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Outward displacement of rocky planetesimals in the early Solar System: Oxygen isotope evidence from angrites.

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Angrite meteorites are ancient inner Solar System meteorites that likely formed inward of Jupiter's orbit based on their isotopic parameters such as ε50Ti vs ε54Cr and Δ17O vs ε54Cr in addition to Fe/Mn ratios of pyroxene1,2. Angrites therefore provide a unique insight into the earliest stages of planetary evolution in the inner Solar System. Here, we report the bulk oxygen isotope composition of nine angrite meteorites, as well as olivine xenocrysts and matrix fractions from three "quenched" angrites. We calculate a new average Δ17O value for bulk angrites, which overlaps with the Δ17O results of terrestrial materials when treated in the same manner, hence supporting the proposition that angrites and Earth may be derived from a similar precursor reservoir[2]. Our analyses of olivine xenocrysts and matrix fractions from Asuka 12209, Asuka 881371 and NWA 12320 define a clear isotopic disequilibrium, with matrix fractions yielding higher $\Delta^{17}O$ values than either the xenocrysts or bulk samples. Microstructural investigations of NWA 12320 reveal the presence of both fully recrystallized and undeformed olivine xenocrysts, indicating that some xenocrysts underwent high temperature events. We interpret these findings in terms of angrite meteorites recording evidence of impact-driven isotopic mixing, possibly in response to early giant planet migration.

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

Angrites constitute a group of unshocked, alkali-depleted basaltic meteorites that are amongst the oldest igneous rocks in the Solar System[3,4]. Based on the mineral chemistry of pyroxene (Fe/Mn ratios) within angrites, it has been suggested that their parent body (APB) originated from a similar planetary reservoir to that of the Earth-Moon system2. Evidence from Mn/Cr isotopic systematic indicates that the APB was larger than 4 Vesta5. 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 to 4556.60 ± 0.26 Ma and quenched angrites with crystallisation ages ranging from 4564.39 ± 0.24 to 4562.2 ± 0.7 Ma (rapidly-cooled) subgroups5. 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 stage between the plutonic and quenched angrites, which has recently been dated to 4560.25 ± 0.18 Ma6,7.

The oxygen isotope composition of bulk angrites display 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 (APB) 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 significant impact processing[10], it has been suggested that the quenched angrites may represent impact melts[11,12]. While this hypothesis has been disputed[3], the recent discovery of variable deformation features in olivine xenocrysts within Asuka 881371 and Asuka 12209 further supports fthe 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 evidence is required to constrain the petrogenesis of the quenched angrites.

We collected high-precision, three O isotope data on bulk-rock samples from nine angrites, encompassing all petrologic subgroups (see supplementary for further details), using laser-assisted fluorination following established procedures[9,15]. In addition, separated olivine and matrix fractions from Asuka 881371, Asuka 12209, and NWA 12320 were also analysed. All samples were treated with a solution of ethanolamine thioglycollate (EATG) to ensure that any isotopic variations were not a result of terrestrial contamination (see supplementary details). All uncertainties are reported at 2SD and N = 2 for all individual sample averages, where 1 N equals four blocks of ten measurements .

While the oxygen isotopic compositions (Δ17O = δ17O – 0.52 x δ18O) for five angrites have been previously reported as homogenous (Δ17O = -0.072 ± 0.014‰8), we observe that the whole-rock Δ17O value of NWA 12320 (Δ17O = -0.017 ± 0.004‰) and matrix fractions of NWA 12320, Asuka 12209 and Asuka 881371 reveal less negative values that are statistically distinct from the other angrite meteorites ($\Delta^{17}O$ = -0.024 ± 0.016‰, -0.001 ± 0.007‰ and -0.003 ± 0.007‰, respectively). Intriguingly, the olivine xenocrysts in NWA 12320, Asuka 12209 and Asuka 881371 display $\Delta^{17}O$ values of -0.065 ± 0.018‰, -0.068 ± 0.015‰ and -0.067 ± 0.008‰, respectively, indistinguishable from the other whole-rock angrite meteorites (Figure 1). These results indicate that the matrix in NWA 12320, Asuka-12209 and Asuka-881371 are isotopically distinct from the rest of the samples. While lesser amounts of matrix may result in undetectable shifts in some quenched angrites, NWA 12320 is dominated by matrix and therefore the whole-rock sample equates to a matrix separate. The origin of such isotopic heterogeneity within magmatic samples requires addition of isotopically distinct material and therefore to calculate an AFL, we have opted to incorporate the $\Delta^{17}O$ values of the olivine xenocrysts in NWA 12320, Asuka 12209 and Asuka 881371 while excluding their matrix fractions and the whole-rock value of NWA 12320. From this, we derive an average $\Delta^{17}O$ value of -0.065 ± 0.016‰, redefining the AFL utilising eleven individual samples. All whole-rock measurements reported here, excluding the NWA 12320 datum, fall within uncertainty of this new AFL, providing considerable evidence that angrites are homogenous in regard to $\Delta^{17}O$ and suggesting that angrite meteorites all originate from a single differentiated parent body that was homogeneous with respect to oxygen isotopes.

A complex history of the xenocrysts in NWA 12320 is apparent from contrasting internal structures revealed by electron backscatter diffraction (EBSD) 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 2). 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 2). A similarly complex history for the xenocrysts within Asuka 12209 is exhibited by various degrees of lattice deformation, from weak deformation bands to subgrain rotation crystallization (see supplementary for further details).

The granular textures seen in NWA 12320 resemble experimental recrystallization textures induced at 1000 °C under dynamic conditions[16], but are also similar to polycrystalline olivine identified in the howardite impact melt breccia Jiddat al Harasis 556[17] and the ureilite impact melt breccia Jiddat al Harasis 422[18]. Most noticeably, there is a measurable oxygen isotope variation between the matrix and whole-rock values for both samples, similar to the quenched angrite meteorites. The authors of these two studies[17,18] conclude that the olivine xenocrysts represent relict grains that underwent recrystallisation in the impact melt. This indicates that at least some olivine grains in NWA 12320, Asuka 12209 and Asuka 881371 remained as relict material and were affected by elevated temperature processes. The identification of angrite-like clasts in howardites, various polymict urelites and CH3 chondrites further supports the proposition that the APB was subjected to impact processing[19,20,21,22]. Moreover, the metal identified within NWA 2999 has been previously attributed to an exogenous source, introduced by impact events[23]. Thus, there is a considerable evidence to support impact mixing on the APB early in Solar System history. The lack of shock deformation features within some 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 (Figure 3). Additionally, the chronological separation between the quenched angrites and plutonic angrites implies that the plutonic angrites were still molten at the time of impact, and therefore did not experience shock induced deformation.

While mantle rheology has been proposed as the cause of the variable deformation seen in some olivine xenocrysts[14], the oxygen isotopic disequilibrium we observe between the xenocrysts and matrix indicates they have distinct origins, with the matrix the product of an impact event that became contaminated by the impactor, and the olivine xenocrysts representing relict material of the APB. The significant difference in $\Delta^{17}O$ values, 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 from JaH 55617, it would only require a small quantity of impactor material to account for the isotopic disequilibrium displayed by matrix and xenocrysts, provided the impactor carried much more positive Δ17O values 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 compositions24,25. However, the majority of carbonaceous chondrite meteorites reveal negative Δ17O values26 and would therefore not induce the more positive matrix values depicted in the quenched angrites. A more viable option would be an ordinary chondrite, which demonstrate positive Δ17O values27, however, a chondritic impactor would add high quantities of metal, which is not observed in angrites. It is therefore suggested that the impactor that struck the APB was an achondrite with similar Δ17O values to the ordinary chondrites, alike NWA 1104228. To induce the isotopic difference between the xenocrysts and matrix based on the Δ17O composition of NWA 11042 (Δ17O =1.03‰)28, an impactor contribution of less than 10% would be required (see supplementary details).

Regardless of the impactor, the combination of oxygen isotopic disequilibrium and evidence of high temperature events causing total recrystallization of relict olivine grains, provide 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[29]. In addition, there is, so far no consensus about the appropriate slope factor that should be used for silicate minerals when calculating $\Delta^{17}O$[29]. 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 VSMOW reference scale[29]. To define an appropriate terrestrial reference line to compare our angrite samples with, we have recalculated the 195 terrestrial samples from a previous study[29], 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‰ (2s). Given that the $\Delta^{17}O$ value of our newly redefined AFL is -0.065 ± 0.017‰ (2s), it is clear that there is overlap between angrites and terrestrial samples (Figure 1). This new oxygen isotope evidence therefore supports previous suggestions, made on the basis of pyroxene Fe/Mn ratios, that angrites and terrestrial rocks are derived from a similar primary source reservoir[2]. Although, it is noted that there is a lack of similarity in terms of nucleosynthetic isotopes including $\varepsilon^{48}Ca$, $\varepsilon^{50}Ti$, $\varepsilon^{54}Cr$ and $\varepsilon^{62}Ni$ in addition to severe volatile and alkali depletion in angrites, unlike terrestrial materials[30,3].

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[31]. CB chondrites are similarly very ancient meteorites that exhibit evidence of mixing with a differentiated body that could have been sourced from the outer Solar System[31]. It has been subsequently suggested that this extreme dynamical excitement is not an expected result of the classical accretion of bodies, and demands the interference of the giant gas planets[31].

There are currently three main competing models to explain the evolution of the Solar System, the 'Grand Tack', 'Low-mass asteroid belt' and 'Early Instability models'. However, based on the evidence of large scale mixing, we favour the 'Grand Tack'. 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[32]. 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. During subsequent outward migration, some of this rocky material was encountered again by the giant planets and scattered back into the inner main disc. Finally, towards the end of the Grand Tack the gas giants moved through the outer icy planetesimal zone, scattering a fraction of them back into the outer main belt[32]. Based on this model, it is entirely possible that angritic material originating from the same reservoir that formed Earth could have been emplaced into the asteroid belt.

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 (4567.18 ± 0.50 Ma[33]). This is within the 4 Ma period in which solar nebular gas is now considered to have persisted[34]. 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 then outwards migration. Consequently, the oxygen isotope disequilibrium recorded by the impact melt and olivine xenocrysts may be the earliest isotopic evidence for the Grand Tack migration.

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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[15,35] In this paper all $\Delta^{17}O$ were calculated using the linearized format[35] with λ = 0.5247.

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

In this paper all $\Delta^{17}O$ were calculated using the linearized format[36].

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‰ ($\Delta^{17}O$)[37]. 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 it is the most suitable technique for this study.

Olivine grains were carefully plucked from the matrix using sterile tweezers. Following the separation, whole rock samples along with matrix and olivine-rich fractions of NWA 12320, Asuka 12209 and Asuka 881371 (~2 mg of material per replicate) were loaded into separate wells in a Ni sample holder. Once the fractions were loaded, the tray was placed in the sample chamber. The sample chamber was next brought down to a vacuum pressure of 10^{-7} mbar. Heater tape was then wrapped around the sample chamber and it was baked out at around 80 °C overnight to remove any adsorbed moisture from the system that could affect the oxygen isotope analyses.

An internal obsidian standard was run with the samples to monitor accuracy and precision. During laser heating, the beam diameter was initially set at 3 mm and the laser power slowly increased to a maximum of 20 W. In order to complete the reaction, the beam diameter was reduced to 1 mm and the laser power increased rapidly to a maximum value of 14 W. This second step ensures that any residual material that may have been missed from the first phase is fully reacted. Following the reaction of the sample, the gas is expanded into a ThermoFinnigan MAT 253 Dual Inlet Isotope Ratio Mass Spectrometer (IRMS).

Unlike other meteorite groups, angrites have very few recorded falls (with Angra Dos Reis being the only recorded fall)[3]. Despite the long terrestrial residence times for angrite meteorites, which range from <0.06 to 0.43 Ma, terrestrial contamination is minor[3]. 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 levels of 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). Tests undertaken on a suite of variably weathered H chondrites indicated that the EATG-wash method was efficient at removing Fe-rich alteration products, without disturbing the primary oxygen isotope composition of the samples[38]. EATG treatment is preferred in comparison to conventional leaching in dilute HCl, as this method can partially remove indigenous glass and feldspathic-rich material. Each meteorite investigated in this project was weighed and placed into centrifuge tubes. Between ~4 and ~12 ml of EATG was added to the tubes using a pipette and shaken to separate the sample into the solution (mixing repeated every 10 mins for 2 hrs). Once the sample has fully reacted with the EATG, the waste EATG is removed and replaced with 50/50 isopropanol alcohol (IPA) and deionized water. This new mixture is shaken four times to ensure the entirety of the sample is treated. The last step is to remove the mixture and replace with straight IPA and mix in the centrifuge for a few minutes. The sample is then removed from the IPA and left to evaporate in ambient temperatures. The weighed, treated samples are then investigated using the same methodology as the untreated samples.

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 then subsequently 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 used for both EDS and EBSD analyses comprised an incident beam ranging between 1-2 nA current and a 20 kV accelerating voltage at a working distance of 12 mm.

Data availability

All data used in the manuscript are presented in the Supplementary Data and are available on request from the corresponding author.

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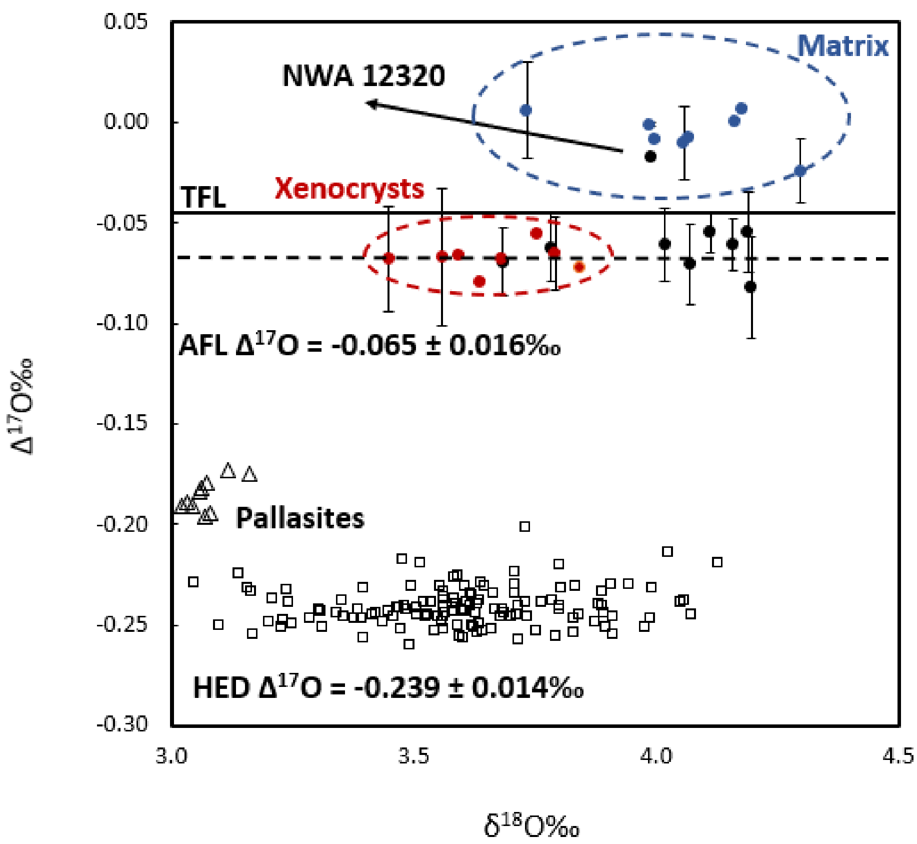

Figure 1: ***Triple oxygen isotope systematics for angrites, pallasites and HEDs.*** *In this diagram we show our angrite data in relation to a TFL line that has been calibrated using a suite of 195 terrestrial samples run under identical conditions to the angrites in this study and calculated using the same slope factor (0.5247) (see text for further details). The fact that our newly defined AFL shows significant overlap with this terrestrial reference line at the 2s level supports suggestions that the APB and Earth formed from the same reservoir. Our data are consistent with the possibility that angrites may represent material from the giant Moon-forming impactor. Olivine xenocrysts from Asuka 12209, Asuka 881371 and NWA 12320 display similar values to bulk angrites (black closed circles) and fall within the newly-defined angrite fractionation line ($\Delta^{17}O$ -0.065 ± 0.017‰), while matrix fractions yield more positive $\Delta^{17}O$ values. This discrepancy in at least three angritic meteorites, suggests unique parent bodies for both the olivine and matrix fractions, requiring early mixing of planetary reservoirs on the angrite parent body. The angrite meteorites are well resolved from both pallasite meteorites (black triangles) and howardite-eucrite-diogenite (HED) meteorites (black squares)*[7].

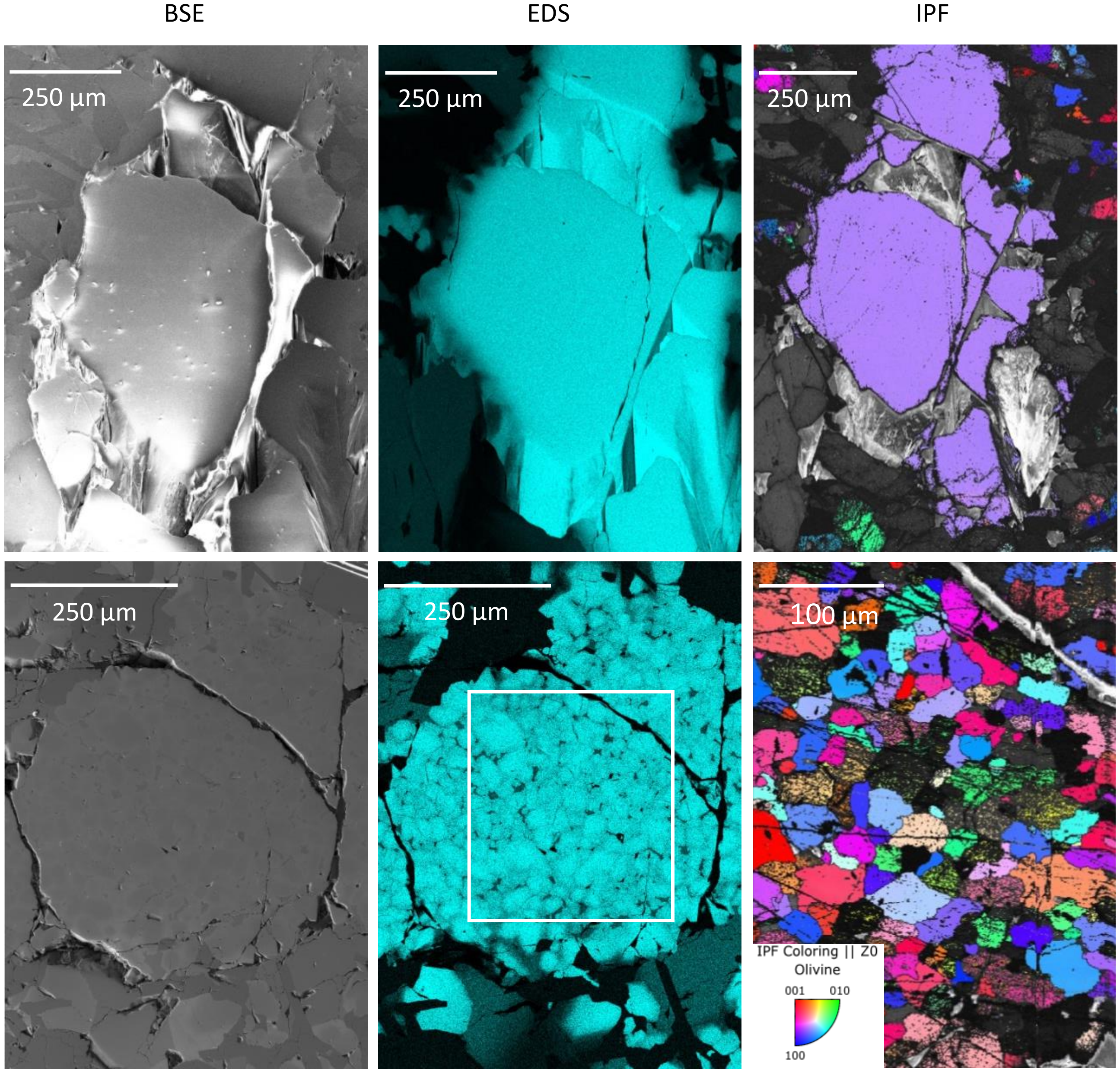

Figure 2: ***Chemical and structural characterisation of olivine xenocrysts in NWA 12320.*** *Two populations of xenocrysts are observed within the sample, with chemically heterogenous recrystallized olivine occurring in close proximity to undeformed grains. Given the low state of deformation within both the undeformed olivine xenocrysts, as well as the majority of angrite meteorites, olivine recrystallization for a subset of xenocrysts must have occurred prior to crystallization of the bulk sample, suggesting that the olivine xenocrysts are relict material that survived impact melting.*

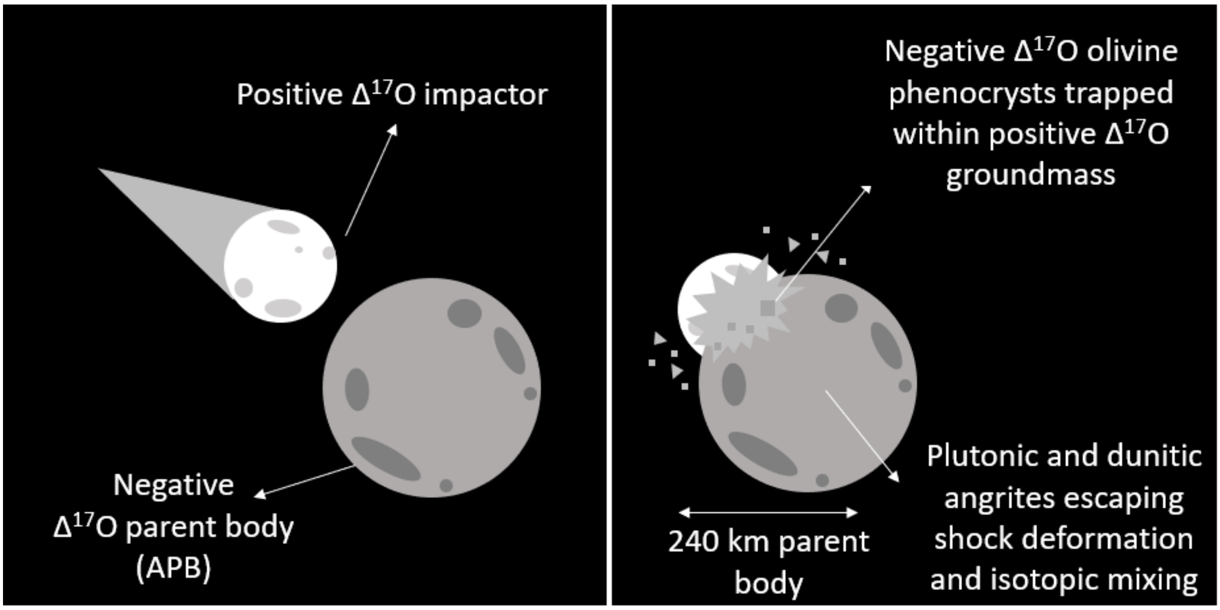

Figure 3 – ***A schematic depicting a possible scenario which causes the oxygen isotopic disequilibrium in the quenched angrite meteorites.*** *An impactor with a positive oxygen isotopic composition, collides with the angrite parent body (APB). Mixing of the two separate bodies occurs and relict olivine grains of the APB are affected by high temperature events. Plutonic and dunitic angrites escape shock deformation and isotopic mixing due to their depth/ distance from the impact site on the APB or due to being molten during the time of impact mixing.*