

- 2 prior to eruption [[indi**qated?]**] by phosphorus distribution
- 3 in olivine from mid-ocean ridges
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14 **ABSTRACT**

15 Melt inclusions (MIs) hosted in euhedral olivine have been proposed to represent 16 droplets of primary melt, protected from processes occurring near Earth's surface during 17 eruption. The complex zoning of phosphorus (P) in some olivines and the presence of a 18 P-depleted zone around MIs indicate a complex history for the host-MI system. We 19 analyzed P in olivine and MIs from two mid-oceanic ridge basalt (MORB) samples from 20 the Mid-Atlantic Ridge (MAR) by electron probe microanalyzer, secondary ion mass 21 spectrometry (SIMS), and NanoSIMS. Phosphorus dendrites in olivine suggest an initial 22 fast olivine growth followed by a stage of slower growth. Dissolution textures around

29 **INTRODUCTION**

30 Olivine-hosted melt inclusions (MIs) are widely used to constrain primary melt 31 compositions because they are isolated from the magma and assumed to preserve the 32 composition of the melt from which their host grew (e.g., Kent, 2008; Métrich and 33 Wallace, 2008; Schiano, 2003). Despite agreement that olivine-hosted MIs are entrapped 34 at relatively shallow levels (<10 km), the origin of melt entrapped in MIs is debated. 35 Some studies indicate that MIs represent the melt composition of the last reequilibration 36 in the mantle (e.g., Hart, 1993; Sobolev, 1996), while others argue that MIs record 37 interaction with the magmatic plumbing system (e.g., Kent et al., 2002; Saal et al., 2005; 38 Kamenetsky and Gurenko, 2007), or localized, grain-scale dissolution-reaction-mixing 39 processes (Danyushevsky et al., 2004). Finally, they can lose their initial water content 40 and change oxygen fugacity (f_{O2}) (Gaetani and Watson, 2000), calling into question the 41 interpretation that the large compositional variations observed in MIs directly represent 42 the heterogeneity of the mantle source.

43 The idea that euhedral olivine grows in concentric layers from core to rim has 44 been challenged due to the discovery of irregular, rhythmic zonation in incompatible, 45 slow-diffusing elements such as phosphorus (P) (e.g., Milman-Barris et al., 2008; Welsch

72 MIs in order to better understand entrapment mechanisms. NanoSIMS profiles of P in

73 olivine allow the residence time of olivine in the system after MI entrapment and before

74 eruption to be estimated. We show that decompression can cause olivine dissolution and

75 MI formation upon reprecipitation on very short time scales.

76 **SAMPLES AND ANALYTICAL METHODS**

77 The selected olivines contain 50–350 µm glassy MIs and are from two different

78 locations on the Mid-Atlantic Ridge (MAR). The first sample is a dredged olivine-rich

79 picrite (sample ARP73-10-03, 36°8372′N, 33°2482′W) from the FAMOUS zone

80 (Bougault and Hekinian, 1974). This sample contains >20% olivine phenocrysts, rare

81 clinopyroxene, and plagioclase xenocrysts (Laubier et al., 2007). The geological context,

82 geochemistry of lavas, and MIs were described in several studies (e.g., Langmuir et al.,

83 1977; Laubier et al., 2007; Gale et al., 2013). The second sample (CH77-DR6-203) is a

84 dredged fresh basalt containing a high volatile proportion from the 14° MAR triple

85 junction (14.12°N, 45°W) (Bougault et al., 1988; Javoy and Pineau, 1991).

86 Major element composition and X-ray element maps of olivine and MIs (Fig. 1;

Fig. DR2 in the GSA Data Repository¹) were acquired by electron probe microanalyzer

88 (EPMA; University of Lausanne, Switzerland). The P content of MIs and olivine was

89 determined by measuring ${}^{31}P$ and ${}^{28}Si$ in monocollection using the CAMECA IMS 1280-

90 HR at the SwissSIMS laboratory (University of Lausanne). High-resolution ${}^{31}P$, ${}^{28}Si$, Al

91 $(^{27}Al^{16}O)$, and Mg ($^{24}Mg^{16}O$) profiles were acquired with a CAMECA NanoSIMS 50L

160 trapped during a period of fast growth, they would be enriched in elements incompatible

- 207 before eruption. MI composition can be affected by olivine dissolution and
- 208 recrystallization processes, at least for P, and cannot be simply taken as representative of
- 209 the mantle source. Care should thus be taken when interpreting MI P and other
- 210 incompatible element compositional variability.

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- 348 Petrology, v. 161, p. 153–173, https://doi.org/10.1007/s00410-010-0518-y.
- 349 **FIGURE CAPTIONS**
- 350 Figure 1. Electron probe microanalyzer (EPMA) phosphorus (P) elemental X-ray map of
- 351 olivine ARP73-10-03_16. P concentrations were determined quantitatively by secondary
- 352 ion mass spectrometery (SIMS). Note higher P concentration in melt inclusions (MIs)
- 353 situated near P-zoned olivine compared to ones located in P-unzoned olivine. Pink arrows
- 354 represent nanoscale SIMS (NanoSIMS) profiles; arrow indicates direction of profile, and
- 355 number is profile number. NanoSIMS profiles used \sim 150-nm-diameter beam. ${}^{31}P/{}^{28}Si$
- 356 profiles 4, 7, and 9 are located at boundary between MI and olivine (profiles 4 and 7) and
- 357 in olivine (profile 9). Interface between MI and olivine was determined based on Al and
- 358 Mg NanoSIMS profiles (Fig. DR3 [see footnote 1]). Peak in rectangle in profile 9 is peak
- 359 fitted in Figure 2. Peak situated at 47 μ m in profile 9 is originally composed of three
- 360 lamellae (indicated by gray rectangles). Because their diffusion profiles overlap, they
- 361 were not fitted.

362

384 about fitting parameters, is available online at

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386 editing@geosociety.org.

FIGURE CAPTIONS

- 7
8 8 Fig.3
- 9

GSA Data Repository 2017359

Manzini et al., 2017, Weekly to monthly time scale of melt inclusion entrapment prior to eruption recorded by phosphorus distribution in olivine from mid-ocean ridges: Geology, doi:10.1130/G39463.1.

Supplementary data

Methodology

Sample preparation

 Polished olivine grains with melt inclusions exposed at the surface were pressed into 1-inch indium mounts. Different glasses and San Carlos olivine were added to the sample mount to be used as reference material for P content determination by SIMS (see below).

To ensure that the melt inclusions were totally glassy, we acquired SEM images with a Tescan

Mira LMU scanning electron microscope at the University of Lausanne.

Electron microprobe

12 All samples were analyzed by a JEOL 8200 Superprobe at the University of Lausanne. Analytical conditions for major element measurements in olivine were 15kV acceleration voltage, 20nA beam current and 5 μm beam size. Counting time was 30 sec for Si, Mg, Mn, 15 Al and Fe and 40 sec for Ca, Ni and Cr. Natural minerals and synthetic oxides were used as standards. Analytical conditions for major element analyses in melt inclusions were 15kV, 10nA and 10 μm. Counting time was 30 sec on all elements, except for K (20 sec) and Na (16 18 sec). A glass standard (KL2-G) was used for SiO_2 , Al_2O_3 . Other elements were calibrated on specific minerals (e.g., a fayalite for Fe). ML3B-G was used as internal standard to check the calibration. Post-entrapment modification due to olivine crystallization (PEC% (% olivine 21 added), Table DR1) at the wall of melt inclusion was corrected for using the Petrolog software 22 (Danyushevsky et al., 2000, 2002; Danyushevsky and Plechov, 2011).

 X-rays distribution maps of melt inclusions and olivines were acquired by electron microprobe using a JEOL 8200 Superprobe at University of Lausanne. Analytical conditions 25 were 15 kV, 10 nA, 0-1 μm probe diameter and 100 ms dwell-time with a 1-2 μm pixel size for major elements and 15kV, 200-250nA, 0-1 μm probe diameter and 100-230 ms dwell-time with a 1 μm pixel size for P, Al, Ni and Ca. X-ray elemental maps display higher count rate 28 for P in MI compared to P-zoned olivine, as a result of the overlapping of the Ca $(K\beta)$ second 29 order peak with the P ($K\alpha$) peak. As olivine contains much less Ca than glass, the interference is negligible in olivine but not in MI. In any case, as olivine is homogeneous in Ca (Figure DR2), the structure on the phosphorus maps and profiles are only caused by variable P concentrations.

Secondary ion mass spectrometer

 P concentrations in olivines and melt inclusions were analysed using a Cameca 1280-HR SIMS at the SwissSIMS laboratory (Lausanne, Switzerland). San Carlos (P = 40 ppm; Welsch et al., 2014) was used as standard for the olivine and BHVO-2G, BCR-2G, BIR-1G (P concentrations are from the certified values furnished by USGS) for the melt inclusions. 40 Samples were sputtered with a 10 kV $^{133}Cs^+$ primary beam of 1 nA. Electron gun was used to compensate sample charge. A 15μm raster was used during pre-sputtering (90 sec) and 42 analyses. Counting time was 0.48 on 29.9 (background), 1.04 s on ^{30}Si and 2.96 sec on ^{31}P . 43 Waiting time was 0.8 s between each mass. Total time for each analysis was ≤ 6 min, 44 including automatic centering of the secondary beam.³⁰Si and $31P$ were detected on an axial 45 electron multiplier. Entrance slit was set to 60 μ m and exit slit to 200 μ m, resulting in ~6000 mass resolving power. Field aperture was closed to 4000 μm. Energy slit was 50 eV.

47 Uncertainty on each point measurement (internal error) was $\leq 5\%$ (2 σ %, average uncertainty 48 of 0.95% 2σ) for P in olivine and <0.8% (2σ %, average uncertainty of 0.2% 2σ) in melt 49 inclusion. The difference in reproducibility is due to counting statistic, melt inclusions having 50 100 times more counts on P. Reproducibility on the standard was $\leq 4.5\%$ (2SD) on the San 51 Carlos and \leq 1% (2SD) on BCR-2G.

52

53 **Nanoscale secondary ion mass spectrometer**

 Profiles in olivines were obtained using the NanoSIMS at the Center of Advanced Surface 55 Analysis (CASA, Lausanne, Switzerland). A $Cs⁺$ primary beam was used to sputter olivine 56 and the ion species ¹⁶O₂, ²⁸Si₂, ³¹P₂, ²⁴Mg¹⁶O₂, ²⁷Al¹⁶O₂ and ³¹P¹⁶O₂ were extracted and measured using electron multipliers. In order to prevent the build-up of a charge on the sample's surface, the surfaces were coated with a 10-15nm gold layer. MRP was > 8000 and no peak overlap was observed.

60 After identification of the area of interest using the CCD camera of the NanoSIMS, a 25 x 25 61 μm image of the area of interest was first acquired (with a 256 x 256 pixel resolution 62 corresponding to a pixel size \sim 100 nm). Using the NanoSIMS software, the locations of the 63 linescan were precisely selected to be perpendicular to the transition identified by EMPA 64 analyses. The line scans were acquired using a two-step approach. First a pre-sputtering phase 65 was performed for 2 cycles with a dwell time of 2sec/pixel, using slightly larger beam size to 66 clean up the surface (diaphragm D1-2, and 3.8pA for the $Cs⁺$ primary beam). The acquisition 67 of the high resolution line scans were performed using a finer beam (D1-3, \sim 150nm, and 68 1.7pA for the Cs+ beam) over 5 cycles with 2sec/pixel. ²⁴Mg¹⁶O⁻ data were used to monitor 69 possible local variation in the ionization and extraction processes as it is expected to be 70 homogeneous in the olivine measured. ${}^{16}O$, ${}^{18}O$, ${}^{24}Mg$, ${}^{27}Al$, ${}^{28}Si$, ${}^{31}P$ and ${}^{31}P^{16}O$ were 71 acquired at the same time. In order to minimize the instrumental drift, we normalized the 72 profiles to 28Si , which is, based on EMPA X-ray maps, homogeneous within the olivine and 73 the MI. The NanoSIMS session was not standardized, resulting in a $31P$ count rate higher in 74 the MI than in P-zoned olivine, due to matrix effects.

75

76 **Diffusion Chronometry and Fit of Diffusion Profiles**

 The 2D profiles were measured perpendicular to the zonation. The following parameters were assumed for the diffusion calculations and fitting of the profiles: 1- each P-enriched band was homogeneous at the beginning or represents a line source; 2- the contact between the P-rich and P-poor zones was a step function. Thin, linear features with no plateau were modelled as a 81 line source, while P-enriched zones displaying a plateau were modeled as step functions. Specifically, a P-peak of a few μm width (e.g. Fig. 2) was modelled as line source, while b) P-

83 enriched zones larger than 10 μm were modelled as individual step functions (e.g. Fig. DR5).

84 We used the diffusion coefficient for P diffusion in olivine reported by Watson et al. (2015)

85 based on their diffusion experiments in olivine which are $log(D_0, m^2/s) = -10.06 \pm 0.80$ and E_a

 $86 = 229 \pm 16$ kJ/mol. Error functions were fit to each of the measured profiles using a weighted 87 Marquardt fitting routine (Press et al., 1992).

88 The uncertainty of the fit was estimated using the χ 2 per point. The uncertainty, expressed as 89 2σ , for each point was estimated from the variation in the part of the profiles considered to be 90 flat. Initial conditions of the model are the bigger source of uncertainty. Since the line source 91 diffusion modeling assumes an infinite band width of P-enrichment, the calculated values are 92 maximum time estimates.

93

94 **MELTS calculations**

95 MELTs version 5.0 was used. The principles of the program are summarised in Ghiorso and 96 Sack (1995). We used the average composition of the individual melt inclusions and 0.15 wt% 97 H₂O. We then determined the liquidus temperature, followed by decompression calculations 98 slightly below the liquidus temperature. Isentropic, isenthalpic and isothermal decompression 99 calculations were done between 2 kbar and 0.1 kbar in steps of 0.1 kbar, with a fixed $fO₂$

- 100 (QFM -2).
- 101
- 102

103 **Figures**

106 Figure DR1: Plot of ${}^{31}P/{}^{28}Si$ ratios obtained during NanoSIMS measurements versus (a) ${}^{48}Si$ 107 counts and (b) $\frac{31}{P}$ counts. The absence of correlation in (a) and the good correlation in (b) 108 indicates that ${}^{31}P/{}^{28}Si$ ratio depends on ${}^{31}P$ concentration only.

110 Figure DR2: X-ray maps for A- Mg concentration in ARP73-10-03 16; B- Al concentration 111 in ARP73-10-03 16; C- Ca concentration in ARP73-10-03 16; D- P concentration in CH77-112 DR6-203 52; E- P concentration in CH77-DR6-203 4; F- P concentration in CH77-DR6-113 203 8. Note the homogeneity of the olivine in major elements (A, B and C), whereas nice P 114 zonations are present in the same olivine (Figure 1). MI are homogeneous in P, except in Fig. 115 DR2-E. Apparent heterogeneity of this specific MI is explained by the presence of a previous 116 SIMS analysis spot, creating a small topography, able to slightly modify the P signal during P 117 imaging by EMPA.

120 Figure DR3:

 $31P^{28}$ Si profiles realized in the ARP73-10-03_16 olivine and melt inclusions for all the 122 measured elements. The contact between MI and olivine can be determined based on the Mg 123 and Al profiles. The location of the profiles is presented in Figure 1.

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128 Figure DR4: Results of the MELTS decompression calculations at fixed $fO₂$ (QFM) between 129 2 to 0.1 kbar for isothermal, isenthalpic and isentropic decompression. Ol is for olivine. Isentropic and isenthalpic decompression causes weak to moderate initial resorption, while isothermal decompression only causes crystallization.

135 Figure DR5: Comparison of the measured ${}^{31}P/{}^{28}Si$ profile in olivine with the calculated curve 136 derived from semi-infinite source diffusion modeling for profile 9, at the contact between P-137 rich olivine and P-depleted halo. Diffusion was 0.8 days at 1270°C, using D from Watson et 138 al. (2015). This estimated time scale is a maximum, since the profile is near vertical, and its 139 width is constraint by one point which could represent an analytical artefact. In fact, the 140 profile width could be significantly overestimated.

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- Figure DR6: P distribution in ARP73-10-03_16 olivine.
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- **Tables**
- Table DR1
- 147 NanoSIMS measurements of the profile 9 realized in the ARP73-10-13_16 olivine.

- 149 Table DR2
- 150 Major element compositions (determined by EPMA) and P concentration (determined by
- 151 SIMS) of the melt inclusions hosted in the ARP73-10-13_16 olivine. Values measured for
- 152 ML3B glass, used as external standard for EPMA glass analyses, are also reported. Major
- 153 element concentrations are given in wt% and P concentrations in ppm. For ML3B FeO
- 154 represents FeO_{tot} .

Table DR2

- Table DR3
- Results of the MELTs calculations for the three different models tested.

Table DR3

167 **References**

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