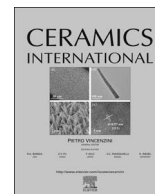




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Corrosion behavior of silicon nitride bonded silicon carbide refractory material by molten copper and copper slag



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ABSTRACT

Silicon Carbide refractories may be used in contact with copper and copper slags. The object for the investigation of corrosion by the melt of copper and the copper slag was SiC siphon block of the slag collector in the runner of the cathode shaft furnace. It is exposed to the permanent flow of copper with small amount of slag. Slag on the surface of the melt is stopped by siphon block and it suffers the most extensive corrosion wear, but the same time it is a good object for investigation, because different parts of this plate are exposed to intensive corrosion by different corrosive agents.

General observations show, that the corrosion of $\text{Si}_3\text{N}_4\text{-SiC}$ by slag is sufficiently more extensive and the wear is about 3 mm per month, while the wear by the flowing copper is below 1 mm per month.

The observation of the cross section of the exposed $\text{Si}_3\text{N}_4\text{-SiC}$ plate on macro level shows 4–5 zones of different color. Microstructural observations show almost no changes of the material in direct contact with molten copper without exposure of air, that suggests slow dissolution of Silicon Carbide and Silicon Nitride in the flowing melt of copper (physical dissolution in case of permanent removal of reactants). The most severe wear of $\text{Si}_3\text{N}_4\text{-SiC}$ refractory is by slag, because the chemical erosion takes place.

1. Introduction

Silicon Nitride bonded Silicon Carbide is known for its corrosion resistance and relative chemical inertness. It is used in Aluminium industry due to resistance to cryolite [1] and in WTE processes [2]. Not much is known on corrosion resistance of $\text{Si}_3\text{N}_4\text{-SiC}$ materials to Copper and Copper slag, although $\text{Si}_3\text{N}_4\text{-SiC}$ refractories are used in shaft furnaces for the melting of cathode Copper [3].

Laboratory corrosion tests may be divided to static cup tests and dynamic rod tests. Static and dynamic laboratory corrosion tests are carried out from 2 to 50 h, but provide limited information, especially in case of relatively slow interactions and high corrosion resistance of materials. At copper plants sometimes “industrial” testing of corrosion resistance is performed. Such kind of testing, although time consuming, opens way to understanding of the processes, that take place at service of materials.

2. Materials and methods

The chemical composition of the materials was determined by standard wet chemistry methods (SiC, Si, SiO_2 , CaO, Fe_2O_3 , Al_2O_3) and by XRD (SiC, Si, SiO_2 , Si_3N_4), the determination of Nitrogen and

Oxygen was performed using the LECO method. The value of SiC content (the first digits in Table 1) was determined by wet chemistry and by XRD (given in brackets). The contents of silicon nitride and silicon oxide were calculated from values of Nitrogen and Oxygen, determined by LECO method and by XRD (given in Table 1 in brackets), α - and β - modifications of silicon nitride were determined by XRD. Si, CaO, Fe_2O_3 , Al_2O_3 concentrations were determined by wet chemistry methods. SiO_2 concentration was calculated from oxygen value, received by subtraction of oxygen from calcium, aluminium and iron oxides. Structures were analyzed at Tescan X-max SEM with EDX Oxford instruments microprobe analyzer.

The object for the investigation of corrosion by the melt of copper and the copper slag was the $\text{Si}_3\text{N}_4\text{-SiC}$ block. The properties of investigated $\text{Si}_3\text{N}_4\text{-SiC}$ refractory material before testing are in Table 1. Open porosity and apparent density were determined according to ISO 5017:2013-01, cold crushing strength and bending strength were determined according to ISO 10059-2:2003 and ISO 5014-97, pore size distribution (Fig. 1) was determined according to ASTM C-493-98.

$\text{Si}_3\text{N}_4\text{-SiC}$ siphon block (3 on Fig. 2a) was used as a slag collector in the runner of the cathode shaft furnace for the re melting of cathode copper [4] (Fig. 2a). It is exposed to the permanent flow of copper

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Table 1
Composition and properties of investigated Si_3N_4 -SiC material.

SiC, %	72,0 (75,2)
Si_3N_4 , %	27,8 (24,4)
α - Si_3N_4 , %	12,0
β - Si_3N_4 , %	12,4
C free, %	0,15
Si free, %	0,21
SiO_2 , %	–
Al_2O_3 , %	0,23
CaO, %	0,25
Fe_2O_3 , %	0,58 (0,4)
Porosity, %	16,6
Cold crushing strength, MPa	160
Modulus of rupture, MPa	35

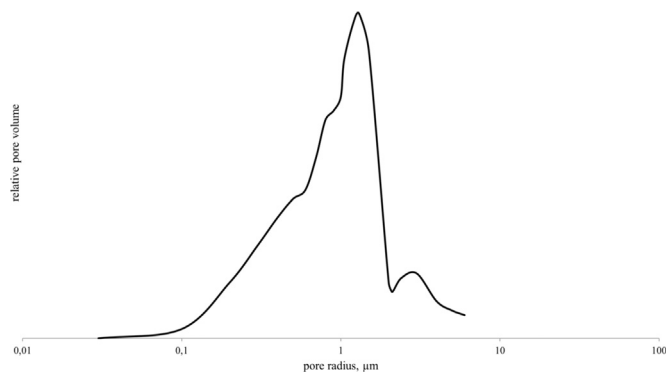


Fig. 1. Pore size distribution in investigated Si_3N_4 -SiC material before testing.

(0,016–0,03 m/s) with thin layer of slag (2) on the copper (1). Slag on the surface of the melt (phosphates and silicates of copper, zinc and aluminium) is stopped by siphon block (3 on Fig. 2a), that suffers the most extensive corrosion wear (Fig. 2b). The lower edge of block, dipped in the melt of flowing Copper, is subjected to erosion and corrosion by pure Copper. We consider it a good object for investigation of corrosion, at least for rough estimation of corrosion resistance, because different parts of this block are exposed to intensive corrosion by different corrosive agents.

3. Results and discussion

3.1. Changes of the geometry of the block after exposure – visual observations and macro structural changes

The corrosion of Si_3N_4 -SiC by slag (Fig. 3, Table 2) is more extensive (the wear is about 3 mm per month), the wear of material by the flowing copper is below 1 mm per month. The cross section of the exposed Si_3N_4 -SiC plate on macro level shows 4–5 zones of different color (Fig. 3), that may show on intrinsic changes in Si_3N_4 -



Fig. 3. The cross section of Si_3N_4 -SiC siphon block after testing.

SiC in zones, not subjected directly to interaction with Copper and Copper slag. Visual observations show almost no changes of the material in direct contact with molten copper (without exposure of oxygen from air), that suggests slow dissolution of Silicon Carbide and Silicon Nitride in the flowing melt of copper (physical or chemical dissolution in case of permanent removal of reactants). The same may be said about the “back” part of the plate, dipped in the copper (not exposed to permanent flow) – there is no wear and there are almost no observable changes in the color of Si_3N_4 -SiC material.

Visual observations of the cut surfaces and simple measurements of the change of geometry had shown (Table 1), that the width of the plate above the level of metal and slag increase by 2 mm (zone 1 on Fig. 3), that takes place most likely due to oxidation. The cut surface is pale-gray, that also may be the evidence of oxidation. The expansion by 5 mm (~7%) of the part, dipped in copper, suggests inner changes in the material.

The most severe wear of Si_3N_4 -SiC refractory is by slag, because the

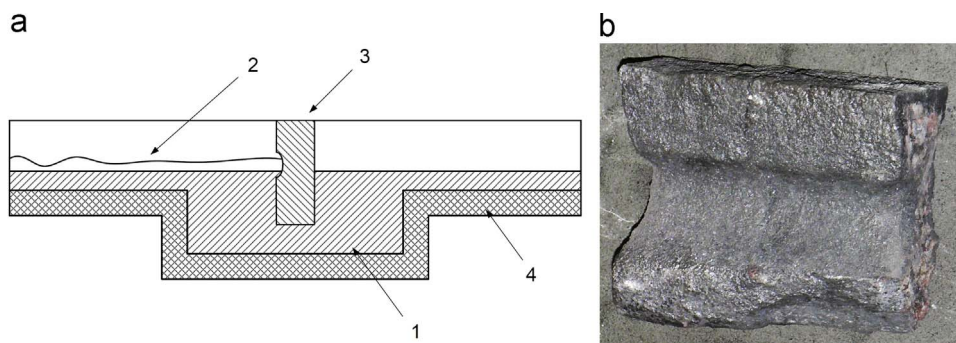


Fig. 2. a The sketch of siphon block of the slag collector in the runner, 1- copper, 2-slag, 3 - Si_3N_4 -SiC siphon block, 4 – refractory; b. general view of Si_3N_4 -SiC siphon block after 8 months of exposure.

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