

STRESS-CORROSION CRACKING SUSCEPTIBILITY  
OF ZIRCONIUM IN FERRIC-CHLORIDE SOLUTION

by

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# STRESS-CORROSION CRACKING SUSCEPTIBILITY OF ZIRCONIUM IN FERRIC-CHLORIDE SOLUTION

## INTRODUCTION

The use of zirconium as an engineering structural material is quite limited other than in atomic reactors where a combination of low neutron cross-section and strength at temperature properties make it highly desirable. However, its resistance to corrosion in a large number of environments coupled with the developments in the last few years in producing high-grade material economically and the refinement in fabrication techniques make zirconium appear attractive for use in industry where severe corrosion conditions are encountered.

Zirconium has already been tried in several production and laboratory applications with a high degree of success where corrosion has been a major problem. McClain and Nelson (12, p. 1-7) have reported the application of zirconium in hydrochloric, sulfuric, and phthalic-hydrochloric acids, wet hydrogen chloride-chlorine atmospheres, and fused caustic. Applications such as tanks, liners, strainers, agitators, exhausters, hubs, fans, valves, pipe fittings, nozzles, pump parts, and many others were found successful in the corrosive environments mentioned. Golden (5, p. 305) has reported the excellent resistance of zirconium to all but the highest concentrations of the mineral acids. He found it resistant to organic acids and inorganic chloride solutions except for ferric and cupric-chloride solutions.

Unfortunately, a material having good corrosion resistance may be susceptible to stress-corrosion cracking when simultaneously subjected to static stress and a corrosive environment. The result can be premature or, as often is, catastrophic failure. The importance of stress in corrosion is well known. Numerous cases can be cited where failure in service is attributed to a combination of stress and an environment which could not cause failure without the additive action of the stress. Several of such cases are discussed in the publications of symposiums on stress-corrosion cracking (1, p. 1-495) (15, p. 1-202). For a designer to make correct choices of materials, apply rational methods of construction and incorporate efficient protection against stress-corrosion cracking he should possess some knowledge of the process, the performance of similar designs in service, and at least the results of laboratory testing.

Except in hot water and steam where stress apparently has no effect (9, p. 620) no experimental testing has been done to evaluate the stress corrosion behavior of zirconium. However, there is no reason to expect zirconium not to be susceptible to stress-corrosion cracking. Most authorities and investigators of this type of corrosion state that most metals are susceptible providing the right conditions are present. Titanium, like zirconium, has excellent corrosion resistance in a wide range of media, but also like zirconium, this resistance is not inherent but associated with a protective surface. Because of this Keifer and

Harple (7, p. 74-76) felt commercially-pure titanium should not be immune to stress-corrosion cracking if conditions were severe enough. They tested for cracking in more than 20 aggressive media with no indications of cracking, but when tested in red-fuming nitric acid they observed severe cracking.

It has been postulated that only alloys are susceptible to stress-corrosion cracking, pure metals being immune (15, p. 48), but even with the most elaborate refining techniques pure metals will likely be pure only hypothetically. Even if pure metals are not susceptible and could be produced the cost of producing them would eliminate such materials from use other than in the laboratories.

Because zirconium does appear attractive as a competitive material in industry where corrosive environments are encountered and because very little experimental testing has been done to evaluate the stress corrosion behavior of zirconium this investigation was initiated.

#### Objective

This investigation was initiated to determine how reactor-grade zirconium behaved when subjected to high-tensile stresses in corrosive environments.

After a preliminary investigation, a primary objective was set to determine if zirconium was susceptible to stress-corrosion

cracking in aqueous solutions of strong oxidizing chlorides. The first concern was to establish qualitatively if zirconium displayed a brittle type of cracking when stressed near the yield strength in this type of environment and then to determine the effects of stress and solution concentrations on the susceptibility and the time required for failure. Secondary objectives were to analyze the cracking that occurred and, if possible, offer an explanation for the mechanism of failure.

#### Scope

An investigation of corrosion phenomena must be quite limited if useful information is to be obtained in a reasonable length of time. Long-time corrosion tests such as those carried on by The American Society for Testing Materials and The British Iron and Steel Institute often involve several years. The accelerated tests are of great utility to study corrosion as long as it is kept in mind that they are accelerated and much caution is used when correlation is made with actual service conditions.

One type of material was used throughout this investigation. This was reactor-grade zirconium and was selected in preference to a very high-purity material, such as that made by the crystal bar method, which at present cost is not likely to find industrial application. While the material was tested in both the hot-worked and the annealed condition, emphasis is placed on the annealed material because there is less chance

of retaining residual stresses. For the same reason no testing was done on material in the cold-worked condition. The mechanical and thermal history of a material often play a major role in stress corrosion but the objectives of this investigation required the measurement of the stress in the test specimens and this is difficult when residual stresses are present.

All specimens were tested in aqueous ferric-chloride solution except for some survey work in a preliminary investigation where a sodium-chloride solution was used. The concentration of the ferric-chloride solution was varied between 3 and 25 weight percent. For this investigation stresses were varied from the yield strength of the material to the unstressed condition.

All testing was done at room temperature because of simplicity in constructing the testing apparatus. Testing was done with and without air aeration of the solution with emphasis on the aerated solution.

#### Background On Stress-corrosion Cracking

"Stress corrosion" means the combined effect of stress and corrosion on the behavior of metals. In the field of corrosion this subject is broken down by the type of stress and the behavior observed. If the stress is cyclic then the behavior is classed as "corrosion fatigue" or "fretting corrosion". If the stress is static then it is termed stress corrosion unless the result of the combined action leads to

cracking or embrittlement of the metal. It is then classed as "stress-corrosion cracking". This is classified further by the nature of the cracks with respect to the internal structure of the metal. If the cracks follow the grain boundaries of the metal, it is intercrystalline cracking and if they propagate through the grains, it is transcrystalline cracking.

A decrease in strength of the metallic structure may be induced by intercrystalline corrosion without the additive action of stress. This type of corrosion is found in high-chromium and chromium-nickel steels and in a number of the light alloys. Such behavior is a case of selective corrosion where the grain boundaries are susceptible; the grain is not attacked. Because stress is not a factor, other than in the sense of a deformed crystal structure at the boundaries, it is not considered stress corrosion.

Almost all the metals and alloys are susceptible to stress corrosion cracking, but for each there exists only certain specific media in which it takes place. The character of the cracking may differ according to the specific medium. For example, carbon steels in sodium and potassium hydroxide and in alkaline and alkaline earth nitrates display intercrystalline cracking, while in hydrocyanic acid transcrystalline cracking takes place (16, p. 24).

Dix (18, p. 575) has proposed a general electrochemical theory on the mechanism that accounts for both inter and transcrystalline

cracking and explains why cracking is observed only in specific corrosive media for a particular metal or alloy. He states that first there must exist in the alloy a susceptibility to selective corrosion along more or less continuous paths, such as grain boundaries, slip planes, or planes of precipitated constituents. In addition the phase or material forming the continuous path must be anodic in the specific corrosive medium to the areas composing the major part of the structure. Next, there must exist a condition of high stress acting in a direction tending to pull the metal apart along these continuous paths. It is interesting to note that no cases of stress-corrosion cracking have been reported where compressive stresses are acting and that compressive stresses generated on the surface of a tension member, by peening, swaging, or some other method, have actually been used to prevent stress corrosion cracking (18, p. 574-575).

If the two conditions exist together and the material is exposed to the corrosive environment producing the specified potential relations, corrosion will start along the anodic paths, forming notches that concentrate stress at their roots.

If the material has a protective coating such as an oxide film considerable time may elapse before the film is penetrated and actual attack of the metal begins. When this does happen small areas, where the film is penetrated, are anodic and concentrate the current from

the relatively large cathodic areas of material still protected by the film. The large current density will promote a very rapid advance of corrosion along the anodic paths. Thus, homogeneous metals can also be susceptible to corrosion cracking.

As corrosion continues along the anodic path the concentration of stress becomes more severe and fissures will begin to develop, destroying any protective film and continuously exposing fresh anodic material to the corrosive. With corrosion proceeding more rapidly the stress will increase at an accelerated rate until the metal fails or the acting stress is relaxed.

Swann and Nutting (17) have discussed a dislocation model to explain stress corrosion along stacking faults and slip planes. Under stress these regions deform first and thus acquire a much higher dislocation concentration which aids the chemical attack of the corrosive atmosphere. The model is illustrated in Figure 1. In (a) dislocations from several parallel slip planes are piled up at a barrier such as a stacking fault near the specimen surface. Chemical attack along the barrier allows these dislocations to run out as shown in (b) and (c) leaving the dislocation free, brittle stage (d). This type of stress corrosion would go slowly during the chemical stage and then change to a rapid brittle stage of cracking.

The above discussion has pointed out that stress not only serves to cause fissures in the metal at the root of anodic notches exposing fresh material to chemical attack but that it can also provide high concentrations of dislocations at barriers which are more susceptible to attack than to adjacent metal.

Stress can also be responsible for forming anodic paths in metals that are not inherently resistant to corrosion but are protected by an impervious oxide film. Such films are generally in compression because they have larger atomic volumes and generally have brittle properties. If a tensile stress is imposed on the material while it is simultaneously exposed to an aggressive environment the oxide film will be broken, exposing unprotected material, and corrosion will proceed in these areas. If the metallic surface is anodic with respect to the surface having an intact oxide film corrosion can proceed very rapidly.

Some observations have shown that the time for complete failure by cracking is independent of the time the stress was applied (15, p. 5). This implies that the only role of stress is to mechanically separate the metal shortly before fracture by supplying the necessary stress concentration. This could be true in some systems but in view of other evidence it is not a generality.

The magnitude of stress required to induce stress-corrosion cracking is generally above the design working stress. However, the

internal stresses generated from volumetric changes in welding or thermal treatment and residual stresses from cold-forming operations are often sufficiently high to promote cracking. The superposition of the applied stress on the internal stress has been observed to cause destruction (16, p. 28).

The obscure nature of internal stress in structural materials has been a major difficulty in predicting the possible occurrence of stress-corrosion cracking by utilizing the results of laboratory tests.

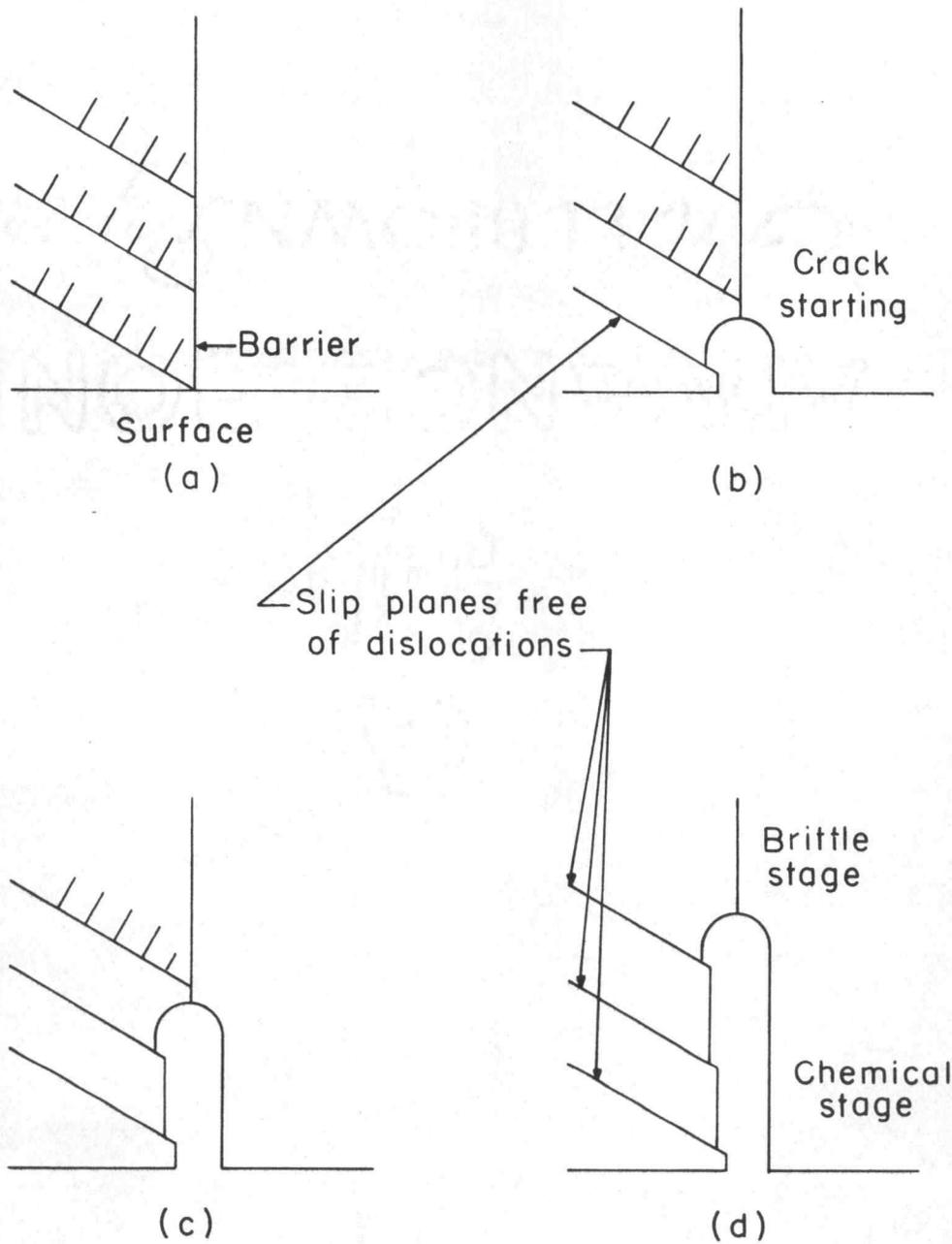


Figure 1.—Dislocation model showing stress corrosion along stacking faults and slip planes

## EXPERIMENTAL TECHNIQUE

### Apparatus For Testing

The constant load method for stress-corrosion cracking susceptibility has two distinct advantages. One - the stress on the specimen can be determined directly and accurately and, two - any elongation or plastic flow of the specimen does not relax the stress. To determine qualitatively whether or not a material is susceptible can be accomplished much easier by the constant strain method of imparting stress to the test methods. This is usually done by bending the specimen and exposing it to the corrosive environment while held in the bent position. Numerous ways of accomplishing this have been devised by various investigators, (11, p. 233-254) (16, p. 206-241). A fifty-percent loss in original stress has been reported by one investigator (16, p. 209) using the constant strain method.

Preliminary tests in this investigation were made by using the constant strain method of inducing stresses in the specimen. The first method used was the hoop method. This was done by bending the ends of a strip around a mandrel into a horseshoe-shaped hoop and then confining the ends after straining. The strained hoop with tensile stresses acting on its outer surface was immersed in the corrosive medium for test. Four specimens were tested in this manner; one each in 5 percent NaCl, 10 percent NaCl, 10 percent FeCl<sub>3</sub>, and 25 percent FeCl<sub>3</sub> solutions. Only the specimen tested in 25 percent FeCl<sub>3</sub> solution indicated any signs of cracking.

Small pits on the edge of the specimen in tension were observed which could have been cracks that developed into pits after forming.

Although useful for qualitative determination of susceptibility, the magnitude of the stress acting on the specimen is uncertain. For this reason no more tests were made using hoop-shaped specimens.

The next method used to induce stress by constant strain was with a direct tension device. Tensile-type specimens were fixed at one end in a rigid frame and the other end gripped by a threaded fixture passing through a hole in the upper member of the frame. A known load was placed on the specimen in a tensile-testing machine and a nut tightened so the load would be retained in the specimen by the frame. Relaxation of the specimen, frame or fasteners was found to occur, consequently, the constant-strain method was abandoned and an apparatus for testing by the constant-load method of inducing stress was constructed.

The apparatus is quite simple; a load is applied to the specimen which passes through a vessel holding the corrosive medium. Figure 2 is a schematic of one of four setups used and Figure 3 is a photograph of two of the setups loaded. Each setup was constructed so the same load could be applied to two specimens. The details of the clamps holding the upper end of the upper specimen and the lower end of the lower specimen are shown in Figure 4. The details of the clamp between the specimens are shown in Figure 5. The clamps were made of Zircaloy-2. Stainless steel

was tried and found unsatisfactory in the atmosphere around the ferric-chloride solution; severe pitting was observed within a few hours.

The rod supporting the upper end of the upper specimen also supports the vessels for holding the corrosive solution and a microswitch which was used to stop an hour-meter when the specimen failed. The microswitch was positioned by an adjustable clamp.

The corrosion vessels were made from Vycor glass tubing. A rubber boot was used for the bottom of the vessel. It was held to the vessel by a rubber band which seated the boot in a groove ground around the vessel 1/2 in. from the bottom. It was held to the specimen by tying with string. The boot not only provided the bottom of the vessel but also made the seal between the specimen and the vessel. This seal must be inert with respect to the corrosion solution, allow replacement of specimens, and not impart any stress to the specimen. Rubber stoppers have been used successfully with round specimens but the rectangular specimens used in this investigation caused leakage to occur when they were tried. A plicene cement, with low-melting temperature was tried with little success because it appeared to react with the ferric-chloride solution. An epoxy cement was also tried but cracked on hardening with leakage resulting. The rubber boot when properly tied to the specimen gave no trouble and did not react with the solution.

Some of the tests were conducted with the corrosive solution aerated with air. The air was obtained from a compressor and filtered through fiber

glass to remove suspended solids. It was then bubbled through a 10 percent solution of sodium hydroxide to remove carbon monoxide, carbon dioxide, and sulfur compounds (2, p. 888). Carbon monoxide and carbon dioxide can form carbonic acid when passed through aqueous solutions. The formation of any acids would change the solution being used as the corrosive. After passing through the sodium-hydroxide solution the air was passed through a water wash and metered through flow meters at 196 cubic centimeters per minute. This flow rate is recommended to insure complete air saturation for the volume of solution used (2, p. 888). Aerators were made by drawing one end of 8 mm glass tubes to capillary size. These provided a very fine steady stream of air bubbles passing through the solution. The aeration system is shown in the photograph of Figure 6 and schematically in Figure 7.

#### Material

Impurity elements at the residual level have been shown to significantly change the corrosion behavior of zirconium and Zircaloy alloys (9, p. 616). Consequently it is important to include impurity analysis in a discussion of corrosion behavior.

The specimens numbered 1 through 26 and P-1 to P-6 were made from a zirconium ingot made at the U. S. Bureau of Mines, Albany, Oregon and designated there as ingot No. 57-D-361. This ingot was made by a sponge-blending process that varied from that recommended for reactor-grade zirconium. (3)



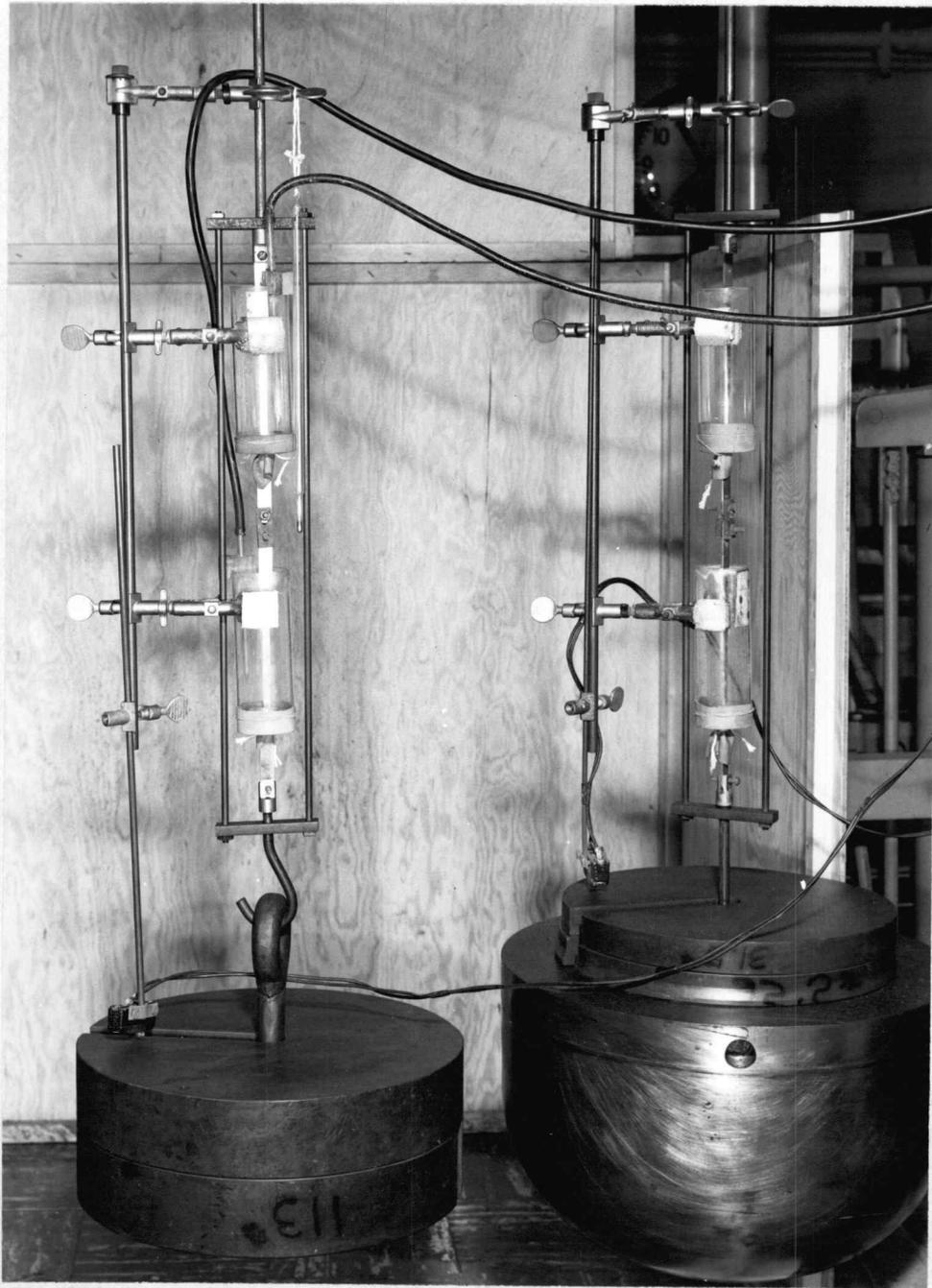


Figure 3. - Photographs of two setups used in stress-corrosion tests.

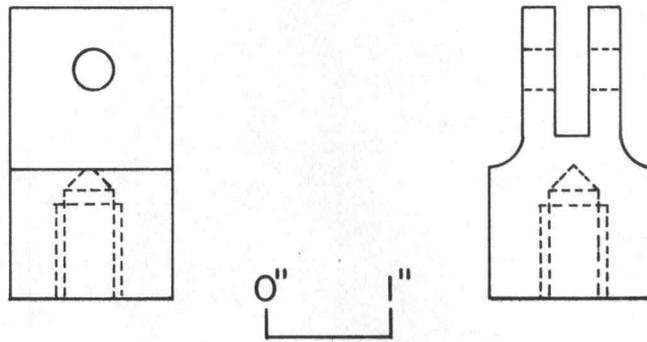


Figure 4.— Specimen clamp

Note:

2 Pieces per clamp

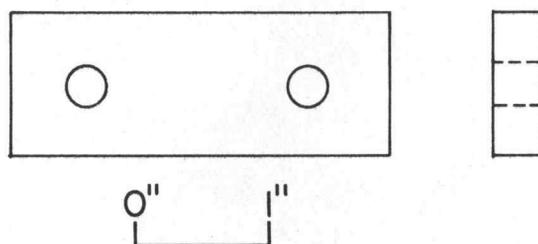


Figure 5.— Specimen clamp

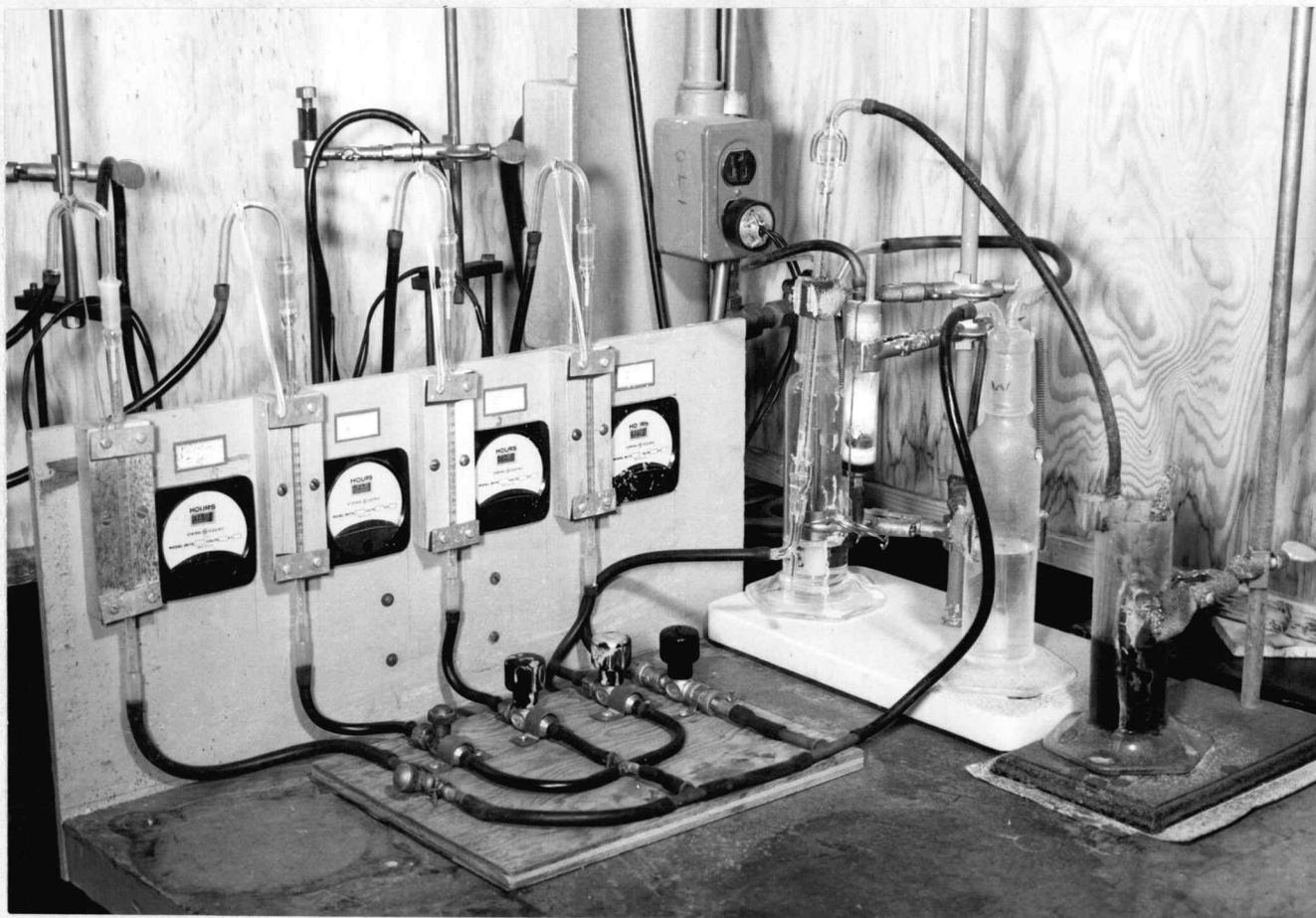


Figure 6. - Photograph of aeration system used in stress-corrosion tests.

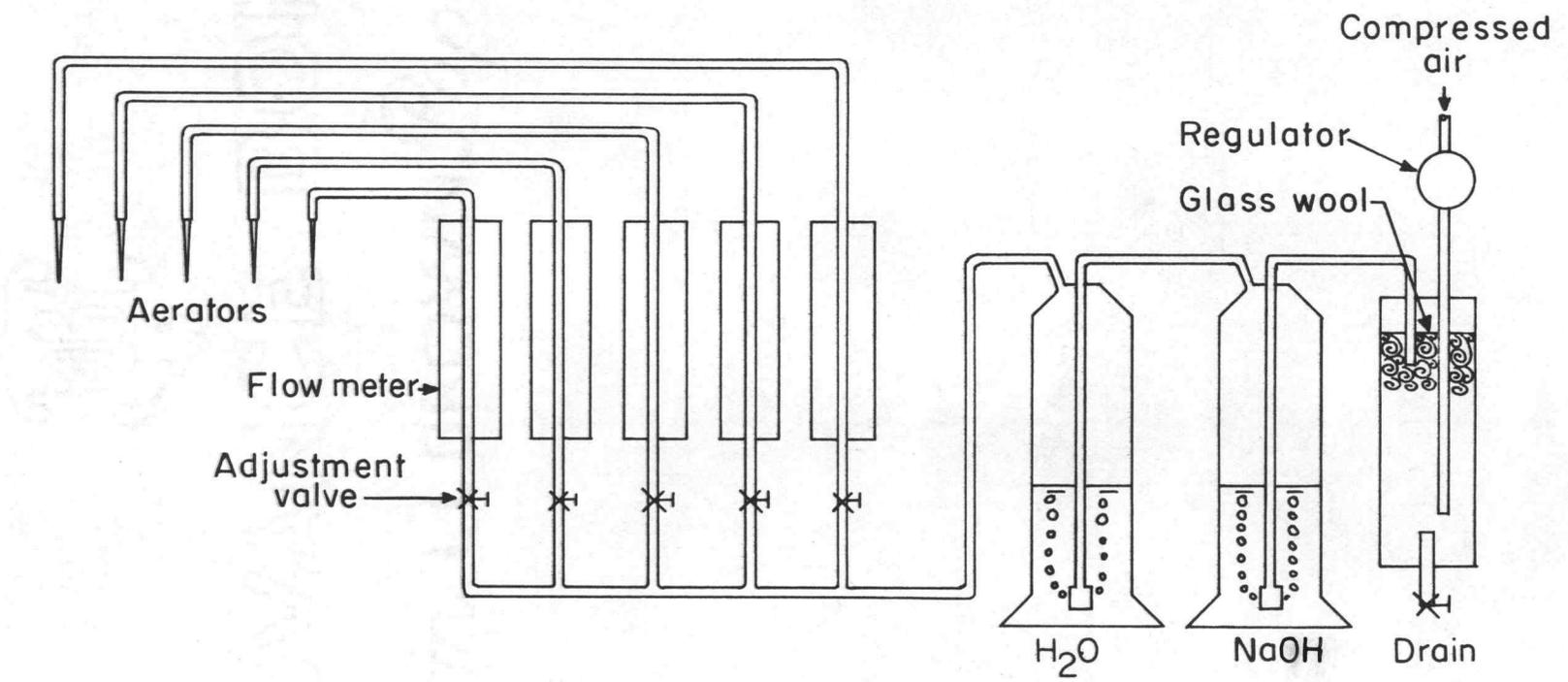


Figure 7.-Aeration system

The remaining specimens were made from a similar ingot, also made at the U. S. Bureau of Mines, Albany, Oregon and designated there as ingot No. 57-D-452. It was processed according to specifications for reactor-grade material. (3)

Both ingots were made from Kroll-processed zirconium sponge by consumable arc melting twice in vacuum. The average impurity analysis of these ingots is shown in Tables 1 and 2 (4, table 3). Iron is the only impurity that appears significantly different in the two ingots being higher in ingot No. 57-D-361.

Both ingots were press forged to 1-3/4 in. slab at 840 C., then hot rolled at 840 C. to 1 in. thickness, surface conditioned by sand blasting and grinding with a No. 36 grit abrasive sleeve and then hot rolled at 840 C. to 1/4 in. The 1/4 in. plate was surface conditioned by sand blasting and grinding with a No. 60 grit abrasive sleeve. Material from ingot No. 57-D-361 was then hot rolled at 840 C. to approximately 0.070 in. thickness and that from ingot No. 57-D-452 was hot rolled to 0.085 in. at the same temperature. All material was then surface conditioned by sand blasting, pickling in a HNO<sub>3</sub>, HF, and water solution, and grinding with a No. 80 grit abrasive sleeve. This was the final condition of the sheet from ingot No. 57-D-361 with an approximate thickness of 0.065 in. The sheet from ingot No. 57-D-452 was reduced from its thickness of approximately 0.079 in. to 0.063 in. by cold rolling. Specimens were machined from these sheets to the dimensions shown in Figure 8.

This type of specimen is a modified version of the ASTM standard rectangular tension test specimen for sheet metals ranging in thickness from 0.005 in. to, but not including, 1/2 in. (10, p. 87). This type of specimen was selected because the material was in sheet form and could be modified by increasing the reduced section and gauge section length to allow the gauge length section to pass completely through the corrosion vessel. Specimens from the sheets fabricated from ingot No. 57-D-452 were vacuum annealed for one hour at 800 C. in a vacuum of less than  $10^{-5}$  mm of mercury.

An additional impurity analysis was made on the material from ingot No. 57-D-361. This was done after the material had been worked to a 1/4 in. plate. This was done as a check on possible contamination of the material during fabrication. The values are shown in Table 3. Comparison of these values with the average ones for the ingot in Tables 1 and 2 shows considerable difference but no serious contamination. Consequently it was felt unnecessary to make an additional analysis on the plate from ingot No. 57-D-452 which was fabricated under the same conditions.

Summarizing, the specimens, numbered 1 through 26, represent hot-rolled sheet from zirconium ingot No. 57-D-361 and the rest, No. 30 and greater, represent cold-rolled, annealed sheet from zirconium ingot No. 57-D-452.

TABLE 1. - Analysis of impurities of zirconium ingot 57-D-361.

<u>Average - parts per million</u>	
O	Si
-	20
N	Mg
30	20
Hf	Ti
-	<50
Cd	Zn
-	<5
B	Ni
0.5	<5
Al	Mn
50	<10
Fe	Mo
500	<10
Cu	V
175	<20
Pb	Co
50	<10
Cr	Sn
<5	75
C	
130	

TABLE 2. - Analysis of impurities of zirconium ingot 57-D-452.

<u>Average - parts per million</u>	
O 1316	Si 100
N 40	Mg 20
Hf -	Ti <50
Cd -	Zn <50
B 0.5	Ni 10
Al 30	Mn 10
Fe 300	Mo -
Cu 50	V <20
Pb 50	Co <10
Cr 50	Sn 110
C -	

TABLE 3. - Analysis of impurities of zirconium plate from ingot 57-D-361.

<u>Parts per million</u>	
O <u>1/</u> 1250	Si <u>5/</u> 110
N <u>2/</u> 40	Mg <u>5/</u> <10
Hf <u>3/</u> 110	Ti <u>5/</u> <20
Cd <u>4/</u> <0.5	Zn <u>4/</u> <50
B <u>4/</u> 1.5	Ni <u>5/</u> 20
Al <u>5/</u> 150	Mn <u>5/</u> 15
Fe <u>5/</u> 960	Mo <u>4/</u> <10
Cu <u>5/</u> 170	V <u>4/</u> <20
Pb <u>4/</u> 35	Co <u>4/</u> <5
Cr <u>5/</u> <40	Sn <u>4/</u> <35

1/ Determined by inert gas fusion method with Leco equipment.

2/ Determined by Kjeldahl wet method.

3/ Determined by spectrograph of oxide buffered with Barium fluoride.

4/ Determined by spectrographic carrier distillation method.

5/ Determined by spark analysis of briquetted oxide.

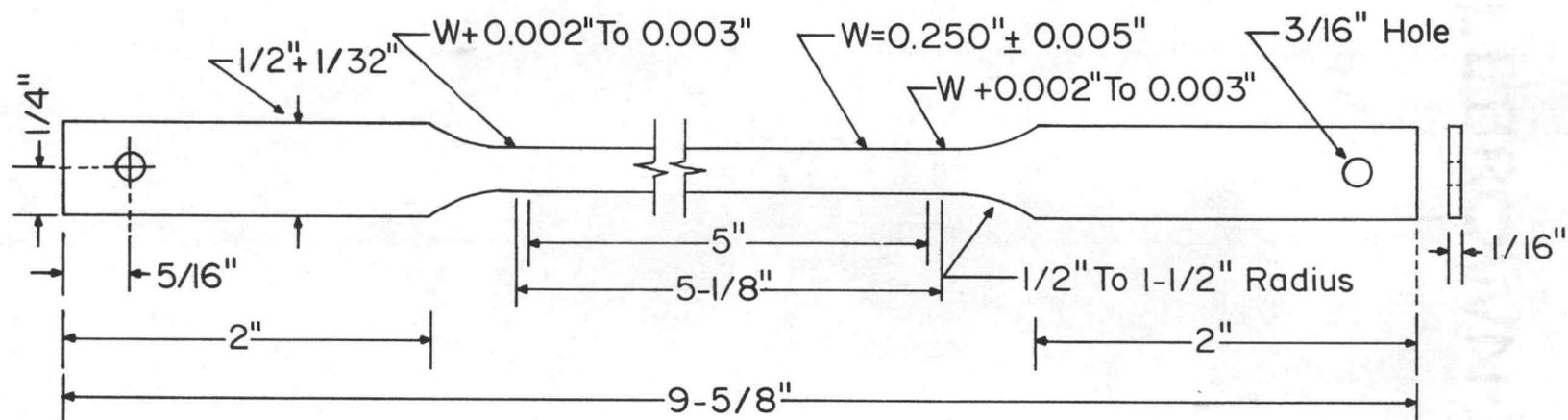


Figure 8.— Dimensions of corrosion specimens

### Mechanical Properties

The mechanical properties of the material used in this investigation were determined primarily to obtain the yield strength so the range of stress levels used in the stress-corrosion tests could be selected. Loading a specimen until it was stressed above the yield strength would plastically deform the specimen thereby changing its mechanical structure. Too small a load might result in a stress too far below the yield strength to have any effect on the corrosion behavior. This has been observed in other systems and has introduced the concept of a so-called "threshold stress" similar to that observed in the fatigue of metals. Only when the stress is above the threshold level does it influence the corrosion behavior of the material. With a known yield strength, then, loads could be adjusted so the stress in the specimen would not cause plastic yielding but still be high enough to promote cracking if the material was susceptible.

Verification of the material's ductility was also desirable because if a specimen should fail by cracking or embrittlement it could be attributed to either the corrosive environment, stress, or a combination of both.

The tensile properties are shown in Table 4. The specimens numbered P-1 to P-6 represent the material from ingot No. 57-D-361 and specimens numbered P-7 to P-14 represent material from ingot No. 57-D-452. The specimens used to determine these properties are the same type

TABLE 4. - Mechanical properties of zirconium specimens.

<u>Specimen</u>	<u>Ultimate tensile stress psi</u>	<u>Yield stress 0.2 percent offset - psi</u>	<u>Percent elongation in 2 in.</u>
P-1	67,700	40,600	20.5
P-2	61,900	41,800	25
P-3	61,600	37,300	27
P-4	61,600	39,400	22.5
P-5	61,800	39,200	25
P-6	<u>62,500</u>	<u>39,800</u>	<u>25</u>
Average	62,850	39,700	24.2
P-7	61,960	-	28
P-8	63,560	38,650	27
P-9	62,530	38,250	
P-10	61,820	42,240	24
P-11	61,860	38,200	25
P-12	61,570	37,950	25
P-13	60,840	37,650	22.5
P-14	<u>61,600</u>	<u>37,730</u>	<u>23</u>
Avg. of 8	61,968	Avg. of 7	38,670
		Avg. of 7	24.9

and in the same condition as those used in the stress corrosion tests. The dimensions are shown in Figure 8.

### Surface Preparation of Specimens

Corrosion takes place at the interface of the specimen surface and the corrosive environment. The preparation or treatment of the specimen surface prior to testing is, therefore, of utmost importance. Grease, dirt, any foreign matter, surface films, or varying degrees of surface cold work (the Beilby layer) can be responsible for considerable variation in corrosion test results. The effect of organic grease when testing in aqueous solutions is quite obvious but the effect of other surface contaminants or conditions, such as the depth of oxide film or the degree of surface cold work, is not so easily discerned. To eliminate variation caused by surface differences then, it is necessary to treat each specimen consistently. This can be done by thoroughly cleaning the surface and pickling until the surface is representative of the internal material.

The specimens for this investigation were first scrubbed in hot soapy water, followed with a rinse in hot flowing tap water and then dried in air. The specimens were then degreased in trichlorethylene, rinsed in ethyl alcohol, and air dried. The clean surface was then pickled to remove oxide and the disturbed surface metal in a manner similar to that specified for zirconium and Zircaloy alloys by the Bettis Plant of Westinghouse Electric Corporation (6, p. 8-9). The pickling solution was as follows:

Nitric acid, sp gr 1.42 . . . . . 39 volume percent  
 Hydrofluoric acid, 48.6 percent. . . 3.5 volume percent  
 Tap water. . . . . Balance

Each specimen was pickled until 0.002 in. was removed from each side. After pickling, the specimens were immediately rinsed in flowing tap water for five minutes, transferred through a distilled water bath, and allowed to dry in room air.

The ends of specimens 1 through 23 were coated with paraffin wax leaving a 2 to 3 in. long section in the middle for exposure to the corrosion solution. This made it possible to measure the exposed area and determine the corrosion rate in terms of weight loss per unit area. The weight loss was determined by weighing the specimen on a Gramatic balance to the nearest tenth of a milligram before and after test. By waxing the ends of the specimen the immersion line effect was also eliminated. This effect is caused by a gradient in the amount of air in a static solution. The quantity of dissolved air is greatest at the air-solution interface and decreases with distance from the solution surface. Oxygen is generally very important in corrosion reactions and a larger quantity of dissolved air means more oxygen available for the reaction. By immersing the exposed, or unwaxed portion of the specimen completely below the surface of the solution no immersion line was encountered. Nelson, Moore, and Quinn (13, p. 15) investigated the oxygen gradient in static solutions of

sea water and concluded that natural occurring currents in the corrosive media caused by convection eliminate the oxygen gradient from the immersion line. LaQue and Knapp (8, p. 4-5) have pointed out that localized corrosion at a well defined immersion line seldom occurs where the liquid is at rest. However, the ferric-chloride solution used in this investigation did wet the specimens causing a very thin film of corrosion solution just above the solution level where a high degree of natural aeration could take place.

On some specimens failure occurred in the waxed portion of the specimen. This was apparently the result of small holes in the wax coating. If the area exposed by the hole was anodic with respect to the rest of the exposed specimen a very high current density would exist at the anodic area. Under such conditions corrosion proceeds very rapidly.

Specimens numbered greater than 23 were not coated with wax on the ends. No significant weight losses were detected on the specimens that were waxed so it was unnecessary to have a known area exposed. All specimens numbered greater than 23 were tested in an air-aerated solution so immersion line effects were eliminated. Elimination of waxed ends also eliminated the possibility of having small anodic areas resulting from holes in the coating.

### Test Solutions

All testing was done in various concentrations of aqueous ferric-chloride solution. The concentrations used were 25, 20, 15, 10, 5, and 3 percent by weight. The solutions were made from laboratory grade  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and distilled water.

The desired concentration was obtained by weighing to the nearest 0.1 gram. Solutions were mixed in two liter batches.

The corrosion vessel for each test held 200 ml of solution and was maintained at this volume throughout the test by the addition of distilled water. This replaced evaporation losses. It is recommended that the volume of test solution should be large enough to avoid any appreciable changes in its corrosiveness either through the exhaustion of corrosive constituents, or the accumulation of corrosion products or other constituents that might affect further corrosion (2, p. 888-889). Two hundred milliliters were found satisfactory for all tests.

### Resistance Measurements

The extent of cracking is difficult to compare by visual observations. After testing the specimens for susceptibility it appeared that the extent of cracking followed some trend related to the variation of stress or solution concentration.

The electrical resistance of a material is proportional to the length and inversely proportional to the average cross-sectional area.

If the material is assumed to be homogeneous throughout the specimens and a constant length taken on each specimen, any change in resistance results from a change in the average cross-sectional area. If the average cross-sectional area of the specimen is known before corrosion testing then any variation in resistance detected after the corrosion test will represent the extent of the corrosion attack and in this case the extent of cracking.

The resistance of each specimen was not measured before testing so to use a change in resistance as a measure of the extent of cracking it was necessary to use a specimen that had not been corroded and compare the resistance of those that had been corroded with it. This was done for two groups of specimens representing the cold-rolled annealed sheet from ingot No. 57-D-452. Only the specimens with the same initial cross-sectional area were measured. The resistance was determined with a Kelvin double bridge to the nearest microhm over a 2 in. length.

## TESTS AND RESULTS

Results of Survey Testing Hot-rolled Sheet

The first group of specimens was tested as a survey under the conditions shown in Table 5. These specimens represent hot-rolled sheet from ingot No. 57-D-361. Two concentrations of  $\text{FeCl}_3$  solution, a low concentration of 10 percent and a high concentration of 25 percent, were used. Two specimens were loaded to approximately the same stress level and one exposed to the low concentration and the other to the high concentration. In this way both the effect of stress and effect of solution concentration could be shown. Specimens No. 9 and No. 10 were used as controls. No.9 was stressed to 35,000 psi in room air and No. 10 was exposed to a 25 percent  $\text{FeCl}_3$  solution without stress.

Two of the specimens stressed at the higher levels, No. 13 and 14, fractured as a result of cracking; one in a 10 percent and one in a 25 percent solution concentration. Specimen No. 3 had several very fine cracks normal to the direction of stress after 216 hours of exposure. A typical example of these cracks is shown in the photomicrograph of Figure 9. This specimen was tested under the same conditions as No. 14 except it was under a slightly lower stress. No. 4, loaded to 35,000 psi in a 10 percent solution, did not show any evidence of corrosion after 218 hours in test, but No. 13 at a slightly lower stress, 33,000 psi, and also in a 10 percent solution fractured after 353 hours in test. The other specimens

in this group, except those used for the controls, showed slight localized corrosion similar to that in the photomicrograph of Figure 10.

Results of Testing Hot-rolled Sheet with  
Variation of Applied Stress

The results of the survey group did show the hot-rolled material susceptible to stress corrosion cracking at stresses near the yield strength. The results indicated cracking would take place in less time in solutions of higher concentration.

A group of specimens of the same material in the same condition were tested in 25 percent  $\text{FeCl}_3$  solution with the stress varied between 35,000 and 27,000 psi. The conditions and results of this group are shown in Table 6. Four of the six specimens tested fractured because of cracking. The corrosion vessel containing specimen No. 20 developed a leak at an unknown time. The test was terminated when this was discovered. The stress on specimen No. 19 was relaxed at an unknown time and this test was also terminated.

The four specimens that did fracture show a definite trend between stress level and time to fracture; time to fracture increasing with decrease in stress. Numerous fine cracks appeared on the surface of these specimens normal to the direction of stress. The photomicrograph, Figure 11, shows a typical sample of these cracks.

Results of Testing Hot-rolled Sheet with Variation  
of Applied Stress in Air Aerated Solutions

It is recommended that corrosion test solutions be aerated for reproducible results even if air-free solutions are desired, in which case an inert gas should be used (2, p. 887-888). To investigate this, three specimens with different applied stresses were tested in air aerated solutions of 25 percent  $\text{FeCl}_3$ . The specimens were of the hot-rolled sheet material from ingot No. 57-D-361. The results are shown in Table 7. All three specimens fractured by cracking. The same effect of stress on time to fracture is shown; increased time to fracture with decreased stress. All cracks observed were normal to the direction of stress. A typical crack is shown in the photomicrograph of Figure 12. It was also obvious that aeration of the solution reduced the time to fracture under the same conditions of those of a non-aerated solution. Less localized corrosion was noted on these specimens than those tested in non-aerated solutions, probably because of the shorter exposure time.

Results of Testing Cold-rolled Annealed Sheet with Variation  
of Applied Stress and Air Aerated Solutions

The results of testing the specimens representing hot-rolled sheet from ingot No. 57-D-361 indicated aeration with air to be a more severe test; cracking occurred in less time. For this reason the specimens representing cold-rolled annealed sheet from ingot No. 57-D-452 were all tested in air aerated solutions. The first group of specimens of this

material was tested in a 25 percent  $\text{FeCl}_3$  solution with variation in applied stress. The conditions and results are shown in Table 8. The specimens after test are shown in the photograph of Figure 17. All specimens failed by cracking. The time for fracture was shown to increase with a decrease in applied stress. Cracking was observed to be intercrystalline and normal to the direction of stress. Figures 13, 14, 15 and 16 show photomicrographs of typical cracks in four of these specimens.

Results of Testing Cold-rolled Annealed Sheet with  
Variation in Solution Concentration and Air Aerated Solutions

To determine the effect of solution concentration on susceptibility, a group of specimens was tested at a constant stress with a variable solution concentration. These specimens represent cold-rolled annealed sheet from ingot No. 57-D-452 and were tested in air aerated solutions. The conditions and results are shown in Table 9. The specimens after test are shown in the photograph of Figure 18. One specimen, No. 31, which was tested at a lower stress is included in the table and photograph. It was a specimen from the group where stress was varied but is included in this series to show the effect of the higher concentration.

All specimens failed by cracking; the time for fracture increased with decreasing concentration. Cracking was observed to be intercrystalline and normal to the direction of stress.

### Results of Electrical Resistance Measurements

Electrical resistance measurements were made to show the extent of cracking on the two groups of specimens representing cold-rolled annealed sheet from ingot No. 57-D-452. These results are shown in Tables 10 and 11. The change of resistance per hour seems to rise to some maximum and then fall as the stress is reduced. This agrees with the visual appearance of the specimens. Those tested at high stresses fractured in a few hours. Cracking was sparse over the test area but what cracks were there were severe. At the low levels of stress the same pattern was observed only the cracks had been enlarged by a pitting action.

The specimens tested at intermediate stress levels were covered with numerous fine cracks. One specimen, No. 44, appeared to have undergone a more uniform type of corrosion although it did fail by brittle fracture.

The specimens tested at constant stress with variable solution concentration show a different trend. The change in resistance per hour decreases as the solution concentration is reduced except for specimen No. 36 tested in a 10 percent solution concentration. There is no explanation to account for this deviation from the behavior of the other specimens.

TABLE 5. - Conditions and results of survey testing in ferric-chloride solution at room temperature.

Representing hot-rolled sheet from ingot 57-Γ-361

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours</u>	<u>Results</u>
15	12,500	10	456	Slight localized corrosion
16	12,400	25	456	Slight localized corrosion
6	12,500	10	216	Slight localized corrosion
5	13,000	25	216	Slight localized corrosion
7	14,500	10	449	Slight localized corrosion
8	14,500	25	448	Slight localized corrosion
1	15,000	10	216	Slight localized corrosion
2	15,000	25	216	Slight localized corrosion
13	33,000	10	408 (353*)	Fracture by cracking; no other corrosion
14	32,000	25	97 (95*)	Fracture by cracking; Slight localized corrosion
4	35,000	10	218	No corrosion observable
3	33,000	25	218	Very fine surface cracks
9	35,000	room air	843	
10	0	25	843	Uniform corrosion

\* Number of hours in test before fracture.

TABLE 6. - Conditions and results of testing in ferric-chloride solution at room temperature.

Representing hot-rolled sheet from ingot 57-D-361

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours</u>	<u>Results</u>
18	35,000	25	41* 95	Fracture by cracking; numerous cracks on all surfaces of the specimen; slight localized corrosion
17	33,000	25	36* 46	Fracture by cracking; numerous cracks on the edge that parted last
19	29,000	25	697	Slight localized corrosion; test terminated
21	28,000	25	502*	Fracture by cracking; numerous fine cracks
22	27,500	25	741*	Fracture by cracking; numerous fine cracks
20	27,000	25	625	Very fine surface cracks normal to direction of stress; slight localized corrosion; test terminated

\* Number of hours in test before fracture

TABLE 7. - Conditions and results of testing in aerated ferric-chloride solution at room temperature.

Representing hot-rolled sheet from ingot 57-D-361

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours *</u>	<u>Results</u>
24	28,000	25	82	Fracture by cracking; other cracks observable on specimen surface; slight localized corrosion
25	30,000	25	28	Fracture by cracking; other cracks observable on specimen surface; slight localized corrosion
26	31,000	25	12	Fracture by cracking; other cracks observable on specimen surface; some severe; slight localized corrosion

\* Number of hours in test before fracture

TABLE 8. - Conditions and results of testing in aerated ferric-chloride solution at room temperature.

Representing cold-rolled annealed sheet from ingot 57-D-452

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours *</u>	<u>Results</u>
31	33,000	25	3	Fracture by cracking; a few other extensive cracks extending from edges; slight localized corrosion
34	31,500	25	22	Fracture by cracking; numerous fine cracks on all surfaces
30	29,500	25	27	Fracture by cracking; numerous cracks; slight localized corrosion
38	28,000	25	14	Fracture by cracking; numerous fine cracks on all surfaces; slight localized corrosion
45	24,500	25	42	Fracture by cracking; numerous fine cracks over all surfaces of specimen

(continued)

TABLE 8. - (continued)

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours *</u>	<u>Results</u>
44	23,000	25	157	Fracture by cracking; uniform corrosion over entire surface; a few extensive cracks and pits
32	22,500	25	62	Fracture by cracking; several extensive cracks on all surfaces; slight localized corrosion
40	19,000	25	132	Fracture by cracking; other cracks present but several obscured by localized corrosion appearing in pits
41	0	25	1,600	Extensive uniform corrosion with localized pits - some extending through the specimen

\* Number of hours in test before fracture.

TABLE 9. - Conditions and results of testing in aerated ferric-chloride solution at room temperature.

Representing cold-rolled annealed sheet from ingot 57-D-452

<u>Specimen</u>	<u>Stress - psi</u>	<u>Solution concentration percent</u>	<u>Time of test, hours *</u>	<u>Results</u>
31	33,000	25	3	Fracture by cracking; a few other extensive cracks extending from edges; slight localized corrosion
35	34,000	20	16	Fracture by cracking; numerous cracks on all surfaces
36	34,000	15	14	Fracture by cracking; numerous on all surfaces; some localized corrosion
39	34,000	10	14	Fracture by cracking; some cracking on all surfaces
43	34,000	5	28	Fracture by cracking; a few additional cracks
42	34,000	3	94	Fracture by cracking; a few additional cracks

\* Number of hours in test before fracture.

TABLE 10. - Resistance measurements of specimens tested in 25 percent ferric-chloride solution with variable stress.

<u>Specimen</u>	<u>Stress - psi</u>	<u>Resistance in 2 in. - milliohms</u>	<u>Change in resistance- milliohms</u>	<u>Rate of change in resistance - microhms/hour</u>
Standard		2.431		
34	31,500	2.586	0.155	7.0
30	29,500	2.732	0.301	11.4
38	28,000	3.004	0.573	40.3
45	24,500	3.102	0.671	15.8
44	23,000	3.175	0.744	4.7
32	22,500	2.902	0.471	7.6
40	19,000	2.567	0.136	1.0

TABLE 11. - Resistance measurements of specimens tested at constant stress with a variable ferric-chloride solution.

<u>Specimen</u>	<u>Concentration - percent</u>	<u>Resistance in 2 in. - milliohms</u>	<u>Change in resistance - milliohms</u>	<u>Rate of change in resistance - microhms/hour</u>
Standard		2.431		
31	25	2.530	0.099	31.0
35	20	2.675	0.244	15.0
36	15	2.691	0.260	19.0
39	10	2.481	0.050	3.6
43	5	2.460	0.029	1.0
42	3	2.546	0.115	1.2

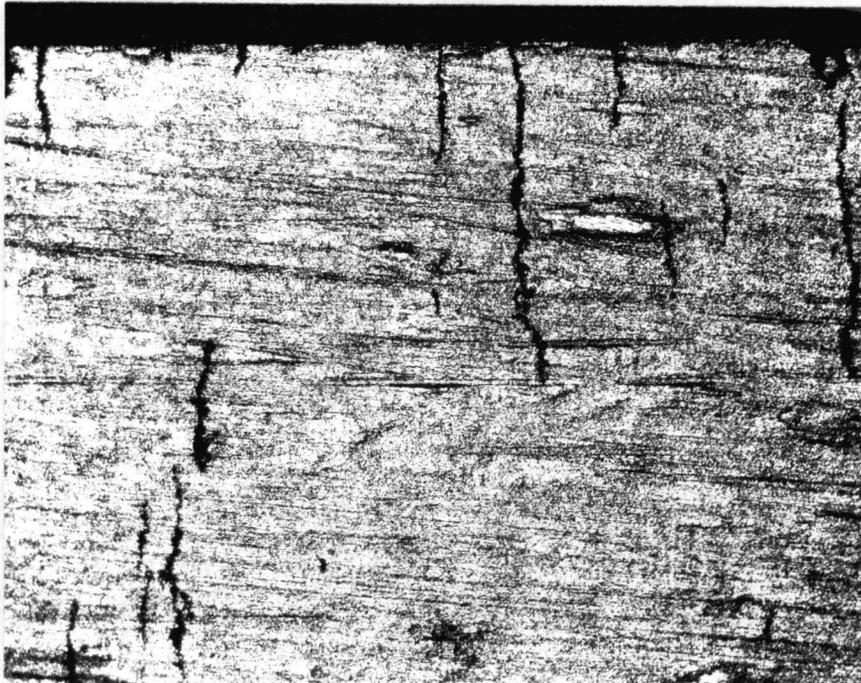


Figure 9. - Photomicrograph showing cracks normal to the direction of stress in specimen No. 3. (50 X)

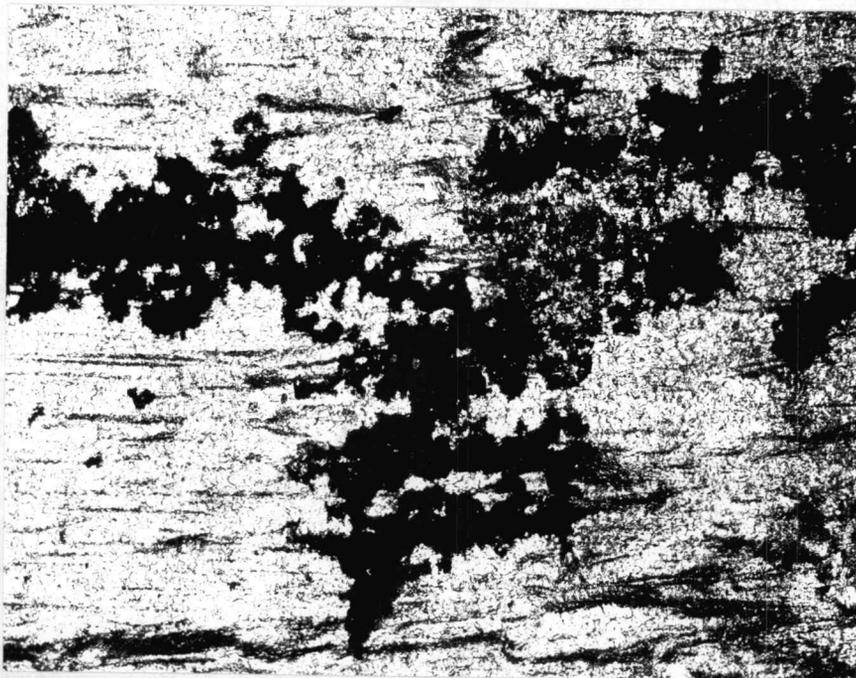


Figure 10. - Photomicrograph showing localized corrosion on the surface of specimen No. 5. (50 X)

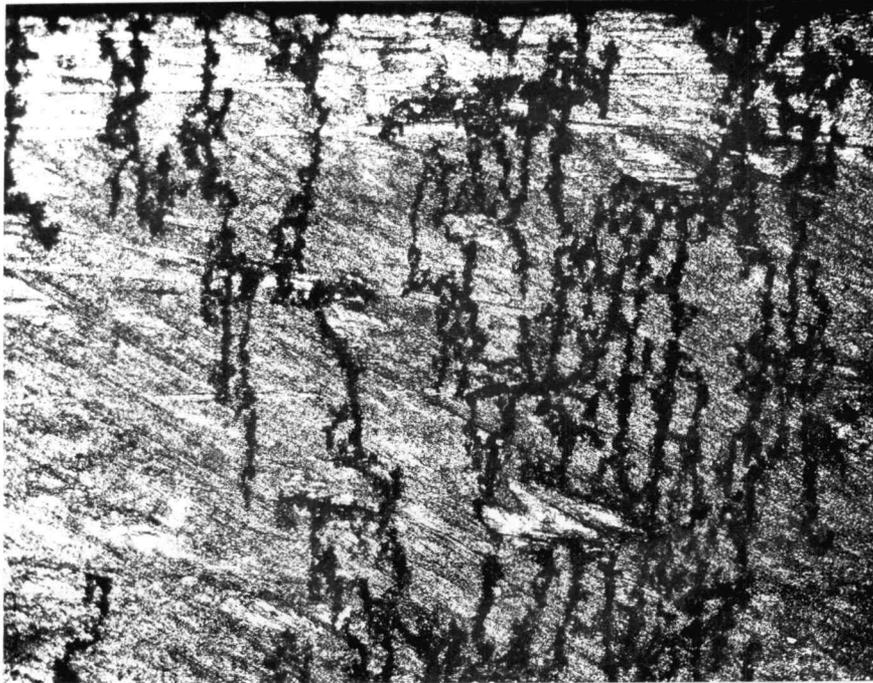


Figure 11. - Photomicrograph showing numerous fine cracks on the surface of specimen No. 21. (50 X)



Figure 12. - Photomicrograph showing a crack normal to the direction of stress in specimen No. 26. (50 X)

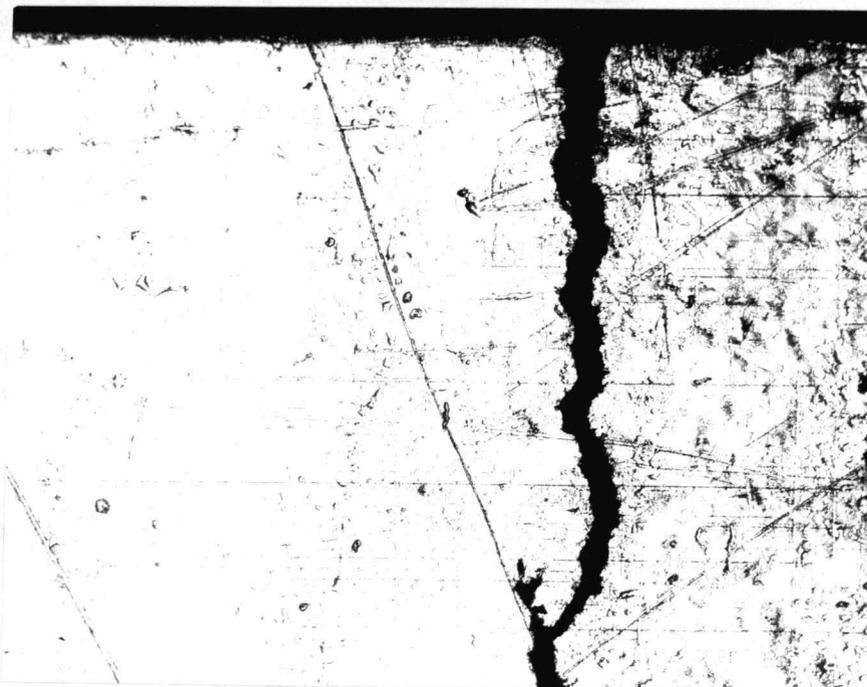


Figure 13. - Photomicrograph showing a typical crack in specimen No. 31. (50 X)

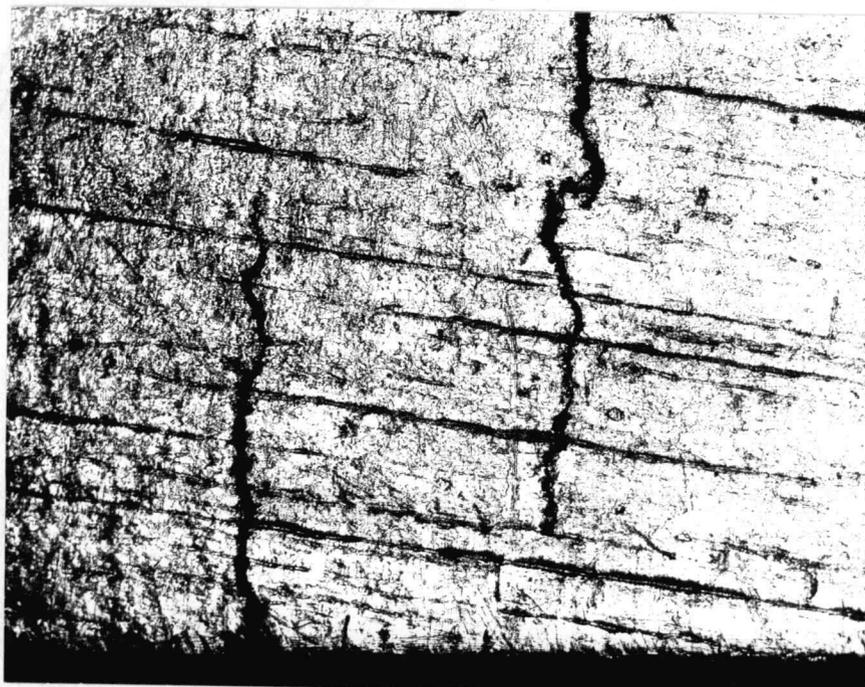


Figure 14. - Photomicrograph showing typical cracks in specimen No. 34. (50 X)



Figure 15. - Photomicrograph showing typical cracks in specimen No. 30. Some localized corrosion is also shown. (50 X)

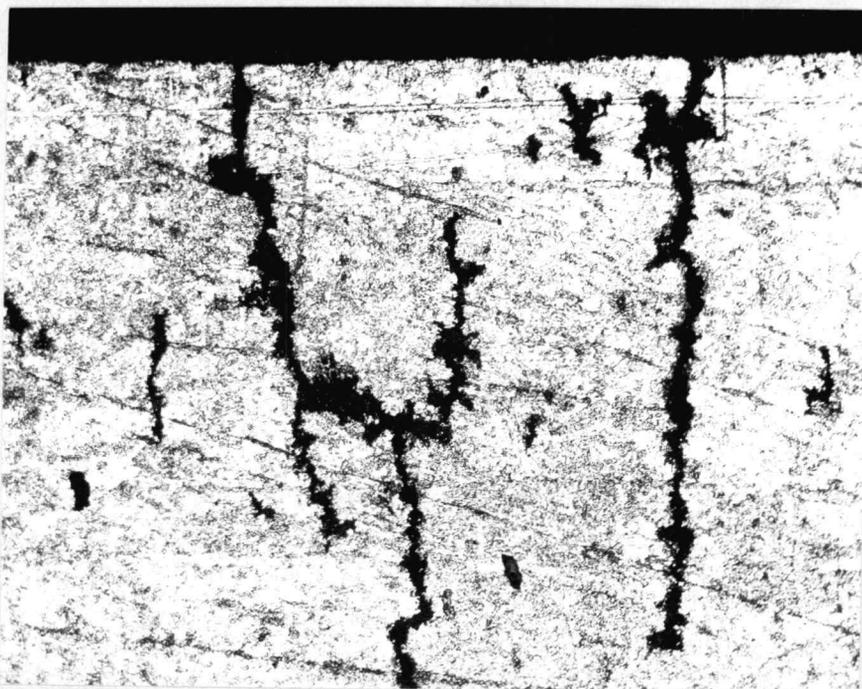


Figure 16. - Photomicrograph showing typical cracks in specimen No. 45. (50 X)

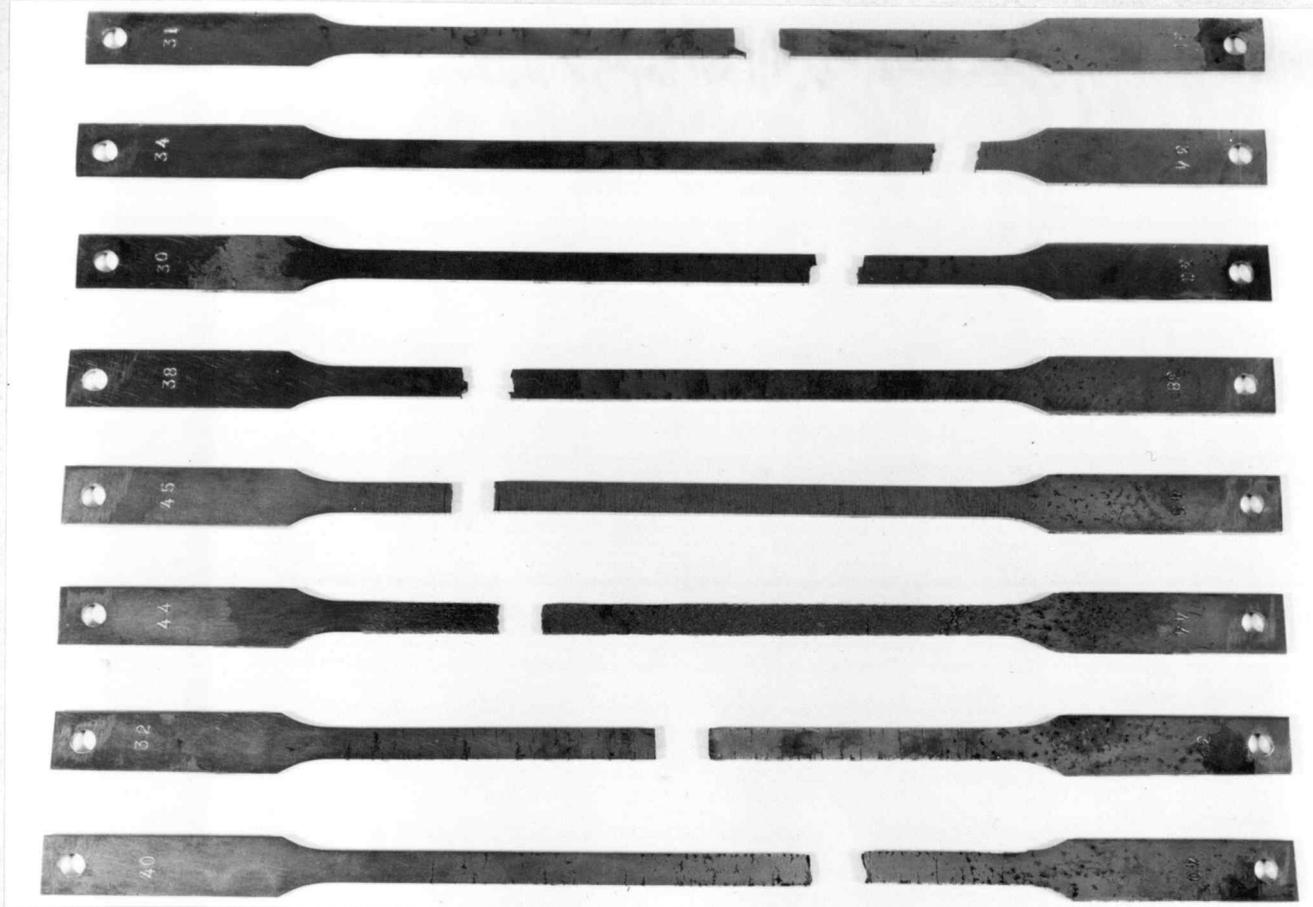


Figure 17. - Photograph of specimens representing cold-rolled annealed sheet from ingot No. 57-D-452. The specimen at the top of the photograph was stressed at 33,000 psi. This was reduced for each of the other specimens progressively towards the bottom of the photograph.

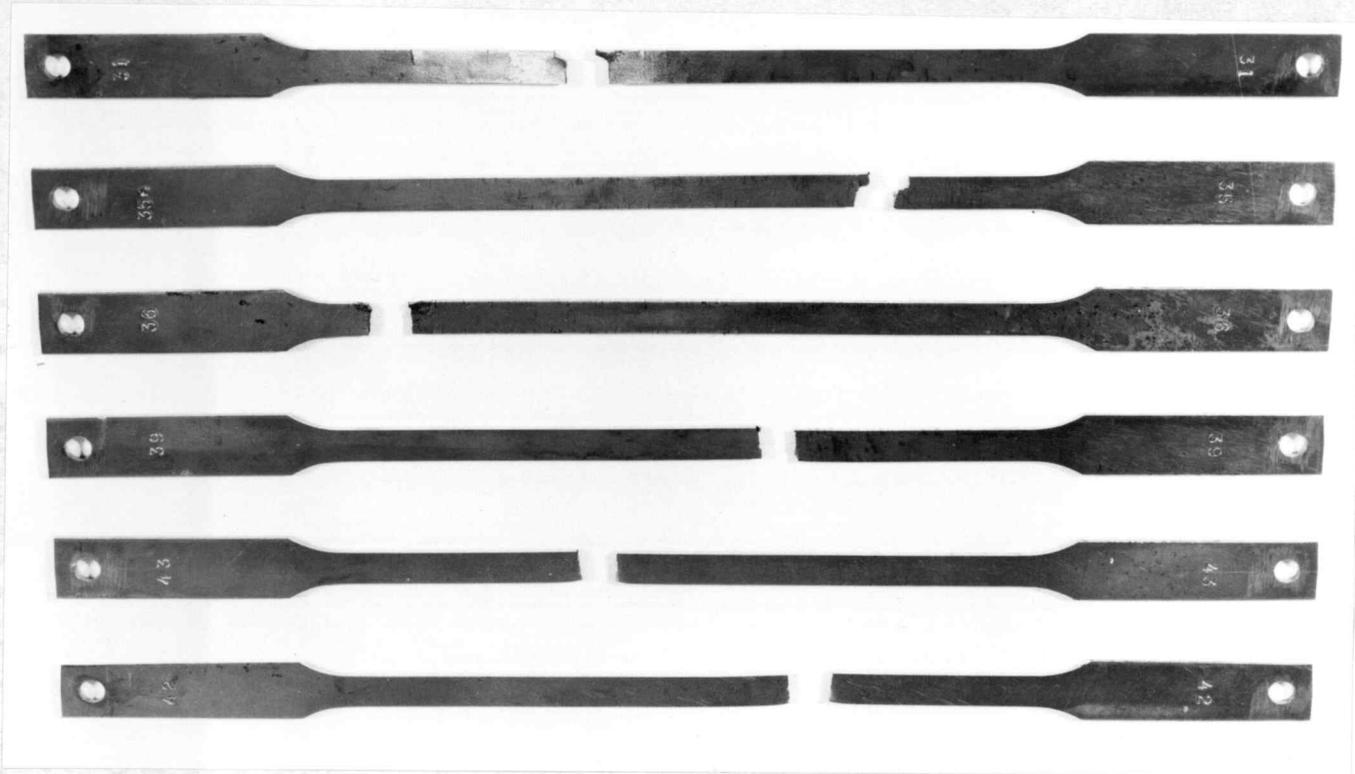


Figure 18. - Photograph of specimens representing cold-rolled annealed sheet from ingot No. 57-D-452. The specimen at the top was tested in 25 percent  $\text{FeCl}_3$  solution. This was reduced for each of the other specimens progressively towards the bottom of the photograph.

## DISCUSSION OF RESULTS

The results of the various groups of specimens that were tested did show the zirconium used to be susceptible to stress-corrosion cracking in solutions of aqueous ferric-chloride. Furthermore, the magnitude of the applied stress, the strength of the solution, air aeration, and the condition of the material were all shown to influence the susceptibility of zirconium. Cracking was also shown to be intercrystalline and normal to the direction of stress.

Probably of primary importance is the fact that zirconium was shown susceptible in the test conditions. Zirconium is not inherently resistant to corrosion, but displays such behavior because of a very tightly adhering oxide film on its surface. The oxide film is the compound  $ZrO_2$  which is the principal, and possibly only, product of the zirconium reaction with oxygen. Unlike titanium, also in subgroup IVA of the periodic chart of the elements, zirconium does not form the lower valence compounds. Titanium forms  $TiO$  and  $Ti_2O_3$  in addition to  $TiO_2$  (9, p. 553-554).  $ZrO_2$  has a monoclinic structure up to 1832 F but minor additions of  $MgO$  and  $CaO$  can produce a cubic modification which is stable at room temperature. The volume ratio for  $ZrO_2$  (molar volume  $ZrO_2$ /atomic volume of zirconium) is about 1.6 (19, p. 39-40). Thus the oxide is in compression and places tensile stresses on the underlying metal. In oxidation studies on Zircaloy-2 sharp changes in oxidation rates are

observed. These have been attributed to the inability of the metal to sustain the stresses imposed by the oxide which cracks and spalls off (19, p. 40). The compressive stresses in the oxide have been estimated from 16 to 48,000 psi (19, p. 40). In an environment where the oxide of zirconium can not be formed a break in the film would not repair itself and the metal would remain exposed. In addition, an applied tensile stress would tend to reduce the compressive stress of the oxide film and at the same time be superimposed on the tensile stress acting on the metal surface of the oxide-metal interface. Thus any penetration of the film by the corrosive environment would initiate attack on the highly stressed zirconium surface and the normal mechanism of stress-corrosion cracking proceed. Harwood (1, p. 13-14) discusses the films on single crystals and on some polycrystalline materials pointing out that these films inhibit creep and deformation processes possibly by acting as barriers to the movement of dislocations. A concentration of dislocations beneath a film surface would produce a high-stress concentration and once the film was broken or chemically removed, spontaneous deformation would occur because the dislocations would move in their original direction. Thus a crevice or crack nucleus would be formed as soon as the film was removed.

It has been suggested that the film is susceptible to penetration in solutions containing acid chloride salts because these salts hydrolyze. Ferric-chloride is one of these acid chloride salts and undoubtedly causes

penetration of the film on zirconium. In addition ferric-chloride is known as a strong oxidizing agent and extremely corrosive (18, p. 132).

Air aeration of the solution resulted in reduced time for failure. This indicates that oxygen is involved in the corrosion reaction. The aeration might have increased the rate of the film breakdown or increased the rate of crack propagation or both. However, the specimens tested in aerated solutions show little localized corrosion, contrary to what would be expected if the aeration influenced film breakdown. On the other hand if the cracks propagate at a higher rate in aerated solutions it would indicate oxygen to be involved in the corrosion reaction of the zirconium metal. The anodic reaction of zirconium in aqueous solutions containing chloride ions is said to be complexed or hydrolyzed (9, p. 665-666). The oxygen could thus have been reduced into hydroxyl ions and accelerated the crack development.

Harwood (1, p. 14-15) also points out that the extension of cracks is largely discontinuous and continued reinitiations are a dominating feature of progressive fracturing. The rapid starting and stopping of individual elements of discontinuous fracturing produce mechanical shock in adjacent material, which can result in fractures and distortions in advance of a propagating crack. The crack will tend to fork or branch. It will stop when it extends into a stress field where there is no significant component of tension normal to the fracture path. The large cracks

tend to grow faster and one crack becomes dominant stopping the growth of others. It generally leads to failure. This was observed on some specimens and is shown in the photomicrographs, Figures 15 and 16.

Stress was shown to have an influence on the time before complete fracture. Variation of the applied stress and the time to fracture is shown graphically in Figure 19 for the specimens representing cold-rolled annealed sheet from ingot No. 57-D-452 and tested in an air aerated solution of 25 percent  $\text{FeCl}_3$ . Increasing the stress does shorten the time before complete fracture. The data indicate that a threshold stress exists near 19,000 psi.

A threshold stress is necessary to produce sufficient stress concentration and consequent deformation to initiate cracking but it is not an absolute quantity; it is dependent on test conditions and specimen geometry. For these specimens tested under the conditions stated it appears that failure by cracking would be unlikely below 19,000 psi. At the lower stresses uniform corrosion would be expected to take place, reducing the cross-sectional area of the specimen until failure occurred as shown by the control specimen, No. 10, tested in 25 percent ferric-chloride with no applied stress. Failure by cracking might be observed with applied stresses below the apparent threshold level because of an obscure stress concentrator such as an inclusion that is rapidly removed by the corrosive environment.

The variation in the rate of change of electrical resistance of the specimens in this same group is shown graphically in Figure 20. It was thought this variation would indicate whether stress influenced the crack nucleation as well as the crack propagation. The extent of cracking in a specimen, meaning both the number of cracks nucleated and their average depth, is shown to increase with increased stress up to some level near 28,000 psi and then fall sharply as the stress is increased. A visual examination of the specimens shows the same trend for the extent of cracking. The specimens stressed near the yield strength have a few severe cracks which would not seriously affect the average electrical resistance. This is also true for the specimens at low stresses. The specimens at the intermediate levels have a much greater number of cracks but the cracks are less severe. Apparently, the greater number of cracks results in a greater reduction of average cross-section and consequently a greater increase in resistance. Considering both the crack nucleation and propagation, increasing the applied stress seems to increase the number of cracks nucleated until some level is reached where the crack propagation becomes so great, failure occurs from one of the first cracks nucleated before many are formed.

A decrease in solution concentration was shown to increase the time to fracture. This is shown graphically in Figure 21 for the specimens representing cold-rolled annealed sheet from ingot No. 57-D-452. The

time to fracture is not greatly affected for the range of concentrations between 5 to 25 percent but a large difference is shown between 3 and 5 percent. The electrical resistance measurements were again made to determine the extent of cracking. The variation of this parameter with solution concentration is shown graphically in Figure 22. At low concentration little change is observed in the rate of change of resistance but as the concentration increases this rate increases rapidly. This indicates that the extent of cracking becomes much more severe as the solution concentration is increased. In conjunction with the visual appearance of the specimens the higher concentrations evidently cause several severe cracks but as it is reduced fewer cracks are nucleated and at the low concentrations crack nucleation is sparse. One of the few nucleated propagates to failure. From these observations it appears that the strength or concentration of the solution determines the time for crack nucleation or, in other words, the time for the penetration of the oxide film; the penetration time decreasing with increased concentration.

A comparison of the data in Table 7 with the data in Table 8 for specimens tested under similar conditions shows little difference for the hot-rolled material and the cold-rolled and annealed material. Specimen No. 26 of the hot-rolled material stressed at 30,000 psi fractured by cracking in 12 hours while specimen No. 34 of the cold-rolled and annealed material stressed at 31,500 psi fractured after 22 hours in test. This behavior

is reversed for the specimens stressed at 28,000 psi; the specimens from the hot-rolled material fractured after 82 hours in test and the specimen from cold-rolled and annealed material lasted only 14 hours. If any difference in cracking behavior caused by the material processing exists, it is obscured by the variation in the data.

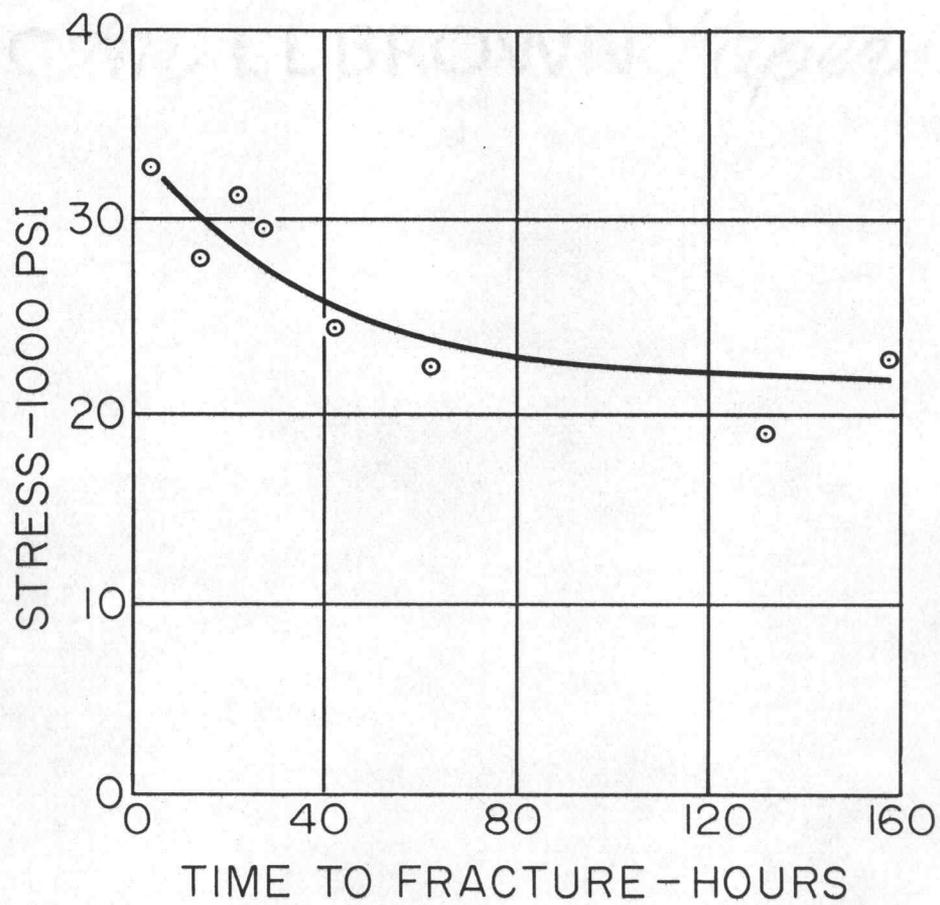


Figure 19.—Influence of stress on fracture time

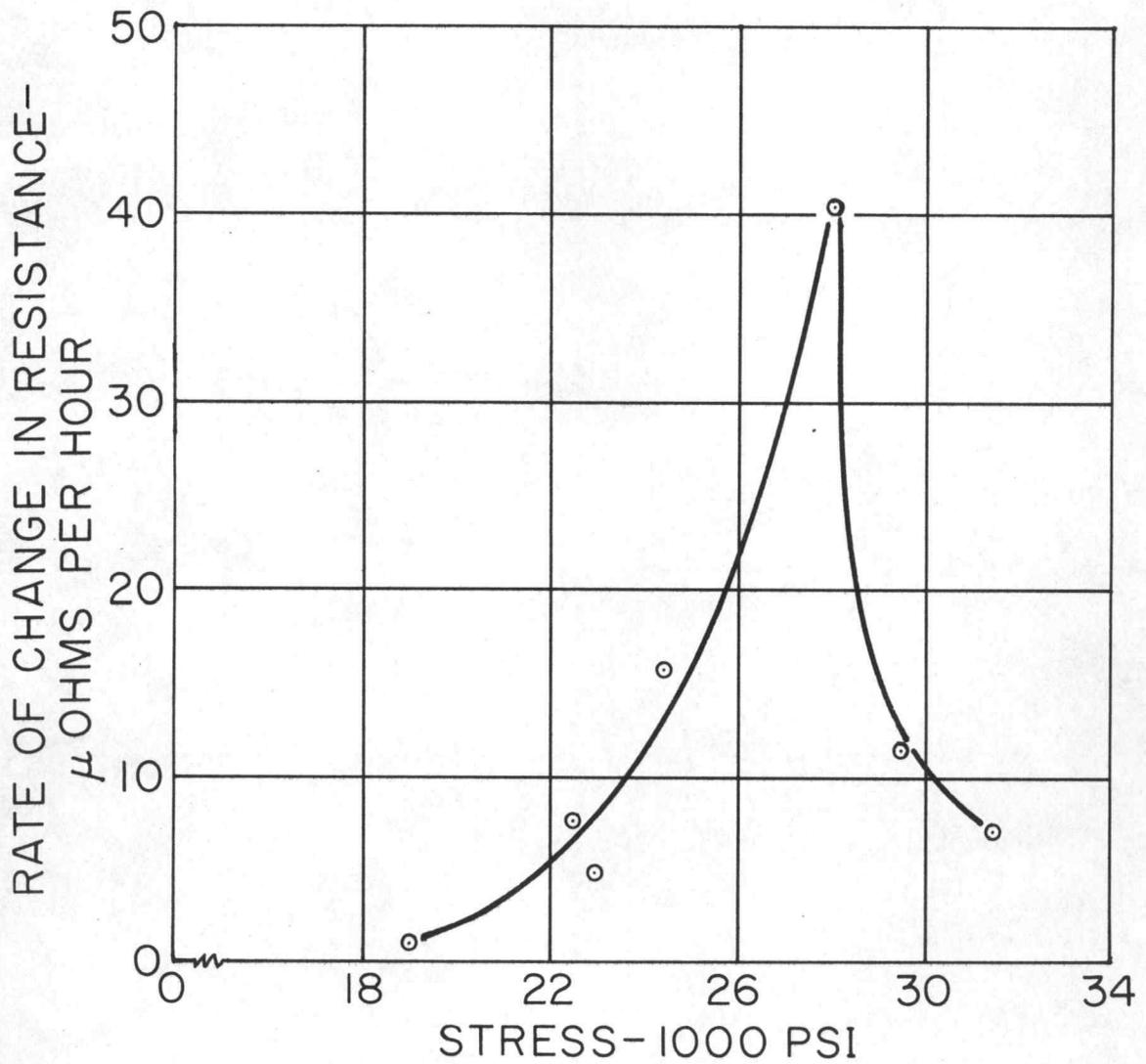


Figure 20.-Rate of change in resistance with variable stress

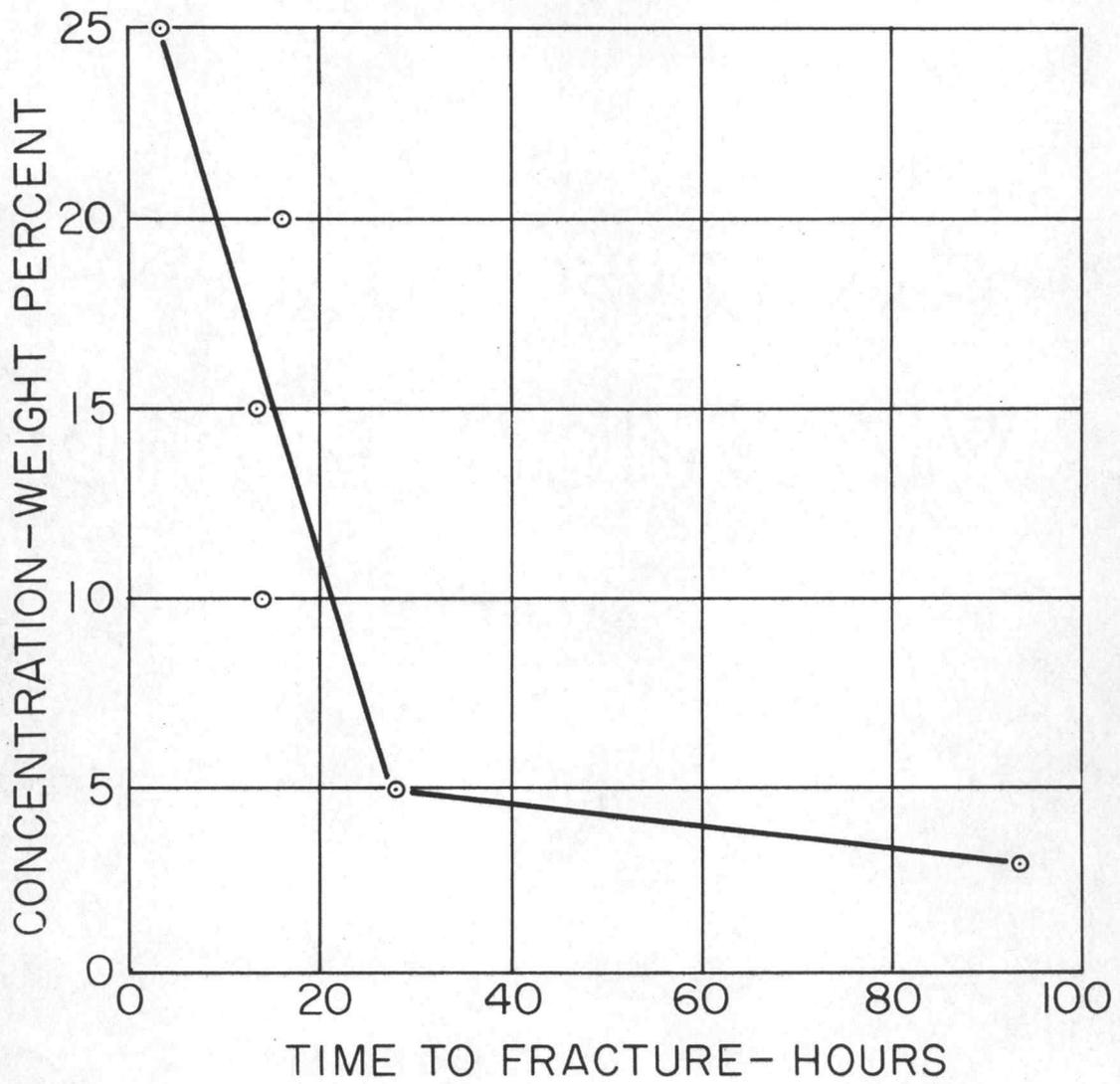


Figure 21.-Influence of concentration on fracture time

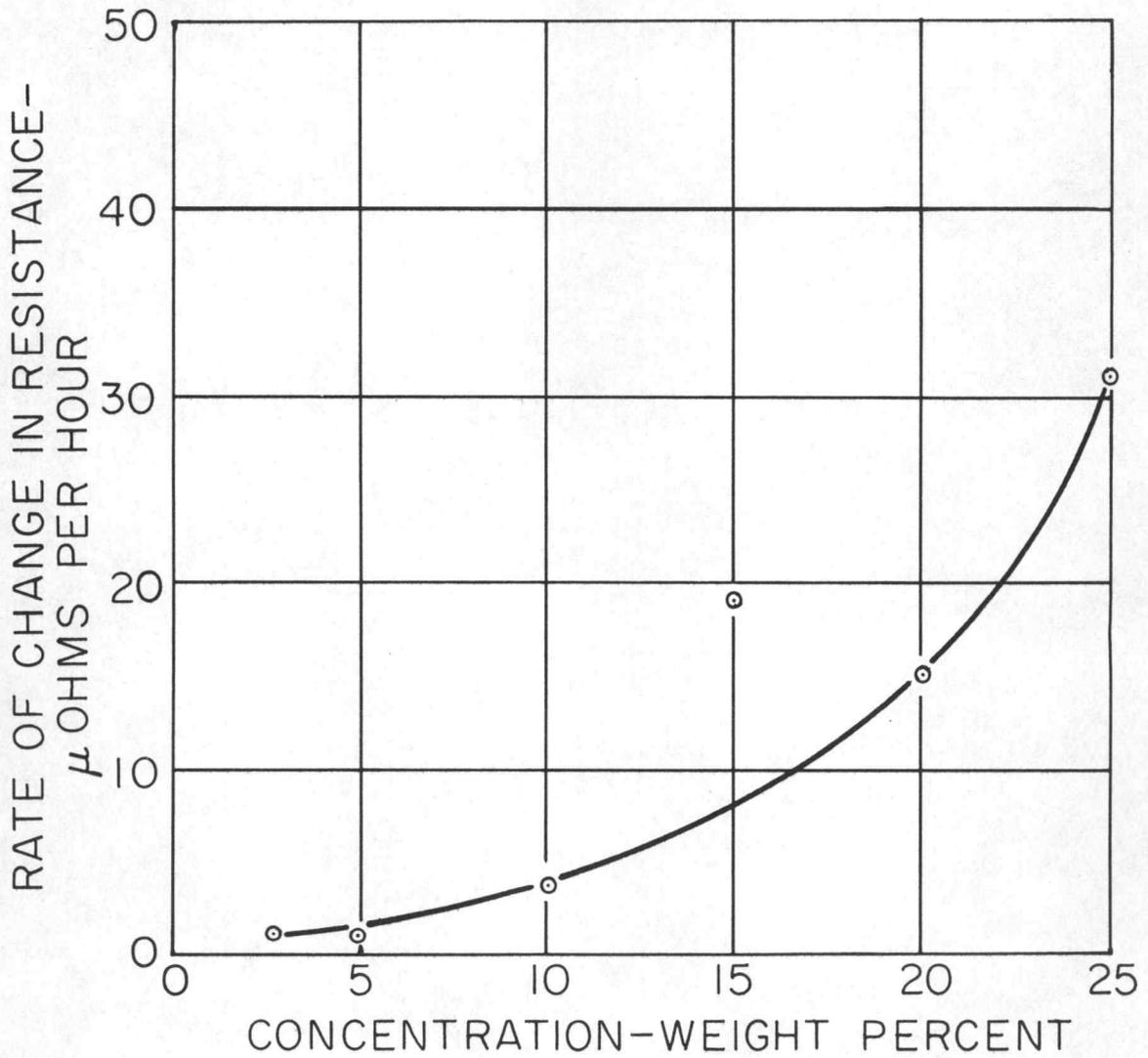


Figure 22.-Rate of change in resistance with variable solution concentration

## CONCLUSIONS

The results of the investigation primarily show the susceptibility of reactor grade arc-melted zirconium to stress-corrosion cracking in aqueous ferric-chloride solutions. Other conclusions based on the results are as follows:

1. The magnitude of the applied stress has a definite influence on the time for cracking to occur; decreasing the stress increases the time to fracture.
2. A threshold stress of approximately 19,000 psi was indicated for susceptibility for the specimens tested under the specified conditions.
3. In high concentrations (20-25 percent) at stress levels near the yield strength the first crack nucleations propagate very rapidly until fracture of the material occurs.
4. In high concentrations at low-stress levels (30-50 percent of the yield strength) crack nucleations are sparse and crack propagation takes place slowly with most nucleations developing into pits.
5. The solution concentration has a definite effect on the time to fracture at stress levels near the yield strength; increasing the concentration reduces the time for fracture to occur.

6. The strength of the solution appears to affect crack nucleation rather than crack propagation indicating that the stronger solution concentrations (10-25 percent) penetrate the oxide film in less time than the weaker concentrations (3-5 percent).
7. In low solution concentrations (3-5 percent) at stress levels near the yield strength crack nucleations are sparse.
8. In low-solution concentrations (10 percent and below) at low stress levels (30-50 percent of the yield strength) the material does not indicate susceptibility.
9. Air aeration of the solution decreases the time to fracture by an order of magnitude from the time observed for specimens tested in non-aerated solutions; apparently it increases the rate of crack propagation.
10. Whether the material was hot rolled or cold rolled and vacuum annealed did not seem to affect susceptibility. If a difference was caused by the material's mechanical history it was obscured by the variation in the data.
11. The constant-load method of testing was found more reliable than the constant-strain method in testing for susceptibility.

The investigation shows the results only for a particular environment, ferric-chloride, where zirconium has poor corrosion resistance, and does not support any general statements about the cracking

susceptibility of zirconium; however, it is possible to conclude that if zirconium is to be used where high stresses are involved, internally or externally applied, and there is some question on the effects of the environment encountered, a test for stress-corrosion cracking susceptibility is warranted.

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