High-pressure stability of acmite

the pressure media. But this approach fails to recognize the possible large frictional effects, which may be either additive or subtractive, and also the fact that the "strength" varies as a function of pressure, temperature, materials, and design of cell. On the basis of studies of the kyanitesillimanite transition in both piston-cylinder and gas-media apparatus, Richardson, Bell, and Gilbert (1968) have conclusively shown that the true sample pressure is at least 1 kb lower than the nominal (calculated) pressure at 12 to 13 kb and 850°C, for the present cell arrangement, due entirely to strength of media. However, no way is now known to extrapolate this finding to any other very different P and T condition. Pressures given in this report have not been corrected, but the frictional sense is indicated where known.

Most runs in this study were piston-out, for two reasons. First, this insured that the melting curve would be approached from the solid field as the run pressure was attained. Second, most of the pressure cylinders used in this study were steel lined. Boyd and others (1967) noted that pressure discrepancies due to frictional effects were smaller in piston-out runs where steel liners were employed.

Temperatures presented for runs were determined with Pt-Pt₉₀Rh₁₀ thermocouples and have an uncertainty of $\pm 10^{\circ}$ C (Bell and England, 1967), with no correction applied for pressure. The containers for most of the charges were Pt70Rh30 capsules. Since iron is least soluble in platinum when in the ferric state, it appeared that there would be no serious loss of iron to the container. To check this assumption, a capsule and charge, which had been run at 1400°C and 10 kb for 30 minutes (table 3, A45) was examined with a Material Analysis Company model-400 electron microprobe. The entire capsule was mounted in epoxy and ground down until a longitudinal section half way through the capsule was exposed. Scans across the quenched glass showed no detectable variation in iron content, either in the center of the charge or near the container walls. Scans from the glassy part of the charge across the interface into the wall showed a smooth drop in X-ray emission to a constant background level within 3 to 5 μ m. For a beam size of about 1 to 2 μ m this drop corresponds to the simple effect of moving across an edge. Thus, no diffusion gradient was detected.

This ferric silicate is one of the first to be studied at very high pressure. Because the environment in the furnace assembly is reducing with respect to ferric iron owing to the graphite heater, a difficulty arose in the piston-cylinder device with the type of charge container used. This container is sealed mechanically with a lid. Some recrystallization of the $Pt_{70}Rh_{30}$ alloy during the run should act to tighten the seal and is normally adequate to hold and isolate liquids and solids. However, reduction of some hematite to magnetite during runs of even a few minutes is evidence of the fact that the capsule is not completely closed to gas (namely, CO/CO_2 from the graphite heater).

All parts of the pressure cell (except the talc sleeve) were dried at 1000° to 1100° C in N₂ for 30 minutes to 1 hour before use. The loaded

capsule was also dried at 850° to 925°C for several hours before use. In some experiments, both the dried assembly and charge were stored in a desiccator until the run was made. The onset of melting is lowered distinctly if the sample is not moisture free. From Bailey's (1969) data, it can be seen that the lowering would be about 350°C at 10 kb for H_2O -saturated samples.

A series of experiments designed to check the stability of acmite under conditions of lowered relative oxygen fugacity was also carried out, employing graphite and iron capsules. Some of the graphite capsules (plus charges) were dried at 850° to 925°C in N₂. The iron capsules plus charges were dried at 160° to 170°C in vacuum for 24 to 48 hours. As will be discussed later, this procedure was inadequate.

EXPERIMENTAL RESULTS

The intention of this study was to determine the pressure effect on the incongruent melting of acmite (point X in fig. 1) and the liquidus



Fig. 2. Acmite composition Na₂O•Fe₂O₃•4SiO₂. All runs were performed in $Pt_{r_0}Rh_{s_0}$ capsules. Solid rectangles represent acmite; half-solid rectangles with horizontal lines represent acmite + hematite + magnetite + liquid; half-solid rectangles represent hematite + magnetite + liquid; open rectangles represent all liquid. A rightdirected arrow indicates a piston-out run (any pressure deviations due to frictional effects would shift pressure to a higher value); a left-directed arrow indicates a pistonin run. Bars extending both directions indicate that history of piston travel during run is unknown. The acmite melting curve is drawn into the 1-atm point (where P_{O_2} = 0.2 atm) determined by Bailey and Schairer (1966). Strictly, the curve should be drawn to a temperature at 1 atm where acmite would melt in equilibrium with hematite + magnetite ($P_{O_2} \approx 10^{-6}$ bars). This temperature is probably not significantly lower than that determined in equilibrium with the atmosphere. Similarly, the curve tentatively suggested as the liquidus should not be drawn to the 1310°C point, but to some lower temperature.

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