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LABORATORY PROCEDURES USED IN THE
HOT CORROSION PROJECT

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1. INTRODUCTION

The objective of the Hot Corrosion Project in the LLNL Metals and Ceramics Division is to study the physical and chemical mechanisms of corrosion of nickel, iron, and some of their alloys when these metals are subjected to oxidizing or sulfidizing environments at temperatures between 850 and 950°C. To obtain meaningful data in this study, we must rigidly control many parameters. In this report, we first discuss the parameters involved and the methods we chose to control them in this laboratory. Second, we cover some of the mechanics and manipulative procedures that are specifically related to data access and repeatability. Third, we describe the method of recording and processing the data from each experiment using an LSI-11 minicomputer. Fourth, we enumerate and discuss the analytical procedures used to evaluate the specimens after the corrosion tests.

2. PARAMETERS

2.1. TEMPERATURE

An electric furnace rated at 130 V, 60 Hz, and 23 A maximum input is used to heat the metallic samples in the corrosive environment. Because the furnace is platinum wound, caution is required in the warming-up process to avoid over-driving, which might burn out the coils; to protect the furnace, a current-limiting controller is used.

The furnace temperature is sensed by a Pt/Pt:10-wt%-Rh thermocouple and is recorded both on a strip-chart recorder and by the LSI-11 computer. Because a temperature profile has been run previously on this furnace, a position variation on the recorder reveals a known temperature change (see the Appendix).

A Pt/Pt:10-wt%-Rh thermocouple probe is located immediately adjacent to the specimen, and, during an experiment, the specimen temperature is monitored using a potentiometer connected to this probe. The greatest temperature

variation from a target temperature of 900°C might be $\pm 10^\circ\text{C}$; the average variation for a typical run is about $\pm 5^\circ\text{C}$.

Because the furnace is mounted vertically, there is a chimney effect, and to minimize this, a close-fitting split collar made of firebrick is placed at the top of the furnace around the quartz sample chamber; a layer of alumina wool is wrapped around the tube above the collar to seal the cracks. The furnace is left open at the bottom.

2.2. CONTROL OF GAS QUALITY

In corrosion testing, gases are often used to alter the nature of the corrosive environment. At the present time, six different gases are available for use in the laboratory. They are purchased in the standard No. 1 cylinders and in the following purity grades:

CO_2	- Coleman Inst. grade	99.99% pure
CO	- U.H.P	99.8% pure
Ar	- U.H.P	99.999% pure
H_2	- U.H.P	99.999% pure
O_2	- U.H.P	99.99% pure
SO_2	- Anhydrous	

Before use in our experiments, the commercial gases are treated as follows:

Argon is first passed through a heated chamber (a 51-mm-diam by 610-mm-long stainless steel tube) that is filled with titanium sponge and maintained at 825°C in its own furnace. This process reduces the oxygen partial pressure to less than 100 fPa. The argon is then passed at room temperature through a chamber filled with ascarite (which is asbestos coated with sodium hydroxide) to remove carbon and finally through a chamber containing magnesium perchlorate (anhydrone) to remove water vapor.

Oxygen is also given the ascarite-anhydrone treatment to remove carbon and water vapor.

Carbon monoxide and carbon dioxide are each run through anhydrone to remove water vapor.

Sulfur dioxide is dried by passing it through a chamber of anhydrous calcium chloride; the substitution is required because of the incompatibility of sulfur dioxide and magnesium perchlorate.

Hydrogen is used without treatment.

Standard-purity argon is available from the house argon system. After passing through an anhydrous dryer, it is used as a protective purge over the electrobalance.

2.3. CONTROL OF FLOW RATES AND COMPOSITION OF GASES

Control of both the flow rates and composition of gases used in our experiments are closely related.

To achieve the desired dynamic flow rate of gas across the sample surface, we chose a total gas flow rate of 20 ml/s. (This may be varied later as experimental evidence might indicate.) The flow rate of any particular gas in the mixture is easily calculated from the proportion of that component gas in the total. For example, a 30:70 mix of carbon dioxide and sulfur dioxide flowing at 20 ml/s delivers $(0.30)(20 \text{ ml/s}) = 6 \text{ ml/s}$ and $(0.70)(20 \text{ ml/s}) = 14 \text{ ml/s}$.

To get precise flow rates, we provided each component gas with its own individual flow regulator. Each regulator consists of a conventional pressure-reducing regulator at the tank and a micrometer needle valve downstream from it. With the tank regulator set to maintain a pressure head of 0.138 MPa, for example, it is fairly easy to obtain a flow rate close to the target rate by adjusting the needle valve. The gas then flows through the appropriate purifying stages described in Section 2.2.

Sensitive pressure regulation is achieved using the apparatus shown in Fig. 1, which is connected to each gas line beyond the purifying stages. This regulation consists of a vent tube with its open end submerged in an inert liquid. The line gas pressure is determined by the height of the liquid displaced; if the pressure exceeds the desired value, the gas bubbles out through the liquid and is vented to the exhaust hood. The pressure can be varied as desired by moving the reservoir flask up or down in relation to the end of the vent tube.

We chose dibutyl phthalate as the liquid used in the pressure regulators because it is inert, dense, and has a low vapor pressure. However, because of the high solubility of sulfur dioxide in dibutyl phthalate, high-grade diffusion-pump oil is used in the sulfur dioxide regulating system. Although SO_2 is slightly soluble in the oil, the oil still maintains its integrity and function.

FIG. 1. Apparatus for sensitive pressure regulation that is connected to each gas line beyond the purifying stages. The open end of the vent tube is submerged in dibutyl phthalate or high-grade diffusion-pump oil. The line gas pressure is determined by the height of the liquid displaced and can be varied by moving the reservoir flask up or down in relation to the end of the vent tube.

The constant-pressure gas is then passed through a calibrated capillary tube that determines the actual flow rate. To measure this flow rate and enable us to regulate it to the desired amount, we installed a U-tube manometer across each capillary (Fig. 2). The fluids used in the manometers are the same as those in the respective sensitive pressure regulators.

Once the pressure-regulation system is calibrated (Section 3.2), any desired flow rate can be obtained. This is done by first selecting the proper capillary for the range desired, then adjusting the fluid height in the sensitive pressure regulator to the value determined by the calibration, and manipulating the gas flow at the needle valve to produce a slow bubbling at the pressure overflow. Readjustments are usually necessary to compensate for system back pressures that result when second or third gases are added to the total flow. We have found that once equilibrium is achieved, very little readjustment is necessary.

The same flow-adjustment procedure is followed for each component gas of any given mixture. Once the flow rate is established for each gas, the gases are combined at the system manifold. The gases then flow through the labyrinth mixer, which consists of a chamber filled with marbles or large beads. The homogeneously mixed gas then goes through either a flow-rate calibration, to an oxygen-activity test cell, or directly to the corrosion test chamber. In any case, the gas is ultimately vented to an exhaust hood that has adequate draw to prevent contamination of the surrounding work space by irritating or toxic gas mixtures.

To date, all corrosion experiments have been conducted using gas mixtures at atmospheric pressure. Calculations indicate that normal barometric-pressure variations have little effect on the flow-rate calibrations.

An oxygen-activity test cell designed to measure the partial pressure of oxygen (P_{O_2}) is used to verify theoretical calculations. As shown in Fig. 3, the gas flows along a zirconia tube. The inside and outside surfaces of the closed end of the tube have both been coated with platinum, which provides electrical conductance at both surfaces. An electrical lead to each platinum surface measures the electromotive force (emf) across the zirconia wall; the system is thus $O_2, Pt | ZrO_2 | Pt, O_2$ (ref). When this system is treated as a concentration cell, the oxygen partial pressure is given by the Nernst equation as:

FIG. 2. Calibrated capillary tube used to regulate gas flow rate. The desired flow rate is obtained by adjusting the manometer fluid-height differential. This is done by selecting the proper capillary for the range desired, adjusting the fluid height in the sensitive pressure regulator to the value determined by the calibration, and manipulating the gas flow at the needle valve so that the gas bubbles slowly at the pressure overflow.

FIG. 3. Oxygen-activity test cell that measures P_{O_2} , the partial pressure of oxygen. Gas flows along the outside of the zirconia tube, and an electrical lead to each surface measures the electromotive force across the zirconia wall. The inside and outside surfaces of the closed end of the zirconia tube are coated with platinum to provide electrical conductance at both surfaces.

$$P_{O_2} \text{ (atm)} = \exp \left[\frac{-E}{T(2.1543 \times 10^{-5})} - 1.5606 \right] , \quad (1)$$

where

T = temperature at test point (K),

E = emf output across zirconia (V) when $P_{O_2}/P_{O_{2,ref}} < 1$.

The gas mixture should have nearly attained thermodynamic equilibrium after passing through the furnace and before reaching the zirconia probe.

Because of the thermodynamic activity of the gases at test temperatures, it is desirable to ensure that they have reacted chemically as completely as possible before they reach the test-specimen chamber. Therefore, just before the gases reach the specimen, they are passed over a catalyst of 0.5% platinum on alumina pellets that is placed in the hot zone of the test chamber. The gases flow through about 15 cm of catalyst before reaching the test specimen.

3. MECHANICAL AND MANIPULATIVE PROCEDURES

Experiments to date have been devoted largely to studying corrosion of high-purity nickel and, to a lesser extent, some nickel alloys containing aluminum or aluminum and chromium.

3.1. SAMPLE PREPARATION

3.1.1. High-purity nickel samples

The nickel is obtained from a commercial vendor in the form of 305 × 12.7-mm-diam rods; copies of the vendor's purity analysis and of a purity analysis made at LLNL are given in the Appendix.

The high-purity nickel samples are prepared as follows:

1. Slice the rod into disks about 2.2 mm thick using an abrasive saw.
2. File off any gross unevenness or burrs.
3. Machine flat the face of a lapping block; a micarta cylinder about 150 mm in diameter by 75 mm long is suitable.

4. Cement the nickel disks to the flat face of the lapping block using 5-min epoxy as a bond. Care should be taken to press the nickel specimens as closely as possible to the block face while the epoxy cures. Attach as many specimens as fit conveniently.

5. After sufficient cure time has elapsed, run the specimens on a lapping wheel until they have all been brought to the same lap height (i.e., as soon as all surface cutting marks have disappeared).

6. Remove the specimens from the block and rebond them again with the other side up. (An easy way to break the epoxy bond is to set the block on a hot plate that is just hot enough to char the epoxy--about 205°C.)

7. Follow the same lapping procedure again after the specimens have been recemented on the block.

8. Remove the specimens from the block (using the hot-plate method) after they have all been brought to a uniform lapped thickness.

9. Drill a hole near the edge of each disk using a No. 51 drill. This hole is used to hang the specimen on a hook during the corrosion test. Deburr the hole.

10. Heat-treat the specimens by vacuum annealing for 24 h at 800°C; do not quench the specimens.

11. Store the specimens in a dessicator jar until they are needed for corrosion testing.

Immediately before a corrosion experiment is begun, each specimen should be treated as follows:

1. Polish each surface of the specimen on a sheet of 600-grit silicon carbide paper laid on a surface plate.

2. Clean thoroughly with acetone and alcohol.

3. Determine the surface area of the disk. Using a 0.001-cm micrometer, take four random diameter readings and eight peripheral thickness readings (Fig. 4).

4. Substitute the average values of these numbers into the following formula for total surface area:

$$A = \pi \left(\frac{D^2 - H^2}{2} + DT + TH \right) \quad (2)$$

FIG. 4. Pure nickel specimen disk. Surface-area measurements are made using a 0.001-cm micrometer.

where

A = total surface area of the circular disk,

D = diameter (average),

T = thickness (average),

H = hole diameter (see Fig. 4).

5. Measure the mass of the specimen to the nearest milligram. The initial mass of the specimen must be known to set the weight suppression on the electrobalance at the start of the experiment or, if the specimen is to be salt coated, to calculate the salt density on the surface of the specimen.

3.2.2. Nickel-alloy samples

There are several major differences in the preparation of nickel-alloy specimens. We have performed corrosion experiments on the following alloys (where Ni-2Al indicates Ni:2-wt%-Al, etc.):

Ni-2Al	Ni-12Al	Ni-20Al
Ni-5Al	Ni-13.3Al	Ni-22Al
Ni-6Al	Ni-14Al	Ni-30Al
Ni-8Al	Ni-16Al	
Ni-10Al	Ni-18Al	

We have also conducted experiments on Ni-Cr and Ni-Al-Cr alloys.

We made 100-g buttons of these different alloys using the following process:

1. Weigh appropriate proportions of the component metals (e.g., 98 g nickel and 2 g aluminum for the Ni-2Al alloy).
2. Combine these metals in a copper crucible inside an electric-arc furnace, which is called a button furnace. This button furnace has a vacuum chamber fitted with a viewing port and a manipulatable tungsten-arc probe. The probe is mounted above the copper crucible that is screw-mounted on a water-cooled base plate. The chamber walls are also water cooled, and a valving system enables the chamber to be evacuated and then back-filled with argon to 0.5 atm. The copper crucible is a 100-mm-diam disk about 38 mm thick with a rectangular depression 57 x 32 mm and about 19 mm deep in the center. The depression has a flat bottom and sides that taper outward toward the top.
3. Vacuum purge the furnace and back-fill it with argon at least twice to ensure an inert environment for the melt. The cooling water must be turned on.

4. Place a filter over the viewport for eye protection.
5. Position the tungsten probe directly over the sample metal in the crucible and strike an arc directly to the metal. The arc probe must be kept at the proper distance and moved over the surface of the melt so that the material melts uniformly into a molten button. Care must be taken to prevent arcing to the copper that would, of course, introduce copper to the alloy button.
6. Allow the button to cool so that it can be handled (about 15 min) and remove it for visual inspection to see if it melted completely and uniformly. It will probably be necessary to remelt the button three to five times, repeating the above procedure to ensure a homogenous, smooth, well-mixed alloy button.
7. Heat treat the button by heating it in a vacuum furnace at 1200°C for 6 h; do not quench the button.
8. Remove surface oxides by sandblasting.
9. Cut the button into slabs and divide the slabs into specimen-sized pieces (usually about 10 x 14 x 1.5 mm thick) using a moving-wire spark cutter. Because the spark cutter leaves a rough surface finish, it is necessary to lap-finish the front and back surfaces of the alloy samples in the same manner used to prepare the pure-nickel samples. To finish smoothing the edges of the specimens, we use a small holding tool that we designed for use with a surface plate and No. 600 silicon-carbide paper (see Fig. 5). By clamping the specimen in the holder and adjusting the leveling screw, the edges can be finished quickly.
10. Drill the hole needed for the support hook. Because some of the alloy specimens are too hard or brittle for this hole to be drilled with ordinary drills, a spark cutter or ELOX machine is used.
11. Face-polish each specimen as described for the nickel specimens. The specimen should look like the sample shown in Fig. 6.
12. Determine the total surface area. This value is needed to monitor the weight gain per unit area during corrosion experiments. The average values from several micrometer measurements of length, width, thickness, and hole diameter can be substituted into the area formula for rectangular samples as follows:

$$A = 2(LW + LT + TW) + \frac{\pi(2T - H)}{2} \quad (3)$$

FIG. 5. Holding tool used to finish specimen edges. By clamping the specimen in the holder and adjusting the leveling screw, the edge can be finished using 600-grit silicone carbide paper.

FIG. 6. Nickel-alloy specimen. Surface-area measurements are made using a 0.001-cm micrometer.

where

A = total surface area,

L = length,

W = width,

T = thickness,

H = hole diameter.

13. Clean and weigh the sample following the instructions given for the pure nickel specimens (Section 3.1.1). The sample is now ready for the corrosion experiment.

3.2. CAPILLARY-TUBE CALIBRATION

To measure gas-flow rates using capillaries, each capillary tube in each gas-supply line must be individually calibrated. The actual gas flow (volume/unit time) through each capillary depends on three factors:

- The fixed size of the capillary hole.
- The differential pressure across the capillary.
- The particular gas being used.

Therefore, we make a separate graph for each capillary and for each gas for which it might be used. On this graph is plotted gas flow (in ml/s) against pressure differential, as indicated by the displacement of manometer fluid (Fig. 2). The Appendix contains several typical calibration charts.

To calibrate the capillaries, we needed to determine the actual gas-flow output, so we designed a gas flow meter that works as follows: A gas inlet and a reservoir for a soap-bubble solution are attached to the bottom of a 100-ml burette; a rubber squeeze bulb is attached to the reservoir (Fig. 7). For the soap-bubble solution, we use either a gas-leak-detector solution diluted with an equal volume with water or a saturated aqueous solution of laboratory glass cleaner. (The latter seemed to work best.)

The gas output from the capillary is connected to the gas inlet at the bottom of the burette. With the gas flowing, the squeeze bulb is used to raise the soap-bubble solution in the reservoir above the gas-inlet opening in the column so that bubbles form at the bottom of the burette and rise to the top. The time required for these bubbles to move between selected marks on the burette is measured with a stop watch, and the gas flow rate is calculated. This procedure is repeated several times and the values averaged to obtain a gas flow rate.

FIG. 7. Soap-bubble gas flow meter used to calibrate the capillaries. A gas inlet and a reservoir for the soap-bubble solution are attached to the bottom of a 100-ml burette; a rubber squeeze bulb is attached to the reservoir, and the gas outlet of the capillary is connected to the gas inlet at the bottom of the burette. With the gas flowing, the bulb is squeezed to raise the soap-bubble solution above the gas-inlet opening so that bubbles rise up the burette. The time required for these bubbles to move between selected marks on the burette is measured with a stop watch, and the gas flow rate is then calculated.

The calculated flow rate is plotted as a function of the pressure differential, which is read from the manometer (Fig. 2). After one rate has been entered as a point on the graph, the pressure differential is changed and a new flow rate determined for that value. This process is repeated until a reasonable number of points on the graph can be connected with a smooth line. Each capillary must be calibrated for each gas in this manner (see Appendix).

3.3. SALT COATING

Several series of corrosion experiments have been conducted using specimens that have been coated with a salt (such as sodium sulfate). Because this coating should be uniform from one specimen to another, we describe the coating method we used:

1. Lay prepared specimens (completely cleaned, polished, weighed, and measured, as described in Section 3) on a flat aluminum plate that just covers the top surface of a hot plate.

2. Prepare the salt solution by dissolving 3 g of the salt in water in a spray-gun jar (about 20 ml).

3. Heat the specimens to 205°C.

4. Spray the salt solution on the specimens using an air brush. Adjust the spray gun to obtain the finest spray mist possible with the least splatter. Spray the specimens with short sweeping bursts and allow sufficient time between bursts for moisture to evaporate from the specimens; this produces an even coating of salt on the surface. Rotate and/or flip the specimens over frequently so that the coating is applied as evenly as possible over the entire front and back surfaces.

5. Determine the salt density. Each specimen must be individually prepared to the correct weight gain. To calculate the salt density on the specimen, the precise weights before and after coating and the surface area of the specimen must be known. It is sometimes necessary to weigh the specimen several times during the salt-coating process to obtain the proper density, which is $1.00 \pm 0.05 \text{ mg/cm}^2$. If too much salt is applied, wash off the salt coating and start over. It should be noted that if the coating is applied too rapidly, the salt will fracture and spall off the specimen.

6. Store coated specimens in a desiccator jar.

4. EXPERIMENTAL PROCEDURE

The following procedure is typical of experimental runs we have performed. A nickel specimen coated with sodium sulfate is exposed to a 65:35 mix of carbon dioxide to sulfur dioxide gas for 4 h at 900°C. For all runs, continuous monitoring of temperature and weight change is needed. Figure 8 shows the experimental chamber and the necessary equipment.

1. Stabilize the furnace temperature at about 900°C.
2. Turn on the argon back-flow through the electrobalance to 3-5 ml/s flow.
3. Set up the gas-metering system to provide the desired 65:35 mix of carbon dioxide and sulfur dioxide at 20 ml/s total flow. (The preliminary setup of proper gas mix and flow should be completed under the conditions prevailing during the experiment but with no sample in the chamber.)
4. Shut off the carbon dioxide and sulfur dioxide gases at the tanks and purge the system with argon for 5 to 10 min.
5. Open the test chamber and hang the prepared specimen on the hook of the wire extension to the balance in the upper (or "out-of-furnace") position. The specimen is chemically insulated from the wire by using a quartz hook, which prevents electrochemical interaction between the hang-down wire and the specimen. (The quartz hooks are made in the laboratory by drawing a quartz rod down to a sufficiently small diameter to fit through the hole drilled in the specimen and then bending it to form a hook. Each hook is good for only one experiment.)
6. Begin the experiment by lowering the specimen into the hot zone of the experimental chamber. The electric furnace is track-mounted and counterbalanced so that it can be easily adjusted up or down; it has a free-travel range of about 22 cm from the top to bottom positions (Fig. 8). Also, the experimental chamber itself is easily opened by removing the gas-line connection at the bottom and unhooking the tension-spring support hooks at the taper joint at the top of the furnace. The quartz tube is very hot, so asbestos gloves are needed, but with a little practice it is easy to coordinate the lowering of the tube with the furnace. Moving them together prevents rapid and wide temperature fluctuations on the quartz tube that tend to degrade it rapidly. A piece of firebrick located strategically below the furnace provides a place to set the tube as the specimen is loaded or removed. With the tube and furnace lowered, the hang-down wire for the

FIG. 8. Experimental chamber, electric furnace, and ancillary equipment. The electric furnace is track-mounted and counterbalanced so that it can be adjusted up or down. The experimental chamber itself is opened by removing the gas-line connection at the bottom and unhooking the tension-spring support hooks at the taper joint at the top of the furnace. A piece of firebrick below the furnace provides a place to set the tube as the specimen is loaded or removed.

balance is accessible, and the support fork described below is set at the top position with the hang-down extension wire (on which the specimen is hung) hooked over the top fork (Figs. 9a and b).

7. Close the chamber by raising the tube and furnace together to their upper positions, rehooking the support springs to the tube, and connecting the gas line at the bottom. Insert the bottom fork through its port in the chamber and adjust it so that the two wires are straddled by the fork.

Each fork consists of a 4.75-mm-long stainless steel tubing with a 0.6-mm-long slot cut about 12 mm longitudinally at one end and a manipulating ring soldered at the other end. A smaller-diameter rod that fits inside the tube is used to push the hang-down wires out of the fork at the appropriate time (Fig. 9c).

At this point, the balance hang-down wire (0.25 mm nichrome wire with a hard solder bead at its bottom end) is threaded through both the top and bottom forks, and the hang-down extension wire (0.50 mm Pt:10-wt%Rh wire) is also threaded through both forks and supported at the top fork by the loop around the nichrome hang-down wire (Fig. 9a). (Pt:10-wt%Rh wire is used for the hang-down wire extension because it is a little stiffer than pure platinum wire and holds its shape better, especially when the sample is dropped into the furnace.)

8. Purge the system with argon for about 30 min.

9. Prepare the computer and the back-up strip-chart recorder to record the experiment. The chart is labeled as in Fig. 10. The specimen identification number is coded to provide the information shown in Fig. 11.

10. Begin the experiment.

- a. Turn on the exhaust hood.
- b. Stop the argon purge and start the flow of the gas mixture.
- c. Wait a few minutes to allow the chamber to reach equilibrium with the gas mixture.
- d. Start the computer and the chart recorder.
- e. Lower the specimen into the furnace by pushing the hang-down wires from first the top and then the bottom forks. The specimen drops to its lowest position (Fig. 9b); the loop at the top of the hang-down extension wire is supported by the hard solder bead at the bottom of the nichrome hang-down wire. The specimen, quartz hook, hang-down extension wire, and nichrome hang-down wire are now all suspended from the electrobalance wire.

FIG. 9. (a) Corrosion specimen in raised position. The hang-down extension wire on which the specimen is hung is hooked over the top fork. The bottom fork is adjusted so that it straddles the balance hang-down wire and the hang-down extension wire. (b) Corrosion specimen in lowered position. The hang-down extension wire has been pushed off the top fork and is supported by the bottom fork. (c) Enlarged cross section of the fork assembly. Each fork consists of a stainless steel tubing with a slot cut at one end and a handle soldered at the other end. A rod inside the tube is used to push the hang-down wires beyond the end of the fork, which allows the hang-down extension wire to slide down the balance hang-down wire.

Sample I.D. _____	Date _____
Chart Speed _____	Range _____
Temperature: _____	
Atmosphere: _____	
Material: _____	
Comments: _____	

FIG. 10. Identification label attached to strip-chart recorder paper. The following information is entered:

Sample identification number: See Fig. 11.
 Date: Beginning date of experiment.
 Chart speed: Chosen to provide reasonable resolution.
 Chart range: Chosen to provide maximum sensitivity.
 Temperature: Temperature of furnace at sample.
 Atmosphere: The gas mixture.
 Material: Composition of specimen.
 Comments: 1. Specimen surface area.
 2. Salt coating (mg/cm^2).
 3. Duration of run.
 4. Special circumstances.

FIG. 11. Specimen identification number code. For example, SA810081 means that the specimen was the first salt-coated alloy specimen run on 10-8-78.

f. Tare the suspended weight on the electrobalance console as quickly as possible so that the indicated weight on the scale is as near zero as possible without going negative.

g. Switch the output to "ON" (from "SHORT") as soon as the balance is tared slightly above zero. The data is recorded on both the chart recorder and computer.

h. Remove the forks from their feed-through ports in the chamber wall and plug the holes with rubber stoppers. This vents all the gas out through the exit tube to the exhaust hood. (For some gas mixes with high percentages of sulfur dioxide, the gas tends to condense in the chamber above the furnace before it is vented up the hood. To prevent this condensation, we installed a couple of reflector infrared heat lamps about 12 cm away from the chamber and directed them on the areas of condensation; reflective aluminum foil was wrapped around the back side of the cube opposite the lamp.)

i. Allow the experiment to run to completion. The computer has already been programmed to run a specific length of time for this run. It is only necessary to maintain constant gas flow and to monitor the sensitivity range of the electrobalance to be sure the data stays within the appropriate range.

11. Remove the specimen after completion of the run using the following procedure:

a. Switch the electrobalance to "SHORT" and turn off the chart recorder after the computer has run the allotted time interval and shut itself off.

b. Turn off the gas-mixture flow.

c. Turn on pure argon gas and purge the system for 3 to 5 min.

d. Turn off the argon and the heat lamps.

e. Lower the experimental chamber and furnace in the same manner as described for sample loading. This exposes the hang-down wire extension in the gap between the experiment chamber and the taper joint.

f. Remove the rubber stopper and insert the upper support fork so that it is next to the hang-down wire. The wire cools rapidly and can be handled within a few seconds.

g. Carefully raise the hang-down extension wire high enough so that it can be reinserted in the top support fork and so that the specimen is above the furnace.

h. Remove the quartz hook with the specimen on it with a pair of tweezers. Check that the hook bent into the bottom of the hang-down extension wire was not straightened by the fall at the beginning of the experiment, as sometimes happens. Reshape the hook if necessary.

5. RECORDING DATA AND PROCESSING DATA

All data are currently being recorded at LLNL on an LSI-11 mini-computer using the diskette storage device. With this system, it is possible to read data directly from the diskettes onto the LLNL Octopus system for data analysis and plotting. Experimental data flow is shown by the block diagram in Fig. 12.

In the laboratory, it is only necessary to determine the desired time for each experimental run; we enter this value as an instruction in the computer and initiate the program. The computer automatically records all data--clock time (seconds from midnight), weight of specimen, and temperature of specimen--for the specified time. At the end of the run, the file must be given its own unique identification file name to make it part of the permanent record on the diskette. The identification number is coded as `exp123456` in Fig. 11.

Complete information on the operation of the computer is provided by the manufacturers of the computer hardware and software.

6. ANALYTICAL PROCEDURES

The completed corroded specimens may be subjected to any of the following analyses, which are conducted in laboratories other than this one:

1. Macrophotos: Specimen photographs (with magnifications of about 5x) show the general appearance and basic type of corrosion encountered.
2. Photomicroscopy: Highly polished and etched cross sections reveal the surface morphology. In preparation for photomicroscopy, specimens are cast in resin to preserve surface-scale structure and sectioned using a slow-speed diamond saw.
3. X-ray diffraction: This process determines the structure of the phases observed.

FIG. 12. Experimental data flow. All data are currently recorded on an LSI-11 mini-computer using the diskette storage device. With this system, it is possible to read data directly from the diskettes onto the LLNL Octopus system for data analysis.

4. Scanning electron microscope: The SEM provides information about the surface topography and chemical analysis by energy dispersive x rays.

5. Gas-sample analysis: Mass spectrometer analysis verifies that chamber-gas mixtures, samples of which are drawn from the chamber output, have the desired composition.

7. ACKNOWLEDGMENTS

I thank David Short and John Truhan for their patient supervision, guidance, and explanations.

KMG

8. APPENDIX

FIG. A1. Temperature profile of electric furnace used to heat the metallic samples in the corrosive environment. The furnace is rated at 130 V, 60 Hz, and 23 A maximum input. Temperature is sensed by a Pt/Pt:10-wt%-Rh thermocouple and is recorded both on a strip-chart recorder and by the LSI-11 computer.

Materials Certification Please Forward with Material																									
Order of Call Form: Lawrence Livermore Labs 7000 East Avenue Livermore, CA 94550												Lot No. 28-12071 Part No. Description 12x12 Nickel Rod													
P.Q. NO. 4580231 MATERIAL: 99.99% Nickel MRC NO.: 25919																									
CHEMICAL ANALYSIS (PPM)																									
C	O	H	N	B	Be	Al	F	Na	Mg	P	S	Cl	K	Ca	Ti	Fe	Cu	Sr	Cr	Ni	Zn	Ga	Se	Zr	Nb
17.0	18.0	2.00	25.00			210.0		10.0	12.0	ND	ND	ND													
Mn	Pb	Ag	Cd	In	Sn	Sb	Te	Ge	W	Pt	Au	Pd													
ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND													
Analysis: ☐ Mass Spectrographic ☒ Vacuum Fusion (Gases) ☒ Emission Spectrographic ☒ Conductometric (Carbon) ☒ Neutron Activation Others < 0.1 or < D.L.																									
DIMENSIONAL INSPECTION: Per customer's specifications.																									
FABRICATION: Rods processed and fabricated to spec.																									
PACKAGING: Water vapor proof container.																									
NOTES																									
DATE: _____ MATERIALS RESEARCH CORPORATION MATERIALS RESEARCH CORPORATION 10000 E. 1st Avenue Livermore, CA 94550 (415) 780-1000																									

MATERIALS RESEARCH CORPORATION		FORM NO. 1000	
LIVERMORE, CALIF. 94550		DATE: 10/10/71	
RE: 28-12071		1. Days	
SPEC. NO. 4580231		M. Number	
Values are approximate values by weight for the impurity elements listed.			
Fe	4.0 ppm	The following elements were not detected:	
Cl	100	< 0.0 Na	
Ca	100	< 1.0 Ba, Cl, Co, Zn	
Al	20	< 0.5 Si, Sn, Pb, Sn	
Si	5	< 1.0 Cu, Fe, Ni, Mn, Fe, Sn	
Ni	5	< 4.0 Ni	
Pb	2.0	< 1.0 Ag, Au	

FIG. A2. Purity analyses for nickel rods. These analyses were obtained from the commercial vendor and made at LLLNL.

FIG. A3. Typical calibration charts of capillary tubes in gas-supply lines. Because the actual gas flow (volume/unit time) through each capillary depends on three factors (the fixed size of the capillary hole, the differential pressure across the capillary, and the gas being used), a separate graph is made for each capillary and for each gas for which it might be used.