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CEND-508 ✓

REACTOR TECHNOLOGY

Joint U. S. - EURATOM Program

EUR/AEC Report. 1233

THE UNITED STATES ATOMIC ENERGY COMMISSION
AND
THE EUROPEAN ATOMIC ENERGY COMMUNITY (EURATOM)

MASTER

Quarterly Progress Report No. 4

July - September 1964

INVESTIGATION OF CATALYTIC RECOMBINATION
OF RADIOLYTIC OXYGEN AND HYDROGEN

October 10, 1964

NUCLEAR
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Quarterly Report

INVESTIGATION OF CATALYTIC RECOMBINATION
OF RADIOLYTIC OXYGEN AND HYDROGEN

July - September 1964

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Contract No. AT-(40-1)-3151

Unclassified
Classification

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Authorized Classifier Date

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I. SUMMARY

The beginning of the fourth quarter marked the initiation of long-term or endurance testing of catalysts and the procurement of corrosion data for both high and low oxygen content steam. This report covers the test loop alterations required for second phase test work, the effect of long-term operation on three of the catalysts and both stress and general corrosion data for conditions typical of virgin reactor steam and for steam conditions which have been altered by the use of a recombiner.

The test loop which had been used for earlier, Phase I work was altered for use in the presently underway Phase II program. Generally, the loop was altered such that H_2 , O_2 and NaCl solution could be added to 900-psig, wet power plant steam which is then used in the tests. This wet (1-3% moisture by weight) steam is then passed over stress corrosion specimens, dried and superheated a few degrees, passed over general surface corrosion coupons, passed over the catalyst bed, passed over a second set of surface corrosion specimens, cooled to wetness again and then passed over a second set of stress corrosion specimens. The steam is then condensed and monitored for chlorides and free oxygen.

The three catalysts were operated for seven weeks before evidence of significant apparently permanent loss of activity occurred. This operation was possible irrespective of the injection of many grams of NaCl but required one shutdown necessary for water and steam cleaning of the catalysts for removal of NaCl deposits. The beds collected more than 50% of the chlorides and other solids under dry steam conditions and after

approximately one week of operation with steam which contained 5 to 10 ppm total solids, it was necessary to remove and clean the catalysts, as both NaCl, O_2 and H_2 "broke through" the beds. This early breakthrough would not occur if high purity reactor steam were used.

Both stress corrosion and general corrosion data taken to date show a marked difference in rates when data for 30-ppm O_2 and 0.2-0.5-ppm O_2 concentration conditions are compared. For situations with and without H_2 , more rapid, more numerous and more severe cracking of Type 304 stainless steel U-bend specimens occurred where the high O_2 content was present.

While only a small amount of general corrosion data have been obtained to date, it appears that higher oxygen contents increase the corrosion rates of the low alloy materials but that the corrosion rate of the Type 304 stainless, which is very low at the test temperatures (approximately 540°F), is not affected noticeably.

II. INTERIM CORROSION TESTS

A. General

An earlier report* described a small amount of stress corrosion data which were obtained by exposing stressed Type 304 stainless steel specimens to high oxygen or low oxygen content wet steam which contained 1-3 ppm of chlorides. These data were taken by using the power plant test loop while loop design modifications were being made and equipment being ordered for Phase II work. Not only did such work utilize the test loop continuously but allowed the procurement of "bench-mark" data needed to plan Phase II effort. No H_2 existed in the steam used in these tests.

The results of these earlier tests indicated that the oxygen content of wet steam does affect the magnitude of chloride-related stress corrosion problems. For those tests, twenty specimens were sheared from a sheet of Type 304 stainless steel and the edges ground very slightly such that they were 1/4 in. wide, approximately 4 in. long and 0.028 in. thick. When exposed to steam within the loop, they were stressed in a U-bend manner such that their ends were 3/8 in. apart. Ten of the specimens (upstream) were exposed for nine days to 900-psig wet steam which contained 0.17-0.25 ppm oxygen and 1.8-2.2 ppm chlorides. The moisture content of the steam was between 2 and 3% by weight. The other ten (downstream) specimens were exposed to the same steam, but after the oxygen content had been increased to 28-35 ppm.

* Investigation of Catalytic Recombination of Radiolytic Oxygen and Hydrogen, Quarterly Progress Report No. 3, USAEC Report GNEC 354, General Nuclear Engineering Corp., Dunedin, Fla., July 10, 1964.

After completion of the nine-day test, each of the downstream specimens was found to contain 30-40 cracks while one of the upstream specimens contained two cracks which had less depth than the downstream cracks. No other cracks were found in the upstream set. All cracks were generally transgranular as can be seen from the photographs of typical cracks shown in Fig. 1.

This test was repeated by placing another set of specimens, one of which was sheared, upstream and a similar set downstream under conditions identical to the ones described above. After six days of exposure many cracks were visible in the downstream sheared specimen but none in the upstream sheared specimen. The remaining eighteen specimens were fabricated by sawing, as opposed to shearing, and were apparently not as susceptible to early crack initiation. No cracks were found in any of the eighteen sawed specimens.

B. Sawed Corrosion Specimens

A third set (20) of specimens was prepared from the same sheet of Type 304 stainless steel. However, all of these specimens were formed by sawing and grinding. The test described above was then repeated. The upstream specimens (10) were exposed to 900-psig, wet steam containing 0.17-0.25 ppm oxygen and 1.8-2.2 ppm chlorides. The ten downstream specimens were exposed to the same steam, but after the oxygen content had been increased to 28-32 ppm. The moisture content of the steam at each point was 2-3%.

Examination under a microscope after three-and-one-half days of exposure revealed that no cracks existed in either the upstream nor the downstream corrosion specimens. The same specimens were then returned to the

loop and exposed for an additional period of five and three-fourths days. A subsequent microscopic and metallographic examination showed no cracks in either the upstream or downstream specimens.

Later, in an attempt to determine if greater specimen thicknesses would accelerate cracking, five of the above specimens and five new, thicker specimens were placed in each of the corrosion specimen positions. The thicker specimens were sawed and ground slightly and were used to achieve higher stresses. Exposure of these specimens for about two weeks produced no cracks. The exposure history for these tests is shown in Fig. 2.

C. General Conclusions

Based on the corrosion data described above, it was concluded that the shearing process, used in preparing the first two sets of specimens, cold-worked the edges of the specimens thereby producing local edge conditions from which the cracks could easily originate. Greater exposure times are apparently required under the above conditions to produce cracks in the sawed specimens. It has been concluded that the higher oxygen concentrations greatly increased the rate of cracking in the sheared specimens. However, it should be mentioned that the upstream specimens were mounted horizontally in a 1-in. pipe while the downstream specimens were mounted vertically; therefore, the behavior of the moisture could have been different.

Visual examination of the corrosion specimen holders indicates that the 28-32-ppm oxygen greatly increased the rate of general corrosion. A photograph of the specimen holders is shown in Fig. 3. Both parts shown, which served as ballast, are made from carbon steel. The downstream holder has corroded to a much greater extent than has the upstream holder. The latter

was exposed to only 0.17-0.25 ppm oxygen while the former was exposed to 28-32 ppm oxygen concentrations. Both holders were used in each of the tests.

III. DESCRIPTION OF TEST LOOP USED IN PHASE II WORK

A. General Description of Test Loop After Alterations

The test loop used in Phase I, after some changes, is being used for Phase II work. A section of 1-in. pipe was added downstream of the catalyst vessel for containment of stress corrosion specimens and a heat exchanger was placed between the catalyst vessel and this container location to increase the local moisture content to the same value as will be present at the upstream corrosion specimen position. A simplified flow diagram of the system to be used during Phase II work is shown in Fig. 4. The stress corrosion specimens are located at points A and B, and the general surface corrosion coupons just above and below the catalyst bed inside the catalyst vessel.

B. Description of Catalyst Holder

A photograph of the catalyst holder is shown in Fig. 5. Its purpose is to allow the long-term testing of three catalysts simultaneously. The length of each of the three catalyst chambers is 24 in. and the ID is 1.18 in. The catalyst holder fits into the 2.9-in.-ID catalyst vessel used during Phase I tests. The desired bed depth of each catalyst is calculated, using data and equations developed earlier. Normally, it is expected that individual catalyst bed depths will be selected such that approximately 98% of the two gases will be removed under idealized conditions. Due to different pressure drops across the respective catalysts, orificing is required to obtain equal flows through each of the three cylinders. The orifice sizes have been determined by air flow tests. The activity of

each catalyst can be checked by operation with the other two catalyst chambers plugged. The plugs are shown in Fig. 5 also. A similar procedure will be used to re-check the individual activities as the test progresses.

IV. ENDURANCE TESTING OF CATALYSTS "D", "E", AND "F"

A. Endurance Testing Without Chloride Injection

The endurance testing of catalysts is accomplished by taking 900-psig wet power plant steam, adding metered hydrogen and oxygen to this steam, producing a small amount of superheat by the use of electrical heaters and a steam heated preheater and then passing the steam over one or more catalysts. This steam is then condensed and monitored for the remaining free oxygen. During the month of August, Catalysts "D", "E", and "F" were tested semi-continuously for 25 days in a parallel flow arrangement. The catalyst volumes were calculated to give 98-99% recombination initially. The resultant bed depths were 7.8 in. of Catalyst "D", 12.6 in. of Catalyst "E", and 8.4 in. of Catalyst "F". The catalysts are contained in the three tubes described above. The steam flow through each catalyst bed was adjusted by orificing such that it was 60 lb/hr. The 900-psig steam was superheated 10-20⁰F before passage through the beds. The bed inlet oxygen concentration was 24-26 ppm, with an excess of hydrogen being added to ensure the presence of a stoichiometric amount, irrespective of small fluctuations in either or both gas inputs.

The oxygen concentration in the effluent ranged from 0.15 to 0.4 ppm. A plot of the effluent oxygen concentration for the month is shown in Fig. 6. As shown in Fig. 6, there was no significant loss in the activity of either of the catalysts.

B. Endurance Testing With Chloride Injection

After the catalyst operation had been observed for 25 days and without any significant change in the combined efficiencies, stainless steel

corrosion specimens were placed in the loop in an effort to obtain chloride-related stress corrosion data and general corrosion data for low alloy piping materials. To obtain the former data, sodium chloride solution was injected in the loop such that the steam chloride content was 3.0-3.7 ppm. The loop effluent oxygen and chloride concentrations for this test are shown in Fig. 7. It can be seen from this figure that a large fraction of the chlorides were being collected by the bed and that this collection gradually poisoned the catalysts. After six days of testing, the chlorides "broke through" and a concentration approximately equal to the calculated loop inlet value appeared in the loop effluent and the effluent oxygen concentration increased to 5.6 ppm. The test was terminated at this time. The respective corrosion data are discussed below in Section V.

An examination of the catalysts revealed that Catalysts "D" and "F" had retained approximately three times as much NaCl as had Catalyst "E". Washing the catalysts in distilled water and with wet steam rejuvenated the three to the extent that the combined flow effluent oxygen concentration was 1.3 ppm under the same conditions as when it averaged 0.3 ppm before the chloride injection was started. Replacing Catalyst "F" and then "D" with new materials proved that these two catalysts had been poisoned by the sodium chloride and that "D" was somewhat worse than "F". The effluent oxygen content with Catalyst "D" and "F" replaced with new catalysts was approximately 0.2 ppm, showing that Catalyst "E" had not lost any significant amount of its original activity.

Catalyst "E" has now been endurance tested for 50 days without loss of activity. It has also demonstrated that it is not very susceptible to

poisoning by collection of solids. While Catalysts "D" and "F" had not lost any activity after 25 days of operation with high purity steam, they both later collected NaCl readily and were poisoned thereby. Washing and steam cleaning did not restore their activities completely. It is possible that they collected and retained more NaCl than Catalyst "E" because of their rougher surfaces.

V. RECOMBINER-RELATED CORROSION TESTS

A. Discussion of Conditions During the First Recombiner-Related Corrosion Test

After Catalysts "D", "E", and "F" had been endurance tested for 25 days, corrosion specimens were placed upstream and downstream from the catalyst bed in an effort to demonstrate the value of a catalytic recombiner in reactor systems. During the earlier 25 days of testing, the oxygen content downstream from the catalyst bed averaged 0.3 ppm and the upstream oxygen concentration varied between 24 and 26 ppm. The steam flow rate was 180 lb/hr at approximately 885 psig. After the corrosion test was initiated, 2 ppm of chlorides (as NaCl) were added to the steam at a point upstream from the first set of corrosion specimens.

Two types of specimens were used to obtain corrosion data; stressed stainless steel specimens and general corrosion specimens. The stressed specimens were taken from Type 304 stainless steel sheet and were 4 in. long and 1/4 in. wide. During testing, they were in the form of U-bends. Eleven of the stressed specimens were sheared from a 0.028-in.-thick sheet and then ground slightly. Nine similar specimens were sawed from a 0.062-in.-thick sheet and then ground slightly. The thick specimens had a prior corrosion test exposure history (see Section II-B above). A tabulation of properties for the thick specimens is given in Table I. Both thicknesses were taken from as-received sheets.

The general corrosion coupons were 1/2 by 1-1/4 by 0.060 in. There were six Type 304, six T-11 and six T-22 coupons.

Five thin-sheared and five thick-sawed stainless steel specimens were placed upstream of the catalyst bed where the steam moisture content was approximately 2%. These upstream stress corrosion specimens were exposed to 24-26 ppm oxygen and approximately 3.6 ppm (avg.) chlorides. The downstream set of stress corrosion specimens included four thick-sawed and six thin-sheared specimens which were exposed to the same steam as the upstream specimens except that the oxygen content and, incidentally, the chloride content, had been reduced by the catalyst bed. The exposure history for each set is given in Fig. 7.

It should be noted that the moisture content of the steam at each stress corrosion position was approximately 2%. Initially, the downstream oxygen content was approximately 10 ppm for 15 hours because of the passage of wet steam through the catalyst bed. After 10⁰F of superheat were obtained at the bed inlet, the effluent oxygen content remained below 0.5 ppm for 3 days. However, the chlorides were apparently collecting on the catalysts during this time and gradually reduced their efficiencies. During the sixth day of corrosion testing, the chlorides "broke through" and the downstream chloride content approached the calculated input value. The effluent (downstream) oxygen concentration thereafter increased to 5.6 ppm. The loop was shut down because of these occurrences, after 140 hours of corrosion test operation.

The general corrosion coupons were exposed to steam which had been superheated 10-15⁰F immediately upstream and downstream from the catalyst bed. There were three coupons of each type of alloy in each position. The upstream specimens were exposed to 24 to 26 ppm oxygen and 2 ppm chlorides.

The downstream (effluent) oxygen and chloride concentrations are shown in Fig. 7.

B. Discussion of the First Set of Stress Corrosion Data

Upon removal of the stress corrosion specimens from the loop, it was immediately apparent that each of the upstream specimens, five thick-sawed and five thin-sheared, had cracked and failed totally. The appearance of these specimens before removal from the rack is shown by Fig. 8, with a closeup view shown in Fig. 9.

The downstream specimens exhibited no cracks visible to the naked eye but examination with a binocular microscope and further metallurgical examinations disclosed five cracks in one sheared thin specimen, with one possible, but somewhat doubtful, crack in a thick specimen. No other cracks were found in this group.

Both upstream and downstream specimens were mounted and polished for metallographic examination. Of the downstream specimens, the thick specimen which contained a possible crack was thoroughly examined at 100X and then greater magnifications. No definite crack was found although it is possible that the evidence of an incipient crack may have been removed during preparation. All the cracks examined metallographically were found to be true transgranular stress corrosion cracks with the normal branched "streak-of-lightning" appearance. Figure 10 shows the appearance of representative failures.

It was obvious from this and previous work that preparation by shearing produced specimens of above normal sensitivity to chloride stress-corrosion failures. High localized stresses are evidently produced in the specimen

edges and cracks are easily initiated in these areas. On the thick-sawed and polished specimens, the localized stress areas are removed and the resultant surface more nearly represents the condition of structural stainless steel when exposed to chloride stress corrosion-producing conditions. Therefore, it was determined that the downstream conditions were such as to produce cracks in only one high-sensitivity specimen out of five but no positive cracks in specimens of normal sensitivity.

As an additional observation, the occurrence of small mounds of black material about the cracked areas on the upstream specimens should be mentioned. These black materials which formed at the cracks are shown in Fig. 9. The material was only loosely attached to the surface, and, from its appearance, could have either been deposited from the gas stream at these locations but more probably resulted from oxidation of the local metal during crack formation. From its appearance and magnetic character, it was plausible to identify the particles as magnetic iron oxide (Fe_3O_4), or a material which contained a large percentage of this oxide. It is of primary interest that this phenomena was not found on the downstream specimens. The oxidation of stainless steel in oxygen containing NaCl has been described¹ with the emphasis on the formation of chromates and chrome oxides. However, localized oxidation of the stainless steel at the failure points to an iron-rich oxide in the high-oxygen, upstream conditions, may be a possibility, although considerably more data would be required before such a mechanism could be formally supported.

1. H. W. Pickering, F. H. Beck, and M. G. Fontana, Rapid Intergranular Oxidation of 18-8 Stainless Steels by Oxygen and Dry Sodium Chloride at Elevated Temperatures, Transactions of the ASM, Vol. 53(1961), p. 793.

C. A Repeat of the Recombiner-Related Stress Corrosion Test

In order to confirm the stress corrosion test results described above, the week-long test was repeated. The conditions during this test were essentially identical to the earlier conditions except (1) the chloride content was reduced by approximately 50%; (2) greater care was taken to prevent the appearance of wet steam on the catalyst, thereby preventing the early appearance of higher chloride and oxygen concentrations downstream; and (3) the test was terminated before the chlorides and oxygen "broke through" the bed. These test conditions are shown in Fig. 11.

An initial microscopic visual examination of the stress corrosion specimens indicated that the results of the second test were identical to or possibly more definitive than the first test data. Each of the upstream (high oxygen, higher chlorides) specimens either had failed completely or essentially. No cracks have been found in any of the downstream (low oxygen, lower chlorides) specimens. A complete metallographic examination is scheduled for each of these specimens.

D. General Corrosion Data

As stated above, six T-11 (1-1/4 Cr, 1/2 Mo), six T-22 (2-1/4 Cr, 1 Mo), and six Type 304 stainless steel surface corrosion specimens were exposed to high oxygen (25-32 ppm) or low oxygen (0.2-1.0 ppm) concentration conditions. These 18 specimens, divided into two sets, were exposed to the slightly superheated steam which existed just upstream or downstream from the recombiner bed. It was apparent from the data obtained by an examination of these coupons that, for a time of approximately 400 hours, the low alloy materials corroded differently. The higher oxygen concentration

conditions produced a rougher surface appearance and resulted in a weight gain which was almost 400% as great as the low oxygen (downstream) conditions. A photograph of representative coupons exposed to high and low oxygen concentration is shown in Fig. 12. An approximate weight gain plot is shown for each set in Fig. 13. There was only insignificant difference in weight gained by the two sets of stainless steel coupons.

Table I PROPERTIES OF THICK TYPE 304 STAINLESS STEEL SPECIMENS

Gauge	No. 16
Tensile strength	85,500 psi
Yield strength	45,800 psi
Elongation in 2 in.	55%
Hardness	RB 80

<u>Chemical Composition</u>						
<u>C</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Si</u>	<u>Ni</u>	<u>Cr</u>
0.06	1.53	0.022	0.013	0.54	9.14	18.43

VI. FUTURE TESTS

Endurance testing of the catalysts will continue. However, the effects of the use of high chlorides concentrations in the catalyst efficiencies may limit future corrosion work. Nevertheless, if possible, it is now planned to place more emphasis on higher temperature, intergranular-related corrosion.

ILLUSTRATIONS

a. Typical crack (100X).

b. Typical crack (60X).

Fig. 1 Microphotographs of cracks in sheared Type 304 stainless steel specimens.

Fig. 2 Histogram of corrosion specimen exposure conditions.

Fig. 3 Photograph of specimen rack ballast plugs after use.

Fig. 4 Flow diagram for system used in Phase II tests.

Fig. 5 Photograph of catalyst container.

Fig. 6 The effect of extended operation on catalyst efficiency.

Fig. 7 Steam conditions for the first recombiner-related stress corrosion test.

Fig. 8 Stress corrosion specimens as removed from the loops. Downstream specimens at top (approximately full scale).

Fig. 9 View of upstream specimens; thin, sheared specimen at left. Part of the black oxide has been removed from the crack in the thick specimen at right (approximately 7X).

a. Cracks in thick upstream specimen (thick, sawed) at 40X.

b. Crack in downstream specimen (thin, sheared) at 100X.

Fig. 10 Representative photomicrographs of the chloride stress-corrosion failures.

EFFLUENT OXYGEN AND CHLORIDE CONCENTRATION, PPM
MOISTURE CONTENT OF STEAM, PERCENT

Fig. 11 Steam conditions for the second recombiner-related stress corrosion test.

Fig. 12 Photograph of representative surface corrosion coupons after 420 hours.

Fig. 13 Weight gain plots for low alloy materials.