

MANUFACTURE OF FERROUS AND NONFERROUS METALS

Controlling the Composition of Nonmetallic Inclusions in a Low-Alloy Manganese Steel

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At the modern level of out-furnace treatment of steel, the steel composition becomes a substantial factor that determines the degree of influence of oxide particles on its properties. Therefore, the development of a method for controlling the inclusion composition is a challenging and technologically feasible problem. In this work, we study the possibility of controlling the composition of nonmetallic inclusions in a low-alloy manganese steel and of providing a given composition of an oxide phase by introducing a controlled amount of calcium, which was determined from the oxygen activity in the steel measured by the emf method, into the liquid steel.

To solve this problem, the study was performed under laboratory conditions in TsNIITMASH at the following stages:

- (i) the study of influence of aluminum and calcium on the oxygen activity,
- (ii) the study of nonmetallic inclusions in metal samples,
- (iii) comparison of the data obtained and finding a relationship in the $[Ca]-a_{Al}-a_O-(CaO)$ system, and
- (iv) the development of a technique for controlling the inclusion composition.

The heats were performed in a VIAM-100 vacuum induction furnace with a 50-kg crucible, whose lining consisted of fused magnesium oxide (70%) and electrocorundum (30%).

As a charge, we used Armco iron with the composition (wt %) 0.027% C, 0.052% Si, 0.015% Mn, 0.006% S, 0.003% P, 0.012% Cr, 0.013% Ni, 0.026% Al, $\leq 0.002\%$ Ti, 0.009% O, and 0.006% N. As alloying elements, we used Kr0-grade silicon, Mr00 manganese, N1 cathode nickel, FNb66-grade ferroniobium, FVd80 ferrovanadium, and graphite as electrode scrap.

The steel was killed by commercial-purity aluminum. Modification was carried out using lumps of calcium-containing powdered wire. The heat mass was 32 kg.

The degree of oxidation was measured using Celox-Al probes and a Multi-Lab Celox II recording device.

Oxygen activity a_O was calculated by the expression used in the method of probes,

$$\log a_O = 1.36 + 0.0059[E + 0.54(T - 1550) + 0.0002(T - 1550)E], \quad (1)$$

where a_O is the oxygen activity (ppm), E is the emf (mV), and T is the temperature ($^{\circ}C$).

A probe was vertically introduced into the melt through the vacuum lock in the upper part of the furnace. The oxygen activity was measured simultaneously with metal sampling. To exclude wrong measurements, the probe container (quartz glass 10–12 mm in diameter and ~ 100 mm in length) was fixed to a sensor (Fig. 1).

The laboratory heats were carried out using the same technological sequence: melting of a charge in vacuum (pressure 2–6 Pa), pumping of argon to the furnace chamber, the introduction of manganese, holding

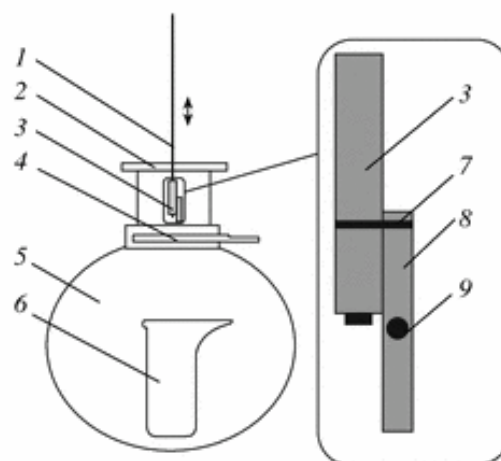


Fig. 1. Scheme of the introduction of an activity meter and a sample container into the melt and the attachment of the sample container to the sensor: (1) rod, (2) vacuum lock, (3) sensor, (4) slide gate, (5) vacuum chamber, (6) crucible, (7) molybdenum wire, (8) sample container, and (9) inlet hole in the sample container.

Table 1. Chemical composition of steel samples (%) taken during experiments

Metal (heat)	C	Si	Mn	S	P	V	Al	Ca
Pure iron (a)	0.010–0.016	0.053–0.057	0.024–0.027	0.006	0.004	–	0.005–0.084	–
Pure iron (b)	0.010–0.015	0.068–0.31	0.034–0.038	0.007	0.005	–	0.038–0.037	Up to 0.0038
Pure iron (c)	<0.01–0.02	0.045–0.072	0.017–0.024	0.006	0.003	–	0.012–0.038	Up to 0.0020
Model alloy (d)	0.012–0.026	0.030–0.073	0.40–0.46	0.040	0.003	–	0.016–0.100	–
Model alloy (e)	0.072–0.082	0.19–0.28	0.061–1.51	0.008	0.006	–	0.014–0.058	Up to 0.0025
Model alloy (f)	0.029–0.036	0.13–0.43	1.20–1.23	0.007	0.004	0.064	0.007–0.011	Up to 0.0045
Model alloy (g)	0.055–0.054	0.31–0.55	1.65–1.69	0.007	0.004	0.065	0.006–0.037	–
Model alloy (h)	0.022–0.031	0.14–0.46	0.29–0.39	0.010–0.011	0.003	0.064	0.009–0.042	Up to 0.0048

of the melt, the measurement of the degree of oxidation and sampling of the initial steel, aluminum killing of the steel, the measurement of the degree of oxidation and sampling, and the sequential introduction of calcium (two to four times per heat). After each addition of calcium-containing materials, from three to five oxygen activity measurements were performed simultaneously with sampling. In this case, the first measurement was performed within 10–25 s after the end of the introduction of calcium, and the next measurements were performed in 2–3 min intervals. The steel temperature in experiments remained constant, 1600°C ($\pm 10^\circ\text{C}$), and the argon pressure in the vacuum chamber was 70 kPa. Eight heats were performed.

Since we studied the influence of only aluminum and calcium on the composition of inclusions and the degree of oxidation of the metallic melt, the oxidation of the steel with atmosphere and slag were excluded (the experiments were performed in vacuum and in the absence of slag) and the sulfur concentration, which (as an impurity) significantly influences the process of modifying oxide nonmetallic inclusions with calcium, was maintained at a certain level (about 0.007%).

Chemical analysis of steel samples (including an analysis of the calcium content) was performed using a Spectro LABS (Germany) multichannel vacuum emission spectrometer according to GOST 18895-97. The carbon and sulfur contents were also determined by IR spectroscopy on a LECO CS-244 (USA) spectroscope.

The content of acid-dissolved aluminum ($[\text{Al}]_{\text{ad}}$) in the experimental steel was analyzed on a Perkin-Elmer 403 (USA) atomic-absorption spectrometer. The resolution of this analysis method was 0.005 abs %.

Qualitative and quantitative electron probe microanalysis of the oxide inclusion composition was performed on a CAMEBAX SX-100 microanalyzer and a JEM 460 LV scanning electron microscope with an INCA-sight (Oxford Instruments) energy dispersive attachment. The inclusion compositions were determined at points. The inclusions were analyzed to find the contents of Ca, Al, Si, Mn, Fe, Mg, and O. A special procedure was used to convert a elemental composition into an oxide composition [2].

The aluminum activity was found from the chemical composition of the steel using information on the first-order interaction parameters.

The chemical compositions of the steels of experimental heats are given in Table 1.

The experimental values of the oxygen activity depending on the aluminum content in the steel are presented in Fig. 2. On the whole, the experimental points are approximated (Fig. 2, curve 1) by the regression equation

$$a_{\text{O}} = 0.4a_{\text{Al}}^{-0.916} \quad (r = 0.84), \quad (2)$$

where a_{O} is the oxygen activity (ppm) and a_{Al} is the aluminum activity (%).

The results demonstrate that the thermodynamic equilibrium between oxygen and aluminum in the experiments was not always achieved and, correspondingly, the metal was overoxidized with oxygen. It

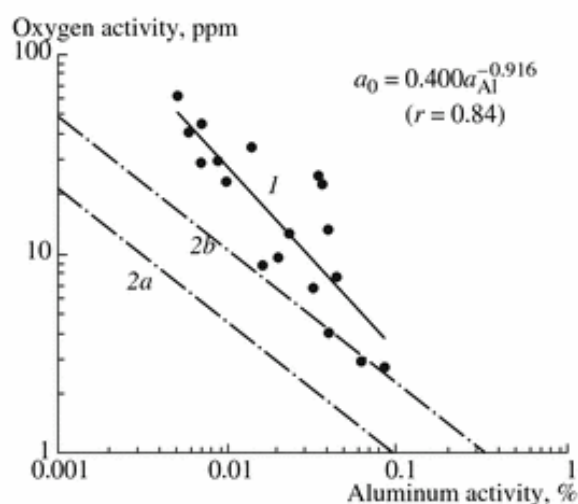


Fig. 2. Effect of aluminum on the oxygen activity in a low-alloy steel: (1) approximating curve; (2a, 2b) the minimum and maximum equilibrium values of a_{O} at $K_{\text{Al}}^{1873} = a_{\text{O}}^3 \times a_{\text{Al}}^2 = 9.54 \times 10^{-15}$ [3] and 1.19×10^{-13} [4], respectively.