

Fig. 7. Typical cooling curve with air jet.

Any phase boundary may be followed only over a limited range of temperature. At low temperatures the rate of reaction is too low to achieve equilibrium in times available. At high temperature, the reaction rate may be so great that quenching becomes impossible. Frequently the role of water may be exploited to extend this range. In exploring the analcite-jadeite + water equilibrium, it was found that at temperatures in excess of 500°C air quenching was too slow, and analcite recrystallized in the quenching process. In this instance, the problem was solved by releasing pressure before air quenching. Jadeite and nepheline albite were unchanged by release of water vapor, and analcite retained its skeleton structure as anhydrous analcite. The rate of re-

action drops very rapidly when water vapor pressure is released, so this expedient is frequently helpful in quenching.

We have found that equilibrium is not readily attained when the initial material is composed of more than one crystalline phase. We had hoped that the shear strain imposed on the aggregate during loading would provide sufficiently intimate mixing so that all components would come to equilibrium. This is not the case. It may well be that rotation of the pistons with respect to each other would promote the attainment of equilibrium due to the resultant intense shearing strain. We shall shortly try this.

We have found three satisfactory methods of introducing water into the system:

(1) Starting material is a hydrate of the composition intended for study (e.g. analcite for the reaction jadeite ≠ nepheline + albite).

(2) Starting material contains a hydrate whose anhydrous product does not take part in the reaction to be studied (e.g. to study the reaction jadeite + water → analcite, start with diaspore + jadeite. The diaspore decomposes to corundum releasing water which combines with jadeite to form analcite. This will work only close to the diaspore-corundum boundary, in the corundum field.

(3) Starting material is glass with adsorbed water. Finely ground glass will absorb roughly 0.5 to 1 percent of water, which is bound to the glass even at elevated temperatures. This technique is not in general suitable for adding a known amount of water but is extremely useful in adding enough water to increase reaction rates. For example, glass of a basalt composition was run at 1000°C, 10 kb for one hour with no observable crystallization. This glass was wet but was not ground very fine. The same glass, with prior fine grinding under water, crystallizes completely in short times at temperatures as low as 800°C. It is presumed that this difference in reaction rate is due to the water adsorbed: the crystalline phases are anhydrous. Water vapor apparently also increases the reaction rate in certain solid-solid transitions.

Proof that equilibrium has been attained at a phase boundary is accomplished by standard techniques—e.g. demonstration that the reaction is reversible. In the case of dehydration boundaries, special techniques are required with this apparatus which are beyond the scope of this paper and will be reported in other papers dealing with such systems.

Experience to date indicates that at hydration-dehydration boundaries, the water vapor pressure closely approximates the total pressure. Such boundaries determined with this apparatus lie on a smooth extension of the boundaries determined at lower pressure in conventional hydrothermal apparatus.

We have the impression that reaction rates are faster in this apparatus than in a purely hydrostatic environment but do not yet have adequate information to prove this. MacDonald (in press), using our squeezer, found that the calcite → aragonite transition went to completion in about 15 minutes at 500°C, whereas in a hydrostatic test at the same pressure and temperature no conversion to aragonite was observed in two hours in an experiment by Griggs.