ENGINEERING RESEARCH INSTITUTE
UNIVERSITY OF MICHIGAN
ANN ARBOR

QUARTERLY PROGRESS REPORT NO. 2

AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEEL

By

C. A. SIEBERT

M. J. SINNOTT

R. E. KEITH

Project 2110

WRIGHT AIR DEVELOPMENT CENTER, U.S. AIR FORCE
CONTRACT AF 33(616)-353

June, 1953

QUARTERLY PROGRESS REPORT NO. 2

AN INVESTIGATION OF INTERGRANULAR OXIDATION IN STAINLESS STEEL

INTRODUCTION

This research project has been undertaken under the sponsorship of the Wright Air Development Center of the U.S. Air Force. Its objectives are fourfold:

(a) to determine the effect of temperatures between 1600 and 2000°F on intergranular oxidation or corrosion;

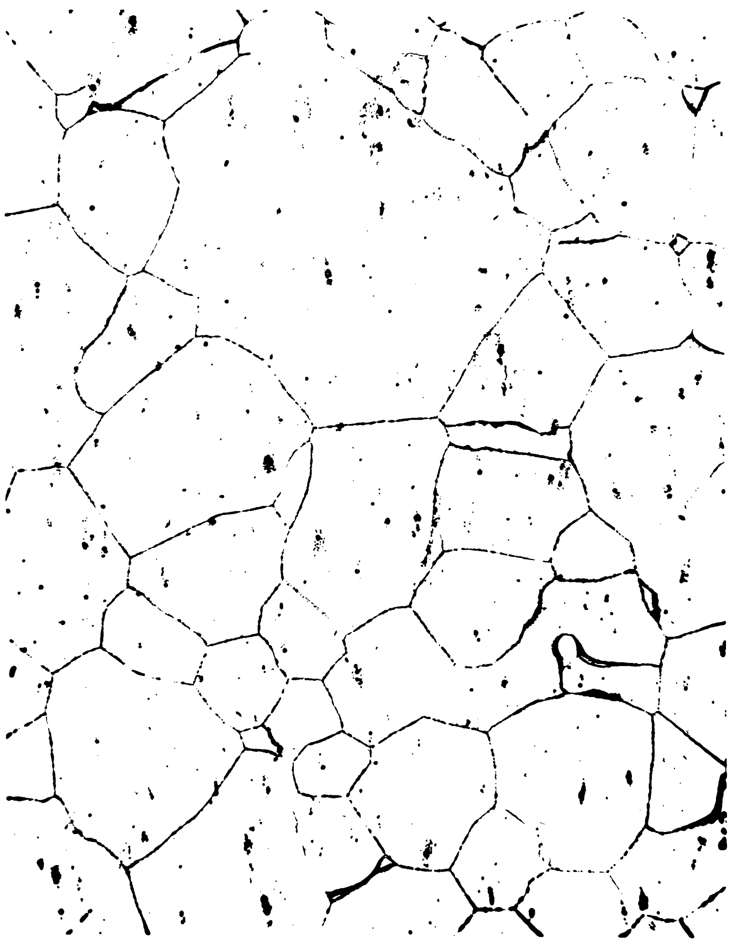
(b) to examine the effects of alloy composition on intergranular oxidation or corrosion;

(c) to determine the nature of the penetrating material in areas of intergranular attack; and

(d) to devise methods of reducing or eliminating intergranular penetration.

MATERIAL

The basic material to be used in this investigation is type 310 stainless steel. Work to date has been carried out on this material using surface-ground specimens measuring 1-1/2 by 1/2 by 1/4 inches. A chemical analysis of this stock is presented in Table I, and a photomicrograph of the material as received is shown in Fig. 1. Future work will be carried out on stock which is of the order of 0.05 inch thick, and attempts are presently being made to obtain samples of this thickness from several heats of type 310 having several compositional variations within the allowable



X 100

Fig. 1. Type 310 Stainless Steel As Received. Hot-rolled and annealed condition. Photomicrograph in plane of rolling. Electrolytic chromic acid etch.

specifications for this alloy. These samples would be used to determine minor compositional effects on intergranular penetration.

TABLE I

COMPOSITION OF TYPE 310 STAINLESS STEEL, WEIGHT PER CENT

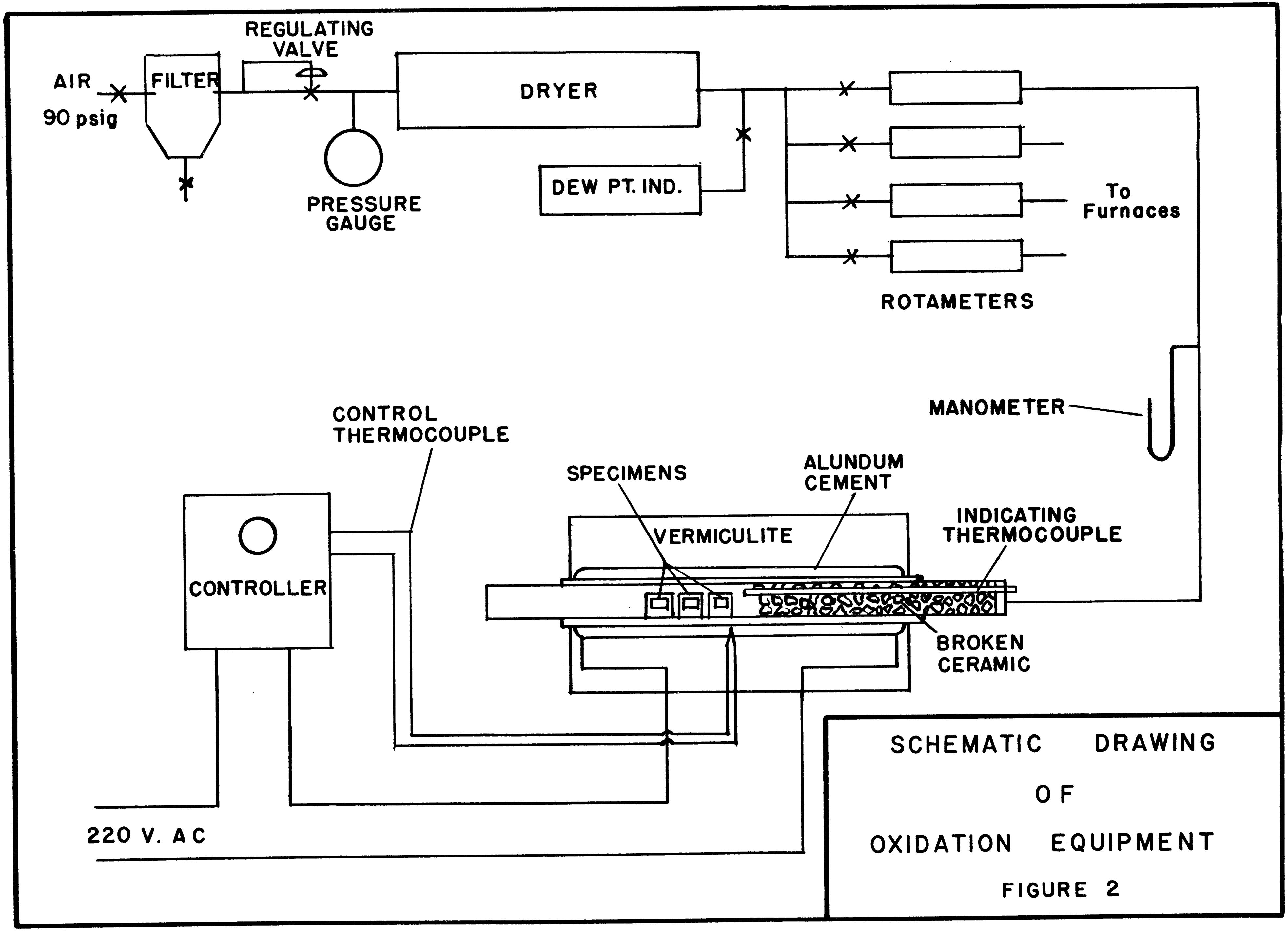
Constituent	Nominal	Analysis
C	0.25 max	0.14
Ni	19.0 - 22.0	19.63
Cr	24.0 - 26.0	24.63
Si	-	0.97
Mn	2.00 max	0.51
P	-	0.015
S	-	0.019

In addition, samples of other types of stainless materials are available, notably types 304, 309 + Nb, 316, and Inconel. Some tests may be run later in the project period using any or all of these materials if it appears desirable to investigate them.

The oxidizing medium to be used in the experiments is atmospheric air. It is anticipated that the effects of moisture and carbon dioxide contents of the air will be determined.

EQUIPMENT AND PROCEDURE

The larger part of the past quarter has been spent in completing the construction of the equipment to be used in the investigation. A schematic drawing of the equipment appears in Fig. 2. Air at approximately 90 psig is first passed through a porous Al_2O_3 filter to remove any entrained oil, sediment, or other foreign matter. The air then passes through a pressure-reducing valve and then through a drying tower filled with activated alumina. A valve is provided to bleed off a sample of the dried air for a dew-point determination at intervals. The air is delivered to a manifold from which four side streams are withdrawn, one to each of the four horizontal tube furnaces. Flow to each furnace is controlled by a needle valve, and is metered by a flowmeter of the rotameter type in conjunction with a manometer. The air is then delivered to one end of a 2-inch sillimanite tube in the furnace. This tube is concentric with a similar, but shorter, 3-inch tube. About the latter tube the chromel heating wire is wound and is imbedded in alundum cement. There is a 5-inch layer



SCHEMATIC DRAWING OF OXIDATION EQUIPMENT

FIGURE 2

of vermiculite insulation contained in an aluminum shell around the heating coils. The furnace draws approximately 12 amperes at 220 volts. The 2-inch tube is packed for approximately one-third its length with broken pieces of ceramic, the purpose of which is to provide a large surface area to preheat the incoming air. A thermocouple well extends into the furnace, as close as possible to the location of the specimens, which are suspended in the center of the 2-inch tube on wire frames. The uniform-temperature zone of the furnace is approximately 8 inches in length. Temperature is controlled by means of a Foxboro controller with the control thermocouple imbedded in the furnace windings.

Specimens are first cleaned in alcohol and are then charged to the furnaces at temperature and with the air flowing. Readings of all instruments and meters are taken twice daily during the runs, which up to the present have been of 100 hours' duration. At the termination of the run, the specimens are withdrawn from the furnaces and quenched into stainless-steel beakers containing distilled water. Quenching is necessary to preserve the high-temperature oxide structures. The contents of the beakers are filtered and the collected oxide scale is washed with acetone and then with ether in order to dry it rapidly. Any scale adhering loosely to the specimen is scraped off and bottled with the filtered scale, care being taken not to damage the specimen surface. The oxide scales thus collected are then pulverized and analyzed by x-ray diffraction methods.

The specimens are each cut into three equal segments and are mounted in cross section in bakelite. They are then polished, using wet silicon carbide papers followed by a polish using a diamond wheel. This results in a surface which, while not scratch-free, is smooth and has well-polished edges with a minimum of rounding. Measurements of penetration are then made on the unetched specimens under a microscope.

RESULTS AND DISCUSSION

A. Literature Survey

The literature survey of the field of corrosion in the stainless steels mentioned in Progress Report No. 1 has been completed, and selected references are being examined in detail. It is already apparent that very little attention has been paid to intergranular penetration in the past. It has been observed and noted, but not explained in anything we have found. Perhaps the most advanced theoretical work on intergranular penetration has been done by the British on several nonferrous systems. It is anticipated that their general conclusions will be valuable in the present study.

B. Oxidation Tests

Tests of 100 hours' duration have been carried out at temperatures of 1600, 1700, 1800, and 1900°F at an air flow rate of 20 feet per minute and penetration measurements made on these specimens. These measurements appear in Table II.

TABLE II

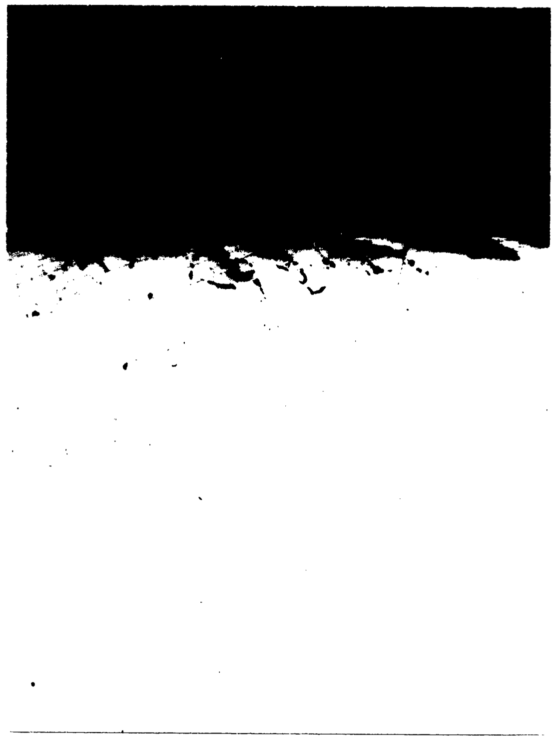
PENETRATION MEASUREMENTS FOR TYPE 310 STAINLESS STEEL
100 Hours at Temperature

Oxidizing Temperature, °F	Intergranular Penetration, in. Depth	Standard Deviation	Number of Measurements
1600	0.0003	0.00007	10
1700	0.0004	0.0001	8
1800	0.004	0.002	8
1900	0.003	0.001	12

In general, there is an increase in the depth of penetration with increasing oxidation temperature. There is also a numerical increase in the standard deviation of the depth of penetration. On a percentage basis, however, the standard deviations do not vary a great deal, all of them falling between 25 and 50 per cent of the depth of penetration.

It will be noticed that the specimen oxidized at 1800°F showed unexpectedly deep penetration. In the case of this specimen, the penetrating material had a markedly different metallographic appearance from the penetration in the other specimens. Referring to Fig. 3(c), it may be noted that the penetrating material at 1800°F has a smooth-edged appearance, whereas the more usual form of penetration is jagged, such as the example at 1900°F in Fig. 3(d). The 1800°F penetrating material is unique in having a red, optically active appearance under polarized light, while the other observed examples of penetration appear dark grey with or without green inclusions, and are optically inactive. We have no explanation at the present time for this difference in penetrating material.

(a) 1600°F



(b) 1700°F

(c) 1800°F

(d) 1900°F

Fig. 3. Intergranular Penetration in Type 310 Stainless Steel. Unetched. Sensitive tint plate. X 500.