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In-Situ Measurements of Alloy Oxidation/Corrosion/Erosion Using a Video Camera and Proximity Sensor With Microcomputer Control

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IN-SITU MEASUREMENTS OF ALLOY OXIDATION/CORROSION/EROSION USING A VIDEO CAMERA AND PROXIMITY SENSOR WITH MICROCOMPUTER CONTROL

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SUMMARY

Two noncontacting and nondestructive, remotely controlled methods of measuring the progress of oxidation/corrosion/errosion of metal alloys, exposed to flame test conditions, are described. The external diameter of a sample under test in a flame was measured by a video camera width measurement system. An eddy current proximity probe system, for measurements outside of the flame, was also developed and tested. The two techniques were applied to the measurement of the oxidation of 304 stainless steel at 910° C using a Mach 0.3 flame. The eddy current probe system yielded a recession rate of 0.41 mils diameter loss per hour and the video system gave 0.27.

INTRODUCTION

The development of alloys for high temperature use in severe environments involves testing of different compositions, preferably under conditions simi- liar to the intended application (refs. 1 to 10). Often the alloys are exposed to a flame which may contain various contaminents. After some period of exposure the change in weight, dimension or some other parameter is measured in an effort to determine the degree of resistance to attack. Some of these measurements may not be valid indicators of metal consumption and most are destructive of the specimen and are therefore performed only at the end of testing. These measurements produce only comparative data and to obtain kinetic information numerous specimens must be tested for various times. Such measurement procedures often contribute to uncertainties in the data because of the many samples involved. In addition, these procedures are expensive in terms of both specimens and manpower.

Obviously, it would be advantageous to be able to make extent of attack measurements in a continuous, in-situ and nondestructive manner.

Two noncontact and nondestructive measurement schemes were investigated and are described here. The first method makes use of a video camera which has the ability to measure the external width of an object while it is in the test flame. This is truly an in-situ technique. The second method makes use of an eddy current sensor which senses the distance from a metallic sample, but only when the test flame is removed. The feasibility of both techniques has been tested by making measurements of the oxidation of 304 stainless steel subjected to a Mach 0.3 burner rig flame. The preliminary results reported here establish that the techniques hold considerable promise for errosion and oxidation testing. Further testing is needed to define the applicability of the techniques to high temperature corrosion measurement.

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MEASUREMENT SYSTEMS

Two in-situ, noncontacting, measurement attack techniques were investigated. One technique makes use of a video camera which has the ability to measure the width of an object by optical contrast differences. This technique can be used to make measurements of a sample under test while it is actually in the flame producing the test conditions. For this width measurement technique resolution is theoretically infinite but in practice it proved to be about ±0.5 mils. The camera measurement is an external dimension determination and thus includes any nonspalled scale or corrosion product that might form on the sample being tested and measured. Measurements have been made in an automated manner only at one position on the sample, and thus at only one temperature, because the contrast threshold was adjusted manually. For the selected contrast setting, continuous width measurements can be made and the results recorded.

The second technique investigated uses an eddy current proximity probe, this sensor measures the distance from itself to a metal object under consideration. In an oxidation/corrosion/errosion context, any increase in this distance, if the probes position is fixed, indicates a recession of the metal surface. Further, if the specimen is a cylinder rotating on its own long axis then the measured change in distance to the metal can be interpreted as a change in the diameter of the metal. In theory the probe used has infinite resolution but in practice it was found to be about ±0.5 mils. The probe must be used at or near room temperature. Therefore, in order to make use of this probe in measuring oxidation/corrosion attack a means had to be devised for removing it from the flame environment during the heating portion of a test cycle. The probe must then be repositioned for each subsequent measurement, to the initial position with a precision of ±0.5 mils or less. Movement of the probe was accomplished with a commercially available stepper motor driven slide mechanism capable of 0.125 mils of movement per step. Return of the probe to its initial position was achieved by use of a second proximity sensor. The metal recession probe must be calibrated to establish, the relation between distance and output voltage. Because this is a function of the surface composition of the metal which may change during oxidation/corrosion testing, the calibration factor must be determined before each distance measurement is made. This was accomplished by making use of the slide's stepper motor which moves the probe a know distance per step. The change in the value of this calibration factor with the progress of the corrosion testing could possibly give some pertinent qualitative information about the change in the surface composition but this aspect was not pursued here. All the functions of slide movement control and measurement were carried out by instructions from a programmable microcomputer.

Several additional refinements were necessary to improve the quality of the measurements, reduced scatter was achieved if at each proximity probe hot section test measurement a reference measurement was also made on a part of the specimen that had not been if the flame. The hot section test distance was then the difference between the reference distance and the hot section distance. In order to implement the reference measurement a vertical movement of the probe was required and this was achieved by using a second slide mounted vertically. Measurement precision was also improved by using a synchronus measurement method. This was achieved by installing a flag on the shaft producing specimen rotation. On each shaft revolution the flag interrupted a light beam and a pulse was sent to the data acquisition system. A proximity reading was made only upon receipt of this pulse. In this way the readings were always made at the same location on the specimen.

Figure 1 is an overall schematic diagram of the measuring systems. Discussion of the function and calibration of each part of the system will be given.

Temperature Measurement and Calibration

In any high temperature environmental testing scheme temperature measurement is a most important factor. This measurement is difficult and in flame systems it presents additional challenges. Figure 2 is a schematic diagram of the setup used in the present study to measure sample temperature and calibrate the measuring devices. A type K thermocouple located in the well in the center of the sample and a two-color optical pyrometer with a 2.5 mm diameter target were calibrated against a type R-thermocouple located about 5 mils under the surface of a 304 stainless steel calibration specimen's surface. The temperature of the R-couple is taken as the surface temperature. The type K working couple and the type R-couple were located adjacent to each other in the same plane perpendicular to the vertical axis of the calibration specimen. The two color pyrometer was aimed the specimen surface directly at the location of the R-couple. It was found that the temperature of the oxidized surface indicated by the two color pyrometer was ±2° C of that indicated by the type R-couple. The type K working couple, located in the center of the half inch diameter specimen, yielded a temperature about 10 to 15° C lower than that given by the type R-couple. For the type K working couple and the two-color optical pyrometer an algorithm was derived and made a part of the software used to convert EMF measurements to temperature. The two color pyrometer was used to monitor the specimens surface temperature above 800° C and the type K working couple monitored the specimen temperature during cooling. This was necessary because the two color pyrometer only measures temperatures above 800° C. The type R-couple was used only for calibration purposes because routine test samples did not have a provision for this couple.

Proximity Probe and Slider System

The proximity probe is an eddy current type which detects the presence of a metallic target and responds by outputting a voltage signal proportional to the distance from the probe to the target. This signal is linear with distance over the detection range for the particular probe used. The probe also responds to changes in metal composition and shape of the target. The probe used here has theoretically infinite resolution and a sensitivity of about a hundred mV per mil of distance depending on the target material and shape. Its maximum detection range is 100 mils of separation from the target. The voltage measurement system used to monitor the probe output has a resolution of 0.1 mV and this thus sets the resolution of the distance measurement system at 0.001 mils. However, all distance measurements were rounded to 0.1 mil because the slider stepper motor used to calibrate the probe produces a 0.125 mil movement per step. Calibration involved measuring the voltage change after a given number of steps and dividing the measured voltage change by the distance moved in mils. This was repeated ten times and the computed average value was used. To assure that changes in metal composition or sample shape did not bias the metal recession measurements during testing, this calibration procedure was followed just before each distance measurement was made.

Figure 3 is a photograph of the proximity probes, sliders, specimen and part of the flame producing burner. The measuring proximity probe is shown located in the hot zone measurement position at the center of the test specimen. The burner is in the heating position but the flame was out for the photographic session. In normal use the burner is rotated away from the specimen and the specimen is allowed to cool before the probe is brought up to the measurement position shown. The horizontal slider is used to transport the probe to a cool storage position while the specimen is exposed to the flame. However, the probe must be returned to the exact same measuring position before each distance measurement is made because the desired information is the metal recession or change in the distance from the fixed probe location to the specimen. The relocation of the measurement probe is done with a second probe, called the positioning probe which is fixed to the horizontal slide support. As the horizontal slider approaches this probe it is servopositioned to the same location, to within 0.125 mils. Now the distance measuring probe output can be measured and will change in accordance with the change in the distance to the specimen. If the metal is receding and the specimen is a cylinder, then the change in distance is the loss of radius. Multiplying this loss of radius by 2 and subtracting from the original known diameter gives the diameter loss of the specimen.

A distance measurement was made at the hot zone location of the specimen and also at a nonheated or reference zone located near the top end of the specimen. The vertical slider was used to move the distance probe to and from this reference zone. By making this added measurement it was possible to compensate for any distortion due to temperature effects on the specimen holder chuck or drift in the measuring system. All readings by the distance probe were made at the same circumferential location on the specimen by using a flag attached to the rotation shaft which, when it passed an optical interrupter, produced a signal which was sent to the data acquisition unit instructing it to make a distance voltage reading. This synchronous reading technique also helped to improve the quality of the acquired data. The shaft rotation flag interrupter also provided a signal to a frequency counter which was used to provide revolutions per minute information.

A series of distance measurements with the proximity probe were made at the hot section without flame exposure to determine the stability of the probe system. The measurement system was allowed to warmup and stablize for two hours before measurements were started. It was found that over a 15 hr period the scatter of the measured distance readings was ±0.5 mil with less than 0.5 mil drift.

Video Camera System

Figure 4 is a photograph of the camera system showing the front of the camera, the zoom lens, pan and tilt support system and the infrared heat filter. The zoom lens was a motorized 16-160 mm, f1.8 type. The image is presented on a video monitor for continuous viewing. The camera and its associated electronics measure the external width of an object along a scan line whose position can be set manually or under program control to any place on the viewed image. In the experiments reported here, the scan line was set at the center of the hot zone of the specimen, at the same level at which the hot zone distance proximity probe measurements were made. The two techniques thus measure at the same cross section location and specimen temperature. The edges of the viewed specimen are detected by intensity contrast. This intensity contrast threshold was set manually, so the width measurement was automatically made for only one light condition. The width measurements, later described, were made at the maximum temperature of the specimen test cycle. These were made in a synchronous manner such that they were always made on the same instantaneous rotational diameter. The measured widths are external measurements and hence include any scale formed and retained on the specimen, i.e., not spalled.

The output of the video system is a voltage which is converted to a length measure by appropriate calibration. Calibration was carried out by panning the camera to an illuminated scale of known length and setting the scan line length to equal this length and recording the voltage output. One calibration point was sufficient because the system was found to be very linear. With the optical system used, the output for a one inch standard length was determined to be 4.22 V or 4.22 mV per mil. The voltage measurement system has a resolution of 0.1 mV so that width measurement resolution is about 0.024 mils. However, all camera width measurements were rounded to 0.1 mil which is considered to be a practical working limit.

The diameter of a test specimen was measured over a 10 hr period without flame exposure in order to determine the stability of the video system. The measurement system was allowed to warmup for 3 hr before measurements were started. It was found that the diameter measurements showed a variation of ±1.0 mil with an upward drift of 1.0 mil over the 10 hr period.

Operation of the Total System

A program was developed for micro computer operation to facilitate unattended proximity probe and camera measurements and printing of appropriate results. Printed information included the average, maximum and minimum temperature of the cycle, the average specimen rpm, test cycle number with the corresponding distance probe calibration factor. Table I is a flow chart for the operation of the total system. Steps A to G are setup steps for the first cycle and require some manual intervention while steps H to Q are repeated under program control, with no manual intervention, until all test heating cycles are completed.

OXIDATION OF 304 STAINLESS STEEL

Initial tests of the system involved the oxidation testing of nominally half inch diameter 304 stainless specimens which were rotated at 600 rpm in a Mach 0.3 flame at 910° C. Jet A-1 fuel was used and contained 0.05 to 0.07 wt.% sulfur. The combustion air was preheated to 235° C. A test cycle consisted of heating at 910° C for 15 minutes then cooling to less than 30° C.

The distance probe data for one test specimen are shown in figure 5. All the distance probe data points from the hot zone measurements were fit by a nonlinear regression analysis assuming a parabolic relation up to 5 hr and a linear relation from 5 to 10 hr. The resulting curve is shown as the solid line associated with the circled points in figure 5(a). Actual data points for the cool reference zone are also plotted in this figure as squares and an estimated line has been drawn through the data points. Taking the difference between the cool zone and hot zone values taken from the respective curves and multiplying by 2 yielded the change in specimen diameter points plotted in figure 5(b). This data was fitted with a line by linear least squares analysis and the slope is 0.41 mils of diameter loss per hour. This is the rate of metal recession. It was expected that this plot would pass through the origin and the perturbations observed during the first hour of testing were not expected and their cause is at present unknown.

There was no appreciable change in the probe calibration factor as testing progressed. This factor remained at 0.135±0.002 V per mil diameter through-out the testing. This indicates no measurable change in specimen composition or shape during testing.

Figure 6 is a plot of test time versus the external diameter as measured by the video camera system. A gradual decrease in the external diameter with a few undulations is observed. The undulations may be due to at temperature spalling of the oxide scale. The straight line shown was drawn by sight. A least squares fit of all the points was not considered to be warranted because the points appeared by inspection to be better represented by the solid line. The slope of the line is 0.27 mils diameter loss per hour. This is somewhat smaller than the value given by the proximity probe and may indicate that oxide scale is retained on the sample. Of course such scale would not be detected by the proximity probe measurement.

DISCUSSION

The two measurement systems investigated have resolutions which are considered to be sufficient for measurements of oxidation/corrosion attack in many alloys at usual use temperatures. However, both techniques exhibit measurement scatter and drift which could cloud true data in the study of very environmentally resistant materials.

The proximity probe, being sensitive to most metals, detects the advance or recession of the metallic surface irrespective of the prescence of a nonmetallic (nonmagnetic) scale. This makes it especially useful for the study of the oxidation of many metallic materials. By its proper implementation the technique can also yield valuable rate data. If the nature of the oxidation/corrosion process is such that metallic particles are present in the developing scale, then this technique will give results not easily interpreted kinetically because the probe will react to these particles giving the impression that the metal is advancing toward the probe or the metal is growing. This may be useful information in and of itself. The proximity probe technique should be especially valuable in errosion studies where none of the above caveats would apply.

The video camera technique measures an external width. If the specimen is a cylinder then a diameter is measured. This has several interesting implications. First the measured values, if the specimen is an oxidizing/corroding material, will depend on the degree of scale formation and its adherence. Therefore, this technique may yield interesting spalling information but probably little interpretable oxidation/corrosion rate data unless the scale spalls nearly completely in each cycle. Second, this technique is independent of the properties of the material. That is, it can be applied to both metallic or nonmetallic materials. Finally, because the camera is remote from the hostile test environment, measurements can be made while the test specimen is actually being exposed this then gives a truly in-situ measurement of attack.

The camera system can also measure 256 grey levels of light intensity and with a hardware modification it can move the scan line under program control at which a diameter measurement or grey level is measured. While this has not been explored this potential may allow the detection of cracks and growth rate measurement of cracks in nonmetallic materials or brittle coated alloys. When coatings separate from the substrate they become very much hotter, even approaching the flame temperatures, thus the camera would detect this occurance by a change in grey level. Also, crack edges usually glow at a temperature higher than the adjacent noncracked specimen.

The feasibility of two new attack measurement techniques has been demonstrated. The preliminary oxidation results reported here establishes that the techniques hold considerable promise for oxidation testing and especially for errosion testing. Further work is needed to more clearly define the appliability of the techniques to high temperature corrosion attack measurements.

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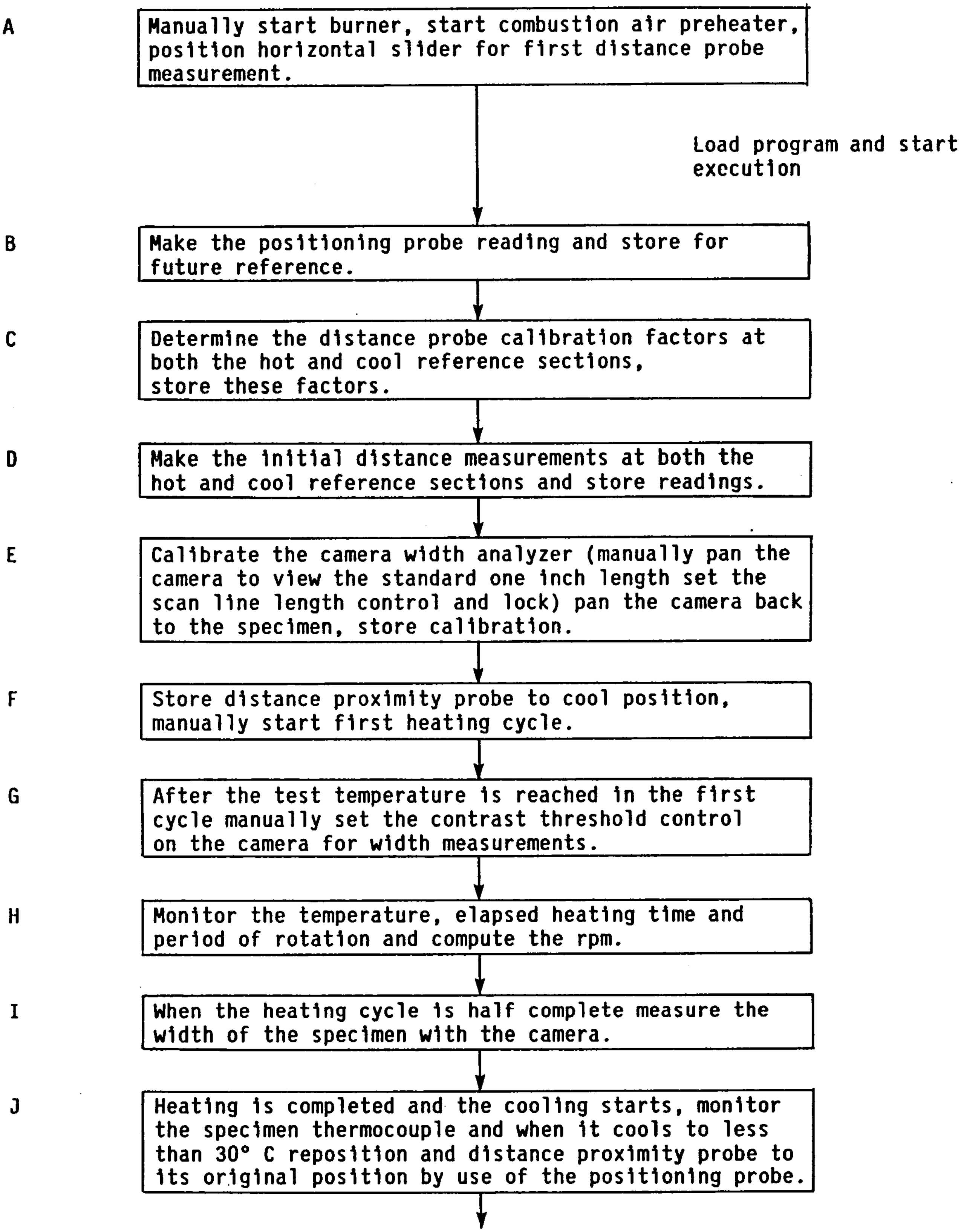
TABLE I. - OPERATIONAL FLOW CHART



A Manually start burner, start combustion air preheater, position horizontal slider for first distance probe measurement.

Load program and start execution

B Make the positioning probe reading and store for future reference.

C Determine the distance probe calibration factors at both the hot and cool reference sections, store these factors.

D Make the initial distance measurements at both the hot and cool reference sections and store readings.

E Calibrate the camera width analyzer (manually pan the camera to view the standard one inch length set the scan line length control and lock) pan the camera back to the specimen, store calibration.

F Store distance proximity probe to cool position, manually start first heating cycle.

G After the test temperature is reached in the first cycle manually set the contrast threshold control on the camera for width measurements.

H Monitor the temperature, elapsed heating time and period of rotation and compute the rpm.

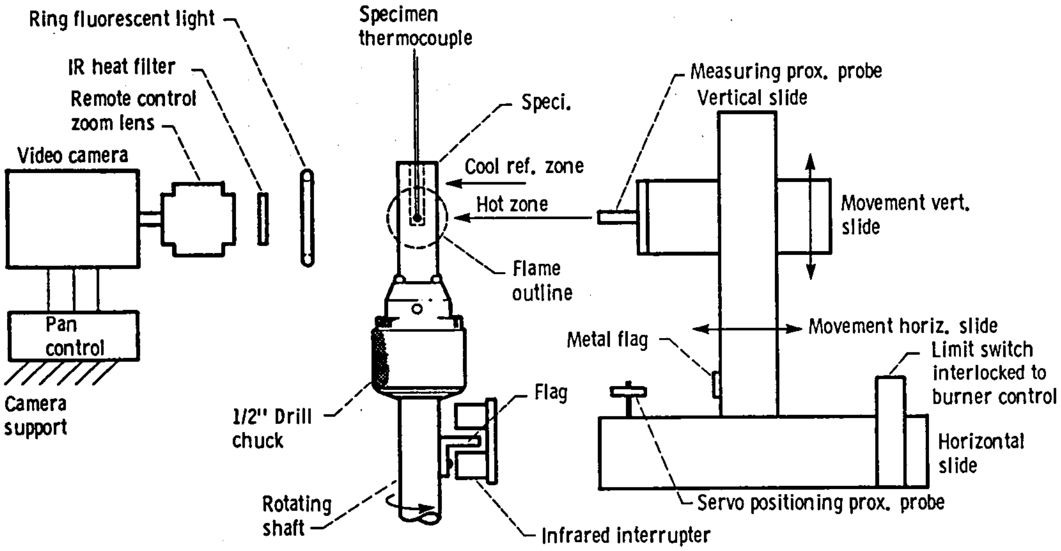
I When the heating cycle is half complete measure the width of the specimen with the camera.

J Heating is completed and the cooling starts, monitor the specimen thermocouple and when it cools to less than 30° C reposition and distance proximity probe to its original position by use of the positioning probe.

TABLE I. - CONCLUDED

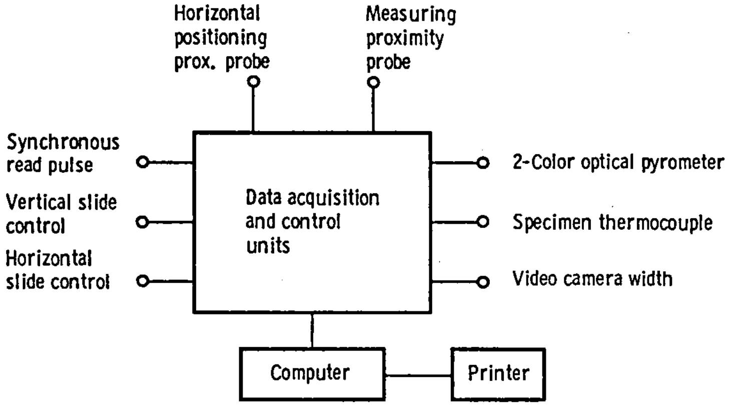
K	Determine the distance probe calibration factor at both the hot and cool sections.
L	Make distance probe measurements at both the hot and cool section.
M	Store the probe in the cool position, start next heating cycle.
N	Print all cycle data.
O	Go to step H unless all cycles are completed.
P	If all heating cycles are completed stop.

NOTES: Steps A to G require manual first cycle intervention steps H to P are repeated with no need for manual intervention.



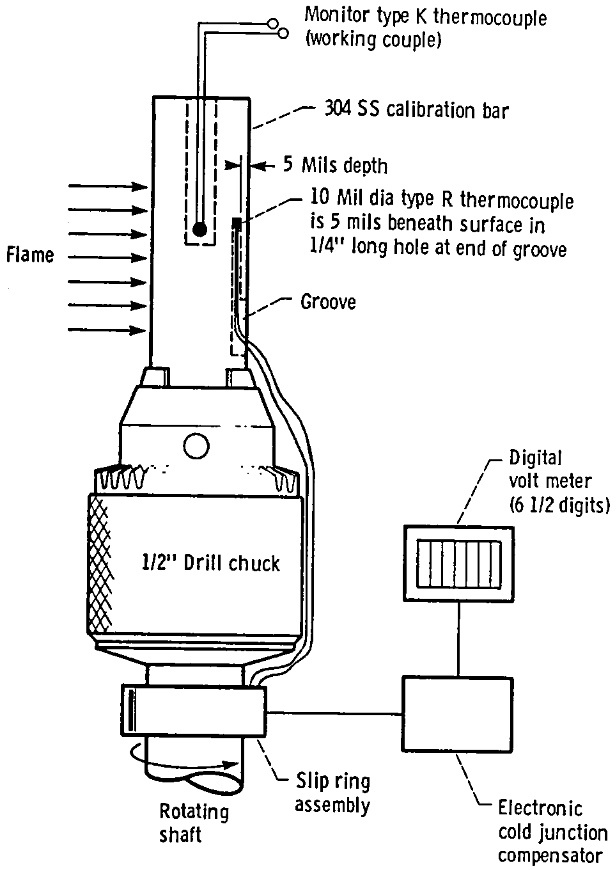
(a) Schematic of measuring systems.

Figure 1. - Schematic of in-situ corrosion measuring systems.



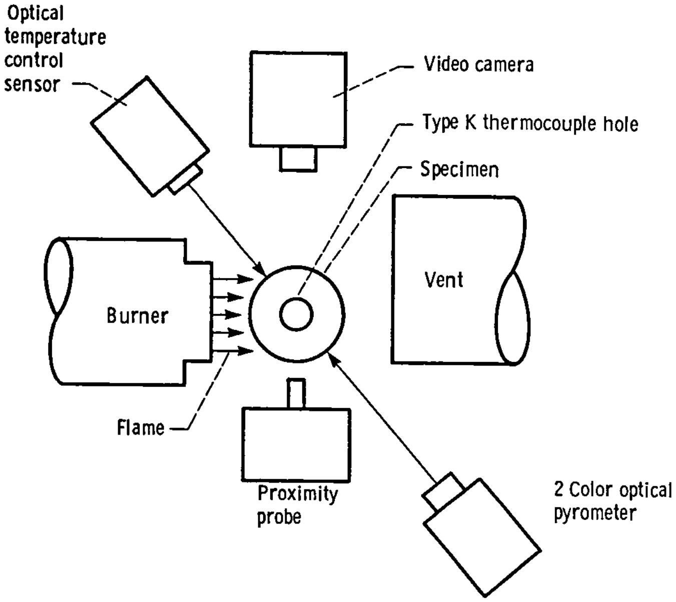
(b) Schematic of control systems.

Figure 1. - Concluded.



(a) Calibration of monitor thermocouple.

Figure 2. - Temperature calibration and equipment layout.



(b) Top view of equipment layout.

Figure 2. - Concluded.

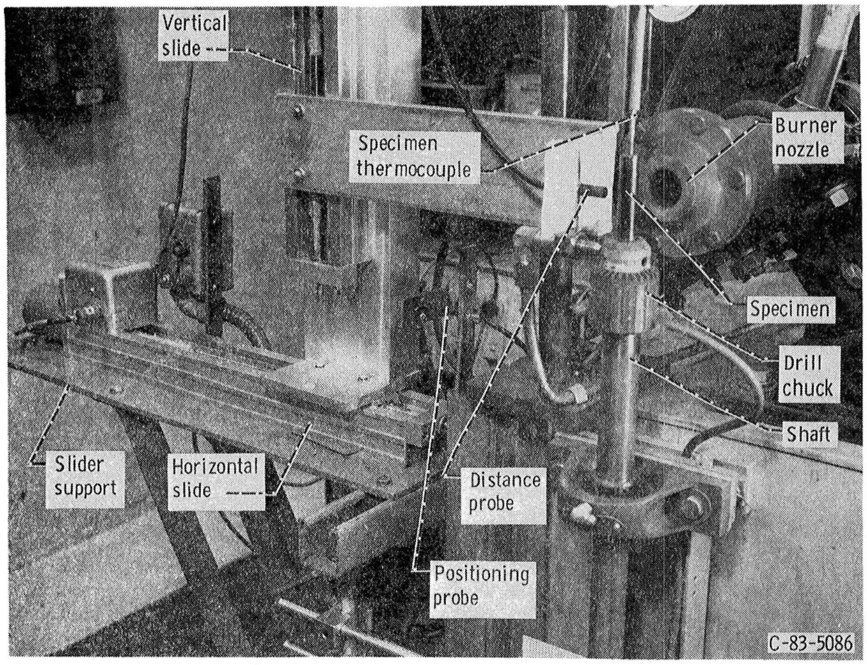


Figure 3. - Proximity probe measuring system.

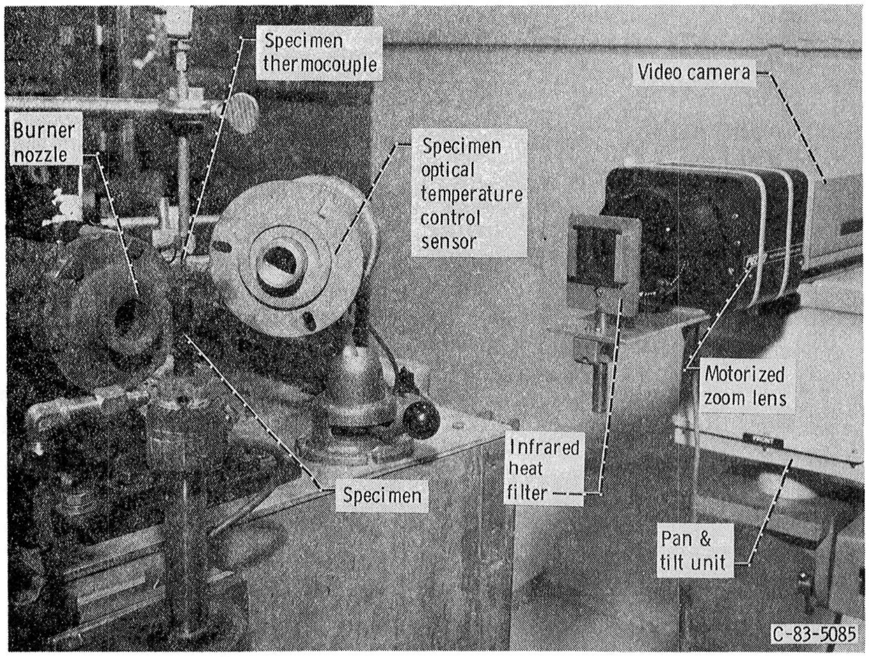
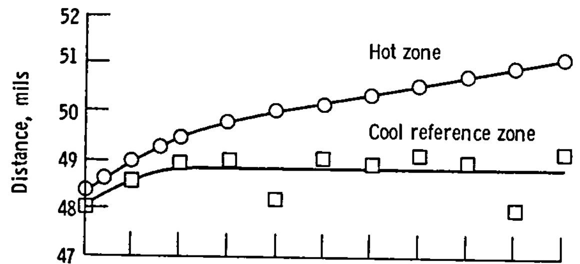
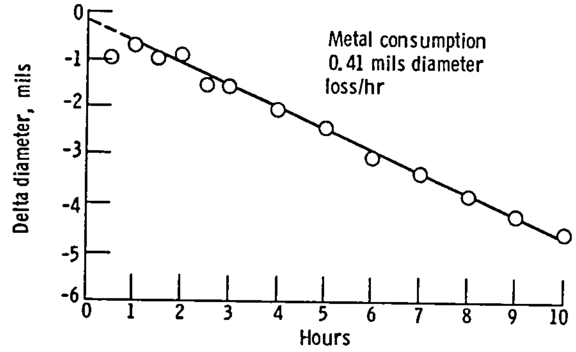


Figure 4. - Video camera width measuring system.



(a) Experimental results.



(b) Metal recession

Figure 5. - Proximity probe measurements for the oxidation of 304 SS at 910° C (15 min heating cycles used).

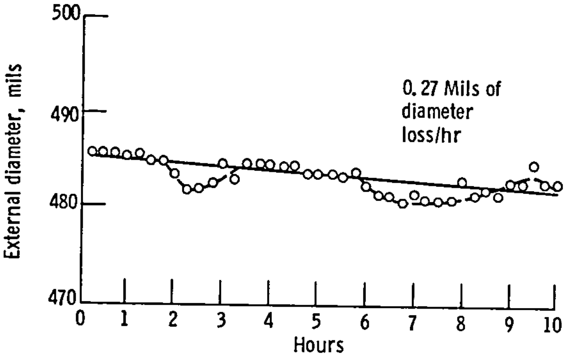


Figure 6. - Video camera measured external diameter of 304 SS specimen at temperature of 910° C and Mach 0.3. (15 min heating cycles used).

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15. Supplementary Notes

16. Abstract

Two noncontacting and nondestructure, remotely controlled methods of measuring the progress of oxidation/corrosion/errosion of metal alloys, exposed to flame test conditions, are described. The external diameter of a sample under test in a flame was measured by a video camera width measurement system. An eddy current proximity probe system, for measurements outside of the flame, was also developed and tested. The two techniques were applied to the measurement of the oxidation of 304 stainless steel at 910° C using a Mach 0.3 flame. The eddy current probe system yielded a recession rate of 0.41 mils diameter loss per hour and the video system gave 0.27.

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