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<th>MEASUREMENT OF PRESSURE AT HIGH TEMPERATURES IN A PISTON-CYLINDER APPARATUS</th>
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<tr>
<td>Author(s)</td>
<td>SHIMADA, Mitsuhiko</td>
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<td>Contributions of the Geophysical Institute, Kyoto University (1971), 11: 213-229</td>
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Kyoto University
MEASUREMENT OF PRESSURE AT HIGH TEMPERATURES IN A PISTON-CYLINDER APPARATUS

By

Mitsuhiko Shimada

(Received September 16, 1971)

Abstract

The pressure in a piston-cylinder apparatus was calibrated at temperatures up to 1000 ºC. The calibration was based on the transitions in bismuth and potassium chloride, and the quartz-coesite transition. The magnitude of friction was independent of pressure and temperature in the range, from 15 to 35 Kb and up to 1000 ºC. The pressure rise from heating was found to compensate for the in-stroke piston friction. The quartz-coesite transition is discussed from the view of the calibration of pressure.

1. Introduction

The most immediate problems in high pressure technique from the view of earth science are the development of the apparatus generating statically ultra high pressures corresponding to the earth’s lower mantle and core, and the high accuracy of the measurement of pressure and temperature, especially the accurate absolute pressure scale at high temperatures, the calibration of pressure at high temperatures, and the effect of pressure on the emf of thermocouples (Akimoto [1969], Pistorius et al. [1967]).

The solid pressure-medium piston-cylinder high-pressure apparatus has become quite popular during recent years. The pressure generated by this apparatus is lower than by the anvil type apparatus, but this apparatus is capable of yielding much more accurate pressure than other types of high-pressure apparatus. However, there are uncertainties as to the pressure loss due to friction between the piston and the walls of the pressure vessel and to the pressure-transmitting media (cf. Tamayama and Eyring [1967]). As the pressure medium, talc is normally used at high pressures and temperatures, and silver chloride is chosen for comparison with talc because of its much lower shear strength.

The true pressure on the sample is usually taken to be the mean between the pressures obtained on compression and decompression, on the assumption of the symmetrical friction. The difference between them is called the double value of friction. Ordinary estimates of the magnitude of friction are based on calibrations using the transitions in bismuth and thallium at room temperatures. These transitions
are very sharp and easily detectable, and the transition pressures determined accurately are used as the standard points of pressure scale. In our laboratory, the pressure was calibrated based on these transitions and the true transition pressures were successfully determined as the mean between the pressures obtained on compression and decompression (Matsushima [1965], Shimada [1966a]).

Friction at high temperatures in the piston-cylinder apparatus has been discussed by some workers. Pistorius et al. [1967] reported from their study of the transition of potassium chloride that the assumption of the symmetrical friction was in error below 500°C. Green et al. [1966], in their study of the quartz-coesite transition at 1100°C, suggested that the symmetrical friction was in error even at 1100°C when talc was used as the pressure medium. Boyd et al. [1966] reported also in the quartz-coesite transition at 1400°C that the magnitude of friction and the symmetricity of friction were varied with the variety of the conditions of the bore of the pressure vessels.

In the present work the magnitude of friction has been determined at high temperatures and our experience that the pressure rise from heating compensates for the in-stroke piston friction was tested (Shimada [1966b]).

2. Experimental method

The apparatus used in this work was a single-stage piston-cylinder device. The hardened steel liner of 12 mm in diameter of bore was used in the carbide bore. The bore of the liner was lapped with 3 micron diamond paste. The pistons, bores of the vessel and outside surface of the pressure cells wrapped with 0.1 mm lead foil were lubricated with the molybdenum lubricant. The pressure-medium configurations in ordinary use are shown in Fig. 1. The temperature gradients near the space normally occupied by the sample are also shown in Fig. 1, (a) in the direction of length and (b) in the direction of radius in O–A section of (a). From this, when the sample size is below 2 mm in length and 4 mm in diameter and the thermocouples are properly located, the temperature in the sample is capable of measuring with the error of ±5°C at 1200°C.

Oil pressure applied to the ram was measured with a Heise gauge with a precision of better than 0.1%. The oil pressure necessary to lift the weight of the hydraulic ram was subtracted from the measured pressure. The nominal pressure on the sample was computed from the measured oil pressure, using the cross-sectional areas of the piston and hydraulic ram. The diameter of the piston was measured after each run.

Temperature was measured with chromel/alumel thermocouples. No correction was made for the effect of pressure on the emf of the thermocouple.

The transition in bismuth was detected by the change of the electrical resistance associated with the transition in thin bismuth wires embedded in the talc pressure-transmitting medium (Fig. 2b(l)). Compression and decompression runs were carried
out at a fixed temperature up to 170°C. Temperatures were believed to have a precision of ±1°C. Bismuth used in this work was of 99.9% from Nakarai Chemical Works.

In potassium chloride, the volume change associated with the transition was measured by means of the piston displacement. Potassium chloride of Nakarai guaranteed reagent-grade was preliminarily compressed at about 5 Kb into rods of 5.4 mm in diameter and 14 mm in length and embedded in the graphite furnace (Fig. 4). Compression and decompression runs were carried out at a fixed temperature up to 1000°C. Temperatures determined have great uncertainties considering the temperature gradients in the furnace shown in Fig. 1, because of the sample amount.
The quartz-coesite transition was determined by means of the quenching method at 800°C. Silicic acid of Mallinckrodt AR grade, containing 16.0% of water and 0.20% of elements nonvolatile with HF acid, was used as the starting material. A few mg of the sample, without ignition, was sealed in platinum tubing of 2 mm in diameter and 0.2 mm in wall thickness. The silicic acid was confirmed to be completely converted into quartz immediately on heating to 800°C at 24 Kb and completely into coesite after subjected to 800°C and 34 Kb for ten minutes. The compression runs were carried out as follows: pressure was firstly attained to about 24 Kb, then temperature was increased to 800°C, and finally pressure was raised to the expected value. In the decompression runs, pressure was firstly attained to about 34 Kb, then temperature was increased to 800°C and maintained for ten minutes, and finally pressure was released to the expected value. Besides the compression and decompression runs, two procedures of achieving the pressure-temperature conditions of runs were carried out in order to examine whether the pressure rise from heating compensates for the in-stroke piston friction and to estimate the magnitude of friction in the runs with the procedures used in the previous works. Those are: (1) pressure was attained near the expected value, then temperature was increased to 800°C (to P, to 800°C), and (2) pressure was raised to about 3 Kb higher than the expected value, then temperature was increased to 900°C and pressure released to the expected value and finally temperature was decreased to 800°C (called in the following sections “double cycle”).

3. Results

(1) The transition in bismuth

Fig. 2 shows the results for typical cycles through the transition and the sample arrangements. The results are summarized in Table 1. A melting point was detected by the abrupt change of the electrical resistance on heating at a fixed pressure.

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>Transition</th>
<th>Transition pressure, Kb</th>
<th>Double value of friction, Kb</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>In-stroke</td>
<td>Out-stroke</td>
</tr>
<tr>
<td>14</td>
<td>I-II</td>
<td>27.16±0.21</td>
<td>24.04±0.19</td>
</tr>
<tr>
<td></td>
<td>II-III</td>
<td>30.68±0.19</td>
<td>27.27±0.24</td>
</tr>
<tr>
<td>100±1</td>
<td>I-II</td>
<td>23.06±0.20</td>
<td>19.85±0.14</td>
</tr>
<tr>
<td></td>
<td>II-III</td>
<td>27.47±0.19</td>
<td>25.08±0.17</td>
</tr>
<tr>
<td>170±1</td>
<td>I-II</td>
<td>20.09±0.11</td>
<td>16.75±0.16</td>
</tr>
<tr>
<td></td>
<td>II-III</td>
<td>24.94±0.19</td>
<td>23.39±0.33</td>
</tr>
<tr>
<td>191±1</td>
<td>melting</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The Bi I–II transition at room temperatures has been very accurately determined by many researchers. In the NBS symposium, 1968, the Bi I–II transition pressure of 25.50 Kb at 25°C was adopted as the international pressure scale (Akimoto [1969]). The phase diagram of bismuth by Klement et al. [1963] and the transition pressures at 25°C are shown in Fig. 3, in which the uncertainties in the Bi I–II and Bi II–III transition boundaries are illustrated by hatched zones. This phase diagram by Klement et al. agrees with those by other researchers in the Bi I–II transition, but the slope of the Bi II–III boundary is rather steeper (Tamayama and Eyring [1967]).

The mean values between the pressures obtained on compression and decompression determined in this work are plotted in Fig. 3. In the Bi I–II transition and a melting point, the determined values are well placed on the phase diagram within experimental errors, but there is discrepancy in the Bi II–III transition. This discrepancy is due to the smearing-out of the transition caused by using talc as the pressure medium (Fig. 2(1)). The transition of bismuth is quite sharp when silver chloride is used as the pressure medium but is not sharp and smeared out when silver chloride is not used, which has been reported by many workers (Strong [1962], Giardini and Lloyd [1963], Lees [1966]). For comparison, the transition of bismuth wire embedded

Fig. 2. (a) Typical cycles through the transition of bismuth wire embedded in talc at 14°C (1), and in silver chloride at 21°C (2). (b) The respective sample arrangements.
in silver chloride was also determined at 21°C (Fig. 2(2)). The determined mean values are 25.67±0.21 Kb and 27.19±0.19 Kb for the Bi I-II and Bi II-III transitions, respectively, which are quite well placed on the phase diagram.

From the above discussion, if the Bi I-II boundary shown in Fig. 3 is true, the mean between the pressures obtained on compression and decompression gives the true pressure in this apparatus with the talc pressure medium.

(2) The transition of potassium chloride

The result for a typical cycle through the transition and the furnace assembly are shown in Fig. 4. The results are summarized in Table 2 and Fig. 5. The linear

<table>
<thead>
<tr>
<th>Temperature, °C</th>
<th>In-stroke</th>
<th>Out-stroke</th>
<th>Average</th>
<th>Double value of friction, Kb</th>
</tr>
</thead>
<tbody>
<tr>
<td>10±1</td>
<td>26.14±0.21</td>
<td>19.60±0.27</td>
<td>22.87±0.24</td>
<td>6.54±0.48</td>
</tr>
<tr>
<td>98±9</td>
<td>24.96±0.33</td>
<td>19.01±0.30</td>
<td>21.98±0.32</td>
<td>5.95±0.63</td>
</tr>
<tr>
<td>295±17</td>
<td>23.77±0.23</td>
<td>19.66±0.15</td>
<td>21.71±0.19</td>
<td>4.11±0.38</td>
</tr>
<tr>
<td>395±21</td>
<td>22.64±0.16</td>
<td>17.69±0.22</td>
<td>20.16±0.19</td>
<td>4.95±0.38</td>
</tr>
<tr>
<td>496±27</td>
<td>20.41±0.18</td>
<td>17.24±0.19</td>
<td>18.83±0.19</td>
<td>3.17±0.37</td>
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<tr>
<td>646±34</td>
<td>21.12±0.08</td>
<td>17.30±0.20</td>
<td>19.21±0.14</td>
<td>3.82±0.28</td>
</tr>
<tr>
<td>793±40</td>
<td>21.19±0.17</td>
<td>17.67±0.25</td>
<td>19.43±0.21</td>
<td>3.52±0.42</td>
</tr>
<tr>
<td>942±46</td>
<td>21.10±0.18</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Fig. 4. A typical cycle through the transition of potassium chloride and the furnace assembly.

Fig. 5. The results on the transition of potassium chloride and the comparison with the results obtained by Pistorius et al. [1967].
transition line, between 19.22±0.08 Kb at 25°C (Pistorius [1964]) and 18.95±0.1 Kb at 1042±2°C (Clark [1959]), were adopted as the true transition boundary according to Pistorius et al. [1967], which are illustrated in Fig. 5. The mean between the pressures obtained on compression and decompression at room temperature is about 16% too high. This discrepancy decreases with increase of temperature, and becomes essentially zero above 450°C. These circumstances quite resemble the results obtained by Pistorius et al. [1967] which are also shown in Fig. 5 by open circles. They concluded that the assumption of the symmetrical friction is in error below 500°C.

Considering in connection with the results on bismuth mentioned above, it is plausible that this discrepancy at lower temperatures is not caused by the unsymmetrical friction in the piston-cylinder apparatus with the talc pressure medium, but by the considerable shear strength of potassium chloride or the sluggishness and smearing-out of the transition.

(3) The quartz-coesite transition

The results of runs are shown in Table 3 and Fig. 6 for four different procedures. The transition pressures obtained for respective procedures and the double value of friction are summarized in Table 4. The mean between the pressures obtained on compression and decompression, 30.3 Kb is considered to be the true transition pressure at 800°C because the symmetrical friction has been verified from the results on bismuth and potassium chloride as mentioned above.

<table>
<thead>
<tr>
<th>Procedure</th>
<th>Pressure, Kb</th>
<th>Time, hr</th>
<th>Products*</th>
</tr>
</thead>
<tbody>
<tr>
<td>To P, to 800°C</td>
<td>24.2±0.1</td>
<td>1/60</td>
<td>Quartz</td>
</tr>
<tr>
<td>In-stroke at 800°C</td>
<td>31.3±0.2</td>
<td>1</td>
<td>Quartz</td>
</tr>
<tr>
<td>31.7±0.1</td>
<td>3</td>
<td>Quartz</td>
<td></td>
</tr>
<tr>
<td>32.2±0.2</td>
<td>1</td>
<td>Coesite + 50% Quartz</td>
<td></td>
</tr>
<tr>
<td>33.0±0.1</td>
<td>3</td>
<td>Coesite</td>
<td></td>
</tr>
<tr>
<td>33.2±0.3</td>
<td>1</td>
<td>Coesite</td>
<td></td>
</tr>
<tr>
<td>To P, to 800°C</td>
<td>33.8±0.1</td>
<td>1/6</td>
<td>Coesite</td>
</tr>
<tr>
<td>Out-stroke at 800°C</td>
<td>29.1±0.2</td>
<td>1</td>
<td>Coesite</td>
</tr>
<tr>
<td>28.7±0.1</td>
<td>1</td>
<td>Coesite</td>
<td></td>
</tr>
<tr>
<td>28.4±0.1</td>
<td>3</td>
<td>Quartz + 10% Coesite</td>
<td></td>
</tr>
<tr>
<td>28.0±0.3</td>
<td>3</td>
<td>Quartz + 6% Coesite</td>
<td></td>
</tr>
<tr>
<td>27.6±0.2</td>
<td>3</td>
<td>Quartz + 7% Coesite</td>
<td></td>
</tr>
<tr>
<td>27.0±0.2</td>
<td>1</td>
<td>Quartz + 6% Coesite</td>
<td></td>
</tr>
<tr>
<td>26.3±0.1</td>
<td>1</td>
<td>Quartz</td>
<td></td>
</tr>
<tr>
<td>To P, to 800°C</td>
<td>28.7±0.1</td>
<td>3</td>
<td>Quartz</td>
</tr>
<tr>
<td>28.8±0.1</td>
<td>1</td>
<td>Quartz</td>
<td></td>
</tr>
<tr>
<td>29.5±0.2</td>
<td>3</td>
<td>Quartz</td>
<td></td>
</tr>
<tr>
<td>29.9±0.2</td>
<td>3</td>
<td>Quartz + 2% Coesite</td>
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</tr>
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<td>30.1±0.1</td>
<td>1</td>
<td>Quartz + 50% Coesite</td>
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</tr>
<tr>
<td>30.6±0.1</td>
<td>3</td>
<td>Coesite</td>
<td></td>
</tr>
<tr>
<td>30.7±0.1</td>
<td>3</td>
<td>Coesite</td>
<td></td>
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</tbody>
</table>
MEASUREMENT OF PRESSURE AT HIGH TEMPERATURES

Table 4. Experimental results on the quartz-coesite transition

<table>
<thead>
<tr>
<th>Transition pressure, Kb</th>
<th>Double cycle of friction, Kb</th>
</tr>
</thead>
<tbody>
<tr>
<td>In-stroke</td>
<td>Out-stroke</td>
</tr>
<tr>
<td>31.9±0.1</td>
<td>28.6±0.1</td>
</tr>
</tbody>
</table>

The transition pressure obtained from the runs, “to P, to 800°C” quite agrees with the mean pressure or the true value, which shows that the pressure rise from heating, or thermal expansion, just compensates for the in-stroke friction. The value from the “double cycle” agrees well with the value on decompression. It is considered that in the double cycle, only the release of pressure of about 3 Kb prescribes the effect of friction but the temperature variation of 100°C does not, i.e. the runs of the double cycle is equivalent to the decompression runs as to the friction correction.

4. Discussion

The friction correction was evaluated from the results on the Bi I–II transition, the KCl transition above 500°C and the quartz-coesite transition. The double values of friction are plotted in Fig. 7 as the function of pressure and temperature. The
magnitude of friction is normally considered to increase with pressure and to decrease with increase of temperature. However, this tendency is not seen from Fig. 7 and it is quite acceptable considering the uncertainties in the obtained values that the magnitude of friction is independent of pressure and temperature in the range, from 15 to 35 Kb and up to 1000°C, the double value of friction being 3.5 Kb. Thus, to obtain the true pressure in our apparatus, it is necessary that 1.8 Kb is subtracted from the observed pressure for the compression runs, and added to the pressure for the decompression runs and the runs of the "double cycle". For the runs of "to P, to T", no friction correction is needed. The previous results published in this contributions are corrected in Appendix.

The phase boundary between quartz and coesite has been determined by many investigators, which are shown in Fig. 8. MacDonald [1956] determined using a "simple-squeezer" apparatus, Takahashi [1963] using a tetrahedral press, Slutskii and Pugin using a piston-cylinder apparatus with the press with parallel platens (Khitarov [1964]), and the others single-stage piston-cylinder apparatuses. The famous determination by Boyd and England [1960], which is almost identical with that by Kitahara and Kennedy [1964], is not shown in Fig. 8 because their friction correction was revised (Boyd et al. [1966]).

Body et al. [1966] determined the quartz-coesite transition pressure, 37.5±0.2 Kb at 1400°C from the numerous runs of compression and decompression with the talc.
MEASUREMENT OF PRESSURE AT HIGH TEMPERATURES

Fig. 8. Results on the quartz-coesite transition determined by various investigators.

pressure medium under the various conditions of the bore of pressure vessels. Green et al. [1966] also determined the transition pressure from the compression and decompression runs at 1100°C using silver chloride with boron nitride and talc as the pressure media and determined the friction correction by comparing the results obtained in silver chloride (31.8 Kb) and talc (34.3 Kb). Our results obtained with the similar method and apparatus are also plotted in Fig. 8. A transition boundary line can be drawn passing through the values obtained by Boyd et al. at 1400°C, by Green et al. in talc at 1100°C and by us at 800°C. It is an important problem whether this new line drawn from the very carefully determined values is the true transition boundary. However, a more important problem is that the softened silver chloride as the pressure medium at high temperatures does not only make the friction smaller than talc, but also the transition pressure obtained in silver chloride is lower than in talc. Green et al. [1966] interpreted that this is due to the larger shear strength of talc. Our unpublished data show that the transition pressure in silver chloride is about 29 Kb at 800°C. It is interesting to note that the linear line between this value and 31.8 Kb at 1100°C by Green et al. is also considered to be a transition boundary and is very close to the result obtained by Kitahara and Kennedy [1964].

As mentioned above, the pressures obtained in our apparatus were calibrated based on the transitions of bismuth and potassium chloride in the talc pressure medium, and the obtained pressure on the quartz-coesite transition in talc was considered to give the true transition pressure. Thus, the discrepancy of the obtained pressures in talc and in silver chloride gives rise to the alternative, either what have been considered to be the true pressures, like the transition pressure of bismuth or thallium, are incorrect, or the use of silver chloride at high temperatures leads to lower pressures than the actual ones. However, it will be very difficult or impossible to demand proof of the former question, since the very accurate experiments on the
transitions have been made by many investigators hitherto (e.g. Kennedy and LaMori [1961], Zeto and Vanfleet [1971]). As regards the latter, the softened part in silver chloride can be considered to be much more compressible than the other parts including the sample. If so, the stress on the softened part becomes lower than the stress on the other parts, differing from the applied pressure, thus the observed pressure is lower than the pressure on the sample. These problems will be discussed in details elsewhere.

Appendix  Corrections for the previous results

The results on the melting of basalt and the basalt-eclogite transition obtained previously (Shimada [1966a, b, 1967]) were corrected in the manner mentioned above. Amounts of water contained in the sample were also carefully redetermined. The "dry"

| Table A1. Chemical analysis and norm of Yakuno olivine basalt* |
|-------------------|------------------|-----------------|----------------|-------------------|
|                   | SiO₂             | Or              | 8.5            |
| TiO₂              | 1.10             | Ab              | 19.0           |
| Al₂O₃             | 15.75            | An              | 29.8           |
| Fe₂O₃             | 3.32             | Di              | 7.6            |
| FeO               | 9.04             | En              | 18.5           |
| MgO               | 8.72             | Fs              | 7.7            |
| CaO               | 7.82             | Fo              | 2.5            |
| Na₂O              | 2.10             | Fa              | 1.1            |
| K₂O               | 1.47             | Mt              | 3.6            |
| H₂O⁺              | 0.97             | II              | 1.6            |
| H₂O⁻              | 0.55             |                 | 99.9           |
|                    |                  | 100.35          |

* Analyst: K. Nishimiya.

| Table A2. Results of runs on melting of natural olivine basalt |
|-------------------|---------------------|------------------|-------------------|
|                   | Corrected pressure, Kb | Temperature, °C* | Time, hr          | Products         |
| P = P_H₂O         |                      |                  |                  |                  |
| 0                 | 1253±5 P             | 1                | melt             |
| 0                 | 1243±5 P             | 1                | melt             |
| 0                 | 1233±5 P             | 1                | melt + crystal   |
| 0                 | 1266±5 P             | 1                | melt + crystal   |
| 0                 | 1208±5 P             | 1                | melt + crystal   |
| 0                 | 1103±5 P             | 1                | crystal          |
| 3.1±0.5           | 1148±5 P             | 1                | glass            |
| 3.1±0.5           | 1103±10 P            | 1                | glass + crystal  |
| 3.1±0.5           | 998±5 P              | 1                | crystal          |
| 3.1±0.5           | 948±5 P              | 1                | crystal          |
| 7.4±0.5           | 1050±5 P             | 1                | glass            |
| 7.6±0.5           | 999±5 P              | 1                | glass + crystal  |
### Measurement of Pressure at High Temperatures

<table>
<thead>
<tr>
<th>Pressure (p)</th>
<th>Temperature (°C)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>7.4 ± 0.6</td>
<td>948 ± 5 P</td>
<td>Crystal</td>
</tr>
<tr>
<td>11.2 ± 0.6</td>
<td>1094 ± 5 P</td>
<td>Glass</td>
</tr>
<tr>
<td>11.2 ± 0.5</td>
<td>1049 ± 5 P</td>
<td>Glass</td>
</tr>
<tr>
<td>11.3 ± 0.5</td>
<td>998 ± 5 P</td>
<td>Crystal</td>
</tr>
<tr>
<td>14.3 ± 0.6</td>
<td>1029 ± 5 P</td>
<td>Glass + Trace Crystal</td>
</tr>
<tr>
<td>14.9 ± 0.6</td>
<td>1030 ± 25 P</td>
<td>Glass</td>
</tr>
<tr>
<td>17.5 ± 0.6</td>
<td>981 ± 5 P</td>
<td>Glass + Crystal</td>
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<tr>
<td>13.5 ± 0.3</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
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<tr>
<td>14.5 ± 0.2</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
</tr>
<tr>
<td>16.7 ± 0.2</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
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<tr>
<td>19.4 ± 0.3</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
</tr>
<tr>
<td>20.3 ± 0.3</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
</tr>
<tr>
<td>21.4 ± 0.3</td>
<td>900 ± 5 C</td>
<td>Crystal</td>
</tr>
<tr>
<td>27.9 ± 0.8</td>
<td>1014 ± 7 P</td>
<td>Glass + Crystal</td>
</tr>
<tr>
<td>26.6 ± 0.4</td>
<td>911 ± 5 P</td>
<td>Crystal</td>
</tr>
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#### 1.64 ± 0.12 wt% of Water

<table>
<thead>
<tr>
<th>Pressure (p)</th>
<th>Temperature (°C)</th>
<th>Material</th>
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<tbody>
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<td>Glass + Crystal</td>
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<tr>
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<td>1011 ± 8 P</td>
<td>Glass + Crystal</td>
</tr>
<tr>
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<td>1159 ± 5 P</td>
<td>Glass</td>
</tr>
<tr>
<td>23.3 ± 0.5</td>
<td>1159 ± 5 P</td>
<td>Glass + Crystal</td>
</tr>
<tr>
<td>25.6 ± 0.4</td>
<td>1009 ± 5 P</td>
<td>Trace Glass + Crystal</td>
</tr>
<tr>
<td>29.1 ± 0.4</td>
<td>1209 ± 5 P</td>
<td>Glass + Crystal</td>
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#### 1.11 ± 0.23 wt% of Water

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<td>Glass + Crystal</td>
</tr>
<tr>
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<td>Glass + Crystal</td>
</tr>
<tr>
<td>12.5 ± 0.6</td>
<td>1209 ± 5 P</td>
<td>Glass</td>
</tr>
<tr>
<td>11.4 ± 0.8</td>
<td>1057 ± 8 P</td>
<td>Glass + Crystal</td>
</tr>
<tr>
<td>15.3 ± 0.8</td>
<td>1209 ± 5 P</td>
<td>Glass</td>
</tr>
<tr>
<td>19.4 ± 0.8</td>
<td>1009 ± 6 P</td>
<td>Crystal</td>
</tr>
<tr>
<td>22.9 ± 0.4</td>
<td>1209 ± 5 P</td>
<td>Glass + Crystal</td>
</tr>
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<td>1056 ± 5 P</td>
<td>Glass</td>
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</table>

#### 0.04 ± 0.02 wt% of Water

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<tr>
<td>12.1 ± 0.6</td>
<td>1260 ± 6 P</td>
<td>Glass</td>
</tr>
<tr>
<td>11.0 ± 0.7</td>
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<td>Glass + Crystal</td>
</tr>
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<td>1266 ± 8 P</td>
<td>Glass</td>
</tr>
<tr>
<td>15.9 ± 0.4</td>
<td>1211 ± 5 P</td>
<td>Glass + Crystal</td>
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</tr>
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<td>1260 ± 7 P</td>
<td>Glass + Crystal</td>
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<tr>
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<td>1195 ± 7 P</td>
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</tr>
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</table>

*P*, temperatures measured with Pt/Pt 13% Rh thermocouples; *C*, those with chromel/alumel thermocouples.
Table A3. Results of runs on the basalt-eclogite transition

<table>
<thead>
<tr>
<th>Corrected pressure, Kb</th>
<th>Temperature, °C*</th>
<th>Time, hr</th>
<th>Starting material**</th>
<th>Products**</th>
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<td>Gl</td>
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<td>1134±5 P</td>
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<td>Cpx + Ga</td>
<td>Cpx + Ga</td>
</tr>
<tr>
<td>14.5±0.2</td>
<td>1108±8 P</td>
<td>5</td>
<td>Pl + Cpx + Ga</td>
<td>Pl + Cpx</td>
</tr>
<tr>
<td>16.8±0.6</td>
<td>1109±7 P</td>
<td>4</td>
<td>Pl + Cpx</td>
<td>Pl + Cpx + Ga</td>
</tr>
<tr>
<td>19.5±0.4</td>
<td>1111±6 P</td>
<td>5</td>
<td>Gl</td>
<td>(Pl) + Cpx + Ga</td>
</tr>
<tr>
<td>20.1±0.2</td>
<td>1109±5 P</td>
<td>4</td>
<td>Gl</td>
<td>Pl + Cpx + Ga</td>
</tr>
<tr>
<td>21.2±0.4</td>
<td>1109±6 P</td>
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<td>Pl + Cpx</td>
<td>(Pl) + Cpx + Ga</td>
</tr>
<tr>
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<td>1112±6 P</td>
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<td>Cpx + Ga</td>
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<td>Cpx + Ga</td>
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<td>1086±5 P</td>
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<td>(Pl) + Cpx + Ga</td>
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<td>Gl</td>
<td>Cpx + Ga</td>
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<tr>
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<tr>
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<td>Pl + Cpx + Ga</td>
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<tr>
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<td>Pl + Cpx + Ga</td>
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<tr>
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<td>Pl + Cpx + Ga</td>
<td>(Pl) + Cpx + Ga</td>
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<td>22.3±0.4</td>
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<td>Gl</td>
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<td>12.4±0.3</td>
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<td>6</td>
<td>Gl</td>
<td>Pl + Cpx</td>
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<td>Gl</td>
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<td>950±5 C</td>
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<td>Gl</td>
<td>Pl + Cpx + Ga</td>
</tr>
<tr>
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<td>6</td>
<td>Gl</td>
<td>(Pl) + Cpx + Ga</td>
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<tr>
<td>22.4±0.4</td>
<td>950±5 C</td>
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<td>Gl</td>
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<td>851±5 C</td>
<td>12</td>
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<td>Pl + Cpx + Ga</td>
</tr>
</tbody>
</table>

* P, temperatures measured with Pt/Pt 13% Rh thermocouples; C, those with chromel/alumel thermocouples.

** Gl, glass; Pl, plagioclase; Cpx, clinopyroxene; Ga, garnet; ( ), small amount; ( ), trace amount.
samples nominated previously in the runs on melting were found to have 0.04 wt% of water, in agreement with comments by Mizutani [1967] and Watanabe [1970]. From the recalculation involved, the amounts of water in the other samples were changed from 0.98 to 1.11 wt% and from 1.60 to 1.64 wt%.

The chemical analysis of the basalt used is shown in Table A1, for convenience. The corrected results of quenching runs of the melting are shown in Table A2, and those on the basalt-eclogite transition are in Table A3 and Fig. A1. The corrected melting relations and transition are summarized in Fig. A2. The "dry" melting curves obtained by the measurement of electrical conductivity of the same basalt by Watanabe [1970] were adopted with a partial revision. The results after correction do not lead to different conclusions than those derived from the previous works. The results on the melting of albite (Shimada [1969]) is corrected and reported elsewhere with new data.

In the previous works, temperature was measured with Pt/Pt13%Rh and
Fig. A2. Effects of pressure and water on the melting of natural basalt and the basalt-eclogite transition (cf. Shimada [1966a, b, 1967]). The “dry” melting relations after Watanabe [1970].

chromel/alumel thermocouples and no correction was made for the effect of pressure on the emf of the thermocouples. In the present article also, no correction was made, because for the present there is no recognized result on the effect of pressure on the emf of thermocouples though considerable efforts have been made (e.g. Getting and Kennedy [1970]), and the thermocouple used in each run is only given in Table A2 and A3.

Acknowledgement

I am indebted to Dr. Shōgo Matsushima for his valuable advice.

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Shimada, M., 1966a; Melting of basalt at high pressures, Zisin, 19, 167–175.


Shimada, M., 1969; Melting of albite at high pressures in the presence of water, Earth Planet. Sci. Letters, 6, 447–450.


