GASES OCCLUDED IN STEEL.

Mr. Thomas Baker, D.Sc., M.Met., F.I.C. (Rotherham), read a paper on "Gases Occluded in Steel."

It is well known that iron and steel are capable of holding in solution or otherwise considerable quantities of gas, and that these are often far from negligible. Many investigators have attacked the problem with the object of ascertaining the composition and volume of these gases and their effect on the physical properties of the metal, but little is known on this subject beyond the part they play in the production of blow-holes, and in a peculiar kind of brittleness which is developed by pickling.

Some years ago the author carried out an investigation with the above-mentioned object, and also to discover the relation, if any exists, between the temperature of evolution of the gas and the critical points of the steel.

For this purpose two crucible steel ingots about six centimetres square were prepared in such a way that whilst one was free from blow-holes the other contained as many as possible. In addition to this material there were also obtained some samples of dead soft steel such as is employed in the manufacture of tin plates. The chemical analyses of these materials are given in Table I.

TABLE I.

	C.	Si.	Mn.	S.	P.	Al.	Fe.
Solid ingot	·90	·088	·096	·023	·019	·033	99·01
Ingot with blow-holes	·81	·080	·050	·028	·019	—	98·84
Soft steel ingot	·13	·011	·370	·053	·075	—	99·36

The apparatus employed for the extraction of the gases consisted of a porcelain tube glazed internally and externally and closed at each end by rubber stoppers. Through one of these passed a glass tube, making connection with a Töpler mercury pump, and through the other two glass tubes into which the leads from the thermo-couple were fused. An electric resistance furnace was employed to heat the tube, and in order to prevent undue heating of the rubber stoppers the ends of the tube were water-cooled. The temperatures were determined by a thermo-couple used in connection with a millivoltmeter.

Before starting an actual experiment it was thought advisable to run a blank; accordingly the tube and pump were thoroughly dried by drawing a stream of dry air through them for several hours. The end of the tube away from the pump was then sealed off and the tube

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exhausted and allowed to stand for 24 hours; the tube was then again exhausted and allowed to stand for a further 24 hours. At the end of this time continued working of the pump failed to pump anything out of the tube. It was next heated for nine periods of 8 hours each to a temperature of about $1,000^{\circ}\text{C}$. and exhausted at the end of every third period. The composition and volume of the gases obtained in these experiments are embodied in Table II.

TABLE II.

	Volume in c.c.	CO ₂ .	H ₂ .	CO.	CH ₄ .	N ₂ .
First period..	0.84	15.48	40.00	—	19.05	25.47
Second period	1.15	18.25	52.20	11.22	5.48	12.78
Third period	1.18	4.23	73.22	13.55	5.93	3.13

No correction has been made for these gases in the appended results on the gases obtained from the various samples of steel.

In this connection it is interesting to note that Fox* found that thoroughly dried oxygen, when swept through an otherwise empty red-hot porcelain tube, persistently yielded small quantities of water—3.5 mgr. during the first hour, 0.7 mgr. from the twelfth to fourteenth hours.

A piece of one of the steels, weighing about 60 grams and having a thermo-couple inserted in one end, was next placed in the tube, the latter exhausted, and the vacuum maintained for 48 hours before starting the heating of the tube. After again exhausting the tube the experiment was commenced and continued over ten days, the period of heating being 11 hours per day; and at intervals during each day the gas was collected and analysed. The volume and composition of the gases are given in Table III.

In the case of the steel with blow-holes, as many as possible were cut open in order to allow the gases contained in them to escape, the object of the author being to obtain the gases from the solid portion of the steel, leaving those contained in the blow-holes for a future investigation.

Some further experiments were carried out to ascertain the effect of such operations as annealing and mechanical work on the volume and composition of the gases. A piece of the sound steel was annealed at a temperature of about $1,200^{\circ}\text{C}$. and the gas extracted in the manner already described; no marked changes were observed in either the composition or volume of the gases. No numerical results are given for this experiment, since they are simply a repetition of those already given for the same steel in the untreated condition.

In order to investigate the effect of mechanical work, an ingot from the same cast of soft steel as that already employed was rolled down from 9 in. square to a plate 9 in. wide by $\frac{1}{2}$ in. thick, and a piece selected from a position corresponding as nearly as possible to that of the piece from the first ingot. The results of this experiment are embodied in Table IV.

* Thesis for Master's Degree in Science, Massachusetts Institute of Technology.

TABLE IV.

Temperature of Extraction.	Volume in c.c.					Percentage Composition.				
	Total.	CO ₂ .	H ₂ .	CO.	CH ₄ .	N ₂ .	CO ₂ .	H ₂ .	CO.	CH ₄ .
<i>Bar—Weight taken, 67.7 grams.</i>										
° C.										
435	1.61	0.06	1.46	0.02	0.07	0.07	3.72	90.68	1.24	4.34
535	2.21	0.02	2.14	—	0.05	0.05	0.91	96.83	—	2.26
610	1.85	0.00	1.71	0.09	0.05	0.05	0.00	92.43	4.86	2.70
679	2.35	0.02	1.80	0.42	0.09	0.09	0.85	76.59	17.87	3.83
722	2.75	0.03	1.43	1.25	0.00	0.04	1.09	52.00	45.45	1.45
788	9.14	0.11	2.48	6.48	0.00	0.07	1.20	27.13	70.89	0.76
823	7.13	0.07	2.43	4.55	0.01	0.07	0.98	34.08	63.81	0.98
872	3.96	0.02	1.81	2.09	0.01	0.03	0.50	45.70	52.78	0.76
927	5.25	0.00	2.48	2.60	0.00	0.17	0.00	47.23	49.52	3.23
Totals..	36.25	0.33	17.74	17.39	0.04	0.64				

TABLE III.

Temperature of Extraction.	Volume in c.c.						Percentage Composition.				
	Total.	CO ₂ .	H ₂ .	CO.	CH ₄	N ₂ .	CO ₂ .	H ₂ .	CO.	CH ₄ .	N ₂ .
<i>Sound Steel—Weight taken, 69.3 grams.</i>											
° C.											
519	6.53	0.07	6.37	0.07	0.02	0.00	1.07	97.54	1.07	0.30	0.00
600	10.67	0.19	8.44	2.00	0.07	0.00	1.78	79.10	18.74	0.65	0.00
672	12.87	0.35	8.07	4.40	0.07	0.00	2.71	62.70	34.18	0.54	0.00
688	22.91	0.75	6.96	15.02	0.12	0.03	3.27	30.38	65.56	0.52	0.13
719	12.61	0.07	4.74	7.72	0.06	0.01	0.55	37.58	61.22	0.47	0.08
765	6.71	0.02	3.35	3.26	0.05	0.02	0.29	49.92	48.58	0.74	0.29
802	4.29	0.00	2.24	1.97	0.08	0.00	0.00	52.21	45.92	1.86	0.00
862	4.59	0.05	2.24	2.22	0.08	0.00	1.08	48.80	48.36	1.74	0.00
936	5.04	0.03	2.53	2.42	0.07	0.00	0.59	50.19	48.01	1.38	0.00
979	5.64	0.01	2.83	2.75	0.04	0.01	0.17	50.17	48.75	0.70	0.17
Totals ..	91.86	1.54	47.77	41.83	0.66	0.07					
<i>Steel with Blow-holes—Weight taken, 63.2 grams.</i>											
530	3.28	0.09	3.04	0.06	0.05	0.04	2.74	92.68	1.82	1.52	1.22
603	2.96	0.06	2.70	0.08	0.06	0.06	2.02	91.21	2.70	2.02	2.02
681	4.36	0.06	2.16	2.01	0.15	0.03	1.37	48.39	46.10	3.45	0.69
696	5.45	0.02	1.49	3.85	0.05	0.04	0.36	27.34	70.64	0.91	0.73
720	5.01	0.03	1.84	3.11	0.06	0.00	0.59	36.50	61.70	1.19	0.00
762	3.80	0.02	1.87	1.81	0.09	0.01	0.52	49.21	47.63	2.37	0.26
825	3.98	0.02	2.01	1.87	0.07	0.01	0.50	50.50	46.98	1.75	0.25
893	4.24	0.03	2.18	1.93	0.10	0.00	0.71	51.41	45.51	2.35	0.00
944	4.48	0.02	2.32	2.08	0.05	0.01	0.45	51.78	46.42	1.11	0.22
1016	4.50	0.02	3.38	1.05	0.05	0.00	0.44	75.11	23.33	1.11	0.00
Totals ..	42.09	0.37	22.99	17.85	0.73	0.20					
<i>Soft Steel Ingot—Weight taken, 66.6 grams.</i>											
411	2.79	0.04	2.68	—	—	0.07	1.43	96.05	—	—	2.51
525	2.37	0.01	2.33	0.01	—	0.02	0.42	98.31	0.42	—	0.84
609	6.05	0.01	5.90	0.13	0.01	0.00	0.16	97.52	2.15	0.16	0.00
667	4.92	0.08	4.58	0.27	0.01	0.00	1.62	93.09	5.48	0.19	0.00
720	7.26	0.31	3.64	3.29	0.02	0.00	4.27	50.13	45.31	0.27	0.00
786	17.19	0.24	5.24	11.63	0.07	0.00	1.40	30.48	67.65	0.40	0.00
836	13.20	0.10	4.19	8.67	0.13	0.11	0.76	31.74	65.68	0.98	0.83
885	7.03	0.01	3.33	3.54	0.13	0.02	0.14	47.37	50.35	1.84	0.28
965	8.03	0.01	3.99	3.89	0.13	0.01	0.12	49.68	48.44	1.61	0.12
Totals ..	68.84	0.81	35.88	31.42	0.50	0.23					

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In Table V the results of all the experiments are collected. From these it appears that the composition is practically the same throughout the series, but that the volume varies considerably. In the case "sound steel" and "steel with blow-holes," the variation in quantity is easily explained, since the latter before and whilst solidifying would boil off the greater part of the gas. In the case of the "soft ingot" also which contained an outer ring of blow-holes, a smaller quantity of gas might reasonably be expected when compared with the sound steel. It is also evident that a mechanical operation such as rolling squeezes out a very considerable portion of the gas, which in the present case amounts to about one-half.

TABLE V.

Description.	Weight of Steel in grms.	Volume of Gas per gm. of Steel in c.c.	Volume of Gas in c.c.	Average percentage Composition.				
				CO ₂ .	H ₂ .	CO.	CH ₄ .	N ₂ .
Sound steel ..	69.3	1.32	91.86	1.68	52.00	45.53	0.72	0.07
Sound steel reheated	47.3	1.40	66.54	1.16	49.55	45.99	2.71	0.59
Steel with blow-holes	63.2	0.66	42.10	0.88	54.56	42.36	1.73	0.47
Soft ingot ..	66.6	1.03	68.84	1.18	52.12	45.64	0.73	0.33
Bar from soft ingot	67.7	0.53	36.25	0.91	49.08	48.12	0.11	1.77

The thermal critical points of these steels occur at the following temperatures :—

	Ac_1	Ac_2	Ac_3	Ar_1	Ar_2	Ar_3
Sound steel and steel with blow-holes	746°			693°		
Soft steel ingot and bar	722°	758°	867°	682°	760°	846°

With the hard steels hydrogen reaches a maximum rate of evolution at 600°, and below this temperature constitutes the greater part of the gas given off. Carbon monoxide is slowly evolved from the beginning of the experiment and reaches *its* maximum rate of evolution at 688°, a temperature which agrees fairly well with that of the Ar_{123} point.

With the soft steel hydrogen again forms the greater portion of the gas collected at temperatures up to 660° and presents a maximum rate of evolution at 609°. It, however, differs from the hard steel in that it has a further maximum at 786°, the temperature at which the carbon monoxide also reaches its maximum rate of evolution. This latter temperature is approximately midway between the A_2 and A_3 points.

Whether these gases are the ones evolved from the steel or are formed by interaction between those actually evolved is a problem that still demands solution.