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# Exploration and Practice of Nitrogen Addition Process for LF Refining Ladle Bottom Blowing Nitrogen Steel Liquid

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**Abstract:** This article discusses and analyzes the law of nitrogen increase in liquid steel and the main factors affecting the nitrogen increasing of molten steel,through the way of adding nitrogen to molten steel by bottom blowing nitrogen gas in LF refining process. It is considered that the main factors affecting the nitrogen increasing instability of molten steel are the initial temperature of LF refining,nitrogen relative element,surface active elements[O]and[S]of steel liquid,and bottom blowing rate of ladle. The large — scale production practice shows that T [O]not more than $50 \times 10^{-6}$ and [S]is not more than 0.020 in LF refining at the initial temperature of not less than 1570. The liquid steel nitrogen enrichment test is carried out by ladle bottom blowing nitrogen gas after 20 min of refining,the flow rate is set as(6.0 ～7.0) NL/min per ton,and it is turned to 2 NL/min at 6 min before the end of refining,the nitrogen increasing rate of liquid steel is basically stable at $(5 \sim 6) \times 10^{-6}$ per minute.
**Keywords:** LF refine; bottom blowing nitrogen gas; nitrogen increase in liquid steel; process practice

## Introduction

Hydrogen and oxygen in steel are generally strictly controlled as harmful elements, but nitrogen has two sides in steel depending on the type of steel being smelted [1]. For example, in pipeline steel production, nitrogen content is generally required to be controlled at $80\% \times 10^{-6}$, otherwise it will deteriorate the impact toughness and welding performance, making it a harmful element; However, in the production process of high-strength steel reinforced with elements such as nitrogen, carbon, vanadium, and titanium, it is often applied as a beneficial element [2]; In addition, in some special steel grades, nitrogen is often present as a special need, such as high nitrogen stainless steel. However, the solubility and absorption rate of nitrogen in steel [3] are influenced by many factors such as temperature, pressure, nitrophilic elements, surface active elements [O] and [S] of the molten steel, and their control laws are not easy to grasp. To study this topic, the author explored and analyzed the control method of adding nitrogen to the molten steel by blowing nitrogen at the bottom of the ladle during the LF refining process under the set conditions, In order to provide useful guidance for the smelting production of steel grades with nitrogen content control requirements.

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## 1 Test equipment and process route

The main experimental equipment includes a 1300 ton mixed iron furnace, a 150 ton desulfurization station, a 150 ton combined blowing converter, a 150 ton argon blowing station, a 150 ton LF furnace, and a 3250mm ultra wide slab continuous casting machine. The composition requirements for the tested steel grades are shown in Table 1.

## 2 Test methods

Selection of experimental process route: mixed iron furnace → desulfurization station → converter → LF ladle refining → slab continuous casting machine.

On the basis of large-scale production, in order to find the ideal experimental parameters under the fresh process, the bottom blowing system of the refining ladle is optimized and

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Table 1 Composition of Test Steel Types

| Experimental steel | C | Si | Mn | P | S | V | N |
|---|---|---|---|---|---|---|---|
| 1# | ≤0. 18 | ≤0.5 | ≤1.6 | ≤0.020 | ≤0.015 | ≤0.10 | 0.0085~0.0150 |

transformed, so that the bottom blowing gas has the function of switching between nitrogen and argon gas. And the following experiments were specifically designed:

(1) Test purpose: Switch the bottom blowing argon gas of LF refining ladle to bottom blowing nitrogen gas under normal pressure, and test the bottom blowing nitrogen increase at the given flow rate regularity.

(2) Test method: First, measure the initial gas content before the start of refining, and then conduct sampling analysis at different refining time periods.

(3) Analysis method: Process the sample into Φ 5×6mm bar sample, requirement: slow precision turning, requirement: the surface of the sample should be free of oil, sand holes, and pores, with a smoothness of Ra 3.2 μm ( ▽ 6), precise analysis is performed using an EMGE-800W/SP oxygen nitrogen hydrogen analyzer.

## 3 Experimental results and analysis

### 3.1 Analysis of initial boundary conditions for refining

Firstly, the initial boundary conditions of LF refining were analyzed, with a focus on the temperature before the start of LF refining, as well as the T [O], [N], and [S] in the steel. Fifty sets of samples were selected, and the experimental results are shown in Table 2.

Table 2 Analysis of Initial Boundary Conditions for LF Refining

| project | Initial temperature/℃ | T[O]/( $10\times10^{-6}$ ) | [N]/( $10\times10^{-6}$ ) | [S]/% |
|---|---|---|---|---|
| Maximum value | 1595 | 82 | 41 | 0.038 |
| minimum value | 1548 | 41 | 26 | 0.012 |
| average value | 1566 | 56 | 33 | 0.019 |
| range | 47 | 41 | 15 | 0.026 |

From Table 2, it can be seen that there are differences and actual control levels in the boundary conditions before LF refining begins. The temperature of the molten steel before refining is mainly affected by factors such as the endpoint temperature of the converter smelting, the insulation state of the ladle, the amount of deoxygenated/alloyed alloy added, and the waiting time of the process. It is generally expected to be as high as possible, not less than 1570 ℃, which can effectively reduce the power transmission time of LF, improve smelting efficiency and smelting effect. In Table 2, the initial temperature of LF refining is generally low, with an average of 1566 ℃, The range deviation is 47 ℃, which will have a certain impact on the stability of the nitrogen addition test for steel liquid; The control levels of T [O] and [S] in steel also exhibit significant fluctuations. T [O] in steel reflects the control level of final deoxidation and oxide inclusions during converter steelmaking, while [S] is mainly influenced by the levels of converter raw material molten iron [S] and converter deoxidation [S]; The basic control of [N] before LF refining is (25-40) × $10^{-6}$, with an average of (30-35) ×$10^{-6}$. Overall, the level of converter smelting and management still needs to be further improved. The significance of analyzing the initial boundary conditions of LF refining is to provide a relatively reasonable explanation for the differential performance of nitrogen addition in steel in subsequent experimental stages.

### 3.2 Analysis of Refining End Point under Different Ladle Bottom Blowing Modes

The main comparative analysis was conducted on the endpoint temperature, T [O], [N], and [S] of LF refining under two conditions: using argon gas throughout the ladle bottom blowing process and switching between argon and nitrogen gas during the bottom blowing process [7]. When switching between argon and nitrogen blowing at the bottom of the ladle, the time is uniformly selected as 20 minutes after the start of LF refining. The nitrogen blowing flow rate at the bottom of the ladle is set to 6.0 NL/min per ton of steel, and it is changed to 2.0 NL/min 6 minutes before the end of refining [8]. 50 sets of random samples are selected for sampling. Selection conditions: 1# steel grade, no abnormalities in the overall refining process, with a cycle controlled within 30 minutes to 35 minutes, and the basic consistency of the bottom blowing flow rate during the smelting process. The test results are shown in Tables 3 and 4.

Table 3 LF Refining End Point [N] Analysis

| project | Argon bottom blowing/( $10\times10-6$ ) | Switching between argon and nitrogen bottom blowing/( $10\times10-6$ ) |
|---|---|---|
| Maximum value | 71 | 128 |
| average value | 38 | 95 |
| average value | 48 | 108 |
| Mean deviation value | 60 | 60 |

When analyzing the LF refining endpoint under different ladle bottom blowing modes, the reason why the selection of experiments and samples is uniformly set is to avoid interference from external conditions as much as possible, which affects the authenticity of data comparison and analysis. Based on Table 3, it can be seen that after switching between

Table 4 Analysis of Other Factors in LF Refining

| project | Argon bottom blowing | | | Switching between argon and nitrogen bottom blowing | | |
|---|---|---|---|---|---|---|
| | temperature/℃ | T[O]/( 10 × $10^{-6}$ ) | [S]/% | temperature/℃ | T[O]/( 10 × $10^{-6}$ ) | [S]/% |
| Maximum value | 1585 | 13 | 0.008 | 1583 | 36 | 0.011 |
| minimum value | 1586 | 19 | 0.003 | 1567 | 22 | 0.003 |
| average value | 1575 | 26 | 0.005 | 1574 | 36 | 0.005 |

argon/nitrogen gas for bottom blowing in LF refining ladle, the nitrogen addition in the molten steel significantly increases, with an average increase of 60% $\times 10^{-6}$, basically equivalent to every minute (5-6) $\times 10^{-6}$; Meanwhile, from Table 4, it can be seen that the control levels of LF refining endpoint temperature, T [O], and [S] are basically consistent [9].

### 3.3 Analysis of nitrogen addition in molten steel under different bottom blowing modes

In order to understand the changes in [N] in steel during the LF refining process using full bottom blowing of argon gas in the ladle and switching between argon and nitrogen gas during the bottom blowing process, 7 time points were selected from 0 min, 10 min, 20 min, 23 min, 26 min, and 29 min after the LF refining began, and sampling analysis was conducted. When switching between argon and nitrogen blowing at the bottom of the ladle, the time is still uniformly selected as 20 minutes after the start of LF refining, and 5 sets of continuous refining process tracking samples are selected for each. Selection criteria: 1# steel grade, no abnormalities in the overall refining process, the cycle is controlled within 30 minutes to 35 minutes, and the bottom blowing flow rate of the ladle is basically consistent during the smelting process.

The initial [N] level of LF refining is basically the same, all within (30-40) $\times 10^{-6}$, during LF refining for 20 minutes, the steel liquid [N] is also at a consistent level, at (35-60) $\times 10^{-6}$, the nitrogen increase in molten steel is (5-20) $\times 10^{-6}$, analyze the possible reasons for its fluctuation, which may be influenced by the different power transmission times caused by the initial temperature fluctuation of LF refining, as well as the influence of surface active elements such as initial T [O] and [S] in the steel liquid [10]. After 20 minutes of LF refining, the slag making and heating period of LF refining are basically completed, and the control level of steel temperature, T [O], and [S] gradually tends to be consistent. For the same steel grade, the difference in external influencing factors will be significantly reduced. Further tracking and analyzing the nitrogen addition law of molten steel after 20 minutes of LF refining, two significantly different experimental results were obtained under two different ladle bottom blowing modes: the nitrogen addition effect of molten steel was significant after using nitrogen for ladle bottom blowing.

### 3.4 Analysis of factors affecting nitrogen addition in molten steel

The main factors affecting the nitrogen addition in molten steel during LF refining process are analyzed, and the following points need to be noted [11]: (1) Temperature of refined molten steel. The starting temperature of LF refining directly affects the refining efficiency and metallurgical effect. A low temperature at the refining station means that the slag making process is slow in the early stage of refining, and the heating time for LF refining is increased. The arc is prone to dissociation, and nitrogen atoms in the air form [N] atoms, which enter the steel liquid. This is one of the reasons for the instability of nitrogen control in LF refining steel liquid; (2) The presence of nitrophilic elements. Elements with strong affinity for [N] at steelmaking temperature include [Al], [Ti], [V], [Nb], [B], etc. They are easy to form stable compounds with [N] element in the steel liquid, causing the dissolution equilibrium of nitrogen in the steel liquid to shift to the right, which can improve the efficiency of increasing [N]. Therefore, for different steel grades, even if other external conditions are the same, The nitrogen increasing effect of steel containing nitrophilic elements may be better than that of steel without nitrophilic elements, which can be verified according to the experimental method introduced in this article; (3) Surface active elements in molten steel. Here, it mainly refers to [O] and [S] in the molten steel. When smelting low-carbon steel, the endpoint oxygen of the converter is relatively high; At the same time, the content of [S] in the initial stage of LF refining is relatively high. Due to the presence of [O] and [S] in the steel liquid, it will inhibit the nitrogen increasing effect of the steel liquid. This situation is generally manifested in the early stage of LF refining. Therefore, the unstable nitrogen increasing effect of the steel liquid in the initial stage of LF refining is related to it; (4) The bottom blowing flow rate of the ladle during the refining process. The setting of the bottom blowing flow rate of the ladle during the refining process needs to consider requirements such as steel composition, liquidus temperature, ladle capacity, etc. It is not only affected by the refining process control, but also limited by the actual equipment situation of each enterprise, and the specific setting may vary slightly. In general, the refining heating and slag making period bottom blowing flow rate is set according to the ton of steel (2.0-4.0) NL/min, the enhanced desulfurization period is set according to the ton of steel (5.0-8.0) NL/min, and the feeding and weak stirring period is set according to the ton of steel (2.0-3.0) NL/min. Different bottom blowing flow rates will affect the nitrogen increasing effect of the steel liquid, which needs to be verified through specific experiments, The author only simulated the nitrogen addition of molten steel under

specific flow rates under specific large-scale production conditions, so this is explained here [12].

Therefore, stabilizing the initial boundary conditions of LF refining is one of the key factors for stable nitrogen addition in steel during the LF refining process.

## 4 Application Practice and Effect of 4 Steel Grades

Guided by the above experimental results and theoretical analysis, the production practice of bottom blowing nitrogen in steel was carried out on Q460 grade high-strength steel for medium and thick plates. Clarify the initial boundary conditions for LF refining as follows: temperature not lower than 1570 ℃, and T [O] in steel not greater than $50 \times 10^{-6}$, [S] shall not exceed 0.020%, and nitrogen addition tests shall be conducted uniformly after 20 minutes of refining. The smelting cycle shall be set to 35-40 minutes according to the specific production rhythm. After 20 minutes, nitrogen gas shall be blown from the bottom of the ladle, and the flow rate shall be set to per ton of steel (6.0-7.0) NL/min. At 6 minutes before the end of refining, it shall be converted to 2.0 NL/min. Under these conditions, 100 furnaces of large-scale production shall be randomly selected for testing. The stability of using ladle bottom blowing to increase nitrogen in large-scale production is good, and the nitrogen content in the steel can be stably controlled within the refining cycle at $(90\text{-}140) \times 10^{-6}$. The nitrogen increase using bottom blown nitrogen mode should generally be within (50-70) compared to not using bottom blown nitrogen mode$\times 10^{-6}$ can fully meet the nitrogen content requirements of steel grades.

## 5 Conclusion

(1) It is feasible to add nitrogen to the molten steel by blowing nitrogen at the bottom of the ladle during the LF refining process. (2) Analysis suggests that the initial boundary conditions for LF refining should be set as follows: temperature should be controlled above 1570 ℃, and T [O] in the steel should not exceed $50\times10^{-6}$, [S] not exceeding 0.020% is crucial, as it can create favorable conditions for subsequent stable bottom blowing of the ladle to increase nitrogen. (3) After 20 minutes of refining, nitrogen addition is carried out by blowing nitrogen at the bottom of the ladle. The flow rate of the bottom blowing is set at (6.0-7.0) NL/min per ton of steel (simulating large-scale production status), and it turns to 2.0 NL/min 6 minutes before the end of refining. At this time, the nitrogen addition rate of the molten steel can be basically stable at 5-6 per minute $\times 10^{-6}$.

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