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Pressure Correction at High Temperature Using the Melting Curve of Pb

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In the recent years, pressure measurement at high temperature has been an important problem. Girdle and belt type apparatus consisting of anvil and cylinder are useful as a pressure generation for material synthesis up to 100 kb. However, it is very difficult to estimate internal pressure at high temperature. Because, a frictional loss at the gasket portion is very complicated and may vary with temperature.

Inoue *et al.*[1] obtained the melting curve of Pb in a cubic anvil type high pressure cell, in which internal pressure was measured simultaneously based upon NaCl internal standard by X-ray technique. The melting curve of Pb thus obtained almost agreed with the data determined in a simple piston cylinder cell.[2]

The piston cylinder device has been well studied and improved in the last two years. A high temperature cell has been developed in which a weak salt such as NaCl is used as a pressure medium.[3] This cell exhibits extremely low friction. Pressures are computed by force/area measurements and are independent of any calibration points. Using the cell Mirwald *et al.*[4] measured the melting curve of Pb to 60 kbar by a differential thermal analysis (DTA) method. This result is the most reliable Pb melting curve.

The purpose of this paper is to estimate the pressure using the melting curve of Pb at high temperature in a girdle apparatus. The melting point of Pb was detected by the DTA. The temperature was determined with a Chromel-Alumel thermocouple without correction of the pressure effect on the e.m.f. The heating and cooling rates were about 100°C/min. Our Pb sample had a stated purity of 99.99%. The pure iron sample container for the DTA under high pressure was employed successfully without any visible evidence of contamination with the sample.

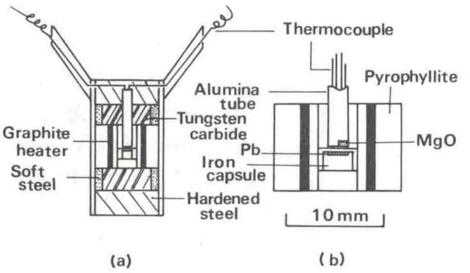

Fig. 1. Sample assembly of the high pressure DTA.
(a) Full illustration of the cell.
(b) Enlarged portion of the container and thermocouple.

We have used an assembly shown in Fig. 1 for carrying out DTA with girdle apparatus. Girdle apparatus with bore of 15 mm diameter was used. Figure 1(a) is full illustration of the cell and Fig. 1(b) is enlarged portion of the container and thermocouple for the DTA. Chromel-Alumel thermocouples were used for the temperature measurement and the differential temperature measurement. Present data on Pb are shown in Fig. 2 and the melting curve of Pb by Mirwald *et al.* is also shown in the figure. The raw data of Mirwald *et al.* for Pb uncorrected for the pressure effect on the e.m.f. of the thermocouple had been fitted to the second degree polynominal

$$P=a_0+a_1(t-a_2)^2 \quad [t \text{ in } ^\circ\text{C}, P \text{ in kbar}] \qquad (1)$$

where $a_0=-44.349$, $a_1=0.838*10^{-4}$ and $a_2=-400$.

The axis of abcissa of Fig. 2 shows nominal pressure calibrated by the electrical discontinuity of BiI-II (25.4 kb), BaI-II (55 kb)

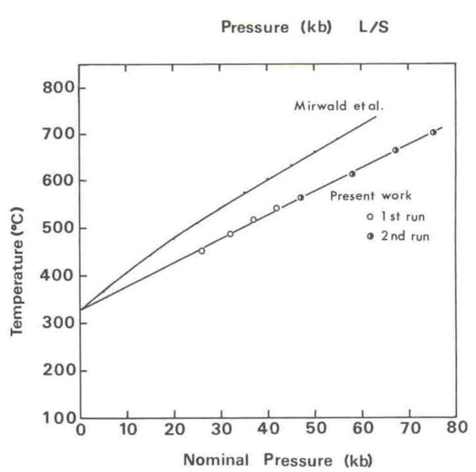

Fig. 2. Melting curves for lead.

and BiIII-V (77 kb) at room temperature. Additionally, we checked BiI-II transition at room temperature by the DTA. No difference of the required load between the electrical resistivity method and the DTA one was detected. The frictional loss at room temperature is not varied between both methods. The difference between the two data in Fig. 2 is attributed to the increase of frictional loss in the girdle type with temperature. Table I shows the variation of the frictional loss of pressure in our girdle apparatus with different

Table I. Pressure variation of the frictional loss with temperature.
T_m: Melting point of lead
P_n: Nominal pressure
P^*: Calculated values by substituting the observed melting point into eq. (1)
ΔP: $=P_n-P^*$

T_m °C	P_n kb	P^* kb	ΔP kb
450	26	16.2	9.8
514	37	25.7	11.3
560	47	32.9	14.1
661	67	50.0	17

pressure-temperature combination. P_n, P^* and ΔP as shown in Table I correspond to the nominal pressure, the corrected pressure from ref. 4 and (P_n-P^*), respectively.

The following points are suggested from these experimental results.

(1) The pressure dependence of the melting points of Pb was observed with a good reproducibility by the high pressure DTA in the girdle apparatus.

(2) The difference between P_n and P^* increases gradually with pressure and temperature. Practice of diamond synthesis also showed the extraordinary frictional loss in the present high pressure cell.

References

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