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Corrosion Rate Measurement Technique with Thin Film Electric Resistance Sensor

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There has been growing interest in corrosion monitoring since it is crucial in aging systems in that it enables one to evaluate corrosion risks and predict the service life of construction materials. In this study, we report the application of new ER corrosion probe using thin metal film for corrosion monitoring. The reliability of the new thin film probe was confirmed by comparison with traditional weight loss coupon. The corrosion rate could be determined successfully within a few days of monitoring at various situations.

Keywords : *corrosion monitoring, electric resistance method, thin film sensor*

1. Introduction

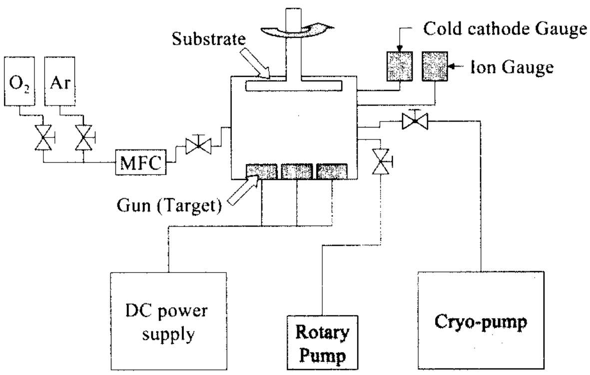
The integrity of the structure material has been major concern of the industry. Corrosion risk and remained service life could not be determined without corrosion rate. Many industries depend on the non-destructive method to estimate the integrity of the structure which could not tell anything about the rate of degradation. To make a proper decision, both qualitative (likeliness of corrosion) and quantitative (corrosion rate) information are requisite. In this study we report the application of new thin film type electric resistance probe for buried pipeline. The measurement of pipe to soil potential has been the only corrosion monitoring activity. The potential could give only the qualitative estimation about corrosion. Sometimes the potential measurement could not give correct information about the pipeline. For example, the corrosion state of the pipeline near electric train railway undergoing interference is hard to be determined from the potential due to the transient nature of the potential.[1),2)] Furthermore the potential itself does not give the answer to the corrosion rate. It only gives qualitative tendency of corrosion. So, many gas transmission companies need an accurate and reliable corrosion monitoring technique. The electric resistance (ER) probes are widely used in other industries to determine the corrosion rate at various environments. In the ER method, one measures the increase in electric resistance of a probe as its cross-sectional area is reduced by corrosion. This method provides the means to automate the corrosion rate and absolute measure of metal loss.[3)] A resistance conductor probe is made of materials of interest and has the shape of wire, strip or tube. One should understand the trade off feature between the probe life and sensitivity. The sensitivity of the ER probe is decided by the thickness of the element: the thicker the probe element the slower the response time. However the application of normal ER type probes for buried pipeline has been limited by their low sensitivity. Denzine and Reading reported that it will take more than 60~70 hours to register a corrosion rate change of 1 mpy with conventional ER probes.[4)]

In this work, we enhanced the sensitivity of the ER probe to very low rate and non-uniform corrosion by using sputtered thin film and multiple narrow patterns. The response of the multi-lined thin film sensor was investigated at various corrosive environments. The detailed behavior of the sensor in various environments was reported and the feasibility of application of the electric resistance (ER) probes for underground pipeline was confirmed by field application. The field application was focused on AC interference which had been hard to estimate with conventional tools.

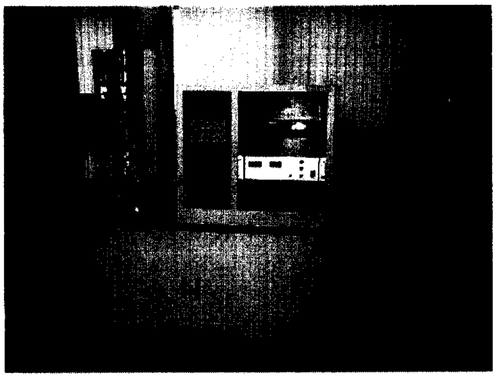
2. Experimental

2.1 Fabrication of thin film corrosion sensor

The thin steel film of 0.6μm thickness was deposited by DC magnetron sputtering of plain carbon steel on the corning 1737F glass substrate and sintered Al_2O_3 plate. The schematic diagram and photograph of the sputtering system was shown in Fig. 1. The resistivity of the film was determined by 4-point method. The film which has

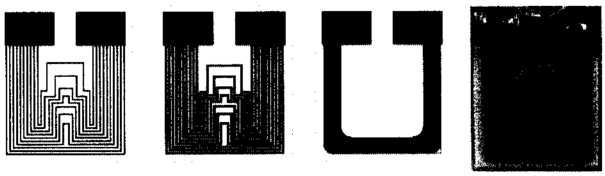


(a) diagram of the sputtering system



(b) photograph of the sputtering system

Fig. 1. The Schematic Diagram and Photograph of the Sputtering System



0.079mm 0.158mm 1.580mm photograph

Fig. 2. The Shape of Thin Film Electic Resistance Sensor

low resistivity ($\leq 5.0 \times 10^{-5}\Omega \cdot cm$) was used for the probe fabrication. The probe with a pattern was prepared by masking and etching in 10% ferric chloride solution by silk screen method. The exposed area of the probe was composed of multiple lines with various width as shown in Fig 2.

2.2 Measurement of corrosion rate

The corrosion rate was determined by two methods. One was direct measurement of the probe resistance. And the other was the measurement of the current through the probe at potential difference of 20 mV across the probe. The average thickness was determined by comparing the measured value to the initial value. The fraction of remained thickness to initial thickness is used to calculate corrosion rate of the probe. No compensation was made for temperature variation.

For the application to corrosion monitoring of cathodically protected steel, the probes were connected to the structure which was protected by independent cathodic protection circuit.

3. Results and discussion

3.1 The corrosion behavior of the deposited thin film

The characteristics of the fabricated probes depend on the quality of the thin film. The control of the incorporated oxygen was important as reported elsewhere.[5] The corrosion behavior of the film with higher oxygen contamination could mislead the test results severely. The property of the film also depends on the surface roughness. Substrates with various roughness were prepared and the film was deposited at same condition. The corrosion potential was measured at the same medium, tap water at 25℃(Fig. 3). The corrosion potential depends on the substrate material and roughness. At the most rough surface of sintered Al_2O_3 plate the corrosion potential was lowest and close to that of the bulk material. When titanium oxide layer of 50nm thickness was used, the corrosion potential was increased more than 200mV. The corrosion potential decrease at rough substrate implied that stress release effect could altered the corrosion behavior of the thin steel film.

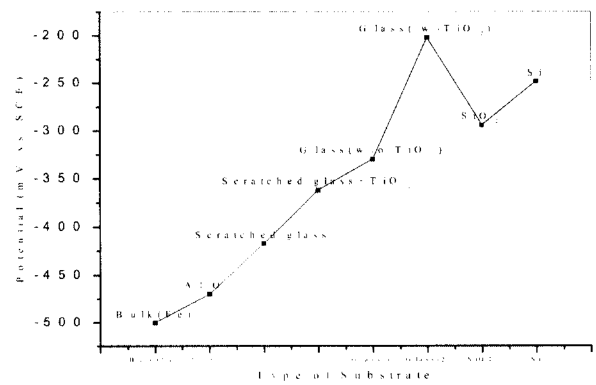


Fig. 3. The corrosion potential measurement at various substrates

3.2 The feasibility test for buried pipe corrosion monitoring

It is well known that the widely accepted criterion for adequate cathodic protection level is -850mV vs. saturated

$Cu/CuSO_4$ reference electrode. However there are many obstacles in determination of the corrosion state of the pipeline. One of the problems lies in transient potential of the pipeline due to interference. As the potential changes with time, it is hard to identify the state of the pipe. Furthermore, the potential itself doesn't tell about the rate of corrosion. The corrosion monitoring system should be sensitive to the protection level and the rate should be determined within reasonable period. Corrosion rate change was monitored at different cathodic protection levels to confirm the possibility of monitoring cathodic protection effectiveness with the thin film probes. The probes were immersed in the NaCl solution of 100 ohm(cm resistivity and the potential have been controlled by the potentiostat with respect to copper/copper sulfate electrode. The results were shown in Fig. 4. At the potential of -850 mV (vs. $Cu/CuSO_4$) most of the initial thickness of the probe maintained after the experiment. Slight decrease had been observed after long exposure. We found that this slight decrease was the result of the corrosion during the measurement. During the measurement the cathodic protection had been interrupted and the accumulated corrosion during measurement decreased the conductance. By altering the measuring interval to several hours, this conductance change had been eliminated. The corrosion rates were increased with the increase of the potential. At the potential of -250mV, which was much higher than natural potential, the corrosion rate was high enough to corrode entire probe within a few minutes. The results showed that the cathodic protection system performance could be determined by the thin film sensor within 12 hours. As the test did not depend on any potential measurement, any kind of interference could be monitored with this sensor. It means that the consequence of stray current from unknown sources could be estimated within reasonable period.

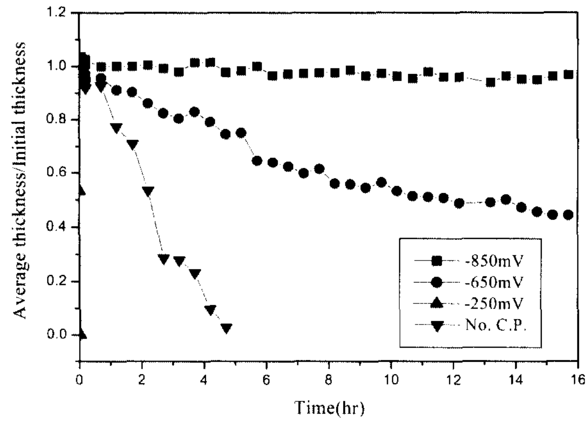


Fig. 4. The corrosion rate change with the cathodic protection potential(solution resistivity: 100 Ωcm, NaCl aqueous solution)

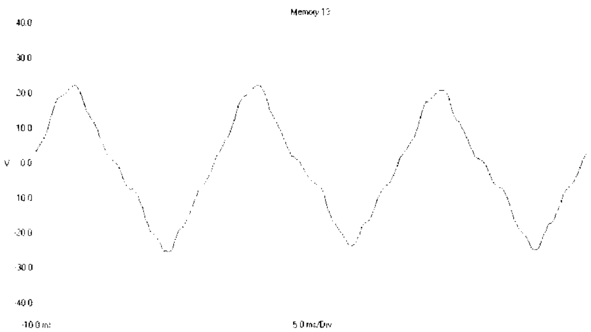


Fig. 5. Typical Pipe-to-Soil Potential Profile(Vertical grid: 10V, Horizontal grid 5msec.)

3.3 Field estimation program

The thin film sensors were installed at 5 sites with metal coupon of 1 cm^2 area for comparison purpose. The sites were selected especially to identify the influence of alternating current interference which were hard to evaluate by routine pipe-to-soil potential measurement. One of the examples of the pipe-to-soil potential taken from a site is shown in Fig. 5. The potential of the pipe was changing with a frequency of 60Hz at a site close to electricity transmission utility. Its average value was around -1V (vs. $Cu/CuSO_4$). We tried to measure the corrosion rate at those situations.

The corrosion rates has been measured with thin film probe and weight loss coupon recovered after 6 months of exposure. The probe station standing near the test box is shown in Fig. 6. The automatic resistance logger was installed in the small box mounted on a small steel pipe(Fig. 7).

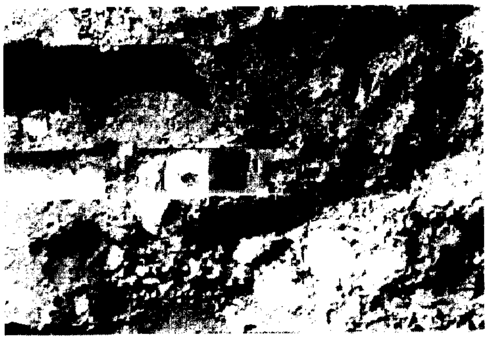
A typical data obtained from the sites was shown in Fig. 8 at a day interval. The corrosion rate could be determined from the following equation.

$$\text{corrosion rate (in mpy)} = \text{slope(1/day)} \times 0.6\text{(micron)} \times 365\text{(day/year)} \times (1/25.4)\text{(mils/micron)}$$

The results were summarized in the Table 1. The current flowing through a coupon of 1 cm^2 area was measured and listed in the table. This value represents the severity of the AC interference. The dominating frequency of the current was also listed in the table.

The correlation of two method was evaluated as shown in Fig. 10. The corrosion rates determined from weight loss coupon was higher than that from the probe. The rate determined from the probe was about 18% of the value from the weigh loss coupon. There could be many reason

(a)



(b)

Fig. 6. Field installation of the resistance probe (a) Small box inclosing a logger was installed near test box, (b) The sensor attached to an end of steel pipe

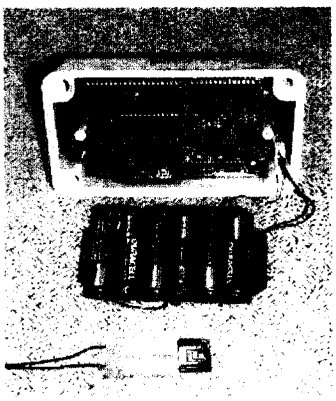


Fig. 7. The automation logging system with a probe

about this discrepancy. One of the cause could be related to the improper insulation of the weight loss coupon. At the edge and corner of the square coupon, the current was concentrated and masking paint layer was disbonded. The weight loss at the disbonded edge was major portion of the weight loss in heavily corroded coupons(Fig. 9) We concluded that the use of probe provide more relevant corrosion rate without artifact from imperfection of the preparation of the coupon.

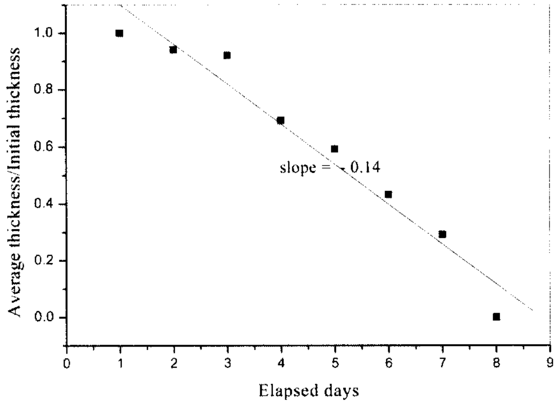


Fig. 8. A Typical Response of the Probe from Program

Table 1. The Summary of the Corrosion Rate from Program

Site number	Corrosion rate by ER (mpy)	Corrosion rate by coupon (mpy)	AC current (mA)	Domination frequency (Hz)
1	1.20	3.3	3.2	180
2	0.17	0.4	2.8	180
3	0.91	5.5	2.5	60
4	0.13	2.5	0.75	60
5	0.27	1.1	1.2	180

Fig. 9. An example of the recovered weight loss coupon

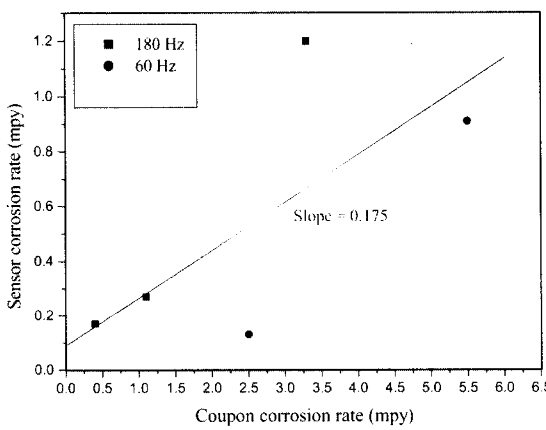


Fig. 10. The corrosion ratio of the sensor corrosion rate to coupon corrosion rate

4. Conclusion

The thin film electric resistance probe was successfully applied for corrosion rate measurement of buried pipeline. The propertiy of the thin film ER probe depends on the corrosion property of the deposited thin film. Precisely controlled process and adequate substrate selection was required to obtain thin film with bulk corrosion properties. With the new design concept and process control thin film type probe could be fabricated. The time required to register corrosion rate of pipeline under interference varied from few minutes to days which was much smaller than conventional weight loss coupon method. The presence of interference and its impact on cathodic protection system could be monitored successfully by installation of the probe connected to the pipeline.

Acknowledgement

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