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LABORATORY CORROSION TESTS OF WELDED LOW-CARBON STAINLESS STEEL

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ABSTRACT

Welded specimens of low-carbon stainless steel (less than 0.06 percent of carbon) were exposed to the corrosion attack of three different solutions. Results of these tests were as follows:

In a copper sulphate-sulphuric acid solution, intergranular corrosion did not occur in either plate or weld metal regardless of the heat treatment.

In boiling nitric acid, intergranular corrosion occurred only in certain heat-treated plates of low-carbon stainless steel. Intergranular corrosion did not occur in welded metal, with or without heat treatment.

In hydrochloric acid of high concentration, the corrosion rate of plate metal was not affected by heat treatment, but the corrosion rate of weld metal was considerably decreased by heat treatment at high temperatures. Intergranular corrosion was not observed in any specimen exposed to this reagent.

These results can be explained on the basis of selective attack by the corrosive agents on certain constituents of the steel samples.

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I. INTRODUCTION

The incorporation of laboratory corrosion tests into specifications for stainless steels has caused considerable controversy regarding the interpretations of results of such tests.

Three types of corrosion are usually recognized, (1) general, which is a simple chemical reaction over most of the surface; (2) pitting, depending upon surface conditions found in localized areas and bearing no relation to structural features; and (3) intergranular, i. e.; localized corrosion progressing along grain boundaries.

The unsatisfactory behavior in service of supposedly corrosion-resistant alloys has been traced in part to intergranular corrosion that is associated with chromium-rich carbides localized in or at the grain boundaries. It is well established that heating within the temperature range of 550° to 800° C ("sensitization") accelerates the formation

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of these carbides and hence increases the susceptibility to intergranular attack. During welding, certain portions of the metal adjacent to the weld are heated to within this range. Consequently, a welding test is frequently used to determine the susceptibility of material to carbide precipitation and intergranular corrosion.

Several laboratory corrosion methods have been recommended for testing materials that have been sensitized. It has been claimed that the results of these tests are a combination of intergranular, pitting and general corrosion and that no one of them is entirely reliable.

The investigation here reported was a study of these laboratory tests to determine their reliability for indicating susceptibility to intergranular corrosion of welded low-carbon 18-8 steel.

II. MATERIAL

The material from which the welded specimens were prepared was $\frac{3}{8}$ -inch (1 cm) plate of a low-carbon corrosion-resisting steel of the 18-8 type, purchased by the United States Navy Department for general welding tests. Arc welds were made with coated welding rods of about the same composition.

The chemical compositions of the plate, welding rod and deposited weld metal are given in table 1, and the type composition of the coating is given in table 2.

TABLE 1.—*Chemical composition of plate, welding rod, and deposited weld metal*¹

Elements	Plate	Welding rod	Weld metal
	%	%	%
Carbon.....	0.054	0.06	0.06
Manganese.....	.40	.52	.50
Phosphorus.....	.025	.029	.031
Sulphur.....	.016	.007	.005
Silicon.....	.30	.34	.14
Chromium.....	18.59	19.26	18.86
Nickel.....	9.30	9.07	9.09
Copper.....	.01	.01	.01
Titanium.....	nil	nil	nil
Vanadium.....	.045	.04	.03
Molybdenum.....	nil	nil	nil
Selenium.....	nil	nil	nil
Nitrogen.....	.033	.032	.054

¹ Analyses made by Chemical Laboratory, Philadelphia Navy Yard, Philadelphia, Pa.

TABLE 2.—*Proximate analysis*¹ of the coating on the welding rod

Loss at 105° C.....	2.6%
Calcium fluoride.....	43.5%
Calcium oxide in excess of calcium present as calcium fluoride.....	8.2%
Magnesium oxide.....	3.6%
Aluminum oxide.....	8.8%
Ferric oxide.....	1.2%
Manganese oxide.....	0.5%
Sodium oxide.....	Present
Carbon dioxide.....	16.5%
Silica.....	12.1%
Manganese dioxide.....	2.6%
Total.....	99.6%

¹ Made by Chemical Laboratory, Philadelphia Navy Yard, Philadelphia, Pa.

Specimens consisting entirely of weld metal were prepared by building up alternate layers of longitudinal and transverse beads on a portion of a plate, as shown in figure 1. On one plate the welding was done continuously, with pauses long enough between beads to permit cleaning off of the slag; on another plate the material was quenched

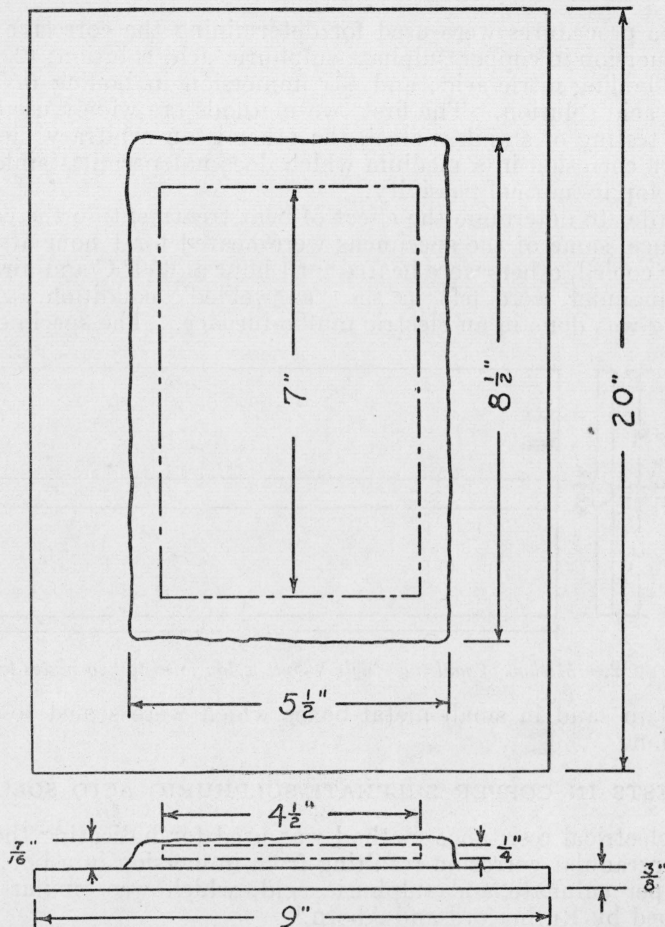


FIGURE 1.—Method of building up solid layers of weld metal

Specimens were taken from the areas within the dotted lines.

in water after each bead was laid and before the slag was cleaned off. The plates were not preheated prior to welding.

Single V-butt welds were made between two sections of plate, as shown in figure 2. The welds were laid in four longitudinal beads. The initial bead at the bottom of the V was retained by means of a backing strap of the same material as the plate. Two pairs of plates were welded in this manner, one of which was quenched after the deposition of each bead, and the other was welded continuously. Specimens containing the welded joint were machined from each pair of plates.

Throughout the work, a coated welding rod $\frac{3}{16}$ in. (5 mm) in diameter was used as the positive electrode and the plate as the negative, with a direct-current arc operated at 23 volts and 170 amperes.

III. CORROSION TESTS

Three procedures were used for determining the corrosive attack: (1) immersion in copper sulphate-sulphuric acid solution; (2) immersion in boiling nitric acid; and (3) immersion in boiling 6*N* hydrochloric acid solution. The first two methods are widely used in corrosion testing of stainless steel, the other is an arbitrary method to measure corrosion in a medium which does not permit stainless steel to develop its normal passivity.

In order to determine the effect of heat treatment on the corrosion resistance, some of the specimens were heated for 1 hour at 980° C. and air cooled, others were heated for 1 hour at 650° C and air cooled; the remainder were left in the "as welded" condition. All heat treating was done in an electric muffle furnace. The specimens were

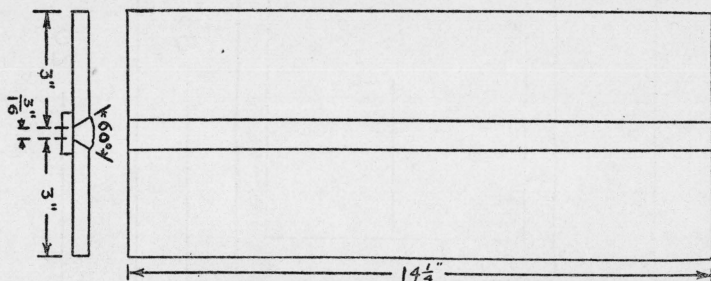


FIGURE 2.—Method of making single V-butt welds joining two plates together.

packed in sand in small metal boxes which were sealed to prevent oxidation.

1. TESTS IN COPPER SULPHATE-SULPHURIC ACID SOLUTION

An electrical resistance method was used for indicating the extent of intergranular corrosion resulting from immersion in a hot solution of copper sulphate and sulphuric acid, which was similar to that described by Rutherford and Aborn.¹

Cylindrical specimens $\frac{1}{8}$ in. (3 mm) in diameter and 4½ in. (11.4 cm) long were used. On specimens of the plate or weld metal, gage marks 1½ in. (3.8 cm) apart were made with a prick punch. On the welded-joint specimens, four gage marks, *A*, *B*, *C*, and *D*, were laid off at ½ in. (1.3 cm) intervals (fig. 3). The two inside marks, *B*, and *C*, were located in the plate adjacent to the weld, that is, between the weld and the zone of pronounced carbide formation. The resistance measured between points *B* and *C* was the combined resistance of the weld metal and bond zones. Measurements between *A* and *B* and between *C* and *D* gave information on the resistance of the two zones that were rich in carbides. Measurements between *A* and *D* were also made.

¹ Trans. Am. Inst. Min. Met. Eng. 100, 293 (1932).

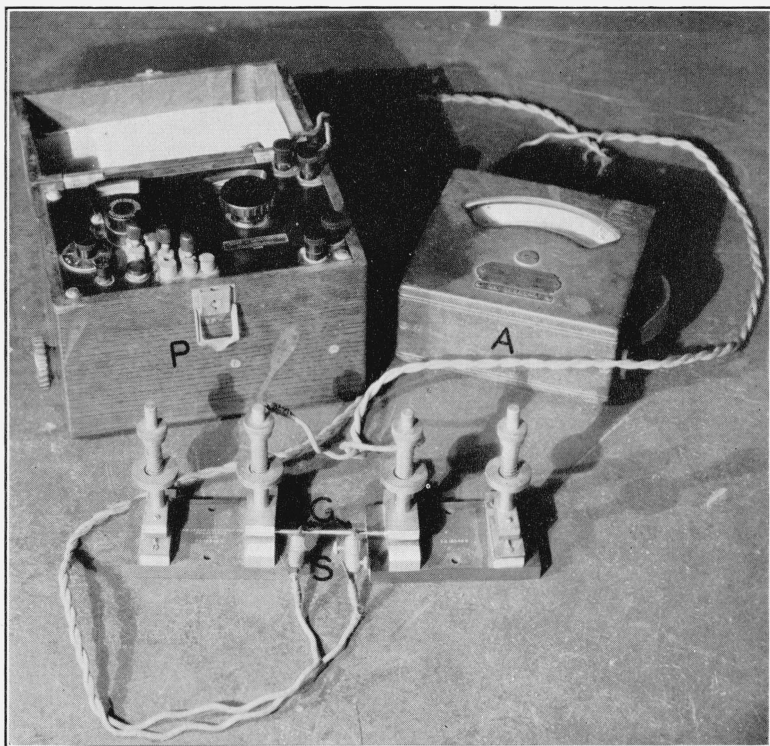


FIGURE 4.—Apparatus for measuring resistance of specimens used in copper sulphate-sulphuric acid corrosion tests.

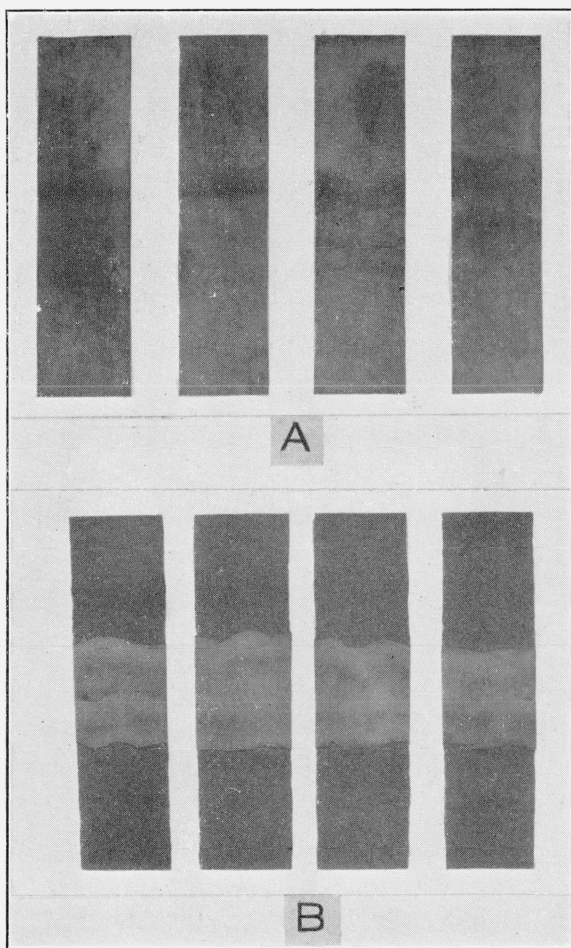


FIGURE 8.—Specimens containing a single V-butt joint after corrosion in boiling nitric acid.

- A. No heat treatment after weld was made.
- B. Heat treated at 650° C for 1 hour.

In using the apparatus, shown in figure 4, for measuring the resistance of the specimens in their initial condition and after corrosion, the copper contact points, *C*, were centered in the gage marks of the specimen *S*. The direct current, measured with the ammeter *A*, was maintained at 5 amperes. The voltage drop across the points was measured with the potentiometer *P*. The resistance was calculated from the values obtained.

The corrosion apparatus consisted essentially of a 1-liter erlenmeyer flask fitted with a reflux condenser. Four test specimens, and one sensitized control specimen that contained 0.10 percent of carbon, were immersed, after initial resistance measurements, in 500 ml of the corroding solution, consisting of 13 g/liter of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ and 47 ml/liter of H_2SO_4 (sp gr 1.84). The solution was maintained at the boiling point for 24 hours. The electrical resistance of each specimen was again determined. The specimens were then treated in a fresh portion of solution for an additional 76 hours, after which the resistance was again determined.

The changes in electrical resistance as a result of exposure to the copper sulphate-sulphuric acid solution were small, that is from -4.6 to $+7.7$ percent, and showed no definite trend, regardless of heat treatment or time of exposure. (The sensitized control specimens, which had a higher carbon content, had an increase in resistance of slightly more than 100 per cent.) All test specimens that had been heat treated for 1 hour at 650°C were reheated at the same temperature for 23 additional hours. These were exposed to the boiling copper sulphate-sulphuric acid solution for 100 hours. The resistances of these specimens were of the same order as of those heat treated for only 1 hour, and indicated that no intergranular corrosion had taken place.

These results agree with those of Rutherford and Aborn for steels containing less than 0.06 percent of carbon, but are in marked contrast with their results for steels containing 0.07 to 0.20 percent of carbon, in which several hundred percent increase in electrical resistance accompanied intergranular corrosion. However, the results of corrosion of the control specimens were quite similar to those of Rutherford and Aborn for similar high-carbon material.

Because the changes in resistance were so small, it seemed probable that no appreciable intergranular corrosion had occurred. Cross sections of ends of the tested specimens were polished and examined under the microscope. Numerous carbides were found at the grain boundaries but no traces of corrosion were detected, even in the specimens of plate metal that had been heat treated for prolonged periods at 650°C .

From these experiments it was concluded that exposure to the copper sulphate-sulphuric acid reagent does not cause intergranular corrosion in this low-carbon material, even after prolonged heat treatment to intensify the formation of carbides. However, the re-

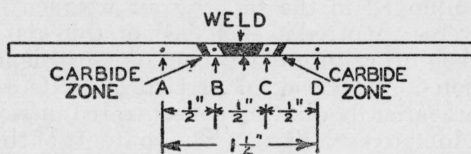


FIGURE 3.—Specimens for copper sulphate-sulphuric acid corrosion, showing location of reference marks.

agent does cause intergranular attack in stainless steel of higher carbon content, as noted in the results of corrosion of the sensitized control specimens containing 0.10 percent of carbon.

Bain, Aborn, and Rutherford² concluded that the copper sulphate-sulphuric acid reagent attacked only those portions of the grain, adjacent to the grain boundaries, that had been depleted in chromium as a result of the formation of carbides at the grain boundaries; and that the reagent did not attack the carbides themselves. The results of the present investigation are in agreement with this conclusion.

2. TESTS IN BOILING NITRIC ACID

The test in boiling nitric acid, described by Huey,³ has been incorporated in many specifications to establish the resistance of stainless steels to corrosion under oxidizing conditions. The procedure was studied by subcommittee IV of committee A-10 of the American Society for Testing Materials with the conclusion⁴ that the test is "capable of furnishing accurate, reproducible, quantitative data."

The apparatus used for corroding the specimens was the same as that used for the copper sulphate-sulphuric acid tests. Rectangular specimens, 1 $\frac{1}{8}$ in. by $\frac{1}{2}$ in. by $\frac{1}{4}$ in. (4.8 cm by 1.3 cm by 6 mm), were employed in the test. Four weighed specimens of a given material were supported in each flask on thin glass rods and were separated from each other to prevent contact corrosion. 500 ml of an aqueous solution of nitric acid (67 percent of HNO₃ by weight, with a sp gr of 1.408 prepared by diluting concentrated nitric acid with distilled water) was added to each flask. The contents of the flasks were maintained at the boiling point for 48 hours. The specimens were then removed, cleaned, dried, and weighed. The process was repeated until each set of specimens had been subjected to five 48-hour tests, 500 ml of fresh acid being used each time. The corrosion rate for each period was computed in inches penetration per month, from the loss of weight, surface area, density of material, and corrosion period (48 hours).

The results obtained from the immersion in boiling nitric acid are given in figures 5 to 7, inclusive. Each plotted point is the average of at least two observations, which were generally in excellent agreement.

Figure 5 shows the results obtained with the plate material. Specimens that were not heat treated showed a very low progressive rate of attack, which was almost identical with the attack of the material that had been heated for 1 hour at 980° C. Specimens heated at 650° C for 1 hour showed a very much greater rate of attack than other specimens, a sharp increase in rate being noted after the second 48-hour corrosion period. At the higher rates of attack, the lack of close agreement in the results obtained in duplicate runs is indicated by the shaded area of the curve.

The results obtained with weld metal specimens are given in figure 6. The effect of heat treatment in promoting the corrosive attack was insignificant. The rates of attack for all the specimens were very low and were similar to the rates for plate metal in the "as rolled" condition or after heat treatment at 980° C. Specimens which had been

² Trans. Am. Soc. Steel Treating 21, 481 (1933).

³ Trans. Am. Soc. Steel Treating 18, 1126 (1930).

⁴ Proc. Am. Soc. Testing. Materials 33, I, 178 (1933).

heated at 650° C corroded at substantially the same rates as those heated at 980° C.

In figure 7 are summarized the results obtained with specimens of the plate containing a welded joint. The corrosion rates were quite similar to those of the plate, probably because the greater portion of each of these specimens was plate metal.

The extent of attack by the boiling nitric acid could be roughly estimated from the appearance of the corroded specimens. The entire

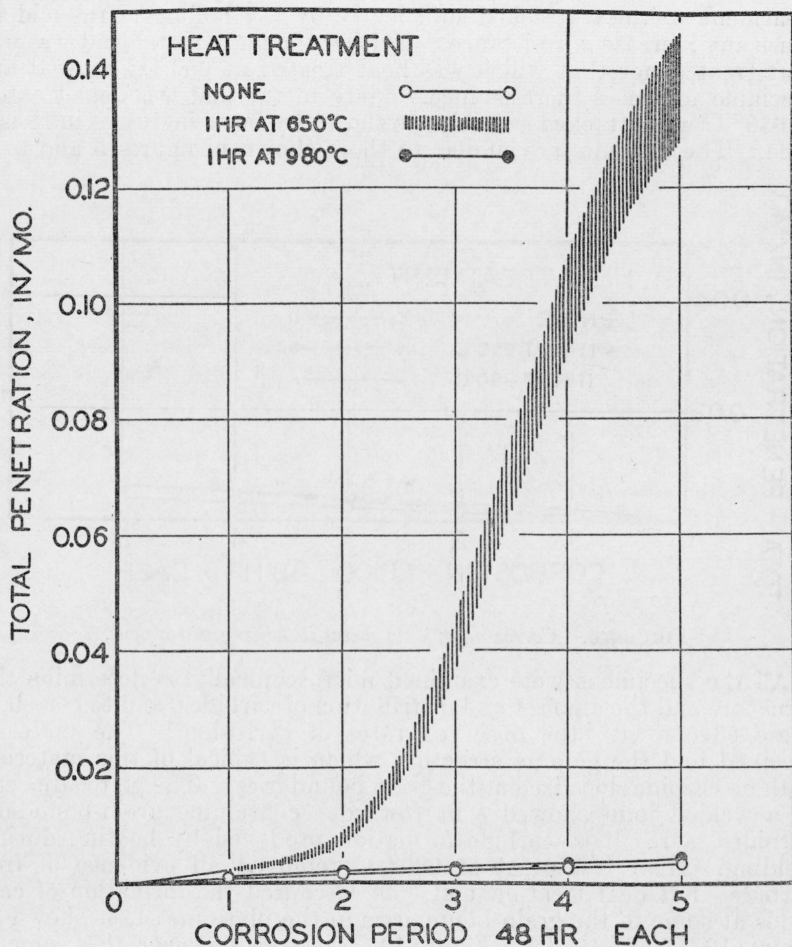


FIGURE 5.—Corrosion of plate metal in boiling nitric acid.

surface of each specimen was uniformly affected. The surfaces of specimens which were attacked slowly (see fig. 5 and 6) were only slightly etched. The surfaces of the specimens that had been heat treated at 650° C, were roughened to a much greater extent. These effects were particularly noticeable in the specimens containing both weld and base metals. As shown in figure 8, the corrosion of "as welded" specimens was relatively slight and quite uniform over the entire surface. The appearance of the specimens that had been heat

treated at 980° C was quite similar to that of the "as welded" specimens. On the other hand, in the specimens which had been heat treated at 650° C, the plate was severely roughened, the weld metal being left in bold relief.

The specimens of plate and weld metals that had been tested in the copper sulphate-sulphuric acid solution and that showed no attack by this reagent were placed in boiling nitric acid and their resistances were measured after each of three 48-hour periods. The results of these tests are shown in figure 9. Weld metal in any condition of heat treatment was not attacked sufficiently by the boiling nitric acid to cause any increase in resistance. Neither the plate metal that was not heat treated nor that which was heat treated at 980° C showed appreciable increases in resistance. Plate metal that was heat treated at 650° C was attacked severely, as shown by large increases in resistance. These results are similar to those shown in figures 5 and 6.

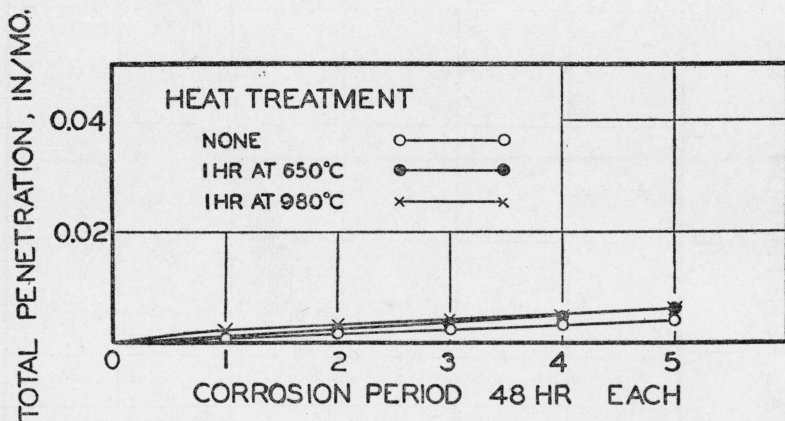


FIGURE 6.—Corrosion of weld metal in boiling nitric acid.

All the specimens were examined microscopically to determine the structure and the amount and distribution of carbides, and to correlate these effects with the observed rates of corrosion.⁵ The plate as received had the gamma structure which is typical of this material, with no carbides localized at the grain boundaries. The plate adjacent to a welded joint showed a narrow zone containing grain-boundary carbides, a result of carbide formation produced by heating during welding. Heat treatment at 980° C removed all evidence of free carbides, but heat treatment at 650° C caused the formation of carbides at some of the grain boundaries in the plate metal, as shown in figures 10 (C) and 10 (D). Figure 10 (D) shows, however, that some of the grain boundaries were free from carbides.

Examination of specimens of the weld metal, built up either by intermittent or by continuous deposition, revealed interesting differences in structure (fig. 10(A) and (B)). After heat treatment at 650° C, both metals consisted of a matrix of gamma iron with numerous small areas of delta ferrite, each of the latter being outlined by carbides. In the intermittently deposited metal (fig. 10(A)) the

⁵ The etchant used for revealing carbides and grain boundaries was 10-percent oxalic acid, the use of which is described in *Trans. Am. Soc. Metals* 24, 26 (1936); also *Etching Stainless Steels*, NBS Technical News Bul. 218, p. 62 (June 1935).

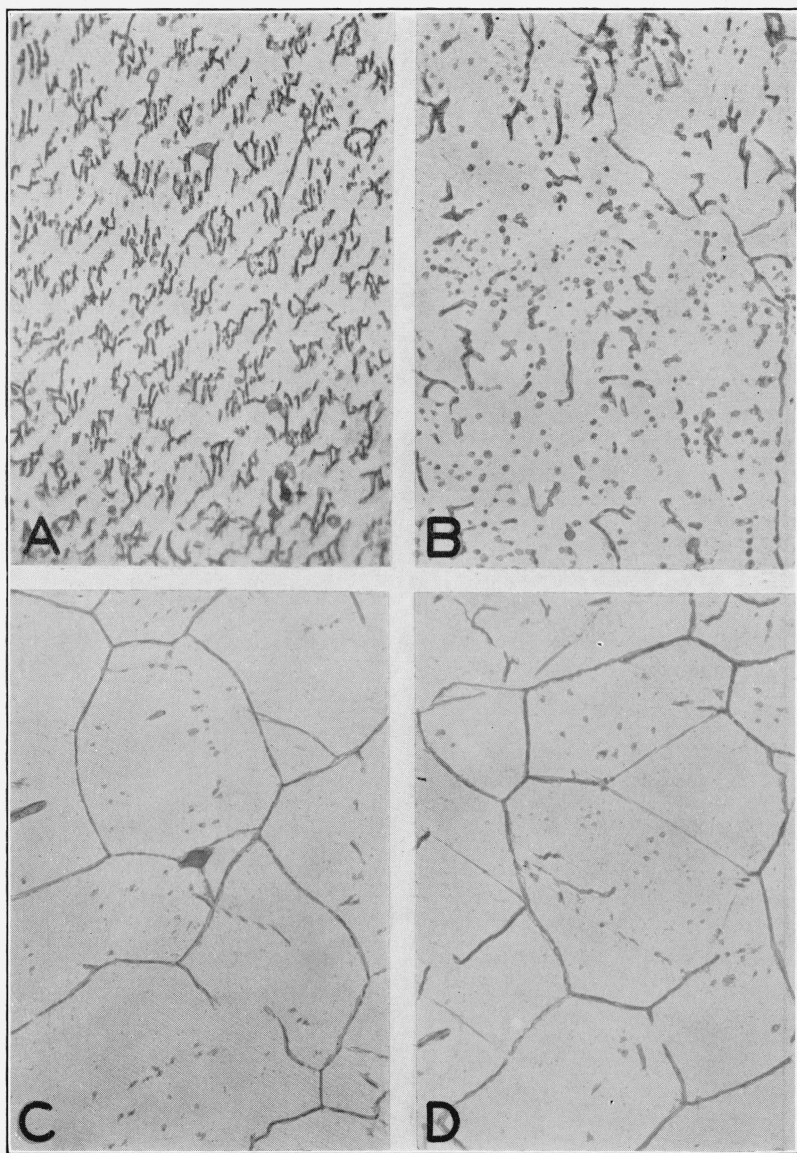


FIGURE 10.—Structures of specimens treated at 650° C for 1 hour.

Etched electrolytically in 10-percent oxalic acid. Magnification, 500 \times .

A. Weld metal, quenched between beads.

B. Weld metal, deposited continuously.

C. Plate metal, etched for carbides only.

D. Plate metal, etched for carbides and grain boundaries.

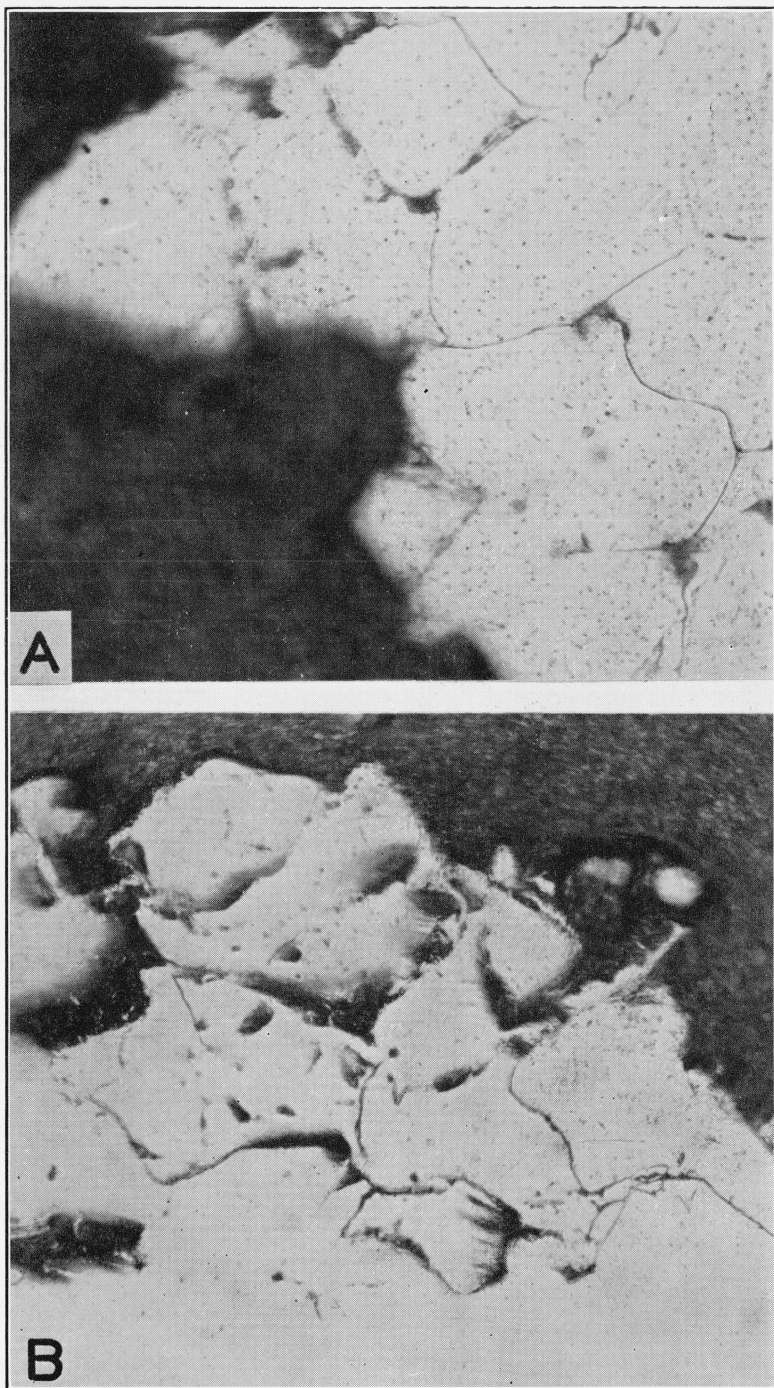


FIGURE 11.—Edges of a specimen of plate metal, heat-treated at 650°C and corroded in boiling nitric acid.

Specimen not etched after corrosion. Magnification, $500\times$.

areas of delta ferrite were elongated and so narrow that the outlining carbides appear to be short lines or dendrites. In the continuously deposited metal (fig. 10(B)) the areas of delta ferrite were larger and were frequently circular in outline. There was no evidence of carbide formation in the gamma areas and it is noteworthy that the carbides did not concentrate at the grain boundaries of the weld metal as they did in the plate metal.

All the specimens that had been corroded in boiling nitric acid showed evidence of nonselective corrosion, but intergranular corrosion

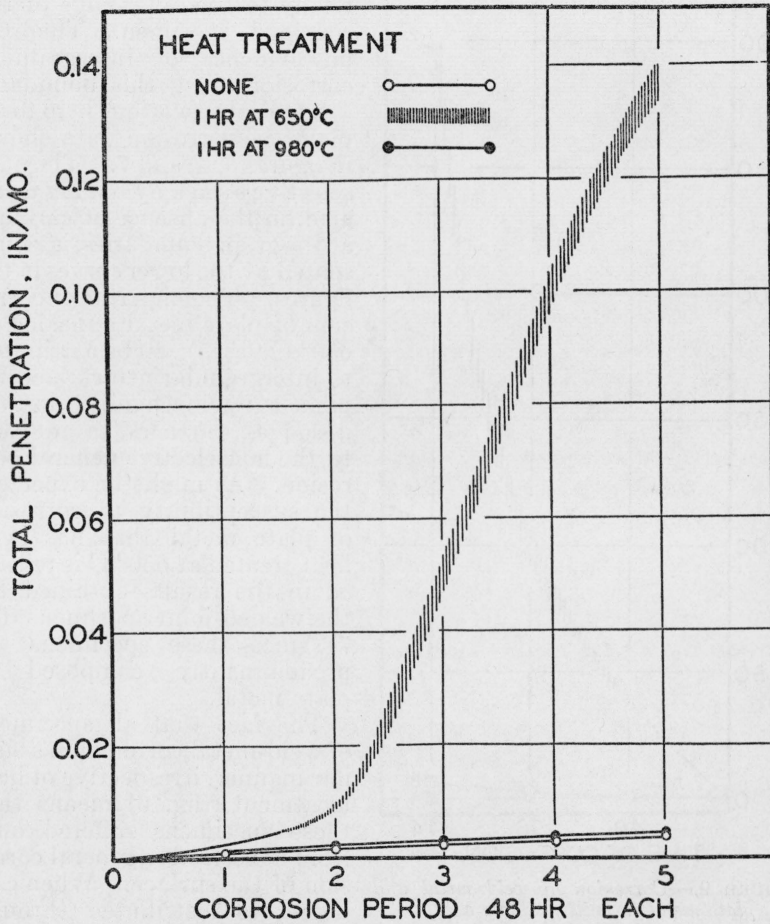


FIGURE 7.—Corrosion of welded specimens in boiling nitric acid.

was found only in those specimens that had been heat treated at 650° C. Figure 11 shows that in some cases intergranular corrosion progressed to such an extent that grains were completely separated or even dislodged. Further evidence of loosening of the grains through intergranular corrosion was obtained in the fourth and fifth corrosion periods of plate specimens annealed at 650° C, when a gray powder collected on the bottom of the corrosion flask. This powder, found only with plate specimens annealed at 650° C, con-

sisted of grains of metal that were evidently detached from the specimens; carbide particles were not detected.

Figure 12 shows a corroded specimen, similar to that of figure 11, after etching with oxalic acid to reveal free carbides and grain boundaries. It is evident that intergranular corrosion occurred only along those grain boundaries where carbide had been formed. In

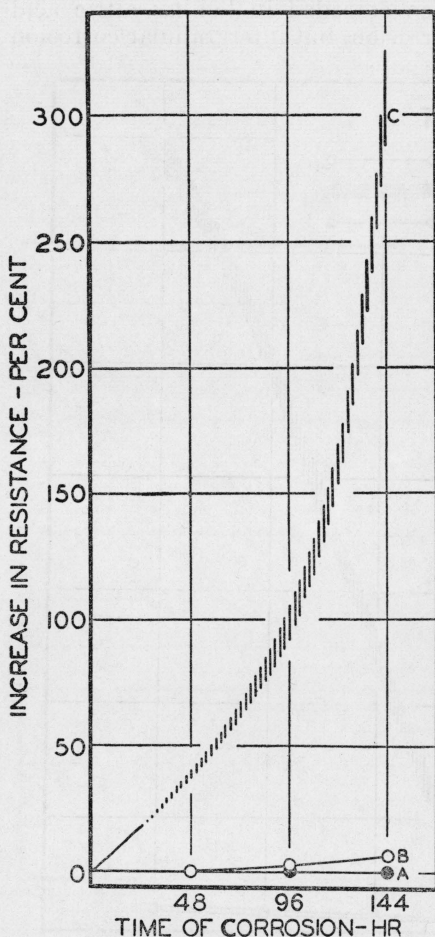


FIGURE 9.—Corrosion of weld metal and plate metal in boiling nitric acid.

- A. Weld metal, not heat treated, heat treated at 650° C, and at 980° C.
 B. Plate metal, not heat treated, and heat treated at 980° C.
 C. Plate metal, heat treated for 24 hours at 650° C.

The attack of boiling nitric acid on low-carbon stainless steels is therefore twofold in nature, and involves a nonselective general corrosion of all exposed surfaces and a selective intergranular attack along grain boundaries where carbides are localized. The intergranular corrosion results in loss of loosened grains and, for this

the upper right corner of figure 12(B) a carbide-free boundary can be identified, extending vertically to the outer edge of the corroded specimen. There is no evidence of intergranular corrosion along this boundary.

These observations help to explain the corrosion data shown in figures 5, 6, and 7. The nonselective attack by boiling nitric acid, in the absence of carbides at the grain boundaries, is represented by the lower curves in the figures. The high rate of corrosion of plate metal annealed at 650° C (fig. 5) can be attributed to intergranular attack, accompanied by mechanical loss of dislodged particles, in addition to the nonselective general corrosion. As might be expected, the susceptibility to corrosion of plate metal that has been heat treated at 650° C is reflected in the results obtained for the welded-joint specimens (fig. 7) since these specimens are predominantly composed of plate metal.

The fact that all specimens of weld-metal corroded in a similar manner, irrespective of heat treatment, (fig. 6) means that these specimens suffered only from nonselective general corrosion of the surface. When carbides are distributed through the metal in the manner shown in figures 10(A) and 10(B), they are not conducive to intergranular attack by nitric acid.

type of steel, is much more destructive than is corrosion of the general type. Since it is known that boiling nitric acid decomposes chromium carbides, it may be assumed that the intergranular attack in these specimens proceeds by the destruction of the carbides in the grain boundaries.

3. TESTS IN HYDROCHLORIC ACID

The opinion is generally held that stainless steels owe their corrosion resistance to the tendency of chromium to form a protective film under oxidizing conditions. Since nitric acid is an active oxidizing agent and probably promotes film formation, the nitric acid test sheds no light on the corrosion of these alloys in the absence of a protective film. On the other hand, hydrochloric acid destroys this protective film. Consequently, some tests with hydrochloric acid

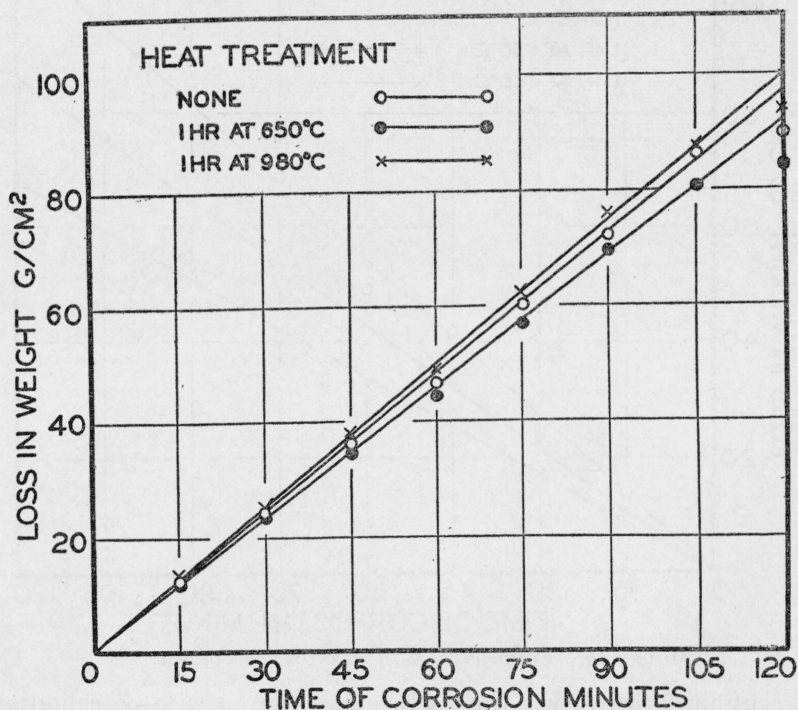


FIGURE 13.—Corrosion of plate metal in hydrochloric acid.

were made to observe the susceptibility of the specimens to corrosion in the absence of a protective film.

300 ml of a solution consisting of equal volumes of distilled water and concentrated hydrochloric acid (1.18 sp gr), was used in a covered beaker of 600 ml capacity. Two specimens similar to those used for the nitric acid tests were immersed for 15 minutes in the boiling solution. The specimens were cleaned, dried, and weighed before and after each 15-minute immersion. Eight 15-minute periods constituted a test run, the solution being replaced with a fresh one at the end of the fourth period. Results of these tests, calculated as loss

of weight in g/cm^2 , are shown in figures 13 to 15, inclusive. Each plate specimen lost weight at approximately a constant rate throughout the duration of a test. Furthermore, as shown in figure 13, the rate of corrosion of a plate metal specimen was not appreciably affected by heat treatment at either 650 or 980°C . Specimens of weld metal that were not heat treated lost weight continuously and at about the same rate as did the plate metal. However, as shown in figure 14, heat treatment of the weld metal had a measurable effect on the rate of corrosion. Heat treatment at 650°C decreased the susceptibility of weld metal to attack, and heat treatment at 980°C decreased it further. In the latter condition the loss of weight of the weld metal was definitely less than that of the plate. The greater

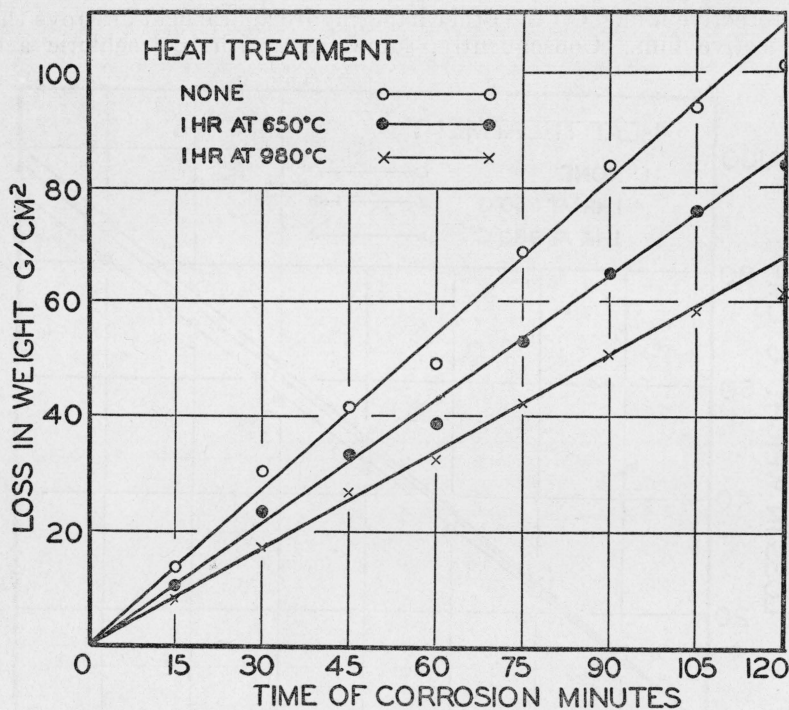


FIGURE 14.—Corrosion of weld metal in hydrochloric acid.

susceptibility to attack of the weld metal in the "as welded" condition, presumably accounts for the greater loss in weight of some of the welded joints (fig. 15).

There is no satisfactory explanation for these differences between weld metal and plate, or for the effect of heat treatment of the weld metal.

There was no evidence of selective attack at the grain boundaries. On the other hand, selective attack of the pitting type occurred on the surface of all specimens, particularly of the weld metal. In welded specimens without heat treatment the weld metal corroded at a faster rate than the plate, as is shown in figure 16 (A). However, after heat treatment of the welded joints at either 650 or 980°C , the weld metal was slightly more resistant than the plate metal.

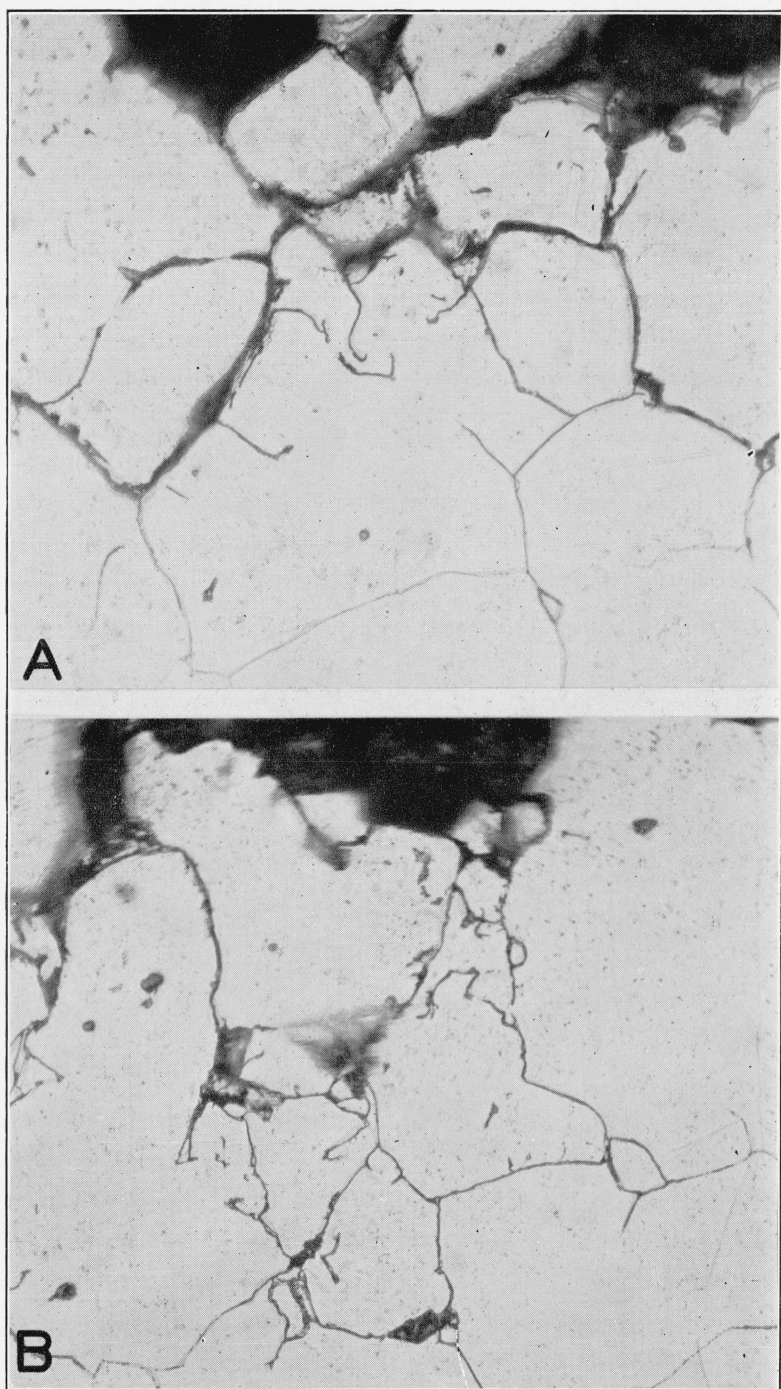


FIGURE 12.—Edges of a specimen of plate metal, heat treated at 650° C and corroded in boiling nitric acid.

Specimen subsequently etched electrolytically in 10-percent oxalic acid. Magnification, 500X.

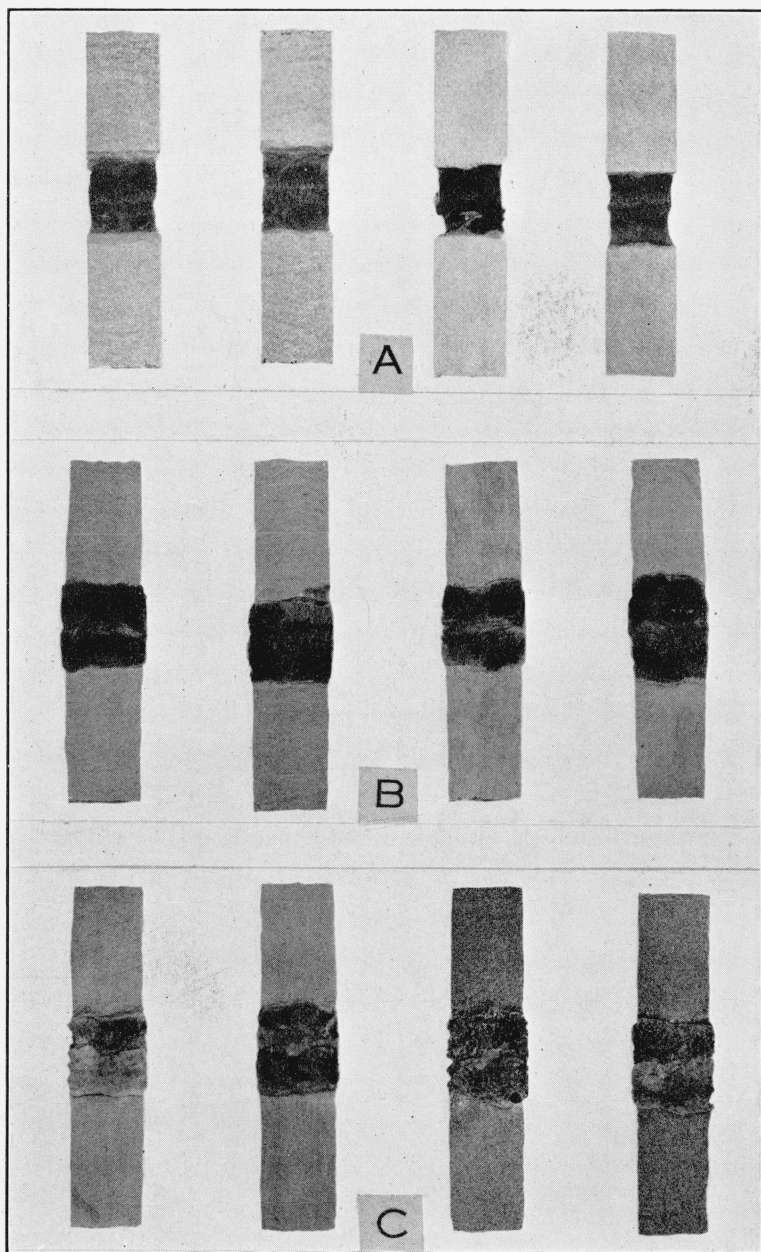


FIGURE 16.—Specimens containing a single V-butt weld after corrosion in hydrochloric acid ($\times 1$)

- A. No heat treatment after weld was made.
B. Heat-treated at 650° C for 1 hour.
C. Heat-treated at 980° C for 1 hour.

These observations cannot be explained by the presence of carbide or its distribution, or by chromium depletion resulting from carbide formation. It is possible that these corrosion phenomena are connected with the presence of delta ferrite in the weld metal, but the connection is not obvious.

IV. SUMMARY

Concentrated nitric acid at its boiling point has previously been used to produce intergranular corrosion in stainless steel. Evidence was obtained that this type of corrosion is the result of the selective attack by the acid on carbides that exist as networks at the grain boundaries. The cohesion between the constituent grains is destroyed

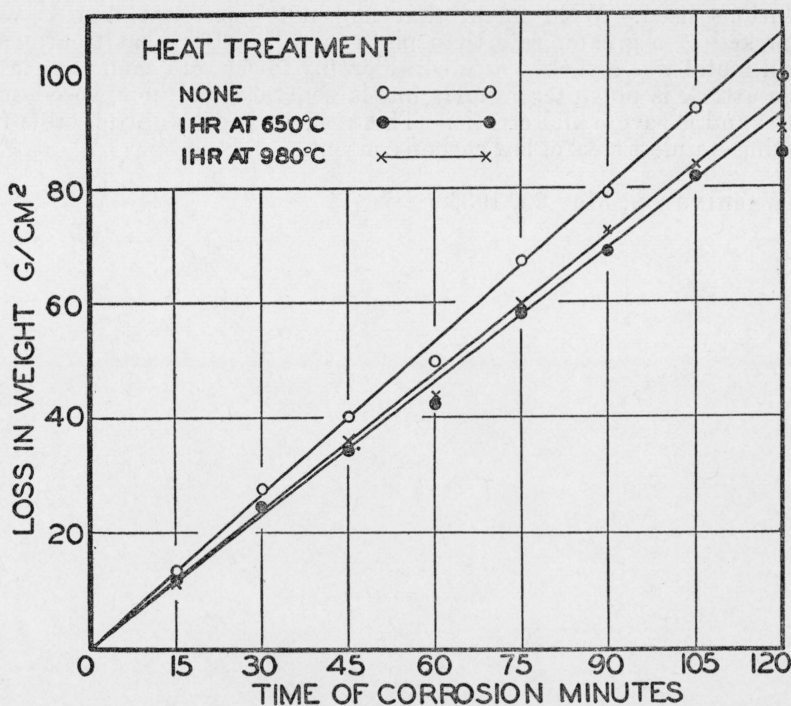


FIGURE 15.—Corrosion of welded specimens in hydrochloric acid.

and the material seriously weakened. Specimens of low-carbon stainless steel (0.06 percent of carbon) develop this type of intergranular corrosion only after the specimens have been heated so as to permit the formation of a carbide network at the grain boundaries. Without such heat treatment, the amount of the grain boundary carbides was not sufficient to render the steel subject to intergranular corrosion by boiling nitric acid.

A solution of copper sulphate in sulphuric acid has also been used to produce intergranular corrosion in stainless steels. It has been established by other investigators that the copper sulphate-sulphuric acid reagent does not destroy the carbides at the grain boundaries but, instead, attacks the adjacent metal that has been impoverished in

chromium through the formation of carbides. Low-carbon stainless steel (about 0.06 percent of carbon) is not subject to intergranular corrosion by this solution, presumably because the formation of carbides does not cause appreciable depletion of the chromium content of the metal adjacent to the boundaries.

Weld metal of approximately the same composition as the plate was not susceptible to intergranular corrosion in nitric acid or in a copper sulphate-sulphuric acid solution, regardless of heat treatment. Carbides were not found in the grain boundaries of specimens that had been heated to facilitate their formation, but were localized in delta ferrite pools scattered throughout the matrix.

Low-carbon stainless steel, exposed to the action of 6*N* hydrochloric acid, lost weight much more rapidly than in either of the other solutions used. Weld metal that had not been heat treated was attacked at a greater rate than plate metal. After heat treatment, weld metal was attacked at a considerably lower rate than the plate. The attack is not intergranular but is general over the exposed surfaces and is severe and erratic. This reagent is of doubtful value for testing stainless steel of low carbon content.

WASHINGTON, May 29, 1936.