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INVESTIGATION OF THE LOWER OXIDE OF SILICON SiO

M. S. Beletskiy and M. B. Rapoport  
 Presented by Acad D. S. Belyankin, 6 Apr 1950

The importance of the lower volatile oxide of silicon, SiO, in electrothermic metallurgical processes is mentioned in the text of the article. Potential applications of results obtained in the study of this compound are (1) condensation of silicon monoxide and (2) subsequent oxidation to silicon dioxide, as a method of depositing a refractory coating or of hardening and waterproofing a surface under treatment. A table is appended.

Considerable interest has been devoted in recent years to studying the role of SiO in the reduction of silicon dioxide. Gaseous SiO is formed as an intermediate substance in a number of production processes including: ferroalloys, silicon, steel products (1), and refractories (2) as well as in silicothermic processes. Moreover, the importance of SiO is well established in the electrothermic process for producing aluminum-silicon alloys from natural aluminosilicates (3), where, due to its comparatively high volatility, SiO exerts an appreciable influence on the final composition of the alloy, altering the ratio of the two elements in the alloy in favor of aluminum in spite of the latter's relatively higher vapor tension. Industrial experience shows that in single- and triple-phase furnaces, 2.5% and 7.6% of aluminum and 15% and 22.5% of silicon, respectively are lost through evaporation.

The development of electrothermic procedures and other high temperature processes in which SiO<sub>2</sub> is reduced, as well as several physicochemical and physical investigations, have dispelled former doubts concerning the formation of SiO in the gaseous state in the course of these processes. Examination of the molecular spectrum of this compound has been conclusive proof (4) of that.

However, no conclusive answer has been given as yet to the problem of whether SiO occurs in the solid state or in condensed form prior to decomposing according to the following scheme:  $2SiO \rightarrow SiO_2 + Si$ , thus forming an equimolecular mixture.

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Attempts to prove the presence of  $\text{SiO}$  in the solid state have, in the majority of cases, been unsuccessful, and investigations of condensed substances actually formed have often proven them to be highly dispersed mixtures of silicon dioxide and silicon.

A considerable step forward along this line was the work done by P. V. Gel'd (5). He produced  $\text{SiO}$  by artificial means in vacuum, measured its vapor tension by the molecular discharge method, studied several properties of this compound, and determined its thermodynamic characteristics. But in spite of this, up to now, the constants of the crystal lattice (the determination of which can give definite proof of the presence of this compound in the solid state), have not been reliably determined.

Two attempts have been previously made at X-ray analysis of  $\text{SiO}$ . Investigating specimens in which he assumed the presence of  $\text{SiO}$ , Baumann (6) established, on the basis of their interplanar distances, that the specimens consisted of mixtures of silicon and cristobalite, and in some cases silicon carbide. Inuzuka has reported (7) that a  $\text{SiO}$  preparation which he investigated had a cubical crystal lattice with the constant  $a$  equals 6.4 Å. The basic cell contained 8  $\text{SiO}$  molecules, and the spatial group belonged to the group  $T_h^c$ .

Beletskiy and Rapoport made X-ray analyses of white formations obtained from the condensation zone of "charge vapors" of an industrial furnace for fusing silicon. The X-ray picture, obtained in filtered iron radiation, had considerable background on which the intensive interference lines were visible. A correction was made for X-ray absorption by the specimens.

The results of these analyses are given in the appended table. They indicate the presence of a mixture of substances with average values of their  $a_1$  equal to 5.41 Å and  $a_2$  equal to 4.34 Å. Substances having such constants would be silicon and silicon carbide.

Be assuming that the white substance was a single compound, this substance by X-ray study was found to have a cubical crystal lattice constant  $a$  equals 6.36 Å, this figure conforming closely to Inuzuka's. By recalculating Baumann's values for the interplanar distances on the basis of the most intensive lines, Beletskiy and Rapoport, with the aid of their own indexes from the appended table, determined the average value for the crystal lattice constant as equal to 6.35 Å; this figure satisfactorily conformed to Inuzuka's data as well as to the data cited above.

From these figures, Beletskiy and Rapoport concluded that Inuzuka investigated a compound which was not  $\text{SiO}$ , but rather a mixture of  $\text{Si}$  and  $\text{SiC}$ ; in other words, approximately the same mixture which was studied by Baumann.

Completely different results were obtained by the Soviet authors in the investigation of preparations obtained as a result of the reduction of  $\text{SiO}_2$  by carbon or silicon at 1,800° or higher in vacuum, or on the reduction of a mixture of  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$  under the same conditions. Several of the properties of the resulting compound were shown to be very similar to those described by Gel'd. Beletskiy and Rapoport's preparation was a yellowish-brown condensate, and isotropic substance with a refraction index, determined by O. I. Arakelyan, of 1.92-1.94. Its density, established with a pycnometer, was  $\rho$  equals 2.13. By X-ray investigation, its cubical crystal lattice constant was found to be  $a$  equals 5.16 Å. No other type of interference being observed on the X-ray picture, it was assumed that this compound was obtained in a pure state.

Based on the figure 2.13 for the density, Beletskiy and Rapoport calculated that with four molecules in the basic cell the molecular weight of the compound is 44.3, which is very close to the theoretical value for  $\text{SiO}$ .

The crystal lattice constant determined by Beletskiy and Rapoport is a specific characteristic of  $\text{SiO}$  in the solid state.

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[Appended table follows.]

No of Specimen	Intensity	$2\tau$	$\frac{\sin^2}{\delta}$	$hKl$	$a^2$	$a^2$	$hKl$	$a^2$
1	Average	37.02	0.096	111	29.15	--	200	40.05
2	Strong	46.21	0.149	200	--	18.79	211	40.35
3	"	61.55	0.256	202	29.16	--	311	40.10
4	Weak	73.18	0.351	113	29.26	--	--	--
5	Strong	78.48	0.395	(110) 2	--	18.89	410	40.14
6	"	95.49	0.544	113	--	18.86	(211)2	41.20
7	Very weak	102.51	0.605	133	29.30	--	--	--
8	" "	106.20	0.636	(120) 2	29.30	--	115	40.76
9	Average	122.03	0.765	(112) 2	29.33	--	522	40.35
10	"	135.97	0.858	115	29.34	--	611	41.28

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