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A Sample Holding Technique for Study of Crystal Growth in Silicate Melts

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Abstract

A thin platinum wire loop is an effective way to hold silicate melts during experimentation in a gas-mixing furnace. This method results in a minimum of physical and chemical interaction between the sample and container but maximum interaction between sample and gas mixture. However, volatilization of sodium occurs while the silicate is molten. By minimizing the chance of heterogeneous nucleation, the method is ideal for experimental investigation of the origin of rock textures.

Introduction

To study the textural features of rocks experimentally, it is desirable that physical interaction of the container and charge be minimized, so that the container is not a prominent nucleation site in the charge. Many containers are unsuitable for textural study of silicate melt crystallization because the melt adheres tenaciously to the container or spreads itself over the container walls, or because heterogeneous nucleation of silicate phases occurs on the container surfaces. The choice of a sample container is further complicated by the need to limit chemical interaction with the charge. All sample containers react chemically with silicate melts to some degree; for example, the commonly used containers made of Pt and Pd alloys extract iron from the silicate.

This paper describes and evaluates a variation of a sample holding arrangement first used by Ribould and Muan (1962) which minimizes these physical and chemical container-sample interactions. The technique has proved ideal for reproducing the textures and phase chemistry features of lunar basalts (Lofgren *et al,* 1974). Variations of the arrangement have been used by McKay (personal communication, 1973), but the technique differs substantially from that recently described by Presnall and Brenner (1974).

Technique Employed

Platinum is the sample container material most often used because it can be employed over the wide range of physical and chemical conditions required by experimental petrology. In Fe-containing systems, however, the Fe rapidly alloys with the platinum and this changes the composition of the charge. In general, there is a problem with the use of crucibles or tubes for holding melted silicates because they have a high contact-area to sample-volume ratio. To minimize this ratio we have developed the technique of Ribould and Muan (1962) for suspending melted samples in a platinum wire loop. By reducing contact between melt and container to a minimum, Fe loss from the sample is minimized, as is the chance of heterogeneous nucleation of silicate phases at the melt-container boundary. The surface tension that holds the melted charge in the loop is sufficient, even in very fluid melts, to maintain an ellipsoidal droplet 4–6 mm in diameter. The technique therefore has the added advantage that the interaction between sample and gas mixture is a maximum, since the ratio of surface area to volume of an ellipsoidal charge is large.

Run Procedure

A powdered starting material (80–100 mg) is pressed into a pellet 6-7 mm in diameter and 2-4 mm thick. The pellet is placed on a loop of 0.008″ diameter platinum wire, attached to the electrodes of a strip heater (Fig. 1). The wire is heated electrically until slight sintering or melting tacks the pellet onto the wire. [In contrast, Ribould and Muan (1962) sintered pellets, enclosed by platinum foil, at 50°C below the solidus temperature for 5 minutes. The pellet

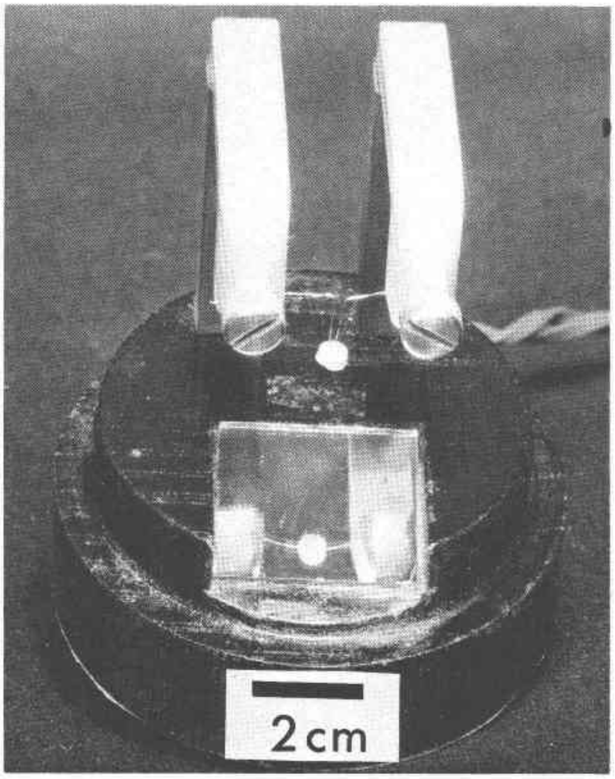

FIG. 1. Pellet in place on platinum loop and attached to strip heater. A transformer and variac comprise the electrical components of the heater.

was then placed in a platinum wire loop, but without tacking by electrical heating.] The loop and charge are then attached to a ceramic bead, itself suspended by a thin wire strung across two heavy duty platinum wires which are encased in a 30 cm long ceramic rod (Fig. 2). During the attachment process a hemostat clamped across the ends of the loop prevents flexing of the wire, which would dislodge the pellet. The ceramic rod and sample are then inserted into a gas-mixing furnace and a run made. Wire of 0.008″ diameter is sufficiently strong to hold a 100 mg charge indefinitely at temperatures up to 1600°C. For smaller charges or lower temperatures, thinner wire may be used.

The resultant charge (Fig. 2) is ideally suited for examination of the surface shapes of crystals on the outside of the charge, for removal of single crystals, and for preparation of thin sections to study texture and phase chemistry. The wire acts as a nucleation site for very few crystals and has little effect on textures developed in the bulk of the charge (Fig. 3). However, some heterogeneous nucleation does occur at the melt-gas interface.

Chemical Changes During Runs

Analyses 1, 2, and 3 in Table 1 compare the compositions of a terrestrial basalt and of two aliquots of the basalt melted at 20–30°C above the liquidus for 1 and 24 hours respectively. The most significant change observed is that Na_2O content of samples decreases by about 1.3 wt percent in a 24 hour run. Analyses 4 and 5 respectively are the starting composition and run product of a synthetic lunar basalt (low initial Na_2O content) for which no Na_2O loss is apparent after 3 hours melting at 1250°C (25°C above the liquidus). Apparently, the greater the initial Na_2O content of samples, the greater the loss of Na_2O during a given run duration. There is no significant change in K_2O content during runs.

The extent of iron loss from the melted basalts to the platinum wire may be gauged from analyses 1-5 in Table 1. For example, 0.9 wt percent FeO (4.8 percent of the total FeO initially present) is lost from the iron-rich lunar basalt during a three-hour melting experiment. The Fe content of the melt is uniform to within 5 μ of the wire. It must be stressed that the run conditions were highly reducing ($f_{O_2} = 10^{-12}$-10^{-13} atm at 1250°C), thereby favoring removal of Fe from the melt. In runs under the more oxidizing conditions typical of terrestrial magmas, Fe loss would be less severe. This rate of Fe loss is tolerable and is considerably lower than was reported by Merrill and

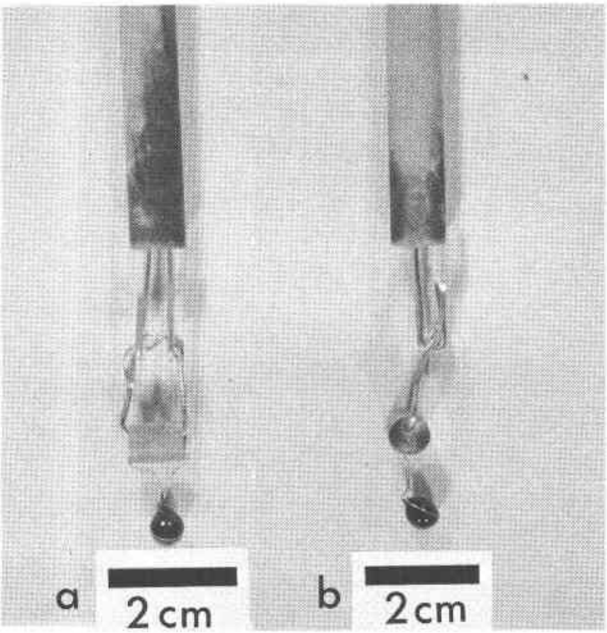

FIG. 2. Melted sample on platinum loop, showing means of attachment to ceramic rod holder (**a.** front view, and **b.** side view).

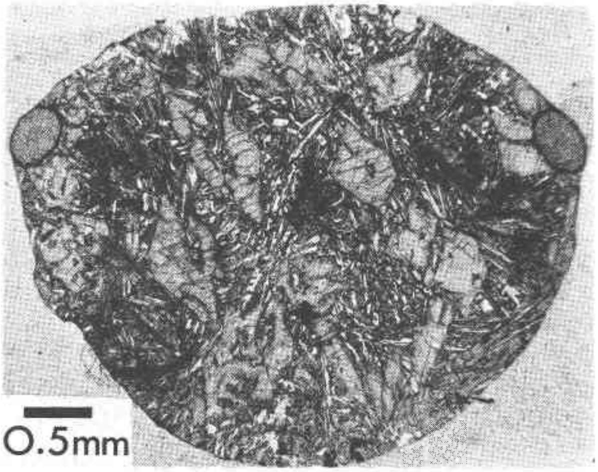

FIG. 3. Transmitted light photomicrograph of a charge of synthetic Apollo 15 basalt cooled from above the liquidus temperature at 2.5°C/hour. Large, subequant, euhedral phenocrysts of pyroxene are enclosed in a matrix of glass and fine-grained pyroxene and plagioclase. The two gray ellipsoids at the margin of the charge are remnants of the platinum loop. There is a slight tendency for increased nucleation of phases near the wire, but crystals at the center of the charge appear to have nucleated homogeneously.

Wyllie (1973) for samples run completely enclosed in platinum capsules.

Electron microprobe analysis was made of the platinum wire in contact with the glass of composition 5 (Table 1). Less than 1 wt percent iron was found at the center of the wire, but an alloy of 40 wt percent iron occurs at the rim. From the profile of the Fe content across the wire, the diffusion coefficient in platinum was calculated as 1.75 × 10^{-10}cm^2/s (calculated using the method described by Medford, 1973).

TABLE 1. Comparison of Compositions of Starting Material and Melted (Glass) Run Products*

	1**	2**	3**	4**	5**
SiO_2	50.19	50.63	51.00	48.70	48.04
TiO_2	1.59	1.57	1.60	1.68	1.76
Al_2O_3	15.39	15.31	15.63	10.70	11.19
FeO	9.72	9.83	9.25	18.90	18.03
MnO	0.19	0.17	0.17	0.26	0.28
MgO	8.53	9.09	9.13	8.94	9.18
CaO	10.81	11.04	11.18	10.41	10.26
Na_2O	3.34	2.70	2.09	0.05	0.08
K_2O	0.24	0.24	0.23	0.09	0.07
	100.00	100.58	100.28	99.73	98.89

*All experiments were performed at an oxygen fugacity of 10^{-12} to 10^{-13} atm. Analysis 1 is by X-ray fluorescence; J.M. Rhodes analyst. Analyses 2-5 are by electron microprobe; C.H. Donaldson analyst.

**1. Basalt recalculated free of H_2O, P_2O_5 and S.
2. 80 mg basalt pellet melted for 1 hour in platinum loop at 1250°C.
3. 80 mg basalt pellet run for 24 hours in platinum loop at 1250°C.
4. Synthetic Apollo 15 quartz normative basalt. (Lofgren et al, 1974).
5. 80 mg pellet of synthetic Apollo 15 quartz normative basalt run for 3 hours in platinum loop at 1250°C.

Summary

The method of containing silicate liquids and charge preparation described in this paper should be of value to experimental petrologists and materials scientists. It is simple, cheap, and provides minimum chemical and physical interaction between the sample and the container, but maximum interaction between sample and gas mixture. The technique is applicable to rock powders, glasses, and solid samples. It is particularly suited to studies of highly reduced melts, such as Fe-rich lunar basalts.

Note Added in Proof

Basalt number 1 (Table 1) was melted at 1250° and $fO_2 = 10^{-8}$ atm for six hours. Fe loss was 2.5 percent of the original amount present and Na loss 2.8 percent of the amount present. Hence, Fe and Na compositional changes in charges run at oxygen fugacities typical of terrestrial igneous rocks are within the error of the electron microprobe analysis.

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