

Evidence for melting mud in Earth's mantle from extreme  
oxygen isotope signatures in zircon

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**ABSTRACT**

The role of sediment melting in Earth's mantle remains controversial, as direct  
observation of melt generation in the mantle is not possible. Geochemical fingerprints provide  
indirect evidence for subduction-delivery of sediment to the mantle, however sediment  
abundance in mantle-derived melt is generally low (0-2%), and difficult to detect. Here we

provide evidence for bulk melting of subducted sediment in the mantle through isotopic analysis of granite sampled from an exhumed mantle section. Peraluminous granite dikes that intrude peridotite in the Oman-United Arab Emirates ophiolite have U-Pb ages of  $99.8 \pm 3.3$  Ma that predate obduction at ca. 85 to 90 Ma. The dikes have unusually high oxygen isotope ( $\delta^{18}\text{O}$ ) values for whole rock (14-23‰) and quartz (20-22‰), and yield the highest  $\delta^{18}\text{O}$  zircon values known (14-28‰; values relative to Vienna standard mean ocean water). The extremely high oxygen isotope ratios uniquely identify the melt source as high  $\delta^{18}\text{O}$  marine sediment (pelitic and/or siliceous mud), as no other source could produce granite with such anomalously high  $\delta^{18}\text{O}$ . Formation of high  $\delta^{18}\text{O}$  sediment-derived (S-type) granite within peridotite requires delivery of sediment to the mantle by subduction, where it melted and intruded the overlying mantle wedge. The granite suite described here contains the most evolved oxygen isotope ratios reported for igneous rocks, yet intruded mantle peridotite below the Mohorovičić seismic discontinuity, the most primitive oxygen isotope reservoir in the silicate Earth. Identifying the presence and quantifying the extent of sediment melting within the mantle has important implications for understanding subduction recycling of crust and mantle heterogeneity over time.

## INTRODUCTION

Subduction of lithosphere to the upper mantle is a fundamental tenet of plate tectonics (Wilson, 1963). Detection of sediment contributions to mantle-derived melt provides evidence for transport of sedimentary material to the mantle via subduction. Geochemical tracers used to detect the presence of sediment in mantle melt (i.e. basalt) include trace elements (Ba, La, Th, and others, e.g., Plank and Langmuir, 1993), oxygen isotopes (Eiler et al., 1995), and chlorine isotopes (John et al., 2010). Calculated sediment abundances are typically low,  $<2\%$  (e.g., Eiler et al., 1995). Here, field observations, petrologic data, and oxygen isotope evidence are used to

show that peraluminous granite dikes intruding peridotite in the Oman-United Arab Emirates (UAE) ophiolite formed by bulk melting of marine sediment in the mantle.

Opportunities to observe *in situ* evidence of melt generated in the mantle are rare, and generally restricted to ophiolites, sections of oceanic crust and mantle exposed at convergent plate margins (Dewey, 1976). The Oman-UAE ophiolite is considered the type-example of an ophiolite, consisting of an intact section of oceanic crust and mantle that was obducted onto the Arabian continental margin in the Late Cretaceous (Fig. 1A) (Searle et al., 2015). The crustal section includes ~8 km of pillow basalt, sheeted dikes, and gabbro above the assumed Mohorovičić seismic discontinuity, defined locally by the transition from peridotite to gabbro (i.e., the petrologic Moho). The underlying mantle section consists of up to 15 km of peridotite (harzburgite), and hosts the granite dike suite studied here (Fig. 1B).

Three distinct suites of granites (*sensu latu*) are recognized in the Oman-UAE ophiolite. Granites intruding the crustal section form a ‘crustal plagiogranite’ suite that ranges from granodiorite to tonalite, has SiO<sub>2</sub> values from 54-72 wt.%, and low K<sub>2</sub>O (<1 wt.%). The crustal plagiogranite suite is metaluminous, contains zircon with  $\delta^{18}\text{O}$  values from ~4-5‰, and formed by fractionation of mantle melt or anatexis of oceanic crust (Grimes et al., 2013; Rollinson, 2015). Two additional granite suites intrude peridotite below the Moho. One of the granite suites in the mantle section has SiO<sub>2</sub> values from 67-75%, is metaluminous, and ranges from granodiorite to tonalite. The other granite suite intrusive to the mantle and the focus of this study is distinct from all other granite suites in the ophiolite in that it has higher SiO<sub>2</sub> values (72-78%), it is peraluminous and is predominately true granite (*sensu strictu*), consisting of near-equal proportions of quartz, plagioclase, and alkali feldspar (Cox et al., 1999; Rollinson, 2015; Searle et al., 2015). The origin of the peraluminous suite is debated; proposed formation mechanisms

include fractionation from mafic melt (Peters and Kamber, 1994), anatexis of subducted pelitic material (Cox et al., 1999; Searle et al., 2015), and mixtures of slab fluids with melted crust and sediment (Rollinson, 2015; Haase et al., 2015). A key observation recognized from field studies (Peters and Kamber, 1994) and confirmed by zircon U-Pb dating (Styles et al., 2006; Rioux et al., 2013; this study) is that the granite suites in the mantle section formed synchronous with ocean crust formation at ca. 99 Ma, and thus pre-date ophiolite obduction from ca. 85 to 90 Ma (Searle et al., 2015; Styles et al., 2006). The pre-obduction intrusive relationship thus distinguishes the Oman-UAE granite suite from other ophiolites where granite intruded peridotite post-obduction, such as the Ronda peridotite in Spain (Priem et al., 1979).

## **METHODS AND SAMPLES**

We present  $\delta^{18}\text{O}$ , U-Pb, and  $\epsilon\text{Hf}$  data for zircon, and  $\delta^{18}\text{O}$  for whole rock and quartz from ten dike samples (see the GSA data repository<sup>1</sup> item DR1 for full analytical methods). Zircon grains were imaged by scanning electron microscopy, analysed for oxygen isotope ratio using secondary ion mass spectrometry (SIMS), and U-Pb age and  $\epsilon\text{Hf}$  by laser ablation inductively couple plasma mass spectrometry (LA-ICPMS) (Items DR1, DR2). Analyses of whole rock and quartz for oxygen isotope ratio were made by laser fluorination (Items DR1, DR2).

Ten peraluminous granite dikes along a ~250 km length of the mantle section of the Oman-UAE ophiolite were analyzed (Fig. 1; Item DR1). The dikes are intrusive to peridotite, and range from 2-8 meters in width (Fig. 1B). Major and trace element data for dikes at each of the three sampling sites (our samples with prefixes 13- and 14-) were reported previously (Rollinson, 2015). The granite dikes are uniformly peraluminous ( $\text{Al}_2\text{O}_3/\text{Na}_2\text{O}+\text{K}_2\text{O}+\text{CaO} > 0.99$ ), high in  $\text{SiO}_2$  (~74-77% wt.%) and low in Fe, Mg, Ca, Na, and all but one has normative corundum (0.4-0.9%) (Item DR1); geochemical data thus distinguish this suite from

metaluminous granites in the ophiolite. The peraluminous granites contain accessory minerals such as muscovite, biotite, garnet, cordierite, andalusite, lepidolite, tourmaline, and zircon (Rollinson, 2015; Item DR1). Some samples exhibit evidence of alteration of various minerals to secondary phases.

## **RESULTS**

### **Zircon U-Pb and $\epsilon$ Hf by LA-ICPMS**

Zircon grains from seven samples were analysed for U-Pb, and yield a single coherent  $^{206}\text{Pb}/^{238}\text{U}$  age of  $99.8 \pm 3.3$  Ma ( $2\sigma$ , MSWD = 1.7;  $n = 16$ ) (Fig. 1C). Analysis of the same grains for Hf isotopes yield  $\epsilon$ Hf values ranging from 2.8 to -4.4 ( $n = 16$ ; Item DR1).

### **Zircon $\delta^{18}\text{O}$ by SIMS**

Measured  $\delta^{18}\text{O}$  for individual 15  $\mu\text{m}$  spots on zircon grains ranges from  $13.8 \pm 0.5\text{‰}$  to  $27.0 \pm 0.7\text{‰}$  ( $2\sigma$ ), with a mean zircon  $\delta^{18}\text{O}$  for the suite of  $19.7 \pm 2.9\text{‰}$  ( $1\sigma$ ,  $n=125$  analyses on 112 zircon grains; Fig. 2; Items DR1, 2). Grains in most samples record minor within-sample variation of  $\delta^{18}\text{O}$  ( $<2\text{‰}$ ), whereas in some samples grain-to-grain variability is up to 11‰. Variable  $\delta^{18}\text{O}$  values also occur within individual grains (Fig. 3A). Multi-spot analysis shows that some grains are homogenous in  $\delta^{18}\text{O}$ , whereas others are zoned by up to ~9‰ (item DR1). The highest  $\delta^{18}\text{O}$  zircon (grain UAE436\_1, Fig. 3A) was analysed an additional 57 times using a ~3  $\mu\text{m}$  spot (Fig. 3B), with  $\delta^{18}\text{O}$  ranging from  $17.7 \pm 1.6\text{‰}$  to  $30.7 \pm 1.5\text{‰}$  in a symmetrical pattern. The ~3  $\mu\text{m}$  spot analyses reveal a 28‰ core surrounded by a 19‰ rim (Fig. 3C). Concurrent analysis of  $^{16}\text{O}^1\text{H}/^{16}\text{O}$  yielded low background corrected values (0-0.0006), which showed no significant deviation during the 57 analyses, indicating the core is not metamict (e.g., Wang et al., 2014).

### **Whole Rock and Quartz $\delta^{18}\text{O}$ by Laser Fluorination**

Measured whole rock granite and quartz  $\delta^{18}\text{O}$  values range from 14.3-19.3‰ and 19.9-22.1‰, respectively (Fig. 2B). The whole rock  $\delta^{18}\text{O}$  values are uniformly high for igneous rocks, although some are lower than predicted based on zircon. The range of measured zircon  $\delta^{18}\text{O}$  values for high-silica granites (~76 wt.%  $\text{SiO}_2$ ) would be in equilibrium with whole rock  $\delta^{18}\text{O}$  ranging from 16-29‰ (Valley et al., 2005). The whole rock  $\delta^{18}\text{O}$  values may thus record variable exchange during low-temperature alteration.

## **DISCUSSION**

### **High $\delta^{18}\text{O}$ S-type Granite in the Oman UAE Ophiolite**

The Oman-UAE peraluminous granite dike suite described here (Fig. 2B) are the highest  $\delta^{18}\text{O}$  igneous rocks known (Cavosie et al., 2011). The previously reported highest  $\delta^{18}\text{O}$  values for igneous zircon (~14‰), quartz (~18‰), and whole rock (~16‰) are from ~1.2 billion year old granitoids of the Frontenac terrane in Ontario, Canada (Fig. 2B; Peck et al., 2004). Formation of the high  $\delta^{18}\text{O}$  Frontenac granitoids is attributed to subduction and melting of continental material during accretionary orogeny. Mean values presented here for zircon (~20‰), quartz (~21‰), and whole rock (~17‰) are significantly higher than those found in the Frontenac granitoids.

Taken together, all available geochemical data for the granite dikes, including high  $\delta^{18}\text{O}$ , peraluminous character, normative corundum, the presence of aluminous minerals (garnet, muscovite and andalusite), and low Fe, Mg, Ca, and Na, identify the suite as having originated through melting of supracrustal rocks, i.e. S-type granite (Chappell and White, 1974). Previous studies of zircon in S-type granite have found anomalously large intra-sample variation in igneous  $\delta^{18}\text{O}$  (e.g., Appleby et al., 2010), similar to findings reported here, that have been attributed to heterogeneities in the melt source. Identification of crustal Nd and Hf isotope

signatures (Haase et al., 2015) and elevated trace element abundance (Rollinson, 2015) in studies of peraluminous granite from the Oman-UAE ophiolite further support their classification as S-type granite.

### **Oxygen Isotopes Constrain a Sediment Source**

We hypothesize that high  $\delta^{18}\text{O}$  pelagic mud (pelitic and/or siliceous) was subducted and melted below the rapidly forming ocean crust during slab rollback (Rioux et al., 2013; Searle et al., 2015). The high  $\delta^{18}\text{O}$  values measured for zircon, quartz, and whole rock in this study uniquely restrict the melt source of the peraluminous granite suite to high  $\delta^{18}\text{O}$  sediment. The range of  $\delta^{18}\text{O}$  zircon from 14 to 28‰, including grains zoned by 9‰ (Fig. 3), further indicates the source was heterogeneous. The extremely elevated  $\delta^{18}\text{O}$  signatures for igneous zircon exclude a mantle source, as whole rock values for peridotite and derivative melts do not exceed ~6‰ (Valley et al., 2005). A global survey of whole rock values identifies shale ( $\delta^{18}\text{O}$  up to 26‰; Payne et al., 2015) as the most likely high  $\delta^{18}\text{O}$  source that would yield peraluminous granite when melted (Nichols et al., 1994). Other possible high  $\delta^{18}\text{O}$  sources include siliceous and carbonate oozes, which have whole rock  $\delta^{18}\text{O}$  values from 25-42‰ (Priem et al., 1979; Eiler, 2001). However, bulk melts of oozes or lithified equivalents would not form peraluminous granite, which limits their contributions, if present. The lower range of calculated whole rock  $\delta^{18}\text{O}$  values based on zircon (e.g., 16-24‰) could potentially represent melt of altered basalt (50 wt%  $\text{SiO}_2$ ,  $\delta^{18}\text{O}$  ~9‰) mixed with chert (100 wt%  $\text{SiO}_2$ ,  $\delta^{18}\text{O}$  ~40‰), although such melts have never been reported. Furthermore, only a melt consisting of shale (60 wt%  $\text{SiO}_2$ ,  $\delta^{18}\text{O}$  ~20‰) and chert could produce a 75 wt%  $\text{SiO}_2$  granite with whole rock  $\delta^{18}\text{O}$  of 29‰.

No appreciable volume of shale is exposed near the Oman-UAE ophiolite on the paleo-continental shelf (Searle et al., 2015; Searle and Cox, 1999). Siliceous schist, marble, and chert,

all with high whole rock  $\delta^{18}\text{O}$ , occur regionally (Grantham et al. 2003), however these lithologies do not have an appropriate composition to form S-type granite when melted (Rollinson, 2015). The lack of shale in the region is not surprising, as the majority of pelagic pelitic mud in modern marine settings is found further offshore and would have been among the first continentally-derived material subducted at ~99 Ma. The zircon  $\epsilon\text{Hf}$  values (Item DR1) are consistent with derivation of the source sediments from the Arabian Nubian Shield, inboard of the Oman-UAE ophiolite (Morag et al., 2011). The U-Pb ages preclude a detrital origin for the zircon grains, which is consistent with a pelagic sediment source (Fig. 1C).

### **Melting Mud in the Mantle**

Data presented here from the Oman-UAE ophiolite provide direct geochemical evidence for sediment melting within the mantle from an intact section of mantle lithosphere. Prior estimates for sediment components in the Oman-UAE mantle granite suites range from 10-30% (trace elements, Rollinson, 2015), to near 80% (Hf-Nd isotopes, Haase et al., 2015), however our results constitute the first report of bulk melting. No indication of mantle-derived melt was detected in the peraluminous granite suite described here, nor is one required, as the granite compositions can be fully explained by bulk melting of high  $\delta^{18}\text{O}$  marine sediment. The peraluminous granite suite described here may thus represent the first report of S-type granite that contains no detectable component of ‘primitive melt’ (c.f. Kemp et al., 2008).

Recognition of the peraluminous granite dikes as nearly pure sediment melts may lead to new insights into related subduction zone processes, including constraining the steady-state volume of subducted sediment needed to source the melts, and the rheology and thermal state of the mantle wedge to allow for semi-brittle fracture formation and propagation during dike emplacement. The initiation of subduction, formation of oceanic crust, melting of sedimentary



material, and intrusion of S-type granite into the mantle wedge all pre-date thrusting of the oceanic crust onto the Arabian continental margin to form the Oman-UAE ophiolite (Searle et al., 2015; Searle and Cox, 1999). These data thus provide ‘ground truth’ confirmation of recently proposed models for sediment subduction and melting beneath supra-subduction zone ophiolites (Furnes and Dilek, 2017). The recognition that small-volume, isotopically evolved, granitoid reservoirs form in the upper mantle as a consequence of subduction also has implications for the compositional heterogeneity of the mantle over time.

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## FIGURE CAPTIONS

**Figure 1.** A: Simplified map of the Oman-UAE ophiolite (Rollinson, 2015) with locations of  
sampling sites. B: Photograph of peraluminous granite intruding mantle peridotite (harzburgite)  
near Al-Dadnah, UAE. C: Terra-Wasserberg concordia of U-Pb data for granite samples from  
this study.

**Figure 2.** A: Histogram of  $\delta^{18}\text{O}$  zircon analyses (15  $\mu\text{m}$  SIMS spots) from peraluminous granite  
in this study compared with global  $\delta^{18}\text{O}$  compilations of igneous (Cavosie et al., 2011) and  
detrital (Spencer et al., 2014) zircon data. Note that 99.8% of detrital and 100% of igneous grains  
are below 13.8‰, the lowest measured  $\delta^{18}\text{O}$  values of zircon from the peraluminous granite

reported here. B: Zircon, quartz, and whole rock  $\delta^{18}\text{O}$  values from Frontenac terrane granitoids (Peck et al., 2004) and peraluminous granite (this study).

**Figure 3.** A: Cathodoluminescence (CL) image of zircon UAE436\_1, displaying locations of the three 15  $\mu\text{m}$   $\delta^{18}\text{O}$  spots. B: Backscatter electron image showing the fifty-seven  $\sim 3 \mu\text{m}$  spot analyses for  $\delta^{18}\text{O}$ . C: CL image of the same surface shown in B with color-coded analytical spots based upon the color scale. The spot with a cross in C is excluded, as it overlapped an inclusion visible in B. A histogram and kernel density estimation (dashed curve) are shown right of the color scale (bin- and bandwidth = 1.5‰). Background corrected  $^{16}\text{O}^1\text{H}/^{16}\text{O}$  (using  $\delta^{18}\text{O}$  standard 91500) is shown as a function of  $\delta^{18}\text{O}$  (vertical line) and is nearly invariant. All  $\delta^{18}\text{O}$  values are in ‰, with average uncertainties shown in A and B.

<sup>1</sup>GSA Data Repository item 2017xxx, material, methods, and full data tables are available online at [www.geosociety.org/pubs/ft20XX.htm](http://www.geosociety.org/pubs/ft20XX.htm), or on request from [editing@geosociety.org](mailto:editing@geosociety.org) or Documents Secretary, GSA, P.O. Box 9140, Boulder, CO 80301, USA.