SERVICE TESTING OF BITUMINOUS COATINGS

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The purpose of any protective coating is to protect the base material from the environment which surrounds it. It follows, therefore, that the protective coating material must in itself have great resistance to the environment to which it is exposed. If the coating does not provide this protection then it has failed in its purpose.

Of necessity, cost is a factor in the selection of a protective coating. It is important, therefore, that the coating exhibit properties which when considered with the type of structure protected, the environment and the cost, will provide an economically sound system. Unfortunately, no one has yet developed a protective coating system which is reasonably priced and fully effective in all applications. Each exposure condition has its own unique characteristics which will take its toll of a coating system. Service testing, which implies testing under actual service conditions, will answer the question: How well is the coating system doing its job?

When considering a protective coating for application to a new facility, two things may be done. First, test panels coated with various systems under consideration may be exposed to environmental conditions considered to be typical of those to be encountered. Short term tests of this type provide valuable screening information and will generally eliminate from further consideration those protective coating systems which are totally unsuited for the job. Second, valuable information may be obtained from operating equipment where experience under actual service conditions may be available. The more complete the history of an existing coating system under operating conditions, the greater the value that can be placed on the conclusions drawn from this experience.

The basis for evaluating protective coatings rests heavily on the properties which the coatings exhibit. It would be well to review briefly some of the particular requirements and more important characteristic properties which coatings must have for exposure to general classes of environment.

In atmospheric service, a protective coating must have good adhesion or bond to the base material, resist actinic effects, rain, wind and dust, and must withstand a wide range of operating temperatures and the associated thermal shock. Also, the coating must be able to resist chemical attack from

the oxygen in the air and such compounds as may be present from a multitude of industrial sources as atmospheric contaminants.

Protective coatings when applied to process equipment may be subject to all the rigors of atmospheric exposure, plus additional specific conditions characteristic only of the process itself, or it may involve exposure to a very specific environment and set of operating conditions. In addition to good bond, the coating system must frequently resist abrasive effects and mechanical action. If the process involves a chemically specific environment, the coating must resist chemical attack and solvent action within economic limits and technical feasibility. In process equipment use of the protective coating may well be subject to extremely wide ranges of temperature.

In total and partial immersion service, such as wharf pilings, water tank interiors, dam gates and the like, a protective coating system is subject to a new set of environmental factors which will have a definite bearing on the serviceability of the coating system. It must have good bond to the base material, but also must have excellent mechanical strength to withstand floating debris, ice and other impact effects. Failure of the coating through cracks or chipping will frequently result in serious corrosion where the base metal is exposed. The coating material itself must be highly resistant to moisture absorption and it should be a nonconductor of electricity. For service where immersion in aqueous environments is involved the coating should be compatible with cathodic protection, i.e., it should resist highly alkaline conditions at the metal surface where cathodic currents enter protective coating faults. Finally, the physical properties should resist a relatively wide range of operating temperatures.

Coatings for service underground are subject to a wide range of variables. Excellent bond to the base material is essential since moisture penetration to the layers between the coating and the base metal can result in aggressive corrosion which may not be controllable with cathodic protection[1]. Mechanically, the coating system must have excellent strength to resist soil movement and stress. As in the case of total immersion service, the coating should resist moisture absorption and have good electrical insulating properties even after long periods of exposure since galvanic currents and in some cases stray electric currents from manmade sources can be aggressively corrosive to the base metal if they pass

through the coating. The coating should be compatible with cathodic protection. For some kinds of service a chemical corrosion inhibitor may be included to assist in controlling local galvanic corrosion. Resistance to microbiological attack is a must since all soils contain many such organisms[2][3]. Depending upon the specific application, the coating may have to resist a wide range of temperature effects although generally not so severe as atmospheric or process equipment conditions.

With these basic properties and the requirements in mind, it should be evident that service testing is about the only all inclusive way to evaluate the merits and limitations of protective coatings.

We have already noted that preliminary screening tests using panels or test coupons are most useful in eliminating totally ineffective materials from consideration on a specific application. Such tests generally require but a short time for completion and provide many practical advantages such as completeness of examination and accuracy of control. Unfortunately, such tests can not provide the complete story nor take into account all of the many variables associated with the final conditions to which the coating is to be subjected.

Service tests generally fall into two categories. The first is the direct tests which may be conducted where it is possible to physically examine the coating system under actual conditions of exposure. This would include coatings on structural members exposed to the atmosphere, on process equipment where it is possible during shut-down periods to directly examine the condition of the coating and, to a limited extent, on underground structures such as pipelines where it is possible to excavate the structure. The second type of test is that conducted using indirect methods to reach conclusions on coating effectiveness. Such tests are performed where it is not possible or economical to examine the coating by direct physical means.

It is not uncommon to set up comparative tests for evaluation of a number of different coating systems for a particular application. This will usually occur at a time when the original protective coating has shown signs of deterioration and it is evident that a complete recoating job will be required in the near future. Test areas selected to cover as many of the variables of exposure as possible are prepared and coated under the close supervision

of either the manufacturer or the plant engineer. The coatings selected for service tests are generally those which have, through experience, given the best service, or are the coatings which preliminary exposure tests have indicated should be most suitable for the specific job. The condition and changes in physical properties of the various coatings are then followed on a regular basis during the period of evaluation, and the final coating selected on the basis of the results.

In carrying out tests on protective coatings on equipment which is in service, it must be remembered that rigorously controlled conditions of the laboratory are not present, and it is most important that comparative data be obtained for all systems under test. It is also essential that excellent records be kept for future reference and comparison. Wherever possible inspection should be done on a statistical basis. This is particularly important in the case of extensive structures where considerable variations may occur from one point to another due to prevailing wind conditions, local environmental factors and temperature differences. It is for this reason that most in-service exposure tests for comparison of different coating systems will involve scattered test sites at quite a number of different locations rather than in one area.

The tests which may be performed on structures where direct access to the coating is possible are generally modifications of standard tests used in exposure testing. Appearance alone may represent a relatively significant comparative test where several coatings are involved; however, caution must be exercised to see that rust stains or other apparent areas of coating failure are truly the result of coating deterioration and are not merely rust stains originating from other areas.

Since bond to the base metal is a particularly important characteristic, some sort of bond or adhesion test should be employed when comparing protective coatings in service. A common test is to scribe two parallel lines 3/4-inch apart and 4 inches long through the protective coating to the base material. A square-ended putty knife of 3/4-inch width is then forced under the coating for approximately 1/2-inch. An attempt is then made to lift the coating from the surface without fracturing the coating film. If the coating pulls away from the base material this is indicative of relatively poor bond. Details of this type of test are described in the American Water Works Association Standard, C-203-62, Section 2.4[4]. On extremely heavy coatings and mastics, lack of bond may be detected by tapping areas of the coated surface

with the handle of a screwdriver or similar implement. Large areas of disbonded coating will have a distinct hollow sound to them. Puncturing of the coating at locations where such sounds are detected will clearly show the area of lack of bond. Blisters in a coating are another method of detecting lack of adhesion or bond between the coating and base material or between layers of the protective coating. For comparative purposes, it is desirable to record the relative size of such blisters and the number of blisters per unit area of inspection.

The presence of moisture at the metal surface is also evidence that bond is not adequate. Such moisture may be present even though no obvious path of entry can be found; this may be the result of penetration of moisture through coating pores with subsequent disbonding. Other evidence of poor bond may be undercutting, flaking or the lifting of a coating from the metal surface over appreciable areas.

If a particular type of service is subject to impact, the resistance of the coating to this type of action must be evaluated. Standard impact tests have been developed for certain specific applications on a controlled basis(4). It is possible, however, to perform a modified impact test using a steel ball dropped from a specific height on a horizontal surface, or swung, pendulum fashion, against vertical surfaces. The weight of the ball and distance of drop will vary depending upon the specific coating under test. The principle behind such tests is that shock on the metal surface will disbond a protective coating a certain distance from the point of impact. By peeling away the disbonded coating around the impact point, a comparison among various coatings is possible using the area of disbonding as the index. Since temperature and other factors have a critical influence on this property, care must be taken to test the different coating systems under comparable conditions.

Pinholes in a protective coating may be found under some conditions by electrical inspection with a protective coating holiday detector. Care must be taken to see that surface moisture on the coating material is not responsible for indications of coating faults. Where there is doubt, a "patch" test may be employed to locate and evaluate this type of defect(5). Fig. 1 shows diagramatically how this test is performed. The coating is first covered with a thin slurry of kaolin or other iron-free clay; the purpose of which is to fill in pinholes and other holidays in the protective coating and provide an electrically conductive path which will retain

moisture. Over the kaolin is placed a layer of absorbent paper, such as paper towelling, followed by a water soaked layer of cotton flannel. A cathode consisting of an aluminum or copper plate is then held firmly against the area of test and a battery, capable of supplying 60 to 100 volts potential, is connected between the base metal and the metal cathode with the polarity as indicated. If coating holidays are present, a current will flow. Since the base metal is the anode, electrolysis will occur and metal ions will migrate into the absorbent paper. The presence of these ions may be detected with a chemical indicator such as potassium ferricyanide where steel is the base material. The number of pinholes can then be counted and recorded per unit area as an index of porosity. For heavy enamel coatings of the coal-tar and asphalt type, it is usual to apply direct current for a period of time which may be calculated in minutes by dividing 100 by the applied voltage.

Where direct examination of the coating is possible, tests may be performed on such things as moisture absorption by removing samples of the coating and subjecting them to laboratory tests. A common test for moisture in bituminous coatings is A. S. T. M. D95[6]. Other effects which should be recorded when a direct examination is made are chalking, alligatoring, softening or embrittlement, color change or other evidence of decomposition of outer layers of the coating.

Where the coating is applied over iron or steel, it is possible to measure coating thickness with electronic instruments which are non-destructive and quite accurate. Variations in thickness are indications of cold flow and can be checked by this type of instrumentation. A thickness-time relationship can then be established over a period of inspections.

Other visual evidence of coating deterioration may be the presence of imbedded foreign material in the coating. This may be of particular concern on structures such as pipelines which have been exposed to underground conditions prior to examination.

Another test which may be indicative of coating quality, is an electrical conductance test. The specific application of this type of test to a coated pipeline is shown in Fig. 2. In general conductance is most important on structures which are normally immersed in an aqueous environment or which are buried in soil. It is, therefore, a test which requires considerably more preparation for inspection than most other tests where direct examination is involved.

As with the electrical "patch" test for coating faults, use is made of electrical instruments and techniques. Referring to Fig. 2, a section of the coating is selected for examination and the pipe is thoroughly cleaned with wet rags to remove adhering soil and foreign matter. As with the "patch" test, the surface is wiped with a slurry of kaolin to retain moisture and provide uniform contact between the coating surface and the cathode. A pad of wet cotton flannel or paper towelling is generally wrapped around the pipe between the coating and the cathode to assure a uniform electrical contact. The area of the test is controlled by the physical size of the pad. Two guard rings are employed on either side of the cathode to prevent electrical leakage current from reaching the cathode from the surrounding environment thus creating an error in the measurement. The coating conductance is calculated by dividing the current collected from the cathode by the voltage applied between the cathode and the pipe surface. Since the guard rings are outside the current measuring network, leakage current from the earth along the coating surface is eliminated. If an appreciable voltage drop occurs across the milliammeter, a correction must be made in the final calculation.

Exposure to an earth environment may result in degrading effects due to soil bacteria. This effect has been the subject of research in recent years and has been reported in many papers (1)(2)(3)(7). Removal of samples for laboratory evaluation appears to be the only practical test for bacterial attack on protective coatings.

To summarize the direct examination approach to service testing: Standard types of tests or their modification may be used for exposure evaluation of coating materials recognizing that the conditions of the tests are subject to many variables not present in a laboratory examination. A complete set of records of the conditions existing during the testing and the properties of the coating system as observed both initially and through all stages of the tests are a must. Tests of this type should be performed on a periodic basis so that a running record will be available. Photographs showing conditions described in the reports are extremely helpful in building a sound record which may be relied on for future reference.

Service testing of structures such as pipelines and other underground installations by indirect means is quite important to certain industries. The most useful tests which fall in this category are ones that can be performed at regular intervals so that a history of the coating system will evolve. Since great quantities of bituminous base protective

coatings are employed in pipeline work, much of the following discussion will relate to this type of structure. It should, however, be remembered that modifications of the techniques described may be employed for other types of exposure, such as pilings, bulkheading, underground storage tanks and similar structures.

While the majority of measurements employed for the indirect evaluation of protective coatings are electrical, one specific test involves a physical type of measurement. This is the evaluation of water-line friction coefficients by the Hazen and Williams formula. Mathematically, the formula for water flow is expressed as

$$v = 1.318\ C\ r^{0.63}\ s^{0.54}$$

where: v = velocity in feet percent

r = the hydraulic radius in feet

s = the hydraulic slope in feet per foot

C = the Hazen and Williams coefficient

Comparisons obtained with various coating systems in pipelines are based on the "C" factor. A good "C" factor generally indicates an effective protective coating in a water main, failure of the coating will be reflected in deterioration of this factor with time. An interesting report covering results of this type of work with various lining and coating materials has been compiled by the New England Water Works Association(8). Where fluids other than water are involved, various friction factor equations are available which are useful for evaluation purposes.

The underground pipeline lends itself particularly well to indirect measurement of exterior protective coating qualities. It is a relatively easy matter to locate major protective coating faults on such a system. This is done by the Pearson electronic holiday detector method illustrated in Fig. 3. An alternating current signal is applied between the pipeline under test and a relatively remote earth electrode or ground bed. The alternating current signal, usually about 1,000 cycles per second, uses the earth path in flowing from the ground bed to the structure. If there are protective coating faults present, the signal current will concentrate at these locations. This produces appreciable voltage gradients in the earth. These voltage gradients at the point of coating holidays are detected by an electronic amplifier connected to metal cleats worn by two people making the survey. Generally the spacing between the individuals in the survey party is about 50 feet. The tone produced

by the voltage gradient in the earth around the holiday is detected and reaches a maximum when one of the operators of the detecting equipment is directly over the coating fault while the other operator is 50 feet away over an area of sound coating. Using this method it is then possible to locate and then excavate the pipeline to determine the reason for the indicated unusual flow of current through the protective coating. Where damage or coating deterioration is not obvious, it may be desirable to perform the "patch" and electrical conductance tests already discussed.

It is also possible on a long pipeline to measure quite accurately the average coating conductance at various points along the line. In this way it is possible to evaluate several different coating systems on a single pipeline provided adequate test facilities are installed. The method consists of establishing millivolt test stations on the pipe which are used to measure the flow of current. Fig. 4 shows a typical arrangement of millivolt test stations used for this purpose. These test stations use the pipe as an electrical shunt to indicate current flow. They are calibrated by inserting a voltmeter, "E", between the two inner leads of the test station and applying a known direct current, "A", to the outer leads. A factor for the test station is then obtained by dividing the number of amperes used by the number of millivolts deflection. The factor is expressed in amperes per millivolt.

By installing such test stations at various points along the pipeline or between sections using different coating systems, it is possible to accurately measure differences in current at successive locations along the line. Thus, if a direct current "I" is applied between the pipeline and a relatively remote ground bed, the relative amounts of current picked up along the structure in different areas can be determined. With known quantities of current picked up within sections, it is necessary only to determine the average electrical driving force or voltage responsible for forcing the current from the earth through the coating to the pipe. This is done by measuring the voltage change "ΔV" produced between the pipe and an earth reference produced by current "I". Calculation of the average conductance for the section is obtained as indicated in Fig. 4. The results are expressed in mhos per square foot of coating. Details of this type of test are given in National Association of Corrosion Engineers Technical Committee Report T-3D[9].

An example of the value of coating conductance as a tool in evaluating protective coatings is given by the graph Fig. 5. This figure shows change in electrical conductance

with time on a section of coated pipeline in service and in this case represents an unusually rapid deterioration for one type of protective coating.

The graph in Fig. 6 shows the same kind of data plotted on semi-logarithmic paper; in this case the coating conductance in a rubble filled area is compared with that in other areas on the same pipeline. The data indicates that even after repair of coating faults in the rubble area, coating conductivity continues to rise at a higher rate than that in areas of normal fill. Long term data of this type is of great value to the corrosion engineer in selecting protective coatings for various applications in underground work.

As noted earlier, similar methods can be employed on structures other than long pipelines provided means are available for measuring the actual flow of current in and out of a given part of the structure in contact with the earth or aqueous environment.

Since the primary purpose of protective coatings is to control corrosion, one method of evaluation is to determine the rate of corrosion on a given structure coated according to a set of specifications. Polarization measurements are one approach to determining such rates of corrosion on buried structures. Methods are described in work done by the National Bureau of Standards(5)(10) and by Dr. Milton Stern(11). The value of corrosion rate data in evaluating protective coating conditions rests in establishing a corrosion rate time relationship. Tests must be performed in an identical fashion at suitable intervals so that comparison is possible.

A modification of corrosion rate data for evaluation of coating condition would be the use of minimum current required for cathodic protection on a given system. Cathodic protection requires that a structure be polarized to a predetermined level. The amount of current required to maintain this polarization over a period of time can be used as an index to the continued effectiveness of the coating. Increasing current requirements to maintain polarization is indicative of deterioration of the coating.

The electrical methods reviewed will indicate something about the effectiveness of the protective coating system; it will not, however, reveal what causes changes in characteristics. To obtain complete data requires some form of direct examination. Unfortunately, most of the tests described for indirect measurement are not of the type that can be readily

formulated into standard procedures. There are entirely too many variables associated with the types of structures involved and local conditions which may significantly affect the accuracy of the tests; a great deal of reliance must be placed on the experience of the engineer in carrying out the tests and in his ability to interpret the results.

Perhaps in time, improvements on techniques and other methods of testing will be developed so that specific problems of indirect in-service testing of coatings will be possible with results that can be compared to the direct access methods.

REFERENCES

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(2) Harris, J. O., "Microbiological Studies Reveal Significant Factors in Oil and Gas Pipeline Back-Filled Ditches", Kansas State University Technical Bulletin 135, December, 1963.

(3) Harris, J. O., "Microbiological Studies Reveal Significant Factors in Oil and Gas Pipeline Back-Filled Ditches", Kansas State University Technical Bulletin 102, May 20, 1959.

(4) American Water Works Association, "A.W.W.A. Standard for Coal Tar Enamel Protective Coatings for Steel Water Pipes", C203-62.

(5) National Bureau of Standards, "Underground Corrosion", Circular 579.

(6) American Society for Testing Materials, "Test for Water in Bituminous Materials", ASTM D-95.

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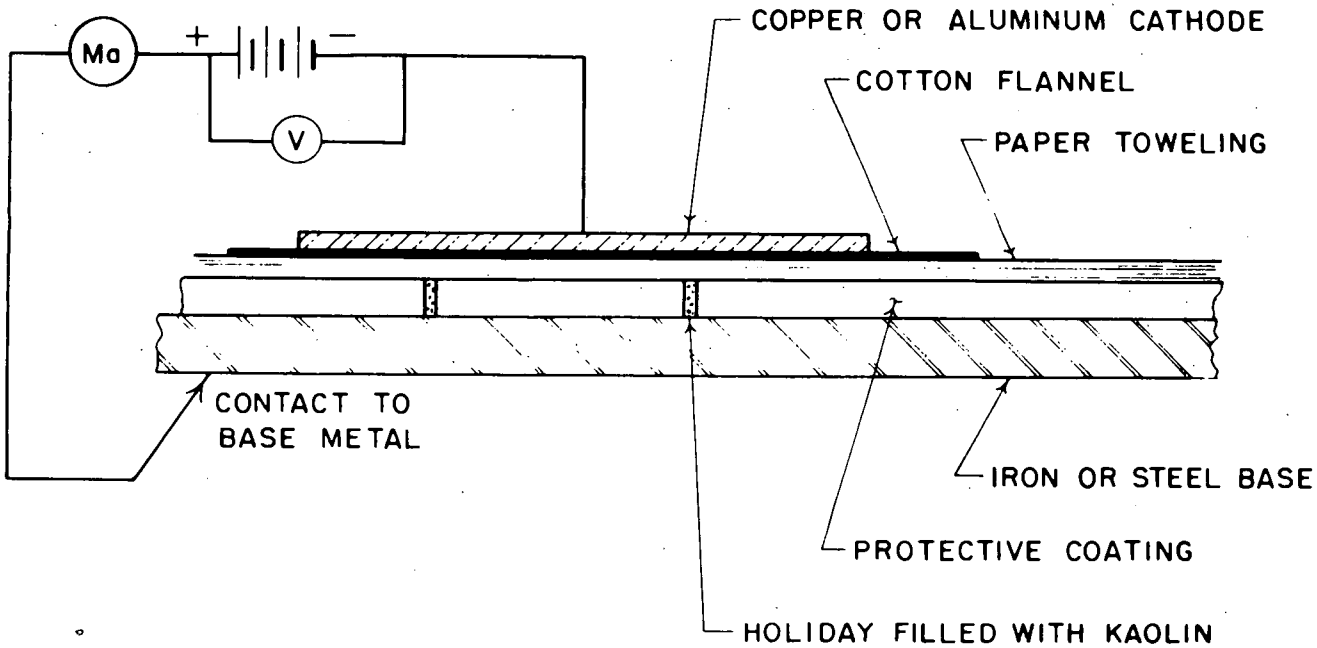
(9) National Association of Corrosion Engineers Technical Committee T-3D Report, "Methods for Measuring Leakage Conductance of Coating on Buried or Submerged Pipe Lines". "Corrosion" Vol. 13, 1957, pp 803t - 805t.

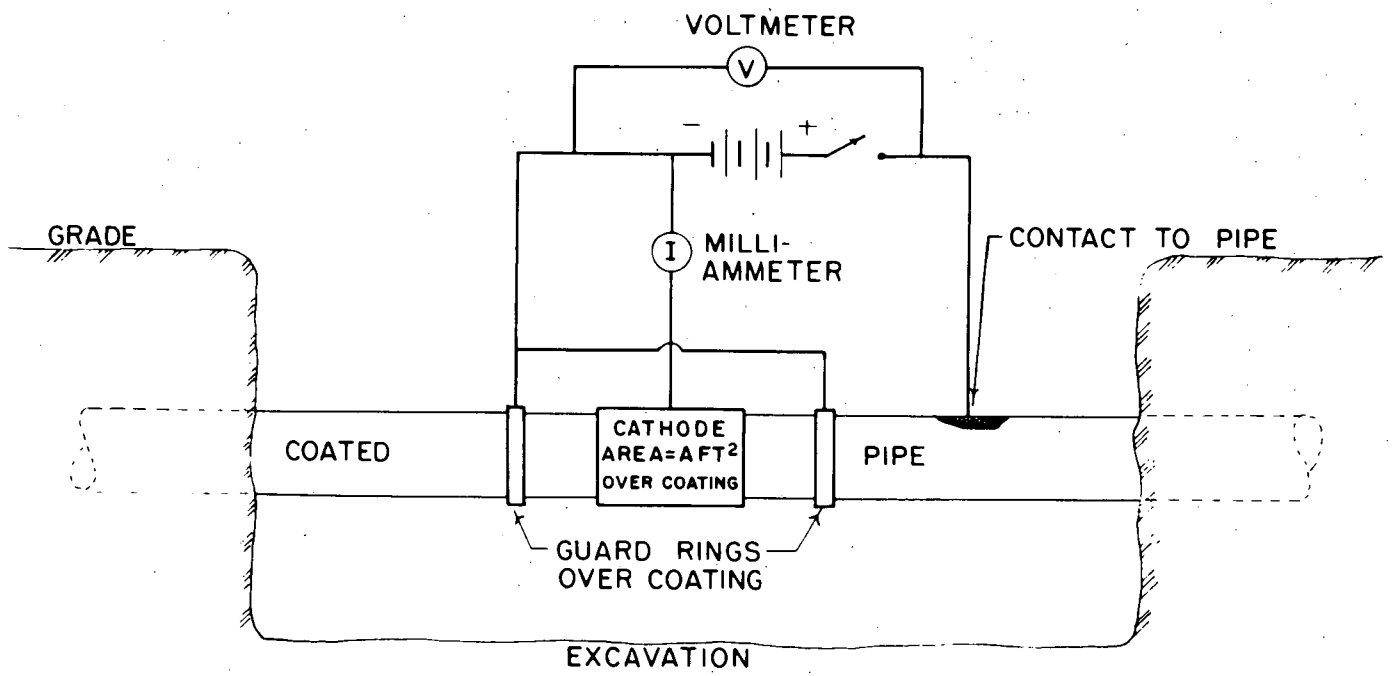
(10) Schwertfeger, W. J., "Corrosion Rates of Ferrous Alloys (Fe - Cr and Fe - Cr - Si) Measured by Polarization Techniques". "Corrosion" Vol. 19, 1963, pp 17t - 25t.

(11) Stern, Milton, "A Method for Determining Corrosion Rates from Linear Polarization Data". "Corrosion" Vol. 14, 1958, pp 440t - 444t.



PATCH TEST FOR COATING FAULTS

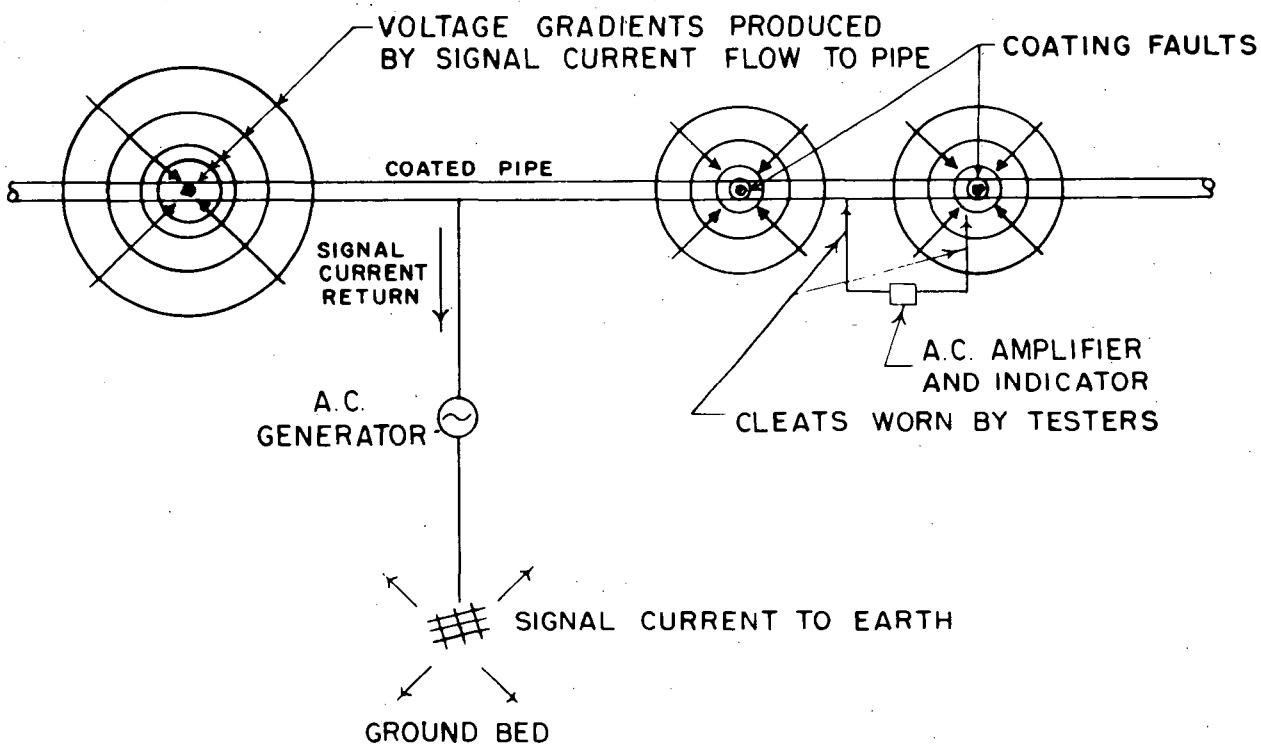
FIGURE 1



$$\text{COATING CONDUCTANCE} = \frac{\Delta I}{\Delta V} \times \frac{1}{A} \text{ mhos/FT}^2$$

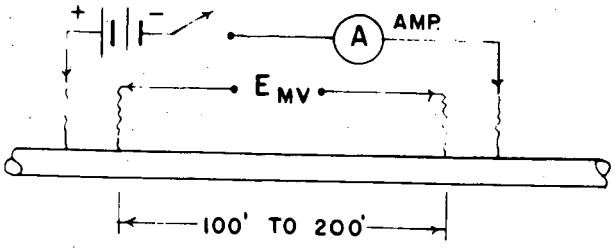
CONDUCTANCE MEASUREMENTS

FIGURE 2



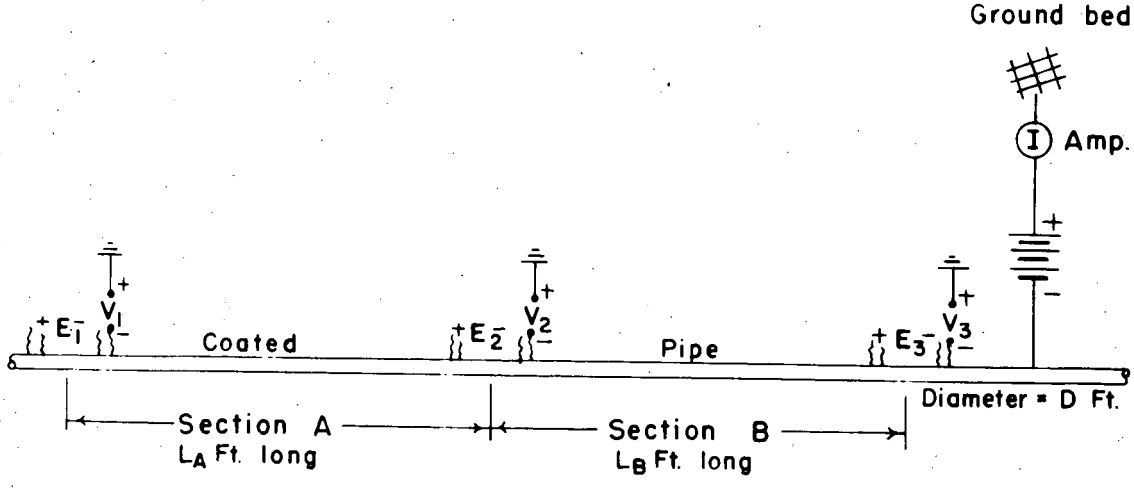
PEARSON COATING FAULT DETECTOR

FIGURE 3



$$\text{Factor} = \frac{\Delta A}{\Delta E} \text{ AMPS/ M.V.} = K$$

CALIBRATION SET UP



$$\text{Average Conductance, Section A} = \frac{\Delta E_2 \times K_2 - \Delta E_1 \times K_1}{\Delta I} \times \frac{2\Delta I}{\Delta V_1 + \Delta V_2} \times \frac{1}{\pi \times D \times L_A}$$

$$= \frac{\text{mhos}}{\text{FT}^2}$$

FIGURE 4

FIGURE 5

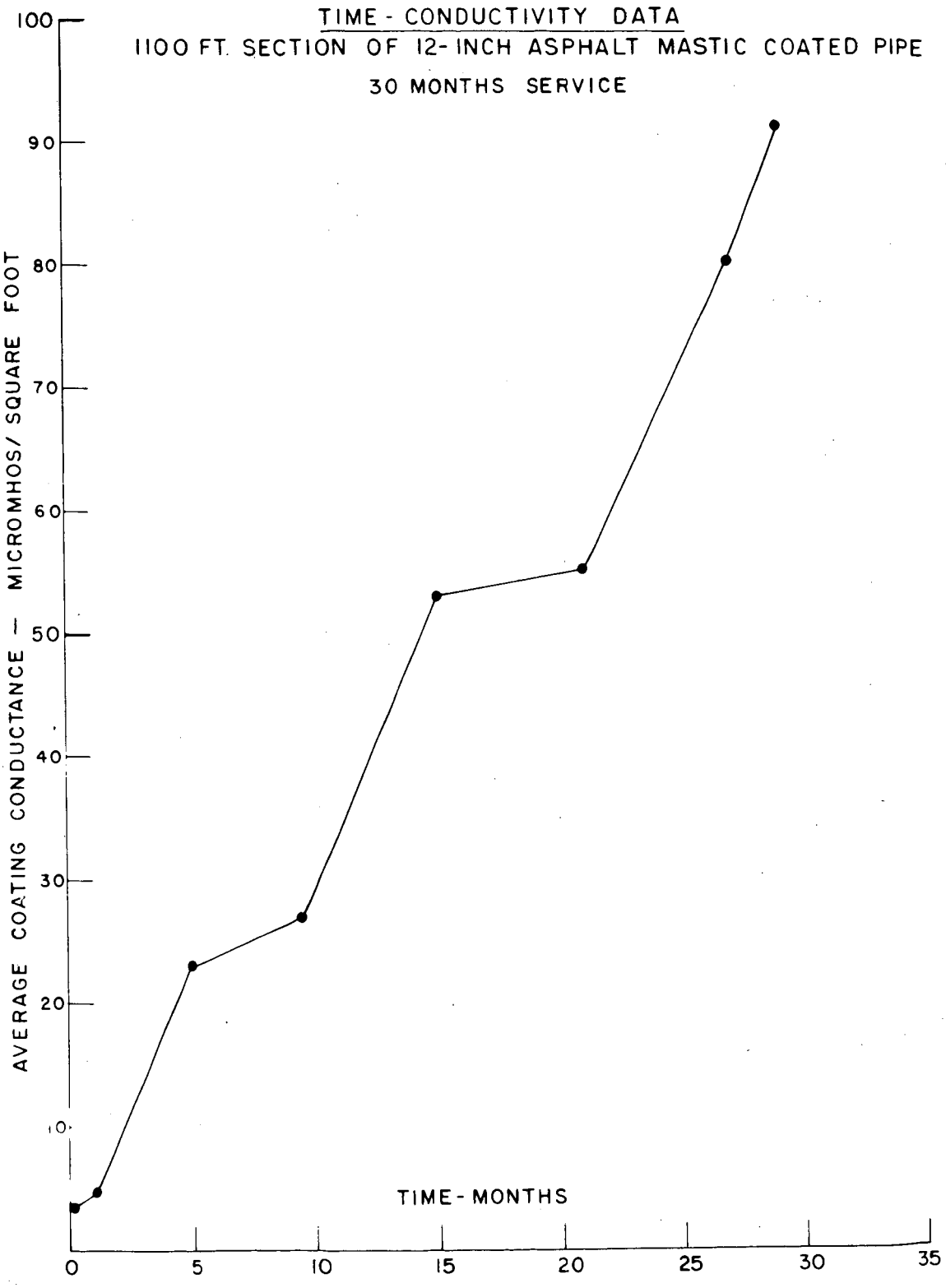
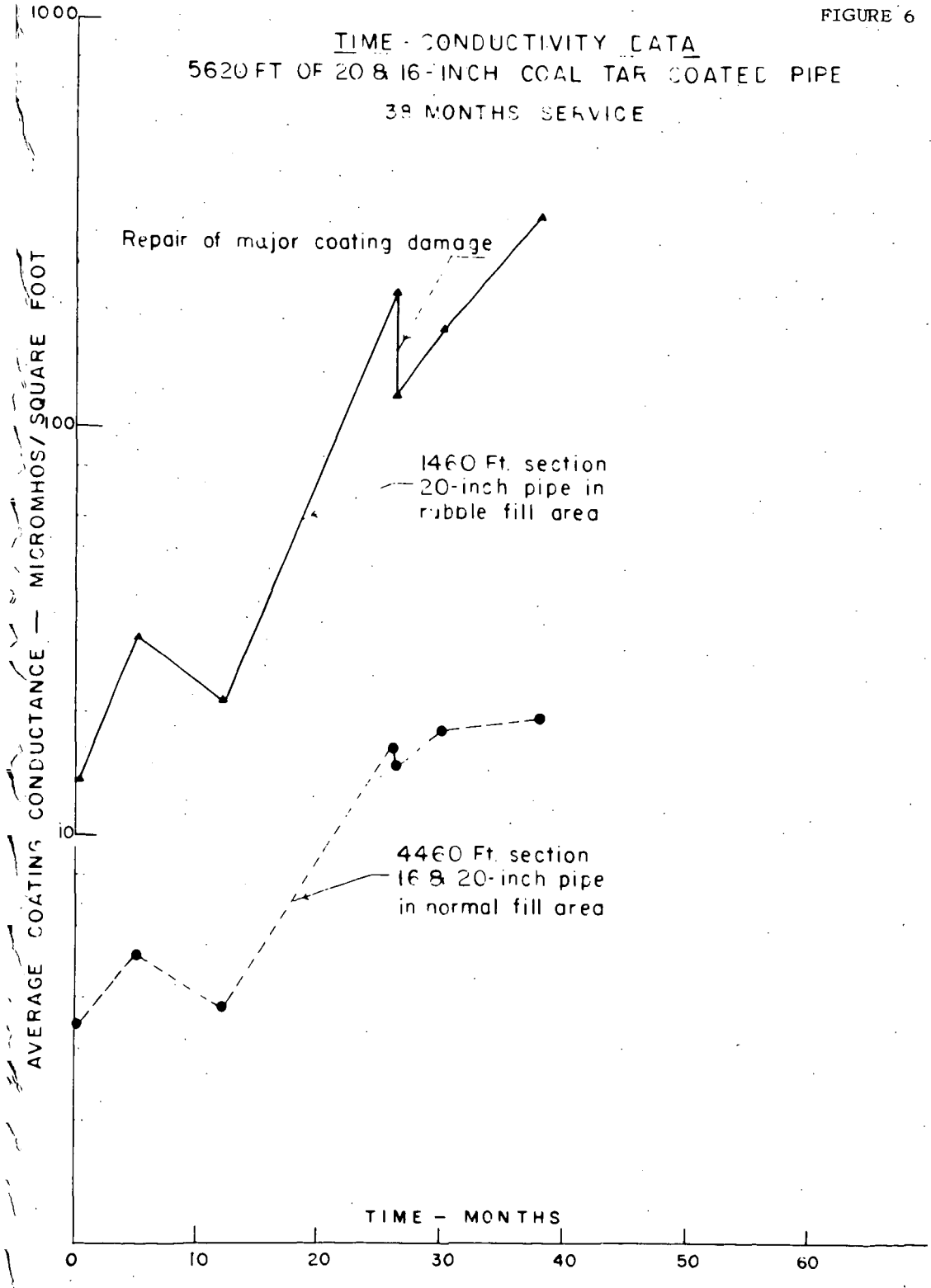

TIME - CONDUCTIVITY DATA
1100 FT. SECTION OF 12-INCH ASPHALT MASTIC COATED PIPE
30 MONTHS SERVICE
AVERAGE COATING CONDUCTANCE - MICROMHOS/ SQUARE FOOT
100
90
80
70
60
50
40
30
20
10
TIME - MONTHS
0
5
10
15
20
25
30
35

FIGURE 6


TIME - CONDUCTIVITY DATA
5620 FT OF 20 & 16-INCH COAL TAR COATED PIPE
38 MONTHS SERVICE
Repair of major coating damage
1460 Ft. section
20-inch pipe in
rubble fill area
4460 Ft. section
16 & 20-inch pipe
in normal fill area
AVERAGE COATING CONDUCTANCE — MICROMHOS/SQUARE FOOT
1000
100
10
TIME — MONTHS
0
10
20
30
40
50
60