Melt Inclusions Come of Age:
Volatiles, Volcanoes, and Sorby's Legacy

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ABSTRACT

Despite nearly forty years of modern research on silicate melt inclusions (MI), only within the past 10-15 years have volcanologists and petrologists come to regularly accept their utility for characterizing magmatic systems. Their relatively slow acceptance was likely due to a number of factors including: 1) Lack of reliable analytical techniques, 2) Concern that MI represent anomalous boundary-layer melts or are altered by leakage or post-entrapment crystallization, 3) Data sets indicative of heterogeneous melts and, 4) Homogenization temperatures greater than those calculated by other techniques. With improvements in analytical methods and careful studies of MI systematics, workers are increasingly convinced of the utility of these features to unravel the complexities of volcanic systems: melt inclusions have "come of age." Recent studies provide compelling evidence for the compositions of dissolved and exsolved volatiles in magma reservoirs. Evidence for immiscibility of gases, hydrosaline brines and pegmatitic fluids demonstrate that magmatic phase relations are often more complicated than can be inferred by inspection of crystalline phases alone.

INTRODUCTION

Volatile gases are the fundamental cause of magma ascent, effervescence, expansion, and fragmentation. Either in dissolved or exsolved form, these gases strongly control the density and viscosity of magmatic liquids. Yet because they are released during eruption, the gases leave little trace of their former presence. Studies of bulk tephra and groundmass glass reveal little about the initial abundance of volatiles in the pre-eruptive magma. Consequently, magmatic inclusions such as silicate-melt inclusions (MI) in phenocrysts represent a vital source of information about the compositions of magmatic fluids during eruptive ascent.

The MI are typically small blebs of silicate melt that are trapped within phenocrysts at magmatic temperatures and pressures. The relatively incompressible phenocryst hosts act as pressure vessels during eruption, keeping any trapped MI from degassing. Through microbeam analysis of the quenched MI, we can quantify the pre-eruptive

concentrations of dissolved volatile gases (i.e., H_2O, CO_2, S, and Cl). We can then estimate the pressures of melt-vapor equilibration at the time of inclusion formation (Anderson et al., 1989). Through studies of MI we also recognize that elements such as B, P, and F can act just as stealthily as some of their better-known volatile brethren, having higher concentrations in MI than degassed matrix glass. Studies of ore metals such as Cu, Zn and Sn show that they also can be transported as volatiles in magmatic systems. Perhaps the most important insight from MI studies has been the revelation that magmatic fluids can be far more diverse than solely silicate melt and gas (Roedder, 1979a; Cathelineau et al., 1988; Roedder, 1992). Recent studies document chloride-, sulfate–, and carbonate-dominated melts, and silicate-silicate immiscibility at relatively low temperatures ($<500°C$); importantly, they have also begun to provide detailed compositional data on these immiscible phases.

Our reliance on MI has come only after several decades of concerted effort to improve analytical techniques, to assess the reliability of MI due to host-inclusion equilibration (pre-, syn- or post-trapping), and to recognize the effects of trapping multiple fluid phases. Additional effort has been placed on characterization of bubbles within MI, which can be present for a variety of very different reasons. In this article, I review some of the history of melt-inclusion studies, with special attention to the changing perceptions of MI and their utility for understanding magmatic volatiles. I also address evidence for trapped magmatic fluids within igneous phenocrysts, including those trapped along with silicate melt in mixed inclusions.

The review is necessarily subjective; it represents the accumulated perspective of a single researcher, and given its brevity, cannot begin to cite all of the many articles and abstracts relevant to the subject. My aim is to provide a starting point for workers interested in MI and the history of their role in the study of volatiles in volcanic systems.

IMPACT OF MELT INCLUSION RESEARCH PRIOR TO THE 1990s

In their pioneering 19th Century studies, Sorby (1858) and Zirkel (1873) gave detailed descriptions and drawings of MI and accompanying fluid inclusions (FI) in a wide variety of intrusive and extrusive rocks (see Table 1 for definitions). They speculated on the origins of bubbles within the inclusions and recognized that MI had marked promise for interpretation of the origin of igneous rocks. Indeed, Sorby (1858, p. 497) in a comment to skeptical colleagues stated: "There is no necessary connexion between the size of an object and the value of a fact, and … though the objects I have described are minute, the conclusions to be derived from the facts are great."

For a variety of reasons, decades would pass before workers would follow Sorby and further scrutinize these tiny droplets of silicate melt in igneous phenocrysts. The first modern studies of MI were undertaken not by volcanologists but by workers interested primarily in the systematics of FI. Studies of FI blossomed in the 1960s, 70s and 80s, particularly in the field of economic geology (Hollister and Crawford; 1982; Roedder, 1984). At this time, the international fluid inclusion community championed the use of MI for studies of lunar samples (Roedder and Weiblen, 1970, 1971), pegmatites, granitoids and volcanic rocks (Sobolev and Kostyuk, 1975; Roedder,

1979b). These papers demonstrated abundant evidence for immiscibility among silicate melt and diverse volatile phases from a variety of igneous rocks (Roedder, 1965; Roedder and Coombs, 1967; Roedder 1979b). Myriad references in the Russian literature refer to "salt melts", "solution-melts" or "melt-brines" (see Sobolev and Kostyuk, 1975), but the implications of this research was not fully assimilated by igneous petrologists in many other countries.

Table 1. Definitions of magmatic inclusions in phenocrysts

Melt Inclusion (MI)	Contains glass or crystallized glass
Fluid Inclusion (FI)	Contains one or more fluids at room temp. and <u>no</u> glass or crystallized glass.
Vapor-rich Fluid Inclusion	FI with > 50 vol.% bubble at room temp.; i.e., low density (potential gassy) fluid.
Mixed Inclusion	More than one magmatic phase trapped.
Hydrosaline Melt	Molten solution of salts and H_2O

Reservations about the utility of MI:

Perusing reviews on volatiles as well as textbooks on igneous petrology written during the 1970s and 1980s, there is little if any mention of MI (e.g., Carmichael et al., 1974; Best, 1982; Burnham, 1979a, 1979b; Holloway, 1979; McBirney, 1984; Maaløe, 1985). The 1987 issue of Reviews of Geophysics contains 5 review articles on igneous petrology and volcanology (Carlson, 1987; McCallum, 1987; Marsh, 1987; Ghiorso, 1987; Self and Francis, 1987). Of some 3000+ references cited in these articles, fewer than 5 relate to MI. The apparent low impact of MI studies on the volcanological and petrological communities can be traced to a number of factors. Some workers were unclear as to whether MI accurately represented the compositions of their host magmas. As stated by Eichelberger (1995): "Interpretation of melt inclusions is not without difficulties: 10^{-14}-g samples are taken to represent a 10^{10}-g system…" Workers cited a variety of processes that could potentially cause an MI to yield spurious data: 1) non-representative "boundary-layers" accumulated near growing crystals, 2) post-entrapment growth of the host crystal from trapped melt, 3) leakage and decrepitation, and 4) diffusion of species through the host crystals (or exchange between host and melt). These problems are real and require caution on the part of the analyst. Because of these potential pitfalls, petrologists in the 1960s to 1980s often seemed leery of MI research findings, and pointed to problems evident in published studies, some of which are discussed below.

MI temperatures appeared to be too high: Much of the work on MI by the international fluid-inclusion community focused on traditional homogenization experiments, primarily to assess magmatic temperatures. The petrological community,

however, had developed a variety of experimentally based geothermometers. These procedures relied on microprobe analyses of co-precipitating minerals or mineral-glass pairs (e.g., Kudo and Weill, 1970; Buddington and Lindsley, 1964; Stormer, 1975; Wood and Banno, 1973), and they were adapted and used successfully in numerous petrological studies (e.g., Ewart et al., 1971; Hildreth, 1979, 1981). In general, the temperatures found from MI homogenization studies were greater than those inferred by analysis of coexisting Fe-Ti oxides and other geothermometers (Sommer, 1977; Beddoe-Stephens et al., 1983). Roedder (1979b) discussed several reasons for high-homogenization temperatures (slow diffusion of melt components, thermal gradients in the stage, diffusive loss of H_2 or H_2O) and cautioned against literal interpretation of temperatures acquired through homogenization experiments. Recent studies have verified that homogenization studies can provide spurious data because of diffusion of volatiles through the host and stretching of the phenocryst container during laboratory experiments at atmospheric pressure (e.g. Massare et al., 2002).

MI volatile contents appeared too high (or too low): In the early 1970s, several workers began to use the electron microprobe to analyze MI and to estimate dissolved volatile concentrations (Anderson, 1974a, 1974b, 1975; Takenouchi and Imai, 1975; Clocchiatti, 1975). Anderson (1974a) found high Cl and S in MI from andesitic glasses of Mt. Shasta (CA, USA). He inferred high H_2O concentrations in MI, 1 to 7 wt.%, by observing that the electron microprobe analytical totals for the oxides were significantly less than 100% (aka, "volatiles by difference"). In a related paper Anderson (1975) concluded that volcanic outgassing rates far exceed the amounts that can be supplied by erupted magmas and that the subsurface reservoirs of Pacaya (Nicaragua) and Kilauea (Hawai'i, USA) were vapor-saturated prior to eruption. Sommer (1977) documented similar evidence for the pre-eruptive Bandelier Tuff magma chamber, and showed there was significant CO_2 dissolved in the magma. Such arguments were novel, and some workers were skeptical. Roedder (1979b) commented: "some workers express little faith in any results based on low electron microprobe totals, particularly when the amounts of water (and other volatile materials) estimated from them are much higher than previously thought." Reviews by Holloway (1979) and Gill (1981, p. 293) were representative of this viewpoint and expressed some doubt as to the accuracy of concentrations reported by Anderson (1974a,b, 1975). Both reviews discussed the possibility that MI may not have compositions representative of the melt from which they grew. This viewpoint was widespread among petrologists: Watson (1976) had pointed out that post-entrapment crystallization of the inclusion host could alter the MI composition. He proposed a useful method for accounting for this process by analysis of inclusions in multiple phases. In a later paper, Watson et al. (1982) predicted that MI might have dissolved H_2O and CO_2 concentrations many times in excess of their actual melt abundance due to buildup of slow-diffusing elements near the borders of rapidly growing crystals. They also estimated that bubbles may nucleate in normally volatile-undersaturated magmas as a result of this process and could mislead interpretation of magmatic conditions. Though such hypotheses are readily testable, no studies actually verified anything more than slight gradients in slow-diffusing elements (e.g., Beddoe-

Stephens et al., 1983). Until workers focused directly on assessing the fidelity of MI compositions (Lu et al., 1995; Manley, 1996), there remained the perception that MI could be unreliable.

MI studies displayed unexpected compositional variability: Another possible hindrance to wide acceptance of MI studies was the large variation in apparent volatile concentrations in MI from some eruptive units (e.g., Muenow et al., 1979; Melson, 1983; Sommer and Schram, 1983; Harris and Anderson, 1983; Palais and Sigurdsson; 1989). There are a number of geological reasons that can result in a spread of H_2O concentrations in MI from volcanic phenocrysts from a single unit. Magma mixing (Hervig and Dunbar, 1992), crystal settling (Anderson et al., 2000), degassing-induced crystallization (Sisson and Layne, 1993) and lava drainback and recycling (Wallace and Anderson, 1998) are all potential reasons to expect variations in volatiles within melt inclusions from a single pumice lump, or crystal. Moreover, MI can decrepitate and leak during or after eruptive ascent (Tait, 1992), and workers must make rigorous efforts to insure that leaked/degassed inclusions are avoided. Those workers familiar with experimental data or bulk whole-rock data on natural samples may have been somewhat leery of early data sets that showed wide and unexpected variations in the chemistry of included melts.

Lack of familiarity with MI: The problems outlined above caused some justifiable skepticism about the utility of MI for petrological and volcanological studies. To overcome the skepticism, it would be necessary to undertake numerous careful analytical studies. However, few of the critics of MI research were active in performing such analytical studies. In part, this stemmed from their general lack of familiarity with MI and their systematics. Studies of volatiles in MI often require grain-by-grain assessment of phenocrysts to observe cracks and capillaries. The most common petrographic mounts such as thin-sections or multi-grain mounts in epoxy are inadequate for inspection of MI populations (Lowenstern, 1995). As a result, to test the utility of MI requires familiarity with the principles of MI preparation and petrography. Those most likely to undertake rigorous studies of MI were those "inclusionists", already familiar with the principles of MI petrology.

Petrological priorities

Another important barrier to widespread use of melt inclusions was the lack of micro-analytical techniques, and the exploding development of high-precision bulk techniques. In the 1970s and 1980s, techniques such as x-ray fluorescence (XRF) and instrumental neutron-activation analysis (INAA) spurred a revolution in trace-element geochemistry of whole rocks. Improvements in mass spectrometers allowed researchers to focus on isotopic studies of terrestrial and planetary samples. The new data were interpreted in light of recently developed plate-tectonic theory and recognition that different magma types and environments had their unique trace-element and isotopic characteristics (Bottinga and Allègre, 1976; Basaltic Volcanism Study Project, 1981;

White and Hoffmann, 1982). At the same time, funding for planetary research increased with creation of NASA and the lunar missions.

Until the 1970s, petrologists lacked analytical tools to routinely characterize the contents of materials only tens of micrometers across. Even today, electron microprobe microanalysis of hydrous glasses remains difficult, and some MI contain high concentrations of light elements such as B and Li which require alternative spectroscopic techniques (e.g., Thomas, 2002) that only recently have become available. Until the 1990s, melt inclusions could not be analyzed reliably for trace elements, and analysis of volatile elements was qualitative. As a result, the broad petrologic and volcanological communities chose not to pursue MI as a means for understanding petrogenesis and volcanic phenomena.

Experimental versus analytical studies of volatiles: Even for studies of magmatic volatiles, other research approaches were preferred. It had long been recognized that magmatic volatiles had a crucial role in the generation, ascent and crystallization of magmas (Bowen, 1928). In the 1960s, 70s and 80s a variety of noteworthy and high-profile experimental studies were undertaken to determine the thermodynamic controls on volatile solubilities (Burnham and Jahns, 1962; Burnham and Davis, 1971; Eggler and Burnham, 1973; Wyllie, 1979; Whitney, 1984). Many workers justifiably reasoned that data on natural samples could not be properly understood without an experimental and thermodynamic basis for interpretation. Money and priorities were directed towards theoretical and experimental studies of magmatic volatiles. In natural samples, volatile concentrations were estimated by bulk analytical techniques on lavas quenched at the surface (e.g., Friedman et al., 1963) or underwater (Moore and Schilling, 1973). H_2O fugacities were later estimated by thermodynamic models based on the partitioning of K and Fe between biotite and feldspar (Wones, 1972), the Na/Ca ratios of the silicate melt (Merzbacher and Eggler, 1984) and other techniques. In general, P_{H_2O} was believed to be far below P_{total} in most igneous systems (Carmichael et al., 1974, p. 326). Given the lack of strong evidence for high abundances of other volatiles (e.g., CO_2, SO_2, H_2, N_2, Cl-bearing species), many workers concluded that most crustal magmas were both H_2O and vapor-undersaturated.

EMERGING IMPORTANCE OF MI IN VOLCANOLOGICAL STUDIES

With time, and with the development of infrared spectroscopy (Newman et al., 1986), the ion microprobe (Karsten et al., 1982) and improved electron microprobe methods for analysis of hydrous glasses (Nielsen and Sigurdsson, 1981), new data on MI compositions became increasingly abundant. The number of articles on "melt inclusion" or "glass inclusion" in the bibliographic database GeoRef® expanded from ~ 8 per year in 1985 to ~60 per year in the late 1990s. As data became widespread, workers became increasingly convinced that MI provided useful and reproducible data to constrain magmatic systems. Concern about boundary-layer phenomena was decreased as workers found little evidence for heterogeneity within individual inclusions (Lu et al., 1995). Post-entrapment changes were also small (Manley, 1996) or could be modeled effectively (Sobolev and Danushevsky, 1994). Other studies found

that variations in melt-inclusion compositions could be attributed primarily to magmatic processes (Dunbar and Hervig, 1992; Sisson and Layne, 1993).

Anderson et al. (1989) published the first large dataset to use infrared spectroscopic techniques to analyze a series of MI from a single eruption, the 760 ka Bishop Tuff. They found high and variable CO_2 concentrations that correlated with H_2O in a manner consistent with isobaric, gas-saturated fractionation of different magma batches. Later work by Anderson and colleagues (Skirius et al., 1990; Lu et al., 1992) and others (Dunbar and Hervig, 1992; Hervig and Dunbar, 1992) spurred the emergence of the Bishop tuff as a "proving ground" for MI-related research. The wealth of carefully collected data, and intriguing geochemical trends demonstrated that MI faithfully record a variety of magmatic processes that cannot be detected by most other avenues of petrologic research. Using the Bishop Tuff alone, recent studies have used MI to assess magma residence times (Van den Bogaard and Schirnick, 1995; Christensen and Halliday, 1996), excess Ar and geochronology (Winick et al., 2001), volatile phase accumulation (Wallace et al., 1995, 1999) and crystal settling (Anderson et al., 2000).

MI are now increasingly used as a volcanological tool to unravel the complexities of very recent eruptions. Dunbar et al. (1989) undertook one of the first studies to use the ion microprobe to quantify H_2O in MI. They analyzed dissolved volatiles in homogeneous glassy MI from the Taupo volcanic center (New Zealand). They found no significant difference between the dissolved volatile contents in phenocrysts from plinian and phreatomagmatic deposits. This non-correlation between pre-eruptive volatiles and eruptive style has been noted by many subsequent workers and is likely due to the influence of pre-eruptive (post-entrapment) degassing and ascent rate on eruptive style (Lowenstern and Mahood, 1991; Bacon et al. 1992; Lowenstern, 1993; see also Mangan and Sisson, 2000). Roggensack et al. (1997) studied the degassing history of eruptions from Cerro Negro (Nicaragua). They found that magmas erupted explosively in 1992 had equilibrated at greater depths than lavas erupted three years later as effusive lavas. Shallow subsurface degassing, plausibly during the three-year period between eruption, led to the decrease in explosivity. In a subsequent paper Roggensack (2001a) looked at the products of an 1867 eruption at Cerro Negro and found that MI glass composition and gas saturation pressures correlated with crystal size, implying that large crystals grew at greater depth (up to >15 km). All crystals apparently formed less than two years prior to eruption. A similar study at Fuego (Guatemala) showed that MI hosted by small, recently formed olivine crystals showed tremendous variability in volatiles and major-element composition, revealing hybridization of a heterogeneous magma column shortly prior to eruption (Roggensack, 2001b).

Similar studies on Italian volcanoes demonstrate the importance of degassing and crystallization in determining eruptive style. Métrich et al. (2001) looked at crystal-rich scoria at Stromboli, which are erupted during persistent and mild strombolian activity, and found that the MI are degassed and contain more evolved glass than MI from compositionally equivalent whole-rocks erupted as crystal-poor pumices during more energetic explosive activity. Evidently, volatile-rich magma batches can either ascend rapidly to produce the explosive eruptions, or they can intrude to shallow depths and

degas quietly to spur the typical strombolian eruptions. Marianelli et al. (1999) were able to differentiate different depths for magma reservoirs feeding the 1944 eruption of Vesuvius including K-tephritic melts saturated with CO_2-rich gas at pressures over 300 MPa. These sorts of studies provide new opportunities for evaluating the relationship between crystal growth, ascent rate, degassing and eruptibility of magma.

Besides these volcanological studies, petrologists have increasingly used MI to understand magma generation and the diversity of melt sources available to volcanic systems (Sobolev, 1996; Frezzotti, 2001). A recent compilation (Hauri et al., 2002) provides an excellent assembly of papers on MI systematics and analysis, primarily focussed on the petrology of mafic magma systems. Clearly, the petrological and volcanological communities have become familiar with the pitfalls of melt-inclusion analysis, and have accepted that valuable data can be collected through careful studies.

NEW OBSERVATIONS OF BUBBLES, FLUIDS AND EXOTIC MELTS

With acknowledgement that glass compositions, including dissolved volatiles, can be determined successfully, we can look critically at MI with more complex petrography. Anyone who has undertaken a study of MI has likely seen at least a few challenging inclusions that defied simple interpretation. Some MI contain large bubbles or bubbles associated with little to no glass at all. Some inclusions contain crystals not normally found as phenocrysts, or melts very unlike those of the whole-rock (Davidson and Kamenetsky, 2001; Thomas et al., 2000).   In some cases, inclusions seem to imply magmatic temperatures, pressures and compositions beyond the norm. Roedder (1979b) wrote: "Theoretical igneous petrology has evolved considerably in recent years, but observational petrography is still capable of providing some surprises and may even inspire some new theories."

Origins of bubbles in MI:

Many studies have avoided bubbles, or inclusions containing bubbles, in an effort to simplify interpretation of MI compositions. However, the size, distribution and composition of bubbles reveal the history of fluid immiscibility, degassing and ascent rate of the host magma. There are at least three reasons why a bubble is found within an MI: 1) it formed during near-constant-volume cooling of homogenous silicate melt, 2) it formed during decrepitation/leakage of the MI and, 3) it represents magmatic vapor that was co-trapped with melt in a mixed inclusion.

"Shrinkage Bubbles": As with all FI, a bubble can nucleate due to isochoric (constant volume) cooling of a trapped homogeneous liquid, causing depressurization and resultant saturation with a low-density phase (Fig. 1a). The bubble that forms is often called a "shrinkage bubble". As discussed by Roedder (1984, p. 50), it should be considered a separate phase formed by immiscibility of the melt and vapor. Homogenization experiments in the laboratory typically show that during heating the bubbles disappear at temperatures greater than when they re-appear during cooling (Roedder, 1979b). Apparently, significant underpressures are necessary to induce bubble nucleation, especially when cooling rates are high. Mangan and Sisson (2000)

also have shown this to be the case for macroscopic rhyolitic systems depressurized rapidly in the laboratory.

When equilibrium is maintained, the internal pressure of the bubble should equal that of the melt. Melt-vapor equilibration should cease at temperatures below T_g, the glass transition temperature, after which the bubble contents may cool isochorically. This should result in high pressures (and thus densities) for fluids within the "shrinkage" bubble. Typically, though, such bubbles appear empty, or at least without a liquid phase, allowing H_2O pressures no greater than that for liquid saturation at 25°C (2.6×10^{-3} MPa). Some workers interpret the empty bubbles to reflect extreme hydration of the glass adjacent to the bubble rim during cooling of the MI (Lowenstern, 1995). It should be noted though, that high-density fluids are sometimes found within MI-hosted bubbles [Naumov et al., 1993 (Fig 2c); this article (Fig 1B)].

The volume of bubbles can range widely, (0.1 – 5 vol.%) and is known to depend on the cooling history of the MI. Small MI often fail to nucleate bubbles because of surface-tension effects (Roedder, 1979b). Many workers have shown that MI from rapidly quenched Plinian pumice often lack bubbles entirely (Clocchiatti, 1972; Sommer, 1977; Beddoe-Stephens et al., 1983). Both theoretical (Tait, 1992) and experimental (Lowenstern, 1994) studies confirm that rapid cooling is likely to permit MI quenching without bubble formation. The latter study found that quartz-hosted MI of peralkaline rhyolites (pantellerites) failed to nucleate bubbles if cooled faster than ~ 300°C/minute from their homogenization/entrapment temperatures. As cooling rates decrease, bubbles have more time to form, to equilibrate with their host melts and thus to grow and to partially crystallize (Skirius et al., 1990). In intrusions, crystallization of the melt at near-magmatic temperatures will allow the MI-hosted bubbles to reach as much as 15-20 % of the total inclusion volume (Student and Bodnar, 1996).

Decrepitation or leakage through capillaries: When the host crystal cools more slowly, within a lava or dense pyroclastic flow, the bubble has time to form, causing the internal pressure of the MI to rise (Tait, 1992). Often these slowly cooled rocks have cracked crystals with MI that contain numerous large bubbles (Lowenstern, 1995; Best and Christiansen, 1997). Best and Christiansen (1997) surveyed scores of Tertiary pyroclastic flow rocks in Nevada and Utah (USA) and noted that broken crystals in ash flow tuffs can often be attributed to the decrepitation of MI during eruption. MI that partially or fully decrepitate are likely to have numerous large bubbles and degassed glass (Skirius et al., 1990). Feldspar hosts are more likely to break than quartz owing to their good cleavage and they can show strain birefringence adjacent to the inclusion (Skirius et al., 1990). Occasionally, degassed/leaked inclusions can be recognized by their glassy nature, when compared to pristine inclusions that cool slowly from H_2O-rich melt and become partially or entirely crystalline. Lowenstern and Mahood (1991) found that leaked peralkaline rhyolite inclusions were glassy and green whereas unleaked inclusions were microcrystalline and blue, presumably from crystallites of alkali amphibole (also see Anderson, 1991).
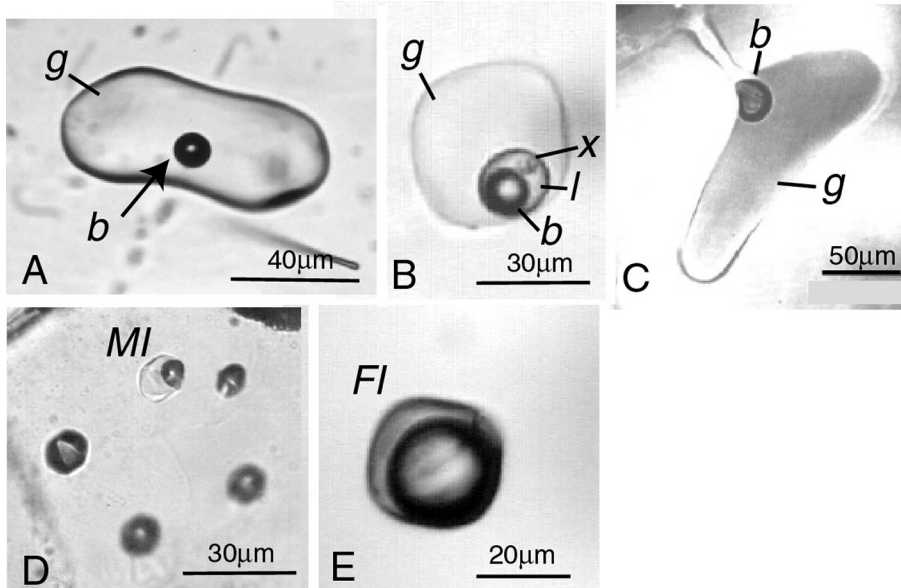
Fig. 1. MI and FI in volcanic quartz. A) MI with glass (g) and associated shrinkage bubble (b) from ignimbrite unit of the 1912 eruption at the Valley of Ten Thousand Smokes, Alaska (VTTS). B) Multiphase shrinkage(?) bubble from the "pink unit" of the "tuff of Pine Grove", (Utah) contains a vapor (b), a crystal (x) and a liquid (l). C) Hourglass inclusion from the 1912 eruption at the VTTS. D) Co-planar MI and FI in a lava from the Miocene Chocaya volcanic center, Bolivia. E) Moderate-density, vapor-rich FI from the "air fall" unit of the "tuff of Pine Grove". All photos by J.B. Lowenstern (unpublished data) except C, from Lowenstern (1993).

Another category of leaked MI has been called "hourglass" inclusions (Anderson, 1991), which consist of glass or crystallized melt connected to the outside of the host crystal by a canal or capillary (Fig. 1C). At magmatic temperatures, melt can be expelled from such inclusions, especially during magma ascent, causing nucleation and expansion of bubbles. The capillary can be sufficiently narrow that it forms a throttle that prevents complete equilibration with the external atmosphere. Anderson (1991) provided a theoretical framework and numerical method for understanding the devolatilization of such inclusions, with the aim of calculating the rate of magma ascent of hourglass-inclusion-bearing quartz. The article contains myriad observations and photos that have proven useful to a scientific community striving to understand the formation of MI and their behavior during magma ascent and eruption.

Presence of a separate vapor phase in mixed inclusions: The simplest evidence for the presence of vapors or other non-silicate fluids is their entrapment either as FI or within mixed inclusions in igneous phenocrysts. Vapor-rich FI that are trapped contemporaneously with (and separately from) silicate melt are common in intrusive rocks (Roedder, 1984, 1992; Frezzotti, 1992; Touret and Frezzoti, 1993; Lowenstern et al., 1997; Audétat et al., 2000; Dietrich et al., 2000; Frezzotti, 2001). Tuttle (1952) noted that whereas intrusive rocks often contained such inclusions, they were rare in volcanic rocks, which instead contained silicate MI. Anderson (1991) discussed a variety of possible reasons that vapor-rich FI are rarely present in the crystals within volcanic rocks. He concluded that any MI with a trapped vapor bubble would create a pressure gradient during magma ascent, preventing the inclusion from sealing. Tait (1992) postulated that MI with co-trapped vapor would be more likely to rupture during eruption. Also possible is that small bubbles are rare in long-lived magma chambers, and bubbles are unlikely to be trapped within sub-cm-sized crystals. As the magma becomes crystal-rich and the liquid is closer to saturation with a pure-H_2O vapor, small bubbles may nucleate and would be trapped more commonly, explaining the ubiquity of vapor-rich inclusions and contemporaneous entrapment of silicate liquid in intrusive rocks.

Nevertheless, some workers have discussed evidence for vapor-rich inclusions in volcanic rocks. Gutmann (1974) showed tubular voids in labradorite phenocrysts that were postulated to result from trapping of a vapor phase. Naumov and colleagues wrote a series of papers (summarized in Naumov et al., 1996) that discussed high-density H_2O-rich fluids in quartz from rhyolites of the Western Carpathians (Slovakia) and other locations. Vaggelli et al. (1993) discussed rare CO_2-rich FI in olivines and other minerals from mafic calc-alkaline eruptions at Stromboli. Métrich et al. (1993) performed 26 homogenization experiments on CO_2-rich FI in olivine phenocrysts found in tephras from the 1989-90 eruption at Etna. The thermometric data indicated entrapment at pressures of at least 100 to 140 MPa at 1100°C. Lowenstern (1995) showed photos of large (~100 μm) inclusions in quartz that had negative crystal shapes but were completely empty. As they were found in the same growth zones as glassy MI, he concluded that they were leaked FI (also see Fig. 1D, 1E). Pasteris et al. (1996) showed photos of much smaller vapor-rich and liquid-rich FI within volcanic phenocrysts from Pinatubo and concluded they were trapped during magma ascent.

Other evidence for entrapped vapors comes from anomalous bubbles within MI that cannot be explained either by shrinkage or leakage. Lowenstern et al. (1991) discovered that bubbles within some quartz-hosted MI in peralkaline rhyolites had anomalous Cu concentrations, and the Cu was located in Cu sulfides associated with salts precipitated on the bubble walls. The heterogeneous nature of the enrichment and association with large bubbles was consistent with occasional trapping of Cu-rich magmatic vapors (see also Lowenstern, 1993). Later studies from other volcanic systems have noted similar behavior for Cu and other ore metals. Yang and Scott (1996) found metal-rich bubbles in MI from submarine basalts of the Manus back-arc basin offshore in the western Pacific. Mineralization in MI-hosted bubbles included

sulfides and chlorides of Cu, Zn and Fe.   Kamenetsky et al. (2002) analyzed small crystals in FI, bubbles in MI and bubbles in matrix glass from a variety of young submarine rocks. They document high volatility of a variety of metals and anions in low-pressure vapors exsolved from mafic magmas. Many of the bubbles in MI were interpreted as magmatic vapor trapped along with melt in mixed inclusions.

"Hydrosaline melts" in mixed inclusions: Perhaps hundreds of articles, primarily from the economic geology literature, have outlined evidence for highly saline fluid inclusions in shallow intrusive rocks. Typically, these hydrosaline melts coexist with CO_2-H_2O-dominated vapors at sub-magmatic temperatures (Roedder, 1984). Roedder and Coombs (1967) carefully documented immiscibility between highly saline fluids and silicate melts from the granite xenoliths of Ascension Island. More recent work on xenoliths and granitic rocks have found abundant evidence for hydrosaline phases, often in equilibrium with a CO_2-bearing vapor phase as well as silicate melt (Frezzotti, 1992; De Vivo et al. 1992, 1993; Yang and Bodnar, 1994; Belkin et al., 1996; Kamenetsky et al., 1999; Gilg et al., 2001; Fulignati et al., 2001; Campos et al., 2002). Webster and Rebbert (2001) studied Cl concentrations in silicate MI from the same xenoliths studied by Roedder and Coombs (1967). They noted a drop in Cl/H_2O with differentiation, consistent with fluid-saturated magmatic crystallization.

Though many of these saline fluids appear to form late during crystallization, hydrosaline melts have also been noted in crystal-poor volcanic rocks. Typically, they are found coexisting with silicate melt in mixed inclusions. For example, Solovova et al. (1991) and Lowenstern (1994) found hydrosaline melt droplets (~60-80% NaCl equivalent) within quartz-hosted silicate melt inclusions from peralkaline rhyolites. The hydrosaline fluids apparently were not trapped alone as discrete inclusions within the phenocryst phases, though similar crystallized salt droplets were found in the outgassed glassy matrix. The presence of the hydrosaline melt, together with a CO_2-H_2O-vapor and silicate melt at low pressure (< 100 MPa) caused buffering of the Cl concentration of the melt at a near-fixed value (Shinohara, 1994; Signorelli et al., 1999).

The hydrosaline melt need not consist solely of alkali chlorides and H_2O. Veksler et al. (2002) describe hydrothermal experiments at 100-200 MPa and 450-900°C where they formed silicate melt, vapor and a hydrosaline brine rich in P, B and F. The skarn-hosted hydrosaline melts from Vesuvius discussed both by Gilg et al. (2001) and Fulignati et al. (2001) crystallized to form carbonates, sulfates and fluorocarbonates as well as chlorides.

"Fluxed melts" and fluid-melt miscibility

Perhaps the most striking new development in melt-inclusion research is the work on pegmatitic melts, including homogenization of crystallized inclusions and quantification of their bulk compositions. Russian workers have studied this field for decades, but only recently have analytical techniques allowed quantification of some of the volatile and light elements that appear to be important in generation of these rock types. Many of these MI come from intrusive rocks, including pegmatites, but as discussed below, some are found in volcanic rocks as well. Kovalenko et al. (1996)

studied MI from the pegmatites of Volhynia (Ukraine) and found them to be poor in SiO_2 (~60 wt.%), but rich in Al, F, H_2O, Li, Rb and a number of other trace elements. Thomas et al. (2000) recently showed that pegmatitic quartz from the Variscan Ehrenfriedersdorf complex (Germany) trapped two immiscible silicate liquids, one higher in silica and lower in H_2O than the other. The fluids were trapped over a range of temperatures, beginning with a single supercritical fluid and separating along a solvus into two fluids that grew more distinct from each other as temperature declined. At 650°C, the more hydrous fluid contained ~38 wt.% SiO_2, 4% B_2O_3 2.6% F, 6% Cl and over 30% H_2O. At the same temperature, the silica-rich melt contained ~65% SiO_2, over 3% P_2O_5, 4.5% F and ~10% H_2O. Both phases contained more than 0.3% Rb_2O. A related study found enrichment of ore-related trace elements, particularly Sn (Thomas and Webster, 2000). Davidson and Kamenetsky (2001) found similar inclusions in subvolcanic rocks from the Rio Blanco porphyry system in Chile and hypothesized that these pegmatitic fluids may have an important role in some ore-forming systems. Sirbescu and Nabelek (2000) found evidence for equilibration between a pegmatitic melt containing Li, Cs, Rb, P and B with a H_2O-rich- and a CO_2-rich fluid. The three fluids coexisted at temperatures of 340°C and 270 MPa, some of the lowest known temperatures for silicate melts in the crust.

Such critical behavior is expected for melt-volatile systems, but at higher pressures and temperatures than demonstrated by Thomas et al. (2000). For example, Bureau and Keppler (1999) used an externally heated diamond-anvil cell to explore miscibility between silicate melts and hydrous fluids. Haplogranite-H_2O showed critical behavior above 900°C at 1.5 GPa. Sowerby and Keppler (2002) performed similar experiments on a synthetic pegmatite and were able to depress the critical pressure to 400 MPa at ~600°C. In comparison, the fluxed melts discussed by Thomas et al. (2000) exhibited supercritical behavior at temperatures > 700°C at only 100 MPa pressure. Such highly fluxed melts probably cannot ascend to the surface without crystallizing, and may never erupt as volcanic rocks. But erupted precursors of such rocks, including melts with very high halogen concentrations are well-documented (Congdon and Nash, 1991; Webster and Duffield, 1994). Moreover, these melts clearly have an important role in the formation of pegmatites and Sn-W, magma-hosted ore deposits.

LOOKING TO THE FUTURE

Improvements in techniques for analysis of MI have advanced our understanding of mantle melting and metasomatism, ore formation, pegmatite differentiation, volcanism, and planetary science. MI reveal that igneous melts can be diverse, can interact with unexpected materials and usually leave behind clues to their history. Sometimes igneous fluids are miscible when we expect them to unmix; other times numerous fluids are present when we expect only one. It is surprising and humbling to learn that micrometer-sized features yield so much information about km-sized magma bodies. Occasionally, analysis of MI may lead us astray. Boundary layers, re-equilibration and crystallization can somewhat alter the composition of these small parcels of igneous fluid. But through careful observation and analysis, we can identify these secondary

processes and reconstruct the history of degassing, crystallization, mixing and unmixing recorded by melt inclusions in igneous rocks.

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