



Intrinsic solidification behaviour of basaltic to rhyolitic melts: A cooling rate experimental study



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ABSTRACT

Dynamic cooling-induced solidification experiments were run using six silicate glasses along the basalt–rhyolite join ($B_{100} = 100$ wt.% of basalt, $R_{100} = 100$ wt.% of rhyolite), i.e. B_{100} , $B_{80}R_{20}$, $B_{60}R_{40}$, $B_{40}R_{60}$, $B_{20}R_{80}$ and R_{100} ; the glasses directly quenched from 1300 °C after a dwell of 120 min (experiment E0) contain 50–400 ppm H_2O , $\ll 1$ area% μm -sized bubble, and Fe^{2+}/Fe_{tot} between 0.34 and 0.46. Experiments were performed in Pt capsules at room pressure and fO_2 of air, between 1300 and 800 °C using three different cooling rates of 0.0167, 3 and 30 °C/min; these cooling rates were run two times: E1–E2 experiments at 0.0167 °C/min, S1–E3 at 3 °C/min, and E4–E5 at 30 °C/min. In experiments E1 to E5, samples were annealed for 120 min at 1300 °C, whereas in the experiment S1 the samples were firstly heated for 30 min at 1400 °C followed by a dwell time of 2400 min at 1300 °C before cooling. In none of the run-products preferential crystallization at the melt/gas interface was observed. B_{100} , $B_{80}R_{20}$ and $B_{60}R_{40}$ run-products have a low tendency to preferentially crystallize on Pt walls, while $B_{40}R_{60}$, $B_{20}R_{80}$ and R_{100} are not affected by the presence of Pt substrata. All run-products show homogeneous textures, except for $B_{60}R_{40}$ and $B_{40}R_{60}$ at 0.0167 °C/min in the E1 experiment. The duplicates of $B_{40}R_{60}$ and $B_{60}R_{40}$ at 0.0167 °C/min and B_{100} at 30 °C/min show differences in crystal content (>4 and <14 area%). $B_{40}R_{60}$ and $B_{60}R_{40}$ duplicated run-products that have the same amount of early-crystallized clinopyroxene and spinel, but different contents in lately-formed plagioclase. The run-products with the same starting composition from E3–S1 (3 °C/min) show a high reproducibility in terms of crystal shape, size, and amount (<4 area%). This demonstrates that the crystallization path is not affected by the different heat treatments above the liquidus temperature, i.e. the time scale of structural re-equilibration (relaxation) and chemical re-homogenisation are shorter than the experimental time scale. Possible chemical heterogeneities on a length scale of several micrometres for R_{100} and several hundreds of micrometres for B_{100} can be removed at 1300 °C within 120 min. A heat treatment at 1300 °C for 120 min significantly reduces the amount of μm -sized bubbles, potentially responsible for the onset of nucleation. The experimental reproducibility is low when the cooling path intersects the tip of the time–temperature–transformation (TTT) curves, i.e. when the nucleation rate is near its maximum (I_{max}). In that case, even small thermal variations in cooling rate and local composition can have large effects on phase abundance and crystal size. Dynamic crystallization experiments can be properly interpreted and compared only if they are texturally homogeneous and the physico-chemical state of the superheated silicate liquid is known. The solidification conditions used in this study mirror those of aphyric lavas and dikes emplaced at shallower crustal levels.

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1. Introduction

The time–temperature history experienced above the liquidus may have strong influence on the crystallization path of a silicate melt and, hence, on texture and composition of crystallized phases.

A silicate liquid kept for long times and/or at high temperatures above its liquidus will have the lowest nucleation rate during successive cooling and will have the highest incubation time of the first crystallizing phase in the sub-liquidus region (Kirkpatrick, 1981; Tsuchiyama, 1983; Lasaga, 1997; Hammer, 2008; Pupier et al., 2008). Two different explanations have been proposed for this effect: 1) an incomplete re-equilibration of short-range order and/or polymeric configurations acquired at liquidus or superliquidus conditions that metastably persist upon cooling (Kirkpatrick, 1981; Corrigan, 1982; Tsuchiyama, 1983; Hammer, 2008) or 2) by the presence of microscopic gas bubbles,

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chemical heterogeneities or “solid foreign particles” that are not fully eliminated during heat treatments and persist in a silicate melt at sub-liquidus temperatures (Dowty, 1980; Lofgren, 1980, 1983; Davies and Ihinger, 1998; Armienti, 2008; Iezzi et al., 2008; Pupier et al., 2008; Iezzi et al., 2011). These two controversial explanations are still unsolved and systematic experimental investigations are not available on physico-chemical features (bubbles, volatile content, un-melted crystals, etc.) of a silicate melt before its solidification; this may limit the interpretation of kinetics data in a single run-product as well as the comparison of experimental results, even derived by the same initial melt. Heating above the liquidus for some time is required to remove such memory effects and to determine the intrinsic solidification behaviour of natural silicate melts as a function of composition.

Reproducibility and homogeneity of cooling-induced solidified products have been poorly investigated and crystallization kinetics is not sufficiently explored for natural melt systems. The few experimental studies reported in the literature mainly focus on basalts, and, subordinately, on andesites, latites, trachytes and rhyolites (Donaldson et al., 1975; Lofgren, 1980; Kirkpatrick, 1981; Berkebile and Dowty, 1982; Lofgren, 1983; Tsuchiyama, 1983; Couch, 2003; Hammer, 2006; Pupier et al., 2008; Iezzi et al., 2011 and references therein). Results from experiments conducted on SiO₂-poor melts at atmospheric pressure and *f*O₂ lower than air show that the sample holder (wire or capsule) and the container material (Pt or Au) can significantly influence the results of solidification experiments (Dowty, 1980; Lofgren, 1980; Berkebile and Dowty, 1982; Tsuchiyama, 1983; Pupier et al., 2008). On the other hand, intermediate (andesitic and latitic) to evolved (trachytic and rhyolitic) silicate melts appear to be poorly or not affected by the sample container (Couch, 2003; Iezzi et al., 2008, 2011; Mollo et al., 2012a). This difference probably relies on the rapid diffusion in SiO₂-poor melts allowing fast transport to existing nuclei. Additionally, surface tension between the nucleus, the melt and (possibly) foreign substrates plays a crucial role in nucleation kinetics (Dowty, 1980; Fokin et al., 2003; Iezzi et al., 2008, 2011). However, the controlling parameter for nucleation and growth was poorly constrained for these relatively SiO₂-rich melt systems.

In order to investigate (i) the variation of intrinsic crystallization behaviours of natural silicate melts as a function of their composition, (ii) the role of superheating before cooling and (iii) the degree of textural reproducibility, we have performed crystallization experiments on six silicate melt compositions from basalt to rhyolite: B₁₀₀, B₈₀R₂₀, B₆₀R₄₀, B₄₀R₆₀, B₂₀R₈₀ and R₁₀₀, where B₁₀₀ = basalt and R₁₀₀ = rhyolite (numbers are wt.% in each mixture). These compositions cover the most part of volcanic rocks. The effect of cooling rate was explored using rates of 0.0167, 3 and 30 °C/min between 1300 and 800 °C. The results of this study show that the superheating effect can be eliminated by annealing all these melts at 1300 °C for 120 min before cooling (Fig. 1), the majority of cooling-induced crystallization can be well reproduced, and the intrinsic solidification behaviour from basalt to rhyolite is strongly different, being basalt crystal-rich (>50 area%) at 30 °C/min and rhyolite glassy even at 0.0167 °C/min.

2. Starting materials, experimental and analytical techniques

2.1. Starting materials

Six different silicate compositions were prepared along the sub-alkaline join basalt–rhyolite. We used natural samples from Iceland (B₁₀₀ = basalt) and Lipari Island (Aeolian Islands, Italy, R₁₀₀ = rhyolite) to prepare glasses of the end-members (Table 1). About 100 g of these two rocks was crushed and then melted in a Pt crucible at 1600 °C in air for 4 h. For quenching, the basalt was poured on a metal plate. Due to its high viscosity the rhyolite could be quenched only inside the crucible by putting it into a water bath. The two recovered glasses were crushed again and re-melted twice at the same conditions, with an intermediate grinding step, to obtain chemically homogeneous glass

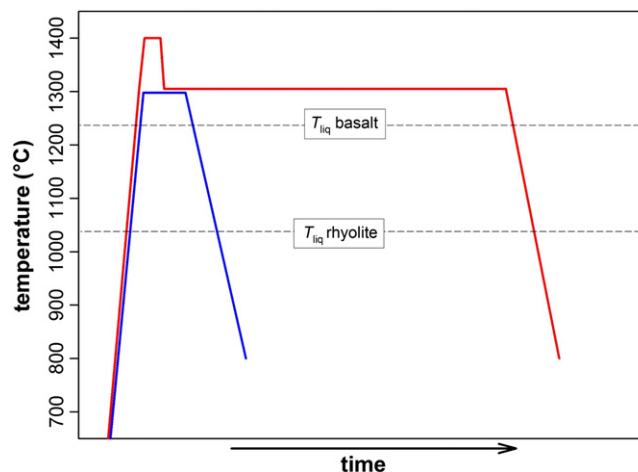


Fig. 1. Schematic thermal paths used in this study; it has been especially drawn to highlight the different heat treatments of experiment S1 (red path) compared with E1, E2, E3, E4 and E5 (blue path) before cooling. The liquidus temperatures of B₁₀₀ and R₁₀₀ end-member melt compositions are also reported.

materials. About 70 g of each glass was crushed and sieved to obtain 1:1 mixtures by weight of two different grain size ranges (<200 and 200–500 μm); such mixture minimizes the number of small bubbles after subsequent re-melting. The two powders were then mixed to produce the four intermediate compositions (B₈₀R₂₀, B₆₀R₄₀, B₄₀R₆₀, B₂₀R₈₀). Each of these batches was then melted at 1600 °C for 4 h in air and rapidly quenched (B₈₀R₂₀ and B₆₀R₄₀ were poured on a metal plate, B₄₀R₆₀ and B₂₀R₈₀ were cooled within the crucible in the water bath). The procedure was repeated twice with intermediate grinding to improve homogeneity. Thus, six homogeneous glass starting materials with systematic chemical variations were obtained. Several glass fragments of each starting composition were analysed by EPMA (see below); the average compositions of the glasses are reported in Table 1 and classified according to TAS (Le Maitre et al., 2002). Several other glass fragments were used to quantify the H₂O amount by FTIR spectroscopy, and the iron speciation by a modified Wilson's method (see below).

2.2. Experimental strategy and techniques

The effect of cooling rate was systematically investigated using rates of 0.0167 (E1–E2), 3 (E3) and 30 (E4–E5) °C/min between 1300 and 800 °C. Afterwards the samples were rapidly quenched. In experiments E1, E2 and E3 all six compositions were used while the highest cooling rate (in E4 and E5) was only applied to B₁₀₀ (Table 2). For all experiments labelled with E, the heating rate was 7 °C/min up to 1300 °C and then followed by a dwell time of 120 min at 1300 °C. To characterize ex-situ the physico-chemical state of all the six melts at the end of this pre-heating, we performed an additional experiment in which the samples were directly quenched from 1300 °C to room temperature in water (experiment E0). In order to investigate the role of pre-heating, an initial heating for 30 min at 1400 °C was followed by a long dwell of 40 h at 1300 °C using a cooling rate of 3 °C/min (experiment S1). The reproducibility of the experiments was checked by duplicates at cooling rates of 0.0167 and 30 °C/min (E1–E2 and E4–E5).

To perform the 32 experiments, mm-sized glass fragments were loaded into Pt tubes (4 mm in diameter and 15–20 mm in length). Each capsule contains ~50 mg of glass chips. All the experiments were run at atmospheric pressure and air redox state.

The experiments E1, E2, E3 and S1 were performed at the Dipartimento di Ingegneria e Geologia (University G. d'Annunzio, Chieti, Italy), in a vertical rapid-quench Protherm PTF16/50/450 furnace equipped with a EURO THERM controller. The temperature was

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