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Fig. 1. Inner part of the DCM

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Fig. 2. Diagram of an ampoule with heater

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Abstract

Full Text

Chemistry

E. Yu. TONKOV, I. E. PAUKOV

THE MELTING CURVE OF SELENIUM UP TO 45,000 kg/cm²

(Presented by Academician Yu. N. Ryabinin, July 13, 1964)

The melting curve of selenium under pressure has been investigated in the range up to 10,000 kg/cm² ⁽¹⁾. Since in tellurium, an analogue of selenium, a maximum was found on the melting curve at about 11,000 kg/cm² ⁽²⁾, it was of interest to study the melting curve of selenium at higher pressures. In the present work the dependence of the melting temperature of selenium on pressure was studied in the range from 13,000 to 45,000 kg/cm². For this purpose a six-punch apparatus was used, the so-called diamond cubic multiplier–DCM ⁽³⁾. Figure 1 schematically shows the inner part of the DCM. Pyrophyllite cube 1, being compressed by six synchronously moving carbide punches 2, partially flows out between them, producing reliable seals 3. The punches are fixed in two split yokes, moving in conical holders. The DCM is actuated by a powerful press, which ensures sufficient plane-parallelism of the die plates. The design of the DCM will be described in greater detail in another paper. The apparatus was calibrated from jumps in electrical resistance during phase transitions in Bi and Tl. According to Kennedy and LaMori ⁽⁴⁾, the transition Bi I–Bi II occurs at about 25,900 kg/cm², and Tl II–Tl III at about 37,500 kg/cm². Wires of Bi and Tl 4, surrounded by silver chloride 5, were placed at the center of the pyrophyllite cube. The calibration curve above 37,500 kg/cm² was extrapolated.

Fig. 1. Inner part of the DCM

Fig. 2. Diagram of an ampoule with heater

Figure 2 shows an ampoule with a heater. Pyrophyllite 1 served as the pressure-transmitting medium. The substance under investigation 2, with a volume of ~0.05 cm³, was placed in a Teflon ampoule 3 with a threaded cap. For uniform

heating of the ampoule, the latter was surrounded by a copper sleeve 4. A thin-walled steel tube 5 with caps 6 was used as the heater. Between the ampoule and the heater there was pyrophyllite 7. The junction of a chromel-alumel thermocouple 8 was placed inside the ampoule. The thermocouple was insulated from the heater by pyrophyllite bushings 9 and then led out through sealing gaskets between the punches.

The melting temperature of the substance was determined in the following manner.

After raising the pressure, uniform heating was carried out at a rate of $\sim 0.3^\circ/\text{sec}$. The thermocouple readings were recorded. At the melting point of selenium, the rate of temperature increase slowed sharply.

Table 1

P , kg/cm ²	Temp., °C	P , kg/cm ²	Temp., °C	P , kg/cm ²	Temp., °C
13500	441	27200	578	38200	648
16000	474	29700	595	39500	664
16000	479	33500	625	39500	666
18000	492	33700	625	41000	664
22200	532	36000	639	44500	679
24000	551	38200	646	45500	680

After the experiment was completed, the sample was cooled to room temperature. Then the experiment was carried out at a higher pressure, and in this way the melting curve was determined over the entire pressure range.

Metallic selenium of “especially pure” grade was used in the work, with an impurity content of no more than 0.002%. Table 1 and Fig. 3 present the results of our experiments and those of Babb⁽¹⁾. The deviations of the experimental data from the graphically smoothed curve are on average $\pm 3^\circ$. In our estimate, the accuracy of pressure measurement was, up to 25 000 kg/cm², no worse than ± 1000 kg/cm²; up to 37 500 kg/cm², ± 1500 kg/cm²; and above that, ± 2000 kg/cm². The effect of temperature on the change in pressure was not taken into account. The chromel-alumel thermocouple was calibrated against a standard platinum-platinum-rhodium thermocouple with an accuracy up to 0.1°. The effect of pressure on the e.m.f. was neglected. The samples used in the work were subjected to spectral analysis in order to determine contamination of the substance by the thermocouple and ampoule. The total contamination amounted to no more than 0.4%. No leakage of selenium from the ampoule was observed.

Fig. 3. Melting curve of selenium up to 45,000 kg/cm²: 1 –data of the authors, 2 –Babb’s data.

The obtained melting curve of selenium in the interval from 13 500 to 45 500 kg/cm² is described by the Simon equation⁽⁵⁾:

Fig. 3. Melting curve of selenium up to 45,000 kg/cm²: 1 –data of the authors, 2 –Babb' s data.

Figure 3: Fig. 3. Melting curve of selenium up to 45,000 kg/cm²: 1 –data of the authors, 2 –Babb' s data.

$$\frac{P}{a} = \left(\frac{T}{T_0} \right)^c - 1,$$

where T is the melting temperature of the substance at pressure P , and T_0 is the melting temperature at atmospheric pressure. The quantities a and c are constants of the equation characteristic of each substance. According to our calculation, $a = 12\,000 \pm 1000$ kg/cm², $c = 2.2 \pm 0.1$, which agrees well with Babb' s data (¹).

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