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Rock Island Arsenal Laboratory

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TECHNICAL REPORT

ELECTRICAL RESISTANCE CORROSION INDICATORS LITERATURE REPORT

By

Robert E. Johnson

SEP 6 1962

Department of the Army Project No. 593-21-062

Ordnance Management Structure Code No. 5010.11.80300-01

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A. C. Hanson

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Laboratory Director

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ABSTRACT

The work of the Air Force, Georgia Institute of Technology, Crest Instrument Division of Magna Products, Inc., and Forest Products Laboratory, was reviewed to establish the degree of sensitivity of electrical resistance corrosion indicators and their applicability to the evaluation of corrosion-preventive oils and compounds.

Laboratory work conducted under carefully controlled conditions indicated a high degree of sensitivity utilizing thin film indicators. Exposed specimens which exhibited no corrosion when examined visually, provided small resistance changes of ~~one~~ to ^{five} percent by careful electrical measurements during the same period. Although each individual electrical indicator detected corrosion, variation in response or lack of reproducibility indicated an undesirable factor for use in field tests.

The adaptation of the corrosometer, originally designed to utilize probe type specimens, to a glass slide having a thin metal film, provided a high degree of sensitivity. Satisfactory results could be obtained only after a thorough check of the electronic system and elimination of variables in the procedure.

There are other matters which must be considered concerning the use of corrosion indicators. The highly sensitized surface presents the possibility of a reaction with contaminant gases and particulate contamination. In addition, with the high concentration of additives in corrosion preventive materials, it is possible that a reaction may occur between the additives and the sensitized surface. Such reactions would be undesirable since they would produce changes in electrical resistance without the presence of a corrosive environment.

RECOMMENDATIONS

Laboratory work should be conducted to refine the current indicators to provide the sensitivity needed in corrosion preventive testing.

**ELECTRICAL RESISTANCE CORROSION INDICATORS
LITERATURE REPORT**

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ELECTRICAL RESISTANCE CORROSION INDICATORS LITERATURE REPORT

OBJECT

To establish the degree of sensitivity of electrical resistance corrosion indicators and relate resistance values to visual deterioration of corrosion preventive oils and compounds.

INTRODUCTION

Current methods of determining the protective characteristics of corrosion preventive oils and compounds involve either a visual observation of an exposed metal surface or a determination of weight loss. The former indicates whether rusting is evident, however, it does not provide a quantitative measurement. The latter provides quantitative data, however, it is lengthy and time consuming. There is a need for a method which will eliminate a visual examination and provide results quickly in a quantitative manner.

A new technique is now available by which corrosion rates may be determined quickly and accurately. This method consists of measuring the change in electrical resistance of a metal specimen placed in a corrosive medium. Since the resistance of an electrical conductor is inversely proportional to its cross section, the change in resistance of a specimen over a period of time can be related to the amount of corrosion that occurs.

At the present time there are two procedures that utilize the electrical resistance technique. The first involves the use of a glass slide that has been coated with a very thin, vacuum deposited film of iron. The iron film can be sensitized with a salt to provide the proper response time for packaging applications. Suitable leads are attached and the resistance is determined by use of an ohmmeter. A similar indicator, without electrical connections, is also available where a visual examination is sufficient. This method has been successfully used to detect the presence of a corrosive atmosphere.

The second procedure involves the use of a corrosion probe. The probe is constructed of the metal for which corrosion data is desired. A currently available instrument, the corrosometer, utilizes an exposed metal specimen as one arm of a modified Kelvin Bridge circuit and a second adjacent arm of the bridge circuit of the same metal as the exposed specimen. The second specimen is coated with a highly

corrosion resistant coating, and therefore retains its original cross section. Both specimens experience the same percentage change in resistance with any temperature change so that the ratio of the two resistances is a function of the cross section of the exposed specimen.

Current procedures in use to determine the protective properties of corrosion preventive materials, generally require that coated and exposed test panels permit the formation of no more than three corrosion dots, none of which is larger than one millimeter in diameter. It is not known whether electrical resistance techniques are sensitive enough to detect such a light rusting condition.

This work will consist of a survey of available literature on electrical resistance corrosion indicators to establish the suitability of the indicators in the detection of incipient corrosion and their application to corrosion preventive testing.

PROCEDURE AND RESULTS (Literature Survey)

A search of the literature revealed numerous references on the subject of electrical resistance corrosion indicators which are described as follows.

Development of Thin Film Indicators

Roller⁽¹⁾ studied the corrosion rates of thin, continuous, adherent, vacuum deposited films at various humidities to determine their suitability as corrosion indicators. This study revealed that differences noted in the rate and amount of corrosion appeared to be due to the thickness of the iron film and the substrate employed. A definite decrease in corrosion was evident when the thickness of the films were too thick or thin, and when a plastic, polymethacrylate resin was utilized as the substrate rather than borosilicate glass.

McCloud, et al.⁽²⁾ also studied different variables and found that thin films corroded earlier. A film thickness of 1000 Angstroms provided the best overall results. No difficulties were encountered in obtaining a good coating if the surface had been degreased and dried prior to deposition of the film.

The work of Roller⁽¹⁾ indicated that adsorbed gas layers on the surface of the glass substrates affected the rate of corrosion. This condition was eliminated by removing the adsorbed gas layers by positive ion bombardment. The presence of adsorbed gas layers was confirmed by Belser and Hankinson⁽³⁾. The gas layer furnished the oxygen necessary for small particles

of the oxides of iron to form at random sites on the film. These subsequently became elements of electrolytic cells in which the iron film acted as the anode in the presence of air at 25° C and 70% R.H. Destructive oxidation of the film thereafter proceeded by electrochemical processes. The gas layer was effectively removed by predeposition heating of the glass substrate to 400°C.

In addition to iron, Belser, et al⁽³⁾⁽⁸⁾ also studied various nonferrous metals. Considering all of the metals tested, iron was the most satisfactory. Films of manganese and bimetal films of iron and copper exhibited corrosion characteristics similar to iron except that their corrosion rates were slightly greater.

McCloud, et al⁽²⁾⁽⁴⁾ found that pure iron does not corrode rapidly enough or reliably enough to be suitable as a corrosion indicator. The surface must be sensitized with a salt in order to provide the necessary rate of corrosion. The need for a sensitized film was confirmed by Belser and Hankinson⁽⁵⁾ who found that films sensitized by a salt or hydrochloric acid corroded at 25°C when the relative humidity rose above approximately 30%. The work indicated that both sensitized and unsensitized films may be needed for a proper indication of corrosive conditions.

In the preparation of a sensitized indicator, McCloud, et al⁽²⁾⁽⁶⁾⁽⁷⁾ utilized ammonium chloride in methyl alcohol. The most suitable method of application was by spraying the thin metal surface. This procedure, however, produced an indicator with a response time which was too rapid for use in normal packaging operations. It was possible that such an indicator would show visual evidence of corrosion prior to placement in a package. This problem was resolved by using a combination of four parts of sodium chromate to one part of ammonium chloride. This provided a proper delay in response time and uniformity of corrosion. The delay can be increased with an increased concentration of sodium chromate. It was pointed out that when films are sensitized, variables other than temperature and humidity have little if any effect.

The work of Belser, et al⁽⁵⁾⁽⁸⁾ included studies of salt treated films in the early experiments. Since the use of salt obscured the effect of all variables other than temperature and humidity, the primary effort was made in the studies of unsensitized films. In conjunction with this work, efforts were made to trace sources of inadvertent sensitization.

The work of Belser and Hankinson⁽⁹⁾ indicated that the films were being sensitized inadvertently by chemical fumes of ferric chloride or nitric acid coming from a nearby chemical laboratory. The contaminated specimens corroded very rapidly upon subsequent exposure. To prevent a recurrence, an inert gas dry box was constructed for use in the preparation of specimens. It was indicated that exposure to room temperature should be held at a minimum.

In their work utilizing unsensitized films, Belser and Engel⁽⁸⁾ conducted extensive studies of bimetal layers of iron and copper. A more noble metal in contact with the iron was used to accelerate the corrosion of iron. It was revealed that films of copper strips plus iron in a "ladder" configuration produced resistance measurements that clearly indicated differences in substrate cleaning. Difficulties were encountered in the use of films prepared by simultaneous evaporation of copper and iron due to problems in the control of alloying proportions. This contributed to a large scatter in corrosion rates observed.

Development of Corrosion Probes

The use of corrosion probes in monitoring corrosion in refinery equipment was investigated by Freedman, et al⁽¹⁰⁾. The principle of electrical resistance was successfully used under all conditions of temperature and pressure encountered in refinery operations. Corrosion rates in vapor and liquid phases, with both oil and aqueous solutions, have been measured.

With the aid of corrosion probes, corrosion rates were determined quantitatively in several hours, as compared to the months required for coupon measurements, water analyses, and unit inspections. The effects of changes in unit operating procedures upon corrosion rates were observed shortly after changes were made. Various corrosion inhibitors were compared rapidly in actual use. The probes were also found to be very useful in establishing corrosion rates in sensitive locations or in new processes where little was known.

The Crest Instrument Division of Magna Products, Inc., Santa Fe Springs, California, currently produces the Corrosometer which utilizes the corrosion probe. The probes can be fabricated from numerous metals in the form of wire, strip or tube, dependent upon the application. The unit provides continuous monitoring of small resistance changes in a corrosive environment.

Belser and Engel⁽⁸⁾ adapted the Corrosometer to study the corrosion of thin metal films. Iron and copper were

simultaneously evaporated onto a glass slide. The coating was divided, with one-half being left unprotected and the other half coated with Glyptal.

Although time permitted only limited tests to be conducted, the data collected indicated the Corrosometer to be a highly sensitive indicator of corrosion of the metal film tested and that it would be useful in more extensive studies of corrosion. Additional work is necessary, however, to obtain a more suitable coating than Glyptal for the reference specimen.

Electrical Resistance Measurements

In the original work on thin metal films, Roller⁽¹⁾ measured the electrical resistance per inch using an ohmmeter. Since the film was only 1/8 inch wide, it was necessary to use a correction factor of 8 to establish the resistance per square. The work revealed that the electrical resistance of an exposed film did not increase to any great extent until corrosion was well advanced and readily observed. At that point the resistance of the specimen increased until almost infinite values were obtained. It was indicated that resistance values utilizing this technique approximated those obtained by more elaborate techniques and provided a suitable means of comparing film thickness and monitoring corrosion.

The work of Belser and Engel⁽⁸⁾ indicated that thin iron films deposited by vacuum evaporation on clean glass slides did not show any visual corrosion after 60 days at 25°C and 70% relative humidity. However, careful electrical measurements indicated small resistance changes of one to five percent during the same period. Attempts to increase the corrosion rates by roughening the substrate, partially oxidizing the film, placing the film in tension, or utilizing bimetal films of iron and copper, were only partially successful. Although corrosion rates increased by a factor of two or three, data scatter was still widespread.

McCloud, et al⁽¹¹⁾ found that electrical resistance indicators corrode at the same rate as visual indicators when factors influencing the rate of corrosion are held constant. Electrical indicators generally have an initial resistance of 10 ohms. The resistance must increase by a factor of 100 to a value of 1000 ohms to provide a general comparison with moderately corroded visual indicators. Currently available instrumentation calls for corrective action when the electrical resistance of the film reached 1000 ohms or more.

Electrical indicators of the card type, having an area of 1.75 square inches and the button type having an area of

0.12 square inches were developed. The card indicators were most suitable due to the larger area available. While tests of replicate specimens indicated only slight scattering of data points, it was revealed that corrosion did not effect the resistance of the film until at least 30% of the area had been corroded. It was revealed that when significant corrosion was present in thin films, hardware items showed only minor corrosion.

Performance Tests Utilizing Electrical Resistance Indicators

The Forest Products Laboratory conducted work (not yet published) to determine the suitability of corrosion indicators utilizing thin film indicators.

This project utilized the different types of visual and electrical thin film indicators to detect corrosive effects on different metals in various environments. Tests were conducted at 80°F and 120°F and relative humidities ranging between 30 and 95% utilizing steel, magnesium, cadmium-plated steel and aluminum specimens. The electrical measurements were obtained by utilizing a portable corrosion meter. The meter was designed to indicate only whether an environment was good or bad. There was no provision made to indicate the degree of corrosiveness. Forest Products Laboratory personnel added a graduated scale so as to be able to determine changes in resistance between inspections.

The test results exhibited widespread variation. In a particular group of thin film test specimens, the minimum failure time for an individual specimen was 13 hours, while the maximum time was 34 hours. Under static or rising humidity conditions the difference in average failure time between the indicators and the metal specimens provided a reasonable time period in which a corrosive condition could be detected and repaired. Under cycling conditions, however, the order of corrosion was reversed. The steel parts, having a greater mass than the indicators, exhibited condensation first. Under such conditions the indicators did not detect the presence of a corrosive atmosphere. It was indicated that this defect could probably be corrected by attaching the indicators directly to the packaged item.

One of the tests conducted utilized an automatic alarm device to signal the presence of a corrosive atmosphere by a flashing light. Upon working with the corrosion meter the indicators showed a corrosive atmosphere.

The published report on the above project will provide complete information and data and possibly determine the basis for the generally positive results obtained.

DISCUSSION

As indicated in the previous section, considerable effort was expended in the evaluation of electrical resistance corrosion indicators. Since this technique provides a rapid means of detecting a corrosive atmosphere, it was necessary to establish their sensitivity in the detection of corrosion.

From the standpoint of handling or use, the indicators are quite sensitive. It was revealed that precautions are necessary to prevent the indicators from being inadvertently sensitized. Chemical fumes commonly present in a laboratory will rapidly accelerate the rate of corrosion and thereby produce large increases in resistance.

In order to successfully utilize electrical resistance corrosion indicators in corrosion preventive testing, it was necessary to know if very minor or incipient rusting could be detected. According to Belser and Engel⁽⁸⁾ thin iron films deposited by vacuum deposition onto clean glass slide did not corrode to such an extent that it was visible to the naked eye after exposure at 25°C and 70% relative humidity in periods greater than 60 days. Careful electrical measurements, however, indicated small resistance changes of one to five percent during the same period. This would seem to indicate the presence of very minor corrosion which could not be detected by the naked eye.

The work of Forest Products Laboratory, however, did not indicate a very high degree of sensitivity utilizing the thin film indicators. Widespread variation in results were evident in tests conducted.

Belser and Engel⁽⁸⁾ adapted the corrosometer for use with a glass slide having a thin metal film. The data collected indicated the corrosometer to be a highly sensitive indicator of corrosion and would be useful in more extensive corrosion studies. It was noted, however, that satisfactory results could be obtained only after a thorough check of the electronic system and elimination of variables in procedure.

There are other matters that must be considered concerning the use of resistance indicators. The development and investigative work on the indicators was conducted under carefully controlled laboratory conditions in atmospheres free of contaminant gasses and particulate contamination. Since the surface of the indicator is so highly sensitized, it is conceivable that a reaction could occur between the metal surface and airborne contamination, such as sulfur dioxide, hydrogen sulfide, charcoal, dust, etc.

The work of Ronceray(11) describes the effects of contamination. It was revealed that highly polished steel surfaces exhibited no corrosion for eight months at static humidities of 95 to 100 percent, using highly purified air and water vapor. When the surfaces were prepared by ordinary polishing procedures, impurities were retained in minute grooves and fissures on the surfaces and corrosion appeared much sooner. Similarly, the introduction of contaminants to the test environment could serve as an accelerator and produce corrosion in a much shorter period of time.

It should also be mentioned that available data on resistance indicators was based upon tests conducted utilizing bare indicators. In order to be successfully utilized in corrosion preventive testing, the indicators must be coated with the material to be tested. Current corrosion preventive materials are highly fortified with additives to provide corrosion protection, resistance to oxidation, water displacing properties and suitable viscosity temperature characteristics. With the high concentration of additives, it is possible that a reaction may occur with the highly sensitized surface of the indicator. This could result in corrosion without the presence of a corrosive environment and thereby produce erroneous results.

Based upon the information obtained in the literature and work in progress, the current corrosion indicators have not been perfected to such a degree that they can replace the test panel in protection tests of corrosion preventive oils and compounds. Prior to initiating a program of this nature, it is recommended that work be conducted to refine the current indicators to provide the sensitivity needed in corrosion preventive testing.

The corrosometer would, however, provide a satisfactory and rapid means of comparing the corrosiveness of various testing environments and would be of benefit in the corrosion preventive program of this laboratory.

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The adaptation of the corrosionometer, originally designed to utilize probe type specimens, to a glass slide having a thin metal film, provided a high degree of sensitivity. Satisfactory results could be obtained only after a thorough check of the electronic system and elimination of variables in the procedure.

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