Evidence of Enriched, Hadean Mantle Reservoir from 4.2-4.0 Ga zircon xenocrysts from Paleoarchean TTGs of the Singhbhum Craton, Eastern India

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Sensitive High-Resolution Ion Microprobe (SHRIMP) U-Pb analyses of zircons from Paleoarchean (~3.4 Ga) tonalite-gneiss called the Older Metamorphic Tonalitic Gneiss (OMTG) from the Champua area of the Singhbhum Craton, India, reveal 4.24-4.03 Ga xenocrystic zircons, suggesting that the OMTG records the hitherto unknown oldest precursor of Hadean age reported in India. HF isotopic analyses of the Hadean xenocrysts yield unradiogenic $^{176}\text{Hf}/^{177}\text{Hf}_{\text{initial}}$ compositions (0.27995 ± 0.0009 to 0.28001 ± 0.0007; $\varepsilon\text{Hf}\[t\] = −2.5 to −5.2) indicating that an enriched reservoir existed during Hadean eon in the Singhbhum cratonic mantle. Time integrated $\varepsilon\text{Hf}\[t\]$ compositional array of the Hadean xenocrysts indicates a mafic protolith with $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of ~0.019 that was reworked during ~4.2-4.0 Ga. This also suggests that separation of such an enriched reservoir from chondritic mantle took place at 4.5 ± 0.19 Ga. However, more radiogenic yet subchondritic compositions of ~3.67 Ga (average $^{176}\text{Hf}/^{177}\text{Hf}_{\text{initial}}$ = 0.28053 ± 0.00003) from the same OMTG samples and two other Paleoarchean TTGs dated at ~3.4 Ga and ~3.3 Ga (average $^{176}\text{Hf}/^{177}\text{Hf}_{\text{initial}}$ = 0.28060 ± 0.00003), respectively, corroborate that the enriched Hadean reservoir subsequently underwent mixing with mantle-derived juvenile magma during the Eo-Paleoarchean.

Zircons, which are the only representatives of the oldest rocks on Earth, preserve robust records of chemical and isotopic characteristics as well as the history of the generation of their parent rocks1-5. Thus far, the oldest recorded rocks on Earth are the 4.03-3.92 Ga gneisses from the Acasta Gneiss Complex, Slave Province, Canada1-7. Zircons older than these have been found as detrital grains within metasedimentary rocks from Western Australia8-13, Western Tibet14, Brazil15 and Southern China16 and as xenocrysts within meta-igneous rocks from Western Australia17 and Central China18. The examination of the HF isotopic compositions of the oldest zircons, can further contribute to a comprehensive understanding of the differentiation of the early silicate Earth3,19-22. Contrasting isotopic signatures and interpretations from such databases have fueled a persistent debate about the nature of the source reservoir of the protolith generating Hadean and Eoarchean zircons and the composition of the earliest crust that developed during the Hadean3,6,12,19-21. HF isotopes are tracers that are very widely used to explicate crustal generation processes19-21. Previously, Lu-HF isotopic data from the Jack Hill’s zircons of Hadean age revealed both supra-chondritic and subchondritic initial $^{176}\text{Hf}/^{177}\text{Hf}$ values23. Highly radiogenic Hadean and Eoarchean zircons reported from the Jack Hill’s metaglomerate24,25 implied the presence of depleted reservoirs, which was in contradiction with concurrent Pb-Pb and Lu-HF isotopic studies yielding predominantly unradiogenic $\varepsilon\text{Hf}\[t\]$ values26,27. The highly positive values observed in previous studies were later described as the artifacts of isotopic mixing between different age domains of zircons with very fine oscillatory zoning28. Here, we report xenocrystic zircons of Hadean (~4.0-4.2 Ga) to Eoarchean age (~3.7 Ga) from

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the Paleoarchean (~3.4 Ga) Tonalite-Trondhjemite-Granodiorite gneisses (TTG), called the Older Metamorphic Tonalitic Gneiss (OMTG), of the Paleo-Mesoarchean Singhbhum Craton of Eastern India and confirm that the OMG holds the hitherto oldest precursor rock recorded in India. We also present Lu-Hf isotopic data from these xenocryst cores and their host zircons that add new information to the Hadean zircon isotopic data repository, augmented with interpretations about the nature of their mantle source and the history of crustal formation events in this craton. In addition, the combined U-Pb and Lu-Hf isotopic data of zircons from two other Paleoarchean TTGs (∼3.4 Ga and ∼3.3 Ga) from different locations within the same craton are also presented to further elucidate the characteristics and heterogeneity of the corresponding mantle source reservoir during the Paleoarchean.

**Geology of Paleoarchean TTGs, Singhbhum Craton**

The Paleoarchean Singhbhum Craton, India, consists of an Archean nucleus of voluminous TTG gneisses and intrusive granitoids of ∼3.5-3.2 Ga age, flanked by three Paleoarchean greenstone successions, which are named the Iron Ore Group (IOGs)26–28 (Fig. 1). The Archean nucleus of this craton is unconformably overlain by Paleoproterozoic volcanics by Paleoarchean tonalitic to granodioritic gneisses26,28,33. According to Hofmann and Mazumder 28, the OMTG represents a suite of TTGs that formed over an extended period between 3.53-3.45 Ga, whereas the OMG represents a supracrustal assemblage that formed as a greenstone succession. The oldest age obtained from the OMTG...
is a whole-rock Sm-Nd isochron age of 3775 ± 89 Ma34. This age was later questioned and subsequently amended by Moorbath et al.35 to be closer to 3.4 Ga. However, other older ages recently reported from the OMTG include an age of 3664 ± 79 Ma, which was derived from a whole-rock Pb-Pb isochron36, and a xenocrystic zircon core age of 3.3 Ga26. A sample of coarse-grained, mesocratic granite gneiss (sample TRBG-1; Supplementary Figure SF2), adjacent to the metabasalt-quartzite sequence of the Eastern IOG greenstone belt to the southeast of Galusihing. A relatively fresh, mesocratic to leucocratic, medium- to coarse-grained, equigranular granite (sample TRG-2; 22°32′34″N, 86°04′52″E) corresponding to the latest phase (phase-III) of the Singhbhum Granite (SG-III26; Supplementary Figure SF2), intrudes both the metabasalt-fuchsite quartzite-chert association (Supplementary Figure SF3A) and the amphibole schist of the EIOG greenstone belt, suggesting that it is younger than both the granite gneiss (TRBG-I) and the EIOG.

Results

Description of zircons and U-Pb data. The 207Pb/206Pb age data of 85 points from 23 zircon grains were obtained from four samples of the Older Metamorphic Tonalitic Gneiss (OMTG; RM-1 & 5), Singhbhum Granite Phase-I (SG-I; sample TRBG-1) and Singhbhum Granite Phase-III (SG-III; sample TRG-2); these data are presented in Table 1 and Supplementary Table S1. Two zircon grains from RM-1 (grain #9) and RM-5 (grain #11) exhibit significantly older ages (Hadean) than the rest of the analyzed grains (Archean to Paleoproterozoic) in this study. Grain #9 is subhedral and displays oscillatory zoning in the cathodoluminescence (CL) image (Fig. 2A); it yields three analyses with 207Pb/206Pb concordant ages of 4031 ± 5, 4036 ± 15, and 4057 ± 8 Ma (Table 1; Supplementary Table S1). A second, relatively smaller, subhedral grain (grain #11) shows a homogenous core in its CL image and yields two concordant ages of 4241 ± 4 and 4239 ± 4 Ma (Table 1), while its rim shows thin oscillatory zoning (Fig. 2D) and yields discordant ages of ~3.8–3.9 Ga (Supplementary Table S1). The differences in age and Hf isotopic compositions between these two grains and the rest of the zircon population, combined with their subhedral grain shapes, indicate that these Hadean zircons are inherited in origin39. The oscillatory zoning and higher Th/U ratios (0.44–0.65) of the Hadean zircons suggest an igneous origin40, although exceptions can occur41. Three analyses from the oscillatory zoned rim of an old xenocrystic core with an age of 4241 ± 4 Ga (grain #11) from sample RM-5 yield >10% discordance, thus reflecting Pb loss, which implies that this rim is probably older than ~3.8–3.9 Ga. In sample RM-5, grain #2 exhibits an inherited core with a concordant age of 3670 ± 7 Ma, which is homogenous in its CL image (Fig. 2C) and is surrounded by an oscillatory zoned growth rim. Another xenocryst from the same sample (grain #9), which has resorbed grain boundaries and broad, faint zoning visible in CL image, yields a concordant age of 3673 ± 7 Ma (Fig. 2C,F). Another older age spot in zircon from sample RM-5 (19.1) yields a concordant age of 3595 ± 12 Ma. The U-Pb analyses of the RM-1 and RM-5 zircons yield 207Pb/206Pb age data that define linear arrays, yielding concordia intercepts at ages of 3393 ± 9 Ma (MSWD = 1.7; n = 3) and 3399 ± 6 Ma (MSWD = 1.6; n = 3), respectively (Fig. 2E,F). Most of the dated zircons exhibit regular oscillatory zoning from core to rim (Fig. 2A–D). Some grains exhibit homogenous cores surrounded by growth-zoned rims (Fig. 2B; grain #23; sample RM-1) but yield a consistent age of ~3.4 Ga (spot 23.1; Table 1). The lower intercept ages of RM-1 and RM-5 are ~900 and ~1200 Ma, respectively which broadly coincide with a ~1.2–1.0 Ga magmatic event related to the late phase of regional dyke swarm emplacement known as the ‘Newer Dolerite Dykes’42. Zircons from samples RM-1 and 5 are euhedral to subhedral, and the presence of irregular boundaries in some grains can be attributed to solid-state recrystallization43. The ~4.0 Ga zircon grain #3 contains K-feldspar, apatite and titane inclusions (Supplementary Figure SF3C), but another Hadean grain
rim of the ~4.2 Ga xenocryst (grain #11) with a discordant 207Pb/206Pb age of ~3.86 Ga yields an unusual

denote initial isotopic ratios calculated using 207Pb/206Pb ages (Ma) of respective spots in zircons. 207Pb/206Pb (4241

Table S2. The Hf isotopic analysis of one spot obtained from the oldest Hadean xenocryst (4241

geneous rims that are visible in CL images (Fig. 3A–D). Zircons from TRBG-1 yield a 207Pb/206Pb upper intercept (0.27995

Hf isotopic compositions of OMTG and SG zircons.

mary43 although exceptions occur47.

Table 1. Selected SHRIMP U-Pb and Lu-Hf isotopic data for samples RM-1 and 5 (OMTG). *176Hf/177Hf (t) values), whereas all data are presented in Supplementary dataset SF1 and 2.

The 176Hf/177Hf compositions of Hadean xenocrysts (≥4.0 Ga) and their host Paleoarchean zircons are summarized in Table 1 and presented in full in Supplementary Table S1), and the core appears to be much brighter than the rest of the grain in the CL image (Fig. 3C).

Hf isotopic compositions of OMTG and SG zircons. The 176Hf/177Hf compositions of Hadean xenocrysts (>4.0 Ga) and their host Paleoarchean zircons are summarized in Table 1 and presented in full in Supplementary Table S2. The Hf isotopic analysis of one spot obtained from the oldest Hadean xenocryst (4241 ± 7 Ma; spot 5-11-1) yields a subchondritic* eHf[t] value of −2.5 ± 1.6 and is similar to the other two younger Hadean xenocrysts of 4031 ± 5 Ma and 4036 ± 15 Ma, which yield eHf[t] values of −4.1 ± 1.3 and −5.2 ± 1.3, respectively (Table 1). The rim of the ~4.2 Ga xenocryst (grain #11) with a discordant207Pb/206Pb age of ~3.86 Ga yields an unusual eHf[t] value of −11.9, which probably due to the underestimated age assignment, considering its discordance. However, the initial 176Hf/177Hf value of this rim (0.27994 ± 0.00008) is remarkably close to that of the Hadean core (0.27995 ± 0.00009) (Supplementary Table S2) indicating same source. Hence, calculating the eHf[t] value of this spot with its upper intercept age of 4.24 Ga as a proxy, yields a value of the ~3.86 Ga rim that is more consistent (−3.2 ± 1.5) with those of the other Hadean zircons. The initial 176Hf/177Hf value of the oldest Hadean xenocryst (4241 ± 4 Ma) in the OMTG is the least radiogenic (0.27995 ± 0.00009; Table 1). Two younger xenocrysts with

| Sample | 207Pb/206Pb age (Ma) | Discordance (%) | Th/U | 176Hf/177Hf (t) | ± error (2 σ) | eHf[t] | ± error (2 σ) |
|--------|----------------------|----------------|------|----------------|---------------|--------|---------------|
| RM-1-3-1 | 4031 ± 5             | 0              | 0.44 | 0.28005        | 0.00007       | −4.1   | 1.3           |
| RM-1-3-2 | 4036 ± 15            | 1              | 0.55 | 0.28001        | 0.00007       | −5.2   | 1.3           |
| RM-1-3-3 | 4010 ± 6             | 12             | 0.44 | —              | —             | —      | —             |
| RM-1-3-4 | 4057 ± 8             | 4              | 0.62 | —              | —             | —      | —             |
| RM-1-5-1 | 3368 ± 12            | 11             | 0.44 | 0.28055        | 0.00007       | −1.8   | 1.3           |
| RM-1-7-1 | 3364 ± 7             | 4              | 1.29 | 0.28049        | 0.00007       | −4.3   | 1.3           |
| RM-1-14-1 | 3402 ± 8             | −1             | 1.10 | 0.28055        | 0.00008       | −1.3   | 1.4           |
| RM-1-1-5 | 3330 ± 23            | 8              | 1.78 | 0.28053        | 0.00008       | −3.6   | 1.3           |
| RM-1-19-1 | 3362 ± 10            | 8              | 1.98 | 0.28047        | 0.00008       | −4.9   | 1.5           |
| RM-1-21-1 | 3365 ± 8             | 12             | 0.47 | 0.28059        | 0.00007       | −0.8   | 1.3           |
| RM-1-22-1 | 3385 ± 6             | 2              | 1.45 | 0.28052        | 0.00007       | −2.8   | 1.3           |
| RM-1-23-1 | 3400 ± 10            | 2              | 2.03 | 0.28049        | 0.00008       | −3.2   | 1.4           |
| RM-5-1-1 | 3396 ± 8             | −3             | 1.84 | 0.28037        | 0.00007       | −0.4   | 1.2           |
| RM-5-2-1 | 3670 ± 7             | 5              | 0.51 | 0.28022        | 0.00006       | −6.6   | 1.1           |
| RM-5-3-1 | 3363 ± 11            | 0              | 1.38 | 0.28054        | 0.00007       | −2.5   | 1.3           |
| RM-5-5-1 | 3341 ± 5             | 10             | 0.76 | 0.28053        | 0.00008       | −3.4   | 1.5           |
| RM-5-6-1 | 3415 ± 7             | −1             | 0.26 | 0.28047        | 0.00010       | −3.7   | 1.8           |
| RM-5-8-1 | 3386 ± 8             | 15             | 1.00 | 0.28054        | 0.00007       | −1.9   | 1.3           |
| RM-5-9-1 | 3673 ± 7             | 0              | 0.57 | 0.28027        | 0.00008       | −4.7   | 1.4           |
| RM-5-10-1 | 3396 ± 6             | −1             | 1.02 | 0.28053        | 0.00008       | −2.0   | 1.4           |
| RM-5-11-1 | 4241 ± 4             | −1             | 0.65 | 0.27995        | 0.00009       | −2.5   | 1.6           |
| RM-5-11-3 | 4239 ± 4             | 0              | 0.65 | —              | —             | —      | —             |
| RM-5-12-1 | 3390 ± 11            | −1             | 1.60 | 0.28056        | 0.00007       | −1.0   | 1.2           |
| RM-5-14-1 | 3433 ± 6             | −1             | 0.97 | 0.28051        | 0.00008       | −1.9   | 1.4           |
| RM-5-15-1 | 3394 ± 8             | 0              | 1.56 | 0.28054        | 0.00006       | −1.8   | 1.0           |
| RM-5-16-1 | 3381 ± 14            | −1             | 1.52 | 0.28054        | 0.00006       | −1.9   | 1.1           |
| RM-5-18-1 | 3394 ± 7             | 2              | 1.13 | 0.28051        | 0.00007       | −2.8   | 1.3           |
| RM-5-19-1 | 3595 ± 12            | 5              | 0.69 | 0.28041        | 0.00007       | −1.5   | 1.3           |
ages of 4031 ± 5 and 4036 ± 15 Ma yield slightly more radiogenic, but altogether subchondritic, initial $^{176}\text{Hf}/^{177}\text{Hf}$ values of 0.28005 ± 0.00007 and 0.28001 ± 0.00007, respectively, which are identical within error.

On the $\varepsilon\text{Hf}[t]$ vs $^{207}\text{Pb}^{206}\text{Pb}$ age (Ma) diagram (Fig. 4), the pre-4 Ga xenocrysts of the OMTG follow an array with a slope of 0.0103, corresponding to a source $^{176}\text{Lu}/^{177}\text{Hf}$ ratio of 0.019 (calculated after Amelin et al.19), which intersects the chondritic uniform reservoir (CHUR) line at 4.497 ± 0.19 Ga (Fig. 4). The source Lu/Hf ratio calculated from the Hadean zircons, although slightly lower, is consistent with the source being typical mafic crust; that ranges from 0.2219 to 0.2021 and is far higher than that of the average TTG crust (0.01) calculated from the oldest

![Figure 2. (A,B) Cathodoluminiscence (CL) image of zircons from OMTG sample RM-1, (C,D) from sample RM-5. Circles represent analyses spot positions with spot numbers and their ages in Ga. Grain numbers are shown beside each grain (e.g. #1). (E,F) U-Pb concordia plots for the zircons from samples RM-1 and RM-5 respectively (ages represent weighted mean of ages in Ma). Separate discordia lines were fitted through different age groups of zircons. Error ellipses are shown at 1σ.](image-url)
The intersection age (4.497 ± 0.19 Ga) of this array with the CHUR reference line is closer to the CHUR extraction age of 4.46 ± 0.12 Ga as the source reservoir of the Jack Hill zircons.

The Eoarchean domains in sample RM-5 with concordant ages of 3673 ± 7 Ma and 3670 ± 7 Ma, yield εHf(t) values of −4.7 ± 1.4 and −6.6 ± 1.1, respectively. Their initial Hf compositions at 3.67 Ga are near identical, e.g., 0.28027 ± 0.00008 and 0.28022 ± 0.00006 respectively, and they are notably higher than those of the Hadean xenocrysts (0.279947–0.280045). However, the oldest Paleoarchean xenocryst, which has a concordant age of 3595 ± 12 Ma, yields an εHf(t) value that is closer to a chondritic value (−1.5 ± 1.3) and a 176Hf/177Hf initial value.

Figure 3. (A,B) Cathodoluminiscence (CL) image of zircons from Singhbhum granite sample TRBG-1 (SG-I), (C,D) from sample TRG-2 (SG-III). (E,F) U-Pb Concordia plots for the zircons from TRBG-1 and TRG-2 respectively (ages represent weighted mean of ages in Ma). Error ellipses are shown at 1σ.

Jack Hill zircons. The intersection age (4.497 ± 0.19 Ga) of this array with the CHUR reference line is closer to the CHUR extraction age of 4.46 ± 0.12 Ga as the source reservoir of the Jack Hill zircons.
A TRBG-1 analytical site with an age of 3361 ± 6 Ma yields an initial Hf composition of 0.28068 ± 0.00007, and a $\varepsilon_{\text{Hf}}(t)$ value of +2.6 ± 1.4 (spot 3.1), which is the only superchondritic value among our entire dataset.

Table 2. Selected SHRIMP U-Pb and Lu-Hf isotopic data for samples TRBG-1 (SG-I) and TRG-2 (SG-III).

| Sample          | $^{207}$Pb/$^{206}$Pb age (Ma) | Discordance (%) | Th/U | $^{176}$Hf/$^{177}$Hf (t) | ± error (2σ) | $\varepsilon_{\text{Hf}}(t)$ | ± error (2σ) |
|----------------|---------------------------------|-----------------|------|--------------------------|--------------|-----------------------------|--------------|
| TRBG-1-1-1      | 3352 ± 6                        | 8               | 0.76 | 0.28060                  | 0.00007      | −0.7                        | 1.3          |
| TRBG-1-3-1      | 3361 ± 7                        | −1              | 0.71 | 0.28068                  | 0.00008      | 2.6                         | 1.4          |
| TRBG-1-6-1      | 3248 ± 12                       | 0               | 0.29 | 0.28060                  | 0.00008      | −3.1                       | 1.4          |
| TRBG-1-8-1      | 3267 ± 6                        | 10              | 0.66 | 0.28061                  | 0.00007      | −2.0                        | 1.3          |
| TRBG-1-9-1      | 3340 ± 6                        | 8               | 0.63 | 0.28051                  | 0.00007      | −4.0                        | 1.2          |
| TRBG-1-10-1     | 3404 ± 7                        | 2               | 0.67 | 0.28045                  | 0.00007      | −4.7                        | 1.3          |
| TRBG-1-13-1     | 3289 ± 10                       | 5               | 1.13 | 0.28056                  | 0.00007      | −3.6                        | 1.3          |
| TRG-2-1-1       | 3377 ± 11                       | 1               | 0.25 | 0.28058                  | 0.00006      | −0.6                        | 1.1          |
| TRG-2-3-1       | 3246 ± 6                        | 6               | 0.77 | 0.28063                  | 0.00006      | −2.1                        | 1.2          |
| TRG-2-4-1       | 3287 ± 6                        | 0               | 0.61 | 0.28060                  | 0.00006      | −2.2                        | 1.1          |
| TRG-2-5-1       | 3285 ± 6                        | 0               | 0.68 | 0.28061                  | 0.00006      | −2.7                        | 1.1          |
| TRG-2-6-1       | 3278 ± 7                        | 1               | 0.99 | 0.28060                  | 0.00006      | −2.5                        | 1.1          |
| TRG-2-7-1       | 3258 ± 7                        | 8               | 0.55 | 0.28061                  | 0.00007      | −2.6                        | 1.2          |
| TRG-2-9-1       | 3291 ± 7                        | 1               | 0.58 | 0.28060                  | 0.00009      | −1.9                        | 1.6          |
| TRG-2-10-1      | 3367 ± 6                        | 7               | 0.63 | 0.28057                  | 0.00007      | −0.3                        | 1.3          |
| TRG-2-14-1      | 3288 ± 7                        | 1               | 0.55 | 0.28057                  | 0.00007      | −3.1                        | 1.2          |
| TRG-2-16-1      | 3264 ± 7                        | 3               | 0.7  | 0.28060                  | 0.00007      | −2.7                        | 1.2          |

Figure 4. $\varepsilon_{\text{Hf}}(t)$ vs. $^{207}$Pb/$^{206}$Pb age (Ma) plot of zircons from samples RM-1, RM-5, TRBG-1 and TRG-2. Data from contemporary Singhbhum TTGs (~3.5–3.3 Ga) are from Dey et al. Note all data from present study are subchondritic and Hadean and Eoarchean xenocrysts follow $^{176}$Lu/$^{177}$Hf = −0.019 array. The isotope trajectory of UCC (Upper continental Crust; $^{176}$Lu/$^{177}$Hf = 0.008) is after Rudnick and Gao.

(0.28041 ± 0.00007) that is higher than those of the older Hadean and Eoarchean age spots. The ~3.3–3.4 Ga age group of zircons from samples RM-1 and RM-5 yields more radiogenic $\varepsilon_{\text{Hf}}(t)$ values than Hadean and Eoarchean age spots, ranging from −0.4 ± 1.2 to −3.7 ± 1.8, except for two data points that fall below −4 epsilon units (−4.9 ± 1.5 and −4.3 ± 1.3). Initial Hf ratios of these spots are identical to those of other age spots with lower $\varepsilon_{\text{Hf}}(t)$ values, implying that they were derived from the same source. Initial $^{176}$Hf/$^{177}$Hf values of the ~3.3–3.4 Ga zircons display a relatively small range of values, varying between 0.28047–0.28057, similar to the average of 0.28053 ± 0.00003. This range also includes $^{176}$Hf/$^{177}$Hf initial values of four discordant age spots (10–15% discordance; Table 1), indicating that despite having undergone U-Pb resetting, their Hf isotopic ratios remain unchanged. The ~3.3–3.4 Ga zircons do not exhibit any particular trend in $\varepsilon_{\text{Hf}}(t)$-time space, but they cluster within the field delimited by the $^{176}$Lu/$^{177}$Hf = 0.019 array defined by the Hadean zircon data and the CHUR reference line ($\varepsilon_{\text{Hf}} = 0$; Fig. 4). This indicates, unlike Zack Hill zircons, the younger zircons of OMTG are not derived from the same source as the oldest crust.

The $\varepsilon_{\text{Hf}}(t)$ values of the 3397 ± 7 Ma zircons from sample TRBG-1 (SG-I) and 3286 ± 6 Ma zircons from sample TRG-2 (SG-III) are all subchondritic, yielding $\varepsilon_{\text{Hf}}(t)$ values ranging from −0.7 ± 1.3 to −4.7 ± 1.3 and from −0.5 ± 1.1 to −3.1 ± 1.2, respectively (Table 2), similar to the 3.3–3.4 Ga age group of the OMTG zircons. However, a TRBG-1 analytical site with an age of 3361 ± 7 Ma yields an $\varepsilon_{\text{Hf}}(t)$ value of +2.6 ± 1.4 (spot 3.1), which is the only superchondritic value among our entire dataset. The oldest age spot (3404 ± 7 Ma) of the TRBG-1 zircons yields an initial Hf ratio of 0.28045 ± 0.00007 and an $\varepsilon_{\text{Hf}}(t)$ value of −4.7 ± 1.3. Likewise, the site with an age of 3361 ± 7 Ma yields the highest radiogenic initial Hf composition of 0.28068 ± 0.00008 among
the entire zircon population. Apart from these, 6 spots (five with <10% and one with 11% discordance) with ages ranging from 3248 to 3352 Ma yield \(^{176}\text{Hf}/^{177}\text{Hf}\) values that overlap (within uncertainty) the average value (0.28057 ± 0.00008; Table 1). The initial \(^{176}\text{Hf}/^{177}\text{Hf}\) ratios of the sites of the TRG zircons with ages of 3377 to 3246 Ma are identical to their average value of 0.28060 ± 0.00003, within analytical error (Table 2). The average initial \(^{176}\text{Hf}/^{177}\text{Hf}\) ratios of TRBG-1 (0.28057 ± 0.00008) and TRG-2 (0.28060 ± 0.00003) are also comparable Table-1.

Discussion

Interestingly, the subchondritic Hf composition (\(\varepsilon\text{Hf}[t] < 0\)) of the oldest (4241 ± 4 Ma) xenocryst indicates the presence of a non-chondritic mantle reservoir as early as ~4.2 Ga. The composition of the earliest mantle reservoirs of Earth has remained controversial. The reported initial \(^{176}\text{Hf}/^{177}\text{Hf}\) ratios of the Bulk Silicate Earth (BSE), i.e., 0.279685 ± 0.0184 or 0.279781 ± 0.1833, are lower than that of the chondritic reservoir, argue against the decades-old paradigm of the chondritic Earth and are explained by the accelerated decay of \(^{176}\text{Lu}\) [56,57]. However, such accelerated decay is caused by the high rate of irradiation of chondritic or eucritic meteorites by \(\gamma\) and/or galactic cosmic rays, a process whose effectiveness has been questioned for the BSE due to the restricted penetration depth of these rays [58,59]. Alternatively, it has been assumed that the Earth was developed from chondritic material but was subsequently modified by either collisional erosion during accretion [50,51] or explosive basaltic volcanism in planetesimals [60]. Hence, we assume that the source reservoir of the Hadean OMG xenocrysts was initially separated from chondritic material, and we interpret our zircon data considering CHUR [48] as a reference frame. The source array of \(\text{Lu}/\text{Hf}\) initial composition of these xenocrysts is comparable to 'mafic protocrust' with \(^{176}\text{Lu}/^{177}\text{Hf}\) values proposed by Kemp et al. [50] (0.020) and Amelin et al. [51] (0.022) calculated from Jack Hill detrital zircon data. The age of separation (~4.5 Ga) of the enriched reservoir from the chondritic reservoir calculated from the Hadean OMG xenocrysts in this study is also very similar to the CHUR separation age of the source reservoir of the Jack Hill zircons (~4.5 Ga [61] or 4.46 ± 0.12 Ga [62]). The development of the ~4.9 Ga enriched reservoir recorded in the Singhbhum craton is also in near-agreement with the estimated age of ~4.5 Ga for the separation of the enriched silicate reservoir upon Earth's solidification, based on recent geodynamic modeling [57–59].

Assuming that the parental magmas of these zircons is likely to be felsic due to the high solubility of zirconium in mafic-ultramafic magmas [63], the Hadean (~4.2-4 Ga) zircons of the OMG were presumably generated from minor silicic melts produced as a consequence of the differentiation or re-melting of pre-4.2 Ga juvenile protocrust of mafic composition. The formation and reworking of juvenile crust were either contemporaneous or separated by a short period of ~100-300 My during the Hadean and Archean epochs [64]. To explain the nature of the enriched mantle reservoir that parented the Hadean OMG zircons, we envisage that such a reservoir may represent an enriched, residual mafic magma generated from a partially solidified magma ocean, analogous to KREEP beneath the lunar anorthositic crust [65,66–68]. This mafic protocrust was presumably reworked and re-melted to generate felsic melt between ~4.2-4.0 Ga without the significant addition of juvenile material from the mantle. The mineral inclusions in the Hadean OMG xenocrysts, including K-feldspar, titanite and apatite (Supplementary Figure SF3C), were likely generated from a differentiated melt. The existence of Hadean mafic protocrust has previously been estimated based on Hadean to Paleoarchean Jack Hill's xenocrysts [31–33], ~3.7 Ga metasediments from Isua [69] and detrital zircons from the Pilbara [70]. Similarly, the source \(^{176}\text{Lu}/^{177}\text{Hf}\) (0.2805) calculated from the OMG validates that a mafic-dominated crust prevailed in the Singhbhum Craton during the Hadean, negating the possibility of the significant presence of upper continental \(^{176}\text{Lu}/^{177}\text{Hf} = 0.0084\) or "TTG-like" crust \(^{176}\text{Lu}/^{177}\text{Hf} = 0.0190\).

Thus, it is necessary to determine the fate of this ancient, enriched Hadean reservoir in the Singhbhum Craton. The Paleoarchean (~3.6 Ga) zircon age domains in sample RM-5 record the second-oldest stage of felsic melt generation; these are slightly more radiogenic than the Hadean ones. Therefore, they were probably generated from the modification of the enriched source of Hadean zircons due to its interactions with juvenile (more radiogenic) mantle melt, as is evidenced by the fact that the \(\varepsilon\text{Hf}[t]\) and \(^{176}\text{Hf}/^{177}\text{Hf}\) initial compositions of these sites (Table 1) are higher than those of the Hadean ones. This also indicates the assumption that the composition of the enriched, subchondritic mantle reservoir in the Singhbhum Craton persisted without undergoing modification until the Paleoarchean (~3.7 Ga). However, the identifiable vertical excursion of \(\varepsilon\text{Hf}[t]\) values in the time-integrated \(\varepsilon\text{Hf}[t]\) plot (Fig. 4) in the Paleoarchean (3.3-3.4 Ga) for OMG zircons and other contemporary Singhbhum TTGs, such as TRBG-1 (~3.4 Ga) and TRG-2 (~3.3 Ga), to near-chondritic values confirms the variable mixing of mantle-derived, juvenile material with material from the old, enriched reservoir during the period between 3.4 and 3.3 Ga. However, the average \(^{176}\text{Hf}/^{177}\text{Hf}\) initial values of the 3330-3433 Ma zircons in samples RM-1 & RM-5 (OMTG), the 3267-3352 Ma zircons in samples TRBG (SG-I) and the 3377-3246 Ma zircons in samples TRG-2 (SG-III) are 0.28053 ± 0.00006, 0.28057 ± 0.00008 and 0.28060 ± 0.00003, respectively. These values are closely comparable except for an age of 3404 Ma with a slightly less radiogenic \(^{176}\text{Hf}/^{177}\text{Hf}\) ratio of 0.28041. Clearly, these Paleoarchean zircons were derived from felsic melts with near identical Hf isotopic values, while minor disparity is most likely due to incomplete mixing between the enriched reservoir with depleted juvenile magma. The results of a previous petrological modeling study [67] suggested that the protolith of the OMG was generated by the 40% partial melting of the OMG amphibolites at garnet stability depths [68]. It is unlikely that the remnants of the earliest Hadean mafic protocrust survived the constant reworking processes until today. Remnants of the oldest mafic protocrust may have been preserved in the amphibolite enclaves within the OMG or these enclaves could represent a modified mafic component developed from interactions between the ancient enriched reservoir and mantle-derived, juvenile mafic magma and was preserved as melting residue of the mafic protolith from which OMG magma was generated. Interestingly, the zircons from the ~3.5 to ~3.3 Ga TTGs of the Singhbhum Craton, which are located near Keonjhar, exhibit suprachondritic Hf isotopic signatures with average \(\varepsilon\text{Hf}[t]\) values ranging from +2.9 to +2.2 [68], suggesting that they were derived from a depleted source reservoir. However, this implies that a separate depleted reservoir, which was probably complementary with the
ancient, enriched reservoir hypothesized in the present study, of Paleoarchean (~3.5 Ga) or even older age, existed under the cratonic lithosphere of the Singhbhum craton and also participated in the generation of TTG magma.

Based on isotopic constraints, it has been suggested that Earth’s accretion was roughly complete 30 Myr after the condensation of the oldest solids from the solar nebula at 4568.2 ± 0.2 Ma. Hence, the separation of the enriched reservoir (4.497 ± 0.19 Ga) in the Singhbhum Craton occurred soon after (~40 My) the accretion of the Earth. Evidence from short-lived isotopes, e.g., $^{146}$Sm–$^{142}$Nd or $^{182}$Hf–$^{182}$W, and long-lived isotopic systems (176Lu–177Hf) suggest that the development of enriched and depleted reservoirs occurred very early in Earth’s history, probably within 100–200 Ma of planetary accretion, and that complementary enriched (Early Enriched Reservoir, or EER) and depleted reservoirs (Early Depleted Reservoir, or EDR) existed during the Hadean. The nature and fate of this EER remains elusive, although its presence has been deduced from the ~3.4 Ga Ameralik dykes of the Amitsoq complex. The highly unradiogenic, Hadean zircons of the OMTG most likely represent product of the EER that existed in the Singhbhum cratonic mantle at ~4.2–4.4 Ga. The presence of ~3.3–3.4 Ga TTG zircons (OMTG and SG) with unradiogenic Hf signals in this study indicates that this enriched reservoir was sustained until the Paleoarchean. Therefore, the development of the EER from the chondritic mantle at ~4.5 Ga raises possible questions about the existence of the complementary depleted mantle. Evidence of depleted mantle under the Singhbhum Craton during the Paleoarchean has already been recorded in ~3.5–3.4 Ga zircon with radiogenic Hf isotope signatures from the TTGs of the Singhbhum Craton.

Is it possible to back-track the oldest depleted Hadean reservoir that is complementary to the enriched one recorded in the Hadean zircons in this study? The most plausible candidate might be the Paleoarchean komatiites (~3.4 Ga) of the Eastern IOG belt, which are the most direct representatives of the Paleoarchean depleted lower mantle (~Nd(t) = 2 to +4) below the Singhbhum Craton. These komatiites may have separated from much older Hadean mantle and may have recorded evidence of early silicate differentiation, i.e., by preserving a record of the Hadean EDR in the highly radiogenic (~Hf(t) = up to +8.2) Paleoarchean (~3.5 Ga) Pilbara komatiites.

U–Pb age constraints clearly indicate that the large-scale generation of continental crust of TTG composition started at ~3.4–3.3 Ga in the Singhbhum Craton. Felsic rocks generated before this time have probably now been completely reworked and recycled, as zircons older than this are only found as xenocrysts here and in earlier studies. A xenocryst with an age of ~3.38 Ga is found (grain #1; spot 1.1; Fig. 3C) to be surrounded by an oscillatory zoned zircon rim with an age of ~3.3 Ga (grain #1; spot 1.2) in sample TRG-2. The site where sample TRG-2 was collected exhibits ubiquitous enclaves of material similar to TRBG-1 (Supplementary Figure SF3C), which suggests that this ~3.38 Ga xenocrystal may have been inherited from SG-I, as it was reworked during the emplacement of the younger SG-III. The oldest concordant age of sample TRG-2 (~3.29 Ga; grain #20, 23) is equivalent to the 3289 ± 10 Ma age spot (spot 13.1), which indicates that probably this age of the tectonomagmatic event that led to reworking of pre-existing SG-I and emplacement of SG-III.

Before the emergence of dominantly TTG crust, mafic protocrust likely prevailed as a thin, buoyant tectonic plate. It is still unclear whether such proto-plates were stagnant, as the heat production of the Earth’s mantle was more than three times greater during the Archean, which led to more rapid mantle convection, thus triggering faster plate movement. Rapid plate movement invokes the possibility of the quick recycling of thin Hadean proto-crust, thus preventing its preservation. Hence, it is possible that the Hadean mafic protocrust in the Singhbhum craton may not have survived long and was recycled and assimilated into more voluminous TTG magma that was generated from a combined process involving the reworking of older, enriched crust and the serial addition of mantle-derived melt during ~3.4–3.3 Ga. The tectonic processes involved in the partial melting of the OGG amphibolites to generate the partial magma of the OMTG are still unclear. However, the geochemical data of the ~3.5–3.3 Ga TTGs of the Singhbhum craton suggest that these TTGs lack the signatures of subduction-derived magma; they are thus considered to have been generated from the reworking of pre-existing mafic crust by the repeated underplating of plume-derived mafic–ultramafic magma during the Paleoarchean.

Conclusions
The combined U–Pb SHRIMP and Lu–Hf isotopic data of ~4.24 and ~4.03 Ga xenocrystic zircons from the ~3.4 Ga TTG of the ‘Older Metamorphic Tonalitic Gneiss (OMTG)’ of the Archean Singhbhum Craton of Eastern India contain records of the oldest crust in India. The essentially subchondritic (~Hf(t) < 0) isotopic signatures of these Hadean zircons indicate that they originated from the reworking of older crust prior to ~4.2 Ga. The calculated $^{176}$Lu/$^{177}$Hf ratio (0.019) of their source reservoir indicates the mafic nature of the older crust that originated from an enriched reservoir that separated from the chondritic reservoir at ~4.5 Ga. However, the younger and almost contemporaneous zircons from the OMTG (3.3–3.4 Ga), Singhbhum Granite Phase-I (SG-I; ~3.4 Ga) and Singhbhum Granite-III (SG-III; ~3.3 Ga) yield more radiogenic Hf isotopic signatures, indicating that this enriched reservoir persisted but underwent mixing with juvenile mantle material during the Eo-Paleoarchean.

Methods
Zircon separation, CL imaging, inclusion analysis and SHRIMP U–Pb dating were carried out at the Beijing SHRIMP Center, Institute of Geology, Chinese Academy of Geological Sciences, using a SHRIMP II following the analytical procedures described by Williams. Zircon crystals were obtained using standard crushing and grinding techniques, followed by separation using heavy liquid and magnetic techniques. The hand-picked crystals were cast in epoxy resin discs and polished. The intensity of the primary O$_2$ ion beam was 5 nA and the spot size was 25–30 μm; each site was rastered for 150 s prior to analysis. Five scans through the mass stations were made for each age determination. The standard used for the calibration of elemental abundances was M257, which contains U = 840 ppm. TEMORA, whose $^{206}$Pb/$^{208}$U age is 417 Ma, was analyzed for the calibration of the $^{206}$Pb/$^{208}$U ratios after every 3 analyses. Detailed CL images of these zircons were captured. All grains were imaged using a CARL-ZEISS MERLIN Compact with GATAN Mono CL4, and the inclusions within Hadean zircons
were analyzed using the same scanning electron microscope with OXFORD IE250. The data were processed and assessed using the Squid 1.0296 and Isoplot 3.0086 programs. Common Pb corrections were based on the measured 206Pb contents. The errors given in Table 1 and the concordia intercept ages for individual analyses are quoted at the 1σ level, whereas the errors for weighted mean ages in the text are quoted at the 95% confidence level.

The in situ Lu–Hf analyses of zircons from all four TTG samples were conducted on the pits generated during U–Pb dating at the State Key Laboratory of Lithospheric Evolution, Institute of Geology and Geophysics, Chinese Academy of Sciences, Beijing, using a 193 nm ArF excimer laser ablation system attached to a Neptune multi-collector ICP MS. The instrumental conditions and analytical procedures were described by Wu et al.91. Each measurement included an ablation time of ~26 s for 200 cycles, a repetition rate of 6–8 Hz, a laser power of 100 mJ/pulse and a spot size with a diameter of 44 μm. Helium was used as the carrier gas for the ablated aerosols. The average 176Hf/177Hf ratios of the Mud Tank92 and Plešovice93 standards obtained in this study after repetitive analyses were 0.282500 (n = 25) and 0.282484 (n = 18), respectively. All Lu–Hf isotopic results are reported with 95% confidence limits.

The calculation of εHf(t) values was based on the 207Pb/206Pb SHRIMP spot analysis ages, chondritic values (176Hf/177Hf = 0.282785, 176Lu/177Hf = 0.033648) and a 176Lu decay constant of 1.865 × 10−11 year−194. Selected 206Pb/207Pb concordant and some discordant (10–15% discordant) age data of zircon spots with Hf isotope values consistent with concordant ones are summarized in Tables 1 and 2. All U–Pb age data and Lu–Hf isotopic data are listed in the Supplementary Material SF1 and 2. During interpretation, zircon 207Pb/206Pb age data with >10% U–Pb discordance and Th/U ratios of <0.15 were commonly disregarded. However, some discordant data, such as those with 176Hf/177Hf ratios identical to those of the concordant population, were included because although their U–Pb ratios have been modified, their original Lu–Hf isotopic ratios were preserved.

**Availability of materials and data.** All data generated or analysed during this study are included in this published article and its Supplementary Tables (SF1 and 2).

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Author Contributions
Trisrota Chaudhuri and Rajat Mazumder conducted fieldwork and prepared the manuscript. Yusheng Wan conducted U-Pb SHRIMP and Lu-Hf isotope analyses and provided CL images and U-Pb Concordia diagrams (Figs 2 and 3) of zircons. Trisrota Chaudhuri prepared Figs 1 and 4 and Supplementary Figures SF1 and 4 using Corel Draw and OriginLab. Mingzhu Ma and Dunyi Liu assisted in U-Pb SHRIMP zircon analyses. All the authors reviewed the manuscript.

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