

# Methodology for the study of melt inclusions in Cr-spinel, and implications for parental melts of MORB from FAMOUS area

Vadim Kamenetsky<sup>a,b,\*</sup>

<sup>a</sup> Department of Geology, University of Tasmania, GPO Box 252C, Hobart, Tasmania 7001, Australia

<sup>b</sup> Petrochemistry and Experimental Petrology Group, Research School of Earth Sciences, The Australian National University, Canberra, ACT 0200, Australia

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## Abstract

Melt inclusions trapped in Cr-spinel in mid-ocean ridge picrites (FAMOUS area, Atlantic Ocean) were studied using a heating stage and analyzed with both electron and ion microprobes. This new technique can provide information on the most primitive magma compositions, olivine–spinel–melt compositional relationships, crystallization temperature and geochemical diversity of parental melts. The study of melt inclusions in spinel can be applied to different high-Mg suites to show the effects of shallow level fractionation, mixing and contamination that usually affect the composition of volcanic rocks and glasses.

*Keywords:* FAMOUS; mid-ocean ridge basalts; inclusions; chrome spinel; experimental studies; olivine; trace elements; melts

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## 1. Introduction

Mid-ocean ridge basalts (MORB) are the most voluminous volcanic rocks on Earth, and have been the subject of numerous detailed petrogenetic studies over the last 30 years. Significant controversy still surrounds models for the MORB-source mantle composition, the nature of parental (primary) MORB magmas, and the conditions and mechanisms of partial melting and MORB magma generation (including pre-eruption fractionation and mixing processes). A key pre-requisite to improved understanding of these processes is positive identification of the range of MORB primary magma compositions. As it is

unlikely that MORB primary magmas are erupted, due to mingling of melts during magma extraction from the upper mantle peridotite, and subsequent subvolcanic magma chamber processes such as mixing and assimilation, the composition of the MORB parental and primary magmas must be inferred or modelled.

Several approaches to the determination of parental MORB magmas have been tried. Experimental peridotite melting studies under various  $P$ – $T$  conditions have made a coherent picture of the range of primary magma compositions that can be produced from fertile to depleted peridotite for partial melting extents  $> \sim 5\%$  (e.g. [1–8]). Numerical modeling, based on these experimentally produced isobaric batch melts, has attempted to derive the range of MORB by pooling of small melt fractions

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\* Address for correspondence: b. Fax: 61 6 2495989. E-mail: dima.kamenetsky@geol.utas.edu.au

in a melting column [9–14]. Another technique to reconstruct parental magmas uses ‘reverse fractional crystallization’ of natural basaltic compositions [9,12,15]. A more direct approach involves the study of melt inclusions trapped in the most primitive phenocrysts in MORB (olivine and plagioclase), using a heating stage, to provide direct information on the composition and trapping temperature of parental melts [15–19].

The objective of this paper is to demonstrate that compositions of melts trapped in MORB spinel crystals can provide important information on MORB petrogenesis and the nature of parental and primary MORB magmas. The advantages of this new method are:

1. Cr-spinel, as a phase cotectic with primitive olivine ( $\text{Fo}_{86-92}$ ), is widespread in MOR picrites and basalts;
2. Cr-spinel often contains primary melt inclusions (e.g. [20–22]);
3. importantly, unlike melt inclusions in silicates (olivine, pyroxene or plagioclase), compositions of melt inclusions in Cr-spinel do not suffer significant post-entrapment modification caused by crystallization on the walls of the host because of the very low Cr content of the trapped melt.

## 2. Samples and method of study

The samples (ARP-73-10-03, ARP-73-10-03a, CH31 DR08-136-02 and CH31 DR08-695; hereafter called as samples *A*, *B*, *C*, *D*, respectively) were obtained by submersible and dredging in the FAMOUS area of Mid-Atlantic Ridge at 36°50'N. They are olivine–porphyritic picrites or picritic basalts with quenched glasses on natural surfaces. Abundant Cr-spinel, as discrete crystals in glass and ground-mass, and as inclusions in olivine, forms euhedral, rarely skeletal crystals up to 0.8 mm in size. These contain numerous melt inclusions, best observed with reflected light on polished surfaces, or with transmitted light if the thickness of specimen is < 50–70  $\mu\text{m}$ . Usually, melt inclusions are randomly distributed in their host grains, but sometimes they occur in more regular zones, outlining relic spinel–melt interfaces. They always have a negative crystal shape and vary in size from 2 to 60  $\mu\text{m}$ . In most

spinel crystals, the melt inclusions are polyphase, and consist of residual glass and daughter pyroxene; however, in sample *B* they are glassy.

Spinel was hand picked after dissolution of crushed rock in concentrated HF. About 200 spinel grains were heated using a heating stage designed in the Vernadsky Institute of Geochemistry, Moscow [23]. For sample *D*, spinels were heated in four separate experiments, each of 10 min duration, to 1240, 1270, 1300, 1330°C, and then quenched (the rate of quenching was  $\sim 500^\circ/\text{sec}$ ). Spinel grains from other samples were heated, and quenched at 1320°C. Finally, spinel grains were mounted in epoxy, and the face of the grain mount was then ground on diamond paste until melt inclusions were exposed.

Heated and unheated (in sample *B* only) melt inclusions in spinel, and spinel inclusions in olivine, were analyzed together with their host minerals using a Cameca SX50 electron microprobe (University of Tasmania, University of Paris VI) under routine operating conditions. Each composition is the average of at least two points. Trace element analyses of melt inclusions were made with a Cameca IMS-4f ion probe (IM RAS, Yaroslavl, Russia) [24].

## 3. Results and discussion

The average compositions of melt inclusions in spinel from sample *D*, homogenized and quenched at 1240, 1270, 1300, 1330°C, are basically identical and do not correlate with the temperature of quenching (Table 1, numbers 4–7). The absence of daughter minerals at the minimum experimental temperature (1240°C) suggests either that the temperature of crystallization of these particular spinels was < 1240°C, or that metastable crystallization of daughter phases in the closed system of an inclusion proceeds at lower (< 1240°C) temperature. Apparently, even significant overheating does not modify the composition of melts trapped in spinel, due presumably to the low solubility of Cr-spinel in basaltic melt. This can be used in melt inclusion homogenization experiments if the exact temperature of crystallization and trapping is unknown. It is essential that the heating of melt inclusions in non-transparent MORB spinel up to 1300–1330°C guarantees remelting of all, if

Table 1  
Compositions of natural quenched pillow rim glasses, melt inclusions and their host spinels

Sample:	B			D			A			C									
	1	2	3	4	5	6	7	8	9	10	11	12							
	(13)	$\sigma$	(9)	$\sigma$	(5)	$\sigma$	(2)	(11)	$\sigma$										
<b>Melt inclusions and natural glasses</b>																			
SiO <sub>2</sub>	49.83	0.42	49.86	0.35	49.1	50.00	0.67	48.92	1.01	49.86	49.60	0.75	49.77	48.36	48.15	50.11	50.57	50.57	
TiO <sub>2</sub>	0.57	0.03	0.60	0.04	0.67	0.67	0.08	0.56	0.06	0.48	0.56	0.07	0.81	0.70	0.68	0.44	0.53	0.53	
Al <sub>2</sub> O <sub>3</sub>	15.73	0.27	15.48	0.13	16.52	15.48	0.43	15.61	0.52	15.16	15.23	0.39	16.58	14.75	14.32	15.53	15.82	15.82	
FeO *	7.44	0.14	7.63	0.22	7.91	7.97	0.28	8.37	0.32	8.69	8.26	0.22	8.56	7.81	7.82	7.57	7.41	7.41	
MnO	0.10	0.06	0.16	0.06	0.08	0.16	0.04	0.15	0.02	0.12	0.14	0.04	0.15	0.18	0.17	0.17	0.20	0.20	
MgO	9.41	0.28	10.29	0.17	9.52	11.05	0.57	11.14	0.53	11.70	11.42	0.30	9.11	12.51	13.64	10.83	10.52	10.52	
CaO	13.21	0.17	13.01	0.10	13.33	13.13	0.82	12.93	0.62	13.00	12.96	0.40	13.12	12.22	11.86	13.49	12.90	12.90	
Na <sub>2</sub> O	1.65	0.04	1.66	0.04	1.67	1.66	0.27	1.51	0.17	1.56	1.48	0.14	1.83	1.45	1.41	1.35	1.40	1.40	
K <sub>2</sub> O	0.09	0.05	0.12	0.06	0.07	0.06	0.03	0.04	0.02	0.08	0.10	0.13	0.10	0.00	0.00	0.07	0.50	0.50	
P <sub>2</sub> O <sub>5</sub>	0.08	0.04	0.06	0.03	0.03	0.08	0.03	0.05	0.02	0.06	0.07	0.05	0.09	0.03	0.03	0.11	0.18	0.18	
Total	98.11		98.88		98.9	100.27		99.28		100.71	99.81		100.13	98.01		99.67	100.03	100.03	
Tq (°C)	<b>1320</b>				<b>1240</b>	<b>1270</b>		<b>1300</b>	<b>1330</b>										
<b>Host spinel</b>																			
	(12)	$\sigma$	(8)	$\sigma$	(3)	$\sigma$	(4)	$\sigma$	(1)	(4)	$\sigma$								
TiO <sub>2</sub>	0.25	0.04	0.27	0.02	0.27	0.06	0.27	0.03	0.26	0.24	0.04		0.34			0.26			
Mg#	72.59	0.58	73.05	0.45	74.19	1.24	73.72	0.64	73.05	74.83	0.63		76.37			74.05			
Cr#	47.55	0.80	48.58	0.83	50.03	0.80	49.87	1.54	49.02	49.82	1.45		51.99			52.69			
Fe <sup>2+</sup> /Fe <sup>3+</sup>	2.85	0.22	2.73	0.16	3.06	0.24	3.08	0.34	2.66	2.89	0.35		2.58			2.74			

A, B, C and D = abbreviations of samples given in the text; 1, 2, 4–7 = average compositions; 1, 4–7, 9, 11 and 12 = heated and quenched inclusions; 2 = natural glass inclusions; 3 and 8 = natural quenched pillow rim glasses; 10 = the calculated [25] most primitive melt in equilibrium with the most magnesian olivine Fo<sub>92.1</sub> at T = 1310°C (composition no. 9 + 3 wt% of olivine); 11 and 12 = melt inclusions in the same spinel grain; figures in parentheses = number of specimens;  $\sigma$  = standard deviation; Mg# = 100 × Mg/(Mg + Fe<sup>2+</sup>); Cr# = 100 × Cr/(Cr + Al) (in mol.%); Tq = temperature of quenching. \* All iron as FeO.

any, silicate daughter phases, and will not change to the composition of the original trapped melt.

The average composition of natural glass melt inclusions and heated/quenched melt inclusions in spinel from sample *B* are similar to each other and resemble the composition of natural quenched pillow rim glass for this sample (Table 1, nos. 1–3). This implies that the majority of phenocrysts were transported to the surface by their parental magma and quenched at the temperature of their crystallization and trapping of melt inclusions. The glassy appearance of melt inclusions confirms this.

Compositions of homogenized melt inclusions in spinel from sample *D* (Table 1, nos. 4–7) are more primitive in comparison with the natural glass of this sample (Table 1, no. 8), being enriched in MgO and depleted in TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub> and Na<sub>2</sub>O. The temperature of melt–olivine equilibrium [25] calculated for this pillow rim glass is 1210°C. The calculated composition of equilibrium olivine Fo<sub>87.4</sub> (at  $K_d^{Fe^{2+}-Mg} = 0.30$  and  $Fe^{3+}/Fe_{melt}^{2+} = 0.09$  estimated on basis of the empirical equation [26]) is very close to the composition of the most evolved (Fo<sub>88.3</sub>) olivine phenocrysts found in this sample. However, melt inclusions are equilibrated with more primitive olivine Fo<sub>89.8–90.4</sub> at higher temperatures (1230–1280°C).

The consistency of a number of chemical and physical parameters between melt inclusions in spinel and naturally quenched glass suggests that melt inclusions trapped in spinel are the most suitable for petrologic studies. Unlike glass inclusions in silicates — olivine (depletion in MgO and FeO), clinopyroxene (depletion in CaO and MgO) and plagioclase (depletion in Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>) — melt inclusions in Cr-spinel generally survive the post-entrapment chemical modification.

Melt inclusions and host spinels show positive correlations between such compositional parameters as Mg# ( $100 \times Mg/(Mg + Fe^{2+})$ ), Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> contents (Fig. 1). The observed relationship between Al<sub>2</sub>O<sub>3</sub> in spinel and melt is close to that predicted in the experimental work of Maurel and Maurel [27]. The composition of spinel may be used as indicator of melt composition (Mg#, Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub>) for a given range and type of compositions, and may eventually allow development of a quantitative model of spinel–melt equilibrium.

Mg# values of spinel inclusions in olivine corre-

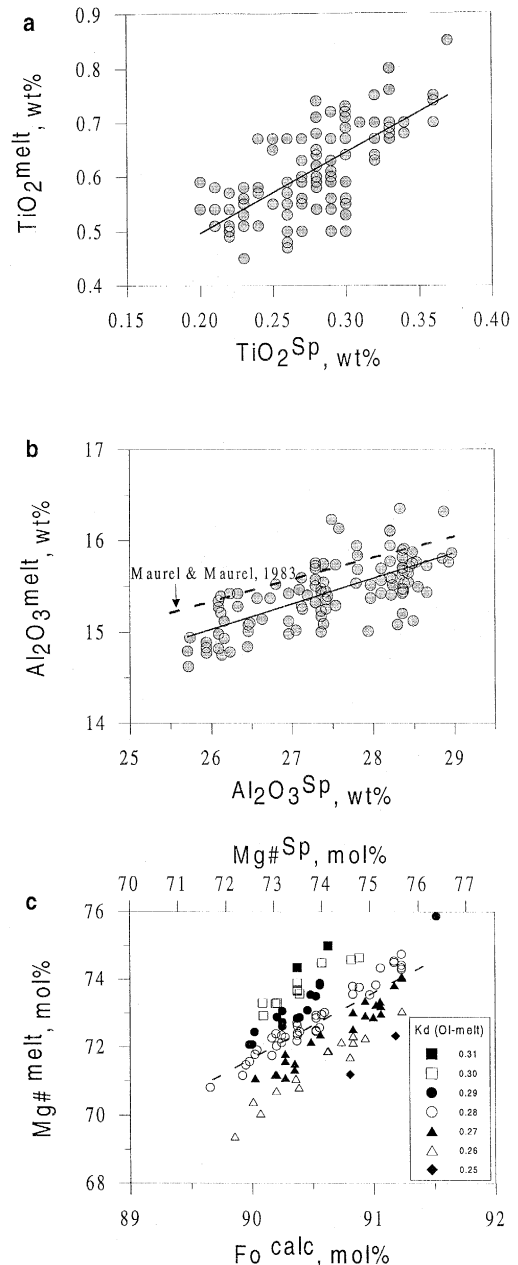


Fig. 1. Relationship between compositions of melt inclusions and their host spinels. The dashed trend in (b) is after [27]. In (c) Fo was calculated using the olivine–spinel compositional relation from Fig. 2.

late positively with Fo values of their host olivine (Fig. 2). Low temperature re-equilibration in this case is limited by the restricted range of decreasing

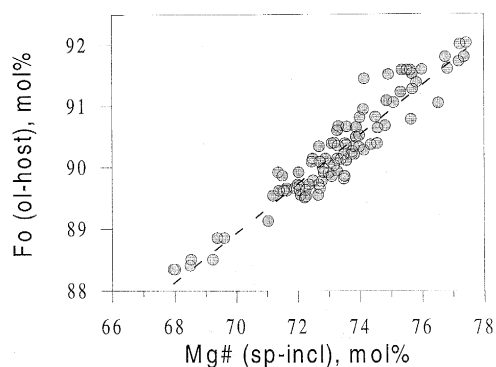


Fig. 2. Correlation between compositions of spinel inclusions and host olivine.

magmatic temperatures ( $\sim 100$ – $120^\circ\text{C}$ , manifested by a narrow range of olivine phenocryst compositions:  $\text{Fo}_{92-88}$ ), and effective quenching by seawater. If melt inclusions are equilibrated with their host spinel, the hypothetical equilibrium olivine composition can be calculated via the Mg# spinel–Fo relationship, as presented in Fig. 1c.

The calculated  $K_d^{\text{Fe}^{2+}-\text{Mg}}$  (olivine–melt) varies from 0.31 to 0.25, averaging  $0.28 \pm 0.01$  (Fig. 1c). The same range of  $K_d^{\text{Fe}^{2+}-\text{Mg}}$  (olivine–melt) was recently reported by Baker et al. [28] from low-degree melting experiments and thermodynamic calculations for similar temperature conditions ( $T = 1250$ – $1300^\circ\text{C}$ ) and melt ( $\text{Mg}\# = 69.7$ – $76.8$ ) and olivine ( $\text{Fo} = 90.3$ – $90.9$ ) compositions. However, as  $K_d = 0.3 \pm 0.03$  [29] is generally accepted for MORB from numerous experimental studies and our melts do not have the extreme variations in  $\text{Na}_2\text{O}$  content (Table 1) that the Baker et al. melts do [28], there is no need to believe that the observed discrepancy in  $K_d$  is real. On the contrary, the simple calculation shows that  $K_d$  is very sensitive to the accuracy of microprobe analyses of coexisting phases. For example, an error of 1 relative percent in determination of Fo or Mg# (melt) values may cause a change in  $K_d$  as large as 0.03 or 0.01, respectively! In our case the observed scatter in Fo for a given Mg# (spinel) (Fig. 2) is sufficient to induce the scatter in  $K_d$  in Fig. 1c. Moreover, the uncertainty in determination of  $\text{Fe}^{2+}/\text{Fe}^{3+}$  (and thus Mg#) in melt always exists and will affect the calculated  $K_d$ .

All studied melt inclusions have very primitive compositions in terms of  $\text{CaO}/\text{Al}_2\text{O}_3$  (0.8–0.9) and

Mg# (69–76). The most primitive melt found in this study (Table 1, no. 9) is trapped in spinel with  $\text{Mg}\# = 76.4$ . It could be in equilibrium with olivine  $\text{Fo} = 91.4$  at  $T = 1280^\circ\text{C}$ . The melt equilibrated with the most primitive olivine found ( $\text{Fo} = 92.1$ ; Table 1, no. 10) should have  $\text{MgO} = 13.5$ – $14$  wt%. This data contributes to the ongoing debate on the nature of MORB parental melts (e.g. reviews in [30–32]). From this study of melt inclusions in spinel we are able to conclude that parental magmas of FAMOUS basalts are neither picritic ( $\text{MgO} = 16$ – $18$  wt%) (e.g. [1,2,5,30,33]), nor resemble the most MgO-rich (9.5–10.5 wt% MgO) MORBs (e.g. [3,4,31,34,35]). Trace element composition of FAMOUS melt inclusions (see below) suggest that parental liquids are an aggregate of magmas formed at different melting extents [10,12]. In general, melt inclusions in the most primitive (earliest on liquidus) spinel can be used to evaluate unmixed and/or unfractionated mantle-derived magmas.

Despite a close similarity in major element composition (except  $\text{K}_2\text{O} < 0.01$ – $0.5$  wt% and  $\text{P}_2\text{O}_5 < 0.05$ – $0.2$  wt%; Table 1), our largely unpublished ion microprobe data show that the melts display a broad and continuous trend of variation of incompatible elements (e.g., (in ppm) La 0.45–10.5; Ba 2–200; Sr 30–240; Zr 16–52; Hf 0.5–1.7;  $\text{La}_n/\text{Yb}_n$

Table 2

Representative trace element compositions (in ppm) of depleted and enriched melt inclusions in FAMOUS spinel

Element	Sample		
	9	11	12
Ba	10.4	45	196
La	0.73	3.3	10.8
Ce	3.6	10.0	18.1
Sr	47	83	221
Nd	2.9	5.0	4.8
Zr	38.2	21.4	21.7
Hf	1.48	0.57	0.91
Sm	1.63	1.05	1.15
Eu	0.55	0.50	0.66
Gd	2.22	2.34	
Dy	3.60	1.77	2.06
Y	17.6	15.3	15.3
Er	2.76	1.53	1.60
Yb	2.76	1.27	1.20

The major element compositions are given in Table 1 (nos. 9, 11 and 12).

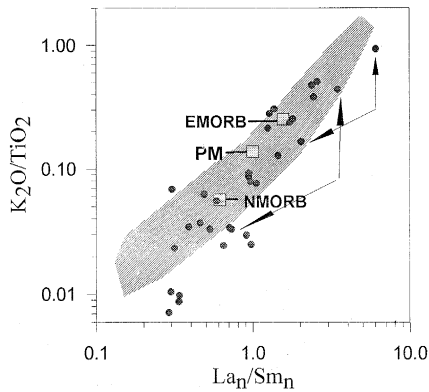


Fig. 3. Correlation between  $K_2O/TiO_2$  and  $La_n/Sm_n$  in melt inclusions. Normalization to the primitive mantle [41]. Arrows point to the pairs of melt inclusions in the same grains of spinel, sample C. MORB field is after [37–40]. PM, NMORB and EMORB are compositions of the primitive mantle, depleted and enriched MORB, respectively [41].  $K_2O/TiO_2$  and  $La_n/Sm_n$  are used as indicators of the enrichment of parental melts. These ratios are not affected by fractional crystallization of primitive MORB cotectic assemblage (ol + sp ± pl ± cpx). Note that, in our case, the variations in  $K_2O/TiO_2$  and  $La_n/Sm_n$  (i.e., the degree of enrichment/depletion) are primarily due to significant variations in  $K_2O$  and La.

0.2–6.5 [36]), which fits and overlaps with the overall field of MOR rocks and glasses (Table 2, Figs. 3 and 4). Surprisingly, contrasting compositions (in terms of concentrations of trace elements more incompatible than Sm–Nd) are recorded from even within a single spinel grain (Tables 1 and 2, nos. 11 and 12; Fig. 4). This suggests crystallization from a continuous spectrum of MORB primary melts or their mixtures. As hypothesized by previous workers [39,42], contrasting melts intermix to give rise to generally uniform N-MORB. Therefore, a primary MORB magma in the strictest sense is very unlikely to be represented as a rock or a natural glass, but can be successfully evaluated from melt inclusions study.

Melt inclusions in spinel offer an opportunity to ‘see through’ magma chamber mixing and contamination processes that have affected the pre-eruption history of most MORBs. The same technique may be able to provide a glimpse of possible primary or parental magma compositions of other major geochemical rock types (ocean island basalts, continental flood basalts, komatiites, subduction related rocks, etc.) before any fractionation, mixing, or contamina-

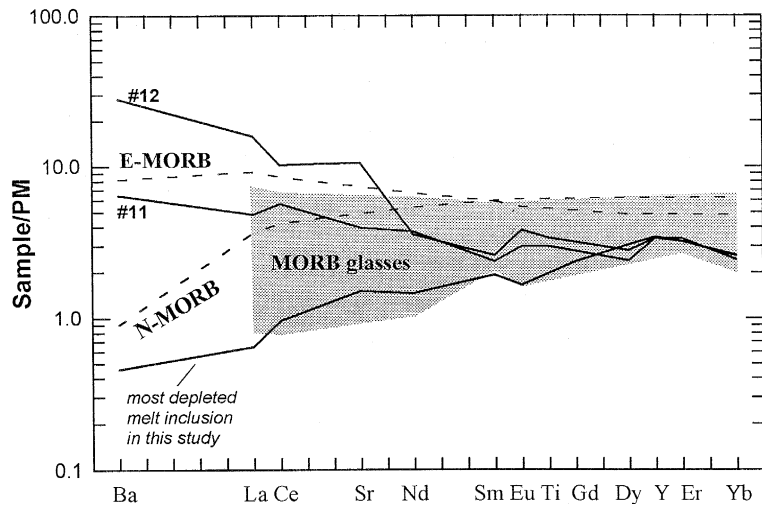


Fig. 4. Trace element compositions normalized to ‘primitive mantle’ [41] of representative enriched and the most depleted melt inclusions in FAMOUS spinel. Patterns 11 and 12 (Table 1 and Table 2, numbers 11 and 12) represent melt inclusions in the same grain of spinel. FAMOUS melt compositions are compared with a range of primitive MORB glasses and glass inclusions [42] and reference types of E-MORB and N-MORB [41]. Depleted melt composition can be modelled as an instantaneous melt fraction at 14% of critical melting of a depleted mantle with 3.3 wt% melt retained in the residue. Enriched melts require a binary mixing of a small amount (< 15%) of the low degree melting fraction (~ 0.3%) of critical melting of a mixed source (MORB mantle + 30–40% ‘primitive mantle’) with 0.3% retained melt and a large volume (> 85%) of the depleted melt. This proportion satisfactorily explains the permanency of major element compositions of FAMOUS melt inclusions. The complete set of trace element compositions and petrogenetic model of the origin of depleted and enriched FAMOUS melts and their mixtures will be published elsewhere.

tion by assimilation of a crust. Finally, as detrital Cr-spinel is common in many sedimentary rocks in fold belts, homogenized melt inclusions in spinels offer great promise towards determining tectonic affinities and implications of mafic and ultramafic rocks in the provenance areas of ancient sediments.

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