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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 an 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 hours.

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 a number of 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 textural evolution of crystal-liquid suspension during cumulate formation by crystal settling. In order to perform such study, it became necessary to develop an easy and quickly repeatable method for high-temperature experiments that is capable of withstanding temperatures of up to 1400 °C in 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., haplobasaltic glass) 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 approximately 1.5 cm edge length, with an 8 mm deep hand-drilled cavity covered by a thin (ca. 2 mm) 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 packaging. The commercial fireclay we used (Uniflex manufacturer; Al_2O_3 38-40 %, SiO_2 50-55 %, TiO_2 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 x 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 hours. The experimental setup is intended as single use only. In order to extract the sample from the capsule after quench, the fireclay shell is to be broken by 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 constrains of the fireclay. The maximum run temperature is specific to the type of fireclay used and should adhere to the manufacture recommendation. For most commercial fireclays, the limit is approximately 1400 °C. Our setup has been employed regularly at conditions of between 1200 to 1390 °C, and exceptionally up at 1500 °C during testing. During experimental runs below 1400 °C, the setup proved stable for durations up to 36 hours. Longer experiments had a significantly lower success rate, with approximately 50 % of cases resulting in graphite burnout. Similar issue occurred at higher temperatures, where the reliable timeframe for the setup was proportionally shorter due to stressing of the fireclay beyond its limits. There are no requirements for specific quench method, the capsule is suitable for both submerging in water and cooling at room temperature as neither of these methods have been observed to damage the container in any way.

In a series of experiments performed with ~60 vol. % of olivine seed crystals, we noticed a presence of large air bubbles jammed within the olivine crystal framework. Volumetric amount of trapped bubbles is comparable to the amount of air present initially in the starting material powder (ca. 50 vol. %). This indicates that thermal expansion of the air during heating of the assembly is roughly compensated by the overpressure built up within the sample cavity. Providing no air escaped the sample, this observation limits the internal overpressure to less than ~5 bars (at 1400 °C) and indicates that our setup ensures ambient to near-ambient experimental pressures.

The employment of graphite capsule buffers oxygen fugacity of the experiments to reducing conditions on the CCO buffer (carbon oxide-carbon dioxide buffer, Holloway et al. 1992). This corresponds to $\log fO_2$ values lower than -10.5 to -8.7 for temperature range of 1200 °C to

1400 °C at 1 bar; according to Jakobsson and Oskarsson (1994). In the series of experiments performed with seeds of natural olivine (~Fo_{90}) as well as a natural basalt powder, we observed that some iron was exsolved out of olivine crystals or of basaltic glass in form of small (order of micrometers) spherical inclusions within the time span of 20 hours (Fig. 1b). The iron reduction caused a shift in the olivine composition to ~Fo_{94}, suggesting fO_2 values laying on or below the IW buffer (iron-wüstite buffer, Médard et al. 2008) with corresponding logfO_2 ranging from -11.95 to -9.72 (between 1200 °C to 1400 °C; Hirschmann 2021). The very reducing environment created by our set up offers the possibility to perform experimental studies, such as those involving the metal/silicate partitioning (e.g., Kilburn and Wood 1997), without the need for a gas mixing furnace and thus avoiding the use of hazardous gases (CO, CO_2).

Implications

Although very simple, the new methodology for high-temperature experiments performed in an ambient (oxidizing) atmosphere presents a unique way to undertake experimental research with a need for a larger volume of material. The method allows full preservation of samples after quenching with no need to expend noble metal crucibles. The convenient attributes of this method could be beneficial for studies of igneous textures and kinetics, which require analysis of numerous samples in full volume. Moreover, the method allows preparation of synthetic starting materials in larger quantities, in case noble metal crucibles are unavailable or reducing environment is needed.

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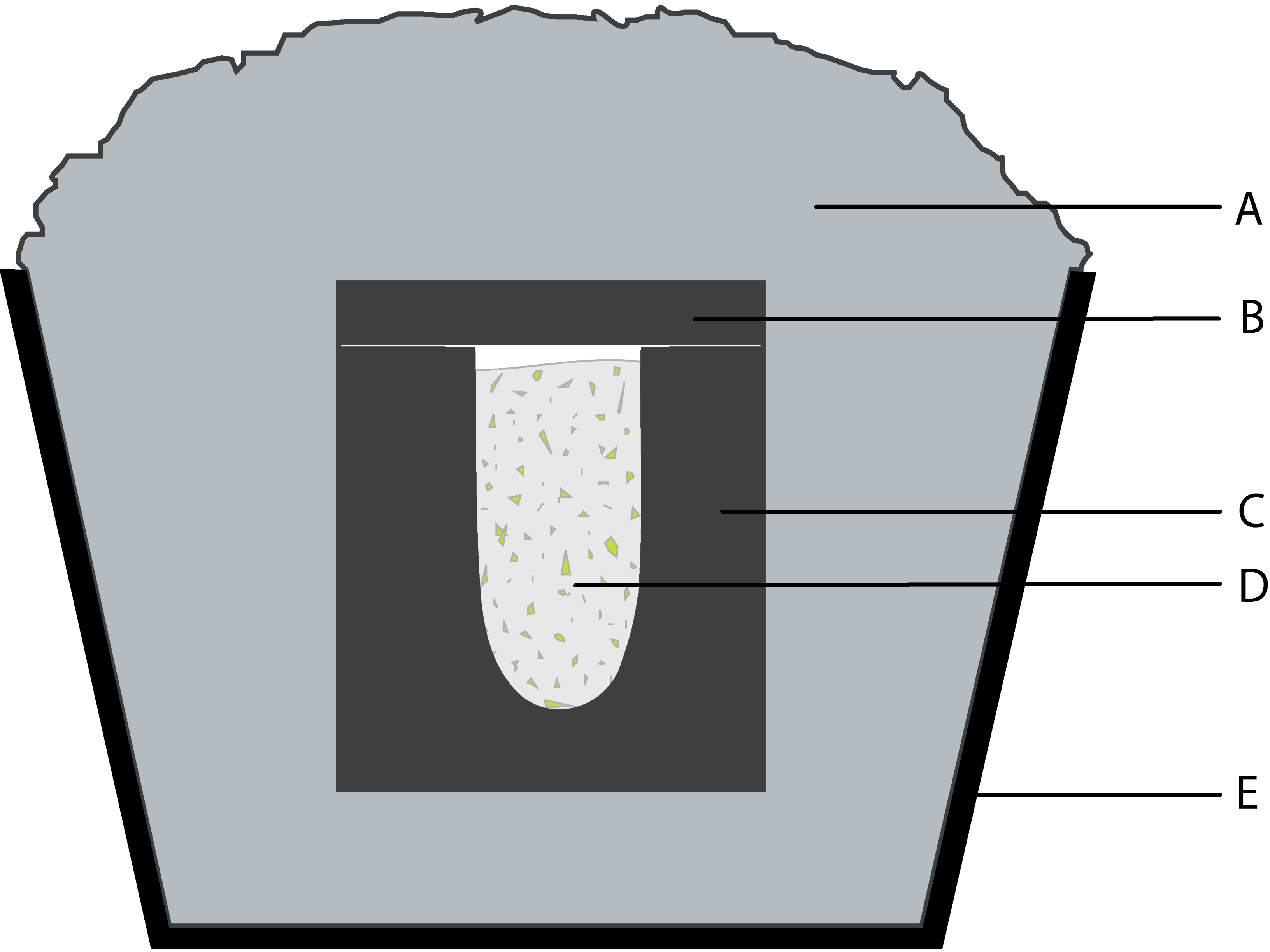
Figure captions

Figure 1. a) An exemplary sample containing olivine suspension in haplobasaltic melt, produced by using the presented setup. Full-scale, back-scattered electron image was acquired for the purpose of textural analysis. b) A closeup of the back-scattered electron image of pure iron spheres exsolved out of natural olivine crystals. Images obtained by using an electron-probe microanalyzer (15 kV accelerating voltage was employed).

Figure 2. a) Cross-section through the enclosed setup. A - fireclay protective layer, B - graphite lid, C - graphite capsule, D – experimental charge, E - silicone baking form (removed prior to use). b) Photograph of two containers placed inside silicone baking forms. The diameter of silicone forms is 50 mm.


A
B
C
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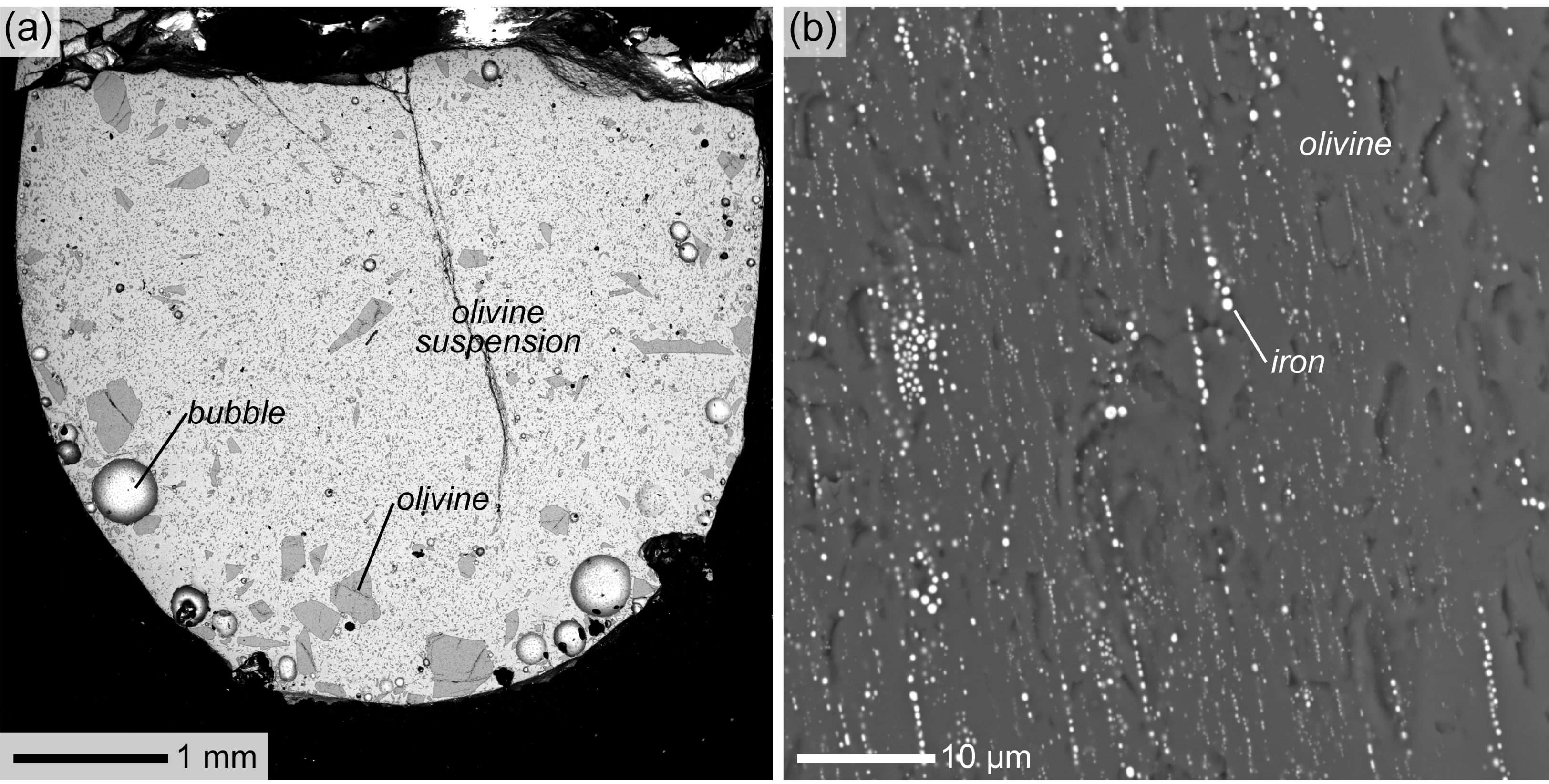
(a)
olivine
suspension
bubble
olivine
1 mm
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