

INSTRUMENTATION FOR MEASUREMENT OF THE EFFECTIVENESS OF VAPOR CORROSION INHIBITORS

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ABSTRACT

Corrosion damage to process instrumentation, computer systems and electrical switch gear due to corrosive environments is frequently being controlled by the use of vapor corrosion inhibitors (VCI). Additionally, these inhibitors are used to protect products that are being packaged for storage and/or shipment, and for lay-up of major equipment systems. Typically, the evaluation of their effectiveness has been by utilizing weight loss coupons or by direct observation of the protected surfaces. Neither technique provides a timely indication loss of protection due to consumption of the VCI or is amenable to remote monitoring.

A more serviceable technique utilizing thin-film electrical resistance corrosion sensors with relatively low cost instrumentation has been developed. This technology offers the advantages of continuous monitoring of the potential corrosive environment as well as the capability to forecast the need for additional VCI without risking damage or deterioration of the protected products or equipment.

Results are presented from operational experience within a chemical laboratory environment as well as test results obtained within a test chamber where different corrosive gases and VCIs were evaluated.

Keywords: Atmospheric corrosion, corrosion monitoring, environmental corrosion, vapor corrosion inhibitor, VCI

INTRODUCTION

There is an increasing awareness of the deterioration in the functionality of electronic, electrical and mechanical systems and their components due to corrosion caused by low levels of corrosive gases and humidity in their operating and storage environments. Environments that were previously thought to be innocuous are proving to be sufficiently corrosive to cause malfunctions or total operational failures after a relatively short exposure time. In many ways, modern electronic equipment has become less tolerant to these environments due to the proclivity toward higher operating speeds, lower voltage and current levels, high density interconnection devices and closer spacing of components and traces on printed circuit boards. Even very small surface penetration and very thin films of corrosion products (from tens to a few hundred angstroms for example) can be sufficient to severely degrade performance or cause a functional failure of a system. The most common types of failures in electronics are from malfunctions of relays, contact, and electrical connectors; all primarily due to corrosion.

Several manufacturers of process instrumentation and computer systems have experimented with coupons and thin-film corrosion sensors which were mounted inside equipment enclosures or strategically placed in the controlled atmosphere storage environments. These provided long-term trend information relative to the corrosivity of the environment, but more timely information is necessary in order to take corrective action when the environment is degrading at an accelerating rate due to depletion of VCIs or other corrosion inhibitors. In some cases the need for VCI replacement was hastened by a breach of the packaging that permitted the escape of VCIs and entry of more corrosive external environment.

With the recent development of more effective and inexpensive **VCIs**, major components and systems in large process plants are being placed in **lay up** with **VCIs** used for the long term corrosion protection. In these applications a cost effective corrosion monitoring system can be critical to the success of a **VCI based** corrosion control program.

In order for a sensor and instrument to be useful in the generally low corrosion rate application it must be capable of providing very high resolution so that quantitative corrosion rate information can be determined over relatively short periods. Counter to this quality, the sensor must have sufficient measurement life so that, under normal circumstance, replacement is generally not necessary over the period of its use. And, the

sensor must be available in a number of different materials that will be responsive to the many airborne corrosive materials that are encountered.

SENSOR

In early experiments it was determined that average indoor ambient corrosion rates were in the range of 10 to 100 angstroms per month (A/mo.) when not under the influence of VCIs, and usually less than 10 A/mo. when VCI protected. A sensor measurement span of 250 A was selected since it would provide a minimum service life of approximately 24 months at a nominal corrosion rate of 10 A/mo.

The basic sensor design consists of a thin metal film configured in a serpentine pattern with two electrical taps at each end and one at the center. This geometry provides two identical resistive elements, one of which is the corrosion measurement surface and the other which is the reference element that provides first order temperature compensation. The reference element is coated with an insulating material to protect it from being attacked by the potentially corrosive environments being measured. The multiple electrical taps permit the precise measurement of the element resistances with an adaptation of the well known Kelvin bridge circuit. Using this configuration will compensate for errors caused by cable resistance. Changes between the electrical resistance of the reference side and the corrosion side of the sensor are then related to corrosion rate.

The sensor elements are fabricated using a vacuum sputtering technique which provides a high material purity and a uniform thickness. The substrate material is soda glass upon which a thin (approximately 25 A) intermediate layer of a proprietary material is deposited in order to increase the strength of the bond between the element material and the substrate. This intermediate layer improves the bonding of the sensor element material to the glass substrate.

Some metals are more sensitive to specific airborne corrosive materials than others, thus a variety of different materials must be considered in order to meet the wide diversity of applications. For initial testing, copper and iron sensors were chosen. Copper, because it is used extensively in electrical and electronic equipment, and iron since the corrosion of steel surfaces is critical in many components and systems.

INSTRUMENT

Since the primary purpose of the instrument is to determine corrosion rates from a few angstroms per month to a few tens of angstroms per month, the design must have the capability of resolving to the nearest angstrom and repeatable to ± 1 A. This would be sufficient to establish reliable corrosion rate information over measurement intervals of a few days.

The instrument design utilizes a high performance, very low power analog-to-digital converter. This integrated circuit includes the capability to directly drive a 3½ digit liquid crystal display and gives 8,000 hours typical battery life with a standard 9-volt alkaline battery. Evaluation testing of the instrument design verified the capability of the design to meet resolution and repeatability requirements. The only drawback to this design is the fact that the integrated circuit configuration prevents the implementation of a Kelvin bridge configuration that will compensate for the resistance of sensor extension cables. Error analysis and subsequent testing determined that the use of extension cables from remotely mounted sensors would have negligible effect on corrosion rate data if the cables were limited to 100 feet in length.

LABORATORY TESTING AND RESULTS

Initial testing was performed to determine the corrosion rate on a copper sensor exposed to an average office environment to simulate how computer equipment, electronic test equipment and related items would be affected. Since these components are frequently used and stored in this quality of environment, testing would establish a baseline value of corrosion rate and aid in the determination of the effectiveness of VCIs for enhanced corrosion protection.

The testing was performed in a 200 liter polyethylene test chamber which had been thoroughly purged with ambient air. Figure 1 illustrates resistivity readings which were taken throughout this experiment. The initial resistivity measurements taken twice daily and the equivalent corrosion rate (on copper) of the room air was determined to be relatively constant at 17 A/mo. over the first four days of the test. After the fourth day VCI's were introduced into the chamber by installing a number of devices which emit the amount of VCI required to protect a volume of 200 liters. These devices, available from Cortec Corporation as VCI-110's, were special hollow yellow polyurethane foam domes filled with a mixture of VCI compounds which volatilize and diffuse through a permeable membrane into the surrounding atmosphere at a controlled rate. They contain a small adhesive base and were mounted to the wall of the test chamber. Subsequent readings were taken on a daily basis. Over the next eight days the resistivity change corresponding to a corrosion rate remained at a level of 17 A/mo. ** The "corrosion" rates given throughout this paper are assumed to correlate with the measurements obtained from the instrument and known corrosion rates measured using other techniques. In actuality, these readings represent any change in the surface which affects conductivity. Beginning with the twelfth day and continuing through the twenty-seventh day of the test, a steady decrease in the resistivity was measured. From the twenty-seventh day until the fifty-seventh day the corrosion rate of the copper sensor under VCI protection was determined to be nearly constant at 4 A/mo. After the reading was taken on the fifty-seventh day, the VCI emitters were carefully removed from the chamber so as to minimize the resistivity changes indicated the corrosion rate (of change in surface chemistry) showed an increase to approximately 8 A/mo. and showed a slightly increasing trend until the conclusion of the test on the eighty-ninth day. This trend is attributed to the slowly decreasing concentration of VCIs as they are depleted in the chamber environment.

After this testing, the used sensors along with an unused, unopened sensor, were sent to an outside laboratory for evaluation of surface chemistry. The surfaces were examined in detail using very sensitive surface analytical instrumentation, ISS (Ion Scattering Spectroscopy) and SIMS (Secondary Ion Mass Spectroscopy). Both these techniques are sensitive to the outer atomic layers of a surface and provided information on how the chemical composition changes from the outer few atomic layers to a depth of about 100 Angstroms. Both techniques showed substantial differences in the surface

chemistry between the used and unused sensors. Typical ISS and SIMS spectral data are shown in Figures 2 and 3. This information not only indicated the presence of CuO but also other contaminants including both organic and inorganic materials. The effect of these other contaminants on the resistivity measurements and the corresponding corrosion rates was not established but the nature of these surface contaminants is such that it would most likely also interfere with subsequent contact performance and degradation.

CONCLUSIONS.

The ISA standard, ISA-S71-04-1985, @ Environmental Conditions for Process Measurement and Control Systems: Airborne Contaminants, classifies a copper reactivity level of <300 A/mo. as mild and that corrosion in such an environment is not a factor in determining equipment reliability. However, as requirements continue for lower operation voltages and lower contact forces, it is quite likely that this reactivity rate will decrease. When comparing the above corrosion rate information to the standard, it is clear that the capability to monitor changes in resistivity of thin Cu films indicative of corrosion and changes in surface chemistry in a protected environment has been well demonstrated along with the ability to measure VCI performance.

REFERENCES

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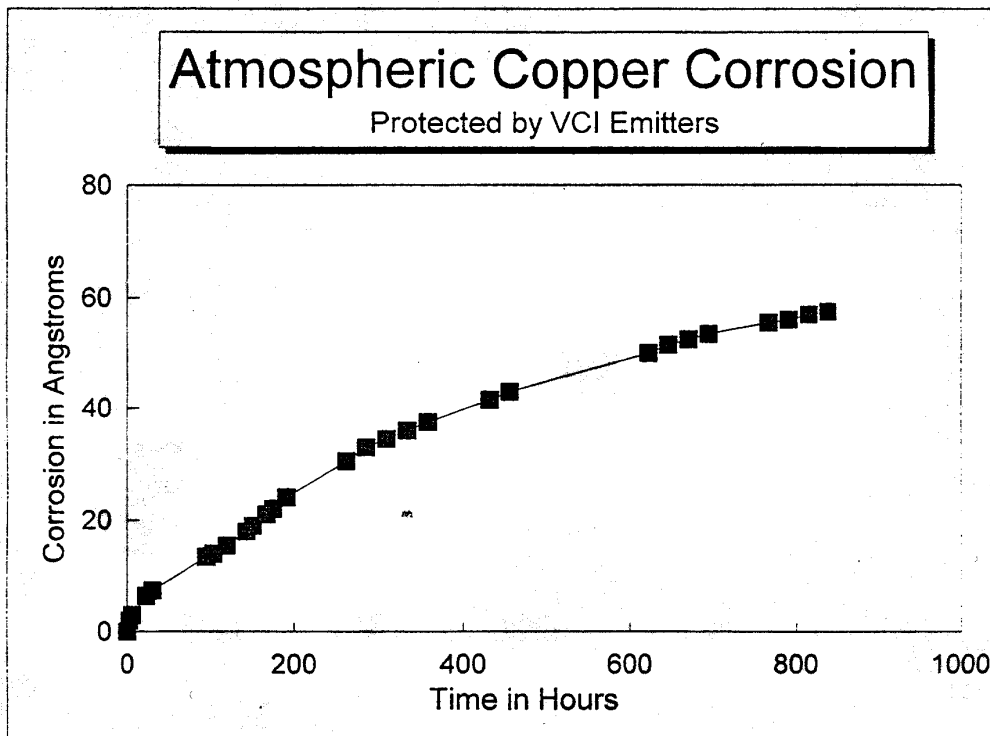


Figure 1. A plot of equivalent total corrosion of Copper (Cu) in Angstroms as measured in test chamber exposed to VCI emitters.

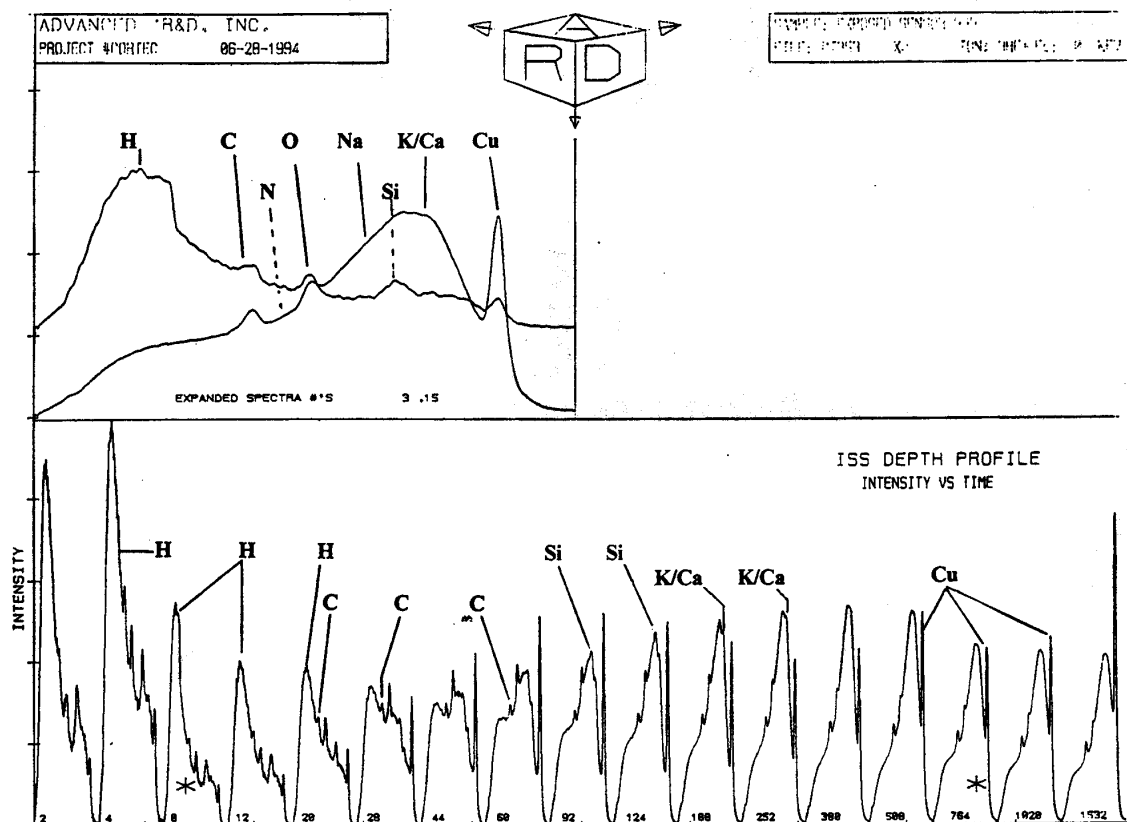


Figure 2. Typical ISS (Ion Scattering Spectroscopy) depth profile of the surface chemistry of a Cu sensor. From the outer atomic layer on the left to about 100Å on the right. Two spectra have been expanded in the upper left corner.

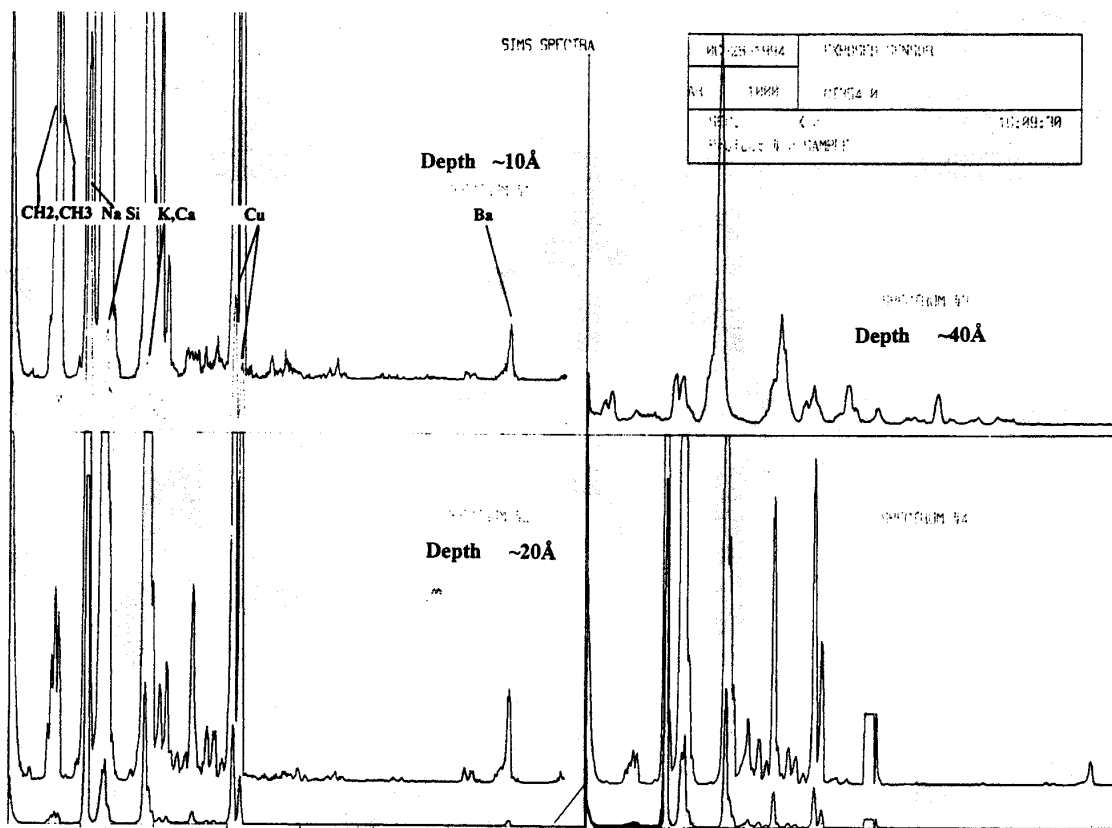


Figure 3. Typical SIMS (Secondary Ion Mass Spectroscopy) of the outer few Angstroms of the surface chemistry of a Cu sensor.