**A. D. MEKHTIYEV, ZH. D. ZHOLDUBAYEVA, B. D. ISSIN, E. G. OGOLTSOVA, A. V. YURCHENKO, D. K. ISSIN**

ISSN 0543-5846
METABK 55(1) 787-789 (2016)
UDC – UDK 661.68:542.6:546.99=111

# METHOD OF SILICON FILTER REFINING FROM HARMFUL IMPURITIES

Received – Prispjelo: 2015-06-03
Accepted – Prihvaćeno: 2016-02-03
Preliminary Note – Prethodno priopćenje

In the article there are considered the types of filters used for silicon refinement, the possibilities of mechanical separation of inclusions when the melt is through the filter, the efficiency of silicon refinement from impurities. There are also considered the advantages of bulk granular filters which consist of the lumpy or granulated elements. There are described the methods of obtaining filtering elements, the functions executed by the filters depending on their type. There are presented the analysis results obtained in filter refinement of silicon which show the impact of different filters materials on the content of impurities.

*Key words:* silicon, filter, filtering, filtering elements, from harmful impurities

## INTRODUCTION

Returning to the matter of the silicon value in our life it is possible to note once again the areas of its application important for a person: as a semiconductor which is used in solar power engineering, in various-purpose instruments; as a vital mineral for a human body, and silicon nanoparticles are used in treatment of cancer diseases for what nanomicrospheres can be used with success in photo-thermal ablation of tumor tissues, as it was shown in the studies in vitro on the cells of breast cancer [1]. Nanomicrospheres represent one-layer spherical nanoparticles consisting of a dielectric silicon sphere ($SiO_2$). The whole technology is based on that curious fact that silicon in the form of nanometer scale particles, unlike larger lumps, is completely recycled by a human body like the silicon acid containing in food.

The greatest application has technical silicon which is also called metallurgical silicon. It is used in metallurgy as an alloying component, a deoxidant and a reducer, for manufacturing electric heating elements and production of acid-proof materials, etc. [2].

If needed, technical silicon is subjected to the further processing for the purpose of deleting impurities from it and obtaining a high-pure product.

The improvement of the technical silicon quality depends directly on the level of its refinement from impurities. At this the known technologies of refinement not always provide complex refinement from the dissolved and chemically bound impurities, they are not always ecologically safe (for example, chlorination). Therefore searching for the new processes of technical silicon refinement combining an extra technical efficiency and ecological safety seems to be topical.

The analysis of the world tendency shows the increased metallurgists' interest in the filter method, its broad application in production of castings, as well as in large metallurgy, for example, in case of continuous pouring of metals. In foundry production of the European industrially developed countries and the USA more than 50 % of all production is made using the filter method of refinement [3].

The use of the existing modern extra furnace methods of metal refinement requires, as a rule, great capital costs for their implementation. The filter method of metal melts refinement, without conceding to the known methods of the refining ability, possesses a number of distinctive features and technological advantages:

– firstly, when filtering the entire volume of liquid metal is exposed to refinement sequentially (at this the process can be carried out directly when pouring metal from the furnace in the ladle, when filling metal in a crystallizer, a mold or a casting mold);
– secondly, the simplicity of construction of the filter and technology of filtering.

The filter method of refinement does not require: capital costs for acquisition of the expensive equipment; preparation of special materials (for example, synthetic slags, fluxing agents, fine powders, etc.); additional floor spaces; it provides stable deleting of nonmetallic and gas inclusions, harmful impurities. At this the filter method permits to refine a metal melt both

A.D. Mekhtiev, Zh.D. Zholdubayeva, D.K Issin, E.G. Ogoltsova, Karaganda State Technical University, Karaganda, Kazakhstan
A.V. Yurchenko, National Research Tomsk Polytechnik University, Tomsk, Russia
B.D. Issin, L.N. Gumilyov Eurasian National University, Astana, Kazakhstan

from primary and secondary inclusions that are formed by the time of filtering, and when filling the filtered metal directly in any form there is actually removed the phenomenon of secondary oxidation.

## METHODOLOGY OF STUDYING LIQUID SILICON FILTERING TECHNOLOGY

The object of studying served metallurgical silicon which is was unrefined and refined by the oxygen purge of the Ushtobe Silicon Plant, which chemical composition is presented in Table 1.

Table 1 **Chemical composition of unrefined and refined technical silicon / *wt %***

| Technical silicon variant | Si | Fe | Al | Ca |
|---|---|---|---|---|
| Unrefined | 98,0 | 0,8 | 0,8 | 0,5 |
| Refined | 99,5 | 0,3 | 0,25 | 0,04 |

For carrying out laboratory experiments for studying the impact of technical silicon filtering for the pollution with harmful impurities, there was designed with use of the computer program “Compass” and manufactured the filtering equipment given in Figure 1 consisting of three main parts: melting crucible 1 (with a hole in the bottom closed by a stopper) mounted directly on the filtering section A which in turn was precisely placed on the metal receiver B [4].

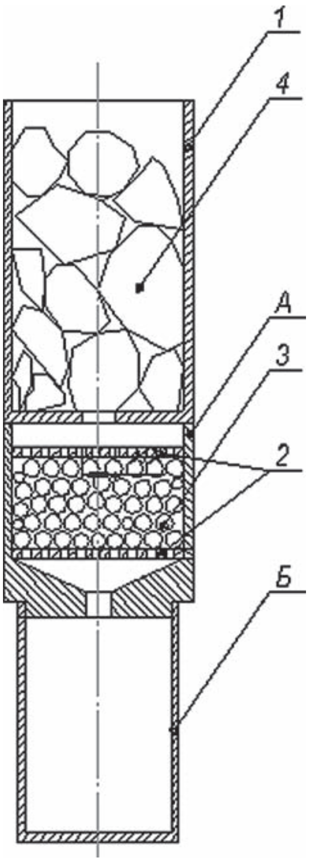


**Figure 1** Filtering equipment assembled: 1 - crucible, 2 - grids, top and bottom, 3 – filtering elements, 4 - charge, 5–metal receiver

Section A is assembled in the following order: on the funnel bottom there is placed flameproof grid 2 onto which there are filled in granules 3 and grid 2 is fined on top. The assembled filtering equipment is placed directly in Tamman’s furnace. The amount of the furnace charge loaded in crucible 1 is defined by the metal receiver B capacity. Filtering elements 3 are made of flameproof materials in the form of granules.

When melting silicon and achieving the required temperature there is carried out the process of filtering the melt by raising the stopper of the melting crucible.

The filtering elements were made of fractional $SiO_2$ and in the form of MgO granules, by means of balling flameproof powders on the dish-shaped granulator (Figure 2).

As the binder there was used a water solution of liquid glass (density of 1,25 ± 0,01 g/cm$^3$) which is not scarce and inexpensive material providing the needed properties to the filtering elements regarding their heat resistance and mechanical durability.

The obtained granules of the required size after preliminary drying in the air, were tempered in the muffle furnace at the temperature 1 100…1 200 K.

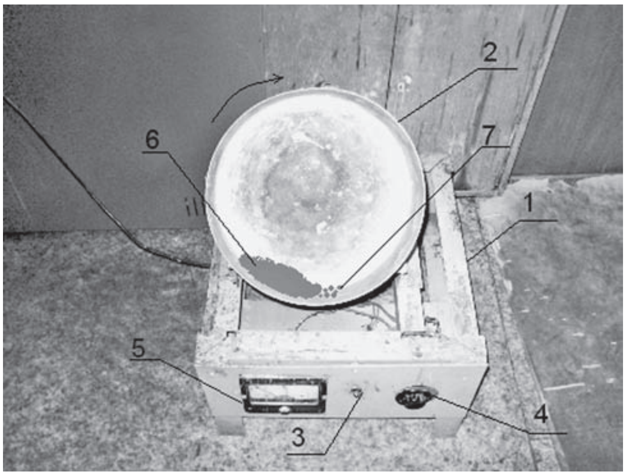


**Figure 2** General view of the dish-shaped granulator: 1 – casing; 2 – dish; 3 – switch; 4 – rotation speed regulator; 5 – voltmeter; 6 – flameproof material powder; 7 – granules

## THE RESULTS OF STUDYING THE FILTER MATERIAL EFFECT ON IMPURITIES CONTENT IN SILICON

The efficiency of the filter refinement was studied on the example of filtering unrefined and refined (blown by oxygen) silicon.

The comparative chemical analysis of unrefined silicon (Table 2) shows the lowering of the content of some impurities [5].

In the filter refinement of the initial unrefined silicon with the use of a $SiO_2$ filter there is observed decreasing the content of Al, Sa and Mg, but at this the content of other impurities remains unchangeable (Figure 3).

Table 2 **Chemical composition of unrefined silicon before and after filtering / wt %**

| Silicon variant | Filter material | Si | Fe | Al | Ca | Mg |
|---|---|---|---|---|---|---|
| Unfiltered | - | 98,0 | 0,8 | 0,8 | 0,5 | 0,03 |
| Filtered | $SiO_2$ | - | 0,8 | 0,3 | 0,05 | 0,01 |
| | MgO | - | 0,8 | 0,4 | 0,5 | 0,03 |

In the filter refinement with the use of a magnesite filter made of there is observed only decreasing the content of aluminum whereas the content of other impurities remains unchangeable.

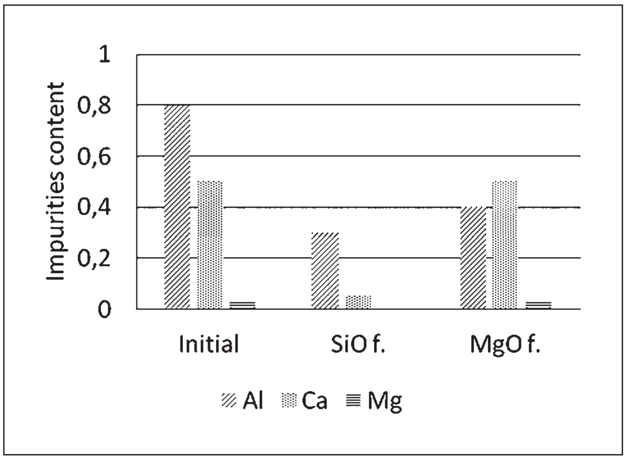


**Figure 3** Impurities content changing in unrefined silicon depending on the filter material

The comparative chemical analysis of refined (oxygen purge) silicon (Table 3) also shows decreasing the content of some impurities.

Table 3 **Chemical composition of refined silicon before and after filtering / wt %**

| Silicon | Filter material | Si | Fe | Al | Ca | Mg | Na |
|---|---|---|---|---|---|---|---|
| Unfiltered | - | 99,5 | 0,2 | 0,1 | 0,02 | 0,07 | 0,06 |
| Filtered | $SiO_2$ | - | 0,2 | 0,06 | 0,01 | 0,04 | 0,01 |
| | MgO | - | 0,2 | 0,07 | 0,02 | 0,07 | 0,06 |

So, in the filter refinement of the initial refined silicon through a quartzite filter the content of aluminum, calcium, magnesium and sodium decreases.

And when using a filter made of magnesite, as well as when filtering unrefined silicon, there is observed only decreasing the content of aluminum.

From the obtained results it follows that a quartzite filter provides the greatest efficiency of purification of liquid silicon (Figure 4).

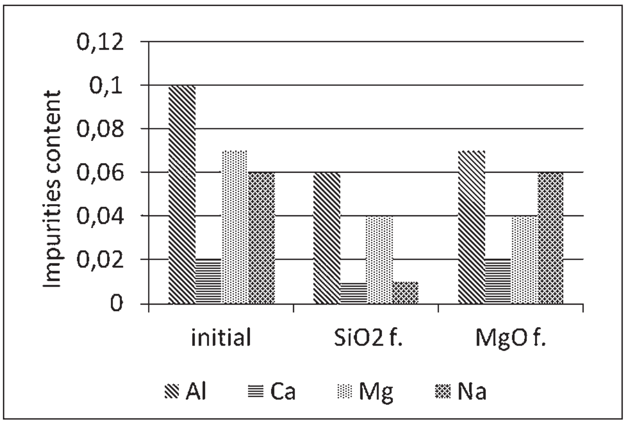


**Figure 4** Impurities content changing in refined silicon depending on the filter material

## CONCLUSIONS

In Table 4 there is shown the extent of the filter refinement of silicon when using a $SiO_2$ filter.

Table 4 **Changing the content of impurities in technical silicon when filtering through a quartzite filter / wt %**

| Varian number | Silicon variant | Al | Ca | Mg | Na |
|---|---|---|---|---|---|
| 1 | Unrefined filtered | 62,5 | 90,0 | 66,6 | - |
| 2 | Refined filtered | 40,0 | 50,0 | 42,8 | 83,3 |

Apparently, a quartzite filter provides decreasing the content of some impurities by 40 – 90 %, including aluminum by 40 – 63 %, calcium by 50 – 90 %, magnesium by 43 – 67 % and sodium by 83 %. Thus when filtering unrefined silicon, there is achieved a higher refining effect (63 – 90 %) than when filtering refined silicon (40 – 83 %).

## REFERENCES

[1] www. Nanonewsnet. ru
[2] Chernyih A.E., Zelberg B.I. Proizvodstvo kremniya. - Irkutsk:Izd. «MANEB» , 2004. –P. 255.
[3] Ten Je.B., Bibikov A.M., Grishin V.M., Kimanov B.M. Upravlenie formirovaniem kachestvennyh otlivok // Litejnoe proizvodstvo. (2003) 5, 5-8.
[4] Kimanov B.M. Ocenka faktorov osazhdenija na fil'tre vzveshennyh v rasplave nemetallicheskih vkljuchenij // Trudy 3-j mezhdunarodnoj Kazahstanskoj metallurgicheskoj konf. Temirtau: KGIU.- 2010.-P. 103 – 106.
[5] Zholdubayeva Zh.D. Razrabotka i issledovanie protsessa filtratsionnogo rafinirovaniya tehnicheskogo kremniya s tselyu povyisheniya ego kachestva: dissertacija na soiskanie uchenoj stepeni Doktora PhD - Almatyi, 2014. - P. 122.

**Note:** The translation of the N. M. Drag, Karaganda, Kazakhstan