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COMPARISON OF COPPER AND GRAPHITE CRUCIBLES FOR SI EXTRACTION FROM TIO_2 - SIO_2 SYSTEM AT PLASMA-ARC HEATING

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Abstract. Plasma arc recovery melting of the quartz-leucoxene concentrate was investigated. Experiments were made in laboratory DC plasma arc furnace in copper and graphite crucibles. The best results were reached in a cold copper crucible. The temperature field of a pool was calculated in hot graphite and cold copper crucibles. It was shown that in graphite crucible diameter of anode spot is more, and material temperature in spot is less, than in copper crucible. This fact was suggested as a reason of the worst refinement in graphite crucible.

Keywords: DC plasma arc, anode spot, reducing, leucoxene, silicon, rutil.

1. Introduction

A considerable part of titanic raw materials in Russia is presented by oil-bearing quartz-leucoxene sandstones of the Yarega field, the Komi Republic [1]. These raw materials are nonconventional for the titanic industry and now are not processed. Thermal plasma technology emerges in processing of ilmenite [2, 3], and as a new frontier in processing of quartz-leucoxene concentrate. Plasma arc carbothermal melting of concentrate during which quartz reduces to SiO and evaporates from the fusion is the cornerstone of the process [4, 5]. Now the research of various options of hardware and technological optimization of the process is proceeded. In this work the results of melting in copper water-cooled and graphite radiation cooled crucibles were compared.

2. Experiment technique

Graphite was used as conventional reducer in plasma reactors. Melting was carried out in the laboratory DC plasma arc furnace under atmospheric pressure. The furnace includes a copper cylindrical water-cooled chamber with a diameter 100 mm and 250 mm high, closed from above by arch through which a copper cooled holder with a graphite electrode was entered. The electrode diameter was 50 mm. The electrode was connected to a negative pole of a source of power supply, the camera - to a positive. Two options of experiments are executed. In the first option melting was carried out directly in the camera which lower part carried out a role of a copper crucible. At the same time diameter of a bathtub was 100 mm, depth ≈ 30 mm. In the second option the melting was conducted in graphite crucible installed in the lower part of the camera. External diameter of the crucible was 90 mm, internal - 75 mm, depth - 43 mm, height on an external surface - 62 mm. Between the camera and the crucible there was a side gap 5 mm wide.

Element	**Si**	**Ti**	**Al**	**Fe**	**O**
Conc. mass.%	17,0	32,8	1,65	2,07	≈ 46

Table 1. The chemical composition of quartz-leucoxene concentrate on the main components.

The electrode was supplied with electric drive and had a possibility of axial movement. The electric arc which current was regulated by ballast resistance was excited by making contact between the electrode and the bottom of the camera or crucible. The electrode had an internal axial channel with a diameter 20 mm. The furnace charge which consisted of graphite powder and concentrate was fed via axial channels in the holder and the electrode to the area of anode spot. Argon as transporting and plasma-forming gas was fed together with furnace charge to the arc column via the channel in the electrode. The furnace charge melted and a bathtub of fusion formed under the influence of the arc. The length of the arc gap was 1 - 3 cm. The gaseous products including SiO and CO were sent to the heat exchanger and then to the filter for collecting condensate. The chemical composition of quartz - leucoxene concentrate is given in Table 1. The dimension of particles was 1 mm. According to the X-ray analysis titanium and silicon in concentrate are present as TiO_2 and SiO_2 oxides. Concentrations of TiO_2 and SiO_2 were 54,7 and 36,4 % respectively. The dimension of graphite particles was 0,1 - 0,2 mm when using a graphite crucible and 0,3 - 0,5 mm when using a copper crucible. Content of carbon in furnace charge in case of graphite crucible was 20,7 % and 11,5 % in case of copper. Ingots in crucibles had a sufficient conductivity. It was possible to excite the arc by making contact between electrode and ingot.

3. Results and discussion

The condensed products of melting were ingot of synthetic rutile and powder on the basis of silicon oxide. The powder was collected from the camera walls, the heat exchanger and the filter. Besides powder and ingot gaseous product formed. It is possible to assume that it was CO. The specific electric power consumption, calculated for graphite crucible as the relation of arc power to feed rate, $Q = P_d/V$, approximately twice exceeded the similar parameter for copper: 39,2 against 20,4 MJ/kg. Despite it, the extent of silicon removal from concentrate

$$\eta = \frac{m_0 - m}{m_{\ 0}} \cdot 100\%, \qquad (1)$$

where m_0 and m - silicon percentage in concentrate and in ingot, for a graphite crucible was 1,4 times less than for copper: 35 and 48% respectively [6]. Lower extent of silicon removal in graphite crucible is caused by lower temperature of fusion. The temperature of fusion was calculated using the mathematical model of bathtub heating [7]:

$$T(r) = 0.282\frac{P_t}{\lambda \cdot r_0}(\phi(r) - \phi(r_s)) + T_s, \qquad (2)$$

where $T(r)$ - temperature field of bathtub surface, r_o - radius of anode spot, r_s - radius of crucible, T_s - temperature of material near the crucible wall, λ - heat conductivity of material, P_t - heat power that supports the temperature field $T(r)$, $\phi(r) = exp(-\frac{r^2}{2 \cdot r_0^2}) \cdot I_0(\frac{r^2}{2 \cdot r_0^2})$, I_o - Bessel's function imaginary argument. Anode spot is restricted by isotherm T_o at which intensive evaporation of material begins [8] ($T_o \approx 2000\ K$ for SiO evaporation [5]). The equation 2 can be transformed into:

$$T_0 = 0.282\frac{P_t}{\lambda \cdot r_0}(\phi(r_0) - \phi(r_s)) + T_s. \qquad (3)$$

For copper crucible $T_s \approx 400K$, for graphite crucible T_s can be solved from equation:

$$P_t = 2\pi \cdot r_s^2 \cdot \epsilon \cdot \sigma \cdot (T_s^4 - T_k^4), \qquad (4)$$

where $\epsilon \approx 0.8$ - the blackness of material, σ - Stefan's - Boltzmann's constant, $T_k = 293\ K$ - the temperature of water-cooled chamber. The heat balance of bathtub can be written as:

$$P_s = P_t + P_r + P_{ch}, \qquad (5)$$

where, P_s - the total heat power, transferred to bathtub from the arc (for discussed laboratory furnace $P_s \approx 0.5P_d$, where P_d is the total arc power), P_{ch} - the heat of evaporation ($P_{ch} = Q_{ch} \cdot V \cdot \eta$, where $Q_{ch} = 8.9\ MJ/kg$ - specific heat of evaporation, V - charge feed rate), P_r - radiation losses from bathtub surface. It was consumed that radiation losses are absent beneath the electrode. In this case

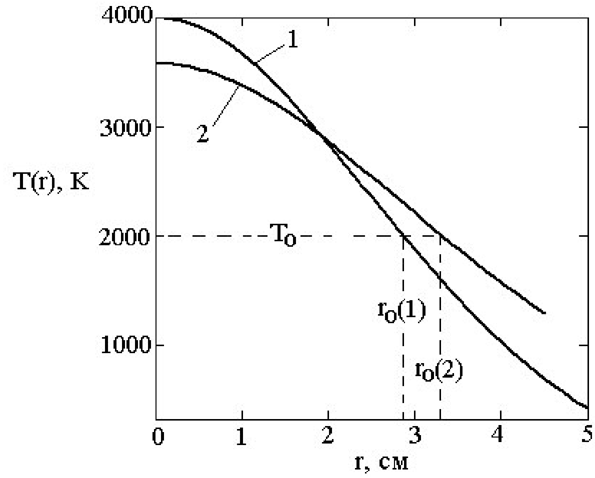

Figure 1. Calculated temperature fields of a bathtub surface. r - radial distance from the center; 1 - copper crucible; 2 - graphite crucible; T_0 - the temperature of the reduction start.

$$P_r = 2\pi \cdot \sigma \cdot \epsilon \int_{r_e}^{r_s} r(T(r)^4 - T_k^4)dr, \qquad (6)$$

where r_e - electrode radius.

Equations (2)–(6) were solved together as a system against P_t, P_r, $T(r)$, T_s and r_o. In Figure 1 the calculated temperature fields of a bathtub surfaces in copper and graphite crucibles are presented. Calculation showed that the peripheral temperature of fusion is higher in hot graphite crucible, than in copper. However this temperature does not affect on reduction velocity because it remains less than the temperature of the reduction start: $T_o = 2000\ K$ [5]. At the same time increase in peripheral temperature leads to considerable losses of radiation heat from bathtub surface and P_t power at the same time decreases. Decrease in concentration of a thermal stream and temperature of material in the region of anode spot is the result of it. In the case of graphite crucible the axial temperature of a bathtub is $T_c = 3580\ K$, whereas in the case of the copper crucible $T_c = 4000\ K$. It was the reason of lower extent of silicon removal in graphite crucible.

4. Conclusion

The efficiency of silicon removal in a hot graphite crucible can be lower than in water-cooled copper. The reason of it is low axial temperature in hot graphite crucible due to high radiation losses from the periphery of the bathtub.

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