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Regulation of carbon and phosphorus content in manganese alloys when processed in an oxidizing gas environment or oxide system

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Регулювання вмісту вуглецю та фосфору в марганцевих сплавах при обробці в окислювальному газовому середовищі або оксидній системі

***Purpose**: Determination of rational methods for reducing the carbon and phosphorus content in manganese alloys. **Research methodology**: Thermodynamic calculations and experimental studies of decarbonization and dephosphorization of a high-phosphorus manganese alloy. **Research materials**: As a high-carbon ferromanganese with a high phosphorus content, a related metal (manganese alloy) obtained during the production of low-phosphorus slag at the Nikopol Ferroalloy Plant was used. Rolling scale was used as an oxidant, with a composition of FeO - 59.5 wt. %; Fe3O4 - 38.9 wt. %. **Research results:** The features of dephosphorization of manganese alloys are considered. According to the adopted "classical" technology, a useful product of dephosphorization of manganese concentrates is manganese slag with a low phosphorus content. Oxidation of phosphorus dissolved in the metal can occur as a result of its interaction with oxygen in the gas phase. The higher the basicity of the manganese slag, the greater the probability of phosphorus oxidation. Given the basicity of the slag, the higher the FeO content in it, the better the conditions for removing phosphorus from the metal. The process of dephosphorization of the associated metal includes the oxidation of phosphorus, the binding of phosphorus oxide into strong compounds (phosphates) and their transition to the slag phase. **Scientific novelty**: The associated metal includes silicon, which has a much higher affinity for oxygen than phosphorus; then, naturally, it will first be oxidized with the formation of silicon oxide with a melting point much higher than the temperature of experimental studies**. Practical significance**: The results obtained show that when oxidizing the associated metal with iron scale at a specific consumption of 114 kg/t of metal, the total degree of silicon extraction was 88.16%, phosphorus - 71.03%. At the same time, the manganese content in the metal decreased by 6.48% due to the recovery of rolling scale.*

***Key words:** manganese alloys, dephosphorization, decarburization, oxidant, rolling scale, ferromanganese, phosphorus oxidation, slag phase, silicon oxidation.*

***Мета:** Визначення раціональних методів зниження вмісту вуглецю та фосфору в марганцевих сплавах. **Методологія дослідження:** Термодинамічні розрахунки та експериментальні дослідження декарбонізації та дефосфорації високофосфористого марганцевого сплаву. **Матеріали дослідження:** Як високовуглецевий феромарганець з високим вмістом фосфору використовувався супутній метал (марганцевий сплав), отриманий при виробництві низькофосфористого шлаку на Нікопольському феросплавному заводі. В якості окиснювача використовувалася прокатна окалина, складом FeO – 59,5 мас. %; Fe_3O_4 – 38,9 мас. %. **Результати дослідження:** Розглянуто особливості дефосфорації марганцевих сплавів. Згідно з прийнятою «класичною» технологією, корисним продуктом дефосфорації марганцевих концентратів є марганцевий шлак з низьким вмістом фосфору. Окислення фосфору, розчиненого в металі, може відбуватися внаслідок його взаємодії з киснем у газовій фазі.Чим вища основність марганцевого шлаку, тим більша ймовірність окислення фосфору. Враховуючи основність шлаку, чим вищий у ньому вміст FeO, тим кращими є умови для видалення фосфору з металу. Процес дефосфорації супутнього металу включає окислення фосфору, зв'язування оксиду фосфору в міцні сполуки (фосфати) та їх перехід у шлакову фазу. **Наукова новизна:** До складу супутнього металу входить кремній, який має набагато вищу спорідненість до кисню, ніж фосфор; тоді, природно, він спочатку буде окислюватися з утворенням оксиду кремнію з температурою плавлення значно вищою, ніж температура експериментальних досліджень. **Практична значимість:** Отримані результати показують, що при окисленні супутнього металу залізною окалиною при питомій витраті 114 кг/т металу, загальний ступінь вилучення кремнію склав 88,16%, фосфору – 71,03%. При цьому вміст марганцю в металі зменшився на 6,48% за рахунок відновлення прокатної окалини.*

***Ключові слова:** марганцеві сплави, дефосфорація, зневуглецювання, окиснювач, прокатна окалина, феромарганець, окислення фосфору, шлакова фаза, окислення кремнію.*

Introduction

The considerable interest of metallurgists in the development of methods for refining manganese alloys is explained by the need to significantly reduce the phosphorus content in alloys and steels, the main source of which is manganese ores. Refining melts obtained on their basis in oxidizing conditions by blowing the melts with gaseous oxygen or by adding solid oxidants is considered ineffective. Under these conditions, part of manganese, an element with a higher chemical affinity for oxygen, can also pass into the slag along with phosphorus. The formed slag based on oxides of these elements, saturated with phosphorus, is a technogenic waste that is difficult to dispose of. A rational solution to problems of this type will allow obtaining an additional amount of steels and alloys, while increasing the coefficient of through-extraction of valuable alloying elements from the starting raw materials with a

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guaranteed low loss. In modem industrial production, special requirements are placed on the consumer characteristics of steel, which largely depend on the composition and undesirable impurities. For most grades of steel, such an impurity is phosphorus, the main source of which is manganese ferroalloys. Smelting ferroalloys with the required phosphorus content is possible only if production is provided with low-phosphorus manganese raw materials. In this regard, the production of ferroalloys standard for phosphorus requires preliminary dephosphorization of concentrates.

Existing methods of refining alloys based on manganese and other elements with a high chemical affinity for oxygen can be divided into several groups according to their physicochemical nature, technological features and type of product obtained.

By the aggregate state of the refining object: refining the alloy in a solid or liquid state to obtain a product - a low-phosphorus manganese alloy and waste, which is a slag saturated with phosphorus compounds.

By the type of main reactions that ensure intensive and fairly complete conversion of phosphorus in a given direction: oxidative, reductive, redox or exchange type.

By the direction of the main refining reaction that ensures the conversion of phosphorus: from the metal to the slag phase in order to obtain a manganese-based metal alloy with a low phosphorus content in it with the achievement of high values of the phosphorus distribution coefficient between the slag and the metal; from the metal to the gas phase by forming its volatile compounds; with phosphorus remaining in the metal phase to obtain low-phosphorus manganese slag.

By the temperature of metal refining: it is most expedient to refine metals and alloys at a temperature that corresponds to the given technological scheme of their production. For example, when refining vanadium slags by blowing with oxygen, the initial physically cold cast iron with a temperature of 1250…1300°C is used. The initial temperature of the accompanying manganese alloy is 1320…1350°C.

By the number of individual stages that solve the corresponding technological tasks by carrying out both reduction and oxidation reactions. With the accumulation of new data, the above classification can be expanded or refined.

1. Relevance of the problem

High-carbon ferromanganese grade FMn70 with 0.35% phosphorus increases the concentration of phosphorus in steel by 3-5 times [1]. In structural steel with a manganese content of 0.5-1%, FMn70 contributes only 0.003-0.005% phosphorus, which is 5-10 times lower than the requirements of the standards. However, in [2] it is shown that in this case, reducing the phosphorus concentration to thousandths of a percent can significantly increase the plastic and fatigue properties of the metal. According to the accepted “classical” technology, the useful product of dephosphorization of manganese concentrates is low-phosphorus manganese slag.

A by-product is an associated alloy with a high phosphorus content, composition, %: Mn 45-55; P 3-4; Si 0.2-0.5; C 3.0-4.0 and other impurities. The formation of an associated high- phosphorus manganese alloy, which has no consumer value, leads to the loss of manganese with this associated alloy, which amounts to 12-22% of the given amount of manganese in an electric furnace with manganese concentrate.

proposed calcium and its alloys as reagents for the depliosphorization of manganese alloys under reducing conditions. However, calcium has a greater affinity for carbon and binds to carbon more readily than to phosphorus, reducing its effectiveness in removing phosphorus. This depliosphorization method is not practical for high-carbon manganese alloys. An increased silicon content in the metal melt reduces the efficiency of its dephosphorization. Therefore, it is necessary' to reduce the silicon content by any conventional method below 0.6% by weight.

2. Analysis of literature data

An increase in the specific phosphorus content (P/Mn) in manganese ore raw materials necessitates an increase in the consumption of low-phosphorus slag to produce ferroalloys with a low phosphorus content. A feature of the electrometallurgical method of dephosphorization of ore and concentrates and one of its main disadvantages is the need to increase the silica content in the slag to 25-26% for a batch process and 29-31% for a continuous process (18-20% in the original concentrate) [3, 4]. An increase in SiO_2 in the slag is necessary to impart high fluid mobility to the slag and create conditions for complete separation by density of the slag and particles of the high-phosphorus accompanying metal. The use of low-phosphorus slag in the smelting of ferromanganese has a negative impact on the quality of the alloy due to the greater amount of silica that is introduced by the slag compared to concentrates, and this leads to an increase in the phosphorus content in the alloy [5]. However, obtaining ferromanganese with <0.55% P and silicomanganese with <0.5% P using only manganese agglomerates is practically impossible.

A reduction in $Si0_2$ is achieved by smelting pig-grade low-phosphorus slag with a high content of Na_2O and K_2O by introducing alkali aluminosilicates into the charge, for example pegmatite (8-10% Na_2O + K_2O; 13-15% Al_2O_3; 70-75% SiO_2) [4, 6, 7]. The authors of [8] proposed calcium and its alloys as reagents for the dephosphorization of manganese alloys under reducing conditions. However, calcium has a greater affinity for carbon and binds to carbon more readily than to phosphorus, reducing its effectiveness in removing phosphorus. This dephosphorization method is not practical for high-carbon manganese alloys. An increased silicon content in the metal melt reduces the efficiency of its dephosphorization. Therefore, it is necessary to reduce the silicon content by any conventional method below 0.6% by weight.

The best results are obtained when the manganese alloy has a silicon content of no more than 0.1%. The greatest efficiency of desiliconization is achieved by introducing oxidizing agents and fluxes into the melt. The oxidizing agent can be in the form of gases (O_2 and CO) or in the form of solid particles of iron and manganese oxides or alkali and alkaline earth metal carbonates, barium oxide. Analysis of thermodynamic data shows that during the dephosphorization of ferromanganese, the formation of gaseous phosphorus or its gaseous compounds PH_3, PC_{13}, PC_{15}, the formation of phosphides of the Ca_3P_2 type under reducing conditions, and under oxidizing conditions of orthophosphates $Ca_3(PO_4)_2$: Na_3PO_4 [9-13].

It is known that reductive dephosphorization occurs most intensively at high temperatures. In addition, this type of dephosphorization is associated with the consumption or formation of very active compounds, such as alkaline earth metal phosphides. Oxidative dephosphorization occurs most intensively at low temperatures and, as a rule, is associated with the consumption of relatively inexpensive, non-scarce materials [14-16]. To dephosphorize manganese alloys, low-melting reagents and mixtures can be used: calcium chloride; sodium chloride; mixtures thereof; sodium chloride and soda and others. Dephosphorization of manganese alloys only with melts of calcium chloride, as well as a mixture of calcium chlonde and sodium chloride, allows one to obtain only a slight reduction in phosphorus content (by 11-25%) [17]. Higher dephosphorization rates were obtained when using low-melting salts with the addition of oxygen-containing components [18-24]. It has been shown that when treated with mixtures of this composition, the degree of dephosphorization of manganese alloys increases, which depends on the amount and ratio of salts used. In parallel with the dephosphorization process, carbon removal occurs, and carbon is removed to a greater extent from alloys with a high phosphorus content. Thus, when processing manganese alloys with molten salts, solid-phase dephosphorization occurs, and the phosphorus content in the alloys decreases by 62-75%, especially when processing mixtures of sodium-containing compounds. Solid-phase dephosphorization technology is of interest not only in terms of producing low-phosphorus manganese alloys, but also for recycling fines obtained during casting, crushing and fractionation of alloys.

Oxidation of phosphorus dissolved in a metal can occur as a result of its interaction with oxygen in the gas phase according to the reaction

$4/5\ [P] + O2_{(g)} = 2/5(P2O5)$ (1)
AG° = -618 000+ 175,0 T,
with oxygen dissolved in the metal
$4/5\ [P] + 2[O] = 2/5(P_2O_5)$ (2)
AG° = -384000+ 170,24 T,
iron oxides contained in the slag
$4/5[P] + 2[FcO] = 2/5(P_2O_5) + 2Fe_{liq.}$ (3)
AG° = -142 000 + 65,48 T.

all cases, the oxidation of phosphorus dissolved in the metal is accompanied by the release of heat. When phosphorus is oxidized by oxygen gas, a significant amount of heat is released. In some processes associated with the processing of phosphorous cast iron (for example, in the Thomas process), the metal is heated due to the heat from the oxidation of phosphorus. An increase in the oxidation of the slag promotes the oxidation of phosphorus, and, conversely, a decrease in the activity of iron oxides in the slag complicates the occurrence of these processes. The presence in the metal of increased amounts of easily oxidized impurities (Si, Mn, C, etc.) complicates the process of phosphorus removal, since these impurities interact with iron oxides and the oxidation of the slag decreases. To remove phosphorus from the metal and retain it in the slag, it is necessary to reduce the activity of $P_2C>5$ in the slag. This is achieved by adding lime (or limestone) to the main slag. The main component of lime, CaO, interacts with P_205, forming strong compounds such as (CaO) (P_2O_5) or (CaO)3 (P_2O5). When metal interacts with slag containing iron and calcium oxides, the following reactions occur:

$2[P] + 5(FeO) + 4(CaO)= 4\ (CaO)\ (P_2O_5) + 5Fe$ (4)
$2[P] + 5(FeO)+ 3(CaO) = 3\ (CaO)\ (P_2O_5) + 5Fe.$ (5)

Analysis of the above reactions shows that for a given slag oxidation, the higher its basicity, the greater the likelihood of phosphorus oxidation. Given the basicity of the slag, the higher the FeO content in it, the better the conditions for removing phosphorus from the metal

Results of experimental studies.

As high-carbon ferromanganese with a high phosphorus content, an associated metal (manganese alloy) was used, obtained during the production of low-phosphorus slag at the Nikopol Ferroalloy Plant. The process of dephosphorization of associated metal involves the oxidation of phosphorus, the binding of phosphorus oxide into strong compounds (phosphates) and their transfer to the slag phase. Mill scale was used as an oxidizing agent (composition, wt.%: FeO - 59.5; Fe_3O_4 - 38.9). The composition of the associated metal includes silicon, which has a significantly higher affinity for oxygen than phosphorus; then, naturally, it will oxidize first to form silicon oxide with a melting point significantly higher than the temperature of experimental studies.

To form a low-melting slag on the surface of the melt (with a melting temperature of less than 1300°C of the ternary system SiO_2-CaO-Al_2O_3 ($Si0_2$ - 62%; CaO - 23%; Al_2O_3 - 15%) additives of lime and alumina were introduced. The amount of additives was determined based on the possible formation of S1O2 as a result of complete oxidation of silicon in the associated metal due to oxygen in the scale.

The process of dephosphorization of associated metal consists of two stages: oxidation of silicon and conversion of its oxide into low-melting slag (first stage); oxidation of phosphorus, binding it into strong phosphates and transferring them to the slag phase (second stage). A sample of the associated metal was melted in an alumina crucible placed in a Tamman furnace. The melt temperature (in the experiments it was 1300°C) was periodically monitored with an immersion thermocouple (BP 5/20). After melting the associated metal, we measured the temperature, removed the resulting slag from its surface and took a metal sample for chemical analysis. Then scale was added in portions to the clean surface of the melt (based on the complete oxidation of all the silicon of the associated metal).

Table 1. Dynamics of changes in the concentrations of silicon and phosphorus in manganese alloy when treated with oxide mixtures

Stage	Material	Metal composition, %		
		Si	P	Mn
Initial sample	Initial high-phosphorus metal	u	1.6	64.60
Sample No. 1	Oxidation of silicon by scale	0.76	1.45	64.71
Sample No. 2	Slag removal CaO, Al2O3 Oxidation of silicon, phosphorus	0.36	1.37	66.86
Sample No. 3	Slag removal. Oxidation of silicon, phosphorus	0.09	0.42	58.23

The specific consumption of scale was 57 kg/t of metal. The specific consumption of slagforming agents (lime and alumina) for the formation of low-melting slag on the surface of the melt was 8.8 kg/t of metal (for lime) and 5.8 kg/t of metal (for alumina). To accelerate the assimilation of additives, the melt was forcibly mixed with a molybdenum rod. After the formation of a liquid-mobile slag phase, a metal sample was taken for chemical analysis. Then all the slag was removed from the surface of the melt (by freezing it onto a steel rod). In order to determine the completeness of silicon oxidation, as well as the possibility of phosphorus oxidation, scale was added in portions to the surface of the melt with a specific consumption of 57 kg/t of metal. After assimilating the scale, CaO and Al_2O_3 were added. The specific consumption of lime and alumina was, respectively, 8.8 kg/t of metal and 5.8 kg/t of metal. To accelerate the assimilation of additives, the melt was forcibly mixed with a molybdenum rod. A metal sample was taken for chemical analysis. Below are the results of the analysis of metal samples (Table 1).

The results obtained show that during the oxidation of associated metal with iron scale with a specific consumption of 114 kg/t of metal, the total degree of removal of: silicon was 88.16%, phosphorus 71.03%. At the same time, the manganese content in the metal decreased by 6.48% due to the reduction of mill scale.

Conclusion

The possibility of dephosphorizalion of carbonaceous high-phosphorus associated ferromanganese, formed during the electrometallurgical dephosphorization of manganese ore, with additives of a mixture consisting of mill scale, lime and alumina is shown.

As high-carbon ferromanganese with a high phosphorus content, an associated metal (manganese alloy) was used, obtained during the production of low-phosphorus slag at the Nikopol Ferroalloy Plant. The process of dephosphorization of associated metal involves the oxidation of phosphorus, the binding of phosphorus oxide into strong compounds (phosphates) and their transfer to the slag phase. Mill scale was used as an oxidizing agent (composition, wt.%: FeO - 59.5; Fe_3O_4 - 38.9). The composition of the associated metal includes silicon, which has a significantly higher affinity for oxygen than phosphorus; then, naturally, it will oxidize first to form silicon oxide with a melting point significantly higher than the temperature of experimental studies.

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