Outward displacement of rocky planetesimals in the early Solar System from angrite oxygen isotopes

Rider-Stokes, B. G.[1], Greenwood, R. C.[1], Anand, M.[1,2], White, L. F.[1], Franchi, I. A.[1], Debaille, V.[3], Goderis, S.[4], Pittarello, L.[5,6], Yamaguchi, A.[7], Mikouchi, T.[8], & Claeys, P.[4].

*[1]School of Physical Sciences, The Open University, Milton Keynes, MK7 6AA, UK. *Corresponding Author;* ben.rider-stokes@open.ac.uk *[2]Department of Earth Sciences, The Natural History Museum, London, SW7 5BD, UK. [3]Laboratoire G-Time, Université Libre de Bruxelles, CP 160/02, 50, Av. F.D. Roosevelt, 1050, Brussels, Belgium. [4]Analytical-Environmental and Geo-Chemistry, Vrije Universiteit Brussel, Pleinlaan 2, 1050, Brussels, Belgium. [5]Naturhistorisches Museum Wien, Mineralogisch-Petrographische Abteilung, Burgring 7,A-1010 Vienna, Austria. [6]University of Vienna, Department of Lithospheric Research, Althanstrasse 14, A-1090 Vienna, Austria. [7]National Institute of Polar Research, Tachikawa, Tokyo 190-8518, Japan. [8]The University Museum, The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku Tokyo, 113-0033 Japan.*

Angrite meteorites are thought to represent ancient basaltic igneous rocks that formed inward of Jupiter's orbit based on their isotopic parameters such as ε^{50}Ti, ε^{54}Cr and Δ^{17}O in addition to Fe/Mn ratios of pyroxene. New bulk oxygen isotope measurements of nine angrite meteorites and olivine 'xenocrysts' and groundmass fractions from three quenched angrites however, reveal clear isotopic disequilibrium, implying an impact melt origin. We determine a new well-constrained average Δ^{17}O value for the angrite parent body (−0.066 ± 0.016 ‰). The average Δ^{17}O value of groundmass fractions from Asuka (A) 12209, A-881371 and NWA 12320 quenched angrites on the other hand, demonstrate higher Δ^{17}O values (−0.003 ± 0.020 ‰). Microstructural investigations of NWA 12320 reveal the presence of both fully recrystallized and undeformed olivine xenocrysts, indicating that some xenocrysts underwent high-temperature processes. These results suggest that angrites bear signatures of impact-driven isotopic mixing, possibly in response to early giant planet migration. The evidence for impact mixing casts doubt on the utility of quenched angrites as a suitable Pb-Pb isotopic anchor, which in turn, has consequences for accurately defining the timeline of other Solar System events.

Key words: Angrites; Oxygen Isotopes; Impact Mixing; Early Solar System Processes; EBSD; Angrite Fractionation Line.

Angrites are defined as a group of unshocked, alkali-depleted basaltic meteorites that are amongst the oldest igneous rocks in the Solar System[1,2]. Based on the mineral chemistry of pyroxene (Fe/Mn ratios) within angrites and the isotopic dichotomy between non-carbonaceous and carbonaceous chondrites, it is likely that angrites originate from the inner Solar System [3,4]. Evidence from Mn/Cr isotopic systematics indicates that the angrite parent body (APB) was larger than the asteroid 4Vesta[5]. On the basis of differing textures and mineralogy, angrites have been principally divided into plutonic angrites (slowly cooled) with crystallisation ages ranging from 4560.74 ± 0.47 Ma to 4556.60 ± 0.26 Ma and quenched angrites (rapidly cooled) with crystallisation ages ranging from 4564.39 ± 0.24 Ma to 4562.2 ± 0.7 Ma[5]. However, there are some angrites, with similar mineralogical assemblages yet differing textures, which do not fit into either of these categories. For example, Northwest Africa (NWA) 8535 has been classified as a dunite (early formed cumulate) and NWA 10463 represents an intermediate type between the plutonic and quenched angrites, which has recently been dated at 4560.25 ± 0.18 Ma[6,7].

The oxygen isotope compositions of bulk angrites have previously been reported as displaying a high level of homogeneity, particularly with regards to ^{17}O-excess (Δ^{17}O), indicating that all the currently identified angritic meteorites originated from a single parent body and likely underwent early isotopic homogenisation in a global magma ocean[8,9]. Although many angrites display limited evidence of shock deformation, indicating that the APB was not affected by multiple impact events[10], it has been suggested that the quenched angrites may represent impact melts[11,12]. While this hypothesis has been disputed[1], the recent discovery of variable deformation features in olivine xenocrysts within Asuka (A)-881371 and A 12209 further support the impact hypothesis[13,14], whereby olivine xenocrysts are relict grains originating from areas of the APB that survived impact melting[13]. However, it has also been argued that the kink bands and sub-grain boundary formation in olivine grains could result from ductile deformation in the APB mantle[14]. Consequently, further work is required to constrain the petrogenesis of the quenched angrites.

We collected high-precision, oxygen three-isotope data on bulk-rock samples from nine angrites, encompassing all petrologic subgroups (Supplementary Figure 1), using laser-assisted fluorination following established procedures[9,15]. In addition, separated olivine and groundmass fractions from A-881371, A 12209, and NWA 12320 were also analysed for their oxygen isotopic composition. All samples were treated with a solution of ethanolamine

thioglycollate (EATG) to ensure that any isotopic variations were not a result of terrestrial contamination (Supplementary Figure 4). For the whole-rock analysis of NWA 12320 (n = 2) we report the typical uncertainty of oxygen isotopic analyses at ± 0.018 ‰, as demonstrated by replicate analyses of homogenous standards[16]. All uncertainties for averages and the AFL (angrite fractionation line) are reported at 2SD.

Results

While the oxygen isotopic compositions ($\Delta^{17}O$ see methods for calculation) for five angrites have been previously reported as homogenous ($\Delta^{17}O = -0.072 \pm 0.014$ ‰[8]), we observe that the $\Delta^{17}O$ values of whole-rock NWA 12320 ($\Delta^{17}O = -0.024 \pm 0.018$ ‰) and the average $\Delta^{17}O$ of groundmass fractions of NWA 12320, A 12209 and A-881371 ($\Delta^{17}O = -0.003 \pm 0.020$ ‰ n = 8) are less negative, and are statistically distinct from the other angrites (Figure 1, Table Supplementary 1 & 2). Intriguingly, the olivine xenocrysts in NWA 12320, A 12209 and A-881371 display an average $\Delta^{17}O$ value ($\Delta^{17}O = -0.068 \pm 0.014$ ‰ n = 8) indistinguishable from the other whole-rock angrite meteorites (Figure 1, Table Supplementary 1 & 2). These results indicate that the groundmass in NWA 12320, A 12209 and A-881371 are isotopically distinct from the rest of the sample. While lesser amounts of groundmass may result in undetectable shifts in some quenched angrites, NWA 12320 is dominated by groundmass and therefore the whole-rock sample equates to a groundmass separate. The origin of such isotopic heterogeneity within magmatic samples requires the addition of isotopically distinct material. Therefore, to calculate an angrite fractionation line (AFL) we have opted to incorporate the $\Delta^{17}O$ values of the olivine xenocrysts in NWA 12320, A 12209 and A-881371, as they formed prior to the groundmass and so pre-date the introduction of any additional isotopically anomalous material. We have excluded the groundmass fractions and the whole-rock value of NWA 12320. From this, we derive an average $\Delta^{17}O$ value of -0.066 ± 0.016 ‰, redefining the AFL utilising eleven individual samples. All whole-rock measurements reported here, excluding the groundmass-dominated NWA 12320 datum, fall within the uncertainty of this new AFL, providing evidence that angrites are homogenous in regard to $\Delta^{17}O$ and suggesting that angrite meteorites originate from a single differentiated parent body that was homogeneous with respect to oxygen isotopes.

Thermal processing of relict olivine grains

Xenocrysts in NWA 12320 have had a complex history as shown from contrasting internal structures revealed by electron backscatter diffraction (EBSD) data and point to two distinct populations of olivine xenocrysts, one of which has a granular texture, clearly illustrated by band contrast images and inverse pole figures (IPF) (Figure 2a-c). The granular olivine xenocrysts have no discernible preferred orientation and are cemented by olivine with a higher Fe content, with each neighbouring grains displaying vastly distinct orientations. This is indicative of recrystallization, after shock-induced mosaicism or fragmentation. A second population of olivine xenocrysts demonstrates unaltered, undeformed grains with little to no internal misorientation (Figure 2d-f). A similarly complex history for the xenocrysts within A 12209 is exhibited by various degrees of lattice deformation, from weak deformation bands to sub-grain rotation crystallization (Supplementary Figure 2).

Discussion

The granular textures seen in NWA 12320 resemble experimental recrystallization textures induced at 1000 °C under dynamic conditions[17], but are also similar to polycrystalline olivine identified in the howardite impact melt breccia Jiddat al Harasis (JaH) 556[18] and the ureilite impact melt breccia Jiddat al Harasis 422[19]. Most noticeably, there is a measurable oxygen isotope variation between the groundmass and the whole-rock values for both samples, similar to those observed in this study for the quenched angrite meteorites. In the case of JaH 556 and JaH 422 it was concluded that the olivine xenocrysts represent relict grains that underwent recrystallisation in the impact melt. This indicates that xenocrysts within NWA 12320, A 12209 and A-881371 are in fact relict olivines grains that were affected by high-temperature processes. The identification of angrite-like clasts in howardites, various polymict urelites and CH3 chondrites lends additional support for the proposition that the APB was subjected to impact processing[20,21,22,23]. Moreover, the metal identified within the plutonic angrite NWA 2999 has been previously attributed to an exogenous source, introduced by impact events[24]. Thus, there is considerable evidence to support impact mixing on the APB early in Solar System history. The lack of shock deformation features within other relict olivine grains, plutonic, intermediate and dunitic angrites may reflect the greater depth at which these angrites formed or that they were more distal to the impact site(s) (Figure 3). Additionally, the chronological separation between the quenched angrites and plutonic angrites implies that the plutonic angrites had either not formed or were still molten at the time of impact, and therefore did not experience or preserve any shock-induced deformation.

While mantle rheology has been proposed as the cause of the variable deformation seen in some relict olivines in angrites[14], the oxygen isotopic disequilibrium we observe between the olivine and groundmass indicates they have distinct origins. Our data clearly indicate that the groundmass was the product of an impact event resulting in isotopic contamination by the impactor, whereas the olivines represent relict material of the APB. The significant difference in $\Delta^{17}O$ values between the groundmass and olivine, implies that the impactor formed in a different region of the protoplanetary disk in relation to the APB. In a scenario analogous to that proposed for JaH 556[18], it would only require a small quantity of impactor material to account for the isotopic disequilibrium displayed by groundmass and relict olivines, provided the impactor was enriched in $\Delta^{17}O$ compared to the APB. It has been recently suggested that angrites record mixing of inner and outer Solar System material (CI and CM carbonaceous chondrites) based on their relatively elevated H and N isotopic compositions[25,26]. However, the CM chondrites have a more negative $\Delta^{17}O$ compared to angrites[27], and while CI chondrites are slightly more positive in $\Delta^{17}O$, they also have extremely positive $\delta^{18}O$ values[27] and would therefore not be capable of the isotopic shift in values observed in the quenched angrites (Supplementary Figure 5). A chondritic impactor with a significantly positive $\Delta^{17}O$ value, such as the ordinary or R chondrites might be proposed[28]. However, a chondritic impactor would result in significantly enriched siderophile elements, whereas angrites are well known for their depletion in siderophile elements relative to chondrites[29,30]. Rumuruti, for example, has a nickel content of 14.4 mg/g[31]. When mixed with the APB (2.5 % impactor), one would expect ~360 ppm Ni, whereas A 881371 has ~103 ppm Ni[29]. We must therefore conclude that the impactor that struck the APB was differentiated, analogous to achondrite meteorites. To satisfy our oxygen isotope constraints the impactor must have had $\Delta^{17}O$ significantly above the TFL. A number of achondrites with $\Delta^{17}O$ values similar to the L chondrites have been identified (Supplementary Table 3). Based on our data, an impactor with the oxygen isotopic composition of these known achondrites would satisfy all the isotopic constraints. However, it must also be recognised that the modern day asteroid belt is highly depleted compared with its likely early Solar System mass[32]. Therefore, it is likely that this achondritic impactor has been destroyed and does not exist in the modern Solar System.

Regardless of the impactor, the combination of oxygen isotopic disequilibrium and evidence of high-temperature events causing recrystallization of relict olivine grains, provides

a compelling case for mixing early in Solar System history, and an impact melt origin for the quenched angrite meteorites (Figure 3).

The terrestrial fractionation line (TFL) is commonly used as a graphical reference when comparing distinct groups of extra-terrestrial samples and is normally quoted as having a $\Delta^{17}O$ value of 0‰. However, there is significant uncertainty about the exact $\Delta^{17}O$ value of the TFL, as this is dependent on the nature of the physical and thermal conditions that affected the reference sample suite used to define it[33]. In addition, there is, so far no consensus about the appropriate slope factor that should be used for silicate minerals when calculating $D^{17}O$[33]. Furthermore, it has been shown that terrestrial rocks and minerals form fractionation arrays that display slight y-axis offsets of approximately −30 to −70 ppm on the Vienna Standard Mean Ocean Water (VSMOW) reference scale[33]. To define an appropriate terrestrial reference line to compare our angrite samples with, we have recalculated the 195 terrestrial samples from a previous study[33], using an identical slope value to that used for the angrites (0.5247) and without an applied y-axis offset correction. This reference line is therefore directly comparable with our angrite data and has a $\Delta^{17}O$ value of -0.048 ± 0.020 ‰. As the $\Delta^{17}O$ value of our newly redefined AFL ($\Delta^{17}O = -0.066 \pm 0.017$ ‰) overlaps with the terrestrial mantle signature (Figure 1) this could indicate that the APB formed from the same O-isotope reservoir as the Earth, although differences in some nucleosynthetic isotope signatures between Earth and the APB (e.g., $\varepsilon^{48}Ca$, $\varepsilon^{50}Ti$, $\varepsilon^{54}Cr$ and $\varepsilon^{62}Ni$) point to their distinct origins[1,34].

It has been suggested that the formation and migration of giant gas planets are crucial to the evolution of planetary systems, yet the timing of these events in our Solar System remains largely unconstrained[35]. In addition to the major impact event required to explain the oxygen isotope variability in angrites proposed here, CB chondrites are very ancient meteorites that exhibit evidence of mixing with a differentiated body that could have been sourced from the outer Solar System[35]. Such extreme dynamical excitement is not an expected result of the classical accretion of bodies, and demands the interference of the giant gas planets[35].

Currently, three main competing models exist to explain the evolution of the Solar System, the 'Grand Tack', 'Low-mass asteroid belt' and 'Early Instability models'[36]. The 'Low-mass asteroid belt' model relies on later dynamical evolution induced by the chaotic evolution of Jupiter and Saturn[36], while the 'Early Instability model' suggests dynamical excitement ~10 Ma after the dispersal of the gaseous disk[36]. The evidence of large-scale mixing

in the first 2-3 Ma of Solar System history presented here is in support of the 'Grand Tack' model and argues against the later dynamical excitement suggested by the other two models. The "Grand Tack" model of giant planet migration implies that Jupiter first migrates inwards and then, as the result of a resonance with Saturn migrated outwards again[37]. During the initial inward drift, rocky planetesimals were scattered inwards to 1 AU or less. The disc here became thickened and formed the feeder zones for Earth and Venus. However, a proportion (14 %) of these rocky planetesimals were also scattered outwards to 3 AU and beyond[37]. During subsequent outward migration, some of this rocky material was encountered again by the giant planets and scattered back into the inner main belt. Finally, towards the end of the Grand Tack, the gas giants moved through the outer icy planetesimal zone, scattering a fraction of them inwards into the main belt[37].

As previously noted, volcanic activity on the angrite parent body took place extremely early in Solar System history, at around 4564 Ma[5] and so, approximately 3 Ma after calcium-aluminium-rich inclusion formation (4567.30 ± 0.16 Ma[38]). This is within the 4 Ma period during which the solar nebular gas is considered to have persisted[39]. Impact dynamics within the inner Solar System with gas still present would have been subdued compared to those that prevailed after the nebula had dissipated. As a consequence, displacement of the APB from the inner Solar System and implantation into the main belt appears to have taken place with only low levels of deformation. If this model is correct, it suggests that angrites preserve an early deformation record that is related to Jupiter's inward and subsequent outward migration. Consequently, the oxygen isotope disequilibrium recorded by the impact melt and relict olivine grains in angrite meteorites may represent the earliest isotopic evidence for the Grand Tack migration. The quenched angrite meteorite D'Orbigny is a commonly used Pb-Pb anchor due to the previous suggestions that quenched angrites are primitive, unshocked, pristine samples[40]. However, we have now demonstrated that some quenched angrites have in fact suffered major impact processing. While D'Orbigny does not appear to demonstrate measurable evidence of impactor contamination in its whole-rock O-isotope composition, the similarity in textures and chemical compositions with the A 12209, A-881371 and NWA 12320 samples strongly indicates a common origin. Any adjustments to the time anchor would require a corresponding correction to the 'model ages' of all materials dated using D'Orbigny as an anchor. This, in turn, has consequences for accurately defining the absolute timeline of Solar System events.

Methods

Oxygen isotope analyses were undertaken by laser-assisted fluorination at The Open University, UK following established procedures[8,15]. Oxygen isotopic analyses are given in standard δ notation, where $\delta^{18}O$ is calculated relative to the international standard, Vienna Standard Mean Ocean Water (VSMOW) as:

$$\delta^{18}O = [(^{18}O/^{16}O_{sample})/(^{18}O/^{16}O_{VSMOW})-1] \times 1000\ (‰)$$

and similarly, for $\delta^{17}O$ using the $^{17}O/^{16}O$ ratio.

$\Delta^{17}O$ represents the deviation of a sample, or group of samples, from the terrestrial fractionation line (TFL) and has proved to be a useful parameter for defining different parent body sources[15,41]. In this paper all $\Delta^{17}O$ were calculated using the linearized format[42] with $\lambda = 0.5247$.

$$\Delta^{17}O = 1000 \ln(1+ \delta^{17}O/1000) - \lambda\ 1000 \ln(1+ \delta^{18}O/1000$$

Laser-assisted fluorination currently provides the highest precision available for oxygen isotope analysis. Replicate analyses of our internal obsidian standard (N = 38) gave the following values: (2SD) of ± 0.053 ‰ ($\delta^{17}O$), ± 0.095 ‰ ($\delta^{18}O$), and ± 0.018 ‰ ($\Delta^{17}O$)[16]. While laser fluorination does not provide spot analysis, unlike secondary ion mass spectrometry (SIMS) or UV laser ablation, the need to resolve slight differences in $\Delta^{17}O$ means that its ability to provide very high precision data make it the most suitable technique for this study.

Olivine grains were carefully picked from the groundmass using stainless steel tweezers to avoid any oxygen isotopic contamination. Following the separation, whole-rock chips along with groundmass and olivine-rich fractions of NWA 12320, A 12209 and A-881371 (~2 mg of material per replicate) were loaded into separate wells in a Ni sample holder. Once the fractions were loaded, the holder was placed in the sample chamber and the system was pumped down to a vacuum pressure of $\sim 10^{-7}$ mbar and baked out at around 80 °C overnight to remove any adsorbed moisture from the system.

Internal obsidian standards were analysed alongside the samples to monitor system performance. Analyses were performed following the standard protocols[15]. Briefly, this involved heating the samples with a CO_2 laser (10.6 μm wavelength) in the presence of BrF5 adjusting the beam diameter from 3mm to 1mm in order to achieve a complete reaction. Following the reaction of the sample, the gas is purified over liquid nitrogen temperature traps and KBr at 120˚C to remove excess BrF_5 and other reaction products (e.g. SiF_4, F_2, etc) before being expanded into a Thermo Finnigan MAT 253 Dual Inlet Isotope Ratio Mass Spectrometer (IRMS) for isotopic analysis.

Unlike most other meteorite groups, only one angrite is a recorded fall (Angra Dos Reis)[1]. Despite the long terrestrial residence times for angrite meteorites, which range from <0.06 Ma to 0.43 Ma, terrestrial contamination is minor[1]. On hand specimen examination, some of the Northwest Africa finds appear to be moderately weathered. This can present a significant problem when attempting to obtain high precision during oxygen isotope analyses. To resolve this issue, leaching of meteoritic finds can remove weathering products, mitigating terrestrial contamination. In this study, we analysed chips of both untreated material and leached material (70 – 170 mg) to determine whether or not the angrites investigated had indeed been affected by terrestrial alteration. This process was conducted using a solution of ethanolamine thioglycollate (EATG), a technique routinely used at the Open University to clean weathered meteorites[43]. EATG treatment is preferred dilute HCl which can partially remove indigenous glass and feldspathic-rich material. Samples were treated repeatedly until no colour change of the solution was apparent, and then washed with water and then 50/50 isopropanol alcohol (IPA). Dried samples are then investigated using the same methodology as the untreated samples.

For petrographic examination, a small chip of NWA 12320 was embedded within a 1-inch round e-poxy mount and coated using a Safematic CCU-010 Compact Coating Unit (<5 μm). The mount was investigated using a Zeiss Crossbeam 550 with an Oxford Instruments Symmetry 2 EBSD detector at The Open University. High-resolution Energy Dispersive X-Ray Spectroscopy (EDS) smart-mapping was collected using an Oxford Instruments Ultim Extreme and an Oxford Instruments Ultim Max detector. The sample was tilted to 70° and an electron beam was used to generate EBSD “maps”, consisting of electron backscatter diffraction patterns (EBSPs) acquired at step sizes ranging from 400 nm. The beam conditions

303 used for both EDS and EBSD analyses comprised an incident beam ranging between 1-2 nA
304 current and a 20 kV accelerating voltage at a working distance of 12 mm.
305
306 **Data availability**
307 All data generated or analysed during this study are either included in the Article or its
308 Supplementary Information.
309
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324 The authors declare no competing interests
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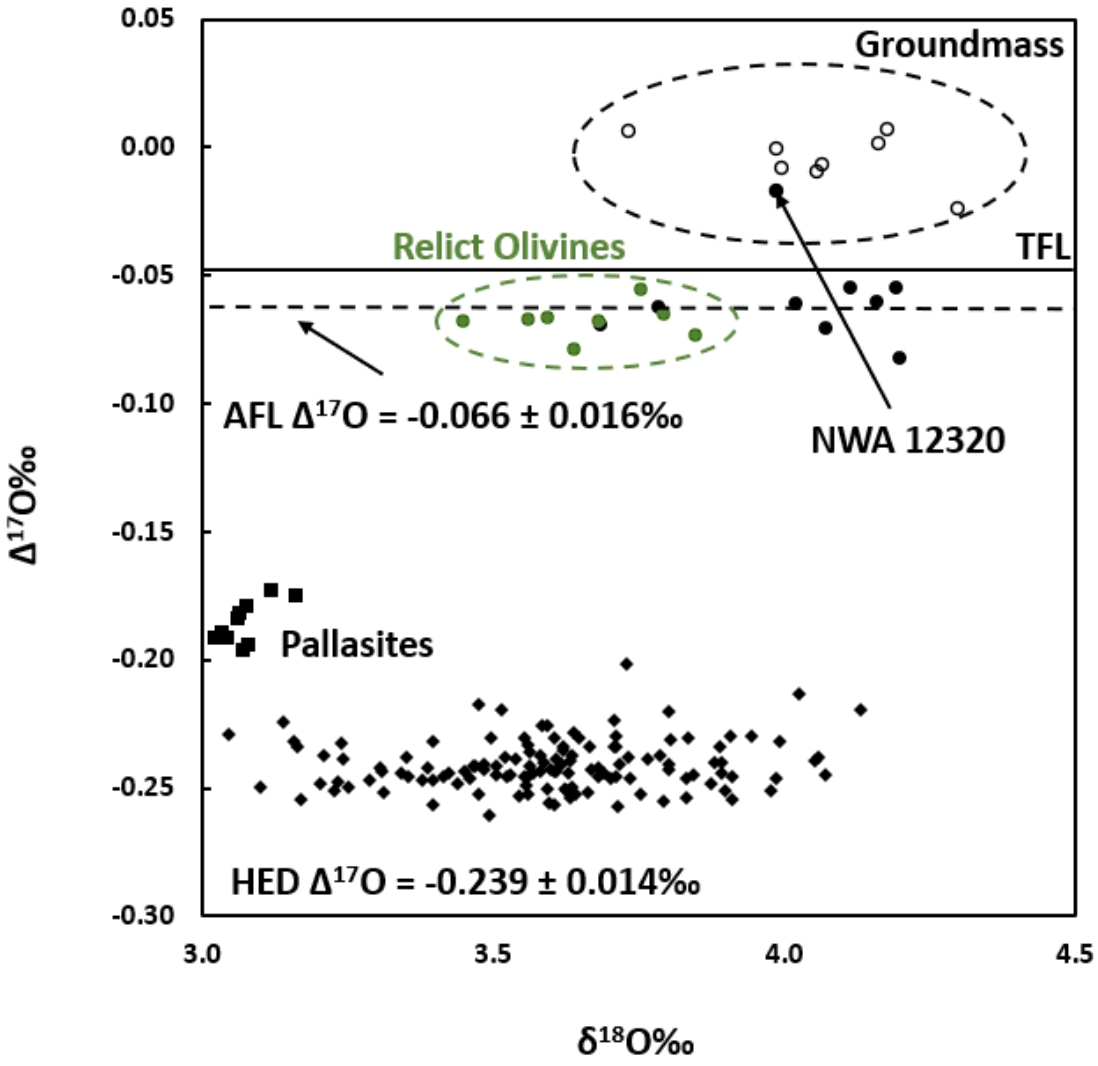

Figure 1: ***Triple oxygen isotope systematics for angrites, pallasites and HEDs.*** *In this diagram, we show our angrite data in relation to a TFL line (−0.048 ± 0.020) that has been calibrated using a suite of 195 terrestrial samples run under identical conditions to the angrites in this study and calculated using the same slope factor (0.5247) (see text for further details). Relict olivines (green closed circles) from A 12209, A-881371 and NWA 12320 display similar values to bulk angrites (black closed circles) and fall within the newly-defined AFL ($\Delta^{17}O$ - 0.066 ± 0.016 ‰), while groundmass fractions (open black circles) yield more positive $\Delta^{17}O$ values. This discrepancy in at least three angritic meteorites, suggests contributions from unique parent bodies for both the olivine and groundmass fractions, requiring early mixing of*

483 *planetary reservoirs on the angrite parent body. The angrite meteorites are well resolved from*
484 *both pallasite meteorites (black triangles) and howardite-eucrite-diogenite (HED) meteorites*
485 *(black squares)[8]. Cross represents 2SD uncertainty[42].*
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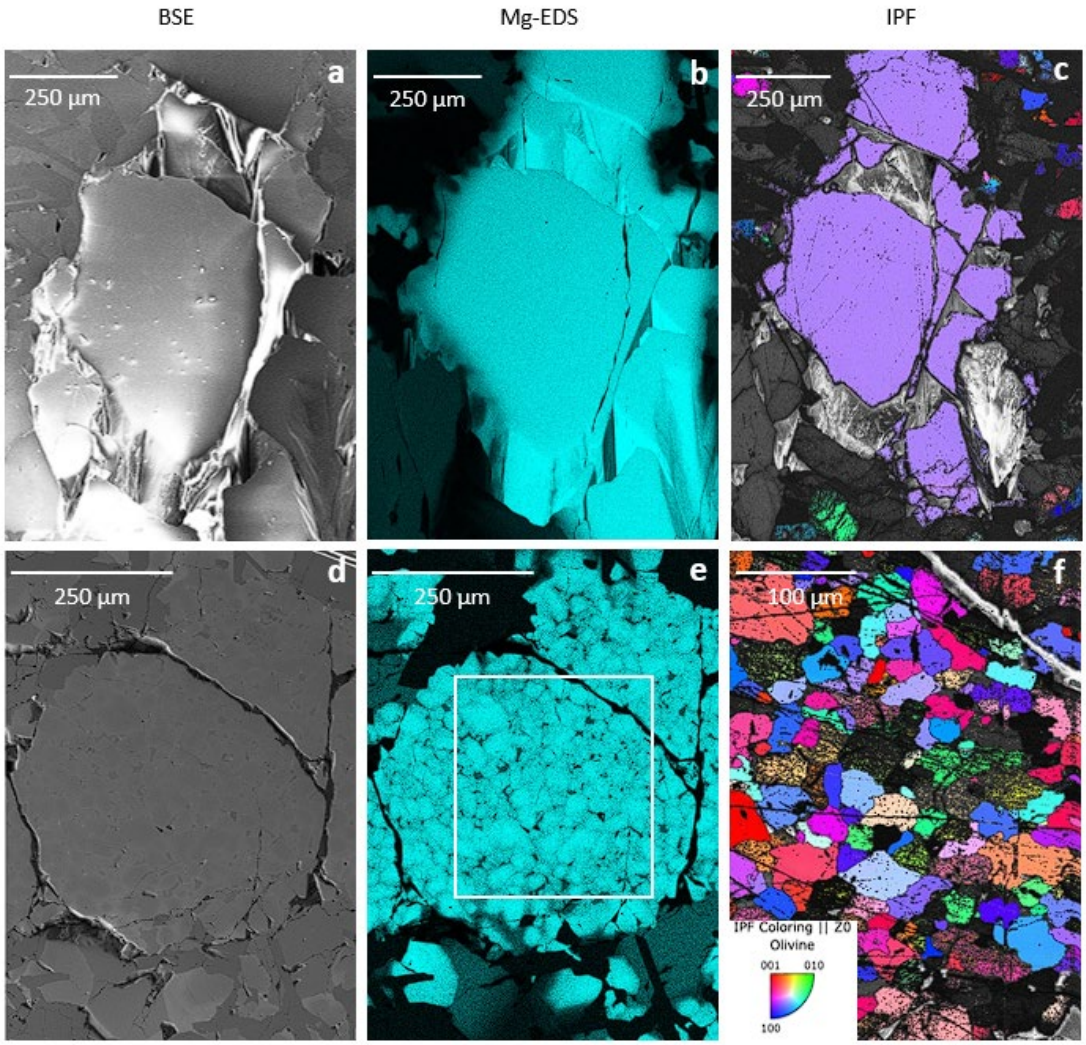

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490 *Figure 2:* ***Chemical and structural characterisation of relict olivine grains in NWA 12320.***
491 *Two populations of olivine are observed within the sample as shown by backscatter electron*
492 *(BSE) imaging (a & d). Both of which are chemically heterogenous, as evidenced by*
493 *magnesium energy dispersive (Mg-EDS) imaging (b & e). Inverse pole figure (IPF) images*
494 *demonstrate the difference between non-recrystalised relict grains (c), displaying a single*
495 *orientation (purple), versus the recrystallized relict grains (f), displaying multiple crystallites*
496 *with differing orientations (multi-colour grains). Given the low state of deformation within*

497 *both the undeformed olivine grains, as well as the majority of angrite meteorites, olivine*
498 *recrystallization for a subset of grains must have occurred prior to crystallization of the bulk*
499 *sample, suggesting that the olivine are relict material that survived impact melting.*
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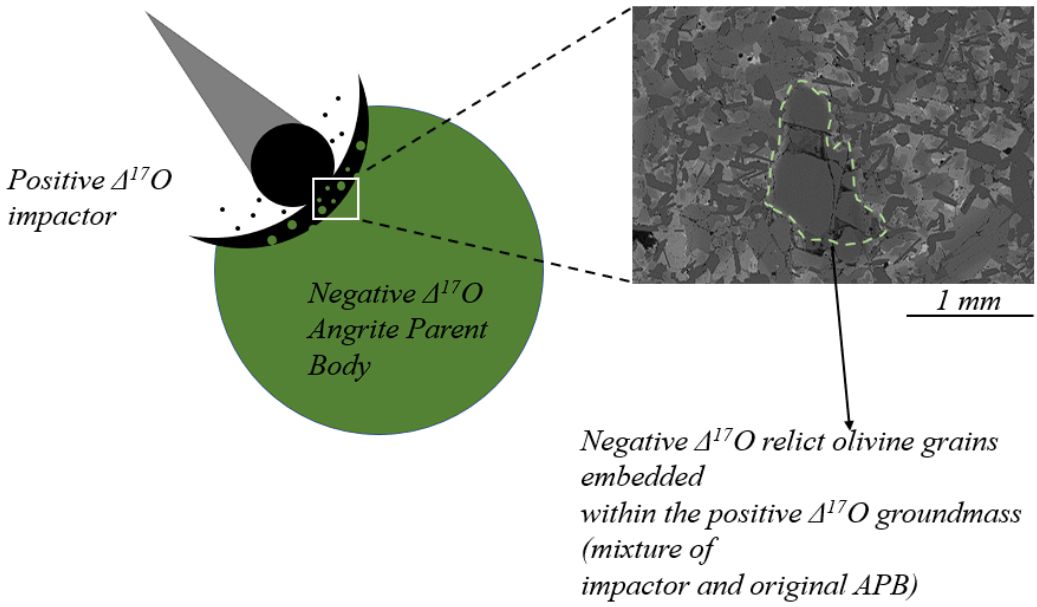

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504 *Figure 3 – **A schematic depicting a possible scenario which causes the oxygen isotopic***
505 ***disequilibrium in the quenched angrite meteorites.** An impactor with a positive oxygen*
506 *isotopic composition, collides with the angrite parent body (APB). Mixing of the two separate*
507 *bodies occurs and relict olivine grains of the APB are affected by high temperature events.*
508 *Plutonic and dunitic angrites escape shock deformation and isotopic mixing due to their depth/*
509 *distance from the impact site on the APB or due to being molten during the time of impact*
510 *mixing. Backscatter electron (BSE) image of the relict olivine grain is the grain depicted in*
511 *Figure 2a-c.*
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