Development of a methodology for studying the influence of technological factors of production on the quality of large ingots from stamped steel grades 5XHM and 56NiCrMoV7

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Abstract. Currently, one of the main problems of metallurgical and machine-building production is the high proportion of defective metal, which is often associated with the formation of flocs in a steel ingot and increased metal contamination by non-metallic inclusions. One of the methods aimed at minimizing the amount of nonmetallic inclusions in deoxidized steel is the use of rare earth and alkaline earth metals as modifiers. The selection of the modifier must be carried out based on the chemical composition of the steel, as well as the technological features of production. In this regard, to assess the effectiveness of the influence of deoxidation and modification technology, it is necessary to develop a comprehensive research methodology that allows taking into account various technological factors of production in the conditions of variability of input parameters. This article presents an analysis of the quality of forgings made of steel grades 5XNM and 56NiCrMoV7, produced according to various technological schemes of deoxidation and modification, based on the use of a complex technique that includes metallographic analysis, X-ray spectral microanalysis, and thermodynamic modeling. Based on the results of the implementation of the method, a conclusion is made about the optimal technological scheme of deoxidation and modification.

1. Introduction

Ensuring the required quality of products is the main task within the existing trends typical for the metallurgical and machine-building industries. Solutions to minimize defects are being actively developed for the production of steel products [1-3]. The reasons for the formation of defects are various, but the most common are the negative effects of hydrogen with the corresponding formation of flocs, increased concentrations of sulfur and phosphorus in steel, excessive iron loss, and the presence of large clusters of non-metallic inclusions (NMI) [4-6].

The most common quality problem of modern steel grades is the increased contamination of products with non-metallic inclusions. Inclusions can have both an endogenous nature of formation associated with the processes of deoxidation, desulfurization and modification, and an exogenous nature caused by the ingress of slag or lining particles into the liquid steel [8-10].

The process of generation of non-metallic inclusions is similar to the formation of solid steel particles during crystallization by the cluster mechanism. Only particles larger than the critical nucleation radius are capable of further growth [11].

Contamination with nonmetallic inclusions has a significant impact on the final mechanical properties of the metal. Depending on the size of inclusions in the metal and the ratio of the average size
of NMI and grain, inclusions can significantly negatively affect the mechanical properties, and improve them. If the inclusions are on average small (< 6 microns) and distributed evenly across the matrix, they begin to harden the metal by the mechanism of dispersion hardening. [12].

In the opposite situation, if the inclusions are either of a significant size or are distributed extremely heterogeneously with local areas of high contamination, they will become sources of crack generation. In this case, as a rule, the inclusion reaches a significant size commensurate with the size of the grain after heat treatment and can lead to cracking [13, 14].

To minimize the negative impact of hydrogen and non-metallic inclusions (in combination), microalloying and modification of steel with rare earth metals (REM) is traditionally used [14]. The hydrogen content in the metal during the implementation of this method is reduced due to diffusion from the metal surface by its release in its gaseous form during vacuum treatment of steel [15].

REM also has an important influence on non-metallic inclusions, affecting both their morphology (causing globularization) and topography. Without the use of REM, most (70-80%) inclusions accumulate along the grain boundaries, which negatively affects the ductility of steel.

As an alternative, use of alkaline earth metals (AEM) is proposed. Combined use of modifiers containing REM and AEM improves the final result of partial removal of excess hydrogen with the resulting non-metallic inclusions and binding of its atomic part to hydride groups [16].

Qualitative and quantitative selection of optimal modifiers must be carried out individually for different grades of steel used for forging production, as well as taking into account the technological characteristics of enterprises. Evaluation of the most appropriate technology for producing forgings with further minimization of non-metallic inclusions and flocs requires the development of a technique that allows a comprehensive analysis of the impact of technological parameters on the quality of the product. This article presents the results of the implementation of various analytical techniques in order to select the most optimal deoxidation scheme in the process of metal processing.

2. Materials and methods

Cast metal and forgings samples from seven industrial heats produced in an electric arc furnace (4 5XHM steel heats and 3 56NiCrMoV7 heats) were examined using the techniques described below. The analysis of production technology revealed three main deoxidation schemes during metal processing (table 1): the first group was deoxidized by vacuum-carbon deoxidation (VCD), the second group was deoxidized with aluminum, and the third group, which had the highest percentage of defects, was deoxidized with a combination of aluminum and calcium.

Table 1. Deoxidation schemes for steel grades 5XHM and 56NiCrMoV7, kg

| deoxidation scheme | No. | CaC₂ | FeMn 78 | Mn95 | FeSi65 | FeSi45 | Al | FeCa40 | Grade       |
|-------------------|-----|------|---------|------|--------|--------|----|--------|-------------|
| VCD              | 1   | -    | 100     | 208  | -      | 750    | -  | -      | 5XHM        |
|                   | 2   | -    | 550     | -    | -      | -      | -  | -      | 5XHM        |
| Deoxidation Al    | 3   | 100  | 750     | -    | -      | 1550   | 20 | -      | 5XHM        |
|                   | 4   | 150  | 1600    | -    | -      | 600    | 60 | -      | 56NiCrMoV7  |
| Deoxidation Al + Ca | 5   | -    | 750     | -    | -      | 400    | 100| 200    | 56NiCrMoV7  |
|                   | 6   | 150  | 1600    | -    | -      | 600    | 60 | 288    | 56NiCrMoV7  |
|                   | 7   | 150  | 700     | -    | 100    | 250    | 50 | 200    | 5XHM        |

In the course of the work, metallographic analysis of contamination with non-metallic inclusions of
metal samples taken during refining and casting of metal, as well as samples from forgings of four heats, was carried out. Samples from forgings were taken from the bottom (B) and sub-feeder (SF) parts in the axial parts of the ingots. Analysis of non-metallic inclusions in both cast and forged samples was performed in accordance with the ASTM E1245 standard. Forgings were additionally evaluated according to the domestic standard GOST 1778-70, method SH6.

To assess the distribution of non-metallic inclusions, X-ray spectral microanalysis was used, which was performed on a Zeiss SUPRA 55VP scanning electron microscope equipped with an Inca (Oxford Instruments) microprobe.

Thermodynamic modeling methods were used to calculate the conditions of slag crystallization. The final composition of the slag and critical temperatures were obtained. The desulphurizing properties of the slag were also studied, and the sulfide capacity was calculated.

### 3. Results and discussion

The greatest interest was represented by technological schemes of steel heats 56NiCrMoV7 (Figure 1), which are characterized by a complex sequence of additives and a long processing time.

In the future, when studying non-metallic inclusions, it will be revealed that these heats will receive the highest values of the contamination score according to GOST 1778-70.

![Additive scheme for heat 6, steel 56NiCrMoV7 (Deoxidation Al + Ca).](image)

Samples of cast metal selected during ladle treatment and casting, according to the results of metallographic analysis of contamination with non-metallic inclusions, turned out to be quite clean. The total level of metal contamination with non-metallic inclusions in the metal samples taken before casting did not exceed 0.017 vol.%. However, the contamination of the forgings metal was significantly higher, especially in the feeder part of the ingot, the level of contamination reaches 0.05 vol.%. In the samples of forged metal, in contrast to cast metal, there are areas with non-metallic inclusions up to 100 microns in size. Such areas stand out sharply from the general range of data and, when analyzed according to the national standard, give a significant contamination score. In the analysis according to GOST 1778, data were obtained that in forgings of steel 56NiCrMoV7 (bottoms 6, 7) contamination of the metal is much higher cast samples, sulphides score was level 3-5, the silicates are deformable at the level of 2-5. In forgings from 5XHM steel (heat 2), the results are better: with a score...
of 0.2 for sulfides, point oxides and non-deformable silicates. In the forgings from the 56NiCrMoV7 (heat 5), the score is at the level of 0.5-2.0 for sulfides and oxides, which is much better than in the other two forgings from this grade. The significant increase in inclusions in heat 6 may be related to the refining time, which was almost 10 hours, which is at least twice the average processing time for stamped steels (about 280 minutes). In addition, in heats 6 and 7, the metal was casted at a 20% faster rate than in heat 5.

Based on the results of thermodynamic modeling, it can be concluded that for cast metal, the dependence of the calculated parameters of the desulphurizing ability of the slag with the practically obtained values is in principle the same, only heat 3 does not correspond, in which large sulfides occur with a significant amount of sulfide capacity (table 2). A similar analysis of the metal contamination of forgings showed that on average the amount of sulfides remains at the same level, but there are areas with a volume fraction greater than 0.3% (4 and 5). In the first case, the ingot is syphon and the last in the series, this fact may explain areas with high bottom contamination against feeder. The second ingot is a vacuum ingot and the first in the series, such a significant contamination of the metal with large-sized NM1 can be explained by the fact that the liquid steel erodes the refractory particles.

### Table 2. Dependence of the volume fraction of sulfides on the sulfide capacity of slag in cast metal and forgings, evaluated in accordance with ASTM E1245, where SF is the sub-feeder part of the forging, B is the bottom part

| No. sample | Sulphide slag capacity, Cs | Cast metal | The maximum proportion of sulfides, vol. % | Volume fraction of sulfides, vol. % | The maximum proportion of sulfides, vol. % |
|------------|---------------------------|------------|-------------------------------------------|-----------------------------------|-------------------------------------------|
|            |                           | volume     |                                           |                                   |                                           |
|            |                           | fraction of |                                           |                                   |                                           |
|            |                           | sulfides,  |                                           |                                   |                                           |
|            |                           | vol. %     |                                           |                                   |                                           |
|            |                           |            |                                           |                                   |                                           |
| 1          | 30                        | 0.003      | 0.004                                     | -                                 | -                                         |
| 2          | 6                         | 0.003      | 0.0033                                    | 0.003                             | 0.006                                     | 0.038                                     | 0.120                                     |
| 3          | 41                        | 0.0028     | 0.2                                      | -                                 | -                                         | -                                         |
| 4          | 39                        | 0.002      | 0.0021                                    | 0.026                             | 0.048                                     | 0.370                                     | 0.160                                     |
| 5          | 21                        | 0.008      | 0.0037                                    | 0.006                             | 0.008                                     | 0.060                                     | 0.340                                     |
| 6          | 6                         | 0.010      | 0.012                                     | 0.031                             | 0.006                                     | 0.100                                     | 0.039                                     |
| 7          | 25                        | 0.017      | 0.068                                     | -                                 | -                                         | -                                         | -                                         |

Based on the results of X-ray spectral microanalysis, it was found that the main type of non-metallic inclusions are oxysulfides of the Al₂O₃+MnS type (figure 2). They are found both in pure form and as part of complex compounds with MnO, SiO₂, MgO, and Ti₅O₇. In two heats (1, 3), the appearance of titanium in the composition of complexes with Al₂O₃-MnS is observed. At heat 5, magnesium is observed in the base metal and an increased content of inclusions and slag (endogenous MgO compounds are found).

CaO in the composition of inclusions is present slightly, on heats with a combined refining scheme only in 4, calcium oxide is the main type of inclusions. In other cases, these are either exogenous inclusions from the slag, or single inclusions for the entire sample. The greatest contamination of the metal with sulfides is observed in samples from heats 7 and 6, which used a combined scheme of deoxidation with aluminum and calcium.

When using the analysis of the thermal nature of the detected non-metallic inclusions, carried out by thermodynamic modeling methods, data were obtained that confirm the results of the X-ray analysis (table 3).

Comparison of calculated data with actual data obtained after metallographic analysis shows convergence with respect to oxides. In two heats (4 and 1), a significantly higher amount of sulfides is calculated (0.002 and 0.003%, respectively, in cast metal after metallographic analysis).
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Figure 2. Complex inclusion of the Al₂O₃-MnS system.

Table 3. The final mass of non-metallic inclusions according to the simulation results, wt.%

| No. of heat | Al₂O₃ | SiO₂ | MnS | 3Al₂O₃·2SiO₂ | Oxide melt | MgO·Al₂O₃ |
|------------|-------|------|-----|-------------|------------|-----------|
| 1          | 0,003 | 0,001| 0,011| 0,004       | 0,007      | -         |
| 2          | 0,004 | 0,002| 0,004| 0,000       | 0,005      | -         |
| 3          | 0,012 | -    | 0,013| 0,000       | 0,001      | -         |
| 4          | -     | 0,002| 0,011| -           | 0,009      | -         |
| 5          | 0,006 | 0,002| 0,009| 0,000       | -          | 0,001     |
| 6          | 0,001 | -    | 0,005| -           | 0,013      | -         |
| 7          | 0,004 | 0,002| 0,014| 0,000       | 0,005      | -         |

4. Conclusion

Based on the results of metallographic analysis, X-ray microanalysis, as well as the results of thermodynamic modeling, it can be concluded that in the process of ladle treatment, the second technology (deoxidation with lumpy aluminum) shows the best results for the contamination of steel with non-metallic inclusions. When using the combined scheme, it is necessary to move the time of adding calcium-containing materials as close as possible to the casting to increase its effectiveness as a modifier.

Slags of all heats are characterized by a high content of MgO (8-18%), which leads to the appearance
of magnesium in the composition of NMI and the base metal. Most-likely source of magnesium is slag-forming, since while such an amount of magnesite can be obtained from the lining, but this would affect the ladle campaign and would be noticed. After analyzing the heat maps, it was determined that even when analyzing a single technology, most of the technological parameters vary significantly, which suggests that there is no established technology or that it is impossible to perform it in production.

Thus, the methodology presented in this article allows us to analyze the possible causes of defects, select the optimal technology for secondary treatment and obtain reproducible results on the quality of forgings in the case of maintaining technological parameters at the same level.

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