



Protective coatings for the graphite facing in calcium–aluminothermal processes

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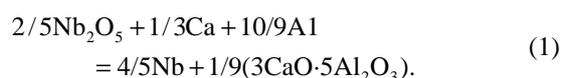
Abstract. Results of pilot plant and full scale studies on the elaboration of high temperature protective coatings for graphite in the process of the reductive melting of niobium pentoxide and NbAl alloys production are presented. Graphite has found extensive application in high temperature processes due to its unique thermophysical properties. However, in these conditions it reacts very easily with air oxygen, liquid metals, oxides, and fluorides. As a result, porous graphite gets impregnated with liquid metal; besides, carbonization of melt and formation of a layer of metal carbides on graphite surface takes place. The layer of metal carbides has a low adhesion with graphite and a major part of them is transferred to the melt. The graphite facing of a furnace is quickly destructed. To avoid these harmful processes, special plasma sprayed protective coatings are used. In this work various coatings (Al₂O₃, Al₂O₃-CaO, ZrO₂, NbC, Al₂O₃ + Nb, ZrO₂ + Nb, NbC + Nb, Nb) were tested in pilot plant and industrial conditions. It was established that the best durability of the protective coating on graphite can be achieved in the case of its three-layer composition: Nb (0.07 mm) + NbC (0.4 mm) + Nb (0.3 mm). After industrial melting of 500–2000 kg ingots about 80% of this coating on the belt of the shaft furnace had preserved. Plasma sprayed protective coatings on the graphite facing extended its operation time, reduced the carbon content in the metal ingots to 0.02% (mass), and slag carbon content to 0.05–0.15% (mass).

Key words: chemical technology, metallotherapy, graphite facing, protective coatings.

INTRODUCTION

Large capacities of the production of metallic niobium, alloys of niobium with aluminium, and master alloys with rare earths using out-of-furnace reduction of metal oxides were concentrated in Estonia at the chemical–metallurgical company in Sillamäe (today Silmet JSC) in the 1980s. The technological solution introduced at Silmet JSC was relatively simple, environmentally friendly, and economically efficient, and it has several advantages over other technologies for the production of metallic niobium, its alloys with aluminium, and master alloys with rare earths [1].

At Silmet JSC two reducers – metallic powdered aluminium and chips of metallic calcium – are used. The reduction process corresponds to the following prevailing reaction:



As a result of the reduction reaction (1) two products are formed: an alloy of niobium with aluminium (3–5% Al) and slag (calcium–aluminium oxides), which have quite a clear separative interfacial area. Due to the high specific exothermic heat of this reaction (2640 kJ/kg of raw mixture) it is possible to carry out the process out of furnace, i.e. without external heat transfer [2]. High temperatures (up to 2700 °C) require implementation of the process in facilities (pot, shaft furnace) with a graphite facing.

The main disadvantage of graphite is its elevated chemical activity at high temperatures. Graphite reacts not only with air oxygen but also with molten metal as well as with slags [3,4].

The solubility of carbon in liquid aluminium is extremely small [5]: at 800 °C it is only 0.1% (mass). However, in the conditions of reaction (1), in the

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presence of high concentrations of carbon, carbide Al_4C_3 in the form of a slag shell on the spots on the graphite facing with a high thermal stress is built up. Similarly, during the reduction reaction (1), calcium carbide CaC_2 is formed on the interface with graphite [5]. The Gibbs free energy of niobium carbide formation is less than that of calcium carbide formation. With increasing temperatures ($>2000^\circ C$) the difference in free energies increases [6]. Reduced to melt, liquid niobium actively reacts with calcium carbide, forming thermodynamically stable niobium carbide NbC on the surface of the graphite facing of the furnace:



Metals of the 5th group (V, Nb, Ta) form carbaceous compounds much more easily than, for example, metals of the 4th group (Ti, Zr, Hf). This can be explained by less free energy of their carbides (MeC) formation as well as by higher diffusivity of carbon in the liquid metals of the 5th group [7].

Protection of graphite with special protective layers enables prolonging its lifetime and producing purer metals and alloys. The best protectors are presumably the high-melting-point oxides, carbides, and borides of W, Mo, Nb, Ti, Zr, etc. [3,4]. For example, plasma sprayed coating of magnesium zirconate with a thickness of 0.3 mm increases the lifetime of graphite pots for uranium and plutonium melting 3 times and totally prevents pollution of melts with carbon [8].

The problem of graphite facing protection needs to be solved for each metal individually. There are only a few examples of the production and application of protective coatings for graphite in the literature. The authors point to weak adhesion of the coating with the graphite base [9]. For example, the adhesive force of plasma sprayed coatings of Al_2O_3 and ZrO_2 with graphite does not exceed 5 MPa [10]. The lattice energy between the carbon atoms in the graphite crystal lattice is extremely high. To activate its surface for achieving good adhesion it is necessary to preheat the graphite significantly (to $700\text{--}1000^\circ C$) before spraying [11]. Application of various underlayers on graphite enables to improve adhesion. Plasma sprayed Al_2O_3 onto presilicided or preboron-silicided graphite has already an adhesive force of $7\text{--}17$ MPa [10,11]. However, this method is not suitable for the treatment of large graphite facing surfaces of reductive furnaces as it requires a special thermal treatment facility [12]. Some authors [13] have suggested that for adhesion improvement the surface of graphite should be made rougher (average microasperity $10\text{--}75 \mu m$). It has been established that plasma spraying of refractory carbide forming metals such as W, Mo, Nb, Ta, Zr, etc. with preheating the graphite basis to $>1000^\circ C$ leads to reactions of metals with graphite and formation of carbides with an adhesive force of about 3.5 MPa. X-ray spectroscopy of protective coatings of metallic niobium

has indicated that starting from the graphite basis preheating up to $800^\circ C$ typical of Nb_2C and NbC lines can be observed. At the preheating temperature of $1200^\circ C$ the content of carbon in the coating makes up already 8.25% [9,14]. Interesting results were obtained in [15], where the protective properties of $ZrB_2\text{--}MoSi_2$ coating were improved by SiO_2 nanopowder admixtures.

The aim of the present study was to test various coatings (Al_2O_3 , $Al_2O_3\cdot CaO$, ZrO_2 , NbC, $Al_2O_3 + Nb$, $ZrO_2 + Nb$, NbC + Nb, Nb) for the shaft furnace shown in our previous paper ([16], fig. 2) for graphite facing protection.

MATERIALS AND METHODS

The thickness of the different coatings listed above was 0.3–0.5 mm. Initial materials were dried, ground, granulated, and classified to get powders with particle diameters of $40\text{--}120 \mu m$. The surface of the graphite facing was precleaned by overblowing with corundum (Al_2O_3 , size 1.5–2.0 mm). For pilot plant tests and full scale operation Russian plasma spraying equipment (plasmotrons) UMP-5 and UPU-3 with argon, nitrogen, and their mixtures was used [17,18].

The phase composition of the coatings was studied with an X-ray diffractometer DRON-2 with monochromatic copper K_α irradiation at a wavelength of 1.5418 Å. The microstructure of the contact zone of the graphite coating was investigated by metallographic analysis using microscopes MIM-7 and MIM-9. The strength of the contact zone was determined on special equipment PMT-3 [17,18]. In the process of the elaboration and selection of appropriate coatings the following requirements were established:

- the coating must have sufficient plasticity and mechanical–thermal resistance
- thermophysical parameters of coating and graphite must be close
- coating must have minimum chemical impact on liquid metal and slag
- adhesion with graphite must exceed adhesion with metal and slag
- possibility of multiple restoration of coating
- in the case of decomposition and shelling material must not pollute liquid metal
- coating material should be relatively cheap and available.

RESULTS AND DISCUSSION

Pilot plant experiments

At the first stage of studies under pilot plant conditions various compositions of coatings (Al_2O_3 , $Al_2O_3\cdot CaO$, ZrO_2 , NbC, $Al_2O_3 + Nb$, $ZrO_2 + Nb$, NbC + Nb, Nb)

for internal surfaces of graphite pots were tested in the process of reductive meltings with a capacity of 10 kg of ingot. The results of the tests revealed that over 50% of all types of coatings, except metallic niobium, were decomposed after melting and were separated from graphite. The reason is that in the process of reductive melting liquid slag reacts actively with oxide type coatings Al_2O_3 and $\text{Al}_2\text{O}_3\cdot\text{CaO}$ (Fig. 1). It was also found that the test coupons from the surface of the coating did not have a clear separation area, which indicates a high velocity of diffusion and mixing of phases in the contact zone. Prespraying on the graphite underlayer of niobium aluminide Nb_3Al did not improve the durability of the main coating of Al_2O_3 . Underlayers of aluminides usually improve adhesion [6], but after reductive melting the underlayer of aluminide was separated from graphite together with the Al_2O_3 coating. The covering of the outer surface of the coating of metallic niobium with lime suspension $\text{Ca}(\text{OH})_2$ helps to preserve the inner layer of niobium and leads to positive results. In this case the skin (shell) of the slag separates easily after melting from the coating surface and the zone of erosive destruction is much smaller.

On the coatings of niobium it is possible to follow self-compaction due to the thermomechanical impact of the liquid metal and slag (Fig. 2). Metallographically self-compaction is proved by the lack of porosity and laminated structure characteristic of plasma coatings. A coating that was in the zone of the maximum temperature and under the impact of liquid metal and slag, suffered from a reduced number of pores due to

compaction and from adding up of some ‘fresh niobium’ from the melt.

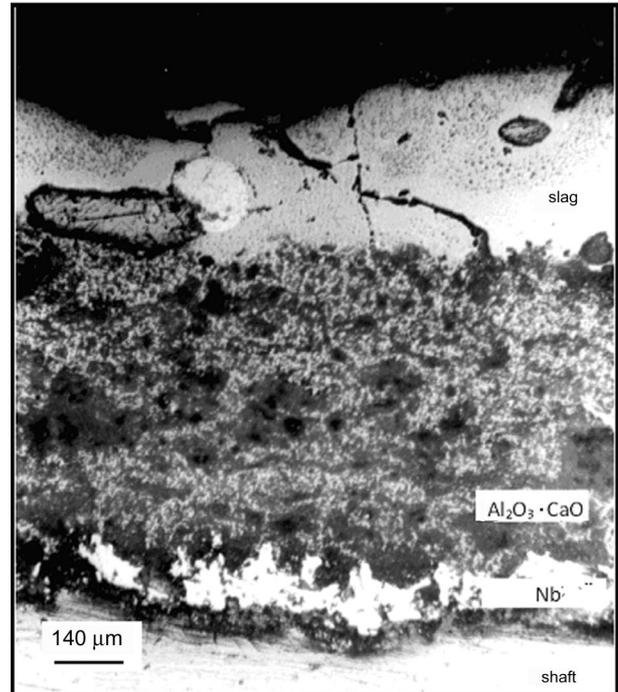


Fig. 1. General picture of the microstructure of plasma sprayed niobium coatings.

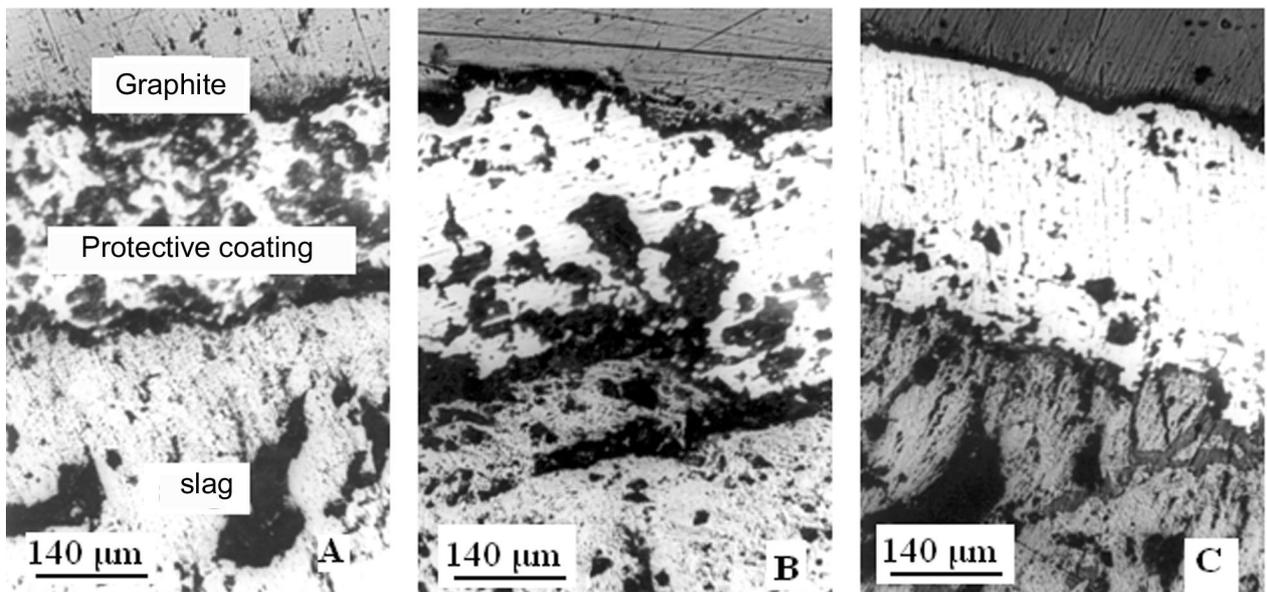


Fig. 2. Microstructure of plasma sprayed niobium coatings: A – after spraying the coating; B, C – after melting; B – middle zone of the melt; C – bottom zone of the melt.

Studies on the microstrength of coatings indicated that niobium coatings reacted with the graphite facing during the reductive melting. The rate of carbon diffusion into the coating was high, and the coating was quickly carbonized during the relatively short melting process. The curves in Fig. 3 indicate that the microstrength increases towards the interface of graphite and the coating. This means that due to the formation of niobium carbides the elasticity of the structure decreases and its fragility increases. Use of an intermediary layer of niobium aluminide Nb_3Al as a barrier significantly decelerates the process of saturation of the niobium coating with carbon. The Nb_3Al layer has a strong impact on the operation properties of the metallic niobium coating, prevents carbonization, and maintains its plastic properties. However, this intermediary layer does not promote adhesion with graphite. Repeated thermal cycles of reductive melting weaken adhesion and lead to the separation of the coating from graphite. Pilot tests with niobium coatings of different thickness showed that the thin coatings and the coatings with a thickness over 1.1 mm have the highest durability (Fig. 4).

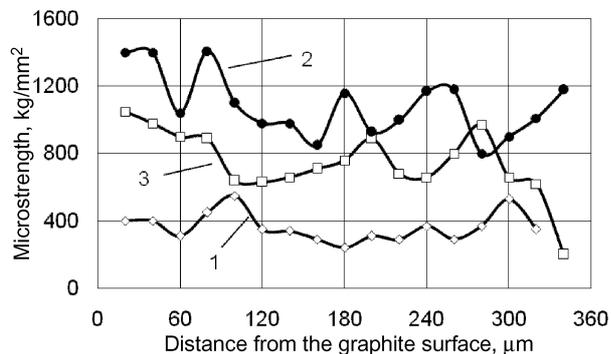


Fig. 3. Distribution of the microstrength in a plasma sprayed coating: 1 – without underlayer; 2 – with an underlayer of Nb_3Al ; 3 – impregnated with ‘liquid glass’.

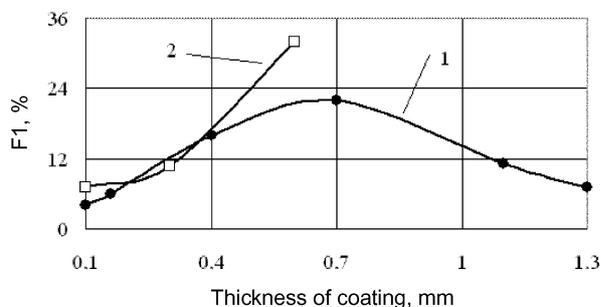


Fig. 4. Relative area of the destructed niobium coating versus the thickness of the layer: 1 – without underlayer; 2 – with an underlayer of Nb_3Al .

The thermal stress created in the conditions of metallothermal melting is responsible for the decomposition of protective coatings. Decomposition takes place on the spots of the concentration of thermal stress – in corners, on edges, etc. According to Kindzheri [7], the maximum thermal stress on the surface of an elastic body at thermal shock is to a large extent determined by the dimensions of the body, especially by its thickness. With increasing thickness the intensity of thermal stress rises. This is why the thin coatings are more thermo- and erosion-resistant in the conditions of melting than thick coatings. On the other hand, these coatings are easily carbonized, which leads to their fragility and destruction during the following meltings. In thicker coatings the thermal stress is created mainly on the surface of their contact with their base (graphite); the outer layer remains at the same time without any special stress, less carbonized, and more plastic [19].

It was established by the authors that the spraying of an intermediary layer of Nb_3Al onto graphite did not improve the adhesion of the main protective niobium coating with graphite. Use of relatively thick coatings with an intermediary layer of Nb_3Al led to the destruction and separation of the coating in a large area of the contact surface (Fig. 4).

In the practice of metallurgy ‘liquid glass’ ($R_2O \cdot nSiO_2$) is often used to increase the resistance of the structure of the coating to thermal shock. This method was also tested by the authors in the process of niobium production from its oxide. It was found that impregnation with ‘liquid glass’ significantly reduced the degree of carbonization of the main coating and increased its thermal durability. Impregnation with ‘liquid glass’ led to a decrease in the microstrength and a higher elasticity of the structure of the niobium coating (Fig. 3). After the first melting the degree of the destruction of the coatings of this type was only 7%.

Full scale experiments

The protective coatings elaborated under pilot plant conditions were tested in an industrial shaft furnace of Silmet JSC, described in our previous publication ([16], fig. 2). The most thermally sensitive graphite surfaces (belt, separation plates of metal receiving hopper) were selected for tests. Technological parameters of plasma spraying of coatings were studied in the production process of $NbAl$ alloys with the mass of ingots per melting of 500–2000 kg.

The impact of the three most important parameters of plasma spraying was studied:

I – current of arc of plasmatron, A

Q – flow rate of the plasma creating gas (nitrogen), m^3/s

L – distance of spraying, m.

In the planning of the experiments the method of Box–Wilson and fractional factor’s plan 2^{3-1} were used [20]. The matrix of the plan and results of the experiments are given in Table 1. The values of the factors in Table 1 are encoded; the actual values are given in parentheses.

Adequacy of the regression equation of the general form of $y = x_0 + b_1x_1 + b_2x_2 + b_3x_3$ was checked by the Fisher criterion. The model has an adequacy of 5% of significance level. Significance of the coefficients of regression b_i was estimated by the Student criterion building up of reliable interval b_i , corresponding to 95% of reliable probability.

The regression equation, including the significance of the coefficients in the coding system, is as follows:

$$F = 17.4 - 2.4I - 5.2Q + 9.4L, \quad (3)$$

where F – durability of the coatings, %.

According to equation (3), the distance of plasma spraying has the strongest impact on the durability of the coatings. Even the smallest changes in the distance due to the vibration of the plasma spraying gun have a deteriorative impact on the quality of the coating in certain places (spots) of the surface. These spots are the primary sources of erosive destruction of coatings on a graphite facing [17,18].

Satisfactory results were achieved by using plasma sprayed niobium coatings with a precover as a ‘lubricant’ of $\text{Ca}(\text{OH})_2$ on the graphite separation plates of the metal receiving hopper. After melting, more than 50–60% of the surface protective coating was preserved. Good results were obtained applying the regime of parameters characterized in Table 1 (run No. 1) and using an underlayer of ‘liquid glass’.

Tests with protective coatings for the belt of the furnace, the second thermally and chemically most sensitive zone, revealed that the degree of the destruction of the protective coating after each melting did not exceed 3–5%.

Quite unexpectedly, it was established that the coating on the belt was strengthened after each melting, probably due to a positive thermomechanical impact of liquid metal. Application of a three-layer coating on graphite – Nb (0.07 mm) + NbC (0.4 mm) + Nb (0.3 mm) – gave

Table 1. Matrix of experimental plan 2^{3-1}

| No. | X_0 | I, A | $Q, \text{m}^3/\text{s}$ | L, m | Durability of coating | | |
|-------|-------|---------|--------------------------|---------------|-----------------------|-------|---------|
| | | | | | $F, \%$ | | |
| | | | | | I | II | Average |
| 1 | +1 | -1(400) | -1(1.2) | +1(0.14) | 38.47 | 30.21 | 34.34 |
| 2 | +1 | +1(480) | +1(12.5) | +1(0.14) | 18.55 | 19.90 | 19.23 |
| 3 | +1 | -1(400) | +1(1.68) | -1(0.10) | 4.07 | 6.41 | 5.24 |
| 4 | +1 | +1(480) | -1(1.2) | -1(0.10) | 12.37 | 9.11 | 10.76 |
| b_i | 17.39 | -2.40 | -5.16 | 9.39 | | | |

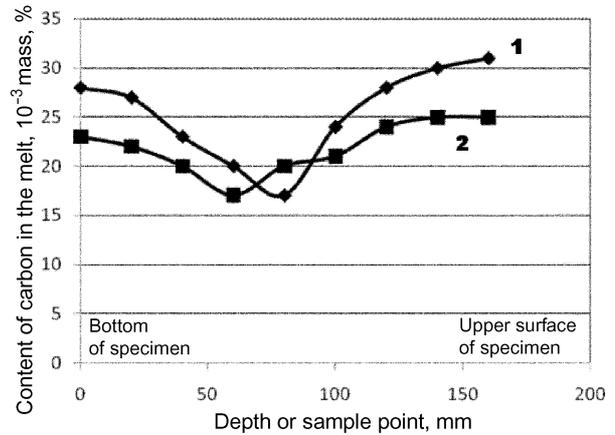


Fig. 5. Carbon content of the melt on the bottom of the specimen and on its upper surface: 1 – without protective coating; 2 – with a 3-layer coating.

the best results. After melting about 80% of this coating surface was preserved.

Because of the protective coatings on graphite the melt of liquid metal was found less carbonized. Application of this three-layer coating enabled to reduce the transfer of carbon into the melt (curve No. 2 in Fig. 5). The coupons were taken by mechanical drilling from the ingot with 20 mm spacing across the whole height from both sides. This fact is very important from the point of view of the quality of the ingots, as Silmet JSC guarantees that the maximum content of carbon in niobium ingots is 0.05% (mass) for grade B and 0.01% for grade A [1].

For the determination of the degree of erosion of the graphite facing analysis of the slag was carried out. To get the corresponding slag sample the shaft furnace at Silmet JSC was operated in this case without any protective coating on graphite. Analysis on the scanning electron microscope ZEISS EVO-50 revealed quite a high content (up to 3% mass) of carbon in the slag [16]. This means that without a protective coating the graphite facing is quickly destructed, ‘burning out’ of graphite takes place, and the liberated gases CO_2 and CO turn the graphite porous and fragile. Semchenko et al. [21] pointed out that the more perfect the crystalline structure of graphite, the higher the starting temperature of its oxidation. However, if the graphite surface is not protected, at 1300°C it will ‘burn out’ very intensively, up to a depth of 10–15 mm.

CONCLUSIONS

After testing various coatings for graphite (Al_2O_3 , $\text{Al}_2\text{O}_3\text{-CaO}$, ZrO_2 , NbC, $\text{Al}_2\text{O}_3 + \text{Nb}$, $\text{ZrO}_2 + \text{Nb}$, NbC + Nb, Nb) in pilot plant and industrial conditions

it was established that the best durability of the protective layer can be achieved in the case of a three-layer coating on graphite: Nb (0.07 mm) + NbC (0.4 mm) + Nb (0.3 mm). After melting of 500–2000 kg of ingots about 80% of this coating on the belt of the shaft furnace was preserved. Use of an intermediary layer of niobium aluminide Nb₃Al as a barrier significantly decelerated the process of saturation of the niobium coating with carbon. A Nb₃Al layer had a strong impact on the operation properties of the metallic niobium coating, prevented carbonization, and maintained its plastic properties. However, this intermediary layer did not promote adhesion with graphite. Impregnation of the niobium coating with ‘liquid glass’ with the following drying significantly reduced its degree of carbonization and increased thermal durability. ‘Liquid glass’ led to a decrease in microstrength and rigidity and to a higher elasticity of the structure of the niobium coating. Results of repeated meltings certified that after each successive melting the destruction zones of the protective coating did not enlarge more than 3–5%, the coating was strengthened in the melting process, and its adhesion with graphite improved as a result of the thermomechanical impact of liquid metal. Plasma spraying of protective coatings on the graphite facing extended their operation time and reduced the carbon content in metal ingots to 0.02% (mass) and the slag carbon content to 0.05–0.15% (mass).

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Grafiitvooderdise kaitsekihid kaltsium-alumotermilises protsessis

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Tänu oma suurepärasele termofüüsikalistele omadustele on grafiit leidnud laialdast kasutamist kõrgtemperatuursetes protsessides. Grafiidil on suur temperatuuripüsivus ja mehaaniline tugevus, kuid kõrgetel temperatuuridel (>1000 °C) reageerib ta aktiivselt õhuhapniku, sulametallide, oksiidide ning fluoriididega. Tulemusena seguneb poorne grafiit sulametalliga ja teisalt toimub metallisulami karboniseerumine ning metallikarbiidide kihi moodustumine grafiidi pinnal. Karbiidide kihil on grafiidiga nõrk adhesioon ja suur osa nendest kandub üle sulametalli ning šlaki massi. Sulatusahju grafiitvooderdis laguneb ruttu. Selle vältimiseks kasutatakse plasmameetodil grafiidi pinnale kantud kaitsekihte. Artiklis on toodud erinevate kaitsekihtide (Al_2O_3 , $\text{Al}_2\text{O}_3\text{-CaO}$, ZrO_2 , NbC , $\text{Al}_2\text{O}_3 + \text{Nb}$, $\text{ZrO}_2 + \text{Nb}$, $\text{NbC} + \text{Nb}$, Nb) katsetamise tulemused piloot- ja tööstuslikes tingimustes nioobiumpentoksiidi taandamisega NbAl-sulamiks. Kaitsekihi suurim püsivus grafiidi pinnal on saavutatud kolmekihilise struktuuri Nb (0,07 mm) + NbC (0,4 mm) + Nb (0,3 mm) puhul. 500–2000 kg NbAl-sulami tootmisel tööstuslikus šahtahjus on pärast sulatusprotsessi ahju “vöö” kohal säilinud ~80% kaitsekihist. Plasmameetodil grafiidile kantud kaitsekiht võimaldab vähendada süsiniku sisaldust metallisulamis kuni 0,02%-ni (mass) ja šlakis 0,05–0,15%-ni (mass).