Bridging the depleted MORB mantle and the continental crust using titanium isotopes

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Supplementary Information

The Supplementary Information includes:

➢ Materials
➢ Methods
➢ Supplementary Text
➢ Tables S-1 and S-2
➢ Figures S-1 and S-2
➢ Supplementary Information References

Materials

The Ti isotopic composition of four geological reference materials from the Geological Survey of the USA, including a Hawaiian basalt (BHVO-2), an Icelandic basalt (BIR-1), a Columbia River basalt (BCR-2) and a Guano Valley andesite (AGV-1) were analysed. Komatiites were collected from five cratons over the world (Pilbara, Kaapvaal, Zimbabwe, Yilgarn and Superior), and have eruption ages of 3.5-2.7 Ga (Table S-1). Only the freshest samples showing spinifex textures and also the chemical compositions closest to the parental magmas were chosen. Details on the samples can be found in Sossi et al. (2016). MORB samples from multiple mid-ocean ridges, including five N-MORB and five E-MORB, which have been studied for Cu isotopes in Savage et al. (2015) (Table S-1).

Methods

The powders of komatiites (= 100 mg) were dissolved following a Parr bomb digestion method described in Sossi et al. (2016). Around 11 to 48 mg rock powders of the rock standards and MORB samples were dissolved in 7 ml Savillex PFA beakers with 2 ml 26 M HF and 1 ml 16 M HNO₃ at 120 °C on a hotplate for three days. After drying down, the samples were dissolved in 3 ml 6 M HCl at 130 °C to decompose the fluorides.

The sample aliquots containing 2 to 6 µg Ti were spiked with a prepared ⁴⁷Ti-⁴⁹Ti double spike, and were then heated at 100 °C to reach sample-spike homogenisation. Afterwards, the sample solutions were dried down, and the re-dissolved sample solutions were subjected to a three-step ion-exchange chromatographic procedure, including Eichrom DGA (50-100 µm particle size) and Bio-Rad AG1-X8 (200-400 mesh) resins, to remove the matrices from Ti (Deng et al., 2018). The Ti isotopic composition of the purified Ti fraction was then measured by a Thermo-Fisher Neptune multi-collector inductively-coupled-plasma mass-spectrometer (MC-ICP-MS) housed at the Institut de Physique du Globe de Paris (France). The sample solutions containing ~300 ppb of natural Ti were introduced in the MC-ICP-MS in 0.5 M HNO₃ + 0.0015 M HF via an APEX HF desolvating nebulizer (Elemental Scientific Inc. USA). A spiked IPGP-Ti standard was analysed in between each two analyses of unknown samples for
the secondary normalisation. The intensities of $^{44}$Ca, $^{46}$Ti, $^{48}$Ti, $^{49}$Ti and $^{47}$Ti were monitored simultaneously under a medium mass resolution (M/ΔM = 5800) with a static mode. After the correction of Ca isobaric interferences, the signals of $^{46}$Ti, $^{48}$Ti, $^{49}$Ti and $^{47}$Ti were used for spike inversion in the IsoSpike software developed by Creech and Paul (2015). The derived data are reported with the δ-notation relative to the standards, IPGP-Ti or OL-Ti, and expressed in ‰:

$$^{49}Ti_{\text{standard}} = \left( \frac{^{49}Ti/^{47}Ti}_{\text{sample}} \right) \times 1000 \quad \text{Eq. S-1}$$

**Supplementary Text**

**Results and inter-laboratory data comparison**

Due to the presence of the small amount of isotopically fractionated Ca within the double spike, a correction of 0.022 ± 0.009 ‰ (2 se, n = 9) on the δ$^{49}$Ti(IPGP-Ti) value has been conducted for all the samples. This change does not affect the isotopic difference between samples in this study. However, if aiming for high-precision inter-laboratory comparison, the analytical uncertainties of ± 0.009 ‰ from the correction of Ca effects and ± 0.011 ‰ from the calibration between IPGP-Ti and OL-Ti have to be propagated onto the corrected or re-normalised values (Deng et al., 2018). After all these corrections, the δ$^{49}$Ti values of four rock standards (BHVO-2, BIR-1, BCR-2, AGV-1), which without specification would stand for the normalization to OL-Ti standard, are consistent with the reported values in Millet et al. (2016) within uncertainty (Table S-1). In addition, the N-MORB samples reported here exhibit an average δ$^{49}$Ti value of +0.000 ± 0.008 ‰ (2 se, n = 5) or +0.000 ± 0.016 ‰ if propagating all the uncertainties above, which is in agreement with the N-MORB average value reported in Millet et al. (2016), i.e. δ$^{49}$Ti = +0.002 ± 0.005 ‰ (2 se, n = 7; Table S-2; Fig. S-1). These corroborate the accuracy of the method in this study.

Komatiites are characterised by a progressive depletion in both the light rare earth elements and heavy Ti isotopes with time (Figs. 1 and 2). In detail, the 3.5-3.3 Ga komatiites have (La/Sm)$_N$ values of 0.91-1.02, with subscript ‘N’ denoting a normalisation to the primitive mantle values from McDonough and Sun (1995), and an average δ$^{49}$Ti value of +0.038 ± 0.018 ‰ (2 se, n = 4). The 2.9-2.7 Ga komatiites show the lower (La/Sm)$_N$ values of 0.37-0.78 and a lower average δ$^{49}$Ti value of +0.003 ± 0.013 ‰ (2 se, n = 5) (Fig. 1; Table S-1). Although being reported with a larger analytical uncertainty of ± 0.035 ‰ (95% confidence interval), similar systematics appears between the komatiite samples reported by Greber et al. (2017a), i.e. the komatiites with the primitive mantle trace element patterns tend to be isotopically heavier than depleted komatiites (Fig. S-1; Table S-2).

The average δ$^{49}$Ti values for chondrites in Deng et al. (2018) and Williams (2014) are 0.04-0.06 ‰ higher than the chondrite average in Greber et al. (2017a). Although the cause of the inter-laboratory discrepancy for chondrites and komatiites is not fully resolved yet (Deng et al., 2018), there are differences for the two analytical sessions on komatiites and chondrites in Greber et al. (2017a). Their first batch including most of chondrite samples has δ$^{49}$Ti values lower by 0.01-0.04 ‰ relative to the second batch (which includes the Allende meteorite) (Fig. S-2; Table S-2). The chondrite and komatiite data in this study, Deng et al. (2018) and Williams (2014) are more consistent with the results of the second batch in Greber et al. (2017a).
**Supplementary Tables**

**Table S1** Chemical and Ti isotopic compositions of komatiites and MORBs.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Location/Type/Lithology</th>
<th>Age (Ga)</th>
<th>MgO (wt. %)</th>
<th>TiO₂ (wt. %)</th>
<th>Δ⁶⁷Sr/⁸⁷Sr</th>
<th>(La/Sm)ᵣ</th>
<th>δ¹⁸⁷⁷⁰⁸⁷⁵¹T i (%)&lt;sup&gt;c&lt;/sup&gt;</th>
<th>2 s.e.⁺</th>
<th>2 s.e.⁻</th>
<th>δ¹⁸³⁷²⁶⁸⁷⁵¹T i (%)&lt;sup&gt;c&lt;/sup&gt;</th>
<th>2 s.e.⁺</th>
<th>2 s.e.⁻</th>
<th>n&lt;sup&gt;b&lt;/sup&gt;</th>
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<td></td>
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<td></td>
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<td></td>
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<td>Ti standard</td>
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<td>0.019</td>
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<td>0.011</td>
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<td>0.016</td>
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<tr>
<td>BIR-1</td>
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<td>2.26</td>
<td>2.41</td>
<td>-0.150</td>
<td>0.009</td>
<td>0.012</td>
<td>0.011</td>
<td>-0.011</td>
<td>0.017</td>
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</tbody>
</table>

| Komatiites                      |                                    |          |             |             |            |           |                             |        |        |                             |        |        |         |
| 179/751        | Pilbara Craton, Western Australia   | 3.515    | 23.54       | 0.42        | 1.02       | -0.121    | 0.015                       | 0.018  | 0.018  | 0.018                        | 0.021  | 9      |
| 331/783        | Kaapvaal Craton, South Africa       | 3.48     | 26.27       | 0.42        | 0.98       | -0.087    | 0.016                       | 0.018  | 0.018  | 0.053                        | 0.022  | 12     |
| 331/777a       | Kaapvaal Craton, South Africa       | 3.48     | 25.10       | 0.41        | 1.01       | -0.089    | 0.008                       | 0.012  | 0.012  | 0.050                        | 0.016  | 4      |
| 176/723        | Pilbara Craton, Western Australia   | 3.28     | 31.13       | 0.30        | 0.91       | -0.118    | 0.007                       | 0.011  | 0.011  | 0.022                        | 0.016  | 3      |
| B-R1           | Zimbabwe Craton, Zimbabwe           | 2.8      | 27.72       | 0.29        | 0.75       | -0.132    | 0.011                       | 0.015  | 0.015  | 0.007                        | 0.018  | 4      |
| B-R2           | Zimbabwe Craton, Zimbabwe           | 2.8      | 27.54       | 0.30        | 0.78       | -0.135    | 0.013                       | 0.015  | 0.015  | 0.005                        | 0.019  | 3      |
| SD5/354.5      | Yilgarn Craton, Western Australia   | 2.7      | 25.72       | 0.39        | 0.56       | -0.121    | 0.006                       | 0.011  | 0.011  | 0.019                        | 0.015  | 7      |
| 422/94         | Superior Craton, Canada             | 2.7      | 22.41       | 0.45        | 0.42       | -0.160    | 0.013                       | 0.016  | 0.016  | -0.020                       | 0.020  | 6      |
| 422/95         | Superior Craton, Canada             | 2.7      | 22.47       | 0.41        | 0.37       | -0.138    | 0.014                       | 0.017  | 0.017  | 0.001                        | 0.020  | 8      |

| Mid-ocean ridge basalts (MORBs) |                                    |          |             |             |            |           |                             |        |        |                             |        |        |         |
| EW9309 2D-1g     | Enriched-type, Mid Atlantic Ridge   | ≈ 0      | 7.60        | 2.04        | 0.704127   | 1.85      | -0.100                      | 0.029  | 0.031  | 0.040                        | 0.033  | 6      |
| DIVA1 15-5       | Enriched-type, Mid Atlantic Ridge   | ≈ 0      | 5.93        | 1.16        | 0.703214   | 1.72      | -0.113                      | 0.015  | 0.017  | 0.027                        | 0.021  | 7      |
| SWIFT DR06-3-6g   | Enriched-type, Southwest Indian Ridge | ≈ 0 | 6.20        | 1.60        | 0.702900   | 1.47      | -0.112                      | 0.010  | 0.013  | 0.028                        | 0.018  | 6      |
| DIVA1 13-3       | Enriched-type, Mid Atlantic Ridge   | ≈ 0      | 7.55        | 1.46        | 0.703000   | 1.70      | -0.104                      | 0.026  | 0.027  | 0.036                        | 0.029  | 7      |
| SWIFT DR04-2-3g   | Enriched-type, Southwest Indian Ridge | ≈ 0 | 6.23        | 1.49        | 0.702465   | 1.39      | -0.094                      | 0.007  | 0.012  | 0.046                        | 0.016  | 6      |
| PAC2 DR38-1g     | Normal-type, Pacific Atlantic Ridge  | ≈ 0      | 7.57        | 1.31        | 0.703214   | 1.72      | -0.113                      | 0.010  | 0.012  | -0.011                       | 0.017  | 6      |
| MD57 D2-8        | Normal-type, Central Indian Ridge    | ≈ 0      | 6.91        | 1.51        | 0.702465   | 0.61      | -0.151                      | 0.008  | 0.012  | -0.011                       | 0.017  | 6      |
| SEARISE1 DR04    | Normal-type, East Pacific Rise       | ≈ 0      | 5.24        | 1.59        | 0.702820   | 0.70      | -0.145                      | 0.011  | 0.014  | -0.005                       | 0.018  | 6      |
| SEARISE2 DR03    | Normal-type, East Pacific Rise       | ≈ 0      | 6.15        | 1.21        | 0.702983   | 0.50      | -0.137                      | 0.007  | 0.011  | 0.002                        | 0.016  | 6      |
| RD87 DR18-102    | Normal-type, Mid Atlantic Ridge      | ≈ 0      | 7.39        | 1.11        | 0.702983   | 0.59      | -0.137                      | 0.006  | 0.011  | 0.003                        | 0.016  | 6      |

<sup>a</sup>The MgO and TiO₂ contents of the komatiites samples are from Sossi et al. (2016), and those of the MORBs are from this study.

<sup>b</sup>The Sr isotopic ratios of the MORBs samples are from the PDB Database (http://www.earthchem.org/pdb).

<sup>c</sup>Subscript "N" represents the normalisation to the primitive mantle values in McDonough and Sun (1995). The (La/Sm)ᵣ values of the komatiites are from Sossi et al. (2016), and those of the MORBs are from this study.

<sup>d</sup>A correction for 0.022 ± 0.009 %, has been conducted for all the samples to account for the effects from the small amount of the highly isotopically fractionated Ca in the used double spike (Deng et al. 2018).
* The analytical uncertainty from the original measurement duplicates.

1 The errors from the correction of Ca effects from double spike have been propagated.

2 The values have been scaled onto the OL-Ti standard using the $\delta^{88_{\text{Ti}}}$ value of $-0.140 \pm 0.011$‰ with error propagation.

3 Number of measurement duplicate.

Table 5-2 Literature Ti isotopic data of MORBs and mantle peridotites from Millet et al. (2016), komatites (Greber et al., 2017a) and Archean TTGs (Greber et al., 2017b).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Location/Type/Lithology</th>
<th>Age (Ga)</th>
<th>MgO (wt. %)</th>
<th>TiO2 (wt. %)</th>
<th>(La/Sm)n</th>
<th>$\delta^{88_{\text{Ti}}}$ (‰)</th>
<th>2 s.e.</th>
<th>2 s.e.</th>
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<th>Reference</th>
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<tbody>
<tr>
<td>A12D8-2</td>
<td>Normal-type, North Atlantic</td>
<td>$\approx$ 0</td>
<td>9.43</td>
<td>0.81</td>
<td>0.55a</td>
<td>-0.143</td>
<td>0.023</td>
<td>-0.003</td>
<td>0.020</td>
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<td>A12D11-1</td>
<td>Normal-type, North Atlantic</td>
<td>$\approx$ 0</td>
<td>8.55</td>
<td>1.17</td>
<td>0.55a</td>
<td>-0.134</td>
<td>0.023</td>
<td>0.006</td>
<td>0.020</td>
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<td>Normal-type, EPR</td>
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<td>7.59</td>
<td>1.33</td>
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<td>Normal-type, EPR</td>
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<td>9.17</td>
<td>1.06</td>
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<td>0.002</td>
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<td>$\approx$ 0</td>
<td>8.23</td>
<td>1.50</td>
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Mantle peridotites

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<th>Sample</th>
<th>Location/Type/Lithology</th>
<th>Age (Ga)</th>
<th>MgO (wt. %)</th>
<th>TiO2 (wt. %)</th>
<th>(La/Sm)n</th>
<th>$\delta^{88_{\text{Ti}}}$ (‰)</th>
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Komatites

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<th>Age (Ga)</th>
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<th>TiO2 (wt. %)</th>
<th>(La/Sm)n</th>
<th>$\delta^{88_{\text{Ti}}}$ (‰)</th>
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<th>2 s.e.</th>
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<td>Schapenburg</td>
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The values have been scaled onto the IPGP-Ti standard using the $\delta^{87}Ti^{GP}$ value of $-0.140 \pm 0.011 \%e$ with error propagation.

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* The MORB samples in Millet et al. (2016) are the normal-type, and the typical average (La/Sm)$_{N}$ value of 0.55 for N-MORBs is used here. The same value has been assumed for the depleted mantle peridotite samples from Millet et al. (2016).

* The average average (La/Sm)$_{N}$ value of the other komatiites from Komatii is shown here.

* The typical (La/Sm)$_{N}$ value of the komatiites from Belingwe in Sossi et al. (2016) is shown here.

* The values have been scaled onto the IPGP-Ti standard using the $\delta^{87}Ti^{GP}$ value of $-0.140 \pm 0.011 \%e$ with error propagation.
Supplementary Figures

**Figure S-1** Comparing the komatiite, MORB and mantle peridotite data of this study with those from Millet et al. (2016) and Greber et al. (2017a). The (La/Sm)\textsubscript{n} range of each group of samples is shown.

**Figure S-2** Comparing the δ\textsuperscript{49}Ti values from two analytical sessions on chondrites and komatiites in Greber et al. (2017a). The first batch of dissolutions (blue labels, including most chondrites) provides the lower δ\textsuperscript{49}Ti values than the second batch (pink labels, including Allende meteorite).
Supplementary Information References


