LETTER

A novel method for experiments in a one-atmosphere box furnace

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ABSTRACT

We present a conceptually simple method to perform high-temperature experiments in a one-atmosphere box furnace that has a negligible cost of materials. The experimental setup consists of two commercially available materials and can be customized to sample or furnace size with few limitations. Furthermore, the design allows easy extraction of samples in one piece, making them eligible for textural analysis.

The setup comprises a graphite capsule and a fireclay shell, the latter of which acts as a heat-resistant protective shield. Containers must be individually hand-crafted, but each can hold multiple samples. The setup can be reliably used in temperature conditions below the heat tolerance limit of the commercial fireclay, commonly ~1400 °C. Moreover, the graphite capsule buffers the oxygen fugacity to strongly reducing conditions during the experiment. The main advantage of our method lies in the utilization of easily accessible and low-cost materials that provides a widely applicable experimental setup easily used at larger scales. The method was developed during an experimental study of magmatic crystal-liquid suspensions and was reliable for experiments lasting for up to 36 h.

Keywords: High temperature, oxygen fugacity, sample capsule, graphite, textural analysis

INTRODUCTION

High-temperature experiments performed at one atmosphere and normal oxidizing environment are commonly performed by using noble metal crucibles, suitable for such conditions without the risk of sample damage or capsule failure (Edgar 1973). However, these materials can raise several limitations for the desired setup, including the size of the container and its cost. Moreover, removal of samples from such containers often presents a complication, forcing the user to either damage the container or fragment the sample to extract all material. In a recent experimental investigation, we focused on the textural evolution of crystal-liquid suspension during cumulative formation by crystal settling. To perform such a study, it became necessary to develop an easy and quickly repeatable method for high-temperature experiments that are capable of withstanding temperatures of up to 1400 °C in an ambient (oxidizing) atmosphere and preserving an intact sample for full-scale textural analysis (Fig. 1a). Thus, a setup has been constructed to allow for the use of low-cost graphite capsules of variable size that have been modified to endure the presence of oxygen without the risk of burnout and sample loss.

EXPERIMENTAL SETUP

The powdered starting material (i.e., haphazardly glassy) placed within the experimental container is separated from the surrounding oxidizing environment by two protective layers (Fig. 2a). The inner layer is composed of pure graphite and represents the capsule itself. In our case, the graphite was cut into cubes of ~1.5 cm edge length, with an 8 mm deep hand-drilled cavity covered by a thin (~1.8–2.8%) graphite lid. Once the cavity is filled with starting material and covered, a fireclay cement is prepared in a separate pot following the instructions listed on the packaging. The commercial fireclay we used (Uniflex manufacturer; Al2O3 38–40%, SiO2 50–55%, TiO2 1.8–2.8%) required a 1:1 mixture of water and sodium silicate solution (aka “water glass”), thoroughly mixed and added to the dry fireclay to create a paste-like substance.

First, the fireclay is carefully applied directly onto the graphite capsule to secure the lid in place and cover the capsule surface until no graphite is exposed. Then, the layer of fireclay is enlarged by placing the capsule into an appropriately sized form (at least 1 cm of free space around the capsule in every direction) and filling in the surrounding space. For this purpose, we used small silicone baking forms with dimensions of 5 × 3.5 cm (Fig. 2b). Once the form is filled with the fireclay, the setup is complete and is left to dry at room temperature for at least 24 h. The experimental setup is intended for single use only. To extract the sample from the capsule after the quench, the fireclay shell is to be broken by a hammer to expose the graphite. It may be necessary to cut open the graphite cube if larger amounts of material were used, otherwise the sample is easily removable by hand.

OPERATION AND ITS LIMITATIONS

The container is to be used as any standard sample capsule and is reliable within the temperature and duration constraints of the fireclay. The maximum run temperature is specific to the type of fireclay used and should adhere to the manufacturer’s recommendations. For most commercial fireclays, the limit is ~1400 °C. Our setup has been employed regularly at conditions of between 1200 to 1390 °C and exceptionally up to 1500 °C during testing. During experimental runs below 1400 °C, the setup proved stable for durations up to 36 h. Longer experiments had a significantly lower success rate, with ~50% of cases resulting in graphite burnout. A similar issue occurred at higher temperatures, where the reliable timeframe for the setup was proportionally shorter due to stressing the fireclay beyond its limits. There are no requirements for a specific quench method; the capsule is suitable for both submerging in water and cooling at room temperature, as neither of these methods has been observed to damage the container in any way.

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