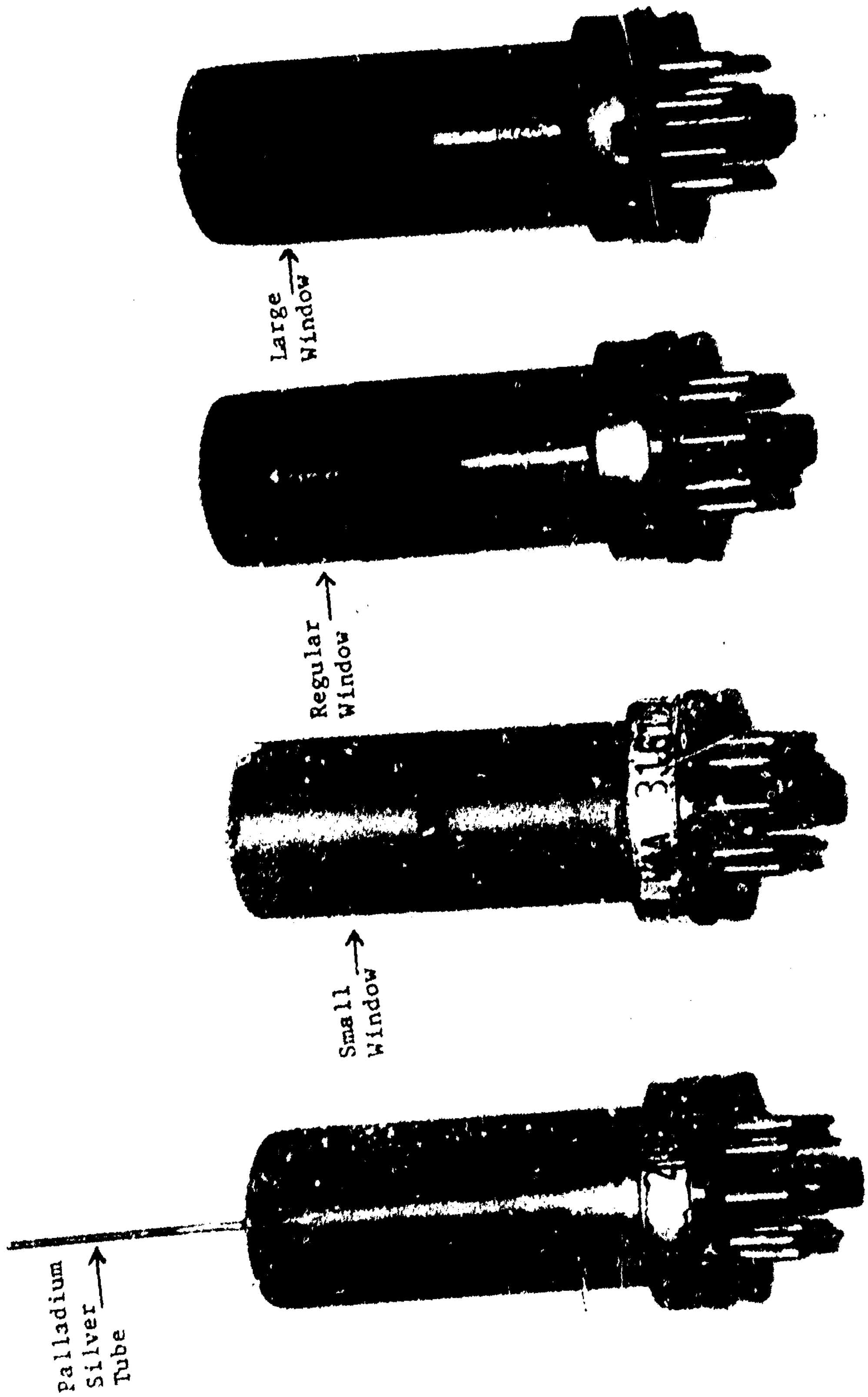

Figure 2. Hydrogen Detection Probes Used in the CDG-2 and LHDG Instruments

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(a) JANKOWSKY, F.J., "Fastener Coating Service Evaluation", Naval Air Development Center Report No. NADC-MA-7151 of Feb 1972.

(b) FISCHER, P., "Evaluation of the Lawrence Hydrogen Detection Gauge for the Prediction of Hydrogen Embrittlement", Naval Air Development Center Report No. NADC-MA-7151 of Feb 1972

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