

MASTER

**The Corrosion of Several Alloys
in Superheated Steam at 482 and 538° C**

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THE CORROSION OF SEVERAL ALLOYS IN SUPERHEATED
STEAM AT 482 AND 538°C

J. C. Griess and W. A. Maxwell*

ABSTRACT

We are investigating the corrosion of several ferritic steels, Incoloy Alloy 800, Inconel 617, and type 304 stainless steel in superheated steam at 482 and 538°C (755 and 811 K). Specimens of the alloys, including welds and U-bends in some cases, are mounted in a once-through loop that receives steam from a fossil-fired power plant. This report presents the data obtained during the first 12,000 h of a planned 30,000-h test.

All ferritic steels have shown similar weight gains and to this time no significant oxide exfoliation has occurred. The other materials have corroded at very low rates. Incoloy Alloy 800 that had been intergranularly corroded during pickling showed no further intergranular penetration in steam. Type 304 stainless steel was oxidized in a nonuniform manner, but even in the affected areas attack was slight. Welded and highly stressed U-bend specimens of Inconel 617 and of Incoloy Alloy 800 welded either to itself or to 2 1/4 Cr-1 Mo steel, showed no evidence of cracking nor any unusual oxide loss.

INTRODUCTION

Corrosion of materials in steam is of concern in any energy conversion system that uses the steam cycle to generate electrical power. However, in conventional fossil-fired electrical generating plants where steam is raised and superheated in tubes by hot combustion gases, the corrosion of the inner walls of currently used tube materials is nearly

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insignificant compared with the corrosion of the outer walls by the hot gases. Consequently, there has been little incentive to determine corrosion rates in steam (or water), so only a few quantitative studies have been conducted in these environments. On the other hand, in either high-temperature gas-cooled (helium) or liquid-metal-cooled nuclear reactors, virtually all the corrosion is produced by steam (or water), so we need to know the corrosion behavior of materials in order to establish accurate corrosion allowances for boiler and superheater tubes in these plants.

Much of the existing quantitative data on the corrosion of 2 1/4 Cr-1 Mo steel in superheated steam were summarized by DeVan and Griess.¹ These data covered the temperature range of 482–593°C (755–866 K). At the highest and lowest temperatures the test times were about 6000 h, but at 538°C (811 K) the tests lasted 14,000 h. The weight gains of all the specimens seemed to approximate parabolic reaction kinetics. McCoy and McNabb^{2,3} exposed a large variety of materials, including some chromium-molybdenum ferritic steels, to supercritical steam at 538°C (811 K) in TVA's Bull Run Steam Plant. Although the maximum exposure time for the ferritic steels was 14,000 h (the data for 2 1/4 Cr-1 Mo steel were summarized by DeVan and Griess), other alloys were in test for as long as 22,000 h. The corrosion behavior of highly alloyed materials in steam has also been examined but generally at temperatures significantly above those of interest for the steam-generating equipment of gas-cooled or liquid-metal-cooled nuclear power reactors. We do not intend to give a complete literature review in this report. However, the studies by Leistikow^{4,5} and by General Electric Company staff made during development of boiling-water reactors with nuclear superheat^{6,7} are probably the most comprehensive studies of highly alloyed materials in high-temperature steam. In these investigations, however, the steam contained low levels of hydrogen and oxygen to simulate the steam raised from boiling-water reactors.

The purpose of this investigation is to determine the long-term corrosion behavior of a variety of alloys in steam at 482 and 538°C (755 and 811 K). In addition to conventional weight-change specimens, we are testing specimens with welds joining both similar and dissimilar metals,

stressed specimens, and specimens of developmental alloys. This paper is an interim report that summarizes data obtained during the first 12,000 h of the test. We now plan to continue the test to 30,000 h.

EXPERIMENTAL

All test specimens in this study are exposed to steam in a once-through loop made of Inconel 617. Figure 1 is a photograph of the loop taken during construction. At the time the photograph was taken the heaters had been installed, but the loop had not been thermally insulated, nor had the condenser been connected. The loop is located at the Bartow Plant of the Florida Power Corporation in St. Petersburg, Florida, and is operated for ORNL by the NUS Corporation. Steam from the superheater circuit of the power plant [nominally 538°C (811 K) and 12.4 MPa (1800 psi)] is cooled below 482°C (755 K) before it enters the loop (upper left in Fig. 1). Then, as steam passes through electrically heated piping, its temperature is raised to 482°C by the time it reaches the first test section or autoclave. After leaving this autoclave, the steam passes through additional electrically heated pipe, which heats it to 538°C (811 K) before it enters the second test section. The steam that leaves the second autoclave is condensed and the condensate is discarded. The loop was made from 25-mm (1-in.) pipe, but the autoclaves, which are flanged on both ends for adding and removing test specimens, have internal diameters of 48 mm (1.9 in.). The mass-flow rate of steam through the loop is 45 kg/h (100 lb/h), which corresponds to a nominal velocity of 1.5 m/s (5 ft/s) past the specimens. The loop pressure is 10.5 MPa (1525 psi).

At the Bartow Plant steam is raised from boiler water, which contains 10 to 30 ppm phosphate and hydrazine (0.01 and 0.02 ppm in the feed water) and has a pH of 9.5 to 10.5. The steam condensate has a pH of 8.6 to 9.0, contains less than 0.4 ppm ammonia, and has a specific resistivity of more than 250,000 ohms-cm.

The weight-change specimens are nominally 73 × 13 × 1.6 mm (2 7/8 × 1/2 × 1/16 in.) with a 4.8-mm-diam (3/16-in.) hole near each end, but some of the welded specimens were slightly wider. The surfaces of all



Fig. 1. The Inconel 617 Loop Showing the Completed Internals with Heaters.

the unwelded and part of the welded specimens were ground on a 100-mesh belt grinder before test; in a few cases the specimens were annealed and/or pickled after grinding. Weight-change specimens were mounted on stainless steel screws that passed through the holes on each end and were separated from each other by thick small-diameter stainless steel washers. Nuts secured the specimens on the screws and these assemblies were placed in stainless steel boats for placement in the loop.

Both autogenous and filler metal welds are being tested. In all cases the gas-tungsten-arc process was used. Autogenous welds of 2 1/4 Cr-1 Mo steel and Incoloy Alloy 800 were made along the middle of 75- to 100-mm-wide (3- to 4-in.) strips of 1.6-mm-thick (1/16-in.) sheet and the test specimens were cut from the sheets so that the weld was across the axial middle of the 13-mm (1/2-in.) width of the specimen. These specimens were not ground.

The filler-metal welds were made from 15.9-mm-thick (0.625-in.) plate. The plates to be joined were machined to produce a 75° included angle with a 0.38-mm (0.015-in.) root face. For Incoloy Alloy 800 either welded to itself or to 2 1/4 Cr-1 Mo steel, Inconel 82 filler wire was used. Inconel 617 was welded to itself with filler metal of the same composition as the plate. In welds of 2 1/4 Cr-1 Mo steel the plates

were maintained at 204°C (477 K) throughout welding and the welded plates were given a post-weld heat treatment of 1 h at 704°C (977 K) in argon. Specimens were cut by a band saw across the thickness of the plate, so that the weld was equidistant from the ends of the test specimen. The flat faces of the specimens were ground on a 100-mesh belt grinder, but neither the face nor the root of the weld was disturbed.

The U-bend specimens were made from welded strips of the same size as the weight-change specimens. The radius of curvature of the "U" was 13 mm (1/2 in.) and the legs of the "U" were pulled parallel to each other. In all cases the weld was at the bottom of the "U." Calculations showed that the material was strained 6.2% at the outer surface. The U-bend specimens were not weighed, but they were examined under low-power magnification without being removed from their holders at the times the other specimens were weighed. Figure 2 shows a set of U-bends mounted in a holder. The welds in the specimens are clearly visible.

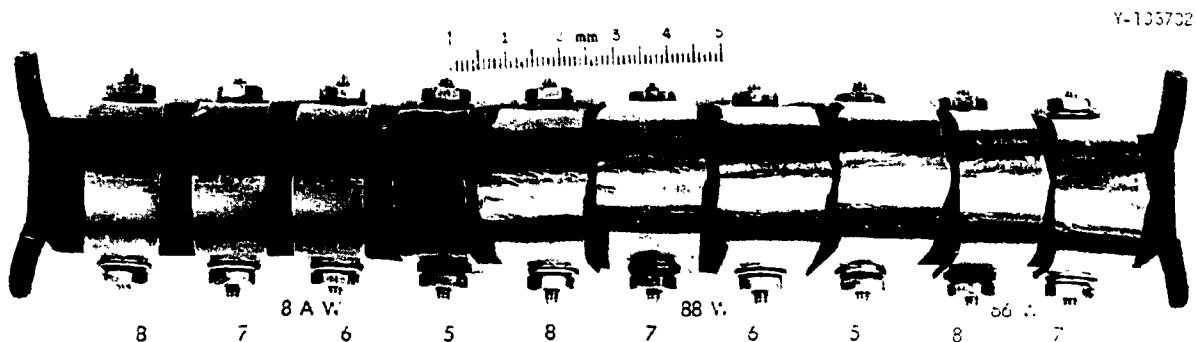


Fig. 2. U-Bend Specimens Mounted in Holder for Exposure in Inconel 617 Loop. The numbers identify specific specimens.

When the test was planned and started it was expected to last for 15,000 h. We initially prepared 25 to 30 unwelded weight-change specimens for each material under each condition. At the start of the test seven specimens of each material were placed in each of the two autoclaves; we planned, after exposure times of 500, 1500, 3000, 5000, and 8000 h, to remove one each of the original specimens from both autoclaves and replace them with new ones of the same kind. The welded specimens were

to be weighed at each of the above times and returned to test. However, after the test had been in progress for some time, it was decided to continue the test for a total of 30,000 h. Therefore, in the future fewer specimens will be removed at intermediate times, but all specimens will be weighed every 3000 to 5000 h. At this time specimens placed in test when operation began (except welds) have been permanently removed after 504, 1621, 3051, 5073, and 8005 h. Those removed after the three longest times were sectioned, mounted, and metallographically polished for examination of the oxide. The most recent examination of the specimens was after 11,751 h.

Certified mill analyses of the materials tested are shown in Table 1. The surface preparation and other pertinent facts about the individual specimens are presented with the data.

Table 1. Composition of Test Materials

Alloy Designation	Composition, wt. %									Other
	C	Cr	Ni	Mo	Fe	Mn	Si	V	W	
Incoloy Alloy 800 ^a	0.08	19.77	33.90		43.46	0.78	0.46			0.44 Ti
Incoloy Alloy 800 ^b	0.05	20.58	31.39		45.28	0.93	0.36			0.46 Ti
Inconel 617	0.07	20.30	57.35	8.58	1.01	0.05	0.16			11.76 Co, 0.76 Al
304 Stainless Steel	0.06	18.04	8.96		Bal.	1.84	0.55			
Sandvik Ht-9 ^c	0.19	11.4	0.50	1.00	Bal.	0.50	0.26	0.27	0.49	
Sandvik Ht-9 ^d	0.20	11.2	0.51	0.97	Bal.	0.50	0.32	0.29	0.51	
Sandvik Ht-9 ^e	0.18	11.3	0.50	0.93	Bal.	0.52	0.35	0.29	0.51	
Sumitomo 9 Cr-2 Mo ^f	0.04	8.80	0.13	2.06	Bal.	0.50	0.29			
9 Cr-1 Mo Steel	0.08	8.79	0.38	1.00	Bal.	0.34	0.46			
Expt 9 Cr-Mo ^g	0.087	9.58	0.08	0.80	Bal.	0.44	0.17	0.15	0.46	0.11 Nb
Expt 9 Cr-Mo ^h	0.095	9.52	0.16	1.55	Bal.	0.50	0.21	0.30	<0.01	0.14 Nb
Expt 9 Cr-1 Mo ⁱ	0.097	9.22	0.08	1.01	Bal.	0.38	0.08	0.22	<0.01	0.15 Mo
2 1/4 Cr-1 Mo Steel ^j	0.11	2.01		0.98	Bal.	0.54	0.22			
2 1/4 Cr-1 Mo Steel ^k	0.12	2.19		1.00	Bal.	0.44	0.26			
Inconel 82	0.02	20.03	72.50 ^l		1.16	3.07	0.09			0.39 Ti, 2.51 Nb

^aUsed for weight-change specimens and for both autogenous and filler metal welds.

^bUsed for mill-anneal specimens.

^cUsed for both annealed and ground specimens.

^dSpecimens supplied by General Atomic - heat 451774.

^eSpecimens supplied by General Atomic - heat 454191.

^fSpecimens supplied by General Atomic.

^gMaterial supplied by Combustion Engineering - heat ESR-KA3177.

^hMaterial supplied by Combustion Engineering - heat ESR-KA3178.

ⁱMaterial supplied by Combustion Engineering - heat 91887.

^jUsed for weight-change specimens and for Incoloy Alloy 800 to 2 1/4 Cr-1 Mo Steel welds.

^kUsed for autogenous weld specimens.

^lTotal Ni + Co.

RESULTS

In most cases the weight gains per unit area experienced by the various materials are presented graphically on linear coordinates in this section. For about the first 5000 h log-log plots of weight gain versus time yielded straight lines of slopes between 0.4 and 0.5, indicating parabolic oxidation kinetics. However, after longer exposure, the weight gains fell below extrapolations from the shorter time data and appeared to increase at a constant rate rather than at a decreasing rate. Longer tests will be required to establish with certainty the oxidation kinetics, but at this time the use of linear coordinates to present the data seems appropriate.

In the following graphs the average weight gains of the specimens of a given alloy that were in the test from the start are plotted as solid or open circles and a curve has been drawn through these points. Since specimens were permanently removed after 504, 1621, 3051, 5073, and 8005 h, the number of specimens used to determine the average decreased from 7 after 504 h to only 2 after 11,731 h. This procedure seems justified because the agreement was always good among replicate specimens put in test at the same time. Deviations in most cases were within 5% and only rarely were greater than 10% of the sample weight changes. Also, spallation of oxide has not occurred except for a very few small flecks of oxide from two of the 2 1/4 Cr-1 Mo steel specimens exposed at 538°C (811 K) for 11,731 h. However, specimens put in test at different times after the test had started show a considerably greater variability in weight gain. The weight gains of individual test specimens added at different times after the start of the test are shown by various symbols without curves drawn through them.

INCOLOY ALLOY 800

All weight-change specimens were prepared at the same time and all were ground on a 100-mesh belt grinder. After grinding, half the specimens were solution annealed at 1177°C (1450 K) for 30 min in argon and air cooled; this group of specimens was then pickled in a HNO₃-HF solution to

remove a thin tarnish film that had formed during annealing. The rest of the specimens were tested as ground.

Figure 3 shows the weight gains of the pickled specimens. The replacement specimens at 538°C (811 K) followed the curve drawn through the weight gains of the original specimens reasonably well, but at 482°C (755 K), the weight gains of the replacement specimens were consistently below the curve. The straight lines drawn through the last three data points correspond to corrosion rates of 3.6 and 5.6 $\mu\text{m/y}$ (0.14 and 0.22 mpy) at 482 and 538°C, respectively.

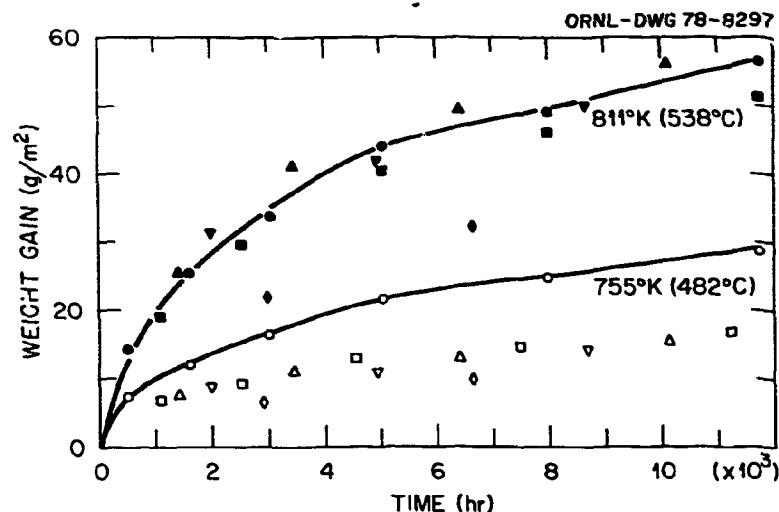


Fig. 3. Weight Gains of Pickled Incoloy 800 Specimens in Superheated Steam. \square — Single Replacement Specimen for One Removed after 504 h. \triangle — Single Replacement Specimen for One Removed after 1621 h. ∇ — Single Replacement Specimen for One Removed after 3051 h. \diamond — Single Replacement Specimen for One Removed after 5073 h. \circ — Average of Specimens in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

Polished cross sections of the pickled specimens showed that severe intergranular attack had occurred. Figure 4 shows photomicrographs of the specimen exposed for 8005 h at both 482 and 538°C and of an as-pickled specimen, as well. The maximum penetration (measured from the original surface on those exposed to steam) was about 200 μm (8 mils), indicating that the pickling had caused the intergranular penetration, and that steam exposure had not caused it to progress. Apparently, the rate of

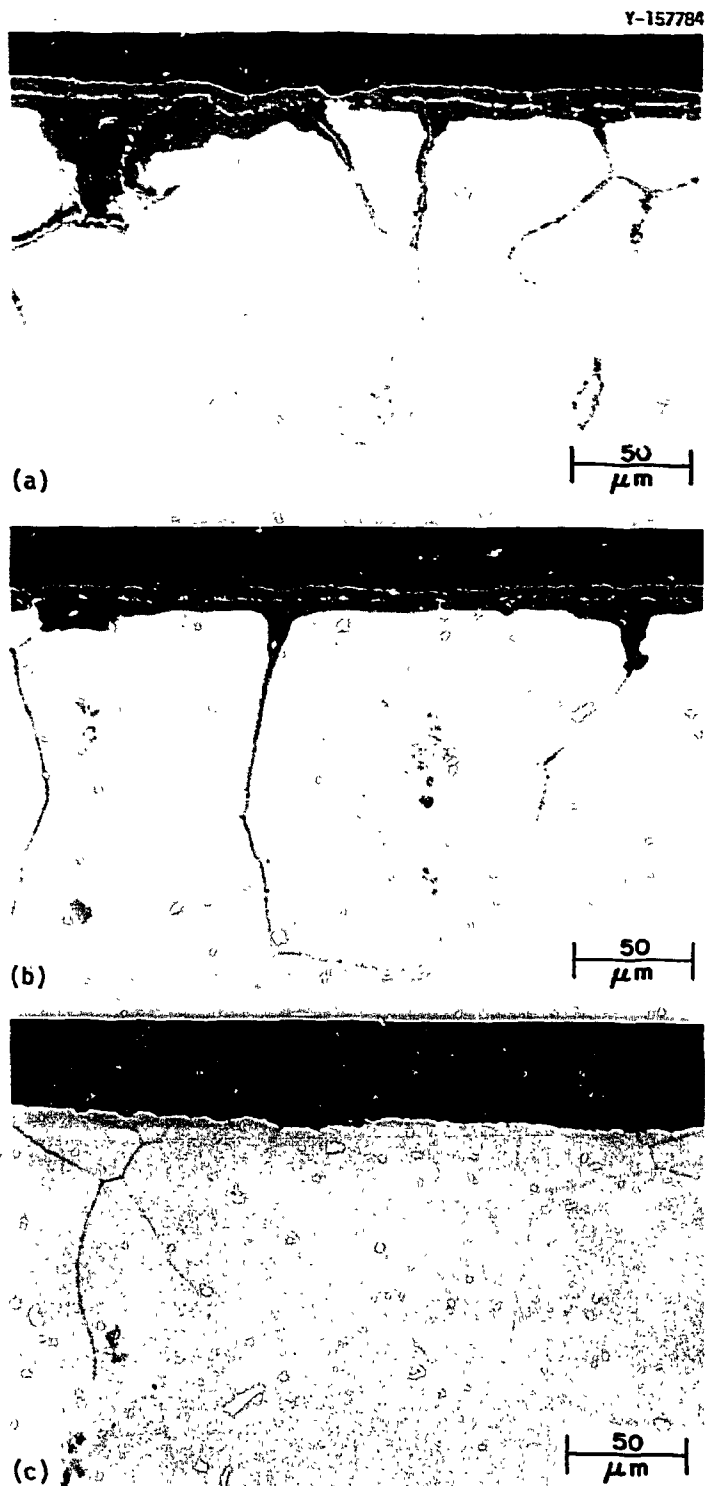


Fig. 4. Photomicrographs of Heat-Treated and Pickled Incoloy Alloy 800. (a) Exposed to steam for 8005 h at 482°C. (b) Same, at 538°C. (c) As pickled.

cooling following the solution anneal was slow enough to allow chromium carbide precipitation at the grain boundaries and the HNO_3 -HF solution attacked the chromium-depleted regions adjacent to the grain boundaries.

The Incoloy Alloy 800 specimens with the ground surfaces corroded only slightly. For example, the average weight gains of the entire specimens exposed for 11,731 h were only 0.85 and 1.6 mg at 482 and 538°C, respectively. Because of these low weight changes the average weight gains of those specimens that started the test are presented in Table 2, rather than in a graph. Replacement specimens corroded at the same low rates. A weight gain of 0.74 g/m² corresponds to a corrosion rate averaged over 11,731 h of 0.19 $\mu\text{m/y}$ (0.008 mpy).

Table 2. Average Weight Gains in Superheated Steam of Incoloy Alloy 800 Specimens with Ground Surfaces

Test Time (h)	Weight Gain, g/m ²	
	482°C (755 K)	538°C (811 K)
504	0.26	0.44
1,621	0.29	0.51
3,051	0.30	0.59
5,073	0.34	0.68
8,005	0.38	0.71
11,731	0.39	0.74

Figure 5 is a photomicrograph of a specimen with ground surface exposed for 8005 h at 538°C. No evidence of intergranular attack was apparent and the corrosion-product layer was so thin that it could not be detected on the polished cross section at a magnification of 500 \times .

A significant difference between the corrosion behavior of ground (cold-worked) and of annealed surfaces of Incoloy Alloy 800 has been reported before,⁴ but because of the intergranular attack on the "annealed" specimens in our test, a direct comparison between "annealed" and ground surfaces is not valid. To compare mill-annealed and ground surfaces of Incoloy Alloy 800, mill-annealed coupons were installed in the loop later in the test. After 2615 h the average weight gain for the seven specimens exposed to steam at 482°C was 1.7 g/m² and for the seven

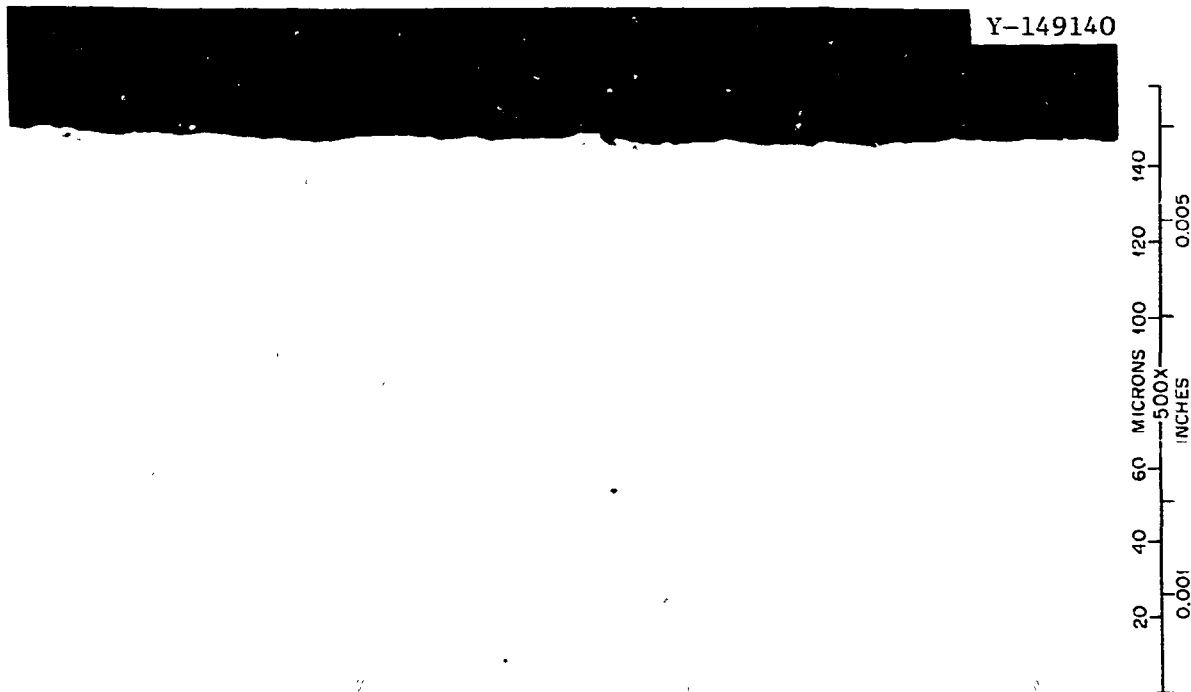


Fig. 5. Photomicrograph of Incoloy Alloy 800 Specimen with Ground Surfaces Exposed to Superheated Steam for 8005 h at 538°C.

tested at 538°C it was 2.0 g/m². Comparison of these values with those shown in Table 2 confirms that grinding increases the corrosion resistance of Incoloy Alloy 800 in steam. The difference is probably greater than indicated because the true surface area of the ground specimens was greater than that of the annealed ones.

The beneficial effect of cold work is further confirmed by comparing the average weight gains of the filler metal welds (ground on flat surfaces) with those of the autogenous welds (mill-annealed and pickled surfaces). In each case four specimens are in test. As shown in Table 3, the filler metal welds show less weight gain.

While we formed the U-bend test specimens with the filler metal welds of Incoloy Alloy 800 welded to itself or to 2 1/4 Cr-1 Mo steel, we found a lack of fusion in a few isolated areas. Figure 6 shows two examples of this defect, one in each type of weld. At the time of the last examination

Table 3. Average Weight Gain of the Welded Incoloy Alloy 800 Specimens in Superheated Steam

Test Time (h)	Weight Gains, g/m ²			
	Autogenous Welds		Filler Metal Welds ^a	
	482°C (755 K)	538°C (811 K)	482°C (755 K)	538°C (811 K)
504	0.70	1.1	0.19	0.33
1,621	0.90	1.4	0.25	0.62
3,051	1.1	1.6	0.40	0.77
5,072	1.2	1.9	0.41	0.93
8,005	1.3	1.9	0.57	1.01
11,731	1.4	1.9	0.67	1.04

^aGround surfaces.

no change had appeared in any of the defects. Also, we have noted no evidence of cracking or unusual scaling in either the filler metal or the autogenous welds.

INCONEL 617

All specimens of Inconel 617, including the welded and U-bend specimens, had ground surfaces that corroded very slowly. Table 4 lists the average weight gains observed on the specimens that had been in the loop since the test started (11,731 h). Specimens added at intermediate times showed similar low weight gains.

Why the welded specimens corroded more than the unwelded ones did is not apparent, but in both conditions the corrosion rates were low. A weight gain of 0.9 g/m² during 11,731 h corresponds to an average metal penetration rate of only 0.26 $\mu\text{m}/\text{y}$ (0.01 mpy).

The U-bend specimens showed no unusual effects and the oxide on the surfaces appeared identical to that on the unstressed specimens. Examination of metallographically polished cross sections showed no localized attack, and surface oxide could not be detected at a magnification of 500 \times .

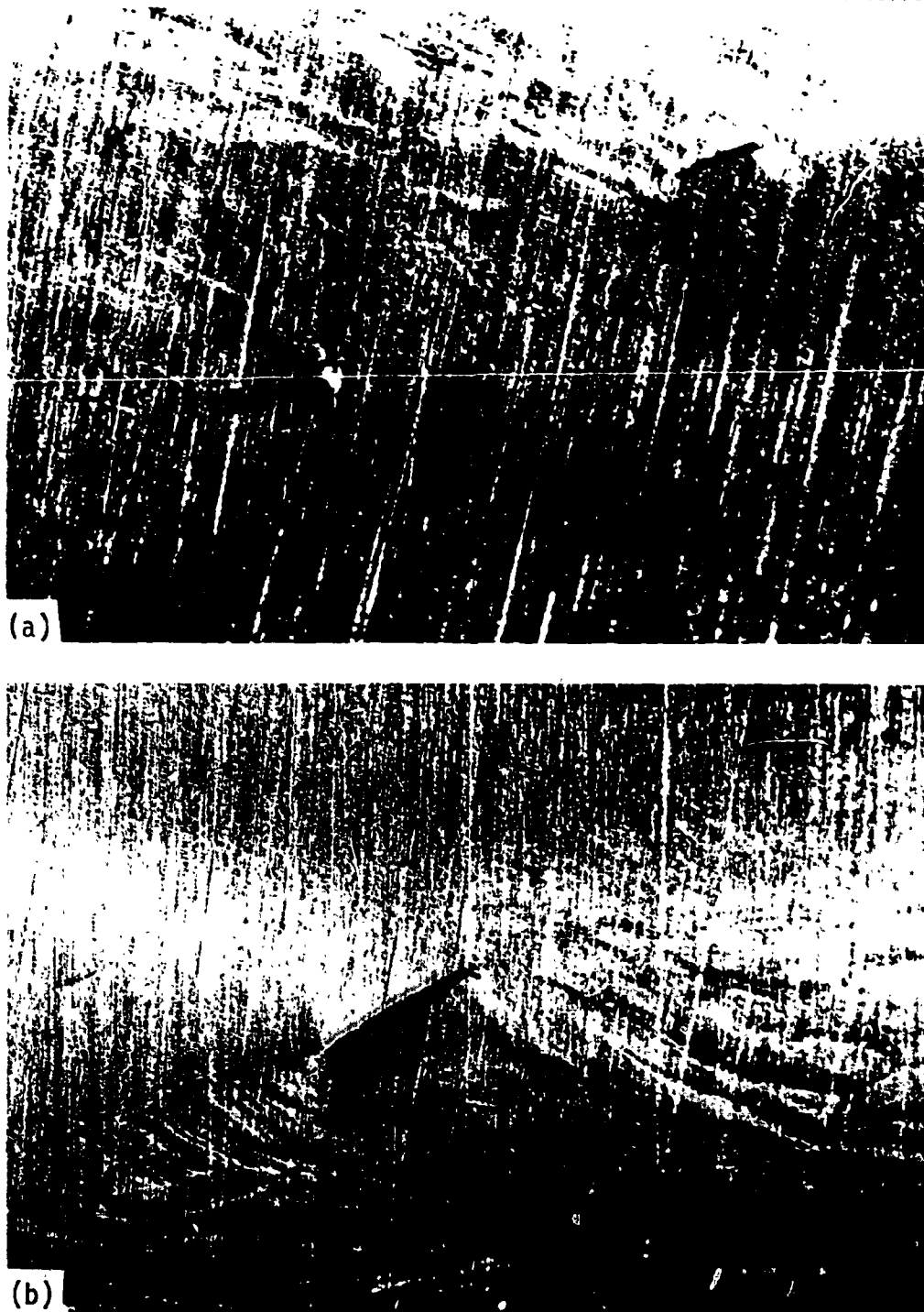


Fig. 6. Small Defects Caused by Lack of Fusion in Welded Specimens. Photographs taken before exposure to steam. (a) Fusion zone between 2 1/4 Cr-1 Mo steel and Inconel 62 filler metal. (b) Fusion zone between Incoloy Alloy 800 and Inconel 62 filler metal. 20 \times . Reduced 18.5%.

Table 4. Average Weight Gains of Inconel 617
Corrosion Test Specimens in Superheated Steam

Time (h)	Weight Gain, g/m ²			
	Unwelded Specimens		Welded Specimens	
	755 K	811 K	755 K	811 K
504	0.10	0.36	0.32	0.40
1,621	0.14	0.34	0.32	0.60
3,051	0.16	0.40	0.39	0.64
5,073	0.16	0.42	0.42	0.73
8,005	0.22	0.43	0.46	0.89
11,731	0.23	0.62	0.50	0.90

TYPE 304 STAINLESS STEEL

The weight gains experienced by the type 304 stainless steel specimens are shown in Fig. 7. Only annealed specimens with ground surfaces were tested. At both temperatures the weight changes of the replacement specimens were lower at a given time than of the specimens originally installed in the loop, but at the higher temperature the corrosion rates are comparable after 5000 h. This difference may be partly attributed to the fact that attack was not uniform during the first 8000 h of test. This is illustrated in Fig. 8, which shows typical corrosion of surfaces exposed for 3051, 5073, and 8005 h at both 482 and 538°C. Based on the linear part of the curves, calculated penetration rates are 1.5 and 4.1 $\mu\text{m/y}$ (0.06 and 0.16 mpy) at 482 and 538°C, respectively.

SANDVIK HT-9

The HT-9 corrosion specimens were made from 81.8-mm-OD (1 1/4-in.) pipe with a 3.6-mm (0.142-in.) wall. The pipe was quartered axially and the pieces were rolled to a thickness of 1.6 mm (1/15 in.). The flats were solution annealed at 1050°C (1323 K) for 30 min in argon, furnace cooled, tempered at 760°C (1033 K) for 30 min in argon, and again furnace cooled. Specimens were then cut from the flat strips. Those specimens

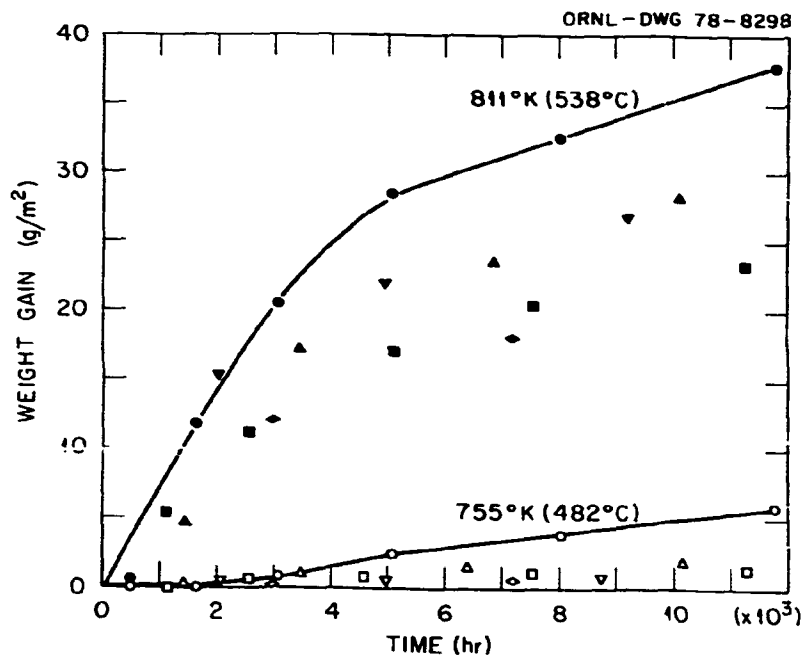


Fig. 7. Weight Gains of the 304 Stainless Steel Specimens in Superheated Steam. \square - Single Replacement Specimen for One Removed after 504 h. \triangle - Single Replacement Specimen for One Removed after 1621 h. ∇ - Single Replacement Specimen for One Removed after 3051 h. \diamond - Single Replacement Specimen for One Removed after 5073 h. \circ - Average of Specimens in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

installed originally were ground on a 100-mesh belt grinder. Later in the test another set of specimens from the same material was included in the test array, but the surfaces were not ground; the flat surfaces were as removed from the furnace.

The weight gains of the ground specimens are shown in Fig. 9. In most cases the replacement specimens agreed reasonably well with those installed originally. Based on the slope of the straight line part of the curves, the corrosion rates are 3.1 and 2.3 $\mu\text{m/y}$ (0.12 and 0.09 mpy) at 482 and 538°C, respectively. The reason for the lower rate at the higher temperature is not apparent, but in both cases the corrosion rates are low.

Cross-sectional views of the oxide formed on specimens exposed for 3051, 5073, and 8005 h are shown in Fig. 10. The oxide on each specimen is composed of the usual two layers plus a third oxidation layer at the

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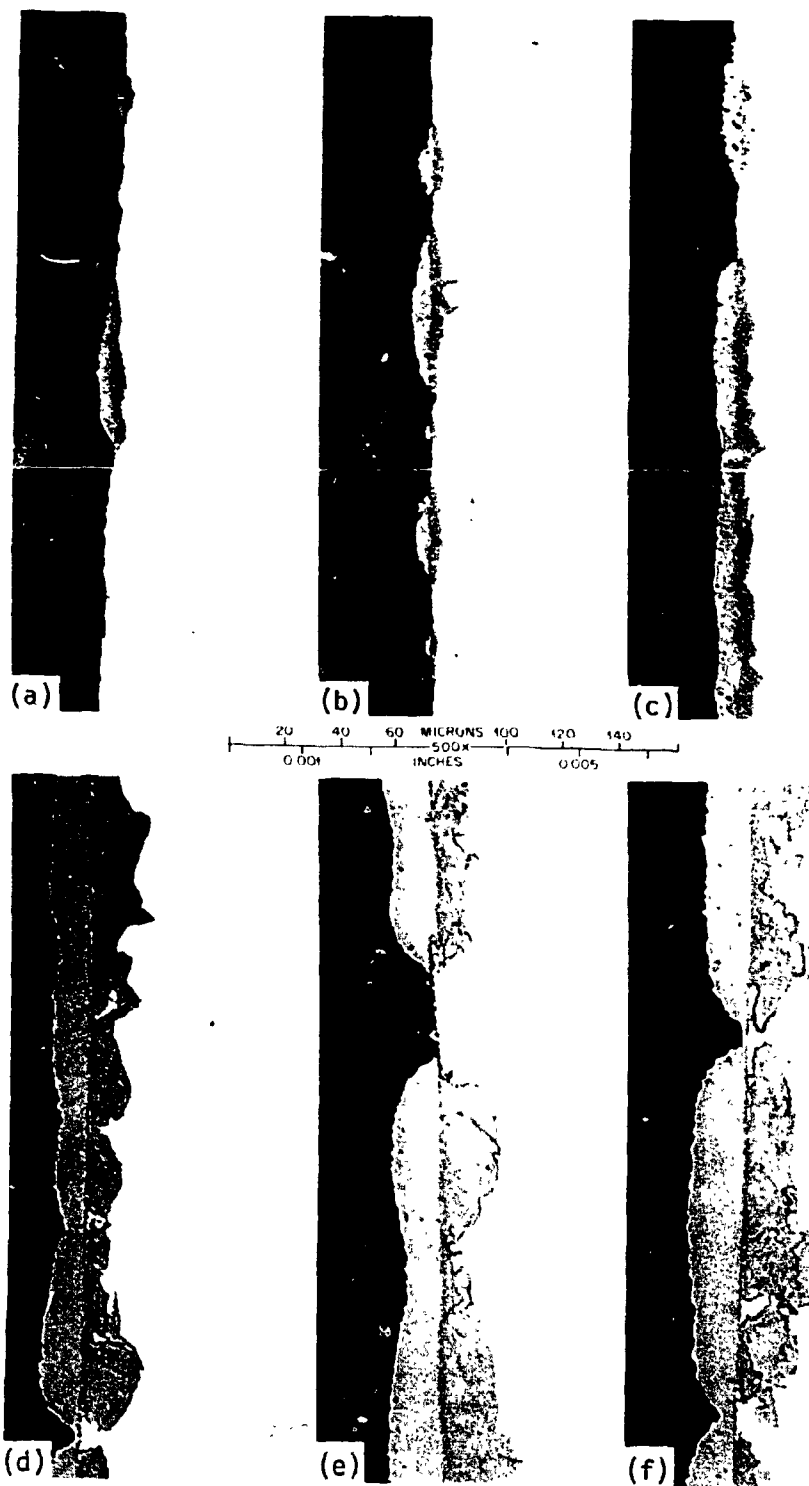


Fig. 8. Cross Sections Through Type 304 Stainless Steel Specimens After Exposure in Superheated Steam. (a) 3051 h, (b) 5073 h, and (c) 8005 h at 482°C (755 K). (d) 3051 h, (e) 5073 h, and (f) 8005 h at 538°C (811 K).

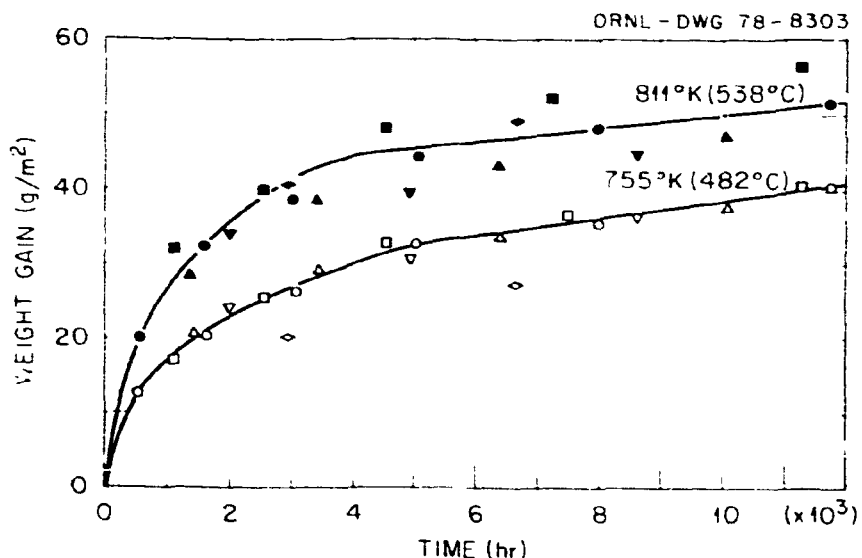


Fig. 9. Weight Gains of Sandvik HT-9 Steel Specimens in Superheated Steam. \blacksquare — Single Replacement Specimen for One Removed after 504 h. \bullet — Single Replacement Specimen for One Removed after 1621 h. \blacktriangledown — Single Replacement Specimen for One Removed after 3051 h. \diamond — Single Replacement Specimen for One Removed after 5073 h. \circ — Average of Specimens in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

oxide-metal interface. Interestingly, the specimens exposed at 482°C had a third layer more distinct than did the ones exposed at 538°C. Electron microprobe analyses showed that both the innermost oxidation layer and the inner layer of the two-layered oxide contained chromium and molybdenum in about the same volume concentration as the substrate metal. The outer layer was essentially pure iron oxide. The composition of the respective layers was the same at each temperature.

At the time this report was prepared the specimens not ground (annealed and tempered surface) had been in test for only 3726 h. Average weight gains at that time were 19.2 and 34.8 g/m² at 482 and 538°C, respectively. Comparison of these values with those in Fig. 9 shows that the annealed specimens gained less weight at both temperatures than did those with the ground surfaces. This may be due to the fact that the underground specimens had a smaller true surface than the ground ones, or alternatively, that the unground specimens had a thin surface oxide film that inhibited attack.

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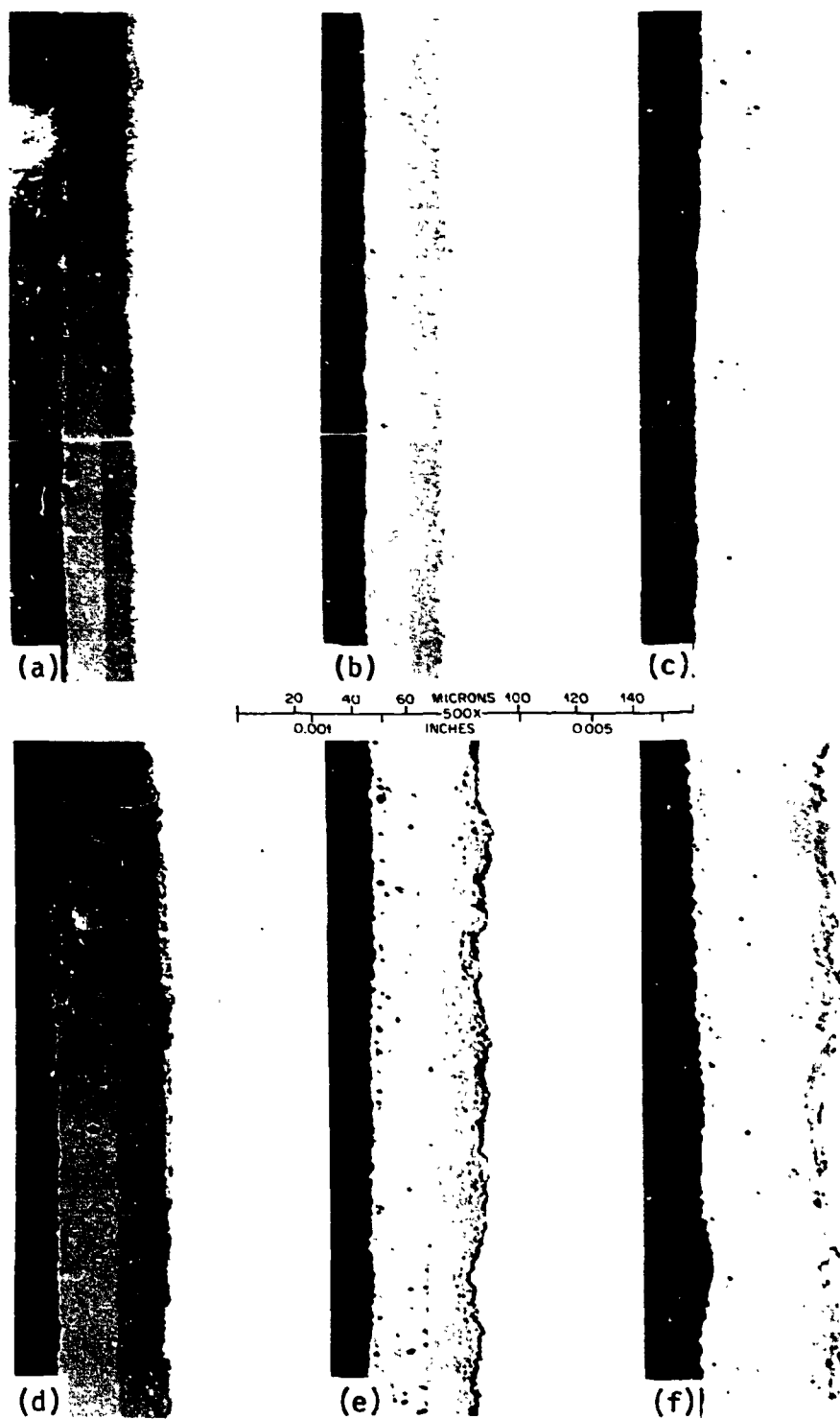


Fig. 10. Cross Sections Through Sandvik HT-9 Specimens After Exposure to Superheated Steam. (a) 3051 h, (b) 5073 h, and (c) 8005 h at 482°C (755 K). (d) 3051 h, (e) 5073 h, and (f) 8005 h at 538°C (811 K).

Specimens of two additional heats of Sandvik HT-9 (heats 451774 and 454191) were supplied to us by personnel at the General Atomic Company. These specimens had been machined from large-diameter thick-wall pipe. One set of specimens was machined from each material so that one face of the specimen was the interior pipe-wall surface. This surface was as received from the mill and was covered with a relatively thick black scale that was not disturbed during preparation. The opposite flat face and the edges were ground before test. Also, specimens from heat 454191 were machined from the middle of the pipe wall and all their surfaces were ground. Some of these latter specimens were subsequently heavily pickled in HNO_3 -HF solution to remove cold-worked surfaces. So far, the testing time of the latter HT-9 specimens has been only 2737 h. Their average weight gains are shown in Table 5.

Table 5. Average Weight Gains of the Sandvik HT-9 Specimens^a after 2737-h Exposure to Superheated Steam

Heat Number	Surface Condition	Weight Gain, g/m ²	
		482°C (755 K)	538°C (811 K)
451774	Mill Oxide — Ground	12.4	23.5
454191	Mill Oxide — Ground	12.2	22.8
454191	Ground	19.1	37.1
454191	Pickled	18.5	45.0

^aSupplied by General Atomic Company.

Values from the two heats of HT-9, consisting of specimens with part of their surface covered with oxide, agreed very well. Comparison of these specimens with the all-ground specimens of one heat shows that the mill scale on one surface had a significantly beneficial effect. The effect of pickling is not clear; there appeared to be little or no effect at 482°C, but at 538°C the average weight gain of the pickled specimen was about 20% greater than that of the specimen with the ground surfaces. The average weight gain of specimens with all-ground surfaces was only somewhat less at the same exposure time than that of similar

specimens of HT-9 in test from the start (see Fig. 9). However, remember that each specimen has been weighed only once, so firm conclusions should await longer exposures.

SUMITOMO 9 Cr-2 Mo STEEL

Specimens of this alloy were also supplied by General Atomic Company, and these specimens were also machined from heavy-wall pipe. One group of specimens was machined so that one of the "flat" surfaces was the interior wall of the pipe, which was covered with a black scale. These specimens were ground on all other surfaces. A second group of specimens was machined from the interior of the pipe wall and all their surfaces were ground. Part of this group was heavily pickled to remove the cold-worked surface.

These specimens have been weighed only once — after 2737 h. Those specimens with mill scale on one face gained average weights of 17.5 and 51.1 g/m² at 482 and 538°C, respectively. Comparable values for those specimens with all-ground surfaces were 23.2 and 51.4 g/m². Pickling of ground specimens had no significant effect on weight gain at either temperature. In contrast to the effect of mill scale on the HT-9 specimens, its effect on the 9 Cr-2 Mo specimens was small: at 482°C its presence appeared to have a slightly beneficial effect, but at 538°C the average weight gain of the specimens with all surfaces ground was about the same as that of specimens partially covered with oxide at the start of the test.

9 Cr-1 Mo STEEL

The weight gains experienced by ground 9 Cr-1 Mo steel test specimens are shown in Fig. 11. The corrosion rates calculated from the slope of the linear parts of the curves are 4.6 and 4.8 μm/y (0.18 and 0.19 mpy) at 482 and 538°C, respectively. The weight changes of replacement specimens agreed reasonably well with those of specimens in test from the start.

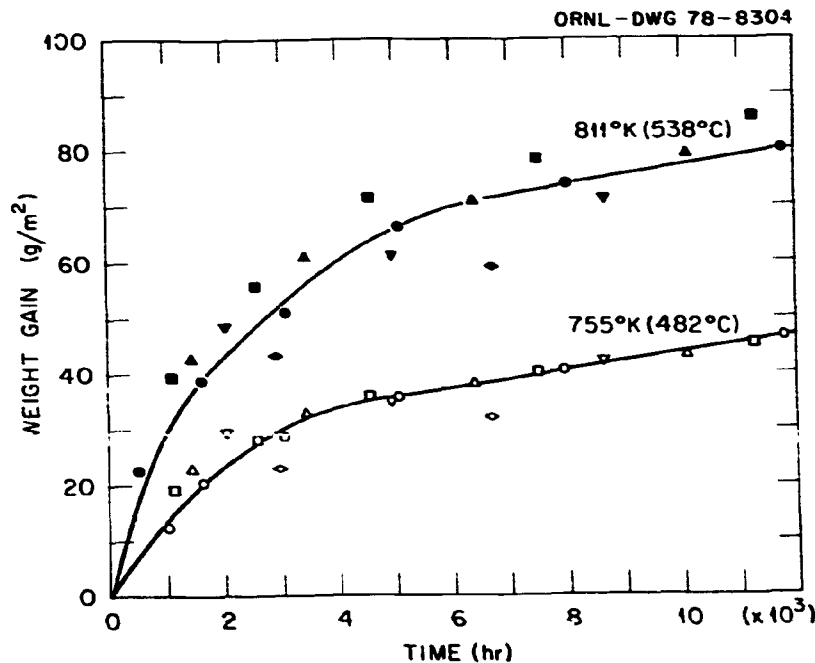


Fig. 11. Weight Gains in 9 Cr-1 Mo Steel Specimens in Superheated Steam. \square - Single replacement specimen for one removed after 504 h. \triangle - Single replacement specimen for one removed after 1621 h. ∇ - Single replacement specimen for one removed after 3051 h. \diamond - Single replacement specimen for one removed after 5073 h. \circ - Average of specimens in test from start of test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

Photomicrographs of cross sections of the oxides after 3051, 5072, and 8005 h are shown in Fig. 12. In each case the oxide film consisted of a two-layered outer scale in addition to a thin inner oxidation layer. In contrast to that of the HT-9 specimens, the inner oxidation layer was more distinct at 538°C than at 482°C. Ion microprobe analyses of the oxide revealed the expected composition: both the inner oxide and the internal oxidation layer contained chromium and molybdenum in the same volume concentration as the alloy, and the outer layer contained only iron oxide.

Some scattered points on the surfaces showed very little attack as compared with the bulk of the surface. Typical examples are evident in Fig. 12. Electron microprobe analyses of metal in these areas showed the composition to be the same as that of the bulk alloy. We cannot currently explain the absence of significant attack on these localized areas.

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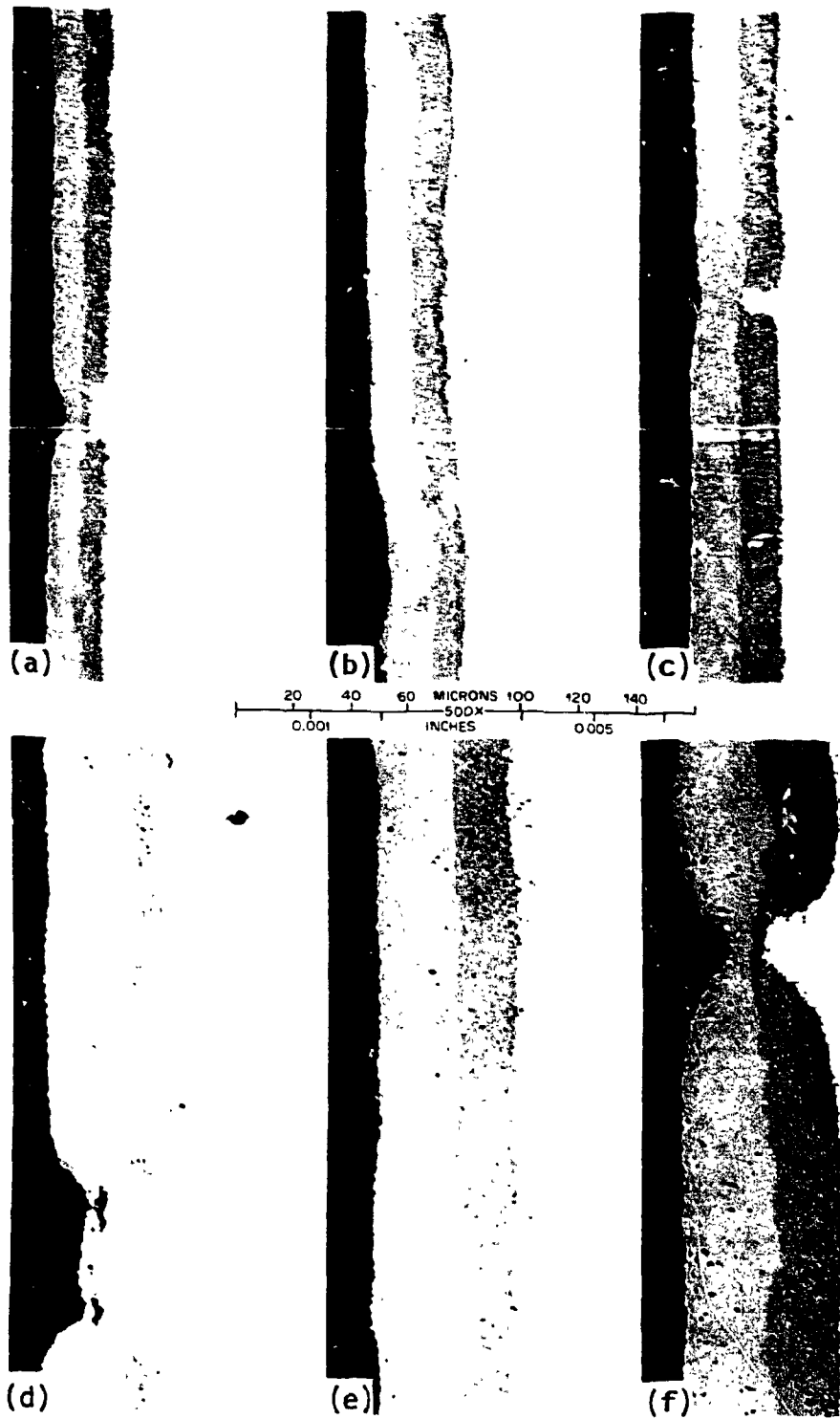


Fig. 12. Cross Sections Through 9 Cr-1 Mo Steel After Exposure in Superheated Steam. (a) 3051 h, (b) 5073 h, and (c) 8005 h at 482°C (755 K). (d) 3051 h, (e) 5073 h, and (f) 8005 h at 538°C (811 K).

2 1/4 Cr-1 Mo STEEL

Three sets of specimens made of this material and having different surface preparations have been in the loop since the test began. After machining, all specimens were ground on a 100-mesh belt grinder; one set was tested in this condition. The rest of the specimens were annealed in argon for 2 h at 732°C (1005 K) and furnace cooled. Half of these were tested without further surface preparation. Impurities in the argon caused the specimens to discolor slightly. To remove this film the remaining specimens (half) were pickled in HNO_3 -HF solution before the test.

The weight gains of the group of specimens installed at the beginning of the test showed no differences attributable to surface preparation. Therefore, all specimens tested for a given time and temperature were averaged to obtain the data presented in Fig. 13. The points through which the curves are drawn were averages of 21 measurements at 504 h, decreasing to 6 measurements at 8005 h. Similarly, each point for the replacement specimens represents the average of three measurements. The only evidence of oxide spallation was a few very small chips of oxide missing from two of the specimens exposed at 538°C for 11,731 h.

The slopes of the straight-line portions of the curves in Fig. 13 correspond to corrosion rates of 4.3 and 4.8 $\mu\text{m}/\text{y}$ (0.17 and 0.19 mpy) at 482 and 538°C, respectively. Generally, the weight gains of the replacement specimens agreed satisfactorily with those of the specimens originally installed in the loop.

Cross-sectional views of the oxide after 3051, 5072, and 8005 h are shown in Fig. 14. The corrosion product was composed of two layers, but the inner oxidation layer found on HT-9 and 9 Cr-1 Mo steel specimens was not detected on the 2 1/4 Cr-1 Mo steel specimens.

Specimens with autogenous welds showed weight gains similar to, but somewhat below, the curves drawn in Fig. 13. Table 6 lists the average weight gains for four specimens tested at each temperature.

U-bends made from strips with autogenous welds across the middle showed no cracks and no unusual tendency for oxide to spall from them.

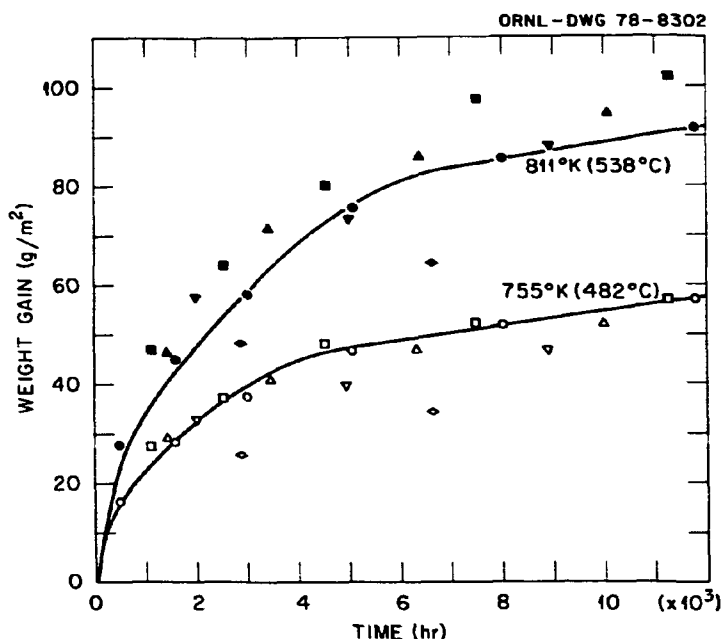


Fig. 13. Weight Gains of 2-1/4 Cr-1 Mo Steel Specimens in Superheated Steam. \square — Average of Three Replacement Specimens for One Removed after 504 h. \triangle — Average of Three Replacement Specimens for One Removed after 1621 h. ∇ — Average of Three Replacement Specimens for One Removed after 3051 h. \diamond — Average of Three Replacement Specimens for One Removed after 5073 h. \circ — Average of Specimens in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

Welded U-bends in which 2 1/4 Cr-1 Mo steel was joined to Incoloy Alloy 800 also showed no unusual oxidation behavior or cracks.

9 Cr-Mo EXPERIMENTAL ALLOYS

Three experimental alloys of 9 Cr-Mo steel with minor alloying additions were supplied to us by personnel of the Combustion Engineering Corporation. We received the materials as 1.6-mm-thick (1 1/6-in.) sheets normalized at 1038°C (1311 K) for 10 min in an argon atmosphere, air cooled, tempered at 760°C (1033 K) for 1 h, and again air cooled. Standard-size coupons were made from the sheets and all surfaces were ground on a 100-mesh belt grinder. All three alloys were from electrosag remelt heats.

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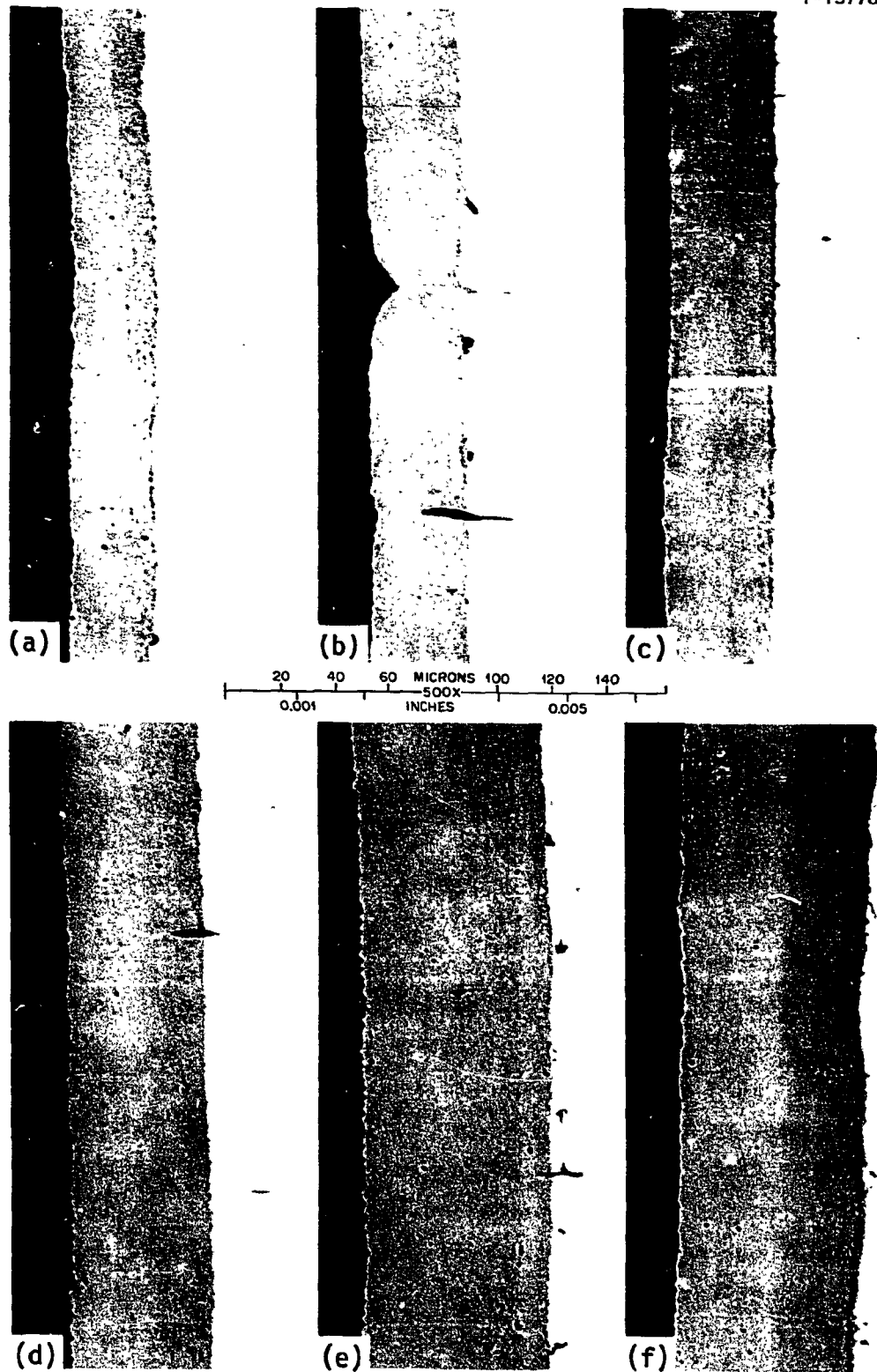


Fig. 14. Cross Sections Through 2 1/4 Cr-1 Mo Steel Specimens After Exposure in Superheated Steam. (a) 3051 h, (b) 5073 h, and (c) 8005 h at 582°C (755 K). (d) 3051 h, (e) 5073 h, and (f) 8005 h at 538°C (811 K).

Table 6. Average Weight Gains of 2 1/4 Cr-1 Mo Steel Specimens with Autogenous Welds Exposed to Superheated Steam

Test Time, h	Average Weight Gain, g/m ²	
	482°C (755 K)	538°C (811 K)
504	14.4	27.0
1,621	24.2	42.0
3,051	31.0	51.5
5,073	38.7	65.9
8,005	43.5	71.2
11,731	47.8	79.4

Figures 15, 16, and 17 show the average weight gains of the specimens. The longest test time to date has been 8680 h. The results from heats ESR-XA3177 and ESR-XA3178 very closely agreed (Figs. 15 and 16), whereas the weight gains of specimens from the third heat (91887) were somewhat higher. The lower silicon content of the alloy may account for this (Table 1). In all cases the weight gains of replacement specimens agreed well with those of specimens that were installed first.

Plots of the logarithm of weight gain versus the logarithm of time give approximately straight lines, except in each case the last point falls slightly below the extrapolated line. Additional exposure time will be required to determine with certainty whether the rate of weight gain is becoming constant or is still decreasing.

DISCUSSION

So far in this study our assessment of corrosion has been limited to weight-gain measurements and, in a few cases, to metallographic examination of corroded surfaces. Weight gain is a valid measure of corrosion only if all the corrosion product remains on the specimen and if the chemical composition of the corrosion product is known. The composition of the corrosion product formed on low-alloy ferritic steels is well known,^{8,9} and for the more resistant alloys we tested, the stoichiometry of the corrosion product can be estimated with sufficient accuracy, particularly in view of the very slight corrosion of the latter alloys (except pickled

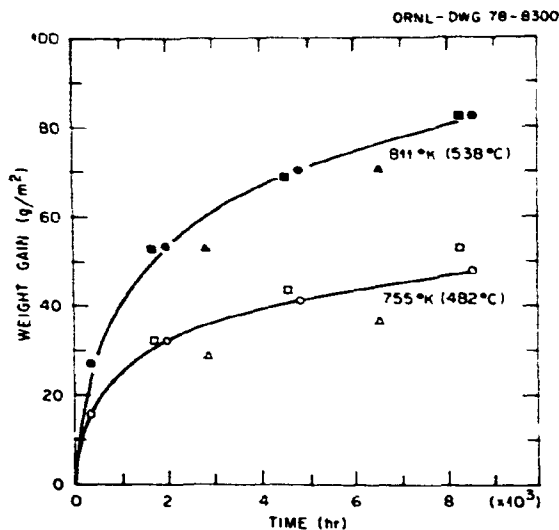


Fig. 15. Weight Gains of 9 Cr Steel (heat ESR-XA3177) Specimens in Superheated Steam. \square — Average of Specimens Added to Loop 323 h after Start of Test. \triangle — Average of Specimens Added to Loop 2022 h after Start of Test. \circ — Single Specimen in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

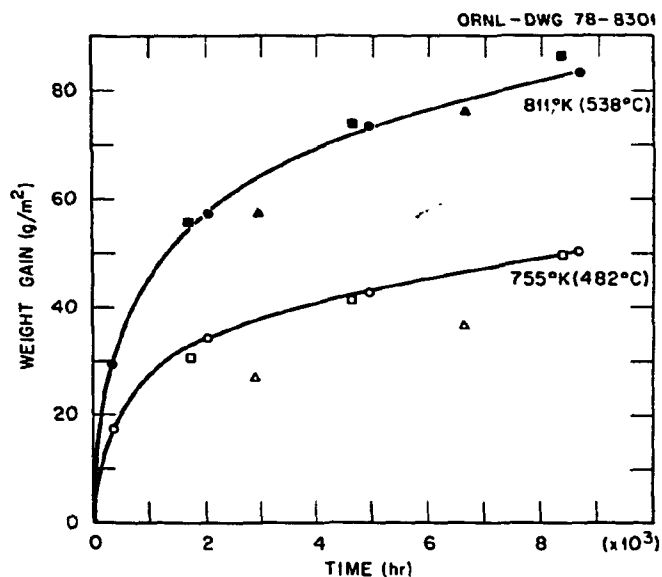


Fig. 16. Weight Gains of 9 Cr Steel (heat ESR-XA3178) Specimens in Superheated Steam. \square — Average of Specimens Added to Loop 323 h after Start of Test. \triangle — Average of Specimens Added to Loop 2022 h after Start of Test. \circ — Single Specimen in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

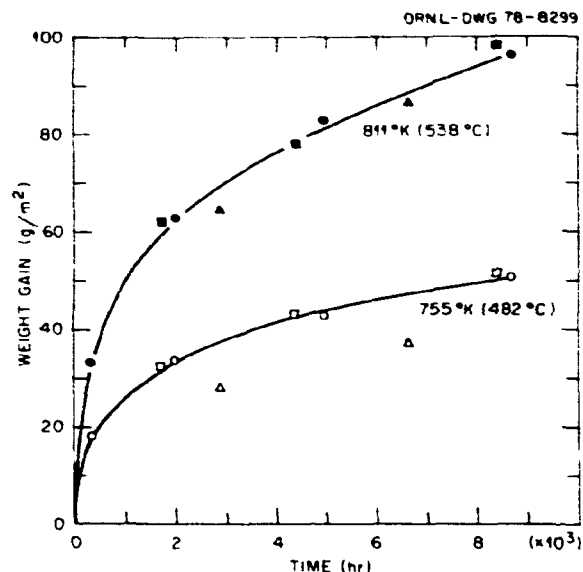


Fig. 17. Weight Gains of Experimental 9 Cr Steel (heat 91887) Specimens in Superheated Steam. \square — Average of Specimens Added to Loop 323 h after Start of Test. \triangle — Average of Specimens Added to Loop 2022 h after Start of Test. \circ — Single Specimen in Test from Start of Test. Solid symbols represent specimens tested at 538°C and open symbols those tested at 482°C.

Incoloy Alloy 800). Visual and metallographic examinations of all specimens have revealed no significant loss of oxide at the longest test times to date. Furthermore, attack was quite uniform, except on pickled Incoloy Alloy 800, on the type 304 stainless steel, and on a few insignificantly small areas on the 9 Cr-1 Mo steel that suffered very little attack. Therefore, weight gain is a reliable measure of corrosion in our tests. Note, however, that at some later time exfoliation or spalling of oxide from the ferritic steels will occur, at which time corrosion will not be directly correlated with weight gain. The loss of a few chips of oxide from the 2 1/4 Cr-1 Mo steel tested at the higher temperature indicates that such a time is nearly at hand for this alloy.

Although this testing program is continuing and any conclusions must remain tentative until corroborated by future test data, certain facts and trends are apparent from the present data. In the following paragraphs we discuss these, and where possible, compare our results with those obtained by others under similar conditions.

For all materials tested, we observed that weight changes of specimens of a given material that were started in test at the same time and

temperature usually agreed well at all time intervals, while weight gains of specimens added at different times after the test had started frequently varied appreciably at comparable exposure times. Apparently, the film that forms during the very early stages of corrosion influences the weight gain for at least the first few thousand hours. The data suggest, however, that the corrosion rate after 3000 to 5000 h becomes similar for all specimens of the same material tested at the same temperature.

As expected, corrosion of Inconel 617, Incoloy Alloy 800, and type 304 stainless steel has been very slight under our test conditions. The corrosion rates of ground surfaces of Inconel 617 and Incoloy Alloy 800 have been particularly low: averaged over the first 11,731 h, the penetration rate was only 0.2 to 0.3 $\mu\text{m}/\text{y}$ in the worst cases. Both these alloys can be safely used in pure steam at substantially higher temperatures than in our test. Hurst and Cowen¹⁰ showed a weight gain of about 1.5 g/m^2 for Incoloy Alloy 800 after 12,000 h in steam at 550°C (823 K). Our value at 538°C (811 K) was only half theirs (0.7 g/m^2) for ground surfaces; it was 1.9 g/m^2 for annealed surfaces after 11,731 h. Hence, our data agree reasonably well with those of Hurst and Cowen. On the other hand, the data presented by McCoy and McNabb^{2,3} for supercritical water show higher weight gains — 8 to 9 g/m^2 after 13,000 h at 538°C.

The sensitized specimens of Incoloy Alloy 800 that had undergone intergranular attack as a result of pickling gained substantial weight, apparently because of their very large true surface area. The depth of intergranular attack did not increase — at least during the first 8000 h of test — indicating that sensitized and pickled Incoloy Alloy 800 components incorporated in a steam system presents no special problems due to steam corrosion. However, when an aqueous phase and low levels of oxygen are present, chloride contamination makes stress-corrosion cracking much more likely in sensitized Incoloy Alloy 800 than in the fully annealed material. Heat-affected zones of Incoloy Alloy 800 welds have shown a high susceptibility to chloride-induced stress corrosion cracking^{11,12} under these conditions.

That cold work increases the corrosion resistance of Incoloy Alloy 800 in steam has been reported by others^{3,5} and our data show this.

Since all corrosion damage to our specimens was slight, it was not as apparent as in the data of Leistikow,⁵ who studied in detail the corrosion of Incoloy Alloy 800 in very high-temperature steam as a function of cold work.

Corrosion of type 304 stainless steel, while greater than that of either Inconel 617 or Incoloy Alloy 800, has also been slight in our test. For the first 8000 h of test, corrosion was not uniform, as Fig. 8 exemplifies, but these photomicrographs suggest that continued exposure will make the scale more nearly uniform. The scale consisted of two distinct layers, the interface between them clearly marking the original surface of the stainless steel. Eberle and Kitterman¹³ determined the elemental composition of scale on type 304 stainless steel formed in 649°C (922 K) steam during 2000 h. The inner layer contained chromium and nickel in concentrations greater than the substrate alloy and the outer layer contained only iron oxide. While their tests were conducted at higher temperatures than ours and they did not obtain quantitative data, our results seem to agree, at least qualitatively, with theirs. McCoy and McNabb³ also examined the corrosion of type 304 stainless steel at 538°C. The weight gain of the single specimen exposed was 26.0 g/m² after 9000 h. This value agrees well with our replacement specimen exposed for the same time. However, the weight gain of their specimen was inexplicably lower after longer test times. A cross section through the specimen after a 12,000-h exposure showed uneven attack, much like that on our specimens.

Our results with the ferritic steels agree reasonably well with those reported by other investigators. Thus, the scale found on these materials consisted of two layers plus an inner oxidation band in the case of the 9 to 12% Cr alloys. Although we have not at this time examined the nature of the scales on the experimental alloys supplied by Combustion Engineering or of those on the alloys furnished by the General Atomic Company, they will undoubtedly have similar structures. Weight gains experienced by all the ferritic alloys at the same exposure time and temperature are quite similar, except that HT-9 appears to gain less weight than do the other alloys.

In Table 7 we have listed the oxide thicknesses on specimens of 2 1/4 Cr-1 Mo, 9 Cr-1 Mo, and HT-9 steels as measured from the photomicrographs shown in Figs. 10, 12, and 14. Assuming the oxide to have a stoichiometric composition of $(\text{Fe}, \text{Cr})_3\text{O}_4$ and a theoretical density of 5.2 mg/mm^3 , we have calculated the expected weight gains and have compared these with the weight gains shown by the curves in Figs. 9, 11, and 13. When one considers the spread of the data shown in the figures and the fact that the actual density of the corrosion product is probably less than the theoretical density, the oxide thickness and weight gain measurements agree adequately. In fact, if the density of the corrosion product were about 80% of the theoretical value, the average calculated values would equal the average measured values.

Table 7. Comparison of Measured Weight Gains with Those Calculated from Oxide Thicknesses

Alloy	Time (h)	Data Collected at 482°C			Data Collected at 538°C		
		Oxide ^a Thickness (μm)	Weight of ^b Oxygen (g/m^2)	Weight Gain ^c of Specimen (g/m^2)	Oxide ^a Thickness (μm)	Weight of ^b Oxygen (g/m^2)	Weight Gain ^c of Specimen (g/m^2)
2 1/4 Cr-1 Mo	3051	32	46	40	52	76	58
	5073	38	55	48	72	105	76
	8005	41	60	52	72	105	85
9 Cr-1 Mo	3051	22	32	30	40	58	53
	5073	30	44	36	50	73	66
	8005	32	47	47	58	85	74
HT-9	3051	25	36	28	40	58	41
	5073	27	39	33	43	63	46
	8005	29	42	36	45	66	48

^aAverage oxide thickness measured from photomicrographs shown in Figs. 10, 12, and 14.

^bCalculated weight of oxygen based on theoretical density of oxide (5.2 mg/mm^3) and its thickness.

^cWeight gain of specimen determined from curves in Figs. 9, 11, and 13.

Tests on 2 1/4 Cr-1 Mo and 9 Cr-1 Mo steels have been conducted by others under conditions similar to ours, so a comparison of our results with theirs is of interest. McCoy and McNabb^{2,3} exposed two sets of specimens to supercritical water at TVA's Bull Run steam plant. Each set was composed of several chromium-molybdenum alloys ranging from 1.1 to 8.9% Cr. One set was exposed for 7000 h and the other for 14,000 h. Log-log plots of weight gain versus time yielded straight lines, but the

slopes were significantly less than 0.5. All alloys behaved nearly the same, thus indicating that chromium concentration in the alloy was not important in determining weight gain. In fact, the authors suggest that differences in silicon content of the different alloys may account for the small differences among the alloys. For the first 5000 h of our test the weight gains of our specimens agreed well with the weight gains of theirs, but after longer times ours tended to be lower.

Hurst and Cowen¹⁰ showed that at 550°C (823 K) in superheated steam 2 1/4 Cr-1 Mo steel began to exfoliate after 3000 h, but up to that time our values were close to theirs. Interestingly, McCoy and McNabb² reported no evidence of exfoliation even after 14,000 h at the slightly lower temperature of 538°C (811 K). Hurst and Cowen¹⁰ also reported that 9 Cr-1 Mo steel in steam at 550°C gained weight logarithmically for about 8000 h, when exfoliation of the oxide began. Again, our average weight gain at 538°C agrees reasonably well with their value. They also showed that increasing the silicon content more effectively reduced the corrosion (weight gain) of chromium-molybdenum alloys than did increasing the chromium content in the range 2 1/4 to 9%. The importance of silicon in these alloys appears to have been overlooked by many investigators. This probably explains why different values for corrosion of chromium-molybdenum alloys have been obtained under the same test conditions.

The corrosion data for 2 1/4 Cr-1 Mo steel summarized by DeVan and Griess¹ also agree with our data. For example, at 482°C (755 K) the penetration of 17 μm in 5000 h is equivalent to a weight gain of 51 g/m², which approximates the value, 48 g/m², shown in Fig. 13.

Leistikow et al.¹⁴ examined the effect of cold work on the corrosion resistance of chromium-containing ferritic steels in steam during 1000 h tests at 550°C (823 K). For a steel containing 0.83% Cr, cold work had no effect on corrosion. A slight and beneficial effect of cold work was noted in an alloy containing 6.73% Cr, but the presence of aluminum also in the alloy may have influenced the results. At higher chromium concentrations cold work had a more pronounced effect in reducing corrosion; the beneficial effect approached that observed in the austenitic stainless steels.⁵ Our results that indicate no effect of cold work (surface grinding) on the corrosion of 2 1/4 Cr-1 Mo steel agree with the data reported by Leistikow et al.¹⁴

The above comparisons show that our test data agree well with those of others for the early stages of test. Not only are weight gains comparable but, furthermore, the oxidation followed essentially parabolic kinetics in both cases. However, after longer test times, in contrast to other data, our results indicate a change in kinetics from a constantly decreasing rate to a low constant rate, even though we find no evidence of scale deterioration or loss. From the remaining 18,000 h of the test, we hope to establish whether or not the present interpretation of our results is correct, that is, that the rate of corrosion becomes constant after about 5000 h.

SUMMARY

This is an interim report that summarizes the data obtained during the first 12,000 h of a planned 30,000-h test of various materials in superheated steam at 482 and 538°C (755 and 811 K). The principal observations during the first 12,000 h were:

1. Inconel 617 and Incoloy Alloy 800 specimens with mill-annealed and ground surfaces corroded at very low rates at both temperatures. Test specimens of Incoloy Alloy 800 that had been sensitized and intergranularly corroded during pickling corroded more extensively than did other specimens on subsequent exposure to steam, but intergranular penetration did not progress.
2. Type 304 stainless steel did not oxidize uniformly, but even in the affected areas corrosion was slight. With increasing exposure time the attack has tended to become more nearly uniform.
3. The ferritic steels developed rather heavy uniform scales, which were thicker at the higher temperature. The scales on Sandvik HT-9 were only slightly thinner than those on 2 1/4 Cr-1 Mo and 9 Cr-1 Mo steels. After about 5000 h the corrosion rates on all three materials were low and constant at both temperatures — generally between 2.5 and 5 $\mu\text{m}/\text{y}$ (0.1 and 0.2 mpy). During the first 11,731 h no significant exfoliation of the corrosion-product oxide has occurred, but two specimens of 2 1/4 Cr-1 Mo steel at the higher temperature are missing a few chips of oxide, indicating that significant exfoliation for that alloy is imminent.

4. Experimental 9% Cr steels supplied by Combustion Engineering Corporation and commercial alloy test specimens supplied by General Atomic Company, some with mill scale on parts of the specimens (HT-9 and 9 Cr-2 Mo), have been in test for shorter periods, but preliminary examinations indicate behavior comparable to that of the other ferritic steels.

5. We have noted no unusual effects on welded specimens, and have observed neither cracking of nor a tendency for oxide to spall from plastically and elastically deformed U-bend specimens.

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