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Role of fluid and melt inclusion studies in geologic research

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ABSTRACT

Although fluid inclusions were apparently known to early naturalists, actual research on fluid and melt inclusions began only in the mid-1800s and grew very slowly for the next 100 years. Russian scientists began systematic studies of inclusions in the 1930s, but it was not until about 1960 that publications mentioning or using fluid inclusions began to increase from a few each year to the present annual level of about 700. Early research focused on ore deposits, first on temperatures and salinities of ore fluids and then on their stable isotopic and major element compositions. Later work extended to fluids in sedimentary and metamorphic environments. Publications using or mentioning melt inclusions only began to increase in number in about 1980 and have grown to today's level of about 200 per year. Early work on melt inclusions focused on igneous rocks with an emphasis on immiscibility and volatile elements and then on rare elements. Recent research on both fluid and melt inclusions has taken advantage of single inclusion analytical methods to investigate speciation and partitioning in both natural and experimental magmatic and aqueous systems.

Key words: evaporite, fluid inclusion, intrusive, melt inclusion, metamorphic, ore deposit, pegmatite, volcanic

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INTRODUCTION

Fluid inclusions have been of interest to naturalists for millennia. The first reference to what were probably fluid inclusions is found in the Natural History of Pliny the Elder written in about 75 AD. In about 400 AD, Claudius Claudianus wrote a poem entitled '*On a crystal enclosing a drop of water*' that almost certainly is about a large fluid inclusion in a quartz crystal. According to Lemmlein (1950; quoted in Roedder 1984), the first specific description of fluid inclusions was by Abu Raihan al-Biruni in the 11th century in his book Kitab al-Jawahir (Precious Stones). In the 13th century, Ahmad al-Tifashi in Cairo wrote 'Azhar al Afkar' (Best Thoughts on the Best of Stones) in which he mentions inclusions ('uyub'), liquid (inclusion) ('ma'), air bubble ('rih'), and mud inclusion ('teen'). Also, at approximately the same time, Albertus Magnus, a German medieval scholar and Archbishop of Cologne, wrote a book on lapidary ('de mineralibus') with a note on fluid inclusions in beryl, that states 'Beryl is a shining and transparent gemstone of pale color. The most precious kind is the one, in which you see water moving when you turn him' (translation from German provided by Albert Gilg). The earliest known description in English was by Robert Boyle (1672), who described a moving bubble in a quartz crystal.

Systematic observations of fluid and melt inclusions really began in the 1800s with the pioneering microscopic studies of Davy (1822), Brewster (1823), Sorby (1857), and Zirkel (1873), but then languished for most of the next century. The lack of follow-up to these original studies is puzzling because Sorby (1858) described aqueous inclusions in vein quartz, with homogenization temperatures that decreased outward from an intrusion, the perfect source for ore-forming hydrothermal fluids. When attention finally returned to fluid and melt inclusions, however, they provided critically important information on a wide and growing range of geologic processes from volcanism to ore formation.

FLUID INCLUSION STUDIES

Fluid inclusion studies that trickled into the literature during the late 1800s and early 1900s focused largely on ore deposits and indicated that they contained fluids with a wide range of compositions. Sorby & Butler (1869) reported CO_2-bearing inclusions in sapphire; Phillips (1875) described aqueous inclusions in the Cornish tin veins; Lindgren (1905) reported salt crystals in fluid inclusions at the Clifton-Morenci porphyry copper deposit; and Newhouse (1932, 1933) described saline fluids without salt crystals in Mississippi Valley-type (MVT) deposits. By the 1930s, Russian geologists had begun fluid inclusion research on a wide range of ore deposits that was summarized by Ermakov *et al.* (1950), Zakharchenko (1950), and Lemmlein (1956) (see also the discussion in Roedder 1984, p.4–6). Work outside Russia was less comprehensive, but included early studies on geothermometry by Ingerson (1947), pegmatites by Cameron *et al.* (1951), and decrepitation by Smith (1952), as well as the historical summary of previous work on fluid and melt inclusions by Smith (1953).

Wider application of these pioneering studies was limited in part by the debate about whether fluid inclusions leaked (Kennedy 1950; Skinner 1953) and by the lack of suitable equipment to measure homogenization and freezing temperatures. Resolution of the leakage dispute by the experiments of Roedder & Skinner (1968) and development of simple heating and freezing equipment for use on microscopes by Roedder (1962, 1963) resulted in wider acceptance of fluid inclusion research. By about 1970, publications using or mentioning fluid inclusions became common and research grew rapidly from a few papers per year to the present rate of over 700 per year (Fig. 1A).

Some of the earliest fluid inclusion studies were surveys showing the range of temperatures and salinities in various ore deposit types (Fig. 2A), including MVT Pb-Zn-Ba-F (Roedder 1967), Bolivian tin (Kelly & Turneaure 1970), and porphyry Cu deposits (Roedder 1971). This work, when combined with analyses of inclusion leachates (Hall & Friedman 1963) and isotopic compositions (Rye & O'Neil 1968), provided support for efforts to determine the different sources from which hydrothermal solutions were derived (White 1957, 1974). Later studies shed light on processes occurring during ore formation, including boiling in epithermal systems (Kamilli & Ohmoto 1977) and halite saturation in porphyry copper and chimney–manto systems (Erwood *et al.* 1979; Wilson *et al.* 1980), as well as on vertical zoning in inclusion fluids in epithermal, porphyry copper, and orogenic gold systems (Bodnar & Beane 1980; Robert & Kelly 1983; Dilles & Einaudi 1992).

After about 1980, fluid inclusion studies began to be applied to problems in sedimentary and metamorphic geology (Hollister & Crawford 1981; Roedder 1984; Goldstein & Reynolds 1994) (Fig. 2B). Topics of greatest interest included characterization of metamorphic fluids (Crawford 1981; Touret 1981) basin evolution and fluid expulsion, including hydrocarbons (McLimans 1981; Burruss *et al.* 1983), variations in chemistry of seawater through time (Channer *et al.* 1997; Timofeef *et al.* 2006), generation of evaporites (Ayora *et al.* 1994), and dolomitization (Machel 1987; Aulstead *et al.* 1988). This work expanded rapidly, and by 2012, the proportion of fluid inclusion studies that focused on ore deposits had decreased from 100% of all papers in the early 1960s to about 35% (Fig. 1A).

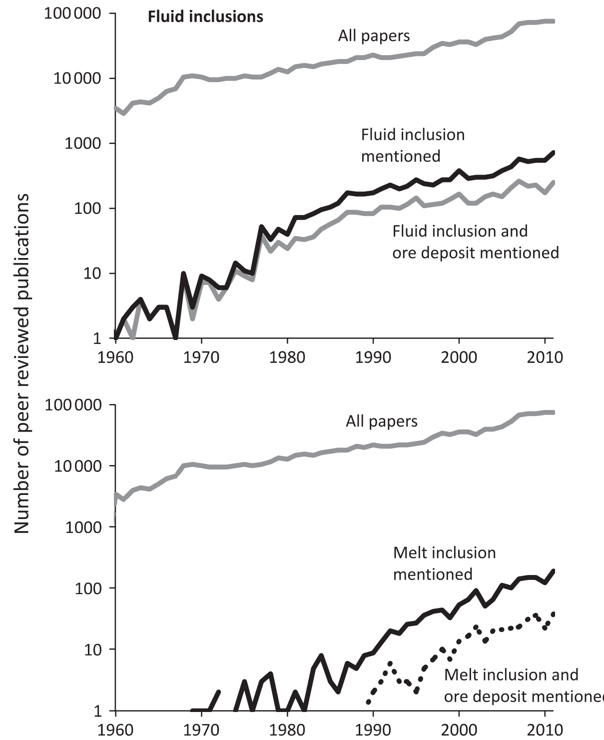

Fig. 1. Growth of peer-reviewed publications that mention fluid or melt inclusions or that mention both ore deposits and fluid or melt inclusions. Note that fluid inclusion studies began to grow sooner than melt inclusion studies, and that ore deposit studies have made up a much greater fraction of fluid inclusion studies. This figure, and the two that follow, is based on Georef searches for publications written in English or with titles or abstracts that were translated into English. This approach is comprehensive with the exception of early studies by Russian and French authors, some of which are mentioned here.

Fluid inclusion studies were enhanced greatly by the development of a method to synthesize inclusions of known composition under controlled conditions (Sterner & Bodnar 1984; Bodnar & Sterner 1985). This led to a burst of studies that used synthetic inclusions to investigate a wide range of geochemical problems (Fig. 3), including a series of important studies of phase equilibria in H_2O-salt-gas systems starting with Bodnar *et al.* (1985) and extending to Lin & Bodnar (2010). Ironically, synthetic inclusions were also used to confirm that fluid inclusions could leak under special circumstances (Hall & Sterner 1993, 1995; Sterner *et al.* 1995).

By about 2000, just as the number of synthetic inclusion studies had tapered off (Fig. 3), analysis of single

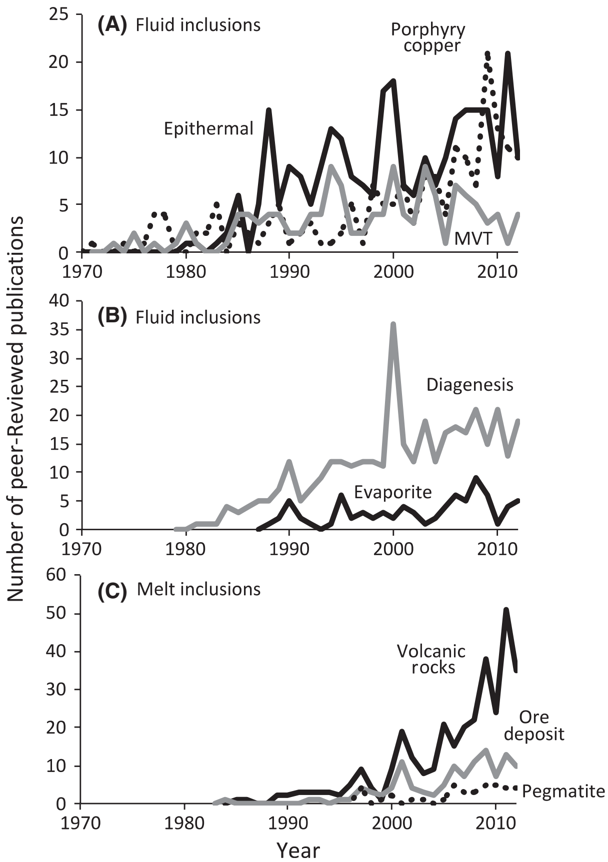

Fig. 2. Growth in number of peer-reviewed publications that mention fluid inclusions in epithermal, porphyry copper, and MVT deposits (A) and in diagenetic and evaporite settings (B) and melt inclusions in volcanic rocks, ore deposits, and pegmatites (C) (data from Georef). There is considerable overlap in some of these categories, especially between MVT deposits and evaporites.

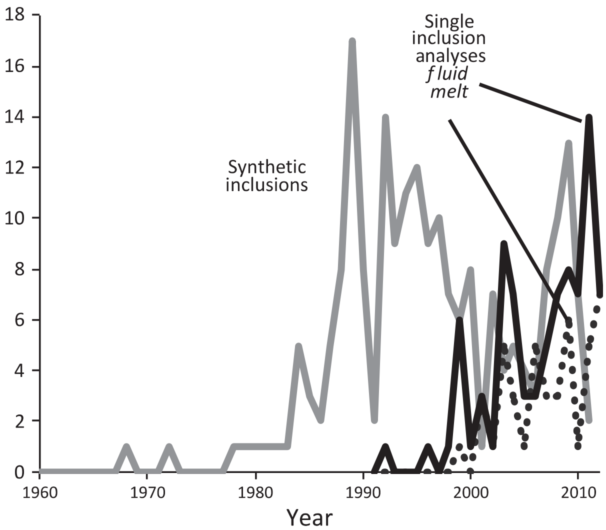

Fig. 3. Change in peer-reviewed publications focused on synthetic inclusions (as mentioned in title) compared with the change in publications that mention single inclusion analyses by PIXE or ICP-MS methods (data from Georef). Note the increase in synthetic inclusion publications after about 2000 related to experimental studies using single inclusion analyses.

fluid inclusions had improved so much that trace elements were detectable. Earlier efforts to analyze individual inclusions had used Raman and quadrupole MS methods that were most effective on gaseous inclusion contents (Blamey 2012; Frezzotti *et al.* 2012). Development and validation of particle-induced X-ray/gamma-ray emission (PIXE/PIGE) and laser ablation, inductively coupled mass spectrometry (LA-ICP-MS) methods for analyzing trace elements in individual fluid and melt inclusions (Heinrich *et al.* 1992, 2003; and Audétat *et al.* 2000a,b; Allan *et al.* 2005) offered a new way to link fluid inclusions to migration of trace elements in the crust. Use of infrared light microscopic methods (Campbell *et al.* 1984) also allowed analysis of single inclusions in opaque ore minerals (Kouzamanov *et al.* 2010). More recently, infrared synchrotron X-ray fluorescence, X-ray absorption near edge structure (XANES) measurements have been used to determine element speciation in single inclusions (Richard *et al.* 2012).

MELT INCLUSION STUDIES

Melt inclusions were also recognized in early studies (Sorby 1858), but little attention was given to them during the next hundred years. Their 20th century renaissance was also much slower; whereas fluid inclusion studies began to grow in about 1960, the rise in melt inclusion studies did not begin until about 1980 (Fig. 1B). The stage for this rise was set between 1940 and 1980, when Russian geologists described observations on igneous rocks, including some pegmatites, which indicated extensive immiscibility in silicate systems (Zakharchenko 1968; Clocchiatti 1975; Sobolev & Kostyuk 1975). Elsewhere, Roedder & Coombs (1967) described complex inclusions containing silicate glass, saline fluid, and gas that were thought to represent 'simultaneous coexistence of immiscible silicate and saline fluid phases in the granitic melt', and Roedder & Weiblen (1970, 1971) showed the importance of immiscibility in lunar samples.

Early melt inclusion studies had their own controversy about leakage (Anderson 1991; Lowenstern & Mahood 1991), as well as the question of whether boundary layer effects around the host crystal meant that the trapped melt did not represent the bulk melt (Watson *et al.* 1982; Bacon 1989). While guidelines for melt inclusion observations and interpretations have improved over the years (Lowenstern 1995, 2003; Bodnar & Student 2006) and have provided a foundation for wider application of melt inclusion studies, controversies remain concerning the validity and interpretation of melt inclusion data (c.f., Danyushevsky *et al.* 2002; Steele-MacInnis *et al.* 2011).

The first generation of modern melt inclusion studies focused on felsic and mafic volcanic rocks to evaluate the degassing of H_2O, CO_2, S, and Cl and the role of volatiles in volcanic eruptions (Harris & Anderson 1984; Anderson *et al.* 1989; Dunbar *et al.* 1989). Melt inclusions in volcanic rocks were also used to evaluate the spatial heterogeneity of volatile contents in magma chambers; rates, locations, and timing of crystal growth; and magma mixing (Metrich & Clocchiatti 1989; Dunbar & Hervig 1992; Christensen & Halliday 1996; Roggensack *et al.* 1997).

Early studies of melt inclusions in intrusive rocks focused on granites enriched in fluorine, tin, and other rare elements (Naumov *et al.* 1977; Kovalenko *et al.* 1984) and on the vapor phase and/or highly saline liquid that was present in many inclusions (Roedder 1984; Frezzotti 1992). These studies went on to estimate the partitioning between magma and vapor or between magma and aqueous phase of volatile constituents (Stix & Graham 1996), as well as ore elements that were concentrated enough to be detected by microprobe (Reyf 1997; Webster *et al.* 1997). Development of PIXE and laser-ablation ICP-MS methods (Fig. 3) allowed an increase in studies of ore elements in melt inclusions from deposit types associated with igneous rocks, including tin and molybdenum deposits (Webster *et al.* 1996; Dietrich *et al.* 2000; Audetat 2010), porphyry copper deposits (Student & Bodnar 2004), geothermal systems (Chambefort *et al.* 2012), carbonatites and rare earth elements (Qin *et al.* 2007; Xie *et al.* 2009), and IOCG deposits (McPhie *et al.* 2011).

THE FUTURE

The much lower detection limit for most elements afforded by PIXE/PIGE, LA-ICP-MS, and other advanced analytical methods has opened a new chapter in inclusion research. Most early single inclusion analytical studies focused on natural inclusions with an emphasis on changes in element concentrations in fluids as they evolved through the ore-forming process or on partitioning of elements between or among magma, fluid, and vapor (Ulrich *et al.* 1999, 2001; Audétat *et al.* 2000a,b; Appold *et al.* 2004). As methods were perfected, they have been extended to analyze inclusions in experimental charges that were generated under known conditions (Simon 2003; Bell *et al.* 2011; Frank *et al.* 2011). These studies are providing basic information on equations of state and solubilities that are needed to develop rigorous chemical models for the generation and evolution of magmas and magmatic fluids, and the migration and deposition of trace elements in all types of hydrothermal solutions. Future inclusion research will undoubtedly expand to take on new questions. Possible directions include age measurements of inclusion fluids (Wayne *et al.* 1996), the use of biomarkers, noble gas isotopes and halogens to trace the source of inclusion fluids (Dutkiewicz *et al.* 2006; Kendrick *et al.* 2011), and the study of microorganisms in fluid inclusions (Naumov *et al.* 2013).

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