Understanding a volcano through a droplet: A melt inclusion approach

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ABSTRACT

This review paper is intended to be a guideline to novices on how to conduct research on silicate melt inclusions in volcanic environments, which analytical techniques are more suitable to gather the desired data and the major pitfalls scientist may encounter. Silicate melt inclusions (SMIs) are small quantities of silicate melt that are trapped in minerals during their growth or crystallization. They contain liquids formed in equilibrium with their host minerals and therefore record the liquid line of descent of magmatic systems. Upon trapping, SMIs become ideally closed to the surrounding environment, and behave as time capsules, giving important information about processes that originated magmas and the nature of their mantle source. A melt inclusions investigation is composed of several steps: (1) a detailed petrographic study to characterize and select representative SMIs, with the aim of identifying Melt Inclusions Assemblages (MIA), the only reliable tool to assess that SMIs obey Roedder's rules and have not re-equilibrated following entrapment; (2) a careful preparation of samples for re-heating experiments and microanalysis; (3) high temperature studies in order to homogenize the SMIs and be able to perform chemical analysis; (4) interpretation of SMIs data, which should always be compared with bulk rock composition and relevant experimentally derived liquid compositions. We suggest that the search for good SMI candidates for study will be achieved by good petrographic analysis of SMIs and detailed petrographic information (size, shape, appearance, position relative to the host and other SMI within the same host). If the goal of a SMIs study is to determine volatile concentrations, assessing the relative time of trapping among SMIs is of paramount importance, as it allows understanding if the variation in volatile concentrations is consistent with a certain magmatic physical–chemical process. Researchers that choose to work with SMIs face several challenges. SMIs are not always hosted in all samples or they can be difficult to recognize (mostly due to their small size). Additionally, due several processes, such as post-entrapment crystallization, boundary layer and sampling melt heterogeneity at the micron scale, they may not be representative of the original trapped melt composition.

1. Introduction

Silicate Melt Inclusions (SMIs) are small droplets of silicate melt, typically 1–100 μm in size, that are trapped in surface irregularities or defects of crystals during their growth in a magma body (Sorby, 1858). They typically contain variable amount of crystals, glass and/or vapor, are very common and usually quite easy to identify in volcanic rocks. Upon trapping in their host crystals, SMIs behave as closed and isolated systems, retaining much of the original composition of the melt (including its volatile content). SMIs are then like time capsules, and can record the pre-eruptive volatile content that usually is lost during degassing or magma differentiation, and allow researchers to trace the evolution of magma from its formation at mantle depth, to its release to the surface, step by step. Therefore, SMIs are an ideal tool to shed light on igneous processes such as fractional crystallization, magma mixing, and fluid exsolution (Kent, 2008 and references therein; Métrich and Wallace, 2008). Some challenges persist for those who choose to work with SMIs: inclusions are not always hosted in all samples or they can be difficult to recognize (mostly due to their small size), and they may not be representative of the original trapped composition due to boundary layer processes and sampling melt heterogeneity at the micron scale. Even when SMIs are representative of the surrounding melt at the moment of trapping, post-entrapment processes may modify or compromise the initial composition. The purpose of this review is mostly propaedeutic; it is meant to be an introduction for beginners to the wonderful world of SMIs, a description of methods to use SMIs in igneous petrology studies, and a guideline on how to conduct a correct SMI study in volcanic rocks while avoiding common mistakes.

2. History of SMIs research

Silicate melt inclusions (SMIs) have been studied for around 160 years, starting with the pioneering works of Sorby (1858) and Zirkel

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(1873), who mainly described SMIs, and tried to give geologic interpretations of their observations. In their studies however, both Sorby and Zirkel provide detailed descriptions and drawings of SMIs and fluid inclusions, recognizing SMIs as a potential tool for the understanding of the origin of igneous rocks. We would have to wait almost a century for the first studies of SMIs in igneous phenocrysts, because the majority of the scientists at that time concentrated more on the applications of fluid inclusions for studies of economic geology (Roedder, 1984). Barrabé and Deicha (1956) were the first to homogenize quartz-hosted melt inclusions by heating at high temperatures; a fundamental contribution in this field was given by the "Russian School" with the development of the heating stage by Yermakov (1950), and the implementation by Dolgov and Bazarov (1965) that led to the "Vernadsky Stage" (Sobolev et al., 1980).

Around 1970, some fluid inclusionists started to apply SMIs to the study of volcanic rocks (Sobolev and Kostyuk, 1975) and lunar samples (Roedder and Weiblen, 1970), while others concentrated on their possible origin and methods to investigate them (Clocchiatti, 1975; Roedder, 1979). In the early 1990s, several scientists focused their research on SMIs applied to volcanic systems, discussing their role in determining the pre-eruptive volatile content of magmas (Roedder, 1992; Johnson et al., 1994; Lowenstern, 1994), describing the information they can provide on plutonic (Touret and Frezzotti, 1993) and magmatic (Lowenstern, 1995; Sobolev, 1996) environments.

At that time, a boost in development of high-precision bulk techniques lead to a lack of micro-analytical techniques for in situ SMIs investigations; before the 1990s, it was not possible to have reliable analyses of trace elements and volatiles in SMIs, and as a result the use of SMIs for volcanological and petrological investigations was scarce. Starting in 1980, new analytical techniques on bulk rock samples, such as X-ray fluorescence (XRF) and instrumental neutron-activation analysis (INAA), allowed researchers to investigate trace elements and isotopes in terrestrial and planetary samples. Researchers were able to correlate each magma type and environment with their unique trace elements and isotopic composition, and to incorporate this information with the new developed plate tectonics theory.

With the development of micro-analytical techniques in the late 1990s, the number of SMI studies increased exponentially and scientists became convinced that SMIs could be very useful and reliable tools in constraining processes in magmatic systems (Table 1). Some authors have devoted their studies to minimizing some of the critical points concerning the use of SMIs, such as the variations in SMIs compositions (Anderson et al., 1989; Skirius, 1990; Dunbar and Hervig, 1992; Hervig and Dunbar, 1992; Sisson and Layne, 1993; Métrich et al., 1999; Severs et al., 2007), the boundary-layer phenomena (Lu et al., 1995; Danyushevsky et al., 2004; Kuzmin and Sobolev, 2004; Zajacz and Halter, 2007; Severs et al., 2009), and the post-entrapment changes (Danyushevsky et al., 2002; Gaetani and Watson, 2002; Baker, 2008; Kent, 2008; Steele-Macinnis et al., 2011).

From the early 1990s, several researchers focused their studies on the role that volatiles play in the determination of the eruptive style of volcanic systems. Multiple studies were published regarding the reliability of volcanic glass and bulk rocks in recording the pre-eruptive volatile content of a melt, mostly due to the loss of volatiles during the rising and eruption of the magma at the surface (Dixon et al., 1995). SMIs, however, represented a portion of the melt that behaves as closed systems (unlike bulk rocks) by preserving the pre-eruptive volatile composition of the magma. With the improvement of micro-analytical techniques, SMIs could be directly analyzed, especially by Fourier Transform InfraRed spectroscopy (FTIR) and Secondary Ion Mass Spectrometry

Table 1
Summary of papers about melt inclusions in volcanic systems from Italy and review papers.

References/year	Type/area of study	Host(s)	Methods					Results		
			Heating	EMPA	SIMS	LAICPMS	Raman/FTIR	P–T conditions	Melt evolution	Volatiles
Vaggelli et al. (1993)	Vesuvius	cpx, fsp	X	X					X	X
Belkin et al. (1998)		cpx, fsp	X	X	X			X	X	X
Cioni et al. (1998)		cpx, fsp	X	X					X	
Lima et al. (1999)		cpx, fsp	X	X				X	X	X
Raia et al. (2000)		cpx	X	X				X	X	X
Webster et al. (2001)		cpx	X	X	X			X	X	X
Fulignati et al. (2004)		ol, fsp	X	X			X	X	X	X
Schiano et al. (2004)		ol		X	X				X	
Fulignati and Marianelli (2007)		cpx, fsp		X			X	X		X
Lima et al. (2007)		ol, cpx	X	X					X	
Balcone-Boissard et al. (2008)		cpx, fsp		X				X		X
Balcone-Boissard et al. (2012)		cpx, fsp		X			X	X		X
Klébesz et al. (2012)		cpx	X	X		X			X	
Cecchetti et al. (2002)	Campi Flegrei	cpx		X			X	X	X	X
Roach (2005)		cpx, fsp		X			X	X	X	X
Cannatelli et al. (2007)		ol, cpx	X	X	X			X	X	X
Mangiacapra et al. (2008)		ol, cpx		X			X	X	X	X
Arienzo et al. (2010)		cpx		X			X	X	X	X
Esposito et al. (2011)		ol		X	X			X	X	X
Mormone et al. (2011)		ol		X			X	X	X	X
Cannatelli (2012)		ol, cpx	X	X				X	X	
Fourmentraux et al. (2012)		cpx		X			X	X	X	X
Métrich and Clocchiatti (1989)	Etna	variable		X					X	
Spilliaert et al. (2006)		ol		X	X			X	X	X
Collins et al. (2009)		ol		X	X	X		X		X
Gioncada et al. (1998)	Aeolian Islands	ol, cpx	X	X	X		X	X	X	X
Doherty et al. (2012)		ol	X	X					X	
Lowenstern (1995)	Review		X	X	X		X			
Frezzotti (2001)			X	X	X	X	X			
De Vivo and Bodnar (2003)			X	X	X	X	X			
Lowenstern (2003)			X	X	X		X			
Wallace (2005)				X	X		X			
Bodnar and Student (2006)			X							
Pettke (2006)						X				
Métrich and Wallace (2008)										

(SIMS). Researchers were then able to determine volatile content (H_2O, CO_2, Cl, F, S, Br) in SMIs and bring new insights into igneous processes (Roedder, 1979; Hauri et al., 2002; De Vivo and Bodnar, 2003; Lowenstern, 2003; Webster et al., 2003a,b; Wallace, 2005; Bodnar and Student, 2006; Audétat and Lowenstern, 2014).

3. How to conduct a SMIs study

### 3.1. The petrographic approach — how to identify and classify SMIs in volcanic rocks

In volcanic rocks, SMIs often consist of glass $\pm$ one or more gas bubbles $\pm$ daughter mineral phases (Fig. 1a–f). There have been several studies regarding the mechanisms of formation of crystallized SMIs (Roedder, 1979, 1984; Lowenstern, 1995; Student and Bodnar, 1999; Frezzotti, 2001; Kamenetsky and Kamenetsky, 2009) and all point out that for any given inclusion size, the slower the cooling rate, the more likely it will be that the melt in the inclusions will recrystallize (Fig. 2). Furthermore, for a given cooling rate, big inclusions crystallize faster than small ones. Student and Bodnar (1999) noted that the composition of the melt also affects the crystallization process, suggesting that H_2O-saturated melts may promote crystallization during cooling.

To establish whether the melt surrounding the host was saturated in volatiles at the moment of trapping, the presence of a bubble and the bubble volume/SMI volume ratio is a first important observation (Roedder, 1979). If the bubble/SMI volume ratios of SMIs, presumably trapped at the same time, is highly variable [e.g., from 1 (fluid inclusion *sensu strictu*) to 0 (bubble-free SMI)], it is likely that the melt was volatile saturated. In volatile-poor melts, it is common to observe a single "shrinkage" bubble, although some inclusions appear completely glassy (Fig. 1d); in volatile-rich melts, a fluid may be present inside the bubble (Kamenetsky et al., 2007; Esposito et al., 2011; Hartley et al., 2014;
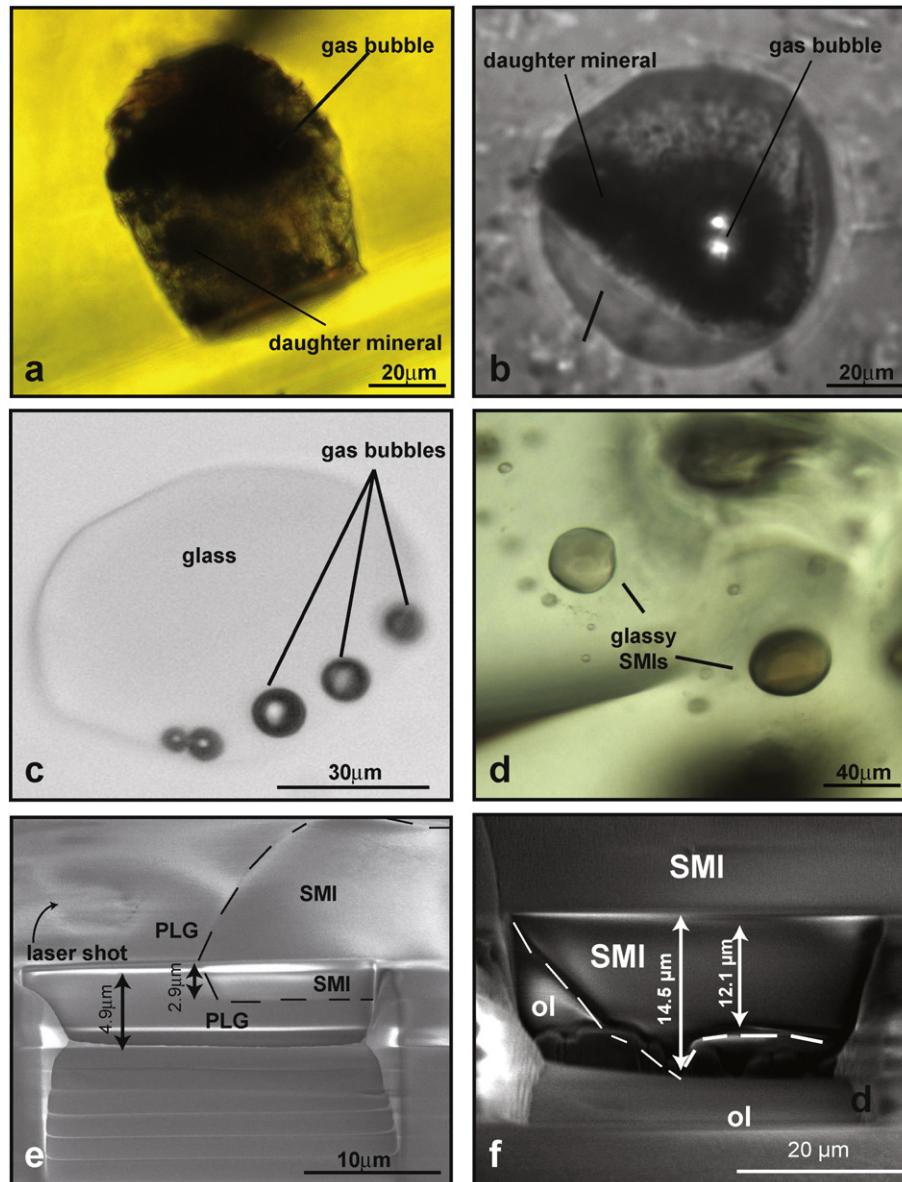
Fig. 1. Silicate Melt Inclusions (SMIs) hosted in volcanic phenocrysts. a) Re-crystallized SMI (glass + bubbles + daughter minerals), hosted in a pyroxene phenocryst from Fondo Riccio scoriae (Phlegraean Fields, Italy); b) re-crystallized SMI (glass + bubbles + daughter minerals), hosted in a pyroxene phenocryst from Minopoli 1 scoriae (Phlegraean Fields, Italy); c) Bubble bearing SMI hosted in feldspars from Bacoli pyroclastics (Phlegraean Fields, Italy); d) Bubble bearing SMI hosted in olivine from Solchiaro scoriae (Phlegraean Fields, Italy); e) Focused ion beam electron microscopic (FIB- EM) image of a plagioclase (PLG) hosted SMI from White Island. The dashed line shows the MI/host interface, note the regularity of the MI/host boundary in the vertical section (modified from Esposito et al., 2014). f) Focused ion beam electron microscopic (FIB-EM) image of an olivine (Ol) hosted SMI from Solchiaro (Phlegraean Fields, Italy). The dashed line shows the MI/host interface, note the irregularity of the MI/host boundary (modified from Esposito et al., 2014).
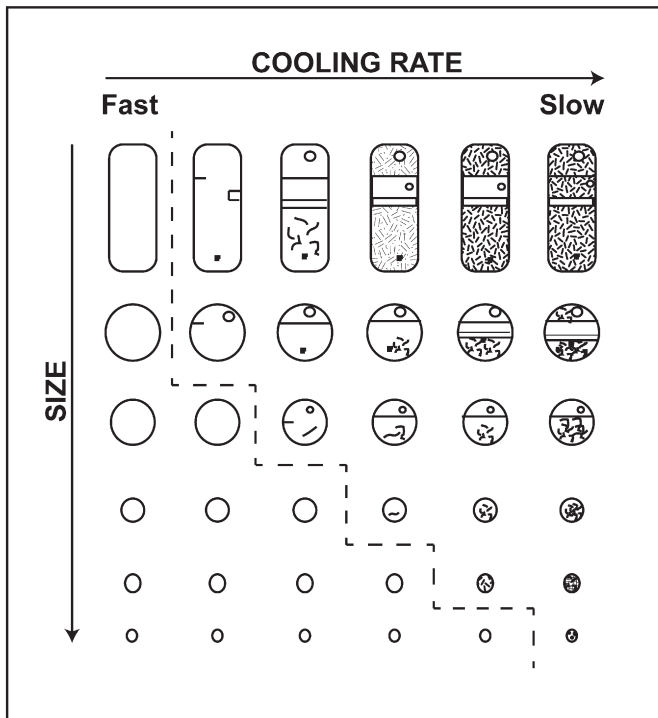
Fig. 2. Schematic diagram showing the effects of cooling rate and size on the nucleation and growth of phases in silicate melt inclusions. Modified after Roedder (1984).

Fig. 3. a) Example of leaked SMI from Minopoli 2 scoriae (Phlegraean Fields, Italy); b) decrepitated SMI hosted in sanidine from Senga (Phlegraean Fields, Italy). Notice the irregular shape of the inclusion.

Moore et al., 2015), which is often detectable using Raman Spectroscopy. However, due to the low vapor density often present in these bubbles, it may be difficult to detect. This problem will be discussed again, later in this review.

While in the past some researchers have generally eschewed studying the SMI bubbles or inclusions containing bubbles (Lowenstern, 1994; Wallace et al., 1999; Helo et al., 2011; Lloyd et al., 2013) due to the belief that they would complicate the interpretation of SMI compositions, others investigated differences in composition between bubble-bearing SMI and bubble-free SMI glasses (Esposito et al., 2011). Based on measurements of CO_2 in the glass by SIMS and CO_2 in the vapor bubble by Raman spectroscopy, Esposito et al. (2011) used a mass balance calculation to estimate that 64% of the original dissolved CO_2 in one SMI was stored in its vapor bubble. Recently, many workers have highlighted that bubble/SMI volume ratios, bubble composition and distribution can give crucial information about the original volatile contents of un-degassed relatively deep melt (Moore et al., 2015 and references therein; Wallace et al., 2015). In particular, Hartley et al. (2014) and Moore et al. (2015) reported that in several basaltic samples, the CO_2 contained in the bubble could account for up to 90% of the total SMI budget.

What is the origin of bubbles contained in SMIs? SMIs may contain a bubble due to heterogeneous trapping of a volatile-saturated melt (silicate melt plus vapor), or may develop a bubble after trapping. Different processes can determine the nucleation of a bubble after trapping of a SMI. Firstly, a bubble can form by differential thermal contraction between the melt and the host during cooling of a homogenous melt (the so-called "shrinkage bubble", Lowenstern, 1994). Secondly, crystallization of the host at the host/SMI interface can occur because of cooling after trapping, known in literature as post-entrapment crystallization (PEC, Danyushevsky et al., 2002). For silicate minerals, if the trapped melt is volatile-rich, the crystallizing phase at the host/SMI interface is always denser than the silicate liquid, providing space for nucleation of a vapor bubble (Steele-Macinnis et al., 2011). Thirdly, a bubble can form because of decrepitation and/or leakage of a SMI (Fig. 3a–b). Finally, a SMI may nucleate a bubble if element such H
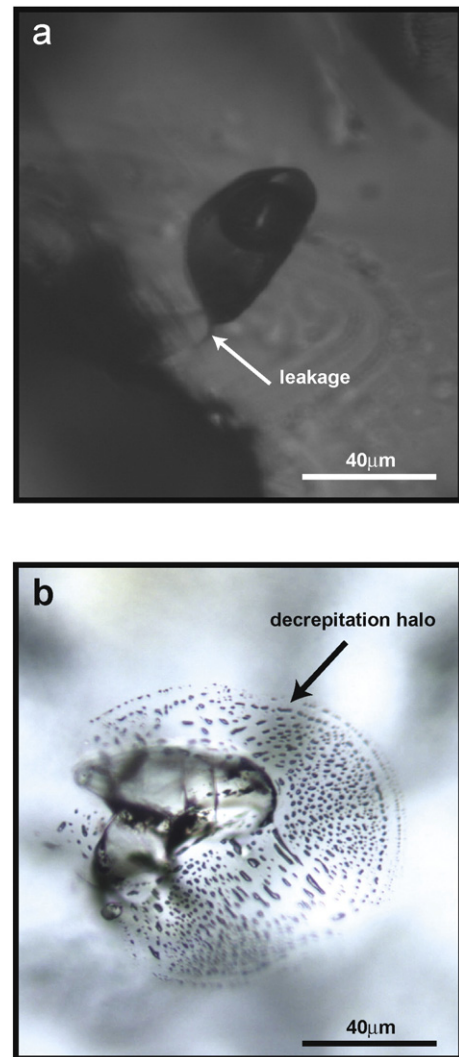
diffuses out from the SMI through the host as recently suggested by Bucholz et al. (2013). In general, small SMIs do not contain bubbles, probably due to surface-tension effects (Roedder, 1979). A bubble has more time to nucleate in a SMI when the cooling rate of the host crystal is slow, to offset the increase in the internal pressure of the SMI. Slowly-cooled rocks often display highly fractured crystals with SMIs containing large bubbles, probably due to the decrepitation of SMI during eruption (Best and Christiansen, 1997).

In 1984, Roedder defined SMIs following the categorization he had given to FIs, i.e. primary, pseudo-secondary and secondary inclusions. A silicate liquid can be trapped as primary SMIs when it is present during the crystal growth, as pseudo-secondary SMIs through fractures that heal before the crystal has stopped growing, and as secondary SMIs through fractures that heal after complete growth of the crystal. It is widely accepted in the igneous petrology community that both primary and pseudo secondary SMIs can be considered "primary", as they were trapped during the growth of the crystals and behaved as time capsules, recording the composition of the silicate liquid during its evolution. However, it is difficult to imagine the mechanism of trapping of secondary SMIs along fractures in volcanic phenocrysts, mostly because in general the intruding silicate phase has higher viscosity than a fluid phase.

Sometimes, the incomplete trap of melt in the host crystal causes the SMIs to stay open during decompression and cooling, by being connected

to the outside of the host only by a narrow capillary (filled by melt or vapor phases that can escape from such inclusions). The resultant leaked inclusions are called "hourglass inclusions" or embayments (Fig. 4a–c) and offer a unique opportunity to characterize the behavior of gases during magma ascent (Anderson, 1991; Liu et al., 2007; Humphreys et al., 2008; Lloyd et al., 2014). However, they should be avoided for other types of studies, as they do not retain the original volatile contents of the melt at the time of trapping.

3.2. How to select representative SMIs

A common characteristic of SMIs is that they show generally a range in composition (major, minor, trace elements and volatiles). In order to decipher and interpret the geochemical variability in a SMI study, the most important consideration is the selection of which SMIs are most suitable for your study, whether that be a study of volcanic processes or other SMI study. In this respect, if an inclusionist wants to assert that an SMI records a reliable composition, it is fundamental to assess the timing of the inclusion entrapment with respect to the host crystal, and the relative formation of the host crystal with respect to the paragenesis of the rock (i.e. whether the host a phenocryst, antecryst, or xenocryst; for the purpose of this discussion, we describe all mineral hosts as "phenocrysts": Doherty et al., 2012). Indeed, the study of igneous processes such as magmatic crystallization need to take into account the temporal classification of SMIs (Cannatelli, 2012).

Inclusions that are formed while the crystals are growing (i.e. primary inclusions) are not always are representative of the original chemical composition because of the possibility that during crystallization they may have re-equilibrated with the host. It is very common to observe isolated or randomly distributed SMIs in volcanic phenocrysts; in this case it is very difficult to associate them to the growth profile of their host. The initial screening for SMIs then needs to be performed using polished thin and thick sections.

Similarly to the definition of Fluid Inclusions Assemblage (FIA; Goldstein and Reynolds, 1994), Bodnar and Student (2006) and Esposito et al. (2014) have suggested to divide SMIs in phenocrysts in Melt Inclusions Assemblages (MIAs), exclusively based on
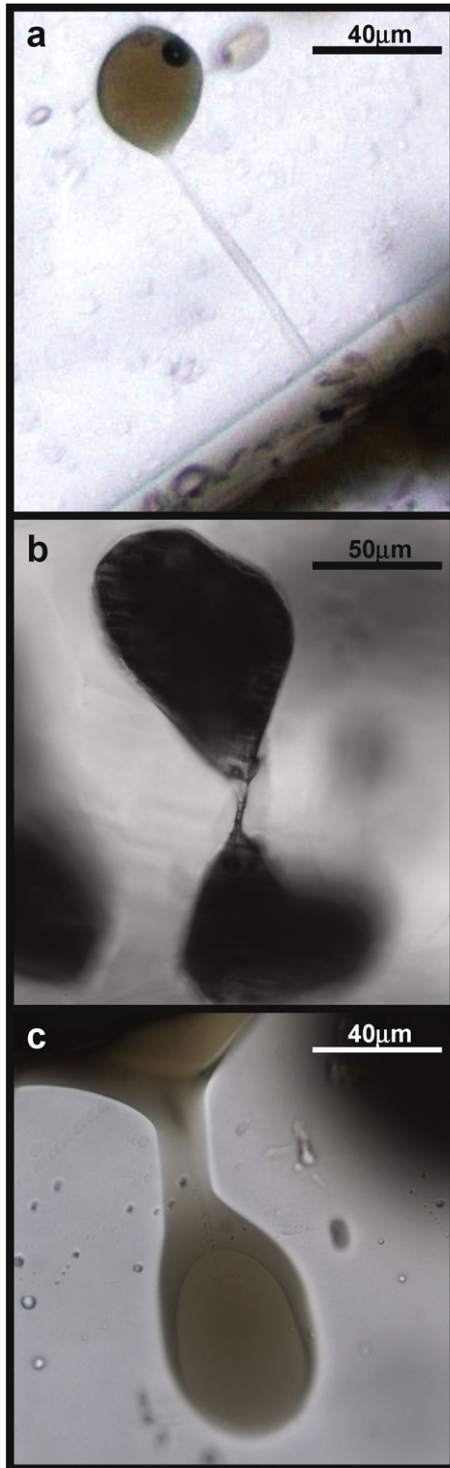
Fig. 4. Examples of bodies of bubble-bearing glass that extend to the crystal rim through a narrow neck, aka hourglass inclusions or embayments. a) Hourglass Inclusions in an olivine crystal from Solchiaro Eruption (Phlegraean Fields, Italy); b) Hourglass Inclusions in a pyroxene crystal from Minopoli 2 eruption (Phlegraean Fields, Italy); c) Hourglass Inclusions in an olivine crystal from Solchiaro Eruption (Phlegraean Fields, Italy).
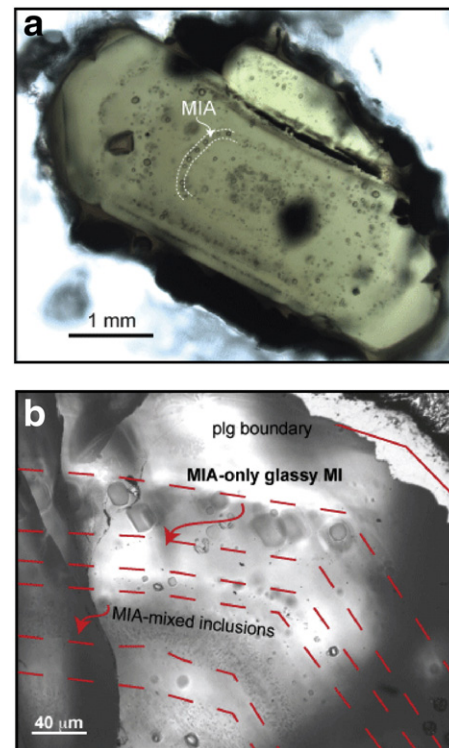
Fig. 5. Melt inclusions Assemblage (MIA). a) Growth zones in a clinopyroxene from White Island scoriae and entrapped SMIs. MIA is highlighted by white dashed curves; b) MIAs hosted in plagioclase from White Island volcano in New Zealand (reflective optical microscope photo). MIAs are marked by the red dashed curves and are parallel to the euhedral boundaries of the phenocryst.

petrographic observations. MIA will be therefore composed by a group of SMIs that were trapped at the same time, same temperature and pressure and from a melt with the same composition (Fig. 5a–b). MIAs can be used to study the evolution of a volcanic system when we have proof that they represent the composition of the melt at the time of trapping and they have not re-equilibrated. To test this hypothesis, we need to be sure that inclusions obey the three Roedder's rules: 1) SMIs must be trapped as a single homogeneous phase; 2) SMIs subsequently behaved as a closed isochoric system; and 3) after trapping nothing has been added to or lost from the SMI. As a crystal grows, different sets of MIAs may be trapped and record information about its crystallization history; when one or more SMI is trapped along the growth zone of a crystal it is easy to group them together in a MIA.

When the relative time of trapping is not possible to assess, it is fundamental to perform a detailed petrographic description of SMI before exposing the SMI to the surface of the host for re-homogenization experiments and microanalysis. The description of SMIs, their size, shape, appearance (e.g., presence of bubble, daughter or co-trapped minerals), is fundamental to better interpret the variability of SMI data (Table 2).

In the past, researchers have been skeptical about using SMIs to investigate igneous processes, such as magma mixing, or degassing-induced crystallization, mainly due to the large variation in measured volatile concentrations from SMIs in volcanic phenocrysts from a single unit. In the last decade, several researchers have devoted their studies to assessing the reliability of data obtained by means of SMIs (Esposito et al., 2014 and references therein), in particular due to the known tendency of SMIs to be modified by post-entrapment processes (Roedder, 1979; Danyushevsky et al., 2002; Kent, 2008 and references therein), to be influenced by boundary layer effects (Baker et al., 2005), or heterogeneity at the micron-scale (Danyushevsky et al., 2004).

3.3. Preparation of samples

In order to perform a SMIs study, rocks needs to be gently crushed and sieved, and phenocrysts handpicked under a binocular microscope. The method of mounting and polishing of the selected SMIs is then dependent on the analytical techniques planned, as different techniques require the SMI-bearing host phenocrysts to be prepared in different ways; this is discussed briefly later in the review. There is no "correct" way of mounting and polishing selected SMI-bearing phenocrysts for study. Each researcher finds the method that works best for them and the materials available in their lab. Some advice, particularly on the mounting and polishing of small crystals can be found in Thomas and Bodnar (2002).

Generally, selected SMI-bearing phenocrysts are mounted on glass slides, or in epoxy mounts and gently ground to produce a flat surface,

which is then polished to a glassy finish using various grades of alumina powder or lapidary polishing disks. The goal of the sample preparation is to obtain the best visibility to describe SMI petrographically, therefore this process may or may not be repeated on the other side of the crystal to produce a double polished wafer (30 μm to 1.5 mm thick) depending on the degrees of transparency inside a phenocryst under the transmitted light of an optical microscope.

A thorough petrographic study is then recommended, where SMIs are divided as being glassy or re-crystallized; re-crystallized SMIs need further investigation in order to determine whether to perform re-heating experiments. Phenocrysts can be removed from the mount by placing them in acetone for 12–24 h, however, once the SMI has been exposed for analysis, the use of acetone should be avoided if possible, particularly if volatile element concentrations are to be studied. This is because, there is the potential for contamination of the volatile analyses (particularly CO_2) due to the precipitation of acetone's volatile components along microfractures on the exposed glassy surface (Esposito et al., 2014). Using a hot needle to release the crystals from the epoxy for remounting at this point is preferable.

3.4. The microthermometry approach

3.4.1. To reheat or not to reheat

To reheat or not to reheat, that is the question. Certain analytical techniques, such as the electron microprobe, require that SMIs are a homogeneous glass in order to obtain representative analysis; a choice of only glassy inclusions though, may result in a biased selection of volatile contents. In other words, if we select only glassy inclusions from a suite of samples that display a wide range of re-crystallized or bubble bearing SMIs, we risk discriminately selecting the range of volatile compositions possible and we may lose important information bearing on the history of the crystal host and the magma around it. It is therefore preferable if there is such variability in SMI textures, to select glassy SMIs, perform re-heating experiments on re-crystallized or bubble bearing SMIs and then analyze all of them in order to compare the range of volatile content of our SMIs.

However, researchers should always keep in mind the possibility that a SMI may undergo a series of "induced" compositional modifications when performing a re-heating experiment (Severs et al., 2007; Gaetani et al., 2012). Olivine hosted SMIs can lose or gain several weight percent of H_2O in a matter of hours (Massare et al., 2002; Portnyagin et al., 2007; Chen et al., 2011), either as a result of re-heating, by rapid diffusion of H^+ and significant proton flux through the host crystal (Mackwell and Kohlstedt, 1990; Aubaud et al., 2004; Demouchy and Mackwell, 2006; Hauri et al., 2006) or after the host has resided into the magmatic reservoir, after the trapping and before the ejection (Bucholz et al., 2013; Lloyd et al., 2013). Careful consideration of the information sought from the analysis must always be made, after a thorough petrographic study. For example, if volatile element contents

Table 2
Example of petrographic descriptions of SMI found in the literature.

Reference	Anderson and Brown (1993)	Esposito et al. (2014)	Moore et al. (2015)	Esposito et al. (2011)	Hartley et al. (2014)
Table ID	Table 1	Table 2	Table 2	Table 2	Table 1
Petrographic description of MI	Host size SMI diameter SMI shape Number of bubbles in SMI	Host type Type of SMI[a] SMI shape Type of MIA[b]	SMI long diameter SMI short diameter Bubble diameter Bubble volume %	SMI dimensions SMI length Number of bubbles in SMI Occurrence of chromite in the SMI	SMI width/depth SMI length SMI volume Bubble in SMI width/depth
	Bubble volume %		Occurrence of daughter minerals	Fe/Mg zoning of the host	Bubble in SMI length
	Occurrence of chromite in the host				Bubble in SMI volume
	Presence of gas inclusions in the host				Bubble volume %

[a] SMI containing only-glass; SMI containing glass + co-trapped mineral; SMI containing glass + vapor + co-trapped minerals; SMI containing glass + vapor + sulfide globule.
[b] MIA composed of only-glass SMIs; MIA composed of SMIs containing glass + co-trapped minerals; MIA composed of all types of SMIs.

are not a focus of study, the presence of a bubble in the SMIs may be of little importance, or if the SMIs are already glassy, there may be no need to fully re-homogenize them.

Unfortunately, SMIs are not always naturally quenched to a homogeneous glass, and SMIs can be affected by various degrees of crystallization after trapping from slightly to strongly crystallized (Fig. 2). In the last few decades much of the work on SMIs was conducted using traditional homogenization experiments, with the aim of obtaining a homogeneous glass for representative micro-analysis. If selected for study, re-crystallized SMIs containing a heterogeneous mixture of crystals and/or glass plus a volatile phase, need to be re-homogenized (i.e. to produce a homogeneous glass), by heating the inclusions back to their homogenization temperature in order to melt the crystalline phases and dissolve the volatiles back into melt. Then the subsequent silicate melt needs to be rapidly cooled (quenched) to form a homogeneous glass (Fig. 6A–H).

In general, the temperatures determined from SMIs homogenization studies are greater than those inferred by analysis of coexisting Fe–Ti oxides, as already hypothesized by Roedder (1979), who suggested that such differences could be attributed to slow diffusion of melt components, the presence of thermal gradients in the stage, or diffusive loss of H_2 or H_2O. Massare et al. (2002) have verified that homogenization studies can provide uncertain data as a result of diffusion of volatiles through the host and stretching of the phenocryst container during laboratory experiments at atmospheric pressure. Danyushevsky et al. (2002) and Sobolev and Danyushevsky (1994) have investigated the changes within the SMIs during reheating due to decrepitation, incorporation of the host mineral or elemental exchange between the SMI and host.
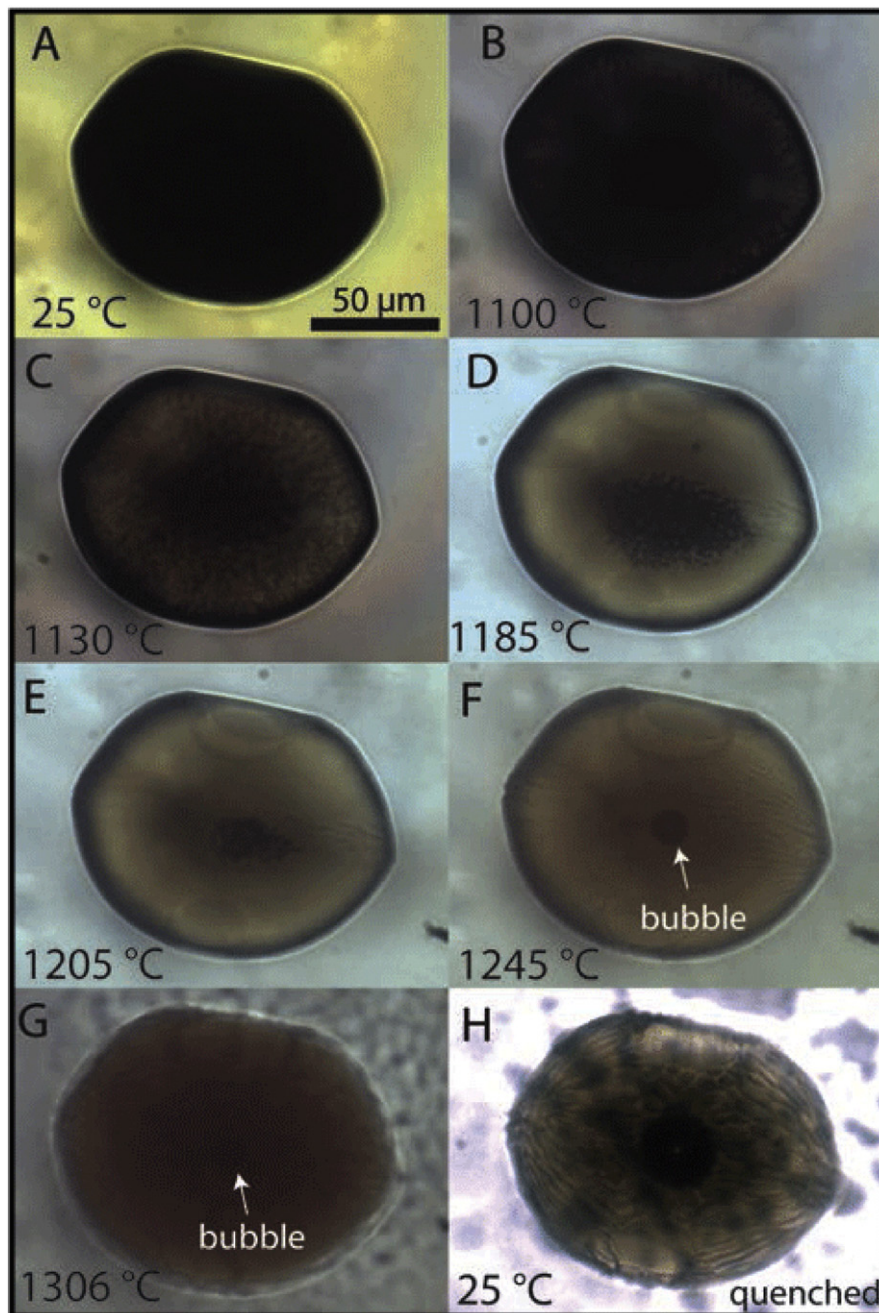
Fig. 6. Re-heating experiment from 25 °C to 1306 °C, performed on an olivine hosted SMI from scoriae of the Solchiaro eruption (Phlegraean Fields, Italy). Details about the re-heating procedure can be found in Esposito et al. (2012).

To avoid reheating the inclusion, some authors have developed methods to detect and quantify volatile elements in the bubble. In a recent experiment, an olivine hosted SMI has been frozen by using a Linkam TS1400XY heating/cooling stage; during the freezing ramp, it was possible to detect a CO_2 rich fluid in the bubble of the SMIs by observing the transition of phases at −57.1 °C. Esposito et al. (2011) and Moore et al. (2015) have used Raman spectroscopy analyses in bubbles of SMIs to quantify the overall CO_2 concentration, with the assumption that the SMI were trapped as a homogeneous silicate liquid phase (i.e., the bubble formed by post-entrapment processes). It is important to note that Moore et al. (2015) estimated that olivine hosted SMI that contain bubbles occupying more than 5% of the volume of the whole SMI, likely were trapped as melt plus vapor (see also Riker, 2005).

If reheating an inclusion is a necessary step, a number of methods can be used, including microscope-mounted high temperature stages, one atmosphere vertical furnaces, High-Pressure Vessels (HPV) and piston cylinder apparatus.

3.4.2. Microscope-mounted high temperature stages

SMIs in igneous phenocrysts can be heated on a Vernadsky Stage (Sobolev et al., 1980), designed to flow He gas over the sample which needs to be deoxygenated by passing it through a tube of powdered Titanium metal heated at 600 °C, in order to prevent oxidation and darkening of iron-bearing phases. The advantage of using the Vernadsky stage is that the SMI can be quenched to room temperature very quickly, from 1500 °C to room temperature in matter of seconds (Cannatelli et al., 2007). This cooling rate is not obtainable with any other heating stage available on the market as far as we know (Esposito et al., 2012).

A newer heating device, the Linkam TS1400XY heating stage, is designed to flow inert gas (He or Ar) through the furnace during heating to prevent oxidation of the sample; the temperature is controlled by a touch-screen system, which also allows the programming heating rates, or "ramps" (Esposito et al., 2012). This "newer" Linkam stage has the advantage, with respect to the older Linkam heating stages, of having a spring mechanism that allows the quick removal of the crystal from the furnace, in order to quickly quench the SMI to room temperature. The advantage of this device over the Vernadsky stage is the possibility of digitally programming several heating ramps during the same experiment and to pause heating at desired temperatures, if needed. Most importantly, the Linkam TS1400XY stage is currently available on the market, while the Vernadsky stage is hard to find for new inclusion laboratories.

There is no universal protocol for choosing a specific heating ramp, the worker may choose their own, however, it is preferable to keep the crystal at a raised temperature for less than 1 h, to minimize the diffusion of volatiles from the SMI during heating. As previously discussed, the migration of H_2O to or from olivine-hosted SMIs can be very quick, a matter of hours (Chen et al., 2011; Massare et al., 2002; Portnyagin et al., 2007). One of the methods used to assess the "ideal" heating rates for a given set of SMIs is to perform kinetic experiments as described by Danyushevsky et al. (2002); the proposed method consists of repeating heating experiments on the same olivine hosted SMI using different heating rates (from fast to slow) and to record the homogenization temperatures. As expected, a positive correlation between the heating rate and the temperature of homogenization is observed (Danyushevsky et al., 2002). Decreasing the heating rate until the temperature of homogenization becomes constant can identify the "correct" heating rate. Based on our personal recent experiments on olivine-hosted SMI, a chosen heating rate of 50 °C/min up to 600 °C, 20 °C/min up to 900 °C and 10 °C/min up to 1175 °C allowed us to perform complete homogenization of the SMIs. It is important however that an appropriate temperature step and constant time intervals are chosen for every grain. Sometimes it is useful to heat the samples in a reduced atmosphere by using a closed or quasi-closed container (e.g., quartz, platinum or gold capsules) or a small droplet of immersion oil on the sample.

SMIs hosted in different phases will homogenize following different heating rates; the size of the SMI will also affect the required rate of reheating and Danyushevsky (2001) provides further discussion on this topic.

3.4.3. One atmosphere vertical furnaces

Heating in a one atmosphere vertical furnace (Sinton et al., 1993; Student and Bodnar, 1999; Fedele et al., 2003; Schiano, 2003) does not allow to direct observation of the changing phases in the SMIs during the heating process; the heating can be done either directly to final temperature or in single steps. The one step method consists of placing crystals into platinum capsules, and introducing them into a preheated furnace. The pre-heated temperature is based on the preparatory experiments carried out to estimate the minimum homogenization temperature. The incremental method consists of incrementally heating the sample, then quenching and taking sample out of the furnace for observation under a microscope. The capsule is attached to a ceramic rod to allow for rapid extraction and quenching in water for both methods. This method allows numerous crystals to be reheated and homogenized at the same time; however, one must assume that all crystals and SMIs were formed under similar conditions and therefore have the same homogenization temperature. This, obviously, is not always the case and a thorough petrographic study to determine the origin of the crystals, and their relation to each other is again, deemed essential.

3.4.4. Internally Heated Pressure Vessel (IHPV), Piston Cylinder Apparatus (PCA) and Hydrothermal Diamond Anvil Cell (HDAC)

Internally Heated Pressure Vessels (IHPV) and Piston Cylinder Apparatus (PCA) are generally used to re-heat SMIs that have high internal pressures, and cannot be reheated by one-atmosphere devices because they will decrepitate (Skirius, 1990; Skirius et al., 1990; Anderson et al., 2000; Student and Bodnar, 2004; Veksler and Thomas, 2002). With these methods, crystals containing unexposed SMIs are loaded in capsules in a vessel pressurized with inert gas (usually pure Ar); the capsules, can be run simultaneously and both pressure and fugacity are always under control. Ramps of heating can be programmed, although the homogenized SMIs can only be observed after quenching. These techniques allow homogenizing SMIs that have been formed at very high pressures (up to 7 kbars). Hydrothermal Diamond Anvil Cells (HDAC) are a high-pressure reheating technique for SMIs that, unlike IHPVs and PCAs, allows direct observation of the phase changes in the crystal (although corrosion inside the chamber may disturb transparency), during the course of the experiment (Li and Li, 2014).

Various researchers have studied SMIs by reheating them with different techniques (Fedele et al., 2003; Student and Bodnar, 2004; Nielsen, 2011) and have concluded that there are no significant differences in the SMIs composition from similar samples reheated using different methods. However, homogenizing SMIs on a stage under a microscope should be the preferred method when dealing with volcanic samples. The advantage of using a microscope-mounted heating stage is the possibility of continual observation over the course of the procedure, allowing a more precise determination of the homogenization temperature, and reducing the risk of overheating; at high temperatures SMIs can decrepitate or the host can start to melt, rendering the SMI, and in fact the entire crystal host, unusable. Continuous monitoring during the heating process also allows checking for physical changes and detecting nucleation/shrinkage of bubbles, not easily achieved by using other re-heating techniques. Working at room pressure though may pose some difficulties, such as not reaching the point of complete re-homogenization of SMIs that have been formed at very high pressure. In this case, it is preferable to use a pressure-controlled furnace, in order to avoid over pressures and consequent decrepitation of the inclusions.

4. Analytical techniques

Recent advances in micro-analytical techniques have broadened the spectra of information, obtainable from the analysis of silicate melt inclusions. The most current techniques will be outlined briefly here.

4.1. SEM/BSE/EDS/WDS

The use of Scanning Electron Microscopy (SEM), particularly imaging using Back Scattered Electrons (BSE), is extremely a useful tool in the study of SMIs. This technique can be used to check for homogeneity prior to EMP, create element distribution maps of an inclusion and to identify daughter or entrapped mineral phases using Energy-Dispersive X-ray Spectroscopy (EDS) or Wavelength-Dispersive X-ray Spectroscopy (WDS; e.g. Klébesz et al., 2012; Chen et al., 2013). However, care must be taken when using the SEM as elements may become mobile when exposed to the electron beam; F, Cl, Ca and P become mobile in apatite and glass, Na and K in plagioclase and glass (Nielsen and Sigurdsson, 1981; Stormer et al., 1993; Morgan and London, 2005; Goldoff et al., 2012).

4.2. Electron microprobe analysis

The use of the Electron Microprobe (EMP) is perhaps the most popular micro-analytical technique in the study of SMIs. The EMP is most commonly used to determine the major and minor element concentration of SMIs as well as some volatile elements (typically Si, Ti, Al, Fe, Mg, Mn, Ca, Na, K, Cr, P, Cl, F and S); H_2O can be calculated by the "water by difference" method (Blundy and Cashman, 2008; Métrich and Wallace, 2008). The high spatial resolution possible with the EMP (with a beam diameter as small as 1–2 μm) allows multiple analyses within relatively small SMI, or multiple point traverses across inclusions to check for homogeneity or post-entrapment crystallization (Danyushevsky et al., 2000).

As previously mentioned, while not strictly a destructive technique, different elements react to the exciting effect of the electron beam in different ways, some becoming mobile over time, leading to errors in analyzed elemental concentrations. Varying the beam conditions (i.e. the size, accelerating potential or beam current) for different elements can mitigate this problem. For example, while more robust elements such as Si, Al and Fe can be analyzed using stronger beam conditions (i.e. an accelerating voltage of 15 kV and a beam current of 10–20 nA), the determination of volatile, and mobile elements concentrations (including Na and K, as previously discussed) may be affected by high accelerating voltage values. If volatile or mobile element concentrations are an important focus of a study, and there is no possibility of using other more accurate analytical techniques for determining their concentrations, they may be analyzed in a separate instrument pass using very gentle beam conditions (i.e. 10–15 kV and 2 nA) and a wider spot diameter. However, a side-effect of using gentle beam conditions is the need for longer analytical time to reduce errors due to counting statistics.

Analytical uncertainty and accuracy in using EMP can vary depending on a number of factors including the elements analyzed, beam and analysis conditions (therefore counting statistics) and individual element calibration. Typically, the analysis of a known analytical standard with similar geochemistry, using the planned beam conditions is performed prior to analysis of the unknown sample to check for instrument drift and accuracy. In all concerns, it is best to discuss your needs with your microprobe technician/operator who may be able to provide advice based on the performance of their individual machine.

4.3. SIMS

Secondary Ion Mass Spectrometry (SIMS) is a slightly destructive micro-analytical technique using a 15–30 μm diameter sputtered beam of primary ions ($^{16}O^-$ or $^{133}Cs^+$) to excavate the sample, then collecting and analyzing the ejected secondary ions using a mass spectrometer. SIMS is particularly useful in determining trace element abundances, as limits of detection can be as low as <1 ppm for some elements (Lowenstern, 1995; Wiedenbeck, 2008; Audétat and Lowenstern, 2014). SIMS is also useful in determining volatile element abundances including H, B, Be, C, O, F, S and Cl and the ratio between stable and radiogenic isotopes (Pettke et al., 2004; Kent, 2008). Typical accuracies are 5–10% for elements >1–10 ppm and 10–40% for lower concentrations.

An internal standard is also required for data reduction using SIMS, typically the Si concentration of the MI, measured using the EMP. As the SIMS technique is less destructive than LA-ICPMS, with a "typical" analysis excavating a hole only 2–5 μm in depth, this preserves a lot of MI material for further study if needed (Kent, 2008).

When determining H or C concentrations (including H_2O and CO_2), the use of indium or specialized epoxy mounts is preferred as the volatility of most epoxy mounts can affect the accuracy of the analysis (Audétat and Lowenstern, 2014). Similarly, when determining C concentration, the sample must be carefully cleaned if carbon coating has been applied for previous analysis (i.e. with the EMP) and the sample recoated in Au to eliminate C contamination. However, contamination can still occur and care must be taken during data reduction to honestly remove analyses where contamination is present. A drawback of the SIMS technique is the requirement of using indium mounts for volatile element quantification. Mounting crystals in indium can be difficult if the crystals are small or very thin and breakage can be an issue but the use of indium can be disregarded if you do not require the quantification of H or C.

Full explanations on the use of the SIMS technique can be found in Hauri (2002), Kent (2008), Lowenstern (1995), Pettke et al. (2004) and the references therein.

4.4. LA-ICPMS

Laser Ablation-Inductively Coupled Plasma Mass Spectrometry (LA-ICPMS) is another tool, which may be used in conjunction with the EMP to determine trace elements, or as a stand-alone instrument to determine major and trace element concentrations. LA-ICPMS is a destructive technique, which ionizes a sample of the material, then analyses it using a mass spectrometer, therefore, should be the final step in the analytical process.

LA-ICPMS analyses individual pre-programmed elements as individual traces and the data must be reduced using specialized software such as the free-to-use AMS software package. Data reduction requires analysis of the background signal, a number of standards and the choice of either using a known concentration (if major elements have been analyzed using EMP) as a benchmark or normalization to a set value (Pettke et al., 2004). Signal contamination from the beam source (i.e. Cs) can be a problem, however, during data reduction, contamination sources can be easily identified and removed. LA-ICPMS can be used to analyze inclusions that have not been exposed (but must be close to the surface) as the beam excavates the sample over time (Pettke, 2006). Additionally, heterogeneous SMI that have not been rehomogenized in the lab can also be analyzed using LA-ICPMS, making sample preparation much easier, and increasing the number of possible targets for analysis (Pettke et al., 2004; Zajacz and Halter, 2007).

While the use of LA-ICPMS for determining both major and trace geochemistry of SMI has distinct benefits, LA-ICPMS cannot be used to analyze H_2O, S, F and Cl or isotopic ratios, and has higher limits of detection for trace elements than SIMS and lower spatial resolution than SIMS and EMPA (Pettke et al., 2004; Kent, 2008). Additionally, the data is reliant on the use of a known concentration of an internal standard, which determines the final geochemical concentrations and therefore the overall quality of the data. This can be difficult to determine if using LA-ICPMS as the sole analytical technique. For these

reasons, the use of EMPA and SIMS for determining major and trace element concentrations is still the most preferred method of analysis for many researchers.

4.5. Raman spectroscopy

Raman spectroscopy has long been used as a tool in the analysis of fluid inclusions due to the high spatial resolution possible and versatility in measuring both fluid and solute/solid phases within an un-exposed inclusion (Burke and Lustenhouwer, 1987; Burke, 2001; Frezzotti et al., 2012). In studies of SMIs, Raman spectroscopy can measure the abundance of volatile elements in a glass (Thomas, 2000; Behrens et al., 2006; Thomas and Davidson, 2012; Thomas et al., 2008a), or un-exposed bubble of a SMI (Esposito et al., 2011; Steele-Macinnis et al., 2011; Doherty et al., 2012). It is a non-destructive technique that requires only a single-sided polish on the sample and has a very small spatial resolution (1–2 μm) allowing a large number of SMI to be analyzed within a single host crystal (Métrich and Wallace, 2008). Thomas et al. (2008b) have shown that Raman Spectroscopy allows the determination of water in glasses down to the parts per million, and independently check with the proton-proton (pp) scattering and infrared (IR) spectroscopy.

One problem when using Raman spectroscopy to analyze SMIs, if the vapor density of the bubble is low, the inclusions and the bubble must be as close to the surface of the sample as possible (<30 μm) to be able to detect the CO_2 Fermi Diad. The deeper the bubble, the weaker the Raman signal will be.

4.6. Other analytical methods

Fourier Transform infrared spectroscopy (FTIR) is another common technique used to quantify CO_2 and H_2O (and their species) in SMIs (Aines and Rossman, 1984; Lowenstern, 1994; Seaman et al., 2006; Métrich and Wallace, 2008). FTIR relies on the determination of the absorption coefficients for the species (of H_2O or CO_2) of interest and measurements of the signal peaks at those wavelengths, which can be occluded by signals of the major elements of the SMIs. FTIR requires a double-polished sample, and knowledge of the sample thickness. This makes preparation of samples for FTIR analysis more difficult than other micro-analytical techniques.

NanoSIMS is a relatively new technique, which expands on the instrumentation of the SIMS technique, allowing for seven ion detectors, as opposed to the five (Wiedenbeck, 2008). Additionally, the extremely high spatial resolution (50–100 nm) possible with NanoSIMS makes it a valuable tool in fields where sub-micrometer measurement is required (Hoppe et al., 2013; Lloyd et al., 2014).

Other less well known analytical techniques in SMI studies include: PIXE (Proton-Induced X-ray Emission, Czamanske et al., 1993), femtosecond laser ablation (fs-LA) inductively coupled plasma-quadrupole mass spectrometry (ICP-QMS) and nanosecond laser ablation (ns-LA) (Borisova et al., 2012) and synchrotron radiation-induced X-ray fluorescence analysis (Rickers et al., 2006) to measure in situ trace element concentrations; X-Ray Tomography to accurately measure the volume of inclusions and their components (Pamukcu et al., 2013); and Time of Flight (ToF) SIMS provides high spatial resolution and ion mass range making selected major elements also measurable (Marques et al., 2011).

5. Determination of volatiles in SMIs

It is widely acknowledged in the scientific community that the volatile content of magma can affect style, frequency, and intensity of volcanic processes (De Vivo et al., 2005; Métrich and Wallace, 2008 and references therein) and it is not news that researchers belonging to the petrological, volcanological and giacimentological communities have used the SMI technique to investigate magmatic processes

(Audétat and Lowenstern, 2014). The main aim of scientists using SMIs has been to trace the evolution of a rock from its source to the surface. While, a whole rock represents a final product of a series of processes occurring over a wide span of time, SMIs, ideally, represent a snap shot of a specific time within the time-span of the rock formation. For this reason, SMIs record information otherwise lost during subsequent processes of the rock formation. One of the most important pieces of information that is lost during crystallization and migration to the surface of magmas is the volatile concentration of early undegassed melt. The importance of volatile contents recorded by SMIs was recognized by several pioneers of the SMIs technique as noted by Roedder (1979) and in earlier Russian literature (Dolgov and Shugurova, 1966). But at these early years, the attempts to measure the volatile contents were hampered by the technology which was unable to directly analyze the concentration of volatiles associated with relatively small volume of glass in SMIs. As mentioned earlier, with the advent of the SIMS and FTIR techniques and even more recently the nano-SIMS, reliable data regarding the concentrations of volatiles dissolved into the glass of SMIs (in particular H_2O and CO_2) has started to be published in the literature (Lowenstern, 1994 and references therein; De Vivo and Bodnar, 2003; Webster et al., 2003a,b).

The use of volatile concentrations recorded by SMIs became of great interest to more researchers because of the potential of volatile elements to track the ascent path of magmas from deep in the crust up to the Earth's surface. In fact, with the assumption that SMIs do not significantly change after trapping, volatile concentrations in SMIs provide an estimation of the depth of formation of the host crystal based on $H_2O–CO_2$–silicate liquid solubility models (Newman and Lowenstern, 2002; Papale et al., 2006). Therefore, if a melt is volatile-saturated, the volatile concentration of a silicate melt is strongly dependent on pressure and the volatile concentration of a SMI can be used as a geobarometer to track the ascent of magmas, especially important for the study of active volcanic fields. If the melt is not volatile saturated, the volatile concentration of SMIs will only give a minimum estimation of the pressure of formation. If phenocrysts crystallized from magma at different times and at different locations within the magmatic reservoir, volatile concentrations of SMIs hosted in different crystals will result in a wide variation that could be used to decipher various igneous processes (e.g. Kent, 2008 and references therein; Métrich and Wallace, 2008 and references therein).

Scientists have used the volatile concentration of SMIs to estimate the budget of volatiles degassed from a deep melt, with the aim of understanding the recycling of volatiles in subduction zones (Wallace, 2005; Plank et al., 2013). In other studies, volatile concentrations of SMIs connected to the outside of the host have been used to estimate ascent rates of magmas (Lloyd et al., 2014 and references therein), while Bartoli et al. (2013) investigated SMIs hosted in garnet from crustal xenoliths to determine the volatile content of anatectic melts.

Recently, post-entrapment modification and the significance of "shrinkage" bubble in bubble-bearing SMIs have been discussed (Esposito et al., 2011; Steele-Macinnis et al., 2011; Hartley et al., 2014; Cannatelli, 2015; Moore et al., 2015; Wallace et al., 2015). The general conclusion of these recent studies is that the volatile contents (especially the CO_2 content) of the original trapped melt are strongly underestimated if the volatile concentration contained in the shrinkage bubble is not considered, as it is common in the literature. For instance, Cannatelli (2015), Wallace et al. (2015), Moore et al. (2015) and Hartley et al. (2014) reported that up to 75% and 90% respectively of the CO_2 concentration of SMIs can be stored in the shrinkage bubble. Another current debate is whether or not volatile elements (especially H) can diffuse out of the SMIs through the host (Bucholz et al., 2013; Le Voyer et al., 2014). Bucholz et al. (2013) reported that if an olivine crystal, containing H_2O-rich SMIs, ascends to a shallower reservoir (H_2O-poor melt), H can be loss from the SMIs in a matter of minutes.

In our opinion, the debates reported above highlight the importance of the selection of "good" SMI candidates to study the volatile evolution

of magmatic system. As described earlier in this review, the search for good candidates is achieved by good petrographic analysis of SMIs (Roedder, 1979) and we suggest that detailed petrographic information (size, shape, appearance, position relative to the host and other SMI within the same host) should be always collected first in any study involving SMIs analysis. In particular, the analysis of several SMIs in a single phenocryst may reveal if the volatile contents are indeed representative of the melt at a definite time in the magma evolution. The relative time of trapping among SMIs may also help to understand if the variation in volatile concentrations is consistent with a certain magmatic physical–chemical process. For instance, a study by Peppard et al. (2001) using cathodoluminescence maps in quartz phenocryst of the Bishop Tuff not only assessed the relative time of trapping of SMIs in single phenocrysts but were also able to provide strong evidence that the variability of the volatile contents among the studied SMIs was the result of phenocrysts sinking into the magma chamber. They concluded that quartz crystals of the Bishop Tuff deposit were sinking based on the relative age of SMIs; the CO_2-rich SMIs were younger than the CO_2-poor SMIs.

It is possible to assess the relative age of formation of SMIs hosted in a single crystal by studying SMIs trapped along a growth zone of a certain mineral. If SMIs in a MIA show the same volatile concentrations, then based on Roedder's rules, there is good evidence that the volatile contents are representative of that "snap shot" of melt along the evolution path of a rock mentioned at the beginning of this section. If the volatile contents of SMIs within a single MIA show significant variability, one needs to explain what type of process may have determined this variation before considering that volatile content representative.

In conclusion, the progress of research aimed at the understanding of the reliability of SMIs in recording the representative volatile contents of un-degassed melt coupled with the advance of technical instrumentation, points toward the selection of SMIs for study as being vital to the investigation of magmatic processes otherwise unrevealed by the whole rock approach.

6. Pitfalls in the study of melt inclusions

As we have seen, the selection of SMIs appropriate for study is the most important aspect of a SMI investigation. Understanding their hosts' crystallization history and their distribution within the host can only be done through thorough petrographic study. Choosing inappropriate inclusions for study is perhaps the most easily avoidable source of error in a SMI investigation. Choosing the method of mounting, polishing and analysis, appropriate to the information sought during the study, is the next potential source of error and advice should always be sought from the instrument operator on the practices for using the analytical equipment and its sample preparation requirements. However, there are other "pitfalls" in the study of SMIs that can trap an unaware researcher.

6.1. Variations in SMIs compositions

In volcanic rocks, compositional changes are common in SMIs after trapping and the amount of alteration is directly related to the rate of cooling in the system (difference between the trapping temperature and the quenching temperature). If the cooling rate of a phenocrysts is fast, then modification to the hosted SMI is constrained; if the cooling rate is slow, the SMIs can be subjected to very high internal pressures and may partially or completely decrepitate, depending on their radius/crystal size ratio (Bindeman, 2005).

6.1.1. Boundary layer phenomena
Several researchers in the past decade have questioned whether some processes occurring either during crystal growth, or after SMIs entrapment, can potentially compromise SMIs composition (Kent, 2008 and references therein). In particular chemical gradients that develop

at the crystal/melt interface during crystal growth, as consequence of preferential incorporation of some elements into the crystal, form boundary layers that, if trapped within, could affect SMIs composition especially with regard to minor and trace elements. As a consequence of such process, elements that are incompatible in the host phase become enriched in the boundary layers, while elements compatible with the host are depleted. SMIs that trap aliquots of the boundary layers will show stronger enrichment in incompatible elements (or stronger depletion in compatible ones) as an inverse function their size, since smaller inclusions will trap a smaller "thickness" of the boundary layer melt.

Several studies have been conducted on natural and synthetic systems (Albarède and Bottinga, 1972; Donaldson, 1975; Petersen and Lofgren, 1986; Muncill and Lasaga, 1987; Aoki and Iyatomi, 1993), and Bacon (1989) proposed a model for the boundary layer effect in major rock-forming minerals with compositions ranging between felsic and intermediate. Sobolev (1996) explained that the coexistence of SMIs in a single crystal which display a large range in trace elements concentrations can be related to disequilibrium conditions, as those SMIs might have been altered by diffusion during entrapment. Experimental work by Faure and Schiano (2005) on SMIs in dendritic or skeletal forsterite crystals shows that SMIs formation is controlled by diffusion, and that those SMIs trap consistent portions of the boundary layer, resulting in compositions quite different from the bulk magma.

While some studies suggest that the boundary layers do not affect SMIs larger than 50 μm (Lu et al., 1995; Zajacz and Halter, 2007; Severs et al., 2009), there is no available information about the minimum size of SMI that can be analyzed before boundary layer processes produce a measurable effect on its composition. Advances in analytical techniques have reduced the size for "analyzable" SMIs, and instrumentations such as LA-ICPMS or SIMS can perform analyses on SMIs as small as 10 μm. It is of paramount importance therefore to determine if data from such small SMIs are reliable source of information on the chemistry of magmas.

6.1.2. Post-entrapment modification
We have discussed that when the cooling rate of a phenocryst is slow, SMIs may experience post-entrapment modifications, such as nucleation of bubbles (Roedder, 1984) and precipitation of some material of the melt on the wall of the inclusion, not always visible under the microscope but detectable by Back Scattered Electron (BSE) images or EMP. Diffusion processes after the SMI trapping result in irreversible changes of the original composition of the SMI (Danyushevsky et al., 2002; Kent, 2008 and references therein). Post-entrapment crystallization (PEC), has been documented by researchers in several studies. Danyushevsky et al. (2000) have described the loss of Fe in olivine-hosted SMIs due to natural cooling and/or re-homogenization experiments and several researchers have developed models to account for such modifications (Danyushevsky, 2001; Danyushevsky et al., 2002; Danyushevsky and Plechov, 2011; Gaetani and Watson, 2002). Cottrell et al. (2002) have demonstrated that PEC is more effective in changing the composition of Sr, Eu and Ba in plagioclase-hosted SMIs than in olivine-hosted SMIs. PEC can occur naturally or during re-heating of SMIs in the laboratory and can affect both volatile, major and trace element concentrations of SMIs. It has been the focus of research of many workers in the past decade (for a more in depth discussion see Audétat and Lowenstern, 2014 and references therein).

The rapid diffusion of hydrogen through the host crystal has induced several researchers to investigate the reliability of H_2O content measured in SMIs. Hauri et al. (2002), Massare et al. (2002), Severs et al. (2007), and Zajacz and Halter (2007) estimated the amount of original H_2O content of a SMI lost during re-heating. Kress and Ghiorso (2004) calculated the effect of PEC on SMIs in an andesite pumice from Popocatépetl using the thermodynamic software MELTS and found that SMIs experienced 7.3% PEC, with the result of an increase in the original H_2O content. More recently, Steele-MacInnis et al. (2011) have numerically

calculated the fraction of the total CO_2 present in the SMIs that is trapped in the bubbles as a result of sidewall crystallization. The post-entrapment modification "problem" is a subject of continued study at this point.

7. Conclusions and final remarks

In this review we have tried to give a summary of the progress in SMIs research, giving a guideline (Fig. 7) on how to conduct a correct SMIs study and explaining ways to avoid common mistakes that we thought to be critical for a reliable SMIs data acquisition.

Here is a summary of suggestions and tips that may be useful for beginners in melt inclusions research.

1. Detailed petrographic approach

Suggestions: Identify a Melt Inclusion Assemblage (MIA): if all the SMIs show the same phase behavior and homogenization temperature, it is the best evidence that they obey Roedder's rules and have not re-equilibrated after trapping. Determine timing of inclusion trapping respect to the host crystal and relative formation of the host respect to the paragenesis of the rock. Describe size, shape, appearance (e.g., presence of bubble, daughter or co-trapped minerals) of SMIs. Observe bubble volume/SMI volume ratio to assess if the melt was

saturated in volatiles at the moment of trapping. Check for chemical zoning and rims around the inclusions.

Tips: If signs of leakage and decrepitation are found, disregard the affected inclusions.

2. Preparation of samples

Suggestions: gently crushed and sieved rocks, handpick phenocrysts under a binocular microscope, mount SMIs on glass slides, or in epoxy mounts and gently grind to produce double polished wafers (30 μm to 1.5 mm thick) if needed.

Tips: Use a hot needle to release the crystals from the epoxy after exposing the SMIs; the use of acetone may contaminate the sample (especially if the volatile element concentrations are to be studied).

3. Microthermometric experiments

Suggestions: Try to re-heat representative SMIs, preferably in one-atmosphere heating stages under the microscope. Carefully document the disappearance of daughter minerals, nucleation/shrinkage of bubble(s), and record the homogenization temperature.
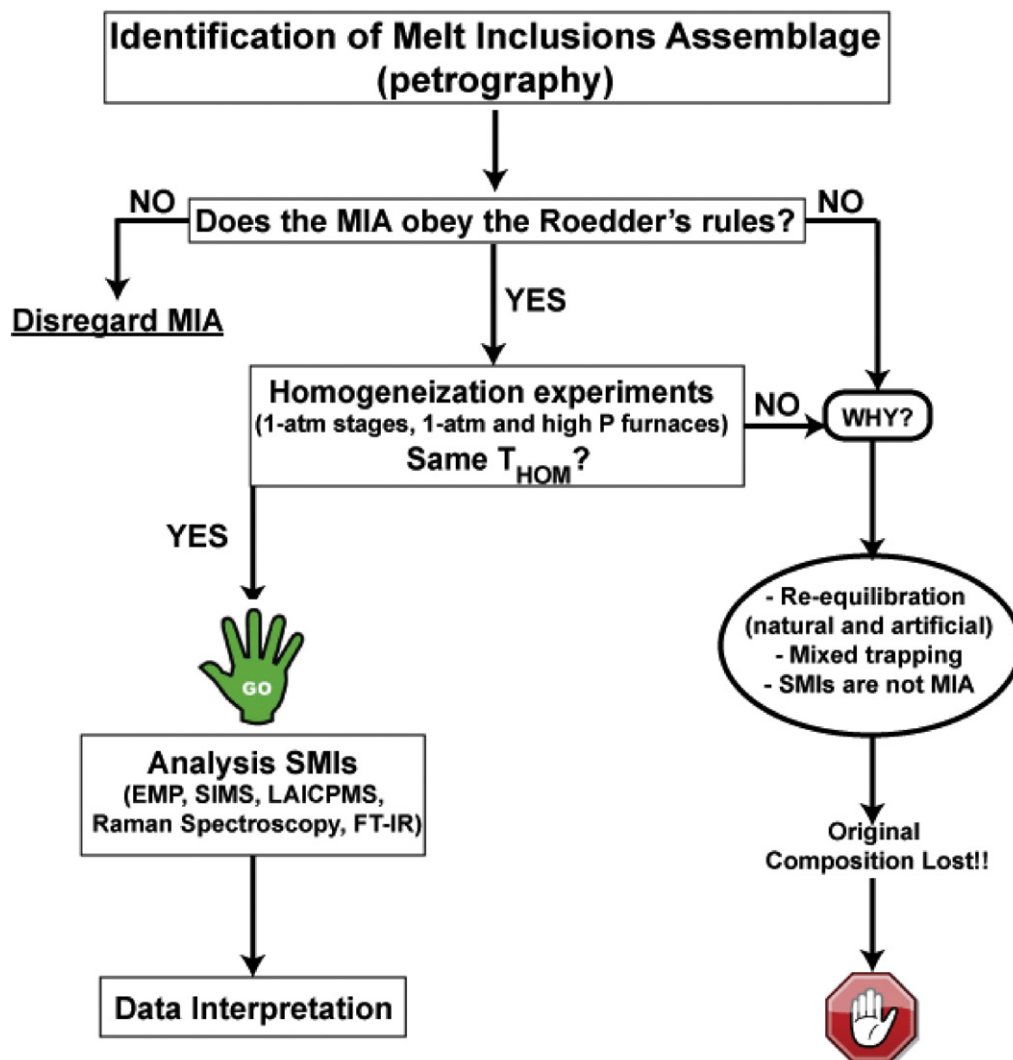
Fig. 7. Flow chart summarizing the procedure that should be followed in a SMI study. The first and most important step is to identify a MIA, a group of SMIs that are trapped at the same time, temperature and pressure and from a melt of the same composition.

Tips: the "correct" heating rate is achieved by several attempts, aiming at obtaining temperatures of homogenization constant for SMIs in MIA.

4. Analyses of SMIs

Suggestions: Analyze naturally quenched SMIs (if present) and experimentally quenched SMIs and compare the results. This can give important information about crystallization history and liquid line of descent in the magma reservoir. Check for homogeneity; create element distribution maps by SEM. Major and minor elements can be analyzed by EMP, trace and (some) isotopic composition can be obtained by SIMS and LA-ICPMS. Volatiles can be analyzed by FTIR or SIMS.

Tips: If a volatile elements study is planned, be sure to analyze SMIs by Raman Spectroscopy to understand if volatile phases are present in the bubble(s) of the investigated SMIs. The determination of volatile, Na and K concentrations by EMP may be affected by high accelerating voltage values, so precautions should be taken to minimize this effect.

5. Interpretation of data

Suggestions: Compare your SMIs data with bulk rock composition and experimentally derived liquid composition (by running thermodynamic models such as MELTS) to make sure your data are reasonable.

Tips: When analyzed for volatile content, it is crucial (if possible) to study multiple SMIs in a single phenocrysts, as they may reveal if the volatile contents are representative of the melt at the time of trapping and may help to understand if the variation in volatile concentrations is consistent with a certain magmatic physical–chemical process.

Day by day, we witness great advances of in situ microanalytical techniques, in particular by SIMS or LA-ICPMS, in the reduction of analyzable sample size and improvement of analytical precision and detection limits. In a near future, scientists will be able to analyze with great accuracy trace and stable isotopes elements in SMIs, that, coupled with advances in simulating natural processes using numerical modeling, will allow the investigation of source composition and melting processes, and will hopefully shed some light into the genesis of magmatic systems.

"…and that there is no necessary connection between the size of an object and the value of a fact, and that, although the objects I have described are minute, the conclusions to be derived from the facts are great." (Sorby, 1858).

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