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INFORMAL QUARTERLY REPORT
July 15, 1942

to

NATIONAL DEFENSE
RESEARCH COMMITTEE
of
OFFICE OF SCIENTIFIC
RESEARCH AND DEVELOPMENT

on

N. D. R. C. RESEARCH PROJECT 4,
CONTRACT NO. OEMsr-499

The Effects of Hydrogen, Nitrogen,
and Oxygen in Armor Plate

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BATTELLE
MEMORIAL INSTITUTE

505 King Avenue
COLUMBUS, OHIO

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N.D.R.C. RESEARCH PROJECT 4, CONTRACT NO. OEMsr-499
THE EFFECTS OF HYDROGEN, NITROGEN,
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INTRODUCTION

Fractional vacuum fusion analyses of six cast and four rolled armor plate samples have been completed.

In performing a vacuum fusion analysis (for "total" oxygen, as contrasted to the "fractional" method used here), the sample is melted at about 1600°C. in a graphite crucible contained in a furnace evacuated to a base pressure of less than 0.0001 mm of mercury. The absorbed and dissolved gases (oxygen, hydrogen, and nitrogen) are liberated, and the various oxides, silicates, and aluminates present are rapidly and completely reduced. Because the melt is saturated with carbon from the graphite crucible, all the oxygen is converted to CO. Nitrides break down to yield free nitrogen, and the hydrogen contained in the steel is evolved as free hydrogen.

By means of a high-speed mercury diffusion pump, the gases from the sample are rapidly pumped from the furnace into an evacuated collection system made of glass tubing. Connected to this system are vacuum gages for measuring changes in pressure as gas is collected.

The gases are then removed (by an automatic Toepler pump) from the collection system and collected over mercury in test tubes. A modified Bureau-of-Mines-type Orsat apparatus is used to analyze the gas.

The fractional vacuum-fusion method used for this work is a modification of the "total" method described above. This modification involves heating the empty crucible to about 2300°C. for out-gassing and pumping off the gases until the amount evolved is very low (about 1 cc per hour at 1600°C.). The temperature is then lowered to 1320°C., and tin equal to twice the weight of the steel sample is dropped into the crucible, and any gas in it is pumped out. The collection system is then isolated from the pumps used in this preliminary evacuation.

The temperature is lowered to 1060°C., and the sample is dropped into the tin which fluxes the steel causing it to melt at this temperature. FeO, the least refractory of the oxides present, is rapidly reduced at 1060°C., and when present the rate of evolution of gas is indicated by a rise in pressure in the system, which reaches a maximum in two or three minutes. When most of the FeO is reduced, the pressure gradually falls until it reaches the base pressure, a point where it remains more or less constant. This pressure is slightly higher than the starting pressure because traces of MnO are now being reduced. This is considered the end of the 1060°C. fraction, so the tube of gases collected is removed for analysis, and a new collection tube is installed. The temperature is then raised to 1170°C.,

where MnO is rapidly reduced, but no more than slight traces of SiO_2 , Al_2O_3 , or other more refractory oxides are broken down. The rate of evolution rises and falls as before. Following the 1170°C . fraction, the temperature is raised to 1320°C . for the reduction of SiO_2 and then to 1600°C . for Al_2O_3 .

These oxides are the ones that may be found in plain carbon steels. However, alloy steels may contain several other oxides, and the fraction in which their oxygen is obtained may be judged roughly by examination of thermodynamic data. Experimental data indicate that Cr_2O_3 is reduced principally at 1170°C ., while TiO_2 appears to be reduced partly in the 1320°C . fraction and partly at 1600°C .

The nitrogen in low alloy, aluminum-killed steels is obtained in the 1600°C . fraction. High-chromium steels which usually contain considerable nitrogen yield most of it at 1170°C .

In regard to more complex compounds, Reeve⁽¹⁾ found that in the case of MnSiO_3 he obtained the MnO and SiO_2 at their respective usual temperatures of reduction.

The accuracy claimed for the method is $\pm .001\%$ O_2 and $\pm .001\%$ N_2 for 10-gram samples. Hydrogen values are generally erratic and are not very significant unless the complete history of the sample is known. For instance, an ingot cooled slowly may be low in hydrogen, while one cooled rapidly may have a quite high hydrogen content. If analyzed at this stage, one could predict that the steel high in hydrogen would be more susceptible to flaking than the other. However, after hot rolling or heat treatment, both steels may show the same low hydrogen analysis even though flakes are present in one of them.

(1) A.I.M.E., 113, 1934, pp. 82-100.

RESULTS

Table 1 gives the chemical analysis, mechanical properties, and ballistic properties of the ten plates that were analyzed for gas content. The data contained in Table 1 were supplied either by the producer or by Watertown Arsenal, with the exception of the residual aluminum content, which was determined at Battelle Memorial Institute.

Table 2 gives the fractional and total gas analysis, acid soluble residual aluminum, the inclusion type (according to Sims and Dahle⁽²⁾ for the cast plate and the Chevrolet method⁽³⁾ for the rolled plate), and impact values for single- and double-width standard Charpy specimens with a key-hole notch.

The tables are divided into two sections; the first gives data relative to cast plate, and the second gives data concerning the rolled plate. In both sections the plates are arranged in order of increasing oxygen content.

DISCUSSION

Table 2 shows a relationship between total oxygen content and residual aluminum. The plates low in total oxygen are high in residual aluminum, and those high in oxygen are low in aluminum in both the rolled and cast materials. There is also a relationship between the inclusion type of the cast plate and the residual aluminum content. Type III inclusions are associated with high residual aluminum contents, and Type I most frequently

⁽²⁾Sims, C. E. and Dahle, F. B. "Effect of Aluminum on the Properties of Medium Carbon Cast Steel", Trans. A.F.A., Vol. 46, 1938, pp. 65-103.

⁽³⁾"Rating of Inclusions, the Chevrolet Method", Metal Progress, Vol. 35, Feb., 1939, p. 169.

TABLE 1. CHEMISTRY AND MECHANICAL AND BALLISTIC
PROPERTIES OF ROLLED AND CAST PLATE

Plate	Chemical Analysis												Deoxidizer
	C	Mn	Si	S	P	Ni	Cr	Mo	Cu	V	Zr	Al	
<u>CAST</u>													
G645	.27	1.48	.40	.017	.022			.51				0.04	
W2332	.27	.78	.35	.033	.035	1.00	.51	.40				0.03	2# Al
P10	.21	1.09	.82	.011	.019				1.61			0.02	
S99	.31	1.06	.25	.038	.019	.47	.59	.49				0.02	
FA5	.28	.79	.18	.016	.020			.52	.72			< 0.01	
AHV2	.29	1.52	.41	.035	.034					.09		< 0.01	2# HC-Fel 3# CaSi
<u>ROLLED</u>													
JT3427	.26	1.05	.22	.014	.018			.54				0.06	
JT3427	.26	1.05	.22	.014	.018			.54				0.06	
CI811	.25	1.58	.24	.021	.015	.98	.52	.44				0.06	2# Al per ton
GL119	.30	.70	.81	.030	.021		.64	.15			.11	0.04	
GL119	.30	.70	.81	.030	.021		.64	.15			.11	0.04	
JT2420	.26	2.05	.19	.015	.022			.51				0.03	
JT2420	.26	2.05	.19	.015	.022			.51				0.03	

		Mechanical Properties						
Deoxidizer	Rolled Plate Direction	Tensile Strength	Yield Strength	% Elongation	% Reduction Area	Izod	Brinell	
		125,000	107,000	17.0	38.2	56.3	252	37 PTI
2# Al		131,500	114,000	12.0	22.7	19.0	277	37 PTI
		134,860	127,450	16.0	36.9		290	37 PTI
		118,350	97,250	21.5	57.8	Charpy: 33.5	248-255	37 PTI
		123,500	108,500	19.5	52.5	46.5	241	37 PTI
2# HC-FeTi; 3# CaSi		104,750	85,500	21.5	52.8	44-45	226	37 PTI
	Longitudinal	144,800	143,800	18.0	67.0	82.0	248-269	37 PTI
	Transverse	144,800	122,850	16.5	61.1	45.0		42
	Longitudinal	144,800	143,800	18.0	67.0	82.0	248-269	37 PTI
	Transverse	144,800	122,850	16.5	61.1	45.0		42
2# Al per ton		128,900	112,100	21.5	63.1	62.0	277	37 PTI
	Longitudinal						277-285	37 PTI
	Transverse							
	Longitudinal						277-285	37 PTI
	Transverse							
	Longitudinal	151,600	138,400	15.5	54.4	56.0	241-286	37 PTI
	Transverse	153,300	137,300	14.0	48.3	38.0		
	Longitudinal	151,600	138,400	15.5	54.4	56.0	241-286	37 PTI
	Transverse	153,300	137,300	14.0	48.3	38.0		

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Ballistics

Pinell	Ballistic Limit	Shook	Plate
252	37 mm M51AP-1936 (+61) f/s. PTP, 2-3/8" x 2-7/16". (Plate 646).	75 mm T12AP-PP, SB. Passed. B. L. on plate 646. Satisfactory plate.	G645
277	37 mm M51AP-1448 (+148) f/s. PTP, 2-3/8" x 2-3/8".	75 mm T12AP-PP. Passed. Satisfactory plate.	W2332
290	37 mm M51AP-1938 (+138) f/s. PTP, 2-5/8" x 2-5/8".	75 mm T12AP-PP, SB. Passed. Satisfactory plate.	P10
48-255	37 mm M51AP-1944 (+112) f/s. PTP, 1-1/2" x 2".	75 mm T12AP-PP, SB. Passed. Satisfactory plate.	S99
241	37 mm M51AP-2047 (+214) f/s. PTP, 2" x 2-3/4".	75 mm MK1 proof slug. PP, SB. 1/2" Bow. Passed. Satisfactory plate.	FA5
226	37 mm M51AP-1490 (+142) f/s. PTP, 2-1/2" x 2-1/2".	75 mm T12AP. PP on companion plate HV-1 Passed. Satisfactory plate.	AHV2
48-269	37 mm M51AP-1603 (+111) f/s. PTP, CP, 2" x 2-1/2" on companion 427B2 plate.	75 mm T12AP-CP, Cl. Passed. Satisfactory plate.	JT3427
48-269	37 mm M51AP-1603 (+111) f/s. PTP, CP, 2" x 2-1/2" on companion 427B2 plate.	75 mm T12AP-CP, Cl. Passed. Satisfactory plate.	JT3427
277	37 mm M51AP-1583 (+89) f/s. PTP, 1-7/8" x 2-3/4".	75 mm T12AP. PP, crack on LB. 75 mm MK1 slug, PP, LB. Satisfactory plate.	CI811
77-285	37 mm M51AP-1611 (+111) f/s. PTP, CP, 2-3/8" x 2-1/2".	75 mm T12AP-PP, MB. 75 mm proof slug. CP, 20" x 11" cracks on LB. Satisfactory plate.	GL119
77-285	37 mm M51AP-1611 (+111) f/s. PTP, CP, 2-3/8" x 2-1/2".	75 mm T12AP-PP, MB. 75 mm proof slug. CP, 20" x 11" cracks on LB. Satisfactory plate.	GL119
41-286	37 mm M51AP-1570 (+90) f/s. PTP, CP, 2-1/2" x 2-5/8".	75 mm T12AP-CP, 2" crack on MB. CL. Satisfactory plate.	JT2420
41-286	37 mm M51AP-1570 (+90) f/s. PTP, CP, 2-1/2" x 2-5/8".	75 mm T12AP-CP, 2" crack on MB. CL. Satisfactory plate.	JT2420

TABLE 2. GAS ANALYSIS, RESIDUAL ALUMINUM CONTENT, INCLUSION RATING, AND IMPACT STRENGTH AT -40°F. OF ROLLED AND CAST PLATE

Plate	Gas Analysis										
	Weight, Per Cent, Obtained in Various Fractions										
	1170°C.			1320°C.			1600°C.			Total	
	O ₂	H ₂	N ₂	O ₂	H ₂	N ₂	O ₂	H ₂	N ₂	O ₂	H ₂
<u>CAST</u>											
G545	.000 ₂	nil	nil	nil	nil	nil	.002 ₃	.0000 ₄	.001 ₈	.002 ₅	.0000 ₄
W2332	.000 ₂	nil	nil	.000 ₃	.0000 ₂	nil	.002 ₆	.0000 ₆	.003 ₆	.003 ₁	.0000 ₈
P10	.000 ₃	.0000 ₂	nil	.000 ₂	nil	nil	.003 ₈	.0001 ₄	.003 ₈	.004 ₃	.0001 ₆
S99	.000 ₅	.0000 ₃	nil	.002 ₅	.0000 ₈	nil	.002 ₁	.0000 ₇	.002 ₀	.005 ₁	.0001 ₈
FA5	.000 ₅	.0000 ₄	nil	.005 ₁	.0001 ₅	nil	.002 ₈	.0000 ₈	.004 ₈	.008 ₄	.0002 ₇
AHV2	.002 ₂	.0000 ₇	nil	.004 ₃	.0000 ₃	nil	.004 ₂	.0000 ₉	.002 ₂	.010 ₇	.0001 ₉
<u>ROLLED</u>											
JT3427	.000 ₂	.0000 ₂	nil	.000 ₆	.0001 ₀	nil	.002	.0003 ₆	.001 ₂	.002 ₈	.0004 ₈
JT3427	.000 ₆	.0000 ₂	nil	.000 ₉	nil	nil	.001 ₃	nil	.001 ₁	.002 ₈	.0000 ₂
C1811	.000 ₂	.0000 ₃	nil	.000 ₄	nil	nil	.002 ₂	.0000 ₁	.001 ₆	.002 ₈	.0000 ₄
GL119	.000 ₄	.0000 ₃	nil	.000 ₅	.0000 ₇	nil	.002 ₄	.0000 ₅	.003 ₄	.003 ₃	.0001 ₅
GL119	.000 ₂	nil	nil	.003	nil	nil	.003	.0001 ₈	.003 ₈	.003 ₅	.0001 ₈
JT2420	nil	.0001 ₀	nil	.001 ₀	.0000 ₈	nil	.002	nil	.002 ₃	.003	.0001 ₈
JT2420	.000 ₃	.0000 ₃	nil	.000 ₆	.0000 ₂	nil	.003 ₂	.0001 ₀	.000 ₉	.004 ₁	.0001 ₅

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Total		Residual Aluminum	Inclusions		-40°F. Charpy Impact Values, Foot Pounds		Plate
H ₂	N ₂		Type	Amount	Single Width	Double Width	
.000 ₄	.001 ₈	0.04	III	Clean	16-3/4	40-3/4	G645
.000 ₈	.003 ₆	0.03	II	Dirty	17-1/2	20	W2332
.001 ₆	.003 ₈	0.02	II & III	Slightly dirty	5 - 16-1/2	33	P10
.001 ₈	.002 ₀	0.02	I	Dirty	29	57	S99
.002 ₇	.004 ₈	<0.01	I	Dirty	14	44-1/2	FA5
.001 ₉	.002 ₂	<0.01	I	Dirty	16-1/4	23	AHV2
.004 ₈	.001 ₂	0.06	O _{<2} S _{<2}	Excellent	Longitudinal 35 ¹ / ₂ Transverse 11 ¹ / ₄ -30 ¹ / ₂	Longitudinal 43-51 ¹ / ₂ Transverse 26-56 ¹ / ₄	JT3427
.000 ₂	.001 ₁	0.06	O _{<2} S _{<2}	Excellent	Longitudinal 35 ¹ / ₂ Transverse 11 ¹ / ₄ -30 ¹ / ₂	Longitudinal 43-51 ¹ / ₂ Transverse 26-56 ¹ / ₄	JT3427
.000 ₄	.001 ₆	0.06	O _{2¹/₂} S _{2¹/₂}	Fair	Longitudinal 41 ¹ / ₂ Transverse 35	Longitudinal 94 ¹ / ₂ -84 ¹ / ₂ Transverse 68	CI811
.001 ₅	.003 ₄	0.04	O ₃ S _{2¹/₂}	Poor	Longitudinal 23 ¹ / ₂ Transverse 20 ¹ / ₄	Longitudinal 41 Transverse 36 ¹ / ₂	GL119
.001 ₈	.003 ₈	0.04	O ₃ S _{2¹/₂}	Poor	Longitudinal 23 ¹ / ₂ Transverse 20 ¹ / ₄	Longitudinal 23 ¹ / ₂ Transverse 20 ¹ / ₄	GL119
.001 ₈	.002 ₃	0.03	O ₈ S _{2¹/₂}	Poor	Longitudinal 30 ¹ / ₄ Transverse 26 ¹ / ₄	Longitudinal 61 ¹ / ₄ Transverse 50	JT2420
.001 ₅	.000 ₉	0.03	O ₈ S _{2¹/₂}	Poor	Longitudinal 30 ¹ / ₄ Transverse 26 ¹ / ₄	Longitudinal 61 ¹ / ₄ Transverse 50	JT2420

occur in those steels having a low residual aluminum content. Type II inclusions occupy an intermediate position. In the rolled plate the oxide inclusions tend to increase as the residual aluminum decreases.

It is apparent that most of the oxygen is obtained in the 1600°C. fraction for those steels high in residual aluminum, while those that have low aluminum contents have most of the oxygen removed in the 1170 and 1320°C. fractions. This trend is in agreement with our knowledge of the relative stability of the various oxide inclusions in steel.

The data from which this report was written are recorded in B.N.I. Notebook 988, pages 1 to 6 and Notebook 968, pages 13 to 24.

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