https://doi.org/10.5194/egusphere-2023-213
Preprint. Discussion started: 30 March 2023

A discovery of nanoscale sulfide droplets in MORB glasses: Implications for the immiscibility of sulfide melt and silicate melt

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https://doi.org/10.5194/egusphere-2023-213
Preprint. Discussion started: 30 March 2023

Abstract

Sulfur forms an immiscible liquid upon saturation in magma, and sulfide droplets were commonly found in fresh mid-ocean ridge basalt (MORB) magmas. In this paper, scanning electron microscopy (SEM) determined that MORB samples were primarily fine-grained and weakly phyric, with hypocrystalline to vitreous textures. A focused ion beam cut from the MORB glasses examined by transmission electron microscopy (TEM) revealed a range of nanoscale sulfide droplets (10–15 nm), featuring rounded shapes and smooth edges. Texturally, the droplets were crystalline and homogeneous in composition. Elemental S, Na, Fe, Cu, and Ni were evenly distributed within the droplets, while the content of element Si, Al and O are less in the droplets. Previous reports have elucidated the immiscibility between sulfide and silicate melts, and the structure of the silicate melt also affects the size distribution of sulfide droplets. This is the first report on nanoscale sulfide droplets within MORB glasses, and those results indicated that nanoscale sulfide droplets were the initial phase of sulfide saturation; such insight may prove useful in understanding how siderophile and chalcophile elements behaved during sulfide crystallization. In addition, this study determined the immiscibility of sulfides and silicate melts occurred in the early nanometer stage, the immiscibility of sulfides in magmatic Ni-Cu sulfide deposits was the key to the formation of magmatic Ni-Cu sulfide deposits. Therefore, all immiscibility phenomena may occur in the nanometer stage during magma evolution.

Keywords: MORB; sulfide melt; silicate melt; TEM; SEM

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1. Introduction

There are two basic differentiation modes in magma evolution: segregation crystallization and immiscibility, with liquid immiscibility the more important for magma evolution (Charlier and Grove, 2012; Kamenetsky et al., 2013; Thompson et al., 2007; Veksler et al., 2008; Magloughlin, 2005). Immiscibility refers to the process by which homogeneous magma decomposes into two melts of very different compositions due to changes in temperature, pressure, composition, etc. Immiscibility has been described in a broad compositional range of natural magmas such as basalt (Skinner and Peck, 1969; De, 1974), fourchite (Philpotts, 1972), lunar rocks (Roedder and Weiblen, 1970), and various volcanic rocks (Philpotts, 1982). Sulfur forms an immiscible liquid upon saturation in magma (e.g., sulfide droplets) (Moore and Calk, 1971; Moore and Schilling, 1973; Patten et al., 2012; Yang et al., 2014). Yeats and Mathez (1976) discovered spherical iron sulfide globules in the walls of vesicles in glassy selvages of tholeiitic basalt. Czamanske and Moore (1977) discovered spherical globules in phenocrysts and glass of submarine basalt from the Mid-Atlantic Ridge.

The sulfur in MORB magma is saturated before magma eruption, and may also be saturated in the source region (Yeats and Mathez, 1976; Czamanske and Moore, 1977). Sulfide droplets were commonly reported in fresh MORB glasses (Maclean, 1969; Holzheid, 2010). Therefore, the well-preserved sulfide droplets in MORB glass provide an opportunity to study the sulfide melt-silicate melt equilibrium. (Yeats and Mathez, 1976; Czamanske and Moore, 1977; Patten et al., 2013), and these investigations indicated the sulfide droplets were commonly micro-sized and displayed zoned and fine-grained textures(Czamanske and Moore, 1977; Patten et al., 2012, 2013; Yang et al., 2014). Furthermore, during mantle melting and differentiation of basalt melt, the sulfide droplet sizes influence the physical behavior of the separate sulfide phases, and the different of sulfide droplets also plays an important role in partitioning the siderophile and cupolophile elements (Bézos et al., 2005; Czamanske and Moore, 1977; Hamlyn et al., 1985; Peach et al., 1990; Rehkämper et al., 1999; Yeats and Mathez, 1976). However, few reports have covered the formation of these sulfide droplets and subsequent scavenge of siderophile and chalcophile elements. A deeper understanding of the initial solidification of sulfide-oxide liquids remains a worthwhile endeavor.

As currently reviewed, most geological processes relate to nanoscale phenomena (Hochella et

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al., 2008; Reich et al., 2011). For example, Hough et al. (2008) reported that nanoparticulate Au develops above Au quartz veins during weathering. Gartman et al. (2014) documented that pyrite nanoparticles comprise a considerable portion of black smoker emissions from hydrothermal vents, and those nanoparticles may be vital sources of iron to the world's ocean (Gartman et al., 2014). Deditius et al. (2018) discovered diverse mineral nanoparticles in magnetite, providing a cautionary note on the interpretation of micron-scale chemical data of magnetite (Deditius et al., 2018). Hawkings et al. (2020) reported that Greenland Ice Sheet meltwaters may provide biolabile particulate Fe that fuels the large summer phytoplankton bloom in the Labrador Sea (Hawkings et al., 2018). This indicated some nano-scale ore-processes play important roles in large-scale systems (Hochella, 2002; Reich et al., 2006; Hochella et al., 2008; Hough et al., 2008).

Here, the material extracted in-situ from the surface of polished MORB glass samples was examined by scanning electronic microscopy (SEM). A focused ion beam (FIB) with transmission electron microscopy (TEM) and high-angle annular dark-field scanning transmission electron microscopy (HAADF STEM) was utilized to (1) investigate micro sulfide droplets in basaltic glass and (2) elucidate the premier stage of sulfide immiscibility in MORB glasses.

2. Geological setting and samples

The MORB samples were dredged along the axis of the SWIR during cruises DY105-17, DY115-19 and DY115-20 by the R/V DaYangYiHao. The Southwest Indian Ridge (SWIR) is an ultraslow-spreading ridge (Dick et al., 2003) extending from the Bouvet Triple Junction (54°50′S, 00°40′W) in the South Atlantic Ocean to the Rodrigues Triple Junction (25°30′S, 70°00′E) in the Indian Ocean. The MORB samples in this study were chosen from the three main ridge segments delimited by the Gallieni (52°20′E) and Melville (60°45′E) fracture zones (Fig. 1). As determined by SEM, the MORB samples were primarily fine-grained and weakly phyric with hypocrystalline to vitreous textures (Fig. 2a). Some phenocrysts were 1-10 μm and relatively rich in Si and O (Figs. 2b-f, 3a-d).

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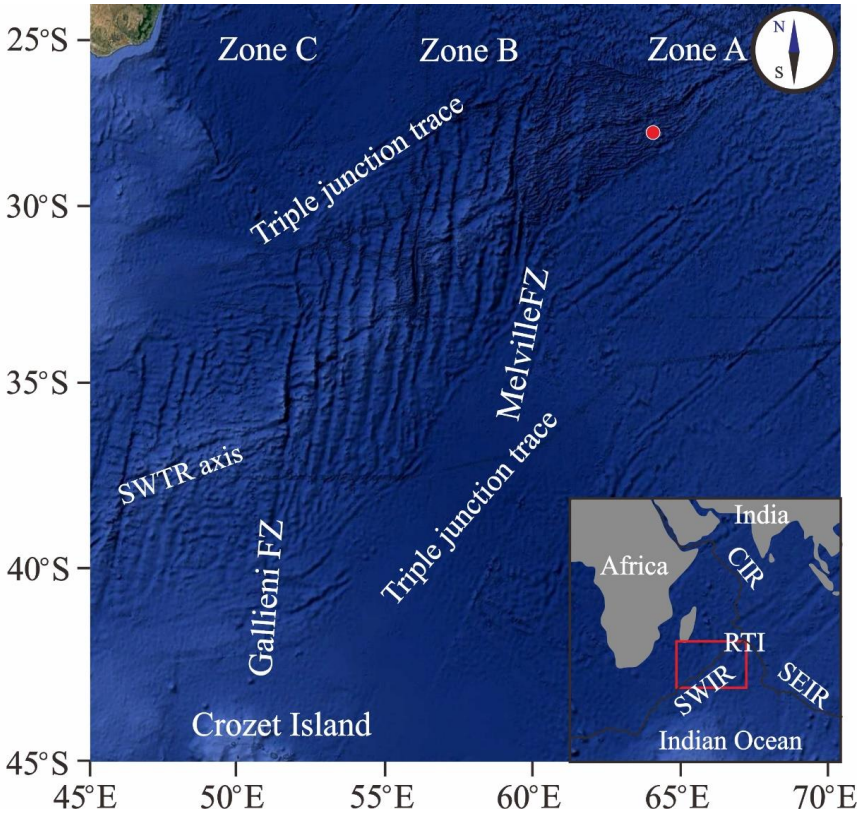

Figure 1. Regional location of the study area

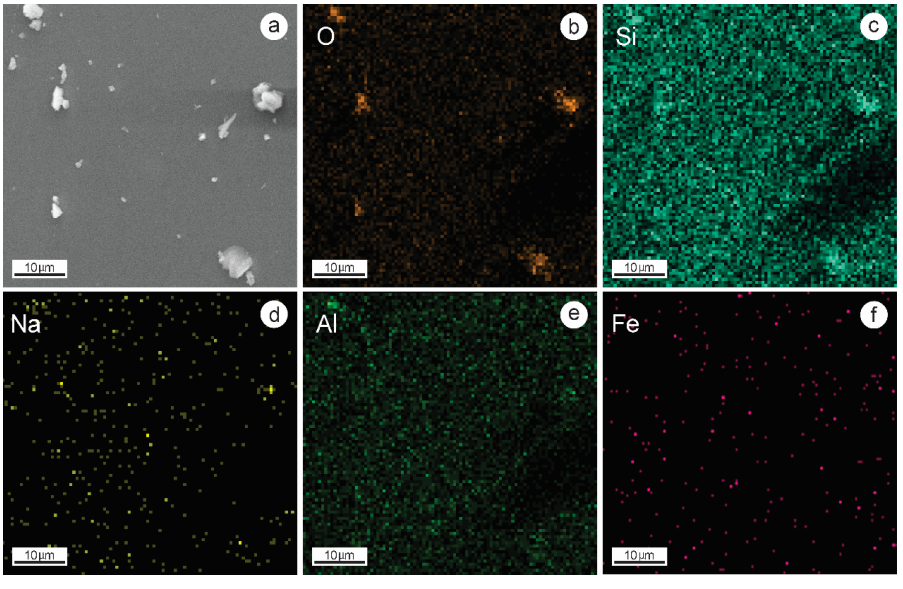

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Figure 2. SEM image (a,) and elemental maps (b-f) of MORB samples

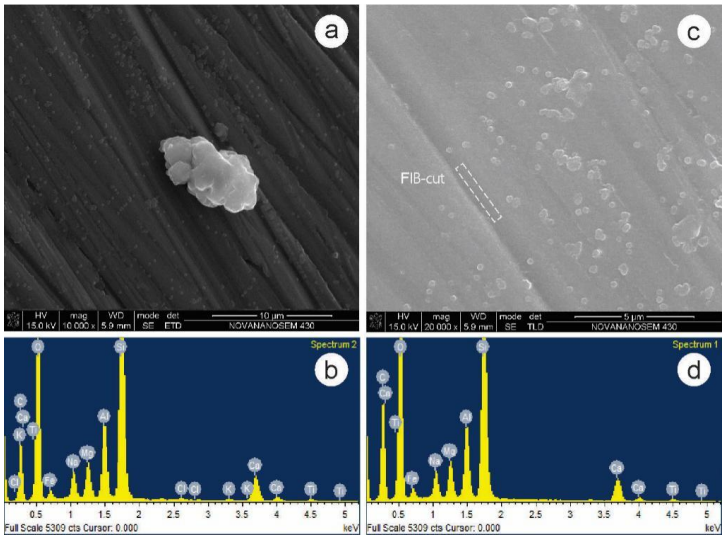

Figure 3. SEM images (a, c) of typical MORB glasses and their corresponding EDS spectra (b, d).

3. Materials and methods

Normal microscopic analyses were conducted on polished thin petrographic sections using a Zeiss-Opton light microscope with transmitted light. Rock platelets for SEM observations were polished and observations conducted using a Hitachi SU8220 at 5 kV and 10 kV. Elemental distribution was determined by energy dispersive X-ray spectrometry (EDS).

A TEM (FEI Tecnai G2 F20) equipped with an EDS detector (0.5–1 wt.% detection limit.) determined the structural/chemical information about the nanoscale sulfide droplets. The maximum TEM acceleration voltage was 200 kV. The TEM foil was extracted to stick on Cu grids via Pt welding, and thinned to thicknesses of 50–70 nm. HAADF-STEM imaging was carried out using an ultra-high resolution and probe-corrected FEI Titan Themis TEM. High-resolution transmission electron microscopy (HRTEM) image processing, including Fast Fourier transform (FFT), was accomplished using Gatan's Digital Micrograph software (version 3.7.4). All analyses, including SEM and TEM, took place at the Sinoma Institute of Materials Research (Guangzhou) Co., Ltd.

4. Results

Fig. 4a shows a FIB-cut of MORB glasses that illustrated several tiny inclusions with relatively

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high contrast. These inclusions differed in size and distributed randomly within the matrix (Fig 4b). Figure 5a also shows a rounded crystalline inclusion (15 nm), based on several diffraction spots recorded in the selected area electron diffraction (SAED) pattern. The lattice fringes were clearly recorded in the HRTEM images except for some lattice defects (Figs. 4c and d). Another crystalline round (~20 nm) inclusion, is shown in Fig. 4d. The well crystalline structure also contained lattice defects as seen in (Figs. 4e and f).

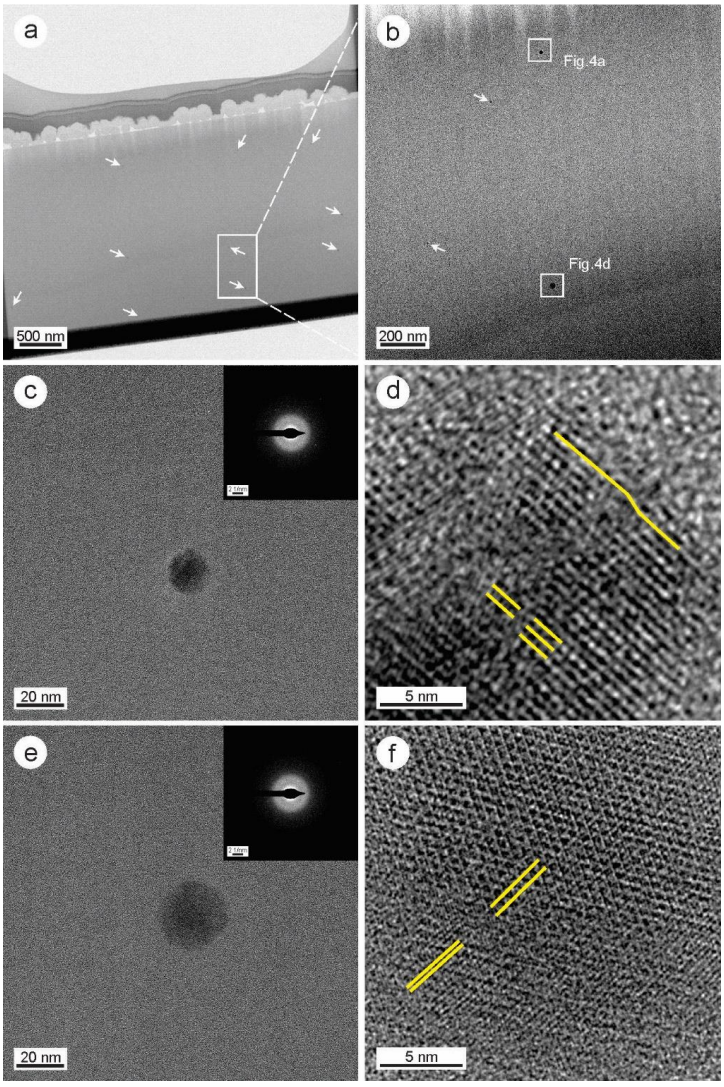

Figure 4. A focused ion beam (FIB)-cut of the MORB glasses (a, b): (a) TEM image. (b) Higher magnification image obtained from the rectangle in a. (c) TEM image showing a nearly

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rounded sulfide droplet with its SAED pattern inserted. (d) HRTEM image the droplet in b. (e) TEM image showing a rounded sulfide droplet with its SAED pattern inserted. (f) HRTEM image of the droplet in e.

Under high-angle annular dark-field scanning transmission electron microscopy (HAADF STEM) imaging, the nanoscale inclusion appeared bright and consisted of metallic elements (Fig. 5). Compositionally, the inclusion primarily included sulfur (Table 1) that indicated these tiny MORB glass inclusions were nanoscale sulfide droplets. According to elemental maps, the sulfide droplet was rich in Fe, Cu, Ni and S (Fig. 6), but lacked Si, O, and Al (Fig. 7).

Table 1. EDS analytical data for the sulfide droplet in this study.

Element	O	Na	Mg	Al	Si	P	S	K	Ca	Ti	Mn	Fe	Ni	Cu
at. (%)	41.16	2.52	2.74	3.56	8.13	0.34	13.06	0.09	2.4	0.36	0.14	11.69	1.51	12.31
wt. (%)	20.68	1.82	2.09	3.02	7.17	0.33	13.15	0.11	3.02	0.54	0.24	20.49	2.78	24.57

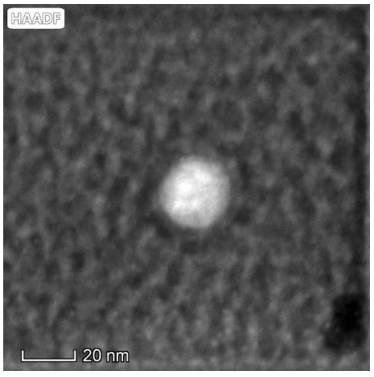

Figure 5. STEM image (a) and elemental maps (b-h) of a typical sulfide droplet within MORB glasses.

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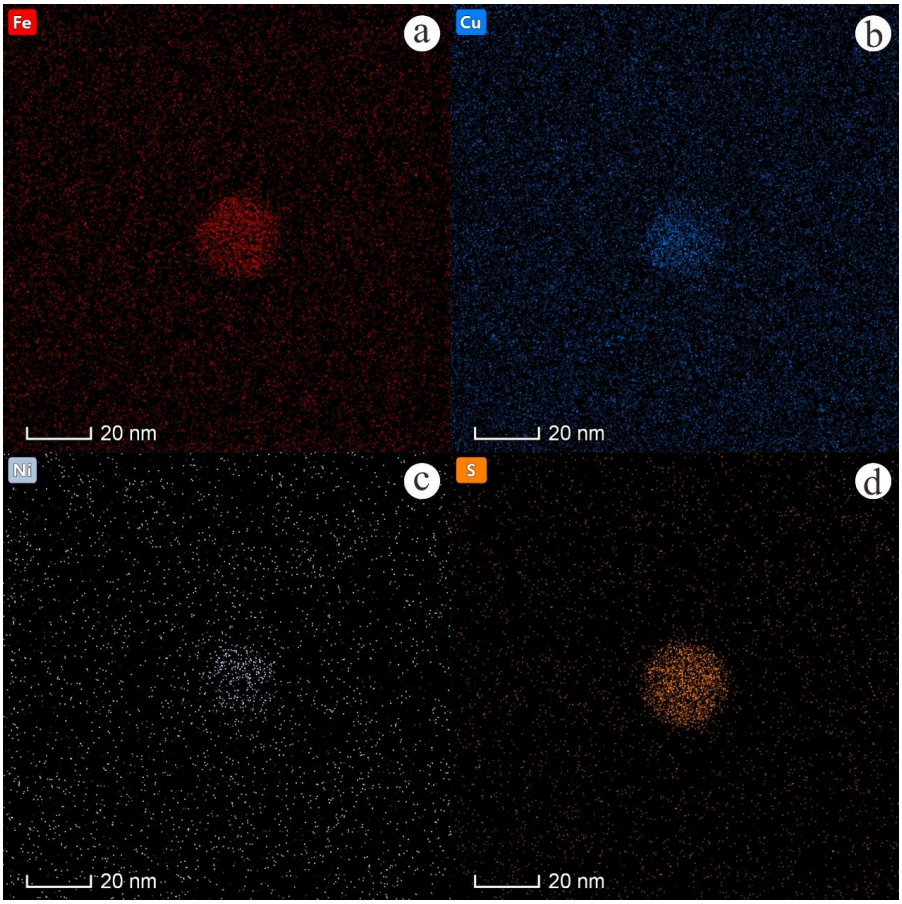

Figure 6. Elemental maps of a typical sulfide droplet within MORB glasses, the sulfide droplets were relatively rich in Fe, Cu, Ni and S.

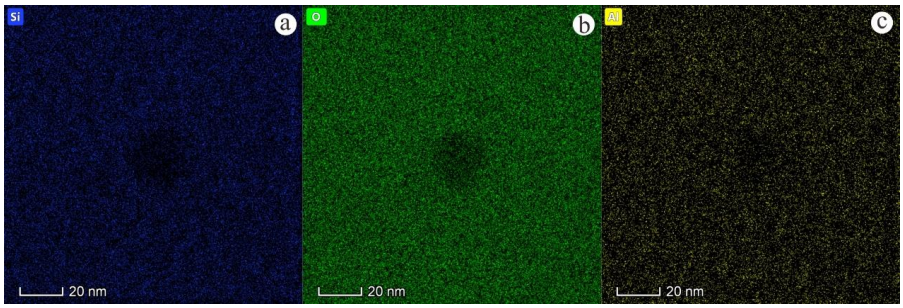

Figure 7. Elemental maps of a typical sulfide droplet within MORB glasses, the sulfide droplets were lack in Fe, Cu, Ni and S

5. Discussion

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5.1 Origin of sulfide droplets

Many studies reported the presence of sulfide globules in olivine crystals and matrix glasses indicated S-saturated fractionation during magmatic evolution (Patten et al., 2012; Yang et al., 2014). On one hand, well-developed sulfide globules in MORBs were thought to take shape prior to the host magma quenching, like those recorded by Mathez and Yeats (1976) and Patten et al. (2012), which were 5–50 μm and round in shape (Yeats and Mathez, 1976; Holzheid, 2010; Patten et al., 2012). Two primary triggers lead to sulfide saturation: incipient crystallization of silicate phases and falling temperature (Mavrogenes and O'Neill, 1999). On the other hand, some embayed sulfides similar to those recorded by Yang et al. (2012) were thought to form from partial dissolution due to decompression. In this study, nanoscale sulfide droplets in natural MORB glass are reported for the first time, thus demonstrating S-saturated fractionation in another way. These recorded droplets (small in size and rounded in shape) were characterized by smooth edges and crystalline features. Consequently, the nanoscale sulfide droplets were more likely to form during the initial phase of sulfide saturation.

5.2 The texture of sulfide droplets

Although sulfide droplets are common in fresh MORB glasses such as phenocrysts and glass, microcrystalline aggregates of plagioclase and olivine, and vesicle walls (Yeats and Mathez, 1976; Czamanske and Moore, 1977; Patten et al., 2012, 2013; Yang et al., 2014), the droplets (i.e., micro-sized) have different textures ranging from a fine-grained intergrowth of MSS and ISS to coarser-grained intergrowth of MSS and ISS with oxide and pentlandite (Yeats and Mathez, 1976; Czamanske and Moore, 1977). The textural differences of these droplets might be controlled by component nucleation and diffusion rates (Czamanske and Moore, 1977). For example, Patten et al. (2013) reported that fine-grained and coarse-grained droplets correspond to the primitive sulfide liquid and the zoned sulfide droplets correspond to the stage where MSS crystallized and co-existed with a residual sulfide liquid (the quenched ISS) (Patten et al., 2013). Compared with those micro-sized sulfide droplets with various textures (Patten et al., 2013; Yang et al., 2012), the nanoscale sulfide droplets recorded in this paper differed significantly in terms of internal textures showing homogeneous compositions without internal fractionation of MSS and ISS phases. Beyond that, the sulfide droplets in this study were crystalline, and their lattices possibly formed during this stage

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(Fig. 4c-f).

On the other hand, the sulfide droplet sizes varied at different stages of sulfur saturation, and the droplet sizes also controlled the textural differences of the droplets. Holzheid (2010) showed the polymerization degree of the silicate liquid controlled the sulfide droplet size distribution. Patten et al. (2012) speculated a positive correlation between the droplet diameter increase and the degree of phase segregation. Beyond that, Peach et al. (1990) reported that the size of a globule should reflect its residence time in the melt and the frequency with which it contacts and consumes other globules. Ostwald's ripening plays an important role in globule size change (Yang et al., 2014). The experimental results show that when the composition of silicate melt becomes immiscible, two-phase nanoscale droplets will soon appear. Small droplets then disappear, which enlarges the larger droplets, and this process is generally accompanied by a reduction in interface free energy (Mazurin and Porai-Koshits, 1984). In this study, the nanoscale sulfide droplets were small (10–15 nm) and have rounded shapes and smooth edges. In summary, sulfides appeared to form very shortly before eruption, followed by immediate supercooling.

5.3 Implications for sulfide droplets scavenge siderophile and chalcophile elements

Sulfide plays an important role in the distribution of sulfurophile elements during mantle melting and basalt melt differentiation (Mathez, 1976; Peach et al., 1990; Bezos et al., 2005). When the sulfide melt droplets segregated, the droplets scavenged siderophile and chalcophile elements such as Ni, Cu, Pt, and Pd from the magma (Holzheid, 2010). Most micro-sized sulfide grains in MORBs contained MSS , the first mineral to crystallize from a sulfide liquid, and ISS, which crystallizes from the remaining liquid (Patten et al., 2013). As such the distributions of different chalcophile elements regarding partition preference commonly behave differently in MSS and ISS. For instance, platinum-group elements (PGE) are scavenged by immiscible sulfides during the evolution of mafic magmas (Keays, 1995; Peach et al., 1990; Rehkämper et al., 1999; Song et al., 2006), but the iridium-subgroup of PGE (Os, Ir, Ru, and Rh) normally behave as compatible elements and partition into MSS (Patten et al., 2013; Yang et al., 2014). Co and Re have a slight preference for MSS, whereas Cu, Zn, Au, Ag, Sn, Te, Cd, Bi, and Pb partition into ISS (Patten et al., 2013). The sulfide droplets reported in Yang et al. (2014) contained Ni-Fe-rich MSS and Cu-Fe-rich ISS phases.

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However, the existing literature does not specify exactly when the sulfurphilic elements enter the sulfide droplets. Different from micro-sized droplets, the nanoscale sulfide droplets in this study compositionally contained Fe, Cu, Ni and S (Fig. 6). These elements might have been scavenged during initial sulfide saturation and behaved similarly in the droplets. With the formation of sulphide beads, the elements Si, O and Al are depleted at the same time. Based on the nanoscale sulfide droplets reported in this paper, chalcophile elements appeared to enter the sulfide droplets and distribute evenly within the sulfide globule in the early stage. As sulfide droplets in MORB provide unique records of near-liquidus phase relations in basaltic systems, such insight help understand how chalcophile elements behaved during sulfide crystallization. With longer residence times, sulfide droplets gradually evolved and featured different textures including MSS and ISS, in which different chalcophile elements had their own preferences.

5.4 Implications of sulfide droplets on magmatic evolution and formation of sulfide deposits

Immiscibility is an important factor in the process of magmatic evolution and mineralization. During magma immiscibility, a single component melt can decompose into two kinds of melts with completely different compositions. After magma immiscibility, the large-scale separation of conjugated two-phase melts affects the overall evolution of magma. The earlier immiscibility begins in the process of magmatic evolution, the better its influence on magmatic evolution, and the better its geochemical and petrological significance (Veksler et al., 2008). Therefore, understanding the nucleation, growth and separation of immiscible droplets is very important to understand the role of immiscibility in magmatic evolution. As one of the immiscible phases, the immiscibility of sulfide droplets and silicate melt plays an important role in magmatic evolution. The immiscibility between sulfide and silicate melts has been clearly elucidated in previous studies. The basic principle of magmatic sulfide deposits origins stems from the sulfur saturation of magma (Mungall and Naldrett, 2008). Sulfide droplets form when sulfur saturation occurs. Magmatic sulfide deposits form when sulfide droplets become sufficiently concentrated enough in a certain area of the magmatic body (Mungall and Naldrett, 2008). The study shows that the formation of magmatic Ni-Cu sulfide deposits is related to the separation and enrichment of sulfur saturated and immiscible sulfide liquids in mantle-derived basic and ultrabasic magmas (Arndt et al., 2005).

According to the elemental maps, the sulfide droplets were relatively rich in Fe, Cu, Ni and S

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(Fig. 6), but lacking in Si, O, and Al (Fig. 7); this indicated the immiscibility of sulfide and silicate melts occurred during the early stage. According to the results, the immiscibility of sulfide and silicate melt occurs in the nanoscale stage, so it can be inferred that the immiscibility in the process of magma evolution may occur in the nanoscale stage.

6. Conclusions

Nanoscale sulfide droplets were first identified in MORB glasses by FIB-cut and TEM analyses. These droplets might form rapidly before eruption and then undergo immediate supercooling. As the initial phase of sulfide saturation, nanoscale sulfide droplets simultaneously scavenged Fe, Cu, Ni, and Na in the early stage rather than selectively concentrating siderophile and chalcophile elements in different parts of the droplet. Furthermore, this new understanding of sulfide and silicate melt immiscibility, which occurs during the early nanometer stage provides a new idea for further study of the immiscibility stage during magma evolution.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

This research was supported by the National Natural Science Foundation of China (Grant Nos. 42102076), project ZR2021QD037 supported by Shandong Provincial Natural Science Foundation, and the Guangdong Basic and Applied Basic Research Foundation (2020A1515010906)

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