Reply to Comment on "Pervasive remagnetization of detrital zircon host rocks in the Jack Hills, Western Australia and implications for records of the early dynamo"

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Determining the history of Earth's dynamo prior to the oldest known well-preserved rock record is one of the ultimate challenges in the field of paleomagnetism. Tarduno et al. (2015) argued that detrital zircons contain records of an active dynamo dating back to 4.2 billion years ago (Ga), 700 million years earlier than previously identified (Biggin et al., 2011; Tarduno et al., 2010). However, this extraordinary claim requires evidence that the zircons have not been remagnetized during the intervening time since their formation. Weiss et al. (2015) argued that such evidence has yet to be provided, a conclusion that we find still firmly holds.

Although mineral thermometry by our group and others has shown that the Jack Hills zircons and their host rocks have not been heated above ~500°C since deposition at ~3 Ga (Rasmussen et al., 2011; Trail et al., 2016), they could have been aqueously remagnetized by alteration of original ferromagnetic minerals or neoformation of new ferromagnetic minerals in cracks and voids after this time. Moreover, the thermal and aqueous remagnetization histories of the zircons prior to deposition at 3 Ga are unknown. These uncertainties mean that the ages of magnetization in the zircons could be hundreds of millions of years younger than their crystallization ages.

Weiss et al. (2015) focused on whether rocks in the Jack Hills region, including those which host the oldest zircons, were remagnetized after deposition at ~3 Ga. They conducted a total of 12 baked contact, fold, and conglomerate tests on 277 specimens, which is nearly an order of magnitude larger than all other paleomagnetic studies of oriented Jack Hills samples to date combined (Cottrell et al., 2016; Tarduno and Cottrell, 2013; Tarduno et al., 2015). Weiss et al. (2015)'s tests yielded either negative results, indicating complete remagnetization in a north-down direction observed throughout the Jack Hills, or inconclusive results due to unstable magnetizations. These results are in stark contrast to two positive conglomerate tests reported by Tarduno and Cottrell (2013) and Tarduno et al. (2015). Another difference is that the dominant magnetization-carrying mineral observed by Weiss et al. (2015) was pyrrhotite (see below) while the latter two studies observed mainly magnetite.

Included in the Weiss et al. (2015) study were conglomerate tests on three individual block samples of pebble conglomerate (EHJH5, EHJH6, and EHJH7) from the Hadean zircon original discovery outcrop at Erawandoo Hill. Claims by Bono et al. (2016) and Cottrell et al. (2016) that this was not our sampling site are without foundation, as described in the Supplementary Material (SM). From each EHJH block, Weiss et al. (2015) analyzed mm to cm-sized subsamples of the

pebble-sized clasts and matrix taken from the decimeter-scale oriented parent block samples. They found that the dominant ferromagnetic mineralogy in most samples is likely pyrrhotite given the dominant unblocking temperature of ~330°C, the identification of the Besnus low-temperature magnetic transition, and quantitative microprobe measurements of Fe:S in sulfides (see SM). Note that by 330°C, many of the samples still had moments of 10^{-9} Am^2, which is ~1000 times above the sensitivity limit of the 2G Enterprises Superconducting Rock Magnetometer in the Massachusetts Institute of Technology (MIT) Paleomagnetism Laboratory [e.g., Fu et al. (2012)]; therefore, the observed lack of directional stability is due to near-total demagnetization of the stable remanence by this temperature rather than magnetometer noise.

Crucially, Bono et al. (2016) accept the central conclusion of Weiss et al. (2015) that the null hypothesis that the remanence directions of the clasts in each block are random can be rejected at the 95% confidence level, meaning they fail the conglomerate test[1]. This means that the clasts in the Hadean zircon-bearing rocks were remagnetized after deposition up to the maximum observed unblocking temperatures of 320-500°C [see Table S3 of Weiss et al. (2015)]. As such, this result does not provide evidence that the zircon magnetization predates deposition at 3 Ga, much less show that the zircon magnetization is a primary record dating back to 4.2 Ga.

The primary subject of the Comment by Bono et al. (2016) is constraining when after deposition at 3 Ga remagnetization of the rocks occurred. Because this is of not much consequence to determining the timing of the earliest dynamo, we recommend that readers not interested in the details of paleomagnetic analysis now skip ahead to the third-to-last paragraph. Bono et al. (2016) focus on the question of whether the three block means are too scattered for the remagnetization to be interpreted as originating from the emplacement of the Warakurna large igneous province (LIP). We agree that the use of a grand mean calculated from the mean directions of just three parent block samples has limited significance given the resulting large 95% confidence interval. Weiss et al. (2015) did not discuss this issue in detail because of its relative unimportance: the main point is that the conglomerate tests fail, which requires remagnetization after 3 Ga. The differences in the $N = 3$ block means and large confidence interval for their grand mean observed by Weiss et al. (2015) are not surprising given the very small N and the following expected error sources:

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[1]Following Cottrell et al. (2016), we instead might say they pass the "inverse conglomerate test."

(a)   multiple overlapping secondary and diachronous overprints, which are clearly visible from non-Fisherian streaking of EHJH6 specimen directions between the ubiquitous north-down direction and a southwest-down direction. Interestingly, the latter direction is very close to the characteristic magnetization direction identified by Cottrell et al. (2016) in the matrix of a nearby Erawandoo Hill sample (Fig. 1A), supporting the possibility of multiple remagnetization events. Cottrell et al. (2016) proposed that this southwest-down direction is related to a metamorphic event at 2.65 Ga. However, the direction also corresponds with the Cambrian portion of Australia's apparent polar wander path (Durocher et al., 2003; Klootwijk, 1980; Mitchell et al., 2010) such that it could be a remagnetization associated with the late stages of the Peterman-Paterson orogeny (Li and Evans, 2011).

(c)   poorly isolated characteristic magnetization components for some samples due to unstable behavior during laboratory demagnetization [i.e., Weiss et al. (2015) Fig. 9C];

(d)   block and specimen orientation errors, along with modest differential tilting of the three parent blocks over the last 1.1 billion years (which we estimate could collectively add differential rotations of up to 10°).

Although the mean directions of the three EHJH blocks are somewhat scattered (Fig. 1A, Table S2), they are oriented in approximately a similar direction as the north-down remagnetization directions observed elsewhere in the Jack Hills by Weiss et al. (2015) (Fig. 1B, Table S2). The grand mean of all of these remagnetization directions (from conglomerate test, baked contact, fold test and other host rock sites) is indistinguishable at 95% confidence from that of the ~1080 Ma Warakurna LIP in local coordinates (which we have recalculated using all available high-quality Warakurna sites from western Australia; Table S3) and indistinguishable at 95% confidence from the characteristic magnetization direction of a 1078.4 ± 4.4 Ma Jack Hills dike that was the subject of three baked contact tests by Weiss et al. (2015) (Figs. 1B and S1 and Table S2). It is the consideration of all these remagnetization directions together, which includes three different types of failed field tests, that led Weiss et al. (2015) to conclude that thermal or aqueous processing by the emplacement of the Warakurna LIP at ~1080 Ma was the most parsimonious source of the remagnetization in the region. Although Weiss et al. (2015) describe how remagnetization scenarios at different times also are conceivable, the evidence for a pervasive Warakurna LIP overprint in the region around the Hadean zircon outcrop is far from a "chimera"

and instead quite compelling. In any case, we remind the reader that when remagnetization of the host rocks occurred does not really matter: the main point is that remagnetization occurred after 3 Ga.

Bono et al. (2016) accuse Weiss et al. (2015) of "gross (90°) errors in orientation and measurement misorientation." We offer two strong pieces of evidence that refute this groundless assertion:

(i)   Weiss et al. (2015) obtained a Warakurna-like local magnetization direction from a dyke with a U-Pb emplacement age of ~1080 Ma as well as from numerous surrounding sites that included failed conglomerate, baked contact, and fold tests. In fact, based on the paleomagnetic direction we measured for the dyke, we predicted that it would have an age of 1.1-1.2 Ga before actually acquiring a U-Pb date for the dyke. These results demonstrate that we can accurately orient and measure paleomagnetic samples.

(ii)   Our failed conglomerate tests, which demonstrated nonrandom magnetization directions at each site, are statistically extremely unlikely to occur by chance if our samples had been misoriented (<1% for each of the EHJH5 and 6 tests, leading to a joint probability of $<10^{-4}$). In contrast, positive conglomerate tests like those reported by Tarduno and Cottrell (2013) and Tarduno et al. (2015) are the much more likely outcome when samples are misoriented. Given the ubiquity of north-down overprints in our samples, it remains puzzlingly that Tarduno and Cottrell (2013) did not observe such remanence directions in their conglomerate test samples. Note that the parent block for Tarduno et al. (2015)'s conglomerate test was an unoriented block sample (their Fig. S1), such that the absolute direction of overprints cannot be recovered.

Bono et al. (2016) state that our interpretation that the remagnetization of Erawandoo Hill likely resulted from the Warakurna LIP "sets an unfortunate precedent for the discipline." This hyperbole is ironic given that over the last several years, we have repeatedly requested the primary demagnetization data and samples for the Tarduno and Cottrell (2013) study from the U. Rochester group; however, they would not provide these data either to us nor even to the *EPSL* editors when requested as part of this Comment and Reply. By comparison, our demagnetization data are available as an online supplement to Weiss et al. (2015) and were analyzed by Bono et al. (2016) as part of their Comment. Even so, we sent some of the standard cm-sized cobble specimens measured by Weiss et al. (2015) to J. Tarduno and R. Cottrell in September, 2014. The exchange

of data and samples is critical for resolving the issues discussed in this Comment and Reply: to establish why Tarduno and Cottrell (2013) observed a positive cobble conglomerate test often with high fractional remanence remaining above 350ºC that is carried by magnetite (with no reported north-down overprint), while all of our conglomerate, fold, and baked contact tests either were negative (with pyrrhotite-dominated remagnetization in the north-down Warakurna LIP direction) or inconclusive. Access to the demagnetization data for Tarduno and Cottrell (2013) would allow us to assess the intensity, direction and thermal stability of magnetization overprints for the 20 out of their 28 samples whose demagnetization data do not appear in their manuscript Fig. 5 and 6. Even the latter vector-component figures are difficult to interpret because the divisions on the published axes are not numbered. Exchange of samples would enable a test whether the differences in results between the MIT and Rochester labs relates to differences in measurement techniques, sample lithologies and/or lightning remagnetization. Reproducibility tests like these form the basis of the scientific method.

We close this Reply by emphasizing what is the critical issue for establishing the existence of a Hadean dynamo from the Jack Hills zircons: determining whether or not the zircons were remagnetized *before* deposition at 3 Ga but still well after their formation at 4.4 Ga and later. Pre-depositional zircon remagnetization is a serious possibility for two reasons. First, it has not been demonstrated that the zircons' ferromagnetic inclusions are primary; in fact, a recent petrographic study showed that only ~12% of iron oxides in Jack Hills zircons are not spatially associated with cracks or annealed cracks (Bell et al., 2015). Second, it has not been demonstrated that the zircons escaped heating above the Curie point of their constituent ferromagnetic inclusions prior to deposition at 3 Ga. The slow diffusion of Pb in zircon means that a 10 million-year-long, ~820°C thermal event, which far exceeds magnetite's 580°C Curie point, will produce just 1% Pb loss from a 100 μm radius non-metamict zircon (Cherniak and Watson, 2000) (Fig. 2). Both of these points mean that a zircon's magnetization could be far younger than its U-Pb age or even disturbance ages inferred from U-Pb discordance.

In an effort to address this issue, Tarduno et al. (2015) argued that, if the zircons had experienced high-temperature metamorphism, the Pb would be redistributed in an inhomogeneous fashion at the nm-scale, resulting in non-systematic Pb/U variations during secondary ion mass spectrometry (SIMS) depth profiling that they did not observe. The above statements misrepresent their SIMS capability in three ways: a) the sputtering process mixes near surface atoms at the ~10

nm-scale, b) the SHRIMP instrument they used cannot truly depth profile as sputtered atoms from both crater bottom and surface are simultaneously accelerated into the mass spectrometer, and c) the 10-20 μm diameter spot they used is three orders of magnitude larger than would be needed to reveal such nm-scale heterogeneities, even if they existed. In any case, even if they were able to detect such inhomogeneities, the studies cited in Tarduno et al. (2015) [e.g., Davis et al. (2008) and Kusiak et al. (2013)] show that their formation occurred during granulite facies metamorphism, indicating temperatures that greatly exceed magnetite's Curie point. Therefore, even the verified absence of such Pb redistribution would fail to rule out complete thermal remagnetization prior to deposition.

In summary, neither the age of magnetization in the Jack Hills zircons nor the existence of a dynamo prior to 3.5 Ga has been established. Bono et al. (2016)'s focus on the events that remagnetized the zircon host rocks after deposition at 3 Ga does little to address this problem and is a diversion from the central point of Weiss et al. (2015): the conglomerate tests failed. Nevertheless, it remains possible that some Jack Hills zircons might have escaped complete remagnetization and retain paleomagnetic records back to the Hadean. We suggest that the best way to resolve the conflicting results from the MIT and Rochester laboratories is through the open exchange of key primary demagnetization data and samples and independent attempts to reproduce the measurements of the two labs on a controlled sample suite. To address the key issue of whether the zircons themselves contain primary remanence, paleomagnetic investigations should be conducted on individual zircons that can be shown not to have been remagnetized since their formation. We invite Bono et al., as well as the wider community, to join us in this endeavor.

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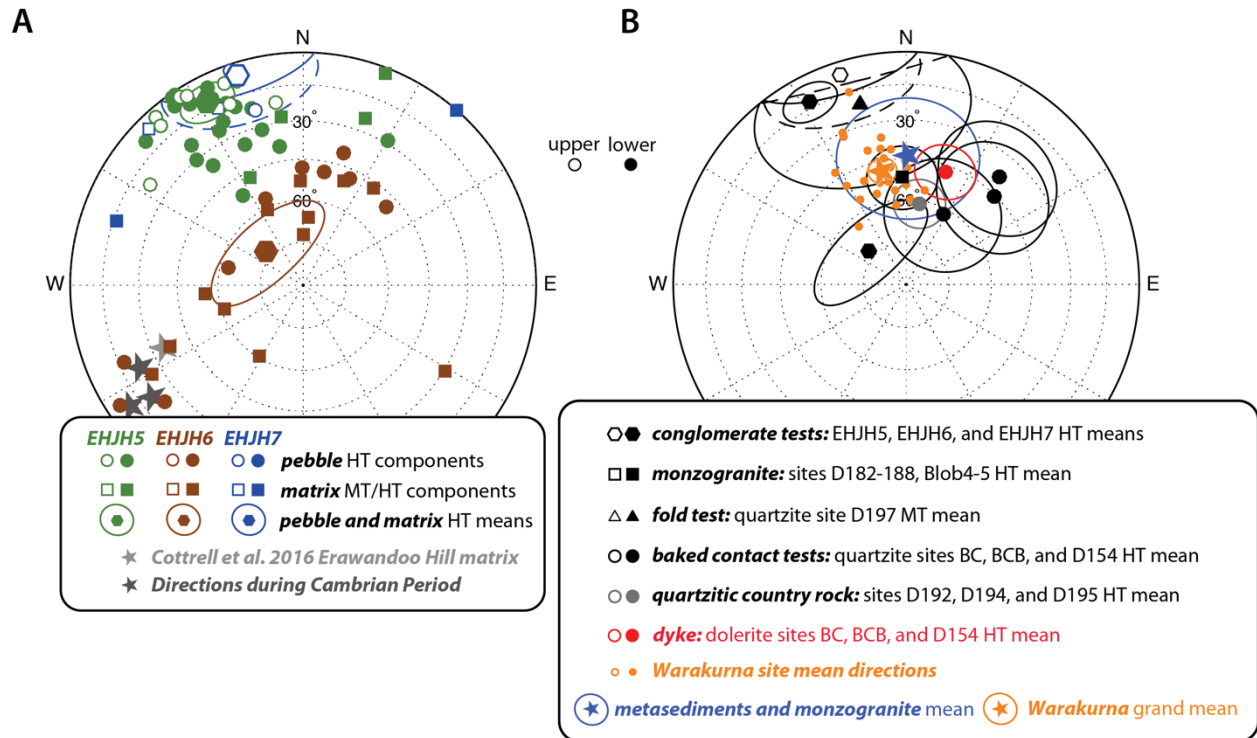
Figures

Fig. 1. Pervasive remagnetization of the Jack Hills and its relationship with the Warakurna LIP. Equal area stereonets show directions of high temperature (HT) and middle temperature (MT) components of natural remanent magnetization using data from Weiss et al. (2015). Open and closed symbols represent projections on upper and lower hemispheres, respectively. Peak unblocking temperatures for the MT and HT components range from 200-580°C, with most sedimentary samples unblocking below ~330°C [see Weiss et al. (2015)]. Points with surrounding ellipses are mean directions and associated 95% confidence intervals. (A) Conglomerate tests on three blocks of pebble conglomerate from the Hadean-zircon site at Erawandoo Hill: EHJH5 (green), EHJH6 (brown), and EHJH7 (blue). Circle and squares represent magnetization components for clasts and matrix, respectively. For all clast subsamples, HT components are shown for all three sites, while for matrix subsamples, MT components are shown for EHJH5 and HT components are shown for EHJH6 and EHJH7. Hexagons and associated ellipses give means and 95% confidence intervals for each of the three blocks (Table S2). Light grey star denotes geographic mean of Erawandoo Hill matrix measured by Cottrell et al. (2016). Dark grey stars denote the directions in Jack Hills coordinates of poles from Australia's apparent polar wander path during the Cambrian at 520-510 million years ago (Billy Creek/Wirre-alpa/Arona Creek

Limestone, Kangaroo Island Red Beds, and Lower Lake Frome Group) (Swanson-Hysell et al., 2012). (B) Evidence from rocks throughout the Jack Hills for remagnetization by the Warakurna LIP. Shown are means for the three EHJH conglomerate tests (hexagons) from (A) compared to means from the monzogranite intrusion (square), a quartzite fold test at site D197 (triangle), three quartzite baked contact tests associated with a 1.1 Ga dyke at sites BC, BCB, and D154 (black circles), and a distal baked contact test at country rock quartzite sites D192, D195 and D195 (grey circle) (Table S2). Small orange circles give the direction for site means of Warakurna LIP-associated sills, dykes, and dolerites in local Jack Hills coordinates (see Table S3 and Fig. S2 for details). The grand mean of all Jack Hills sites excluding the dyke (blue star) is indistinguishable from the dyke mean (red circle) (Table S2) and indistinguishable from the grand mean Warakurna LIP in local Jack Hills coordinates (orange star).
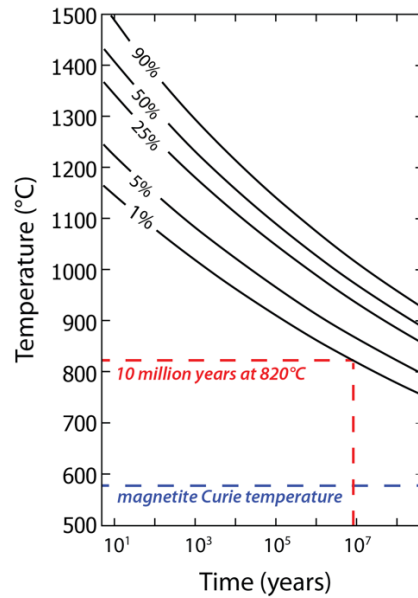
Fig. 2. Conditions for diffusional Pb loss in crystalline zircon. Shown are the time-temperature conditions for a thermal event that will lead to 1%, 5%, 25%, 50% and 90% Pb loss from a 100 μm radius grain. Blue line shows 580°C Curie point of magnetite, while red lines show that a temperature event to 820°C lasting for 10 million years will produce just 1% Pb loss. Such an event would be undetectable by the U-Pb methods used by Tarduno et al. (2015). After Cherniak and Watson (2000).