

# Study on the Method of Taking a Sample for Determination of Oxygen in Molten Steel

Shizuya Maekawa  
Yoshitaka Nakagawa

**D**ETERMINING quickly and precisely the oxygen in the bath during the steel refining process is an important basic subject in the control of the refining operation and in comprehending the mechanism of steel making reactions.

Although much improvement and progress have been seen recently in the determination of oxygen in steel and it has become possible to determine it with accuracy and some rapidity, there has not been any perfected sampling method to ascertain the oxygen content in the bath during the refining process.

Various researches on methods of sampling molten steel for oxygen analysis have been carried out but these methods can be divided into two main groups, namely, collecting samples directly from the furnace, and taking a spoon to removing them from the furnace and pouring them into a mould or the suction methods.

The first method includes those described by Hare, Peterson and Soler<sup>1</sup>, Kobayashi and Kimura<sup>2</sup>, Bassett, Dougherty and Fitterer<sup>3</sup> in which the suction tube was directly inserted into the bath in the furnace. This method also includes those described by McCutchen and Raution<sup>4</sup>, Tawara, Yasuda and Sato<sup>5</sup>; Brower and Larsen<sup>6</sup>, Girardi and Hilty<sup>7</sup>; Huff, Baily and Richards<sup>8</sup>; Gilbert and Baily<sup>9</sup>; Shimose and Kakiuchi<sup>10</sup>; and the authors<sup>11</sup> in which a covered mould is placed in the bath to get samples and is the so-called bomb method. The second method is to remove the sample from the furnace with a spoon and to pour it into a mould, and it is described by Borower and Larsen, Herty<sup>12</sup> and Yonezaki<sup>13</sup>. The latter method also includes one in which suction is made directly from the spoon<sup>14,15</sup>. There are numerous methods as can be noted, but each having its merits and defects it is difficult to state which sampling method would be most suitable. In Table I is given a summary of the advantages and disadvantages of the methods.

The final conclusion on the practicability and rationality of the various sampling methods is not possible yet at the present stage. The main reason is that the methods are imperfect in

principle and the extent of the imperfection cannot even be evaluated clearly. It can also be said that due to there being no established method to ascertain the standard value for the purpose of attaining the true oxygen content of the bath in the furnace at a desired time and due to other apparent defects, it had not been possible to make comparative studies.

It is therefore necessary to establish a sampling method of obtaining the true amount of oxygen in the bath and from this to perfect the most rational and practical sampling method by numerous comparative studies of simple methods. The author conducted several experiments for this purpose and was able to obtain an almost satisfactory result. The outline is as follows:

## Method of vacuum sampling from bath

Of the methods of sampling from bath which have already been published, the suction system<sup>1, 2, 3</sup> required complicated installation and much technical skill, and has almost never been employed. Of the sampling methods which are used the bomb method has the least defects in principle but there are yet many problems that are being discussed. The problems are:

- (a) The dipping of the mould of considerable size and of low temperature into the bath causes local boiling of the molten steel bath around the mould and lowers the O content.
- (b) Air in the mould being drawn in oxidation would increase the O content.
- (c) Possibility of slag being drawn in the mould would increase the O content.
- (d) Releasing gas during solidifying process would decrease the O content.
- (e) Consumption of mould in actual use and the requirement of time for preparation of sample after sampling.

Problem (a) is not a peculiarity of the bomb method but is a problem for all methods that dip the sampling instruments into the furnace. To prevent this it is required to use an instrument as small as possible with preferably small heat capacity and heat conduction.

It is only possible to prevent (b) by making the sampling instrument a vacuum, and for (d) it will be required to catch the evolved gas and conduct an analysis separately.

Dr. Shizuya Maekawa, D.Sc., Chief of Chemical Metallurgy, and Mr. Yoshitaka Nakagawa, B.Sc., Research Engineer, Third Section of Research Laboratory, The Muroran Plant, The Japan Steel Works, Ltd.

**TABLE I**  
*Comparison of sampling methods and their respective advantages and disadvantages.*

Position of samples being taken	Sampling method	Advantages and disadvantages
Inside of the furnace	Taking sample by dipping the mould directly into the bath (Al is put into the mould beforehand).	<ol style="list-style-type: none"> <li>1. Proficiency is required in taking samples.</li> <li>2. Difficult to prepare the sample afterwards.</li> <li>3. Severe wear of moulds.</li> <li>4. By dipping the mould into the bath the temperature drop and change in O<sub>2</sub> content of the bath is considered.</li> <li>5. This sampling method is excellent. There will be an effect of air in the mould.</li> <li>6. Shows a tendency to decrease in O<sub>2</sub> content caused by gas released during solidification.</li> </ol>
	Taking sample by dipping suction apparatus directly into bath.	<ol style="list-style-type: none"> <li>1. Complicated equipment, difficult operation.</li> <li>2. Theoretically very good but difficult for daily operation.</li> </ol>
Outside of the furnace	Taking out a sample with spoon and sucking up molten steel from the spoon (Al may or may not be added beforehand).	<ol style="list-style-type: none"> <li>1. Will be affected by oxygen in the air.</li> <li>2. Shows a tendency to increase in oxygen content caused by reduction of slag when Al is added to spoon.</li> <li>3. Due to molten steel being drawn out of furnace, O<sub>2</sub> content change caused by temperature drop must be considered.</li> </ol>
	Taking out a sample with a spoon and pouring it into a mould (Al may or may not be added beforehand).	<ol style="list-style-type: none"> <li>1. Shows a tendency to decrease in oxygen content caused by gas released during solidification.</li> <li>2. Simple in operation.</li> </ol>

Therefore the only sampling instrument that would satisfy the conditions described above is the vacuum sampling method, although it has many practical difficulties. With this in consideration an improved vacuum sampling method, as described below, was made.

#### *Molten steel suction tube*

The sampling instrument made of quartz has its tip prepared<sup>15</sup> as shown in Fig. 1 and made vacuum. It has a narrow neck at 6 to 7 cm from the tip. Cold iron is placed in the upper part of it for cooling the bath sucked up and 50 to 80 mm Hg of argon gas is also sealed into it; both prevent the violent suction of molten iron to the top of the tube.

#### *Preliminary experement of suction tube*

Several preliminary experiments were conducted with respect to the sipping of molten steel into the suction tube, the conditions of the suction tube

(inside pressure, advisability of the presence of cool iron, etc.) and the amount of gas released during solidification.

(a) *Experiment procedure*: The following sampling methods were conducted at the same time utilising a 35 kVA high frequency furnace to melt approximately 600 g. molten steel. The first method was vacuum sampling method by a suction tube. The next method was to use the same type of sampling tube and to suck up by using lubricator. The third method to be explained in detail later was a

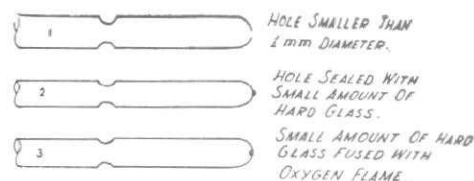


Fig. 1.  
Manufacture of suction tube.

method to pour molten steel into a mould in which aluminium had been placed. The last method was to put aluminium in the molten steel and then to pour it into the mould.

(b) *Analysis method*: Equipment illustrated in Fig. 2 was used for the vacuum fusion method.

The conditions of the analysis were:

Extraction temperature	1900°C
Extracting time	20 min.
Crucible blank	0.2 cc/20 min.
Heating temperature of crucible	higher than 2,100°C.
Heating time of crucible	approximately 3 hrs.
Advance treatment of graphite crucible	with hydrochloric acid.

(c) *Experimental results*: The experimental results are shown in Table II.

It was ascertained by the experiment that it was difficult to fuse the powder of hard glass on the tip of the suction tube in the same condition but if the temperature was high enough it was melted within 10 seconds and the molten steel would enter it. It was also ascertained that an addition of even 1% of aluminium greatly affected the amount of gas that was released during solidification and that sucked molten steel stopped at the appropriate position and was satisfactory. Samples were obtainable by sealing 50 to 80 mm Hg of argon gas in the suction tube and by setting cooling iron and a constriction.

Based on the results from the experiment the following sampling instrument for molten steel and procedure was set up.

### Sampling instrument

The suction tube made by the method described previously was placed in a quartz tube of considerable size for protection purpose with a wooden or copper cap over the suction tube as shown in Fig. 3 and

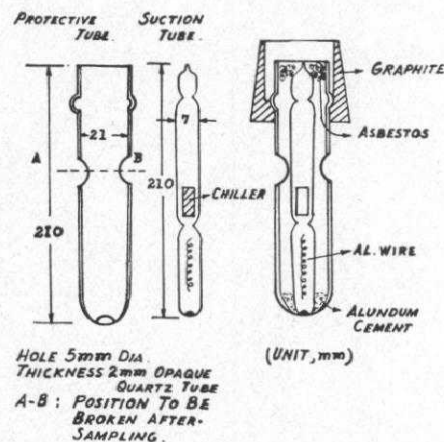


Fig. 3.

Sampling for determination of oxygen in bath.

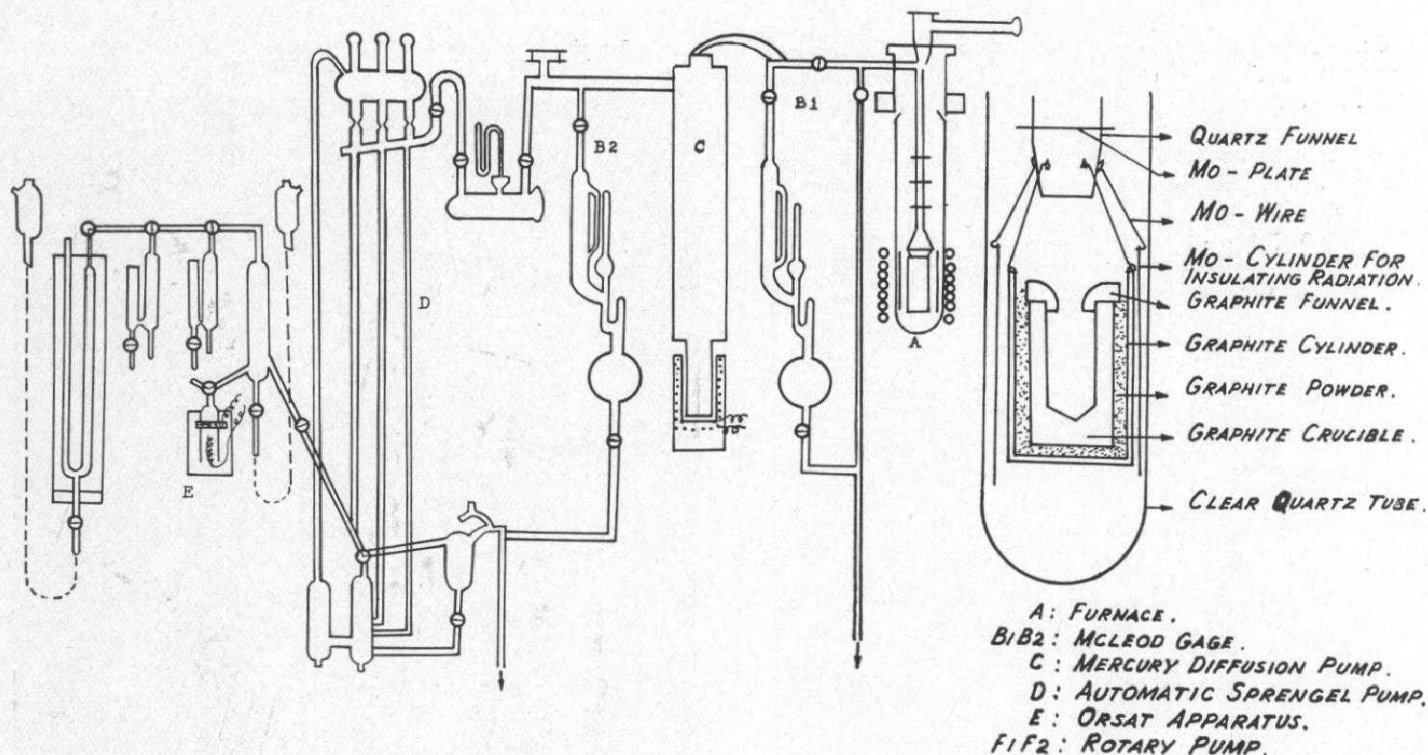


Fig. 2.

Schematic diagram of the vacuum fusion apparatus.



TABLE II  
Preliminary experiment results

Sample No.	Molten Steel temperature (°C)	Time of dipping suction tube (sec)	Value of Oxygen determined %				Gas released by the time of molten steel solidifies in B method	Components of Molten steel		
			A	B	C	D		%		
								C	Si	Mn
1	1580	-	0.017	0.016	-	-	0.0001	-	-	-
2	"	3.5	0.014	0.016	-	-	0.0002	-	-	-
3	1550	4.9	0.016	0.014	-	-	0.0002	-	-	-
4	"	2.3	0.016	0.013	-	-	0.0001	0.09	0.08	0.37
5	1500	5.8	0.009	0.009	-	-	0.0001	-	-	-
6	"	18.1	0.008	0.008	-	-	0.0003	-	-	-
7	1550	2.8	0.007	0.008	-	-	-	-	-	-
8	"	10.5	0.007	0.006	0.008	0.006	-	0.01	0.30	0.66
9	1580	15.6	0.018	0.017	-	-	0.0012	-	-	-
10	"	46.5	0.019	0.022	-	-	0.0104	-	-	-
11	"	-	0.014	0.014	-	-	-	-	-	-
12	"	13.7	0.020	0.029	0.023	0.020	0.0010	0.20	0.04	0.12
13	"	5.6	0.022	0.041	-	-	0.0001	-	-	-
14	"	10.3	0.017	0.018	-	-	0.0002	-	-	-
15	"	4.3	0.024	0.025	0.044	0.025	0.0018	0.14	0.03	0.14
16	"	11.0	0.016	0.012	-	-	0.0001	-	-	-
17	"	4.0	0.017	0.020	0.024	0.019	0.0004	0.22	0.04	0.15
18	"	-	-	-	0.030	0.033	-	-	-	-
19	"	2.5	0.012	0.013	-	-	0.0003	-	-	-
20	"	4.0	0.024	0.020	-	-	0.0003	-	-	-
21	"	2.0	0.022	0.022	-	-	0.0007	-	-	-
22	"	4.0	0.020	0.022	0.021	0.016	0.0016	0.15	0.03	0.12

A: Suction method in which a lubricator is used.  
C: Casting method. (Al in mold)  
\*: Computation of O<sub>2</sub>%.

B: Vacuum sampling method.  
D: Casting method (Al in spoon).

Fig. 4a. This was attached to the tip of the sampling instrument which was protected by a graphite as shown in Fig. 4. The sampling instrument can be operated by 1 to 2 men.

The purpose of the wooden or copper cap was to protect the tip of the suction tube from coming in contact with the slag. Also in the suction tube, there was a pure aluminium sheet of 0.2 mm thickness, 1 mm width and weighing 0.05 to 0.1 g in a spiral shape for killing purpose.

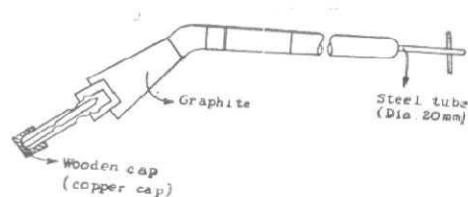


Fig. 4.

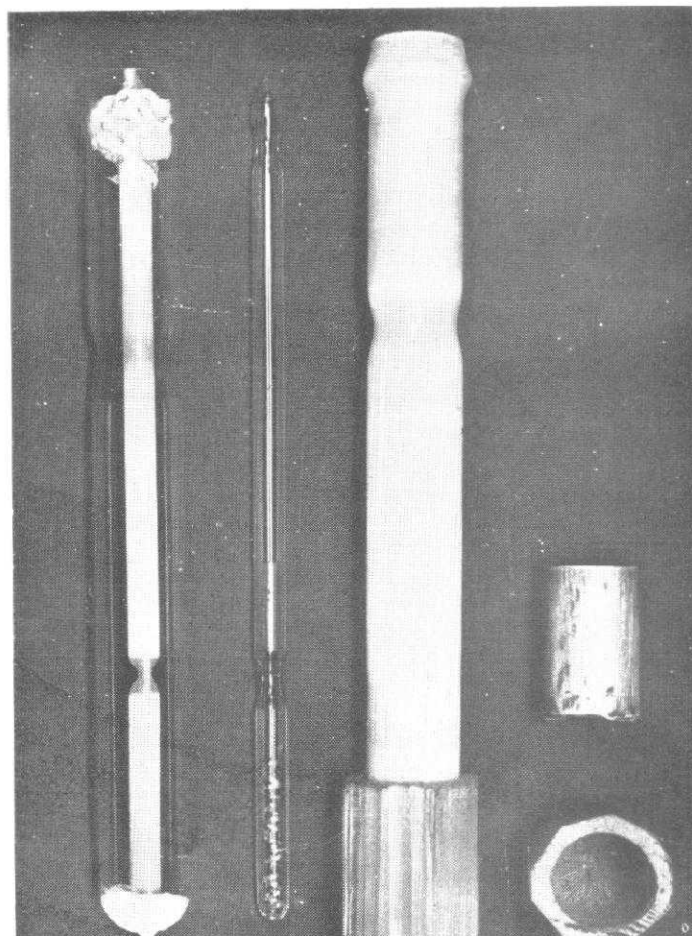
Instrument for taking a sample for determination of oxygen in molten steel bath.

### Sampling procedure

The suction tube which had been placed in the protecting tube was attached to the sampling instrument and inserted into the open hearth furnace

Fig. 4a.

Sampler for determination of the oxygen in bath.



through a sampling window. It was then immediately dipped into the bath (to the depth of approximately 10 cm). If after 3 to 5 seconds the wooden cap should float, the position of the sampler required to be moved a little bit. After holding the sampling instrument for 3 to 30 seconds (in accordance with the temperature of the bath), it was removed from the furnace and the protecting tube was broken by hold A and B of Fig. 3 with chopsticks. The suction tube was removed and the tip was immediately placed in mercury to cool. It was held there. Later the quantity of gas released in the suction tube was calculated and then the suction tube was broken to obtain the sample inside. The sample was lightly polished on the surface and used as analysis sample. The amount of sample taken was from 5 to 15 g, the average weight being 12 g. The total quantity was sent for analysis.

#### Comparison of various sampling methods in actual bath

A comparative study was made of the vacuum sampling method mentioned above, the bomb method that is being employed here and the casting method after spooning out.

#### Out line of bomb method employed and casting method after spoon out

(a) *Sampling by bomb method*: In the bomb method sampling procedure, a mould was used to obtain a sample from the furnace illustrated in Fig. 5. The wooden upper cover was threaded firmly to the mould body with 3 mm wires. This was attached to a Derrick and dipped at 1 metre into the furnace from the main furnace door to get the sample. The mould is charged with 0.5% of pure aluminium (0.15 mm thickness, 8 mm width) in spiral shape.

(b) *Casting method*: Molten steel was scooped

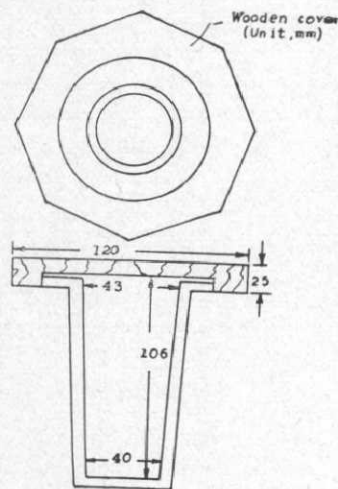


Fig. 5.  
Mould used in bomb method.

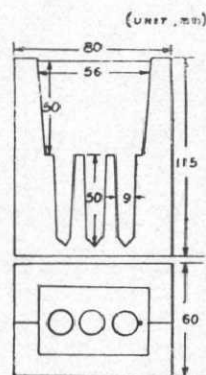


Fig. 6.  
Mould used in casting method.

up with a spoon (capacity approximate 2 kg) and after quickly removing the slag poured into a mould shaped as in Fig. 6 while agitated aluminium of approximately 0.5% was being added. But as there were possibilities of the slag being reduced and  $Al_2O_3$  becoming mixed and also of the aluminium in the molten steel oxidising quickly with the oxygen in the air and increasing the analytical value when aluminium was added to the spoon, a fixed quantity of aluminium was placed in the mould in advance and molten steel was poured into the mould from the spoon from which slag has been removed as much as possible. (The first method will be called "method A" and the second "method B").

(c) *Segregation and comparison of samples from bomb method and casting method*: The samples from the bomb method and the casting method were of considerable size and there were possibilities of segregation within the samples. A study of segregation as well as a comparison of samples taken from the basic and acid baths at the same time was made with the analysis samples cut out at position as shown in Fig. 7. The results of the study are shown in Table III.

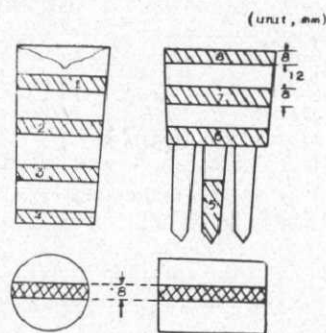


Fig. 7.  
Positions of samples been taken and their symbols.

From the results it was decided to accept positions No. 2 and No. 3 of Fig. 7 as analysis samples from the bomb method sample. On the sample with legs there was some difference due to the positions but position No. 5 was accepted to represent this method after comparing with the bomb method. Of the two methods the casting method had the higher

TABLE III  
Segregation and comparison of samples taken by bomb method and casting method.

Furnace type	Sampling period	Kind of casting method	No.	Chemical composition %			O <sub>2</sub> Content (%)							
							Position of sample in bomb method				Position of sample in casting method			
				C	Si	Mn	1	2	3	4	5	6	7	8
Acid open hearth furnace	After pig addition	A	1	0.27	0.07	0.09	0.017	0.014	0.014	0.014	0.014	0.014	0.016	0.017
			2	0.26	0.08	0.09		0.013			0.015		0.016	
		B	3	0.25	0.09	0.09	0.013	0.012	0.013	0.013	0.015	0.016	0.016	0.013
			4	0.25	0.10	0.09		0.014			0.015		0.019	
Basic open hearth furnace	After Ferro-Manganese addition	A	1	0.16	0.03	0.29	0.023	0.021	0.022	0.023	0.042	0.036	0.038	0.050
			2	0.15	0.02	0.31		0.026			0.035		0.029	
		B	3	0.14	0.03	0.28		0.031	0.024	0.026	0.035	0.032	0.031	0.040
			4	0.13	0.02	0.29		0.030			0.033		0.036	

value but the difference between A method and B method of the casting methods could not be ascertained.

#### Comparative experiment procedure of various type of sampling methods

Experiment procedures are shown in Table IV. Although it is ideal to take samples at the same place and at the same time, and to compare the results for all sampling methods, this was almost impossible actually. During the experiment the order of sampling methods was in principle started with the vacuum sampling method followed by the bomb method, casting method and finally the suction method (Sucked up from the spoon just prior to being poured for casting method). Although endeavours were made to take samples for the various methods continually with the least possible time in between, it required some time due to the operation being conducted in front of the furnace. But judging from the decarburisation condition of the steel bath at that time, the actual change of oxygen content in the molten steel was considered to be within analytical error.

Shown in Table V is the time relation of various methods.

Time required for vacuum sampling through all the other accompanying treatment was within 60 seconds. As the time required for the suction method to get sample in the spoon was the same as in the casting method the actual lapse of time was approximately 45 seconds.

TABLE IV  
Experiment procedure.

Furnace type	Acid open hearth furnace	Basic open hearth furnace
Furnace capacity	Normal 35 ton	Normal 15 ton
Fuel	Producer Gas	
Refining process	Cold charge Lime process	Cold charge Usual method
Kind of steel	Medium carbon steel	Low carbon killed steel
Time of sampling	Various period during refining	
Number of charge	2	3
Outline of various sampling methods		
Vacuum method	As above	
Bomb method	Same as above	
Casting method	Same as above B method	
Suction method	A small hole of 2 mm dia. is made on the tip of suction tube which is identical with vacuum suction method. On the opposite end a rubber tube is attached to a lubricator. The tip is dipped into molten steel with slag removed, which has been taken with a spoon, and then sucked up with the lubricator. Aluminium has been placed in the tube beforehand.	

#### Experiment results

Oxygen analysis values of samples from various sampling methods from bath during refining in the open hearth furnace are shown in Table VI.



TABLE V  
Relation of time in various methods (sec.)

Furnace type	Vacuum method		Bomb method	Casting method	Suction method
	Time of tube being dipped into molten steel	Time from suction tube being removed from molten steel to it being cooled in mercury	Time from suction tube for vacuum sampling being removed to the end in bomb sampling method	Time from suction tube for vacuum sampling being removed to the end in spoon sampling method	Time from suction tube for vacuum sampling being removed to the end in suction sampling method
Acid open hearth furnace	6 - 13	20 - 40	2 - 45	30 - 45	40 - 60
Basic open hearth furnace	16 - 30	20 - 40	2 - 45	30 - 45	35 - 60

### Comparison of sampling methods

The result of study of the difference in analysis values of the vacuum sampling method, as compared with the other methods from the results obtained in Table VI is shown in Table VII. Oxygen analysis values are all higher for all sampling methods with both types of furnaces when compared to the vacuum sampling method. In the vacuum method and bomb method in the acid open hearth furnace there was no difference at all; other methods had apparent differences.

As shown in Fig. 8 on the relation of oxygen analysis results of samples taken by various methods and the C content of molten steel (analysis by bomb method sample) at that time, the oxygen analysis value by the vacuum sampling method was appreciably lower than many of the results obtained in the past. It can be seen that the relation of C-O was very close theoretically when considering the effects of Si and Mn being together and the difference of the temperature of the bath. A separate report on this subject is being considered.

TABLE VII  
Test of significance of various sampling methods.

Furnace type	Comparison of methods	Results of test
Acid open hearth furnace	Vacuum method ~ Bomb method	$n = 9, \bar{\alpha} = 0.0006, \gamma_e = 0.0015, t_0 = 1.28$ $t_0 \leq t \quad (\beta = 8, \alpha = 0.05) \text{ no difference}$
	Vacuum method ~ Casting method	$n = 9, \bar{\alpha} = 0.0082, \gamma_e = 0.0065, t_0 = 3.720$ $t_0 > t \quad (\beta = 8, \alpha = 0.05) \text{ difference exists}$
Basic open hearth furnace	Vacuum method ~ Bomb method	$n = 8, \bar{\alpha} = 0.0126, \gamma_e = 0.0074, t_0 = 4.796$ $t_0 > t \quad (\beta = 7, \alpha = 0.05) \text{ difference exists}$
	Vacuum method ~ Casting method	$n = 9, \bar{\alpha} = 0.0122, \gamma_e = 0.0063, t_0 = 5.865$ $t_0 > t \quad (\beta = 8, \alpha = 0.05) \text{ difference exists}$

TABLE VI  
Effects sampling methods on oxygen analysis value of bath in open hearth furnace.

Furnace Type	Sample No.	Refining period	Chemical composition %			Vacuum method					Bomb method	Casting method	Suction method
			C	Si	Mn	Weight sample gr	Evolved CO gas cc	Oxygen content of evolved CO gas %	Sample Analysis Value %	Total Oxygen %	Analysis value		
Acid open Hearth Furnace	1	Oring Per.	0.79	0.05	0.06	13.3	0.11	0.0006	0.0083	0.009	0.011	0.017	
	2	" "	0.47	0.02	0.05	10.8	0.12	0.0008	0.0111	0.012	0.011	0.018	
	3	Liming Per.	0.23	0.05	0.06	25.5					0.021	0.038	
	4	After lim. per	0.19	0.06	0.06	12.5	0.08	0.0005	0.0166	0.017	0.020	0.037	
	5	Reducing Per.	0.24	0.04	0.06	10.8	0.03	0.0002	0.0173	0.018	0.019	0.030	
	6	" "	0.17	0.03	0.08	15.4	0.04	0.0002	0.0180	0.018	0.019	0.030	
	7	" "	0.18	0.10	0.09	16.8	0.05	0.0002	0.0197	0.020	0.019	0.029	
	8	Before tapping	0.19	0.27	0.52	9.5	0.02	0.0002	0.0159	0.016	0.017	0.020	
	9	Melt down	1.02	0.06	0.09	4.5			0.0059		0.007	0.029	
	10	" "	1.15	0.16	0.21	8.5			0.0051		0.010	0.015	
	11	Before liming	0.61	0.02	0.06	7.0	0.18	0.0019	0.0137	0.016	0.016	0.018	0.012
	12	Before Fe-M	0.35	0.05	0.07	9.5	0.15	0.0009	0.0132	0.014	0.013	0.018	0.012
Basic open Hearth Furnace	1	Deoxidizing	0.11	0.02	0.13	9.2	0.06	0.0005	0.0385	0.039	0.060	0.053	
	2	" Period	0.14	0.02	0.13	8.0	0.04	0.0004	0.0308	0.031	0.037	0.036	
	3	" "	0.10	0.02	0.16	12.5			0.0295		0.036	0.046	
	4	Melt down	0.31	tr	0.13	5.9	0.20	0.0020	0.0133	0.015	0.022	0.022	
	5	Before S.P.	0.19	0.04	0.20	6.6	0.10	0.0010	0.0214	0.022	0.041	0.028	0.032
	6	Before Fe-Mn	0.20	0.01	0.16	9.5	0.26	0.0020	0.0165	0.019	0.023	0.039	0.028
	7	Melt down	0.34	0.02	0.16	7.0	tr	tr	0.0102	0.010	0.016	0.027	0.013
	8	Oring Per.	0.28	0.01	0.15	11.5	tr	tr	0.0138	0.014	0.018	0.019	0.027
	9	Before S.P.	0.24	0.01	0.15	3.5	0.02	0.003	0.0112	0.012		0.030	0.027
	10	Before Fe-Mn	0.26	0.01	0.16	7.0	0.11	0.011	0.0089	0.010	0.027	0.037	0.028

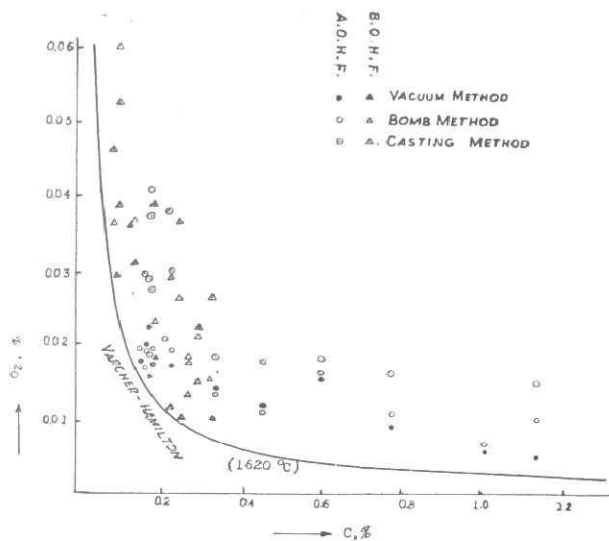


Fig. 8.

Relation between analysis value of various sampling method and carbon content.

### Consideration of the results

Summarising the above results it can be said that :

- In the case of determining the oxygen content in the acid open hearth furnace the vacuum sampling method was very closely similar to the bomb method but apparently differed from the casting method. The casting method showed a higher value in general.
- In the basic open hearth furnace the vacuum sampling method had lower value in comparison with the bomb method, suction method, and casting method.
- The amount of gas that was released during solidification varied with the chemical components of the molten steel, the temperature and condition of solidification and it was difficult to draw a conclusion from the experiment results, but it was believed that an excessive amount of gas was released when the contents of Si and Mn were low and the content was about the amount of medium carbon steel. But in most cases the amount of gas released was within analytical error.

From these results, the rationability of the vacuum method reported here was studied with the following results.

- The vacuum sampling instrument was much smaller than the former bomb method mould and the heat capacity was extremely small and besides it had very low heat conduction. Also, after the wooden cover was removed and the position was slightly changed there was no fear of the temperature of the bath changing quickly around the sampling instrument as it was held in one position for several seconds after being moved. Even

though the steel bath may boil locally it will do so to an extent that can be overlooked. (b) The fact that the vacuum method was relatively similar to the bomb method in the acid open hearth furnace was evidence that the effect of air in the mould was limited. In the case of the basic open hearth furnace the fact that the value was higher than that in the vacuum method was considered due to the properties of the slag rather than peculiarities of the molten steel.

The viscosity of the acidic slag was relatively high while the viscosity was low in the basic and as oxidation was small in the acidic and high in the basic, it can be considered that aluminium was oxidising by the mixing of slag. As the bomb method showed a higher value than the vacuum method which was considered to be only slightly affected by boiling, it became necessary to re-investigate boiling in the bomb method to study whether the effect was only limited or within an amount that could be completely overlooked.

- The high value in the casting method was apparently due to oxygen in the air and this effect was completely obscured in the case the vacuum sampling method. As gas released during solidification was almost entirely caught, there was expected to be no trouble.

### Conclusion

Although the sampling method for oxygen analysis of molten steel in the furnace is currently the subject of much discussion and is not yet perfected, the vacuum sampling method which is almost perfect and relatively simple was invented and gave its results. It was also considered that with some improvement the bomb method would give true reading under certain conditions of the molten steel.

By this method the relation of C-O in the open hearth furnace was studied and a value closer to equilibrium than previously attained was obtainable. Especially during the last stage of refining, a value almost equal to equilibrium was attained. When the effect of Si and Mn activities of C and O is considered, it is thought that the measured value will become even closer to equilibrium value. A separate report will be made on this subject.

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## Bibliography

- <sup>1</sup> W. Hare, L. P. Peterson and G. Solar : Am. Soc. Metals, 25 (1937), 889.
- <sup>2</sup> S. Kobayashi and K. Kimura : J. Iron and Steel Inst. Jap. 25 (1939), 222.
- <sup>3</sup> S. Kobayashi : J. Iron and Steel Inst. Jap. 28(1942), 641.
- <sup>4</sup> J. G. Bassett, J. W. Dougherty and G. R. Fitterer : Open Hearth Proceeding 39 (1956), 44
- <sup>5</sup> K. C. McCutchen and L. J. Rautio : Trans. A.I.M.E., 140 (1940), 133.
- <sup>6</sup> S. Tawara, Y. Yasuda and N. Sato : J. Iron and Steel Inst. Jap. 28 (1942) 1051
- <sup>7</sup> T. E. Brower and B. M. Larsen : Trans. A.I.M.E. 162 (1945), 712.

- <sup>8</sup> D. J. Girardi and D. C. Hilty : J. of Metals, July (1952), 703.
- <sup>9</sup> G. H. Hulf, G. Bailey and J. H. Richards : J. of Metals, Nov (1952), 1162.
- <sup>10</sup> S. Gilbert and G. R. Bailey : J. of Metals, Dec. (1954), 1383.
- <sup>11</sup> K. Shimose and Kakinai : J. Iron and Steel Inst., Jap. 41 (1955), 343.
- <sup>12</sup> S. Maekawa and Y. Nakagawa : Gakujitsu-Shinka-Kai in Japan Rep. No. 19 (1955).
- <sup>13</sup> C. H. Herty : Bl. Fu. and St. Pl. (1930), 468.
- <sup>14</sup> N. Yonezaki : Yahata Seitetsu Re. Dep. Report (1953).
- <sup>15</sup> Gakujitsu Shinko Kai in Japan Rep. No. 19, 3557 (Fuji-Seitetsu) (1954).
- <sup>16</sup> Gakujitsu Shinko Kai in Japan Rep. No. 19, 3559 (Kobe Seiko) (1954).

## DISCUSSIONS

*Mr. N. G. Banerjee, N.M.L.:* The vacuum fusion apparatus is generally operated at 1,560°C for the determination of oxygen in steel, in Europe and America, and this temperature has been established both practically and theoretically from thermodynamical calculations. I would like to ask Dr. Maekawa to comment on the reasons that have led him to operate at 1,900°C when a lower temperature is sufficient.

I fully endorse the author's views that the mode of sampling is the most important factor when bath sample is analysed. I would request the author to let us know the method he has adopted to analyse the gas collected in the vacuum tube during solidification of the sample.

*Dr. S. Maekawa (Author):* In reply to the comments made by Mr. N. G. Banerjee, it may be mentioned that we have determined the residual alumina on the sample by chemical methods after it has been melted during the vacuum fusion analysis. We observed that when the vacuum fusion analysis was operated at lower temperature the residual alumina obtained was considerably high.

The residual alumina obtained at the different operating temperatures are shown below :

Temp. C°	Residual alumina %
1550	about 50
1600	20
1700	4
1800	0
1900	0

From our observation it is clear that the reduction of alumina is not complete below 1,700°C. Though much of the previous work on the vacuum fusion analysis has been carried out at an operating temperature below 1,650°C, the recent trend is to operate at higher temperature. From our experimental data and practical experience we are convinced that better result is obtained when the vacuum fusion is operated at 1,900°C and the analysis is more rapid at this temperature than at a lower temperature.

The analysis of the gas, collected in the suction tube during the solidification of the sample, has been carried out in a Micro Orsat Gas analysis apparatus.

