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Technical Note

Kinetics of high-temperature thermal treatment of boehmite-based alumina in vacuum to produce pure alumina

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ABSTRACT

The last stage of the hydrothermal oxidation of aluminum is based on high-temperature treatment of boehmite-based alumina in vacuum furnace to produce pure alumina. This process of alumina purification is studied experimentally with the use of a thin highly-porous layer of the material at different temperatures and durations of thermal treatment. The theoretical analysis of experimental data indicates that the high-temperature thermal purification of alumina is well described by the Arrhenius-type kinetic equation. Both the activation energy and pre-exponential factor of this process as a whole are retrieved from the laboratory experiments. A comparison with the experimental data showed that theoretical predictions based on the simplest one-stage sublimation model are acceptable for potential engineering estimates of the remaining impurities at rather different thermal regimes of high-temperature treatment. The latter is expected to be useful to optimize the industrial production of pure alumina.

1. Introduction

The present study is concerned with the last stage of the dispersed pure alumina production using a specific but widely used procedure of the hydrothermal oxidation of aluminum powder [1–9]. In the beginning of the process, the aluminum powder is fully oxidized during few seconds in water steam at temperature about 570 K. The product of this oxidation is boehmite, AlO(OH), in the form of single crystals with sizes from 10 to 200 nm, and the primary crystals are then agglomerated into the particles with the size about 10 μm. The complete process including the described first stage was realized in experimental plant with the use of a continuous flow reactor [10]. The next stage of the alumina production is a thermal treatment of boehmite at first in muffle furnace at 870 K for crystallized water removal and then in vacuum furnace at about 1800 K for α-Al_2O_3 obtaining. We are focused on the last stage of high-temperature treatment of the material which is alumina containing some impurities.

It should be noted that α-Al_2O_3 is widely used due to its advantages as compared to other phases of the same substance [11,12]. The unique thermal, mechanical, electrical, and near-infrared optical properties of porous alumina ceramics are also important for diverse engineering applications [13–21]. As an example, alumina particles are widely used in advanced technologies such as thermal spraying [22–30] and even in experimental studies of steam explosion phenomenon as applied to possible severe accident in nuclear reactors [31,32]. Therefore, the thermal treatment of boehmite used to produce pure alumina continues to attract an attention of researchers over the world [33–35].

The particular objective of the present study is two-fold: (1) to provide the experimental data for the high-temperature treatment in the laboratory vacuum installation and (2) to suggest an approximate theoretical model for thermal kinetics confirmed by experimental data for the remaining impurities. It is expected that the model developed will be useful to choose appropriate regimes of a similar high-temperature treatment in the industrial applications.

2. The experimental procedure and some results of measurements

The vacuum furnace designed at the Joint Institute for High Temperatures has been used for the high-temperature treatment of dispersed boehmite to obtain chemically pure α-Al_2O_3. The general view of the laboratory installation is presented in Fig. 1. The nominal power of the furnace is equal to 100 kW and the maximum temperature which can be reached in the furnace is about

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Nomenclature

A	pre-exponential factor
d	diameter of single cylinder
E	activation energy
p	pressure
R	universal gas constant
t	time
T	temperature

Greek symbols

ζ	logarithmic parameter of impurity
ξ	relative fraction of impurity

Subscripts

a	activation
f	final
i,j	numbers of experiments
0	initial

Fig. 1. General view of laboratory installation: 1 – control terminal, 2 – vacuum furnace, 3 – regulator of furnace power; 4 – water cooling system; 5 – vacuum system.

2300 K. The regulation of furnace power from 10 to 100 kW makes possible the experiments at various temperatures. The high vacuum in the furnace is an important property of the installation.

The design of the vacuum furnace with the molybdenum crucible of volume about 29 l includes a thermal insulation which prevents unfavorable heat losses as shown schematically in Fig. 2. It is interesting to look at the fragment of a dispersed layer of thickness $H \approx 7$ mm using a close-up image presented in Fig. 3. The material pellets look like randomly oriented cylinders, and the porosity of this thin dispersed layer is rather high. The latter is an important basis for the choice of a kinetic-type theoretical model of high-temperature purification.

The chemical impurities to be removed (or minimized) during the high-temperature treatment are specified in Table 1. The last column of this table shows that Fe is the main component of initial chemical impurities. Therefore, we will focus on more reliable data for Fe-containing impurities.

A representative series of laboratory experiments was specially chosen to include a wide range of the main parameters of thermal treatment. The measured time variations of temperature and pressure during these experiments are presented in Fig. 4. The standard tungsten-tungsten-rhenium thermocouple characterized by the maximum error about ±5 K at $T < 1300$ K and ±10 K at temperatures up to $T = 2500$ K was used in the measurements. One can see in Fig. 4a that this error is sufficiently small for all the temperature regimes under consideration. The Edwards pressure sensor WRG-S-NW25 was used to control the gas pressure. It goes without saying that pressure measurements shown in Fig. 4b confirm the high vacuum conditions supported during the experiments.

The impurities (including all the components) before and after the high-temperature thermal treatment in the above described vacuum furnace are presented in Table 2. The chemical analysis was carried out using a special laboratory technique based on inductively coupled plasma mass spectrometry (ICP MS) with a preliminary dissolution of the sample [36]. Three small samples were examined in every case with the use of the iCAP6500 Duo Thermo Scientific device. These measurements were very accurate, but a considerable discrepancy between the results for three samples in the same experiment were sometimes observed because of natural differences in local impurities. A relative error in concentration of impurities increased at small values of ξ defined as a ratio of the dimensional concentration to that before the experiment. It is difficult to estimate this error, but the result for experiment 4 seems to be not quite reliable. The latter statement is based on a comparison of several additional experiments characterized by low final impurities. Perhaps, this result is explained