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Density Change in Silicon upon Melting

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The density of both solid and liquid silicon has been measured in the vicinity of its melting point. From the average of several determinations, the increase in density upon melting is $9 \pm 1\%$.

BY observation of the mass of the sprout formed at the surface upon freezing a known mass of silicon, a density change of $10 \pm 1\%$ has been reported.¹ The measurement of density change is not feasible by conventional techniques due to the reactivity of silicon. Furthermore, quartz vessels become soft at the melting point of silicon (1415°C).

A technique has been devised to measure the density of molten silicon in a more direct way with a precision of approximately 1%. This is done by observation of the volume of a small known mass of silicon contained in a calibrated quartz capillary of sufficiently heavy wall to provide a dimensionally stable container during the melting. The density of solid silicon at the melting point was deduced from the lattice constant, obtained by extrapolation of x-ray diffraction data taken at temperatures up to 950°C .

To determine the density in the molten state, silicon samples were heated in air in quartz capillary tubing, 20 to 40 mils i.d. and about 300 mils o.d. The average bore diameter, over a $\frac{1}{2}$ -in. length of tubing, was computed from the length of a known mass of Hg. In the samples of tubing used (approx 2 in. in length), the diameter was constant within 1%. To observe the effect of heating on the diameter of the capillary, a tube was heated in air at 1450°C for 15 min. On re-measuring the diameter it was found to have changed by about 1%.

Samples of silicon, about 0.5 in. in length, were ground and etched to fit inside a capillary of known bore. The sample was then weighed. The capillary con-

taining the sample was placed on an alundum platform inside a tube furnace in air at a temperature of about 1425°C . The sample was discarded unless the silicon melted and formed a single, bubble-free column.

The length of the column was readily observed by using a reading microscope, and this length was stable during the run, indicating a negligible reaction with the silica. The microscope was calibrated in each run by observing the total length of the quartz tube, which had been measured with a micrometer. The microscope calibrations were averaged. Corrections due to the expansion of the quartz are negligible.

From the length, diameter, and mass of the molten silicon, the density was calculated. The results for five successful runs, as described above, give an average density of molten silicon at 1425°C of 2.49 g/cc with an average deviation of ± 0.02 g/cc.

The density of solid silicon at high temperature was determined from x-ray diffraction of powdered silicon, using a hot camera technique which has already been described.² The cell size was measured at temperatures up to 950°C . By extrapolation of these results to the melting point of silicon, one obtains a density of 2.2861 g/cc at 1415°C .

Hence, the density of silicon increased by $9 \pm 1\%$ upon melting.

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¹ H. von Wartenberg, *Naturwissenschaften* **36**, 373 (1949).

² W. L. Bond, *Rev. Sci. Instr.* **29**, 654 (1958).