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Purification of hexafluorosilicate of ammonium with fractional desublimation

R.R. Mamedov, F.A. Voroshilov
Tomsk Polytechnic University, Lenin Avenue, 30, Tomsk, Russia, 634050

Development of non-traditional technologies to produce cheap poly –Si "electronic" and "solar" quality is an urgent modern requirement. These technologies include a method of obtaining with fluoride technology [1]. It consists of the following operations: 106luorinetion of original siliceous raw materials, sublimation separation of fluoroproducts into fractions, fine purification of fluorosilicate of ammonium from impurities, obtaining of silicon oxide and its subsequent recovery. Sublimation can not clean $(NH_4)_2SiF_6$ from impurities of most metals and non-metals. The admixture of boron being present in the form of oxygen compounds of boron with sublimation purification can not be not removed. For the production of silicon of "solar" quality it is necessary to reduce the boron content to < 0.001% and phosphorus to <0.002 %, so the stage of fine cleaning is required.

There occurs formation of borate of ammonium NH_4BF_4, which sublimes in the temperature range from 266 – 363 ° C in the fluorination of feedstock, and is sublimed approximately in the same range $(NH_4)_2SiF_6$ (250 – 330 ° C). It is assumed that a significant reduction in the concentration of boron in the fluorosilicate of ammonium being cleaned can be achieved in the fractional desublimination [2].

To test this hypothesis there was assembled a unit consisting of three hermetically interconnected aluminum cups, two of which are heated with the heaters, and the third is cooled with water. Each of them has a volume of 0.8 dm3. The top of the cups are covered with lids for thermocouple. To reduce heat transfer between the compartments and the environment the whole installation was insulated. The temperature control in the zones of heating and fixing temperature is done with the help of Oven TRM138. Data from the 6 channels of the process (2 for each volume) are transmitted through the interface 485 to a computer for recording.

Hexafluorosilicate of ammonium being sublimated from the first volume enters the second volume, where the temperature is kept somewhat lower than the sublimation temperature. A part of the product is condensed, and a part of the gas flow passes into the third volume, where the complete condensation occurs. We conducted a series of experiments in which the temperature in the first volume was maintained at 350 ^oC and the temperature in the second volume was changed in the range of 150 – 250 ^oC. The preliminary experiments showed that adjusting the temperature in the second volume it is possible to reduce the concentration of boron to the value of 0.005%. Experiments on the determination of optimal conditions for purification from impurities of hexafluorosilicate of ammonium will continue.

References:

[1] The technology of synthetic silicon oxide // www.ftortechnology.ru
[2] An apparatus for processing of fluorine ammonium silicon products // Patent 2233694