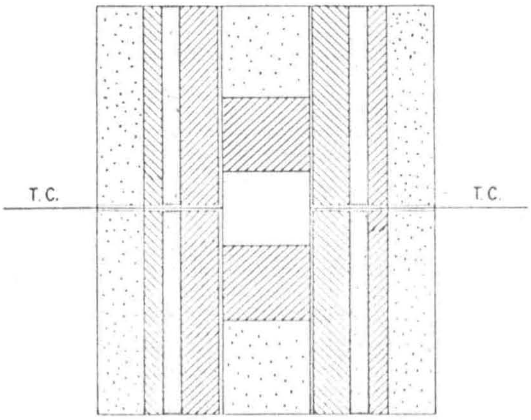

Fig. 1. High-pressure chamber. Light stippling is massive pyrophyllite (lava), crosshatched areas are alumina, and the heavy stippling is the graphite heater. Sample at the center is enclosed in Pt foil. Thermocouple wires pass through the cell in protection tubing (*not shown*). (×50.)

by hydrothermal techniques was first reported by Roy,[5] but later details have shown that this phase is not identical with natural andalusite.[6] In addition, two phase diagrams based on deductions from field observations and other considerations have been published.[7,8] As yet the phase relations of the three varieties of Al_2SiO_5 are not completely understood and more data are needed in both moderate pressure temperature regions and higher pressure-temperature regions (e.g., above 30,000 bars and 1500°C) where the interest may be less geological and more exploratory in its emphasis.

It is the higher pressure-temperature region which was chosen for further study because of curiosity about the decomposition of kyanite at high pressures and temperatures as compared with its decomposition to mullite and quartz at 1 atm. The latter reaction is accompanied by a 21% increase in volume. By comparison, a 5% increase in volume would result from the formation of corundum and coesite from kyanite.

II. Experimental

(1) High-Pressure Equipment

An Elmes 300-ton hobbing press was adapted for experimental use.[9,10] The belt apparatus and the basic chamber design were the same as those which have been described by several investigators.[9–11] The particular cell used in the present study is shown in Fig. 1. The internal resistance heater was a sleeve of graphite for most runs, although Ni or Pt heaters were used occasionally.

The material to be run was placed in a tube made from 0.0005-in. foil of Pt or 80Pt20Rh. The amount of foil and the amount of sample material used in each run was kept constant. The cylindrical sample after pressing was about 1.5 mm high.

(2) Pressure Measurement

Bourdon-type gages were used to measure oil pressure in pounds per square inch on the ram. The gages were calibrated in terms of pressure in the chamber, measuring the resistance change at the Bi and Ba phase transformations at room temperature at 25,300 and 59,000 bars, respectively.* The gages were calibrated several times during the investigation. On the basis of other experience with the belt equipment, the uncertainty of the pressure measurement is considered to be ±2000 bars. There are some qualifications which should be considered: (1) The calibration was made at room temperature only and (2) the assumption was made that the pressure was transmitted hydrostatically.

In the more usual calibration procedure with the belt a piece of calibration wire is embedded in an AgCl sleeve which is surrounded by pyrophyllite. The flow properties of AgCl presumably result in a more hydrostatic distribution of pressure. Since the actual runs are in Al_2O_3 sleeves, which on a structural basis would be expected to have more limited flow compared with AgCl, calibration runs were also made by embedding the wire in Al_2O_3. Within the limits of error of measurement the phase transformations in Bi and Ba took place at the same pressure as in the pyrophyllite-AgCl cell.

At high temperatures the reaction within the pyrophyllite results in different crystalline phases plus liquid (or glass) so that the pressure-transmitting properties change. Some assurance of the reproducibility of the pressure measurement at high temperatures was gained by reasonably good agreement with the melting of Pt as a function of temperature.[11]

Pressure was automatically controlled during a run by means of a Bristol pressure controller recorder.

(3) Temperature Measurement

A power-vs.-temperature plot was first determined for each type of cell and heater by making several runs with a thermocouple in the cell shown in Fig. 1. The thermocouple was insulated from the rest of the cell by alumina thermocouple tubing. The Pt foil of the sample container completed the circuit between the two dissimilar wires so that the junction was at one side of the cell. The reproducibility of the runs with thermocouples was found to be no larger than ±50°C and in general was closer to ±25°C in the range over which more data were collected (25,000 to 40,000 bars). No correction was made for the small change in emf as a function of pressure. In the figures summarizing the data, an overall uncertainty of ±75°C for the temperature measurement is shown.

After the power-temperature curves were determined, most runs were made without thermocouples. In this way considerable time was saved without appreciable sacrifice in accuracy. The temperature was hand-controlled with a variable transformer.

(4) Other Observations Relating to Accuracy of Data

It was found in the earlier runs that rather steep temperature gradients existed lengthwise in the pressure chamber. If the final length of the sample was of the order of 3 mm, it was obvious from macroscopic examination that the ends had been

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[5] D. M. Roy, "Hydrothermal Synthesis of Andalusite," *Am. Mineralogist*, **39** [1/2] 140–43 (1954); *Ceram. Abstr.*, **1956**, September, p. 201*a*.

[6] Shigeo Aramaki and Rustum Roy, "Revised Phase Diagram for the System Al_2O_3–SiO_2," *J. Am. Ceram. Soc.*, **45** [5] 229–42 (1962).

[7] Akiho Miyashiro, "Stability Relations of Kyanite, Sillimanite, and Andalusite, and the Physical Conditions of Metamorphic Processes," *J. Geol. Soc. Japan*, **55**, 218–23 (1949).

[8] J. B. Thompson, Jr., "Thermodynamic Basis for Mineral Facies Concept," *Am. J. Sci.*, **253**, 65–103 (February 1955); *Ceram. Abstr.*, **1957**, October, p. 259*e*.

[9] H. T. Hall, "Ultra-High-Pressure, High-Temperature Apparatus: The 'Belt'," *Rev. Sci. Instr.*, **31** [2] 125–31 (1960).

[10] H. P. Bovenkerk, F. P. Bundy, H. T. Hall, H. M. Strong, and R. H. Wentorf, Jr., "Preparation of Diamond," *Nature*, **184**, [4693] 1094–98 (1959).

[11] H. M. Strong and F. P. Bundy, "Fusion Curves of Four Group VIII Metals to 100,000 Atmospheres," *Phys. Rev.*, **115** [2] 278–84 (1959).

* When this work was done, the pressure calibration was based on the Bi I–II and the Ba II–III transformations at 24.8 and 77.4 kbars, respectively. Since that time new values for these transitions and others have received greater acceptance (25.3 and 59, respectively; see G. C. Kennedy and P. N. LaMori, pp. 304–13 in Progress in Very High Pressure Research (Lake George Conference), John Wiley & Sons, Inc., New York, 1961) and the data have been replotted on the basis of the new calibration.