

## Friction Effects and Pressure Calibration in a Piston-Cylinder Apparatus at High Pressure and Temperature

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The pressure of a piston-cylinder apparatus was calibrated at a temperature of 1100°C. The calibration is based on the quartz-coesite phase transition. Pressure losses are considerable and a correction of -11% at 1100°C and 35 kb is indicated for a compression run with talc as the pressure-transmitting medium. This correction was evaluated by comparing results obtained with talc and silver chloride pressure-transmitting mediums.

### INTRODUCTION

In recent years increasing use of piston-cylinder apparatus at high pressure and temperature has been made in phase-equilibrium studies. It has been shown that hydrostatic pressure conditions are more closely attained in this type of apparatus than in an anvil apparatus [Boyd and England, 1960a]. However, there are uncertainties as to the pressure loss due to friction between the piston and the walls of the pressure vessel and due to friction and other possible effects inherent in the talc pressure-transmitting medium. We have investigated the magnitude of pressure losses in the piston-cylinder apparatus at high temperature by using two different pressure-transmitting mediums. Talc is normally used, but it has a substantial shear strength. Silver chloride was chosen as the pressure medium for comparison with talc because of its much lower shear strength.

Early estimates of the magnitude of pressure losses were based on calibrations using the bismuth and thallium transitions at room temperature [Boyd and England, 1960b]. They used silver chloride and talc pressure-transmitting mediums and determined the correction needed to account for pressure loss in the talc medium, assuming that silver chloride has a negligible strength. They concluded that a friction correction of -13% is needed at room temperature. They attributed the pressure loss to the shear strength of talc, and, since this will decrease with increasing temperature, they suggested that at high temperatures the friction

correction would be closer to  $-8 \pm 5\%$  [Boyd and England, 1960a; Boyd, 1962]. In later work Boyd and England [1963] considered that at high temperatures the shear strength of talc is very low, and they no longer applied a friction correction.

Kitahara and Kennedy [1964], in their study of the quartz-coesite transition, applied a friction correction of -12% at 17.1 kb, -8% at 30 kb, and -7% at 41 kb for a compression cycle. They estimated this correction from a study of the melting point of mercury at different pressures [Klement *et al.*, 1963]. Their friction correction was determined at any specific pressure as half the difference between the compression and decompression strokes. They assumed that the pressure loss on a compression run was the sum of the piston-cylinder friction and friction in the talc.

Newton [1965], in work at pressures of 4 to 8 kb and temperatures of 640 to 860°C and using a piston-cylinder apparatus similar in design to Kennedy's apparatus, applied a pressure correction of -1.5 kb over the 4- to 8-kb pressure range; this represents a -37% to -19% correction. It was determined using the LiCl melting curve at about 700°C as the calibration point.

### EXPERIMENTAL METHOD

The quartz-coesite phase transition at 1100°C has been chosen as the calibration point, since a considerable amount of high-pressure work in this laboratory has been done in the neighborhood of this temperature. Also, experience showed that when the sample temperature



was 1100°C at pressures greater than 30 kb, there was no significant melting of the silver chloride pressure medium. Some minor melting in the immediate vicinity of the 'hot spot' may have occurred. The nearness of the temperature to the melting point of the silver chloride would also reduce its strength and so improve it as a pressure-transmitting medium. Silver chloride pressure cells were made by melting the silver chloride, pouring it into a mold, and then machining it to the required dimensions (Figure 1c).

The quartz-coesite transition is rapid at 1100°C if some moisture is present, so that run times of an hour length are adequate. A great deal of work has been done recently on establishing the quartz-coesite curve with piston-cylinder and other apparatus, and adequate results are available for comparison.

The apparatus used is based on the design of *Boyd and England* [1960b], and the experimental procedure is similar to that described by *Boyd and England* [1960a, b] and by *Ringwood and Green* [1964]. Temperature is measured with a Pt|Pt-10Rh thermocouple. Temperature calibration of a normal assembly with

talc as the pressure medium showed that the temperature gradient in the space normally occupied by the specimen capsule is less than 5°C. In a normal run, once thermal equilibrium is reached, the temperature varies by approximately 5°C on either side of the control point. Hence temperatures are believed to have a precision better than  $\pm 10^\circ\text{C}$ . Oil pressure applied to the ram is measured with a Heise gage with a precision of better than 0.1%. The nominal pressure on the sample (i.e., assuming perfect transmission of applied pressure) is computed from the measured oil pressure, using the known cross-sectional areas of the piston and hydraulic ram.

The starting materials consisted of very finely ground mixes of either (1) 94% quartz, 4% coesite, and 2% silicic acid or (2) 90% coesite, 5% quartz, and 5% silicic acid. The coesite used in these mixes was prepared from silicic acid subjected to a temperature of  $900 \pm 60^\circ\text{C}$  and a pressure of 40 kb for 2½ hours. The first mix was used for most of the runs. A run was considered to be in the quartz field when the coesite had demonstrably disappeared and in the coesite field when the amount of coesite increased measurably. A few runs on the second mix demonstrated reversibility of the reaction.

Approximately 10 to 20 mg of undried sample was sealed in a platinum tube of wall thickness 0.015 cm. Three different ways of achieving the pressure-temperature conditions of a run were used:

1. Single-stage compression. The pressure was raised to the required value, then the temperature increased to 1100°C. This method resulted in final inward piston movement when used with talc (Figure 1a) and talc + boron nitride pressure mediums (Figure 1b). It was intended also to use it for the silver chloride + boron nitride pressure medium (Figure 1c), but, when the temperature was increased, expansion of the assembly resulted in a pressure excess of 1 to 1½ kb, so that in actual fact the run was over-pressed and pressure had to be released. Thus, with the silver chloride, the single-stage application of pressure and temperature resulted in a decompression run.

2. Double-stage compression. The pressure was increased to about 2 kb below the required value. The temperature was then raised to

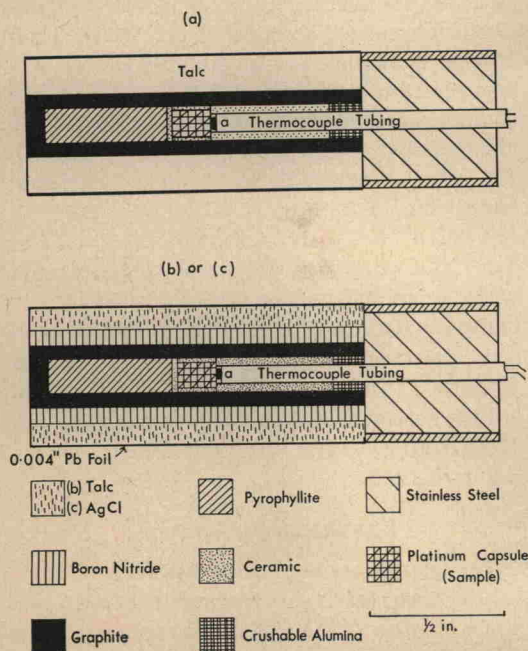


Fig. 1. Detail of types of pressure cells used in the calibration. Thermocouple junction, in contact with sample capsule.