ARTICLE IN PRESS

VOLGEO-06325; No of Pages 8

Journal of Volcanology and Geothermal Research xxx (2018) xxx–xxx

Contents lists available at ScienceDirect

Journal of Volcanology and Geothermal Research

journal homepage: www.elsevier.com/locate/jvolgeores

Volatile contents of primitive bubble-bearing melt inclusions from Klyuchevskoy volcano, Kamchatka: Comparison of volatile contents determined by mass-balance versus experimental homogenization

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ARTICLE INFO

Article history:
Received 31 October 2017
Received in revised form 9 March 2018
Accepted 12 March 2018
Available online xxxx

Keywords:
Melt inclusion
Raman
Volcanic degassing
Shrinkage bubble
Volatiles
Experimental rehydration

ABSTRACT

Primitive olivine-hosted melt inclusions provide information concerning the pre-eruptive volatile contents of silicate melts, but compositional changes associated with post-entrapment processes (PEP) sometimes complicate their interpretation. In particular, crystallization of the host phase along the wall of the melt inclusion and diffusion of H^+ through the host promote CO_2 and potentially S or other volatiles to exsolve from the melt into a separate fluid phase. Experimental rehomogenization and analysis of MI, or a combination of Raman spectroscopy, numerical modeling, and mass balance calculations are potentially effective methods to account for PEP and restore the original volatile contents of melt inclusions. In order to compare these different approaches, we studied melt inclusions from a suite of samples from Klyuchevskoy volcano (Kamchatka Arc) for which volatile compositions have been determined using experimental rehydration, Raman spectroscopy, and numerical modeling. The maximum CO_2 contents of melt inclusions are in agreement (~3600–4000 ppm), regardless of the method used to correct for CO_2 in the bubble, but significantly more uncertainty is observed using mass balance calculations. This uncertainty is largely due to the lack of precision associated with the petrographic method of determining bubble volumes and may also be related to the presence of daughter minerals at the glass-bubble interface.

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1. Introduction

Information concerning the pre-eruptive volatile contents of magmas provides important constraints on local volcanic processes and global cycling of various elements in the Earth system. For example, the pre-eruptive concentrations of CO_2 and H_2O in the melt affect the depth and intensity of volcanic degassing and the explosivity of volcanic eruptions (Metrich and Wallace, 2008). Mantle temperatures can be estimated based on the H_2O concentration in the melt (Sobolev and Danyushevsky, 1994; Portnyagin et al., 2007; Gazel et al., 2012), and the CO_2 content of a melt may be related to the composition of the source lithology (e.g. anhydrous vs carbonated peridotite). The CO_2 concentrations of early-forming melts also have implications regarding the amount of CO_2 subducted into the mantle (e.g. Wallace, 2005), how deep carbon-bearing phases are subducted (Dasgupta, 2013), and how much subducted carbon eventually outgasses into the atmosphere (Burton et al., 2013). Much of our knowledge about magmatic volatile budgets comes from remote sensing and in situ sampling at active volcanoes (Burton et al., 2013). While these methods are effective for active volcanic systems, they cannot be applied to extinct or dormant volcanic systems. Furthermore, studies of diffuse degassing (e.g. Chiodini et al., 2004) suggest that volatile fluxes from a single point-source may significantly under-estimate the total volcanic degassing flux. As an alternative, melt inclusions preserve samples of pre-eruptive melt and provide a valuable tool for determining the volatile contents and degassing behavior of magmas (Roedder, 1979; Roedder, 1984).

Although melt inclusions can be a robust source of information, various post-entrapment processes (PEP) can modify the composition of the melt inclusions (e.g. glass, fluid) and complicate the interpretation of melt inclusion data to determine the volatile budget of the melt that was trapped in the inclusion. As a result, it is often difficult to determine whether compositional variations within a group of (presumably) coeval melt inclusions reflect local variations in melt chemistry during trapping or reflect processes that have occurred after the melt inclusion formed. For example, when a melt inclusion is trapped, post-entrapment crystallization (PEC) leads to depletion of elements that

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https://doi.org/10.1016/j.jvolgeores.2018.03.007
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Please cite this article as: Moore, L.R., et al., Volatile contents of primitive bubble-bearing melt inclusions from Klyuchevskoy volcano, Kamchatka: Comparison of volatile content..., J. Volcanol. Geotherm. Res. (2018), https://doi.org/10.1016/j.jvolgeores.2018.03.007

are compatible in the host mineral (Roedder, 1979; Danyushevsky et al., 2002). Furthermore, because the relative change in molar volume (or density) of the host mineral is less than that of the melt during cooling, the volume change associated with crystallization results in the formation of a “shrinkage bubble,” depressurization within the inclusion, and degassing of volatile components (particularly CO_2) into the bubble (Roedder, 1979; Esposito et al., 2011; Moore et al., 2015; Aster et al., 2016). Additionally, it has also been shown that H_2O can be lost from olivine-hosted melt inclusions as the inclusion cools (Roedder, 1979; Sobolev and Danyushevsky, 1994) as a result of diffusion of H^+ across point defects in the host mineral (Mironov and Portnyagin, 2011; Gaetani et al., 2012). Thus, it is necessary to correct the volatile concentrations of melt inclusions to obtain the original concentration in the trapped melt, and a range of experimental and numerical methods have been used to do this. These include reversing changes that occurred during cooling experimentally by re-heating the melt inclusion and using a combination of microanalytical techniques, numerical modeling, and mass balance calculations to reconstruct the bulk composition of the trapped melt.

In the experimental approach, melt inclusions are heated and homogenized (dissolution of all solid and volatile phases to produce a homogeneous melt/glass, in ideal cases) under controlled temperature, pressure, and oxygen fugacity. This may include optical monitoring of the melt inclusion during heating on a microscope-mounted heating stage, heating in a tube furnace at one atmosphere, or heating in either a cold-seal or internally-heated pressure vessel (Student and Bodnar, 1999). While experimental homogenization works well for inclusions trapped at temperatures less than ~1000 °C and hosted in quartz (Bodnar and Student, 2006), melt inclusions trapped at higher temperatures and hosted in olivine and some other phases are often problematic – especially for inclusions that are relatively H_2O-rich (see Esposito et al., 2012). It has been shown that melt inclusions lose more H_2O during longer heating experiments (Massare et al., 2002; Severs et al., 2007; Bucholz et al., 2013). The change in density of the melt resulting from H^+ diffusion causes depressurization to occur, promotes the formation of shrinkage bubbles, and results in a homogenization temperature that exceeds the original trapping temperature (Danyushevsky et al., 2002). Thus, a consequence of H^+ diffusion is that H_2O-rich olivine-hosted melt inclusions often contain a bubble after 1 atm reheating experiments, and overheating the inclusion beyond the trapping temperature would compromise the composition of the inclusion by dissolving excess olivine into the melt. To solve this problem, Mironov et al. (2015) describe a method in which melt inclusions are heated in a pressure vessel in the presence of a hydrous glass under conditions similar to those presumed to be present when the melt inclusions were trapped (Mironov and Portnyagin, 2011). Because of the experimentally-generated water fugacity gradient, H_2O diffuses into the melt inclusions and rehydrates the melt to its original H_2O content, and samples are rapidly quenched (~150 °C/s) to prevent diffusive loss of H_2O following the experiment. As a result, CO_2 and other volatiles dissolve back into the melt at the temperature of trapping and do not require overheating.

As an alternative to the experimental approach, it is sometimes desirable to reconstruct the bulk compositions of melt inclusions without reheating them, such as when there is a need to avoid damaging a precious sample (e.g. Harvey and McSween, 1992; Goodrich et al., 2013), to preserve chemical gradients that record information about kinetically-limited processes (e.g. Newcombe et al., 2014), or because the equipment required for controlled heating experiments is unavailable. In these cases, the composition of the trapped melt can be reconstructed by determining the compositions and relative proportions of the various phases in the inclusion and estimating the bulk composition of the melt inclusion using a mass balance approach. Then, a numerical approach may be used to account for the effects of post-entrapment crystallization by incrementally adding host phase back into the melt until the calculated composition of the melt is in equilibrium with the host. This method works best with samples erupted as fine-grained tephras because H_2O loss is limited by the relatively rapid cooling (Lloyd et al., 2013) and inclusions that contain only glass $\pm$ vapor. It has been demonstrated in several recent studies that the CO_2 content of glassy, bubble-bearing melt inclusions can be determined based on Raman analysis (Esposito et al., 2011; Hartley et al., 2014; Moore et al., 2015; Aster et al., 2016) or cryometric analysis (Naumov et al., 2006) of the vapor bubble combined with other in situ microbeam analyses to determine the major, trace, and volatile composition of the glass. Additionally, the composition and density of the fluid exsolved into the bubble over the cooling interval between trapping and eruption can be estimated by numerical modeling (e.g. Anderson and Brown, 1993; Wallace et al., 2015; Aster et al., 2016).

Because of the benefits listed above, the approach of using mass-balance calculations to restore the CO_2 contents of unheated melt inclusions erupted in tephras is gaining acceptance, but there are a few notable disadvantages associated with this approach. For example, uncertainties incurred by mass-balance calculations associated with the method are not well understood. Previous studies have reported that minerals containing C, H, S, F, and Cl commonly form at the glass-vapor interface in melt inclusions (e.g. Kamenetsky et al., 2002; Esposito et al., 2016), but these minerals are rarely considered in studies to determine the composition of the vapor phase and/or the volatile content of melt inclusions (Kamenetsky et al., 2007; Moore et al., 2015; Esposito et al., 2016). Additionally, restricting sampling to fresh tephras and lavas with rapidly-quenched inclusions limits the availability, quality, and representativeness of sample material. For these reasons, it is useful to compare the compositions of unheated melt inclusions to inclusions that have been experimentally treated, but there are few studies have directly compared results from the Raman mass-balance approach with compositions determined after experimental homogenization (e.g. Wallace et al., 2015). To explore the relative merits of both approaches, we used Raman analyses and a mass balance approach following the methods described by Moore et al. (2015) to analyze melt inclusions from a suite of samples from the Klyuchevskoy volcano (Kamchatka). Previous studies (Mironov and Portnyagin, 2011; Mironov et al., 2015) determined the compositions of these same and similar melt inclusions after experimentally rehydrating and homogenizing the inclusions. We also use the method described by Wallace et al. (2015) to numerically estimate the amount of CO_2 exsolved into the bubbles.

2. Sample description

The melt inclusions analyzed in this study are hosted by olivine (Fo > 84) from lava and tephra samples from the eruption that formed the ~3 ka Bulochka cinder cone (V. Ponomavera, personal communication) on the flank of Klyuchevskoy volcano in the Kamchatka arc. The inclusions in this study are separated into three groups according to their host lithology and method of study: 1) unheated (as found) inclusions in olivines that had been separated from tephra samples, 2) recrystallized melt inclusions in olivine from a lava flow that were heated at 1 atm under dry conditions (Mironov and Portnyagin, 2011), and 3) inclusions from the same lava flow that were heated at ≥300 MPa in the presence of a hydrous glass (Mironov et al., 2015). Hereafter, these samples are referred to as *unheated*, *dry reheated*, and *experimentally rehydrated*, respectively. The proportion of CO_2 contained in the bubble was determined for all three groups using Raman spectroscopy. We present new analyses of the inclusion glass in the unheated group only; previously reported glass compositions are used for dry reheated and experimentally rehydrated groups (Mironov and Portnyagin, 2011 and Mironov et al., 2015 respectively).

Melt inclusions from the *unheated* tephra samples were prepared by polishing olivine crystals to expose the glass without breaching the bubble, as described by Moore et al. (2015). The splitting of the Fermi diad (Δ, cm^{-1}) could be quantified in approximately 95% of the inclusions analyzed. Vapor bubbles were analyzed by Raman spectroscopy in the

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