May 30, 1967 HAJIME NAKAMURA 3,322,530

METHOD FOR ADDING ADDITIVES TO MOLTEN STEEL

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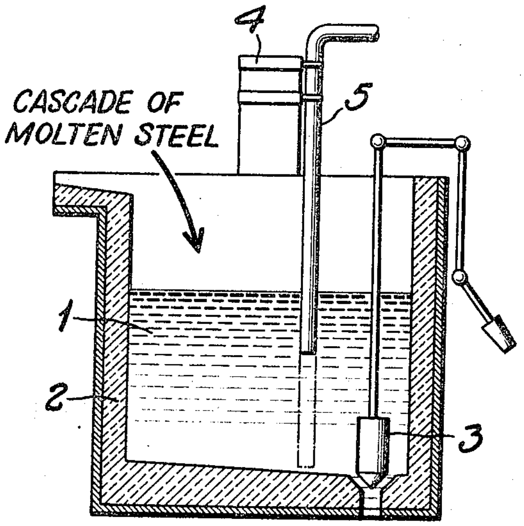

FIG. 1.

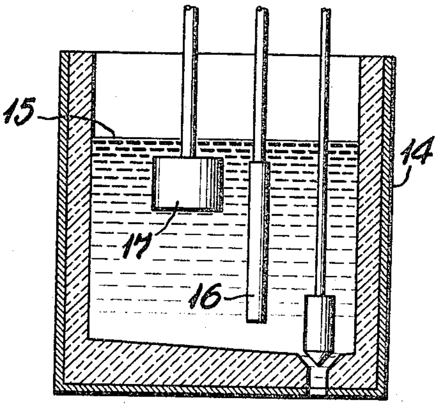

FIG. 4.

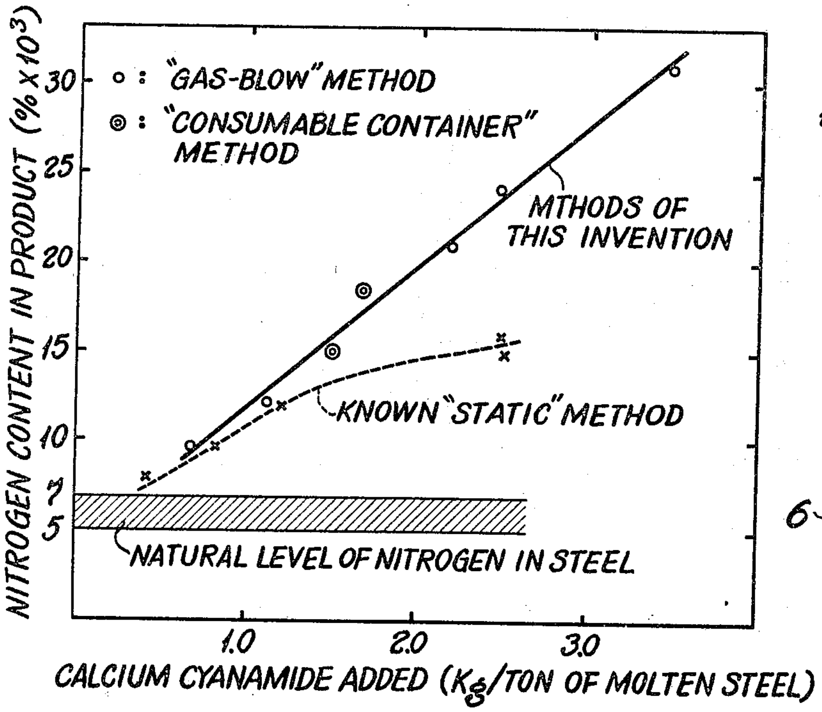

FIG. 2.

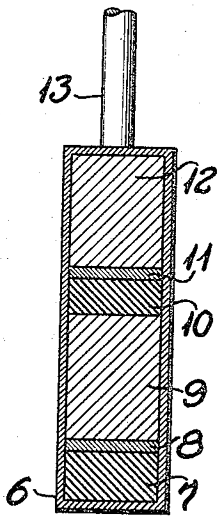

FIG. 3.

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METHOD FOR ADDING ADDITIVES TO MOLTEN STEEL

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13 Claims. (Cl. 75—53)

This invention relates to a method for adding additives to molten steel. More particularly, this invention relates to a method for adding additives to molten steel refined by any way of oxidation refining or both oxidation and reduction refining, the steel being tapped to a ladle equipped with a suitable number of transport pipes in which the additives are provided or through which they are fed to said molten steel. Said transport pipes are so designed as to be consumed in the molten steel from its lower end in approximate synchronism with the rise of said molten steel surface as the latter is accumulated in the ladle in such a manner as to release said additives gradually and steadily in a zone of the molten steel bath that is approximately constant in relative position with respect to the rising surface of said steel bath. The primary purpose of this invention is to provide low carbon unalloyed steel, or low carbon low alloyed steel enriched with nitrogen, and also to provide a process to produce such steels economically and efficiently as well as to provide a new steelmaking process in which the reduction refining may be carried out during and simultaneous with the tapping. Other purposes and applications of this invention will be elucidated in the course of disclosure.

As for effective and economical industrial methods for increasing the nitrogen content of molten steel, I have previously proposed four ways: (1) a method in which the nitrogen gas or nitrogen containing inert gas is blown directly into the steel bath under a pressure that is appropriate to give rise to a proper stirring motion within the bath, (2) a method in which a nitrogen compound is blown into the bath on stream of nitrogen or inert gas, or a gaseous mixture thereof, (3) a method in which calcium cyanamide which is specifically doped with a deoxidation agent is blown into the bath on stream of inert gas or non-oxidizing gas (these three methods will be referred to as the "bath" method hereinafter for the sake of simplicity), and (4) a method in which nitrogen compound is fed on stream of nitrogen gas or inert gas to the bottom portion of the slag layer immediately above the surface of the molten steel bath, namely to the interface between the slag and the melt (this method will be referred to as the "interface" method).

These methods are all immediately applicable to a large industrial steelmaking furnace such as an open hearth furnace, large electric arc furnace or pure oxygen overblowing (the LD) converter furnace, and by using any one of these methods the molten steel in a quantity of as much as 100 tons or over can be enriched with nitrogen to a desired nitrogen content within a few minutes, or at least 10 minutes. However, it is not to be disregarded that even such a short nitrogen enriching stage can become a substantial burden, when the very shortness of the entire steelmaking time is considered to be of utmost importance in such case as, for example, the pure oxygen overblowing converter process (the LD process).

The difficulty can be overcome by carrying out the nitrogen enrichment at the ladle, in other words, in the molten steel which is being tapped or already tapped and in the ladle. For this practice, a number of methods has been proposed. For example, steel smelted by high frequency furnace up to 1 tone capacity and tapped into a ladle was effectively enriched with nitrogen up to about 0.038% by adding nitrogen containing ferromanganese or water-free calcium nitrate (H. J. Wiester et alii, Stahl und Eisen, volume 77, pages 773–784, 1957). (This method will be referred to as the "throw-in" method hereinafter.)

However, since steels tapped into a ladle by conventional industrial method are usually often covered by a thick layer of slag, large amounts of nitrogen enrichment agent thrown into the steel are oxidized and lost upon passing through the slag layer. Therefore only a low and unstable nitrogen yield can be gained.

Alternately, the nitrogen enrichment agent may be previously laid along the bottom of the ladle and the molten steel may be tapped onto said agent in the same way as commonly employed for adding various alloying elements. (This method will be referred to as the "static" method.) In this case, although the efficiency and the stability of nitrogen enrichment may be improved over the "throw-in" method, the reaction of the nitrogen enrichment with molten steel tends to take place violently, and dangerously, so that a portion of the agent inevitably flows away and is lost. Therefore, the efficiency and the precision of nitrogen enrichment is far less than those of this invention, to say nothing about the incapability of the "static" method to attain a nitrogen content over a certain limit.

The above described "bath" methods and the "interface" method are applicable to steel being tapped or already tapped into a ladle. However, these processes are not devoid of shortcomings. Namely, as the molten steel bath in a ladle is often over a few meters deep and hence the uniformity of nitrogen content throughout the steel may not always be assured and, further, the manipulation of the conduit pipe for the nitrogen enrichment agent is not easy as it is at the front of a steelmaking furnace.

In order to overcome the difficulties described above, I have discovered after many tests and experiments that when a pipe, so designed as to possess a consumption (melting) rate which is approximately the same as the rate of the rise of the metal surface within the ladle, and to maintain its free outlet end at a certain depth relative to the rising metal surface, is used either as the conduit pipe through which the desired additives are to be fed into the melt or as a container in which said material is previously stored (such a pipe will be simply referred to as the "consumable transport pipe"), one can perform the addition of additives or the nitrogen enrichment operation quite easily, economically, and with remarkable efficiency without affecting the steelmaking time itself in any way.

As for the nitrogen enrichment agent to be used in this method, agricultural calcium cyanamide is most satisfactory, but other nitrogen containing compounds that readily dissociate at a high temperature to liberate nitrogen gas, such as manganese nitride, chromium nitride, lithium nitride, or nitrogen containing ferroalloys of these elements, can be used just as well. Additives include elements that are commonly referred to as the normal alloying elements, such as nickel, chromium, molybdenum, vanadium, copper, and others, or metallic elements that are to serve as the nitrogen fixer, such as aluminum, beryllium, or columbium, or deoxidational alloying elements such as aluminum, ferrosilicon or ferromanganese, or deoxidizers such as calcium carbide. Those additives may conveniently be in a readily meltable form such as powder, granular, ribbon or tablet.

These additives or nitrogen enrichment agents (these will be simply referred to as the "additives" hereinafter) may be previously stored in the consumable transport pipe (the "consumable container" method), or they may be fed into the molten steel through the consumable transport pipe on stream of a suitable gas such as nitrogen, inert gas or non-oxidizing gas (the "gas-blow" method).

One factor rather critical in practicing this invention, which was found in the course of trials, is to hold the relative depth of the free outlet of the consumable transport pipe at about 5 cm. to 75 cm. from the rising surface of the accumulating molten steel. This can be achieved by, for example, selecting the wall thickness and diameter of the pipe so that natural rate of its melting in the steel bath from the lower end is approximately the same as that of the rise of the molten steel surface. The required wall thickness and the diameter can be found by calculations, but one can readily determine a proper pipe after a few experiments or trials. When the relative depth is too small, the carrier gas tends to splash the additives out into the air, or else, the nitrogen liberated from the agent tends to escape before being absorbed in the steel sufficiently, and a main portion of the nitrogen is lost. Because of the violent agitation at the melt surface and the overlying slag layer, an exact measurement is impossible. Nevertheless it was found empirically that the presence of a molten steel layer of at least 5 cm. above the free end of the consumable pipe assures satisfactory nitrogen enrichment.

I shall now describe operational examples in which the principle of my invention was applied to the electric arc furnace steelmaking process as illustrated in the accompanying drawing in which:

FIGURE 1 is a schematic drawing of a ladle in vertical cross-section for practicing one embodiment of this invention;

FIGURE 2 is a diagram to show the efficiency of nitrogen enrichment of molten steel due to this invention as compared with that due to a known method;

FIGURE 3 is a schematic drawing of a consumable transport pipe in vertical cross section that is convenient for slag reduction refining of the molten steel at the ladle; and

FIGURE 4 is a schematic drawing of a ladle in vertical cross-section for another embodiment of this invention.

FIGURE 1 illustrates schematically an exemplary large ladle equipped with a steel pipe for a convenient practicing of the "gas-blow" method of this invention, wherein **1** is the accumulating molten steel bath, **2** is the heat resistive lining of the ladle, **3** is the stopper, **4** is the hanger handle of the ladle, **5** is the consumable transport pipe whose consumed portion is indicated by broken line. Although in the figure, the consumable transport pipe **5** is fixed against the hanger handle of the ladle **4**, this pipe may equally be well held by any other means, for example, by human hands. FIGURE 2 shows the nitrogen yield in the product steel as function of the amount of nitrogen enrichment agent (calcium cyanamide) added per ton of molten steel. The solid line indicates the results due to my methods while the broken line represents those due to a known method.

Example 1

As soon as the tapping of steel into the ladle was commenced, an agricultural calcium cyanamide of the composition listed in Table 1 was blown into the melt on nitrogen gas under a pressure of about 1 to 1.5 kg./cm.^{2} according to the "gas-blow" method. As the tapping period was slightly over 1 minute for a 3 ton heat and slightly over 2 minutes for a 10 ton heat, the feeding rate of the calcium cyanamide was so adjusted that the desired quantity would be fed to the melt in slightly less than 1 minute or slightly less than 2 minutes in the respective cases.

The dimensions of the ladle were about 100 cm. in diameter at the upper opening and about 120 cm. deep for 3 ton heat about 115 cm. in diameter at the upper opening and about 160 cm. deep for 10 ton heat. The diameter and the wall thickness of the consumable transport pipes used and other pertinent operational data are presented in the Table 2 under Charges 1, 2, 5 and 6. Actual measurements showed that the relative depth of the outlet end of the consumable transport pipes was in any one of the cases always about 20 cm. to 30 cm.

Immediately succeeding the completion of tapping, metallic aluminum for Charges 2 and 5, columbium for Charge 1, and beryllium for Charge 6 was added to the melt. This was done in order to convert the nitrogen into beneficial metallic nitride which is to precipitate out throughout the granular structure of the steel during the hot working process or heat treatment process that was to follow, thereby making the steel highly ductile. The results thus obtained are summarized in the Table 2 as the Charges 1, 2, 5 and 6, and in FIGURE 2 by single circles.

TABLE 1.—CHEMICAL ANALYSIS OF AGRICULTURAL CALCIUM CYANAMIDE, PERCENT

$CaCN_2$	55
CaO	33
C	12

TABLE 2.—CHEMICAL ANALYSIS OF STEELS AND OTHER OPERATIONAL DATA

Charge	Molten Steel, ton	Calcium Cyanamide Added, kg./ton	Nitrogen Content, Percent $\times 10^3$			Nitrogen Yield, Percent		Carbon Content, Percent $\times 10^2$		
			Before	In Product	Increment	Apparent	Net	Before	In Product	Increment
1 [1]	3	0.7	5	9.5	4.5	63.8	30.6	11	12	1
2 [1]	10	1.1	5	12	7	52.0	30.4	14	16	2
3 [2]	10	1.5	6	15	9	47.6	28.6	11	13	2
4 [2]	3	1.7	7	18.5	11.5	50.6	32.3	13	15	2
5 [1]	3	2.2	6	22	16	47.6	33.6	9	12	3
6 [1]	3	2.5	7	34	17	45.6	37.4	10	13	3
7 [1]	10	3.5	6	32	26	43.6	35.4	14	18	4
Mean							32.6			
8 [3]	10	0.4	6	8	2	95.2	23.8	12	12	0
9 [3]	3	0.8	5	9.5	4.5	56.8	26.8	10	11	1
10 [3]	3	1.2	6	12	6	47.8	23.8	11	13	3
11 [3]	3	2.5	7	16	9	30.5	17.2	12	13	1
12 [3]	10	2.5	6	15	9	28.6	17.2	14	17	3
Mean							21.8			

TABLE 2—CHEMICAL ANALYSIS OF STEELS AND OTHER OPERATIONAL DATA—Continued

Charge	Soluble Aluminum Content, Percent			Other Element, Percent			Dimensions of Consumable Transport Pipes, mm.	
	Added	In Product	Yield	Si	Mn	Others	Outer Diameter	Wall Thickness
1 [1]				0.25	0.98	Cb: 0.07	21.7	2.8
2 [1]	0.184	0.136	73.9	0.14	1.34		34.0	3.2
33 [2]	0.168	0.160	95.2	0.39	1.23		89.1	4.2
4 [2]	0.167	0.144	86.2	0.36	0.60	Ni: 0.95 Cr: 0.80 Mo: 0.15	89.1	4.2
5 [1]	0.166	0.105	63.2	0.32	0.86		21.7	2.8
66 [1]				0.27	0.77	Be: 0.03	21.7	2.8
7 [2]				0.45	1.99	V: 0.15	34.0	3.2
Mean			[1] 68.6 [2] 90.7					
8 [3]				0.30	0.62			
9 [3]	0.030	0.025	31.3	0.27	0.89			
10 [3]	0.121	0.069	57.0	0.15	1.05			
11 [3]	0.121	0.084	69.4	0.20	0.71			
12 [3]	0.121	0.068	55.4	0.22	0.92			
Mean			53.3					

[1] The "gas-blow" method of this invention.
[2] The "consumable container" method of this invention.
[3] The known "static" method.

Example 2

Electric arc furnace molten steels of 3 ton and 10 ton were treated by the "consumable container" method. The same calcium cyanamide as the foregoing example was used for the nitrogen enrichment agent. Granular aluminum (Charge 3) or aluminum, nickel, chromium, molybdenum, vanadium (Charge 4) or vanadium (Charge 7) were thoroughly admixed with the calcium cyanamide. The mixture was then compactly stuffed into the consumable transport pipe of diameter and wall thickness as listed in the Table 2 under Charges 3, 4 and 7 until the apparent specific gravity of the calcium cyanamide became about 1.2 to 2. The holding position of the pipe was the same as in FIGURE 1. Although both ends of the pipe were sealed in this example, it was found that the ends may be left open. Other factors such as the tapping time, the relative depth of the outlet and such were the same as in the Example 1. The results from this example are summarized in Table 2 as Charges 3, 4 and 7, and in FIGURE 2 by the double circles.

Another factor that may become critical in practicing my methods is that, depending on the state of on-rushing molten steel cascade, special care may have to be taken to protect the transport pipe from untimely melt down due to molten steel splashes. This can be done in many ways. For example, the pipe may be made in double structure or it may be encircled by a suitable material which is heat resistive enough at relatively lower temperatures but readily melts away at the temperature of the molten steel.

In order to outline the merits of my methods over those of known methods for nitrogen enriching the molten steel, the "static" method which is often practiced today for its apparent simplicity and fairly high efficiency was applied to the electric arc steelmaking process. Namely, the nitrogen enrichment agent, the same calcium cyanamide of the previous examples (Table 1), was laid on the ladle bottom onto which the molten steel was tapped.

The results are summarized in Table 2 as Charges 9 through 12 and presented in FIGURE 2 by symbol crosses. The particulars of the smelting furnace, the steelmaking process, the dimensions of ladles, the tapping time and other pertinent factors were the same as in the previous examples. Metallic aluminum was added into the melt immediately following the completion of tapping.

Examination of Table 2 and FIGURE 2 clearly shows that (1) the nitrogen enrichment of molten steel may be executed by either one of my methods or the known "static" method; however, (2) that my two methods and the known method differ greatly in the efficiency of nitrogen enrichment, namely, while the nitrogen yield in either one of my methods is 32.6%, in the "static" method it is only 21.8% (Table 2); (3) that the nitrogen enrichment efficiency of my methods is very stable or constant up to the saturation nitrogen content of about 0.035% to 0.040%, whereas it tends to tail off for nitrogen contents of over about 0.015% in the "static" method, this being probably because a substantial portion of the nitrogen enrichment agent heaped up on the ladle bottom gets burnt up before ever having an opportunity to react with the molten steel; (4) the yield of aluminum is 90.7% in the "consumable container" method and 68.6% in the "gas-blow" method of my invention, whereas it is only 53.3% in the "static" method (Table 2); (5) although carburization of steel due to the carbon content of nitrogen enrichment agent is unavoidable, particularly when the agricultural calcium cyanamide is used as such, the extent of the carburization due to agricultural calcium cyanamide in my methods are well controlled as to be perfectly predictable, whereas in the "static" method it is so random as to make exact prediction impossible (Table 2).

The present methods of nitrogen enrichment are superior to my other previous methods, the "bath" method and the "interface" method which are mentioned earlier, if the intrinsic difference in their respective applicability is disregarded. Namely, (1) since the nitrogen enrichment of the present methods is performed on the melt being tapped, entirely independent of the smelting process itself, the entire steelmaking duration suffers no prolongation whatsoever despite the addition of this step; (2) the hardship that must be imposed upon the operator in handling the conduit pipe in front of hot furnace in the "bath" or "interface" method is entirely avoided in the present methods; (3) where the nitrogen yield on the agricultural calcium cyanamide is about 17.5% (oxidation slag) to about 21% (reduction slag) in the "bath" method or about 15% in the "interface" method, it is as high as about 32.6% in the present methods; (4) where the aluminum yield is about 25% when forcibly added into the melt at the ladle after having been enriched with nitrogen by either the "bath" or the "interface" method, it is as much as about 68.6% ("gas-blow" method) or about 90.7% ("consumable container" method); this being probably due to the fact that the steel, tapped while being subjected to the steady interaction with the calcium cyanamide, is in a state of more thorough deoxidation by the reaction with the dissociation product from the calcium cyanamide, a factor that will not fail to assure a high yield for elements which otherwise react with oxygen in steel and are lost as nonmetallic inclusion.

Inasmuch as the principle of this invention can be directly applied to other operations, for example, addition of deoxidizer, an entirely new method of steelmaking is here evolved. Namely, I have succeeded in performing the reduction refining process by applying the methods of this invention to the steel which is being tapped to the ladle, thereby improving the yield of various deoxidizers greatly while reducing the entire steelmaking time by a large margin.

Example 3

A 10 ton charge composed mainly of scrap steel was melted in the same electric arc furnace as in the foregoing examples and smelt in the known method of oxidation refining by using pure oxygen gas. The heat was then tapped, without performing the customary reduction refining at the furnace, into a ladle that was equipped with three consumable containers which are schematically illustrated in FIGURE 3. In FIGURE 3, **6** is a steel pipe of inner diameter about 13 cm. and wall thickness about 4.5 cm. whose both ends are closed, **7** and **10** are the layers of deoxidizer calcium carbide of about 10 cm. thick compactly stuffed to an apparent specific gravity of about 2.0, **8** and **11** are the layers of granular aluminum that are to act partially as a deoxidizer and partially as an alloying element, **9** and **12** are the layers of granular ferrosilicon and ferromanganese of about 45 cm. thick that are to act partially as a deoxidizer and partially as an alloying element, and **13** is the stay rod. Thus, about 5 kg. of calcium carbide, about 15 kg. of ferrosilicon, about 30 kg. of ferromanganese and about 0.5 kg. of aluminum were stored in a consumable container, whose wall thickness and diameter were so chosen as to melt away in synchronism with the rise of melt surface in the ladle in the same manner as described in the Example 2 (the "consumable container" method). The relative depth of the container outlet was always about 20 to 30 cm., while the other factors such as the dimensions of the ladle, the manner of container pipe holding, the time of tapping were all the same as those in the previous examples.

Immediately following the completion of tapping, that is the completion of reduction refining in this particular case, the heat was teemed into ingot in an ordinary way.

TABLE 3.—CHEMICAL ANALYSIS OF STEEL, PERCENT

	C	Si	Mn	P	S	Sol. Al	O_2
Furnace	0.10	0.0	0.20	0.018	0.015	0.0	0.035
Product	0.15	0.28	0.82	0.019	0.013	0.021	0.007
Yield	------	85	92	--------	--------	89	--------

Table 3 which presents the chemical composition of the product steel in comparison with that before tapping indubitably indicates that an effective reduction refining was achieved. The merit of this method may be seen in the following points in respect to a normal electric arc furnace smelting process. Namely, (1) the time needed for the reduction refining process in this method is nothing more or less than the time needed for tapping, namely only about 2 minutes at most, whereas it is at least a few tens of minutes when performed at the furnace according to the normal way of smelting, (2) the yield of various additives is about 85% for silicon, about 92% for manganese, about 89% for aluminum in this method, whereas it is only about 63% for silicon, about 60% for manganese and about 40% for aluminum in the customary method, (3) therefore, the ease and the accuracy of controlling the additives, and the economy of fuel or electric power associated with this method are evident.

The primary factor that makes this method of reduction refining very successful appears to reside in the gradual and steady delivery of deoxidizer into the melt and its immediate reaction with the latter, while in the conventional methods, the deoxidizer inevitably comes in contact with air before it begins reacting with steel. In other words, the deoxidizer, the calcium carbide in this case, reacts with the molten steel without ever touching air and reduces the steel thoroughly and locally in the slag reduction reaction into which portion the other additives are now delivered to enjoy a remarkably improved yield. This process is evenly diffused throughout the steel bath by the stirring movement of the bath itself.

Although a transport pipe with both ends closed containing a stratification of various additives composed of well defined layers was used in the above example and in FIGURE 3, the ends of the pipe need not be closed or the additives need not be clearly stratified. In fact, either one or both the ends can be left open and the additives may be stuffed so as to find themselves along a predetermined concentration gradient. Alternatively, the number and the relative position of the consumable transport pipes may be so chosen that during a predetermined period in the tapping only the calcium carbide is delivered to perform the deoxidation refining, then upon closure of that stage, or overlapping with the latter period thereof, the other transport pipes for remaining additives start participating. For this variety, a number of pipes may conveniently be employed in parallel or series disposition, in a manner that is shown schematically in FIGURE 4 in way of illustration. In FIGURE 4, **14** is the ladle, **15** is the level the molten steel is expected to attain ultimately on the completion of tapping, **16** is the consumable transport pipe for calcium carbide, **17** is the consumable transport pipe for other additives which would be ferrosilicon, ferromanganese and aluminum had this variety been applied to the foregoing example. The wall thickness and the diameter of these pipes may be selected individually according to their particular requirements, and they need not be the same.

The improvement in the cleanness of the ingot or the diminuation of the ingotisms gained in my other methods of the "bath" or the "interface" method, that is considered attributable to the action of carrier gas, is also obtained in this new method due probably to the action of the liberated nitrogen and the stirring motion of the molten steel itself in conjunction with the proper usage of gas and additives.

Having disclosed heretofore the principle of my invention and several examples in which the principle was applied in practice, it will be immediately and indubitably understood by all those who are skilled in the art of smelting of steel or indeed of any metal that the principle and applications of my invention are not to be limited to those specific examples given and that many modifications are possible without deviating from the principle of my invention, which is only to be bounded by the appended claims.

I claim:

1. A method comprising adding a nitrogen enrichment agent to molten steel which is being tapped into a receptacle and which thereby has a continuously rising surface by supplying the agent to the molten steel by a consumable pipe which is placed in said steel and is consumed by the steel at a rate to supply said agent thereto at a lower discharge end of the pipe which continuously rises and remains at a depth of 5–75 cm. relative to said rising surface of the molten steel.

2. A method as claimed in claim **1**, wherein the nitrogen enrichment agent is injected into the steel via said pipe by means of an inert gas.

3. A method as claimed in claim **2**, wherein the gas is blown into the molten steel at a pressure of about 1.0 to 1.5 kg./cm.^{2}.

4. A method as claimed in claim **1**, wherein the nitrogen enrichment agent is loaded into the consumable pipe.

5. A method as claimed in claim **1**, wherein the pipe is charged with layers of deoxidizer and alloying metals.

6. A method as claimed in claim **5**, wherein the pipe is a steel pipe with an internal diameter of about 13 cm. and a wall thickness of about 4.5 cm.

7. A method as claimed in claim **1**, wherein the enrichment agent is calcium cyanamide.

8. A method as claimed in claim **7**, wherein the calcium cyanamide is added in an amount of about 0.7–3.5 kg./1 ton.

9. A method as claimed in claim **1**, wherein the tapping time is about 1–2 minutes.

10. A method as claimed in claim **1**, wherein at least one end of the pipe is sealed.

11. A method as claimed in claim **1**, wherein the pipe is protected against premature consumption due to splashing steel with a material which is heat resistive at relatively low temperature but which is consumed in the molten steel.

12. A method as claimed in claim **1** comprising enriching the steel up to about 0.040% nitrogen content.

13. A method as claimed in claim **1**, wherein said steel pipe has an outer diameter of about 20–130 mm. and a wall thickness of about 2.8–45 mm.

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