HIGH PRECISION OXYGEN THREE-ISOTOPE ANALYSES OF PROBABLE COMETARY MATERIAL FROM A GIANT CLUSTER IDP. C. Defouilloy[1], D. J. Joswiak[2], D. E. Brownlee[2], N. T. Kita[1]. [1]WiscSIMS, Department of Geoscience, University of Wisconsin-Madison, Madison, WI 53706, USA, [2]Department of Astronomy, University of Washington, Seattle, WA 98195, USA.

Introduction: Interplanetary Dust Particles (IDPs) are particles constantly falling on Earth and that can be collected by aircraft flying in the upper atmosphere. Collected IDPs can originate from either asteroids or comets. The Giant Cluster IDP (U2-20GCA) is a loose aggregate of thousand particles, both fine and coarse [1]. It shows similar texture, mineralogy and chemical compositions as comet particles [2-6] and is thus likely of cometary origin. Previous works on fine grains from this Giant Cluster showed a large range of oxygen compositions among its fine grain particles [2]. In order to explore further the possible link between this IDP and comets, we analysed the oxygen 3 isotope composition of several coarse silicates from this IDP. First results of this study have been reported in [7] and [8].

Experimental: Five distinct particles, ranging from 5 ot 15 μm in diameter, were analysed. Particles LT7, LT14, LT26, LT16 are bi-crystalline particles containing both pyroxene and olivine phases while LT19 is a polycrystalline grain containing various phases and one olivine grain. Mg# in the silicate phases varies from 77 to 96. The Mg# in multi-phase particles is consistent between mineral, with the exception of LT14, where olivine and pyroxene have clearly distinct Mg# (77 and 96 respectively). Particles were mounted in a 100 μm wide acrylic cubes which were each pressed into a 1.4 mm diameter indium metal substrate with a San Carlos olivine grain, at the center of a 1 inch aluminum disk.

To facilitate aiming at those small particles, we marked the desired SIMS analysis positions using a Zeiss focused ion beam (FIB) field emission (FE)-SEM Auriga (UW-Madison) by removing a 1 μm x 1 μm square of the carbon coating. The focused Ga^+ beam with an accelerating voltage of –30 keV was set to 5 pA, for a dose of 0.4 nC/ μm^2 [9].

Oxygen three-isotope ratios were acquired with the Cameca IMS 1280 ion microprobe at the WiscSIMS laboratory of the University of Wisconsin-Madison over 2 sessions.

Prior to each unknown sample analysis, secondary $^{16}O^-$ ion images were obtained by 10 μm x 10 μm primary beam rastering, in order to identify the FIB marks. Due to the absence of coating, the FIB marks appear as bright spots. With the recent addition of a NanoDeflector to the WiscSIMS IMS 1280, primary beam adjustment was greatly improved and the ion image was centered with a precision of 0.1 μm [9].

The analytical conditions were similar to those detailed in [9] and [10]. The primary Cs^+ beam was set at an intensity of 3 pA, for a beam size of ~1.5 μm in diameter. The conditions remain the same for ion imaging as for isotope analyses. Secondary ions $^{16}O^-$, $^{17}O^-$, $^{18}O^-$ were detected simultaneously on the multicollection system; $^{16}O^-$ by a Faraday cup and $^{17}O^-$ and $^{18}O^-$ by electron multipliers. The mass resolving power (MRP at 10 % peak height) was set at ~ 6000. The contribution of the tailing of $^{16}O^1H^-$ ion on $^{17}O^-$ was estimated by the method described in [11] though the correction (< 0.1 ‰) was negligible. Each analysis lasted 20 minutes, giving typical internal precisions of 1.5‰, 2.3‰ and 2.6‰ (2σ) for $\delta^{18}O$, $\delta^{17}O$, $\Delta^{17}O$, respectively.

One to four analyses were performed on each particle, with at least one per silicate phase and bracketed by eight analyses performed on San Carlos olivine (Fo_{89}) grains mounted in the same disks, within 500 μm of the sample particle. The external reproducibility of the running standards was on average 1.4‰, 2.1‰, and 1.6 ‰ for $\delta^{18}O$, $\delta^{17}O$, $\Delta^{17}O$, respectively, which were assigned as analytical uncertainties to unknown samples [12]. Instrumental biases of all silicates were corrected from the calibration using multiple standards with known oxygen isotope ratios that cover the range of compositions of the unknowns: additional olivines (Fo_{60}, Fo_{100}), three low-Ca pyroxene (En_{97}, En_{85}, En_{70}) and a diopside standard.

Results: Oxygen isotopic ratios in the Giant Cluster IDP grains vary from –0.8 ± 4.1‰ to 2.6 ± 2.0‰ in $\delta^{18}O$. Results are shown in Fig. 1, along with data previously reported in [7,8]. Grains containing both olivine and pyroxne do not show significant difference in the phases' respective oxygen isotope ratios. Therefore, we average the compositions of all the analyses on a given particle. SEM imaging after the SIMS sessions confirmed accurate aiming, with SIMS pits located within < 0.2 μm of FIB-marks with exception of particle LT7. However SEM imaging confirmed that the misplaced spots on LT7 were within the correct phase. Data are in good agreement with previous IDP studies, spreading between the terrestrial fractionation line (TFL) and the primitive chondrule mineral (PCM) line, though they do not show as large a spread along the PCM line. They also appear to be similar to oxygen

compositions of ^{16}O-poor silicates grains from the comet Wild 2. While ^{16}O-rich compositions have been measured in fine grains [2], no ^{16}O-rich particles have been found in coarse grains of the Giant Cluster IDP, though the sampling is still limited.

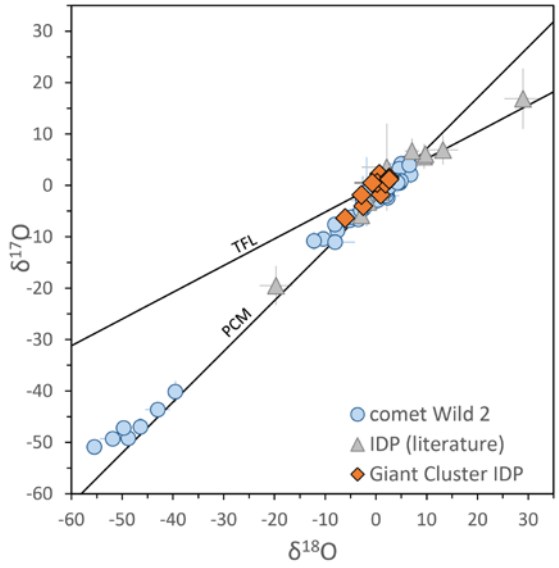

Fig. 1: Oxygen three-isotope ratios Giant Cluster IDP olivine and pyroxene, compared to comet Wild 2 [12-16] other IDP studies [17-19]. TF and PCM represent the terrestrial fractionation line and the primitive chondrule line (~ slope 1 line), respectively.

Discussion: We compared the Giant Cluster IDP with cometary and meteoritical material by combining Δ^{17}O and Mg# data. Our data from the Giant Cluster IDP seem to form two clusters: one cluster shows homogeneous data with Mg# = 79 ± 2 and Δ^{17}O = 0.1 ± 1.3 (2σ), while the other one shows high Mg# (ranging from 93.6 to 99.2) and Δ^{17}O spreading from –3.2 ± 1.4‰ to –1.9 ± 1.4‰ . The compositions displayed by these two clusters are consistent with the range of compositions shown in comet Wild 2 (Fig. 2), even though Wild 2 data are more scattered and also show particles with ^{16}O-rich, high Mg# compositions as well as particles with lower Mg#, in the 60-68 range. While Wild 2 Δ^{17}O-Mg# compositions have been shown to be close to CR chondrites [7], the spread of Δ^{17}O shown in the Giant Cluster particles with high Mg# overlaps other carbonaceous chondrite trends, as well as ordinary chondrite compositions (Fig 2.). This spread could be the result of variable conditions in the reservoir where those particles formed, or could alternatively be the result of a mixing of particles from different origins which were later transported and accreted to the parent body of the Giant Cluster. Further work is required to fill some of the gaps in the current data, or to assert that those gaps are significant and represent a distinction from compositions found in other bodies (comets or meteorites). It seems that material from the Giant Cluster IDP formed in similar conditions as particles from comet Wild 2.

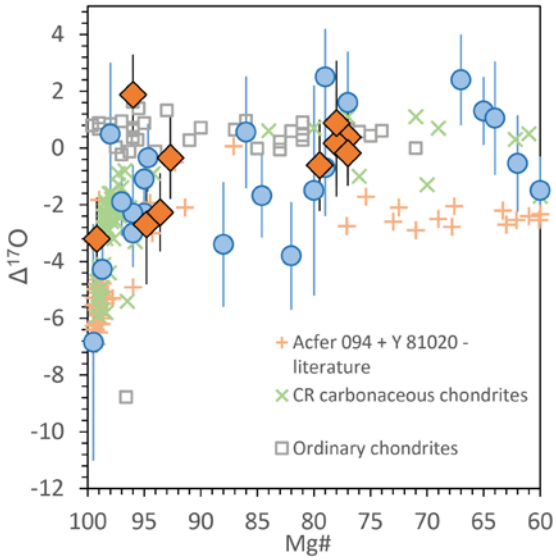

Fig. 2: Relationship between Mg# and Δ^{17}O in ferromagnesian GC-IDP in comparison with Wild 2 particles and chondrules in primitive chondrites. Stardust literature data from [12-16]. Chondrites data from [20-24].

References: [1] Brownlee D. E. et al. (2011) *LPSC XLII* #2235. [2] Messenger S. et al. (2015) *LPSC XLVI* #2603. [3] Pepin et al. (2015) *LPSC XLVI* #1706. [4] Langevin et al. (2016) Icarus 271, 76-97. [5] Bentley et al. (2016) Nature 537, 75. [6] Joswiak et al. (2017) MaPS, 52, 8, 1612-1648 [7] Defouilloy et al. (2016) *LPSC XLVII* #1584. [8] Defouilloy et al. (2016) Goldschmidt conference [9]Defouilloy et al. (2017) *EPSL*, 465, 145-154. [10] Nakashima D. et al. (2011) *MaPS*, *46*, 857–874. [11] Heck P. R. et al. (2010) *GCA*, *74*, 497–509. [12] Kita N. T. (2009) *Chem. Geology*, *264*, 43–57. [13] Nakashima D. et al. (2012) *EPSL*, *357*, 355–365. [14]] McKeegan K. D. et al. (2006) *Science*, *314*, 1724-1727. [15] Nakamura T. et al. (2008) *Science*, *321*, 1664-1667. [16] Ogliore R.C. et al. (2015), *GCA*, *166*, 74-91. [17] Nakamura-Messenger K. et al. (2011) *MaPS*, *46*, 1033-1051. [18] Bridges J. C. et al. (2012) *EPSL*, *341-344*, 186-194. [19] Ogliore R.C. et al. (2012) *Astroph. J. Lett.*, *745*, L19. [20] Tenner T. J. et al. (2013) *GCA*, *102*, 226-245. [21] Ushikubo T. et al. (2012) *GCA*, *90*, 242-264. [22] Kita N. T. (2010) *GCA*, *74*, 6610-6635. [23] Connolly H. C. and Huss G. R. (2010) *GCA*, *74*, 2473-2483. [24] Tenner T. J. et al. (2015) *GCA*, *148*, 228-250.