Refractory materials in comet samples

D. J. JOSWIAK1*, D. E. BROWNLEE1, A. N. NGUYEN2, and S. MESSENGER2

1Department of Astronomy, University of Washington, Seattle, Washington 98195, USA
2Robert M. Walker Laboratory for Space Science, ARES, NASA JSC, Houston, Texas 77058, USA
*Corresponding author. E-mail: joswiak@astro.washington.edu

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Abstract—Transmission electron microscope examination of more than 250 fragments, >1 μm from comet Wild 2 and a giant cluster interplanetary dust particle (GCP) of probable cometary origin has revealed four new calcium-aluminum-rich inclusions (CAIs), an amoeboid olivine aggregate (AOA), and an additional AOA or Al-rich chondrule (ARC) object. All of the CAIs have concentric mineral structures and are composed of spinel + anorthite cores surrounded by Al,Ti clinopyroxenes and are similar to two previous CAIs discovered in Wild 2. All of the cometary refractory objects are of moderate refractory character. The mineral assemblages, textures, and bulk compositions of the comet CAIs are similar to nodules in fine-grained, spinel-rich inclusions (FGIs) found in primitive chondrites and like the nodules may be nebular condensates that were altered via solid–gas reactions in the solar nebula. Oxygen isotopes collected on one Wild 2 CAI also match FGIs. The lack of the most refractory inclusions in the comet samples may reflect the higher abundances of small moderately refractory CAI nodules that were produced in the nebula and the small sample sizes collected. In the comet samples, approximately 2–3% of all fragments larger than 1 μm, by number, are CAIs and nearly 50% of all bulbous Stardust tracks contain at least one CAI. We estimate that ~0.5 volume % of Wild 2 material and ~1 volume % of GCP is in the form of CAIs. ARCs and AOAs account for <1% of the Wild 2 and GCP grains by number.

INTRODUCTION

The return of samples from comet Wild 2 by the NASA Stardust (SD) spacecraft provided an unprecedented opportunity to study the range and diversity of materials present in a known Kuiper Belt comet (Brownlee et al. 2006). The discovery of two calcium-aluminum-rich inclusions (CAIs) and other high-temperature objects such as chondrule fragments was a major surprise and has contributed to an increased understanding of the origins of outer solar system bodies (McKeegan et al. 2006; Zolensky et al. 2006; Nakamura et al. 2008; Simon et al. 2008; Matzel et al. 2010; Brownlee et al. 2012; Joswiak et al. 2012; Nakashima et al. 2012, 2015; Ogliore et al. 2012, 2015; Frank et al. 2014; Gainsforth et al. 2015). The intimate details of comet refractory materials reveal direct information on the high-temperature processes involved in making a portion of the solids accreted by comets. The refractory materials found in comet samples are similar to those found in chondrites but detailed comparison between the two sources is challenging because the comet materials are limited in size and number. Although constrained by sampling limitations, comparison of these materials with those in chondrites provides insight into how these materials were distributed across broad regions of the early solar system.

Results to date suggest that the mix of refractory materials accreted by comets may differ from the mix of refractory materials commonly found in chondrites. These differences, as well as variations in refractory in the different chondrite groups, may relate to differences in timing and transport processes in different regions of the early solar system. The comet refractories may include lesser abundances of the most refractory silicates and oxides than found in chondrites.

In this report, we discuss results from analyses of two new CAIs discovered in SD tracks and two CAIs and two other refractory objects from a giant cluster IDP (GCP) which likely has a cometary origin. We also
present new and extended results from the CAIs “Inti” and “Coki,” which were previously reported (McKeegan et al. 2006; Simon et al. 2008; Matzel et al. 2010). All the refractory objects discussed in this paper including “Inti” and “Coki” and those in the giant cluster IDP were discovered and extensively studied at the University of Washington. In addition, we provide new O isotope measurements from one of the previously unreported CAIs from comet Wild 2. This is only the second O isotope measurement obtained from a Wild 2 CAI.

A goal of this work was to determine the abundances and types of refractory materials in comets and compare them with those found in meteorites. Unlike refractory materials in chondrites, which have been studied for decades, the broad properties of refractory materials in comets are not well known. The mineralogical and petrological properties of these materials, observed range of the refractory objects, abundances of small CAIs, along with isotopic compositions and comparison to refractory inclusions in chondrites provide constraints in models of outward transport of early solar system materials. In this paper, mineralogical and chemical information, phase abundances, petrographic structures, O isotopes, and bulk compositions are used to provide a contextual relationship between the comet refractory fragments and the data are compared to comparable objects in chondritic meteorites, and similarities and differences are discussed. Preliminary results of some of the fragments discussed here are given in Joswiak et al. (2013) and Joswiak and Brownlee (2014).

Summary of Previous Studies: Refractory Materials in Comet Wild 2 and IDPs

To date, only two confirmed CAIs from comet Wild 2 have been found. These include “Inti,” a CAI aggregate from track 25, the first refractory inclusion discovered in Wild 2 (McKeegan et al. 2006; Simon et al. 2008) and “Coki,” a mineralogically less refractory object found in track 141 (Matzel et al. 2010). Oxygen isotopes measured on “Inti” showed that it was $^{16}$O-rich similar to unaltered CAIs from primitive chondrites (McKeegan et al. 2006). In “Coki,” O isotopes have not been measured but a $^{26}$Al/$^{26}$Mg isotope study showed no excess $^{26}$Mg in the fragment and a low initial $^{26}$Al/$^{27}$Al ratio suggesting formation at least 1.7 Myr after the onset of CAI formation (Matzel et al. 2010). A third CAI candidate from track 110 was reported by Schmitz et al. (2009) but no specific minerals were unambiguously identified.

Studies of refractory Wild 2 fragments that are not CAIs were done on two probable Al-rich chondrule fragments. The first, from track 154, consisted largely of Al-rich diopside with smaller amounts of pigeonite, forsterite, and enstatite (Bridges et al. 2012). Oxygen isotopes measured on the fragment were distinct from CAIs and thus the authors suggested that the fragment was most similar to Al-rich chondrules. A second fragment, “Bidi,” the terminal particle (TP) from track 130, consisted of subequal amounts of forsterite, anorthite, and clinopyroxene, and based on these minerals, their specific mineral compositions, its bulk composition, O isotope measurements, and other properties, Joswiak et al. (2014) concluded the fragment was most similar to Al-rich chondrules in chondrites.

Two reports have identified Wild 2 forsterite fragments with possible AOA affinities. One fragment, the TP from track 112 was a Fo$_{97-99}$ olivine with an $^{16}$O-rich composition (Nakamura-Messenger et al. 2011). The authors suggested that the forsterite was most akin to primitive AOAs in chondrites whose O isotopic compositions are similar to CAIs. In an O isotope study of SD fragments from several tracks, Nakashima et al. (2012) measured $^{18}$O-rich compositions in three Mn-rich forsterite fragments. Low-iron, Mn-enriched (LIME) forsterites have been identified in AOAs in chondrites (Weisberg et al. 2004) in addition to interplanetary dust particles (IDPs) (Klöck et al. 1989). In both of the Wild 2 studies, however, petrologic links to AOAs are uncertain as no other minerals typically observed in AOAs were found with the AOA forsterite candidates.

IDPs collected in Earth’s stratosphere are also an important source of cometary materials (Brownlee et al. 1995; Bradley 2005; Joswiak et al. 2007; Nesvorný et al. 2010). In particular, highly fragile and porous cluster IDPs have physical and chemical properties consistent with unequilibrated, uncompacted comet regoliths that are largely composed of loosely bound rock and mineral grains, ices, and organic materials. Although the cometary origin of cluster IDPs is not yet proven, a significant body of evidence supports a cometary origin for some of them (see below). Like comet Wild 2, both chondritic porous IDPs (CP IDPs) and cluster IDPs contain diverse rock and mineral fragments many of which appear to have high-temperature origins. Large cluster IDPs such as GCP, the IDP discussed here, contain thousands of ~1–20 $\mu$m mineral and rock fragments and therefore are an important source of cometary material. Large numbers of grains are required for meaningful assessment of refractory components that occur at the percent abundance level in cometary materials.

Christoffersen and Buseck (1986) provided the first report of CAI mineral assemblages from IDPs. Found in a 10 $\mu$m fragment from a 50 $\mu$m cluster IDP named “Spray,” the authors report grains containing diopside + spinel symplectite intergrowths with smaller amounts of fassaite, diopside, perovskite, and other phases. Zolensky (1987) found assemblages of
perovskite + melilite + hibonite and possibly spinel in stratospherically collected IDPs, and in a companion paper, McKeegan (1987) reported on oxygen isotope measurements from the same IDPs and two others which largely showed $^{16}$O excesses demonstrating their extraterrestrial origins. A summary of these refractory minerals from IDPs and several Antarctic micrometeorites which may have links to CAIs in chondrites is given in Greshake et al. (1996).

**SAMPLES AND ANALYTICAL PROCEDURES**

**Nomenclature**

We use the word “fragment” to designate a large (>1 μm), distinct mineral or polymineralic grain. For SD tracks, fragments are isolated in aerogel, and in IDPs, they are discrete minerals or small rocks that are not components of larger grains. Fragments in the giant cluster particle often have associated fine-grained minerals, carbonaceous material, silicate glass, and/or magnetite (from atmospheric entry heating) attached to their exterior surfaces, but these materials are not considered here. Because of the sizable number of tracks that were in the Stardust collector and the large numbers of samples within most bulbous tracks, SD sample names can be somewhat awkward. Here we label the Wild 2 refractory fragments with a compound designation consisting of the track name and fragment number. TuleF4, for instance, is fragment number 4 from a track we named Tule (track 80). Because 23 CAI fragments were studied in track 25, fragments from this track are labeled IntiF1 to IntiF23 and the designation “IntiFx” is intended to be fragment nonspecific in discussions where specific fragments from the track are unimportant. Previous reports on some of the fragments discussed here follow slightly different conventions. In the O isotope study by McKeegan et al. (2006), the TP from track 25 was simply designated “T25 Inti.” This fragment corresponds to IntiF1 in this report. Three fragments from track 25 were also discussed by Simon et al. (2008) and are referred to as “Inti,” “Inti-B,” and “Inti-C.” These fragments are the same as IntiF1, IntiF2, and IntiF3, respectively. Similarly, Matzel et al. (2010) measured $^{26}$Al-$^{26}$Mg isotopes in fragment 2 from track 141 and simply referred to it as “Coki.” Here, the refractory inclusion “Coki” corresponds to CokiF2. In addition, we use the names giant cluster IDP, giant cluster particle, and GCP interchangeably.

**Comet Wild 2 Samples**

Refractory-rich fragments were found in five Stardust tracks (track 25 [C2054,4,25,0,0], track 80 [C2092, 2, 80, 0, 0], track 130 [C2061, 3, 130, 0, 0], track 141 [C2061, 3, 141, 0, 0], and track 172 [C2119, 1, 172, 0, 0]) from a total of 205 individual fragments that we examined in detail in 19 SD tracks. Although several of these fragments have previously been reported on, we provide additional mineralogical observations, bulk chemical compositions, and set them into context with other refractory-rich fragments. Two refractory inclusions which have not previously been reported are CAIs from track 172 (WF216) and track 80 (TuleF4).

A summary of the mineralogy, fragment sizes, textures, and other properties of the five refractory-rich Wild 2 fragments is provided in Table 1. Except for BidiF1, an ARC fragment, all fragments were found in type B tracks (Fig. 1) (Hörz et al. 2006; Burchell et al. 2008). Type B tracks are composed of bulbous cavities with one or more roots and typically contain numerous dispersed mineral and rock fragments. These tracks likely are formed from impactors composed of loose unconsolidated coarse and fine-grained components that separated during capture in silica aerogel (Hörz et al. 2006; Joswiak et al. 2012).

Because of similar mineral assemblages and mineral compositions, it is likely that all the CAI fragments in track 25 were part of a single weakly bonded aggregate particle from Wild 2 that fragmented during capture in silica aerogel. This is supported by potted butt FESEM images of the TP that show a somewhat porous morphology indicating internal structural weaknesses in the prefragmented impacting particle. Only one CAI fragment was found in each of the tracks 80, 141, and 172 even though numerous fragments were examined in each track.

**Giant Cluster IDP**

A total of 50 largely translucent fragments ranging in size from ~3 to 40 μm were hand-picked from a single large cluster IDP. The particle was collected in 1980 on a series of U2 aircraft flights with a cumulative duration of 61 h above 20 km altitude. The particle was collected by the University of Washington program and hardware that preceded the NASA stratospheric IDP collection program. The particle was named the U2-20 Giant Cluster IDP or U2-20 GCP and these names should not be confused with the similar terminology later used by the NASA U2 program that contains particles labeled U220. The entire IDP, thought to have been ~350 μm in diameter before collection, appears to be a large example of a common chondritic composition porous IDP dominated by anhydrous phases. Many fragments originally appeared to be opaque and were only revealed to be transparent after
stripping of encrusted black surface debris with a needle. It is likely to be the largest cluster IDP studied and consists of thousands of submicron and larger particles that “pancaked” during collection, which cover a region >1.5 mm on the collector flag (Fig. 2). Although most of the individual grains that we observed consist of olivine, pyroxene, or olivine–pyroxene mixtures ± sulfides, four fragments (P3-4, P6-14, P1-1, and LT1) are composed of refractory minerals including spinel, anorthite, and Al,Ti clinopyroxenes (diopside/Al-diopside/Al-Ti diopside/augite) (Table 1).

### Sample Preparation

Aerogel keystones from the SD collector containing tracks 25, 130, 141, and 172 were produced at the University of California, Berkeley or the Johnson Space Center curatorial facility (Westphal et al. 2004). We did not have the entire track 80 but were issued an allocation containing only a portion of the bulb region. Entire tracks, or in some cases, portions of tracks, were flattened to <100 µm in thickness between clean glass slides and embedded in acrylic resin. Fragments in each

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**Table 1. Refractory fragments from comet Wild 2 and a giant cluster interplanetary dust particle (GCP).**

| Sample       | Track | Size (µm) | Object Type | Texture   | # slices | Mel | Sp | Cpx | An | Fo | En | Inclusions       |
|--------------|-------|-----------|-------------|-----------|----------|-----|----|-----|----|----|----|------------------|
| Wild 2       |       |           |             |           |          |     |    |     |    |    |    |                  |
| IntiFx       | 25    | 10 × 15<sup>1</sup> | CAI         | Nodular   | 10<sup>1</sup> |   |    |     |    |    |    | RMN, Osb         |
| WF216        | 172   | 2         | CAI         | Nodular   | 8        |   |    |     |    |    |    | Osb              |
| TuleF4       | 80    | -1        | CAI         | Nodular   | 1        |   |    |     |    |    |    |                  |
| CokiF2       | 141   | 3 × 5     | CAI         | Nodular   | 1        |   |    |     |    |    |    |                  |
| BidiF1       | 130   | 4 × 6     | ARC         | Layered?  | 3        |   |    |     |    |    |    |                  |
| Giant cluster particle (GCP) |       |           |             |           |          |     |    |     |    |    |    |                  |
| P3-4         |       | 8 × 13    | CAI         | Nodular   | 5        |   |    |     |    |    |    | Rim, RMN         |
| P6-14        |       | 8 × 10    | CAI         | Nodular   | 1        |   |    |     |    |    |    | Rim, Osb         |
| P1-1         |       | 2 × 7     | AOA/ARC     | Granular  | 1        |   |    |     |    |    |    |                  |
| LT1          |       | 5 × 8     | AOA         | Coarse    | 3        |   |    |     |    |    |    | RMN              |

<sup>1</sup>Terminal particle (IntiF1). Column 6 indicates number of microtome slices used to measure bulk composition.

Mel = melilite; Sp = spinel; Cpx = clinopyroxene; An = anorthite; Fo = forsterite; En = enstatite; CAI = calcium-aluminum-rich inclusion; ARC = Al-rich chondrule; AOA = amoeboid olivine aggregate; RMN = refractory metal nugget; Osb = osbornite.

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**Fig. 1.** Cross sections of five Stardust tracks and locations of refractory (solid circles) and nonrefractory (open circles) fragments. Specific locations of 28 fragments studied from the bottom of the bulb in track 172 not shown. Track numbers and University of Washington track names are shown above and below each track. Track 80 shown at half-scale. Note: The upper portion of track 25 is missing.

**Fig. 2.** Transmitted light image of giant cluster interplanetary dust particle (GCP) on collector flag. The cluster particle consists of thousands of rock and mineral fragments varying from submicron to >40 µm in size and is believed to have been derived from a comet. Pancaked character indicates its fragile and porous nature. All refractory IDP fragments in this study were extracted from this particle.
track were studied optically and large-scale Z-focused mosaic images were constructed. Large isolated fragments or groups of closely spaced fragments were separated into individual pieces by cutting across the tracks with a fresh razor blade. Each piece was mounted on the end of an acrylic cylinder ~1 cm high and successively smaller stepped mesas were cut with the uppermost mesa 100–200 μm in size. The embedded fragments were microtomed into slices typically 70 nm thick. The microtomed sections were placed onto commercially obtained 200 mesh Cu or Au TEM grids coated with 10 nm thick carbon films for transmission electron microscope (TEM) examination.

Because the giant cluster IDP was collected in silicone oil, each hand-picked fragment was washed in hexane, an effective solvent for removing most of the encapsulating silicone oil. Following washing, individual fragments or small groups of fragments were embedded in acrylic or epoxy cylinders for ultramicrotomy. Microtome sections were produced following the same procedures used for the Wild 2 samples. See Matrajt and Brownlee (2006) and Joswiak et al. (2012) for more details of the sample preparation techniques.

Electron Microscopy

Examination of the Wild 2 and GCP fragments was done with a Tecnai TF20 200 keV field-emission STEM equipped with a bright- and dark-field CCD camera and high-angle annular dark-field (HAADF) and secondary electron (SE) STEM detectors. High-resolution lattice fringe images or selected area electron diffraction (SAED) patterns were used to measure atomic spacings in some minerals. Measured d-spacings and associated crystal plane angles obtained from the diffraction patterns were compared with X-ray diffraction data (XRD) of minerals listed in the JCPDS Mineral Powder Diffraction File Data Book (Bayliss et al. 1980) to confirm mineral atomic structures. Camera lengths for the electron diffraction patterns were calibrated using ring patterns from an in-house produced evaporated Al thin film. High-resolution images were calibrated from well-known mineral standards. We estimated that relative errors for HRTEM images and d-spacings in SAED patterns are ~5%, while angular measurements have errors of less than ~2°.

Low-background energy dispersive X-ray (EDX) spectra were obtained from samples placed in a beryllium sample holder capable of tilting on two orthogonal axes. Although most minerals analyzed in this study were robust, broad beam analysis techniques were used to minimize sample damage. This was done by condensing the electron beam to no less than ~50–100 nm in conventional TEM imaging mode. Anorthite was the most susceptible mineral to beam damage thus extra care was taken on this phase. All EDX spectra were collected in conventional TEM mode (except refractory metal nuggets whose spectra were collected in STEM mode) with an EDAX 30 mm² light element detector and quantified using k-factors (Cliff and Lorimer 1975) obtained from mineral and glass standards and a NIST-produced thin film standard (SRM2063a). Background fitting and peak integral measurements were done using EDAX Genesis software with acquisition times typically between 45 and 90 s. Detection limits are usually ~0.1 wt%, while relative errors for major elements are approximately 5% and 25–30% for minor elements except for overlapping element peaks in some spectra (TiKβ overlaps VKα; CrKβ overlaps MnKα) where errors are higher. The refractory metals Os, Ir, Mo, Ru, Pt, and Rh were measured by a standardless technique. Internal Genesis software k-factors for Os-, Ir-, and Pt-L lines and Mo-, Ru-, and Rh-K lines were used in the quantification. Although the accuracy of the measurements obtained on the refractory metal nuggets (RMNs) could not be verified due to lack of a suitable metal standard, the quantified results are expected to be representative due to good background and spectrum peak fits and reasonable internal system k-factors. Slight overlaps, however, between the Os, Ir, and Pt-L line peaks are likely to increase the relative errors in these elements.

Element maps were collected on microtomed sections from eight refractory fragments. The maps were collected from summation of numerous rasters in STEM mode with a resolution of 512 × 400 pixels, ~6–24 nm pixel size, and dwell time of 200 μs. Because bulk compositions are an important part of this study, quantitative bulk compositions of fragments were carefully obtained. This was done by collecting a HAADF STEM image of a microtome slice and then drawing an outline overlaying its perimeter followed by rastering the entire slice within the outline. The Genesis EDX software provides a specific irregular raster mode allowing this capability. In fragments where more than one microtome slice was used, bulk compositions from each slice were normalized to magnification before a total fragment bulk composition was calculated. Although grain sizes are small and some sample loss with the SD particles may have occurred during capture, the bulk compositions measured on the fragments are valid because grain sizes are significantly smaller than rastered areas. The number of microtome sections used to measure bulk composition varied between fragments and is listed in Table 1. To properly measure the bulk compositions, care was taken to ensure that the rasters did not include silica aerogel in SD fragments or fine-grained matrix in GCP fragments.
In SD fragment TuleF4, the rounded morphology of the fragment and its discontinuous pyroxene rim suggested that abrasion during capture in aerogel significantly eroded the rim of the fragment, thus a second bulk composition was calculated assuming the rim was originally composed of $10 \times$ the pyroxene abundance currently observed in the fragment. Similarly, the SD fragment CokiF2 appears to also have suffered rim loss from capture, and bulk compositions of the actual fragment and $5 \times$ additional pyroxene were calculated. The bulk compositions of TuleF4 and CokiF2 were calculated using average mineral compositions, and mineral modes were measured from mineral maps of micromtome sections.

TEM studies were complemented by collecting backscatter images of the potted butts of most of the fragments. This facilitated improved understanding of grain relationships, which can often be disrupted in micromtome sections due to redistribution of shards during ultramicrotomy. The BSE studies were obtained at 12 keV with a JEOL JSM 7000F FESEM or a FEI Sirion FESEM located at the Materials Science and Engineering department and Molecular Engineering and Sciences Institute (formerly Molecular Sciences Nanotech User facility) at the University of Washington.

Oxygen Isotopes: NanoSIMS

Oxygen isotope studies were performed on two micromtomed sections from the 2 μm Wild 2 refractory inclusion WF216 with a Cameca NanoSIMS 50L ion microprobe located at the Johnson Space Center. Prior to the O isotope studies, the micromtome sections were examined in the TEM, and mineral maps showing the locations of all phases were produced. Ion images of $^{16}\text{O}$, $^{17}\text{O}$, $^{18}\text{O}$, $^{28}\text{Si}^+$, and $^{27}\text{Al}^{16}\text{O}^-$ were simultaneously acquired in electron multipliers by rastering a ~0.5 pA 16 keV primary Cs$^+$ ion beam over the samples, and measurements were performed at a mass resolving power of $>10000$ for oxygen. An electron flood gun was used for charge compensation. For slice L, 13 separate image analyses were conducted with at least five measurement planes for each analysis. Following each image analysis, the secondary ion beam was recentered in the entrance slit of the mass spectrometer to ensure optimal reproducibility of each isotopic measurement. It was found that the secondary ion beam did not shift significantly during the analysis of slice L. A second section (slice E) was measured but non-CAI fragments were small, often $<1$ μm in

Isotopic compositions were determined with custom-written image processing software by defining regions of interest in the images that corresponded to the CAI. The software was also used to apply corrections for electron multiplier dead time, quasi-simultaneous arrival, and instrumental mass fractionation. The $^{27}\text{Al}^{16}\text{O}^-$ secondary ion images were used to definitively identify the locations of the CAI in the micromtome slices.

RESULTS

In this section we provide mineralogical and textural details, based largely on TEM studies, of each refractory fragment. Readers are also referred to previous reports in Simon et al. (2008) (track 25, IntiF1, IntiF2, and IntiF3), Matzel et al. (2010) (track 141, CokiF2), and Joswiak et al. (2014) (track 130, BidiF1) for additional details and complementary studies. Representative compositions of minerals from Wild 2 and the GCP refractory fragments are provided in Tables 2–6. Bulk compositions of the refractory fragments are given in Table 7.

Comet Wild 2: Mineralogy and Petrography

Track 25 fragments (IntiFx): CAI

Track 25 was a ~1.5 mm long bulbous track containing a ~$10 \times 15$ μm terminal particle and numerous transparent grains that could only be seen when the track was embedded in acrylic that matched the aerogel refractive index. Visible fragments were distributed throughout the bulb and in several short side-roots (Fig. 1). We studied 32 fragments in the track including the three discussed by Simon et al. (2008). Of these, we found 23 CAI fragments which were primarily composed of spinel, Al,Ti clinopyroxene, and anorthite. SE and BSE images of the TP and some of the other fragments show pronounced internal pore spaces which are likely to have contributed to the fragmentation of the primary impacting Wild 2 particle during collection in silica aerogel. Variable gray-levels in the BSE images show concentric circular minerals indicating the impactor was a cluster of weakly bound nodules (Fig. 3). No Si-rich glass with nanophase Fe,Ni metal and sulfides common to most other type B tracks was observed; however, at least eight non-CAI fragments were found including pentlandite, En$_{98}$ several olivines (forsterite, Fo$_{76}$, Fo$_{80}$, and Fo$_{85}$), and two Kool grains (assemblages of FeO-rich olivines + Na,Cr-rich Ca pyroxenes + Na-rich plagioclase feldspar or Na,$\text{Al}$ silicate glass ± minor spinel believed to have high-temperature origins [Joswiak et al. 2009]). Most of these non-CAI fragments were small, often $<1$ μm in
size and were likely fine grain matrix material originally adhering to the dominant mass of the impacting particle.

To further understand mineral textures and their petrographic relationships, element maps were obtained from fragment 15 (Fig. 4), which was lodged in the middle portion of the bulb. The bright-field image (Fig. 4a) shows a relatively complete microtome section, and composite RGB maps (Figs. 4b and 4c) illustrate that spinel (yellow, Fig. 4b) is largely concentrated toward the fragment center, while Al,Ti clinopyroxene (blue to purple, Fig. 4b) is the principal phase on the exterior. Anorthite (teal, Fig. 4b) is variously present in contact with both spinel and clinopyroxene. Its abundance is low in this fragment compared to others observed in the track. Because spinel contains a moderate amount of Cr (Cr$_2$O$_3$ = 0.67–3.1 wt%), this element (Fig. 4d) clearly delineates the locations of this mineral. The Ti map (Fig. 4e) shows that Ti is concentrated in clinopyroxenes located in the central portions of the fragment. In general, the images suggest a nodular structure to the fragment akin to some fine-grained, spinel-rich inclusions (FGIs) found in CV3 and

Table 2. Representative compositions of spinels (normalized oxide wt%).

| Source | Wild 2 | Wild 2 | Wild 2 | Wild 2 | GCP | GCP | GCP |
|--------|--------|--------|--------|--------|-----|-----|-----|
| Track # | 25 | 172 | 80 | 141 | – | – | – |
| Frag | 15 | 216 | 4 | 2 | – | – | – |
| Name | IntiF15 | WF216$^1$ | TuleF4$^1$ | CokiF2$^1$ | P3-4$^1$ | P6-14 | LTI$^1$ |
| Ref | 1132 | 1156 | 765 | 322 | 1245 | 1335 | 960 |
| SiO$_2$ | b.d. | 3.4 | 1.3 | 19.1 | 24.6 | b.d. | 2.3 |
| TiO$_2$ | 0.25 | b.d. | 0.51 | 0.22 | 0.90 | b.d. | 1.5 |
| Al$_2$O$_3$ | 71.2 | 70.1 | 74.3 | 53.2 | 54.9 | 67.9 | 42.8 |
| Cr$_2$O$_3$ | 1.8 | 2.2 | 3.9 | 3.6 | 17.3 | 5.1 | 22.0 |
| FeO | b.d. | 0.84 | 0.19 | 1.2 | 1.7 | 0.29 | 13.9 |
| MnO | b.d. | b.d. | 0.22 | 0.39 | 0.44 | b.d. | 0.17 |
| MgO | 26.8 | 23.5 | 19.3 | 19.0 | 24.6 | 26.8 | 16.7 |
| CaO | b.d. | b.d. | 0.32 | 2.9 | 0.23 | b.d. | b.d. |
| V$_2$O$_5$ | b.d. | b.d. | 0.29 | b.d. | b.d. | b.d. | 0.72 |

Cation formulas based on three oxygens

| Si | 0.000 | 0.081 | 0.031 | 0.449 | 0.000 | 0.000 | 0.62 |
| Ti | 0.004 | 0.000 | 0.009 | 0.004 | 0.017 | 0.000 | 0.62 |
| Al | 1.994 | 1.954 | 2.076 | 1.474 | 1.641 | 1.926 | 1.38 |
| Cr | 0.034 | 0.041 | 0.073 | 0.067 | 0.347 | 0.096 | 0.475 |
| Fe | 0.000 | 0.017 | 0.004 | 0.024 | 0.035 | 0.006 | 0.317 |
| Mn | 0.000 | 0.000 | 0.004 | 0.008 | 0.009 | 0.000 | 0.004 |
| Mg | 0.949 | 0.830 | 0.680 | 0.667 | 0.932 | 0.960 | 0.679 |
| Ca | 0.000 | 0.000 | 0.008 | 0.073 | 0.006 | 0.000 | 0.000 |
| V | 0.000 | 0.000 | 0.000 | 0.004 | 0.000 | 0.000 | 0.013 |
| Total | 2.982 | 2.922 | 2.886 | 2.770 | 2.989 | 2.989 | 2.960 |

1$^1$Analysis includes minor to moderate overlap with host.
GCP = giant cluster particle; b.d. = below detection.

Table 3. Representative compositions of melilite in Wild 2 fragments (normalized oxide wt%).

| Track # | 25 | 25 | 25 | 172 |
|---------|----|----|----|-----|
| Frag 1 (TP) | 6 | 10 | 216 |
| Name | IntiF1 | IntiF6 | IntiF10 | WF216 |
| Ref | 1398 | 611 | 639 | 1106 |
| SiO$_2$ | 21.6 | 23.6 | 24.9 | 26.0 |
| Al$_2$O$_3$ | 38.5 | 34.6 | 32.9 | 34.7 |
| FeO | b.d. | b.d. | b.d. | b.d. |
| MnO | b.d. | b.d. | b.d. | b.d. |
| MgO | 0.24 | 1.9 | 0.95 | 0.98 |
| CaO | 39.7 | 39.9 | 41.3 | 38.3 |
| Na$_2$O | b.d. | b.d. | b.d. | b.d. |

Cation formulas based on seven oxygens

| Si | 0.980 | 1.133 | 1.072 | 1.168 |
| Al | 2.059 | 1.768 | 1.853 | 1.835 |
| Fe | 0.000 | 0.000 | 0.000 | 0.000 |
| Mn | 0.000 | 0.000 | 0.000 | 0.000 |
| Mg | 0.016 | 0.065 | 0.131 | 0.066 |
| Ca | 1.935 | 2.018 | 1.945 | 1.845 |
| Na | 0.000 | 0.000 | 0.000 | 0.000 |
| Total | 4.990 | 4.983 | 5.001 | 4.914 |
| Ak | 1.5 | 7.0 | 12.4 | 7.3 |

TP = terminal particle; b.d. = below detection.
Table 4. Representative compositions of clinopyroxenes (normalized oxide wt%).

| Source | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP |
|--------|--------|--------|--------|--------|--------|--------|--------|-----|-----|-----|-----|-----|-----|-----|-----|
| Track# | 25     | 25     | 172    | 172    | 80     | 141    | 141    | 130 | -   | -   | -   | -   | -   | -   | -   |
| Frag   | 15     | 15     | 216    | 216    | 4      | 2      | 2      | 1   | TP  | -   | -   | -   | -   | -   | -   |
| Name   | IntiF15| IntiF15| WF216  | WF216  | TuleF4 | CokiF2 | CokiF2 | BidiF1| P3-4| P6-14| P6-14| P6-14| P1-1| P1-1| LT1 |
| Ref    | 1131   | 1399   | 1152   | 1101   | 770    | 327    | 304    | 529 | 1537| 1336| 1322| 1327| 1192| 1187| 932 |

| | SiO₂ | TiO₂ | V₂O₅ | Al₂O₃ | Cr₂O₃ | FeO  | MnO  | MgO  | CaO  |
|-----|------|------|-------|-------|-------|------|------|------|------|
| Track# | 25 | 25 | 172 | 172 | 80 | 141 | 141 | 130 | - |
| Frag  | 15 | 15 | 216 | 216 | 4 | 2 | 2 | 1 | TP |
| Name   | IntiF15 | IntiF15 | WF216 | WF216 | TuleF4 | CokiF2 | CokiF2 | BidiF1 | P3-4 | P6-14 | P6-14 | P6-14 | P1-1 | P1-1 | LT1 |
| Ref    | 1131 | 1399 | 1152 | 1101 | 770 | 327 | 304 | 529 | 1537 | 1336 | 1322 | 1327 | 1192 | 1187 | 932 |

| Source | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP |
|--------|--------|--------|--------|--------|--------|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Track# | 25     | 25     | 172    | 172    | 80     | 141    | 141    | 130 | -   | -   | -   | -   | -   | -   | -   |
| Frag   | 15     | 15     | 216    | 216    | 4      | 2      | 2      | 1   | TP  | -   | -   | -   | -   | -   | -   |
| Name   | IntiF15| IntiF15| WF216  | WF216  | TuleF4 | CokiF2 | CokiF2 | BidiF1| P3-4| P6-14| P6-14| P6-14| P1-1| P1-1| LT1 |
| Ref    | 1131   | 1399   | 1152   | 1101   | 770    | 327    | 304    | 529 | 1537| 1336| 1322| 1327| 1192| 1187| 932 |

| | SiO₂ | TiO₂ | V₂O₅ | Al₂O₃ | Cr₂O₃ | FeO  | MnO  | MgO  | CaO  | Total | Fs | Wo | En |
|-----|------|------|-------|-------|-------|------|------|------|------|-------|----|----|----|
| Track# | 25 | 25 | 172 | 172 | 80 | 141 | 141 | 130 | - | 3.946 | 3.918 | 3.992 | 3.973 | 3.817 | 4.012 | 3.920 | 3.831 | 3.946 | 3.979 | 3.968 | 4.017 | 4.029 | 3.984 | 3.938 | 4.020 |
| Frag  | 15 | 15 | 216 | 216 | 4 | 2 | 2 | 1 | TP | - | 3.946 | 3.918 | 3.992 | 3.973 | 3.817 | 4.012 | 3.920 | 3.831 | 3.946 | 3.979 | 3.968 | 4.017 | 4.029 | 3.984 | 3.938 | 4.020 |
| Name   | IntiF15 | IntiF15 | WF216 | WF216 | TuleF4 | CokiF2 | CokiF2 | BidiF1 | P3-4 | P6-14 | P6-14 | P6-14 | P1-1 | P1-1 | LT1 |
| Ref    | 1131 | 1399 | 1152 | 1101 | 770 | 327 | 304 | 529 | 1537 | 1336 | 1322 | 1327 | 1192 | 1187 | 932 |

| Source | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP | GCP |
|--------|--------|--------|--------|--------|--------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Track# | 25     | 25     | 172    | 172    | 80     | -   | -   | -   | -   | -   | -   | -   | -   | -   | -   |
| Frag   | 15     | 15     | 216    | 216    | 4      | -   | -   | -   | -   | -   | -   | -   | -   | -   | -   |
| Name   | IntiF15| IntiF15| WF216  | WF216  | TuleF4 | -   | -   | -   | -   | -   | -   | -   | -   | -   | -   |
| Ref    | 1131   | 1399   | 1152   | 1101   | 770    | -   | -   | -   | -   | -   | -   | -   | -   | -   | -   |

GCP = giant cluster particle; TP = terminal particle; b.d. = below detection.
Other than sub-10 nm V-bearing osbornite (TiN) inclusions, which are commonly found in at least half of the CAI fragments, perovskite was the most refractory mineral observed. In all 23 CAI fragments examined, we found only a single occurrence of the phase and in only one microtome slice from the terminal particle. An EDX spectrum of the perovskite showed that it is composed largely of Ca, Ti, and O. Small peaks of Mg, Al, and Si in the spectrum were likely from its Al,Ti clinopyroxene host. If the host composition is subtracted from the EDX analysis, a stoichiometry close to CaTiO₃ is obtained. Measurement of the atomic spacing and interplanar angles of several (hkl) planes from a fast Fourier transform (FFT) of a high-resolution image of the phase confirmed the perovskite structure. We also observed melilite (C₂₃Al₁₅₋₁₈.₆) but found it in only a few microtome sections from the TP and two additional smaller fragments located in the bulb, thus melilite was a minor phase in the overall preimpacting particle. Spinel was observed in all CAI fragments in the track and was often found in cores with anorthite and Al,Ti clinopyroxene defining layers toward the exteriors similar to IntiF15 (Fig. 4). We also found CAI fragments that did not fit this pattern and the phases were in more texturally ambiguous

Table 5. Representative olivine and low-Ca pyroxene compositions (normalized oxide wt%).

| Source | Wild 2 | GCP | GCP | GCP | GCP | GCP |
|--------|--------|-----|-----|-----|-----|-----|
| Track # | 130 | - | - | - | - | - |
| Name | BidiF1 | P6-14 rim | P1-1 | LT1 | P3-4 rim | P6-14 rim¹ |
| Frag | 1 (TP) | - | - | - | - | - |
| Ref | 538 | 1471 | 1188 | 926 | 1243 | 1324 |

| Cation formulas based on four (olivine) or three (pyroxene) oxygens |
|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| Si | 0.875 | 0.969 | 0.997 | 1.016 | 0.982 | 0.998 |
| Al | 0.007 | 0.000 | 0.000 | 0.000 | 0.003 | 0.006 |
| Cr | 0.011 | 0.002 | 0.000 | 0.000 | 0.003 | 0.006 |
| Fe | 0.053 | 0.003 | 0.011 | 0.154 | 0.014 | 0.004 |
| Mn | 0.001 | 0.009 | 0.011 | 0.154 | 0.011 | 0.005 |
| Mg | 1.949 | 2.039 | 2.023 | 1.795 | 0.926 | 0.908 |
| Ca | 0.002 | 0.009 | 0.000 | 0.007 | 0.036 | 0.003 |
| Total | 3.007 | 3.030 | 3.023 | 2.982 | 2.001 | 1.994 |
| Fo | 97.4 | 99.9 | 99.4 | 92.1 | 94.9/3.7 | 93.1/6.5 |

¹0.39 wt% TiO₂ not shown.
GCP = giant cluster particle. TP = terminal particle.

Table 6. Compositions of refractory metal nuggets (RMNs) in Wild 2 and GCP fragments (normalized wt%).

| Source | Wild 2 | Wild 2 | GCP | GCP |
|--------|--------|--------|-----|-----|
| Track # | 25 | 25 | - | - |
| Fragment | 1 (TP) | 9 | - | - |
| Name | IntiF1 | IntiF9 | P3-4 | LT1 |
| Ref | 1350 | 619 | 1289 | 722 |
| Host | Anorthite | Spinel | An₉₅ | Fo₀₂ |
| Os | 1.1 | 13.1 | 7.1 | 9.5 |
| Ir | 1.1 | 12.3 | 6.7 | 10.2 |
| Mo | b.d. | 11.9 | 1.6 | 3.8 |
| Ru | 0.8 | 9.2 | 4.2 | 6.8 |
| Pt | 1.7 | 13.0 | 7.5 | 27.2 |
| Rh | b.d. | 1.4 | b.d. | 1.9 |
| Ni | 2.8 | 0.8 | 5.4 | 11.7 |
| Fe | 92.4 | 38.3¹ | 67.4 | 28.9¹ |

¹Fe abundance includes host.
TP = terminal particle.
relationships. Anorthite is ubiquitous and present in all fragments. EDX measurements show a restricted range of compositions varying from An96 to An100. Anorthite Na concentrations were below detection limits in four fragments. Spectral analyses of 10 osbornites showed variable amounts of vanadium with Ti/V ratios varying from 0.9 to 22.8 (atomic). Of these, six were near the chondritic Ti/V ratio of 8.2. Most osbornites were found in anorthite, although a few were observed in spinel and a single occurrence was seen in pyroxene. Chi et al. (2009) examined microtome sections from the terminal particle and reported that most osbornite inclusions were in pyroxenes, although they were also found in anorthite and spinel.

**Fragment WF216 (Track 172): CAI**

Track 172 (Wawa) was a classic 0.92 mm long bulbous track containing an 8 \times 8 \mu m terminal particle, two large fragments in smaller roots, and a large number of dispersed fragments throughout the bulb (Fig. 1). We studied 32 fragments from the track including FeO-bearing enstatite grains. FeO-bearing olivines. Fe-sulfides. Kool grains. LIME forsterites. and the TP, which consists of an enstatite + anorthite + kamacite + Fe-sulfide assemblage. A single small (\sim 2 \mu m) isolated CAI (WF216) was found near the base of the bulb (Fig. 1). The CAI fragment is composed of concentrically zoned minerals with spinel occupying the core followed successively by anorthite and Al-bearing, low-Ti clinopyroxene toward the exterior. A minor amount of melilite (Am_{7.3-15.2}) largely concentrated near the interior was observed in two microtomed sections. Bright- and dark-field images, an HAADF image, and RGB element maps of a microtome slice obtained from the interior of the fragment are shown in Fig. 5. The spinels are Mg,Al-rich and like spinels in track 25 contain moderate amounts of Cr (Cr_{2O3} = 2.2–3.6 wt%). Anorthite (An_{96-98}) displays a euhedral morphology and encloses spinel. Sub-10 nm Ti,V-rich inclusions, which are likely to be the mineral osbornite, are present in both spinel and anorthite (Figs. 5a and 5c and green hotspots in f) but apparently are absent from clinopyroxene and melilite. From 10 EDX analyses, clinopyroxene was found to have a wide variation in Al_{2O3} (2.0 to 20.2 wt%) but low TiO_2 abundances (TiO_2 = 0.20–0.53 wt%). Single CAI nodules like WF216 are not commonly observed in FGIs in chondrites, which typically occur together in large numbers in aggregates. We note that two additional Wild 2 CAIs in this study, TuleF4 and CokiF2, were also observed as single nodules (see below).

**Fragment TuleF4 (Track 80): CAI**

Track 80 is a very large \sim 0.5 cm long bulbous track with three uncharacteristically short roots each containing separate particles (Fig. 1). Discrete fragments from the track bulb were studied by Stodolna et al. (2012) and Joswiak et al. (2012). These authors reported a large diversity of mineral and rock fragments including olivines, pyroxenes, an Al-diopside + augite + enstatite assemblage, Fe-sulfides, magnetite (possibly frambooids), cristobalite, and other phases. During a later examination of additional track fragments from the bulb, we found a LIME forsterite, a Kool grain assemblage, and a single \sim 1 \mu m CAI composed of spinel grains poikilitically enclosed in anorthite, which is in turn rimmed by Al,Ti clinopyroxene showing that the refractory inclusion has a nodular mineral structure (Fig. 6). The entire fragment is encased in a thin veil of compressed aerogel. Measurements on anorthite showed no detectable Na.

| Source       | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | Wild 2 | GCP | GCP | GCP | GCP |
|--------------|--------|--------|--------|--------|--------|--------|--------|-----|-----|-----|-----|
| Track #      | 25     | 172    | 80     | 80     | 141    | 141    | 130    |     |     |     |     |
| Frag         | 1\textsuperscript{1} | 216    | 4      | 4      | 2      | 2      | 1\textsuperscript{1} |     |     |     |     |
| Name         | IntiF1 | WF172  | TuleF4 | TuleF4 | CokiF2 | CokiF2 | BidiF1 | P3-4| P6-14| P1-1| LT1 |
| # sections   | 10     | 8      | 1      | 1      | 1      | 1      | 3      | 5   | 1   | 1   | 5   |
| SiO\textsubscript{2} | 37.5  | 44.1   | 36.6   | 45.4   | 45.9   | 46.7   | 52.0   | 47.9| 51.2| 45.6| 44.7|
| TiO\textsubscript{2} | 0.63  | 0.12   | 0.49   | 2.1    | 1.3    | 3.3    | 0.37   | 0.44| 0.69| 0.72| 0.31|
| Al\textsubscript{2}O\textsubscript{3} | 32.4  | 27.6   | 40.7   | 24.9   | 28.6   | 19.6   | 10.2   | 22.0| 13.2| 21.0| 7.4 |
| Cr\textsubscript{2}O\textsubscript{3} | 0.34  | 0.38   | 0.72   | 0.49   | 0.07   | 0.11   | 0.72   | 0.53| 0.38| 0.15| 0.15|
| FeO          | 0.17   | 0.34   | 0.13   | 0.17   | 0.13   | 0.18   | 0.13   | 1.3 | 1.3 | 1.2 | 0.66|
| MnO          | b.d.   | b.d.   | 0.05   | 0.08   | b.d.   | b.d.   | 0.03   | 0.26| 0.16| 0.18| 0.24|
| MgO          | 11.0   | 7.7    | 5.0    | 8.2    | 3.4    | 8.3    | 25.6   | 11.1| 18.0| 16.8| 35.5|
| CaO          | 18.0   | 19.5   | 16.2   | 18.6   | 20.6   | 21.7   | 9.6    | 16.3| 15.2| 14.7| 4.2 |
| Na\textsubscript{2}O | b.d. | b.d.   | b.d.   | b.d.   | 0.07   | 0.04   | 0.09   | 0.32| 0.31| 0.70|     |

\textsuperscript{1}Terminal particle.
\textsuperscript{2}10 \times average pyroxene composition added.
\textsuperscript{3}5 \times average pyroxene composition added.

GCP = giant cluster particle; #sections = number of microtome sections measured to calculate bulk; TP = terminal particle; b.d. = below detection.

Refractory materials in comet samples 1621
Enclosed within the anorthite are discrete spinel grains up to 300 nm in size (Fig. 6b, yellow and Fig. 6c, red). Although we did not obtain a stoichiometric analysis of the spinel (Table 2), EDX spectra showed they are largely composed of Mg and Al with a moderate amount of Cr ($Cr_2O_3 = 3.9$ wt%) similar to spinels in the CAIs in tracks 25 and 172. Clinopyroxene was observed as a rim on only ~half of the fragment (Fig. 6b, purple) suggesting some of this phase was removed from abrasion during capture in aerogel. This is consistent with its rounded external morphology and encasement in compressed aerogel and similar to the rounded exterior observed in the CokiF2 fragment. The fragments TuleF4 and WF216, just discussed, may be the smallest CAIs ever reported.

**Fragment CokiF2 (Track 141): CAI**

The refractory inclusion CokiF2 was found in the main root midway between the TP and the base of the bulb (Fig. 1). Because the fragment is discussed by Matzel et al. (2010) and Joswiak et al. (2012), we provide only a brief summary here. CokiF2 is a small refractory inclusion, 5 x 3 μm in size, composed of anorthite ($An_{97-100}$) that poikilitically encloses rounded submicron Cr-bearing ($Cr_2O_3 = 3.6-4.3$ wt%) Mg,Al spinel grains (Fig. 7). Sub-
100 nm inclusions composed of V and Nb were observed in the anorthite. A partial rim of Al,Ti clinopyroxene is present around the exterior. EDX analyses show the clinopyroxene is composed of variable Al (Al$_2$O$_3$ = 4.4–19.5 wt%) and Ti (TiO$_2$ = 2.6–10.0 wt%) and in some locations minor amounts of V (V$_2$O$_5$ = 0.0–0.37 wt%) were present. Similar to the CAI in track 80, the rounded shape of the fragment suggests that the pyroxene and possibly anorthite was scoured from the surface during capture in aerogel. A small amount of Al silicate glass, a likely capture product, was found in some regions around the exterior of the fragment. CokiF2 is similar to the other Wild 2 and GCP CAIs in that it has a mineralogically zoned nodular structure.

Fragment BidiF1 (Track 130): ARC

Of the five Wild 2 refractory fragments discussed here, BidiF1 is the only non-CAI fragment and the only fragment from a carrot-shaped (Type A) track (Fig. 1). Impactors that produced type A tracks were composed of competent particles that did not significantly fragment during capture. The ~5 µm terminal particle consists of a subequal mixture of olivine (Fo$_{97-98}$), anorthite (An$_{95-100}$), and high-Ca pyroxene (augite) (Fig. 8). A detailed discussion of the mineralogy, O isotopic composition, and possible origin of BidiF1 is given in Joswiak et al. (2014) and readers are referred to this source for further details. Based on its mineral assemblage, bulk composition, O isotopic ratios, and other factors, the fragment is most likely related to an Al-rich chondrule.

Giant Cluster Particle (GCP): Mineralogy and Petrography

P3-4: CAI

The refractory inclusion P3-4 is the second largest CAI in this study and was extracted from the giant cluster particle. The CAI consists of nodules of

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**Wild 2: IntiF15**

Fig. 4. (a) Bright-field image, (b–c) composite RGB element maps and (d–f) individual Cr, Ti, and Fe element maps of a microtome section of one Wild 2 fragment from track 25 (IntiF15). In (b), yellow regions represent spinel, blue to magenta regions denote clinopyroxenes, and cyan regions signify anorthite. A partial cocoon of compressed aerogel (green) is visible surrounding the lower portion of the fragment in (c). The Fe element map (f) demonstrates that the primary phases in IntiF15 are Fe-free. The small amount of Fe, visible in a few locations along the periphery is present in sulfides and not considered primary. Minor amounts of the melilite observed in three fragments and the trace amount of perovskite found in a single microtome slice are not present in this fragment.
anorthite which are surrounded by Al,Ti clinopyroxene and then low-Ca pyroxene (En95Wo4) in a discontinuous rim (Fig. 9). Submicron spinel grains are poikilitically enclosed by the anorthite. The concentric mineral layers and the presence of voids between the nodules suggest independent formation of each nodule similar to the Wild 2 CAI Inti.

With the exception of enstatite on the rim, the major minerals closely mimic those observed in the Wild 2 CAIs. One difference, however, is that the spinels in P3-4 contain significantly higher Cr contents (Cr2O3 = 12.3–17.3 wt%) and small amounts of Ti and Fe were also observed (Table 2). RGB maps from microtome sections show that anorthite (An96) is the dominant phase comprising each nodule (Fig. 9c). In addition to the spinel grains, refractory metal nuggets (RMNs), ~50 nm in size, were observed in the anorthites (Fig. 9a, inset) (Joswiak and Brownlee 2014). The RMNs are largely composed of Fe and Ni but have moderate amounts of Os, Ir, Pt, Mo, and Ru (Table 6). A discontinuous rim of Al,Ti clinopyroxene is directly in contact with the anorthite, and Al and Ti are variable (Al2O3 = 8.8–16.7 wt%; TiO2 = 1.4–6.4 wt%). In an electron energy loss spectroscopy study in the TEM, Joswiak and Brownlee (2015) showed that the Ti valence states in the clinopyroxenes are variable suggesting that the nodules were formed in environments of varying oxygen fugacity. Surrounding the exterior of each nodule is a partial accretionary rim of enstatite (En95-97, Wo0.8-3.7) which is variously in contact with anorthite or clinopyroxene. The fragment itself is partially wrapped in fine-grained Fe-rich matrix material.

**P6-14: CAI**

The 8 × 10 μm fragment P6-14 is an aggregate of fine-grained nodules composed of concentric minerals encased in Fe-rich fine-grained matrix (Fig. 10) and is mineralogically and texturally similar to P3-4, just...
discussed. Up to seven individual nodules consisting of anorthite with submicron spinel inclusions and surrounded by Al,Ti clinopyroxene and forsterite (Fo99+) ± enstatite rims were observed in the fragment (Fig. 10). Anorthite (An$_{95-98}$) comprises a significant portion of the interior of most nodules and is in direct contact with clinopyroxene, forsterite, and enstatite. Like P3-4, the Mg-Al spinels contain significant Cr (Cr$_2$O$_3$ = 5.1–16.0 wt%) and minor Fe (FeO < 0.29 wt%). We observed ~25–100 nm osbornite inclusions in the anorthite (Fig. 10c, inset). This is the only refractory inclusion from the giant cluster IDP where osbornite was found; unlike P3-4, no RMNs were observed. Highly variable Al and Ti abundances were measured in the clinopyroxenes ranging from Al$_2$O$_3$ = 0.93 to 30.2 wt% and TiO$_2$ = 0.18 to 22.0 wt%. In some analyses, small amounts of V (V$_2$O$_3$ < 0.95) were also present. In a Ti valence study of the Al,Ti clinopyroxenes, Ti$^{3+}$/ (Ti$^{4+}$ + Ti$^{4+}$) ratios were found to vary from 0.11 to 0.73 indicating the nodules may have formed under widely different oxidation conditions (Fig. 10e) (Joswiak and Brownlee 2015). Although the textural and mineralogical properties of P6-14 are very similar to P3-4, a major difference is that the accretionary rim on P6-14 is composed of both olivine and low-Ca pyroxene. Both olivine and pyroxene have low Fe concentrations (Fo$_{97.5-99.5}$, ave. Mg/(Fe + Mg) in enstatite = 0.99; Table 5). The Mn/Fe (atomic) ratios in all forsterite and enstatite analyses are >1 and therefore are similar to LIME silicates in chondrites, which were suggested to have condensation origins in the solar nebula (Klöck et al. 1989).

P1-1: AOA/ARC

This fragment from the giant cluster particle is unlike those previously discussed as it does not have a nodular mineral structure and spinel is absent. The fragment, ~2 × 7 μm in size, is composed of subequal amounts of forsterite, anorthite, and clinopyroxene (Fig. 11) and mineralogically resembles the SD fragment BidiF1, which is a likely Al-rich chondrule fragment (Joswiak et al. 2014). Like BidiF1, olivine in P1-1 is forsteritic (Fo$_{97.5-99.5}$, while anorthite contains small to moderate amounts of Na$_2$O (An$_{94.9-97.8}$). Both phases are in contact with one another and with clinopyroxene. In the anorthite, small amounts of MgO (<0.28 wt%) and FeO (<0.28 wt%) were measured and no inclusions were observed. Apparent solar flare tracks were observed in one grain. Clinopyroxene has variable Al$_2$O$_3$ (4.9–13.5 wt%) and TiO$_2$ contents (0.12–3.6 wt%). Based on its mineral assemblage and bulk.

Fig. 6. Bright-field image and composite element maps of fragment TuleF4 found in the bulb wall in track 80. In (b), yellow regions correspond to spinel grains which are surrounded by anorthite (cyan) and a discontinuous rim of Al,Ti clinopyroxene (purple). Fragment is partially enclosed in compressed aerogel (green regions in c). A few ~100 nm Fe-sulfides (blue) are enclosed in the compressed aerogel in the upper rim. The rounded shape of the fragment and its discontinuous pyroxene rim suggests additional Al,Ti clinopyroxene was present on the impacting particle but was eroded away during capture.
composition (discussed later), P1-1 is likely to be either an AOA or Al-rich chondrule fragment.

**LT1: AOA**

This $5 \times 8$ μm fragment from the giant cluster IDP is primarily composed of a compact assemblage of olivine, anorthite, and Al,Ti clinopyroxene; contains numerous inclusions of spinel and Fe,Ni metal; and minor amounts Na and Al silicate glass (Fig. 12). The mineralogical diversity, mineral compositions, and bulk composition of LT1 are distinct from the other fragments in this study.

EDX analyses and element maps show that olivine (Fo$_{88.95}$) is the modally dominant phase. Diffraction patterns obtained on the mineral confirmed the olivine structure. At least two types of inclusions are present in the olivine. The larger inclusions, which are relatively numerous, are $\sim$100 nm Mg-Al-Cr-Fe-rich spinel grains (Table 2), whereas smaller $\sim$50 nm Fe,Ni metal inclusions contain variable amounts of Os, Ir, Ru, Mo, Pt, and Rh and are best described as refractory metal nuggets (Table 6). In situ studies of refractory metal nuggets in chondrites indicate that RMNs are largely observed in CAIs (Schwander et al. 2015). To our knowledge, RMNs have not been reported in AOA.s. However, Hewins et al. (2014) reported Pt-Fe-rich RMNs in olivine in Al spinel+forsterite objects from the CM chondrite Paris. We note that at least one RMN from LT1 is dominated by Fe and Pt. Small amounts of phosphorus were also observed in some of the Fe,Ni metal inclusions.

The spinel grains are compositionally dominated by Mg, Al, Cr, and Fe (Table 2), and also contain small amounts of Ti, V, and Mn. In one spinel inclusion observed along the edge of its olivine host, a small amount of phosphorus was unexpectedly found. In another region along the periphery of the fragment, a 50 nm wide amorphous rim is present on Fo$_{92}$ olivine (Fig. 12b, inset). Detailed examination of the rim shows that it is composed of an outer 10 nm wide Si- and O-rich layer which overlies a $\sim$40 nm wide inner rim with $\sim$Fo$_{92}$ composition. Embedded within this inner rim are discontinuous patchy regions rich in Fe or Al and Cr. Although the exact origin of the rim is unclear, the outermost Si- and O-rich layer may be a result of alteration during exposure to solar radiation.

Al,Ti clinopyroxene is the second most abundant phase in the fragment. EDX analyses show that it is generally low in Fe (average FeO = 0.18 wt%) and
Fig. 8. a) Bright-field image of a microtome slice from probable Al-rich chondrule BidiF1, the terminal particle from track 130 (Joswiak et al. 2014). The fragment consists of Fo97 olivine, anorthite, and Al,Ti clinopyroxene which is partially surrounded by a cocoon of compressed silica aerogel. Bright interior regions show plucking during microtoming. b) Composite RGB element map delineating Fo97 olivine (red), Al,Ti clinopyroxene (purple), and anorthite (teal). Compressed silica aerogel—which does not contain Mg, Al, or Ca—is not visible in this image but can be seen in (a). Rounded anorthite exterior on left-hand side of image suggests rounding from abrasion during capture. An = anorthite, Cpx = clinopyroxene, Fo = forsterite.

Fig. 9. Bright- and dark-field images and composite element map of a microtome slice of refractory inclusion P3-4 from giant cluster IDP (GCP). Element map (c) shows that the particle is composed of two concentrically layered nodules which are composed of anorthite cores (cyan) surrounded by Al,Ti clinopyroxene (blue to purple). Anorthite cores contain submicron inclusions of spinel grains and ~50 nm refractory metal nuggets (inset in [a]). A discontinuous accretionary rim of enstatite grains (red) surrounds the nodules. A region of fine-grained Fe-rich silicate material is present on the lower left side of the nodules. Sp = spinel.
contains variable Al (Al$_2$O$_3$ = 1.9–8.8 wt%) and Ti (TiO$_2$ = 0.69–5.6 wt%). Small amounts of V and Cr were observed in two spectra. One triple junction from the intersection of three clinopyroxenes was noted.

Anorthite is low in abundance compared to olivine and clinopyroxene. Compositionally, we measured relatively low Na$_2$O contents in the phase (Na$_2$O = 0.20 wt%, An$_{98}$) except in one location where anorthite was juxtaposed with Al,Ti clinopyroxene and forsterite/enstatite rims. Submicron spinels occur as inclusions in the anorthite. Ti EELS spectra (A–E) obtained on Al,Ti clinopyroxenes in nodules compared to Ti$^{3+}$ and Ti$^{4+}$ standards. The L3 and L2 fine structures demonstrate that large variations in Ti$^{3+}$/Ti$^{4+}$ ratios are observed within single nodules and between nodules that are in close proximity to one another. Capital letters with EELS spectra correspond to lettered solid white circles in (d) (after Joswiak and Brownlee 2015). Sp = spinel, f.g.r. = fine-grained region.

Although the fine-grained material is in contact with the major phases, it does not appear to have reacted chemically with these minerals and is likely to have a different origin than the primary fragment. The mineral assemblage, mineral compositions, and bulk composition suggest that LT1 may be related to an altered AOA (fragment).

Oxygen Isotopes

Oxygen isotopes were measured in fragment WF216, the second CAI from comet Wild 2 in which O isotopes have been obtained. The O isotopic composition from a microtome slice from WF216 is plotted in Fig. 13 along with measured isotopic compositions of the Wild 2 CAI IntiF1 (McKeegan
et al. 2006) and spinel + anorthite + Al,Ti clinopyroxene-rich FGIs from CV3 chondrites (Aléon et al. 2005). The O isotopic composition from WF216 ($\delta^{17}$O = $-26 \pm 12\%_o$ and $\delta^{18}$O = $-22 \pm 6\%_o$), which plots along the CCAM line at intermediate $\delta^{18}$O and $\delta^{17}$O values, was obtained from a mixture of spinel, anorthite, and Al,Ti clinopyroxene. In the assemblage, spinel is relatively minor compared to the other phases. The small amount of melilite observed in some portions of the CAI was not present in the microtome section in which the O isotopes were obtained.

**DISCUSSION**

**Giant Cluster Particle: Evidence for a Cometary Source Region**

The giant cluster IDP, which is composed of thousands of individual submicron to $\sim$40 $\mu$m grains, has many properties similar to comet Wild 2 materials and CP IDPs many of which are likely to have cometary origins (Brownlee et al. 1995; Wurm and Krauss 2005; Joswiak et al. 2007; Ishii et al. 2008;
Nesvorný et al. 2010). For instance, GCP was clearly a very fragile, porous collection of grains as is evident from its spray of fragments disseminated across a large area of the collector flag (Fig. 2). Optical, TEM, and SEM observations of 50 solid fragments from the particle have shown that the IDP is mineralogically diverse. We have observed fragments dominated by single minerals (olivines, pyroxenes, and Fe,Ni sulfides) to polymineralic grains largely containing mixtures of silicates and/or silicates+sulfides. In some fragments, textures and Fe/Mg ratios of coexisting pyroxenes and olivines indicate equilibrated assemblages, while the silicates in other fragments appear to be chemically unequilibrated. Many studies of comet Wild 2 fragments have also shown a diverse and often unequilibrated range of mineral and rock fragments (Zolensky et al. 2006, 2008; Nakamura-Messenger et al. 2011; Joswiak et al. 2012). In GCP, large grains are often partially encased in anhydrous Fe-rich fine-grained matrix or are associated with carbonaceous material or silicate glass and GEMS (glass with embedded metal and sulfides) (Keller and Messenger 2011; Bradley 2013) which appear to be unrelated to the coarse grains they enclose.

Further evidence tying GCP to a cometary source includes the following. (1) Uncorrelated Mn/Fe ratios of FeO-rich olivines mimic the Mn/Fe ratios in FeO-rich olivines observed in Wild 2 olivines (Frank et al. 2014; Brownlee and Joswiak 2017). Unlike FeO-rich olivines from chondrules from unequilibrated CO or ordinary chondrites where Mn and Fe are distinctly correlated and loosely correlated Mn/Fe trends observed in unequilibrated CR chondrites (Berlin et al. 2011), the Mn/Fe ratios found in the FeO-rich olivines from GCP (and Wild 2) show no correlations. This suggests that FeO-rich olivines from GCP and Wild 2 were not derived from any specific chondrite region but represent a broad mix of olivine material from diverse regions in the nebula. (2) Kool grain assemblages are observed in numerous GCP fragments. Kool grains—assemblages of FeO-rich olivines + Na,Cr-rich Ca pyroxenes + albitic feldspar or Na,Al silicate glass ± minor spinel—are chemically and mineralogically unique grains likely to have igneous origins which so far have only been observed in Wild 2 samples and CP IDPs (Joswiak et al. 2009) and are absent in chondrites. (3) GCP have similar external morphology to grains collected in situ at low-impact velocities with the Cosima dust collector on the Rosetta spacecraft (Schulz et al. 2015). Although compositions and mineralogy were not determined, images of grains derived from the surface of comet 67P/Churyumov-Gerasimenko demonstrate highly fragile and probable porous aggregate structures. (4) GCP have high presolar silicate abundance. Ion images with anomalous N and C and estimated presolar abundances obtained from NanoSIMS measurements of O isotopes from microtome slices of GCP fragments are similar to primitive IDPs and consistent with a primitive parent body source such as a comet (Messenger et al. 2015). (5) Noble gas measurements from GCP fragments are similar to Wild 2 fragments (Pepin et al. 2015). (6) V-rich TiN inclusions are observed primarily in anorthites present in refractory assemblages in both Wild 2 and GCP but not in...
chondrites. (7) Enstatite whiskers are present in GCP. Enstatite whiskers are direct nebular condensates commonly found in CP IDPs (Bradley et al. 1983) and recently in Wild 2 samples (Stodolna et al. 2014). (8) Carbon-rich nature of GCP and presence of GEMS in some fragments (unpublished data) similar to CP IDPs. Thus, numerous physical, chemical, and mineralogical properties of GCP are consistent with its derivation from a comet.

Size Biases of Refractory Comet Samples

Burchell et al. (2008) showed that the frequency of small particles impacting the Stardust cometary collector tray greatly exceeded larger ones. Based on detailed examinations of 20 aerogel cell blocks, the authors found many more 10 μm impacting particles than 100 μm particles. In our study, the largest Wild 2 fragments examined were ~15 μm and most were significantly smaller ranging down to the ~1 μm nodular CAI from track 80 (fragment TuleF4) (Table 1). Similar size grains were observed with the refractory fragments extracted from the giant cluster particle. Thus, any refractory inclusions or other refractory materials captured by the Stardust spacecraft or extracted from the giant cluster particle are limited to small sizes. In chondrites, CAIs vary from micron-scale to >1 cm in size; however, most CAI studies have focused on larger (>100 μm) objects. Accordingly, it is not straightforward to compare the refractory materials in the comet samples with those in chondrites. Typical intact coarse-grained Type A, B, and C CAIs, Al-rich chondrules, and AOAs from chondrites would be absent in the collected comet materials because they are too large. However, small refractory objects in chondrites do exist. For instance, nodules in fine-grained inclusions (FGIs), CAIs < 50 μm, and individual nodular AOAs are objects whose sizes can approach the sizes of the comet samples (Ruzicka 1996; Lin and Kimura 1998; Krot et al. 2004a). It is possible that small CAIs and fragments of CAIs and other refractory materials, analogous to the refractory comet grains, are present in chondrite matrix and are simply unknown due to a lack of studies.

An important size bias that should be considered with comet Wild 2 samples is the fragmentation and abrasion that many of the impacting particles experienced during hypervelocity capture in aerogel, which reduces the sizes of the initial captured particles. Some fragments, however, will suffer little to no physical degradation during capture such as impactors composed of competent single mineral or rock grains. This is observed in some smaller carrot-shaped (Type A) tracks where little to no debris is present along track walls and whose terminal particles are not highly rounded.

Detailed examinations of SD track particles from this and other studies that we have done (Joswiak et al. 2012), have shown that many of the fragments within bulb and/or root regions were pieces of single
previously larger coherent mineral or rock grains that broke apart during track deceleration. For example, studies of the >20 CAI fragments from track 25 suggest that the observed fragments are representative of their actual mineralogy before collection as most have very similar mineral textures and assemblages. It is clear, however, that many Wild 2 impactors did lose rim material from abrasion during capture thus the observed sizes of these fragments are necessarily minimum values.

These effects can impact the ability to accurately measure bulk compositions in the Wild 2 samples. Errors in bulk composition would likely show a fragment to be artificially more refractory because it is often the less refractory phases in the samples, particularly olivine or Al,Ti clinopyroxenes, which would be lost from abrasion during capture as these phases typically reside toward the fragment exteriors. Because of these effects, additional bulk compositions were calculated on fragments TuleF4 and CokiF2 by adding additional average Al,Ti clinopyroxene compositions (see above) where textural evidence suggested that some Al,Ti clinopyroxene was lost during capture.

**Moderate Refractory Character of Refractory Wild 2 and GCP Fragments**

Comparison of the most refractory grains from comet Wild 2 and GCP to mineralogically comparable objects in chondrites demonstrates that the refractory fragments in Wild 2 and GCP do not include the entire range of refractory minerals observed in chondrites (Fig. 14). The six cometary CAI fragments (IntiFx, WF216, TuleF4, CokiF2, P3-4, and P6-14) are mineralogically dominated by anorthite + Al,Ti clinopyroxene with lesser amounts of spinel, minerals that are not the major constituents of the highest temperature CAIs in primitive chondrites. In five of the six CAI fragments, spinel is typically observed as inclusions, largely in anorthite. Melilite, a common phase in many Type A and B CAIs (MacPherson 2005), is only observed in minor quantities in two of the comet fragments and the more refractory perovskite is a trace constituent found solely in the TP of track 25 (IntiF1). The most refractory minerals found in CAIs in chondrites, including hibonite, grossite, and corundum, are conspicuously absent in the Wild 2 and GCP CAIs.

Mineral compositions in the Wild 2 and GCP CAIs are also reflective of more moderate temperatures. Compared to Type A and B CAIs, spinels in the Wild 2 and GCP fragments have elevated Cr$_2$O$_3$ contents and are most comparable to spinels in some Type C CAIs, FGIs, and ARCs (Fig. 15). Chromium is a moderately refractory element (transitional to refractory), which suggests that the spinels either equilibrated at temperatures below the Cr condensation temperature (1296 K; Lodders 2003) or reacted with lower temperature Cr-bearing nebular gas. Additionally, the A$_2$O$_3$ and TiO$_2$ contents of many clinopyroxenes in the comet CAIs are somewhat lower than clinopyroxenes in Type A and B CAIs (Fig. 16).

A similar pattern is observed with the refractory metal nugget inclusions present in the IntiFx and P3-4 fragments compared to RMNs in CAIs in chondrites. Hosted by anorthite (or olivine in LT1), RMNs in the Wild 2 and GCP CAI fragments have variable amounts of Os, Ir, Mo, Ru, Pt, and Rh but are compositionally dominated by Fe and Ni (Table 6) (Joswiak and Brownlee 2014). This is in contrast to RMNs in primitive chondrites where RMNs and their hosts are more refractory (Berg et al. 2009; Harries et al. 2012; Hewins et al. 2014) than counterpart RMNs and hosts in the comet CAI fragments. When plotted on the condensation equilibrium curves of Berg et al. (2009), equilibration temperatures of ~1380–1510 K are obtained for the comet RMNs compared to ~1490–1610 K for the RMNs in chondrites. Thus, the RMNs observed in the comet samples are less refractory than RMNs in CAIs in chondrites (Fig. 14). The lower condensation temperatures of RMNs observed in the Wild 2 and GCP samples are consistent with the onset of significant Fe condensation compared to the more refractory metals.

Paradoxically, osbornite, a highly refractory mineral, is commonly observed in refractory fragments from Wild 2 and GCP, including IntiFx, WF216, and P6-14 (Table 1). Although typically ~10 nm in size, it has been observed as inclusions up to 75 nm (Fig. 10c). In the large majority of osbornites that we analyzed, the Ti/V ratios were often variable with Ti abundances exceeding V. In some osbornites, however, we found that the Ti/V ratios were ~chondritic (Ti/V = 8.8, atomic) although in at least one inclusion the V concentration was above Ti (V/Ti = 1.1, atomic). The mineral is exceedingly rare in chondrites but was found in a CAI in the Isheyevo CH/CL-like meteorite (Grokhovsky 2006; Meibom et al. 2007). Osbornite was also observed in the CH chondrite ALH 85085 where it was associated with spinel and diopside, a possible CAI (Weisberg et al. 1988) and in CAIs in some E3 chondrites (Fagan et al. 1999). Unlike the TiV nitrides in the Wild 2 and GCP samples, it was C-bearing, significantly larger, and found as a polycrystalline aggregate ~30 × 60 µm in size. To our knowledge, it has not been reported as ~10 nm inclusions in chondrite minerals. Thermodynamic calculations indicate that osbornite was formed by condensation in the solar system.
nebula at ~2000 K and in environments with enhanced C/O ratios (Ebel 2006). The common presence of this unusual high-temperature phase may be attributed to formation of ~10 nm condensates that preceeded the formation of the major Wild 2 refractory phases. Accordingly, its presence does not actually contradict the observation that most Wild 2 CAIs are only moderately refractory in character, but it does provide evidence that comets contain exotic condensates that appear to have preceeded formation of more common refractory phases.

The Al-rich chondrule fragment BidiF1 and AOA/ARC object P1-1 are also moderately refractory. These fragments are composed of forsterite + anorthite + Al, Ti clinopyroxenes, and do not contain spinel or melilitie and are mineralogically similar to ARCs from chondrites. In chondrites, ARCs are assemblages of olivine + Al,Ti-rich clinopyroxene + Ca-rich plagioclase feldspar ± spinel ± low-Ca pyroxene ± silicate glass and are texturally similar to ferromagnesian chondrules and likely formed in the nebula by crystallization of liquid droplets (MacPherson and Huss 2005; Tronche et al. 2007). ARCs define a trend between type C CAIs, the least refractory type of CAI in chondrites, and ferromagnesian chondrules involving solid–gas chemical reactions of their precursor grains in the nebula (Krot et al. 2002; MacPherson and Huss 2005; Nagahara and Ozawa 2008; Nagahara et al. 2008).

The fragment LT1 from GCP, a forsterite-rich grain which contains modest amounts of anorthite and Al,Ti clinopyroxene + inclusions of Al-Mg-Fe-Cr spinel and Fe,Ni metal, is compositionally and mineralogically the least refractory of the nine comet fragments in this study. Mineralogically, LT1 resembles AOAs, which are observed in all carbonaceous and some ordinary chondrite groups (Scott and Krot 2005). In chondrites AOAs are principally composed of olivine, clinopyroxene, anorthite, and spinel ± minor kamacite, melilite, or perovskite (Komatsu et al. 2001; Krot et al. 2004b). Pristine AOAs generally are 16O-rich and have compositions and mineral textures consistent with condensation (Krot et al. 2004b; Weisberg et al. 2004; Aléon et al. 2005; Itoh et al. 2007; Ruzicka et al. 2012), while altered AOAs may contain anorthite, FeO-rich olivines, FeO-rich pyroxenes, and a large number of other phases (Krot et al. 2004b).

In chondrites, refractory objects including CAIs, ARCs, and AOAs can be arranged, based on bulk
composition, from the most to least refractory as follows: hibonite-rich inclusions → grossite-rich inclusions → Type A CAIs → Type B CAIs → Type C CAIs/FGIs → ARCs → AOAs. Refractory grains found in Wild 2 and GCP only comprise materials from the three least refractory categories in this sequence, i.e., FGIs, ARCs, and AOAs, indicating that the Wild 2 and GCP collections do not contain the entire range of refractory materials that the nebula produced and that are found in chondrites.

Wild 2 and GCP CAIs Compared to Fine-Grained Inclusions (FGIs)

Most of the CAIs from comet Wild 2 and GCP have distinctive textural and mineralogical properties consisting of nodules of very fine-grained minerals composed of anorthite and spinel in cores surrounded by outer layers of Al,Ti clinopyroxene ± forsterite ± enstatite. Fragments WF216, TuleF4, and CokiF2 are single nodules, and P3-4 and P6-14 are aggregates of several nodules cemented together, while the IntiFx fragments are likely to have been an aggregate of several dozen nodules prior to capture by the Stardust spacecraft. These properties—nodular textures, aggregate structures, core to rim mineral sequences, and grain sizes—are similar to many fine-grained, spinel-rich inclusions observed in CV3 and other chondrites. In the CV3 chondrite Efremovka, Krot et al. (2004a) described an FGI (E42) that is composed of a large number of <40 μm nodules which have spinel cores that are surrounded by anorthite and Al,Ti clinopyroxene. Some melilite is present in the FGI mantle with spinel and Al-diopside and tiny perovskite grains are present in the spinel in the core. The FGI is surrounded by a thin forsterite rim. In the reduced CV3 chondrites Efremovka and Leoville, FGIs in general consist of aggregates of nodules with concentrically arranged minerals composed of spinel-rich cores (± hibonite ± perovskite), which are surrounded by various amounts of monomineralic layers of diopside, melilitie, and anorthite (Krot et al. 2004a). In a 1.8 × 2.4 mm anorthite + spinel-rich inclusion in the anomalous carbonaceous CV3 chondrite Ningqiang, Lin and Kimura (1998) found abundant 6–15 μm grains composed of spinel cores, which were successively rimmed by layers of anorthite and Al,Ti clinopyroxene. In other portions of the anorthite + spinel-rich inclusion, 20–50 μm nodules were composed of spinel cores with successive layers of melilitie, anorthite, and clinopyroxene. The authors interpreted the anorthite and pyroxene as alteration products from reactions of melilitie with nebular gas. Similar nodules in Kainsaz, a CO3 chondrite, were reported in an extremely fine-

![Fig. 15. Histograms of Cr$_2$O$_3$ contents in spinels from refractory materials in comet Wild 2 and the giant cluster particle (GCP) compared to spinels in Types A, B, and C CAIs, fine-grained, spinel-rich inclusions (FGIs), and Al-rich chondrules (ARCs). Data sources: Type A CAIs—Grossman (1975) and MacPherson and Grossman (1984); Type B CAIs—Grossman (1975), Kurat et al. (1975), Kornacki and Wood (1985), McGuire and Hashimoto (1989), Davis et al. (1991), and Caillet et al. (1993); Type C CAIs—Wark (1987), Lin and Kimura (1998), and Krot et al. (2007); FGIs—Holmberg and Hashimoto (1992) and Krot et al. (2004a); ARCs—Sheng et al. (1991), Krot et al. (2002), and Krot and Keil (2002).]
grained porous inclusion by Holmberg and Hashimoto (1992). Like the nodules in the previous studies, those in the Kainsaz inclusion are composed of spinel grains with mantles of melilite, anorthite, and Al,Ti clinopyroxene. In the Paris meteorite, which is believed to be the most pristine CM chondrite studied to date, Hewins et al. (2014) described nodular CAIs with nearly identical mineral structures as FGIs. CAI 268, for instance, is a concentric 160 μm refractory inclusion composed of a spinel core (+inclusions of hibonite, perovskite, and grossite) with a mantle of gehlenite which is overlain by anorthite and diopsid/fassaita.

The Wild 2 and GCP CAIs have significant textural and mineralogical similarities to FGIs in chondrites. Compared to the comet CAIs, the FGIs generally are somewhat more refractory as they contain higher proportions of minerals that condensed at high temperatures including hibonite, perovskite, and melilite. FGIs also contain higher proportions of spinel, generally, than the comet samples. These more refractory minerals then reacted with nebular gas at lower temperatures to produce anorthite and Al,Ti clinopyroxene (Krot et al. 2004a). Accretionary forsterite rims were later added in Mg-rich nebular gas. In the Wild 2 and GCP CAIs, anorthite and Al,Ti clinopyroxene are the modally dominant phases. The minor amounts of melilite, rare perovskite (IntiF1 only), and perhaps the spinel inclusions are likely surviving relict minerals. The absence of hibonite in the Wild 2 and GCP CAIs shows that the comet CAIs, if formed in a manner similar to FGIs, must have been altered to a greater extent than the nodules in their FGI counterparts. Because the nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers. Individual nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers. Individual nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers. Individual nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers. Individual nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers. Individual nodules in the Wild 2 and GCP fragments occur either singly or in small numbers (assuming the Wild 2 nodules were not in large aggregates prior to capture), they would have been directly exposed to nebular gas more than nodules in the FGIs, most of which were largely shielded due to their aggregation in large numbers.

![TiO2 versus Al2O3 concentrations (oxide wt%) in Ca-rich pyroxenes from refractory materials in comet Wild 2 (red and orange symbols); the giant cluster particle (green symbols); and various moderately refractory objects in chondrites (blue symbols) including Type C CAIs, FGIs, ARCs, and AOAs. Data sources: Type C CAIs—Wark (1987), Krot et al. (2007); FGIs—Lin and Kimura (1998), Krot et al. (2004a); ARCs—Sheng et al. (1991), MacPherson and Huss (2005), Krot et al. (2007); and AOAs—Komatsu et al. (2001), Alén et al. (2002), Krot et al. (2004b), Weisberg et al. (2004). Fields for Type A and B CAIs from Lin et al. (2003) and Simon et al. (1991), respectively. Inset shows expanded view of data from lower left-hand corner of plot. FGI = fine-grained spinel-rich inclusion, ARC = Al-rich chondrule, AOA = amoeboid olivine aggregate.](image-url)
nODULES and is the only CAI in Wild 2 and GCP to contain perovskite. The greater degree of alteration in the comet nodules is further supported by the differences between the forsterite rims on FGIs in chondrites compared to the Wild 2 and GCP fragments. In the chondrites, forsterite rims do not surround individual nodules but enclose entire FGIs, whereas in the CAIs from Wild 2 and GCP, forsterite (and enstatite) rims, where they occur, surround (discontinuously) individual nodules whether they occur singly or in aggregates. In summary, CAIs from Wild 2 and GCP are texturally and mineralogically similar to single nodules or small groups of nodules from FGIs found in chondrites, but the Wild 2 and GCP nodules (or small aggregate clusters) were either formed at lower temperatures or were more extensively altered in the nebula compared to their chondrite counterparts because the nodules from the comet samples were smaller and were not as well protected from nebular gas like most nodules in the much larger FGIs.

Wild 2 and GCP Fragments Compared to AOAs

Amoeboid olivine aggregates are composed of micron-sized grains of forsteritic olivine; Fe,Ni metal; and Al- and Ca-rich minerals including spinel, anorthite, Al,Ti clinopyroxene, and occasionally melilite (Krot et al. 2004a; Weisberg et al. 2004; Ruzicka et al. 2012). Oxygen isotope studies have shown that unaltered AOAs have similar \(^{16}O\)-rich compositions as pristine CAIs and therefore may have formed in similar nebular environments (Krot et al. 2004a; Itoh et al. 2007; Yurimoto et al. [2008] and references therein). Unlike most CAIs, AOAs contain forsterite and Fe,Ni metal indicating that if these objects are products of condensation, they likely condensed in slightly cooler nebular gas than CAIs. Although all the objects in this study are composed of relatively refractory Al- and Ca-rich minerals, some also contain olivine, thus an important question is whether they may be related to AOAs or AOA fragments.

GCP fragment P1-1, whose mineral assemblage is of \(\text{Fo}_{98-99}\) olivine, Al,Ti clinopyroxene, and \(\text{An}_{95-98}\), is similar texturally and mineralogically to some AOAs in the LL3.0 chondrite Semarkona (Itoh et al. 2007) and like some of the AOAs, Fe,Ni metal is absent. However, the bulk composition of P1-1 is somewhat more refractory than expected for AOAs and more akin to Al-rich chondrules (Figs. 17 and 18). Thus, P1-1 has properties consistent with both AOAs and ARCs and therefore we cannot rule out possible links to either object type. The fine-grained nodules of GCP fragment P6-14, whose minerals consist of \(\text{An}_{96-98}\) (with spinel inclusions), Al,Ti clinopyroxene, \(\text{Fo}_{99}\) olivine, and \(\text{En}_{90}\text{Wo}_3\) low-Ca pyroxene are also generally consistent with some AOAs. In approximately 10% of AOAs in primitive chondrites, low-Ca pyroxene is present with forsterite (Krot et al. 2004a). However, the bulk composition of this fragment is significantly more refractory than AOAs as shown on the larnite-corundum-forsterite ternary diagram in Fig. 17. It plots close to the anorthite-forsterite (+spinel) phase boundary and is relatively distant from bulk AOAs. Thus, we interpret the nodular texture and bulk composition of P6-14 as an object more likely related to FGIs than AOAs.

GCP fragment LT1, a mixture of \(\text{Fo}_{88-94}\) (plus inclusions of Fe,Ni metal ± refractory metals and Cr- and Fe-rich spinel), \(\text{An}_{90-98}\) plagioclase feldspar, and Al, Ti clinopyroxene, is the one fragment in the study whose properties are generally most consistent with AOAs. The elevated Fe content of the olivine and spinel and enrichments of Na in the plagioclase feldspar indicate that if LT1 is an AOA, it is more akin to an altered AOA than a pristine counterpart. Of the Wild 2 and GCP objects presented here, LT1 is the least refractory (Fig. 17). On the \(\text{Al}_2\text{O}_3 + \text{CaO} \ versus \ \text{MgO} + \text{Si}_2\text{O}_3\) diagram (Fig. 18), LT1 plots with AOAs from chondrites. Alternatively, the fragment may have affinities to AOA/Al-rich chondrule-like objects similar to those reported in the CV chondrite Leoville and the ungrouped carbonaceous chondrite Adelaide where fayalite contents in olivine range up to Fa3,9 and elevated Na contents in plagioclase feldspar are as high as Ab\(_{0.09}\) (Krot et al. 2004c; electronic appendix). Spinel minerals in these objects also have slightly elevated FeO and Cr\(_2\text{O}_3\) abundances consistent with some alteration. Itoh et al. (2007) have suggested that some AOAs may have minor amounts of melt. We note that small amounts of glass have been observed in LT1.

Finally, several objects in this study (WF216, TuleF4, CokiF2, and P3-4) contain primary Al- and Ca-rich minerals but lack olivine. All have nodular textures with concentric minerals generally consisting of anorthite surrounding spinel (inclusions) overlain by Al, Ti clinopyroxene. As previously discussed, it is known that fragments can lose material due to abrasive removal during capture. Could forsterite have been present on some or all of these refractory objects prior to collection? In the SD tracks in this study, we have observed micron-size and smaller isolated Mn-rich forsterites that were physically distant from these refractory fragments which conceivably could have been eroded from the refractory object exteriors during capture. Mn-rich forsterites (LIME) have been observed in some AOAs in chondrites (Krot et al. 2004a; Weisberg et al. 2004). Although the amount of material removed during capture for any particular fragment is unknown, because no forsterite was observed to be
directly associated with any of these fragments, it was likely not present or if it existed at all was present only as thin rims. A small amount of forsterite as a rim would not dramatically alter the bulk compositions of these fragments and therefore it is most likely that these objects are related to fine-grained inclusions rather than AOAs.

Refractory Wild 2 and GCP Fragments Compared to Wark-Lovering Rims

The refractory fragments in Wild 2 and the giant cluster IDP presented here are texturally and mineralogically similar to many Wark-Lovering rim (WLR) sequences, which are observed on most CAIs in primitive chondrites (Ruzicka 1997; MacPherson 2005; Simon et al. 2007; Dyl et al. 2011; Bodénan et al. 2014). In general, WLRs consist of a fine-grained (<a few microns) FeO-poor spinel ± perovskite ± hibonite inner layer, which is successively overlain by fine-grained Al-rich melilite (often being replaced by anorthite), Al,Ti-rich to Al-rich clinopyroxene, and then forsterite. The WLR from a type B CAI in the reduced CV3 chondrite Leoville, for instance, has many characteristics of the Wild 2 and GCP CAIs including pyroxenes with wide ranges in Al₂O₃ and TiO₂ content (Al₂O₃ = 0.48–12.4 wt%; TiO₂ = 0.09–3.88 wt%) and an anorthite layer containing tiny FeO-poor spinel inclusions (Toppiani et al. 2006). Similarly, an inner to outer sequence of spinel, Ca pyroxene, and olivine is present on a type A CAI from the CR chondrite EET92-21 (Weisberg et al. 2004; their fig. 1m).

Did the refractory nodules in Wild 2 and GCP form in similar environments as WLRs? Oxygen isotope measurements obtained on bulk IntiF1 showed that the fragment is ¹⁶O-rich and falls on the carbonaceous
Fig. 18. Bulk $\text{Al}_2\text{O}_3$ + CaO versus $\text{MgO} + \text{SiO}_2$ oxides of Wild 2 and giant cluster IDP (GCP) refractory objects compared to refractory materials in chondrites. Specific Wild 2 and GCP fragments (green and red solid symbols) are labeled in the figure (see Table 1). The plot shows that the comet samples are most comparable to moderately refractory materials in chondrites. Most of the bulk analyses plot on the slope = –1 line because the four oxides comprise most of the bulk of the samples. Objects that deviate from the line contain oxides in addition to $\text{Al}_2\text{O}_3$, CaO, MgO, and $\text{SiO}_2$ and show increasing deviation with less refractory character. Three diagonal lines from lower left to upper right show $1 \times$, $10 \times$, and $20 \times$ ($\text{Al}_2\text{O}_3 + \text{CaO})/(\text{MgO} + \text{SiO}_2$ wt% solar ratios. Bulk chondrite data from Wark (1987), Wark and Boynton (1987), Ruzicka (1996), Komatsu et al. (2001), Krot et al. (2004a, 2004b), MacPherson and Huss (2005), and Grossman et al. (2008). Plot after Grossman (2010). $10 \times$ additional average rim pyroxene added to bulk composition (see text). $25 \times$ additional average rim pyroxene added to bulk composition (see text). FGI = spinel-rich, fine-grained inclusion, WLR = Wark-Lovering rim, ARC = Al-rich chondrule, AOA = amoeboid olivine aggregate.

Chondrite anhydrous minerals line (CCAM) at $\delta^{18}\text{O} = –39.5$ to $–41.6_{oo}$, $\delta^{17}\text{O} = –38.7$ to $–42.0_{oo}$, $\Delta^{17}\text{O} = –18.9$ to $–20.4_{oo}$ (McKeegan et al. 2006). Recent isotopic analyses obtained from rim minerals on Type A CAIs in primitive carbonaceous chondrites (Leoville [reduced CV3], QUE 99177 [CR3.0], and ALHA 77307 [CO3.0]) were also uniformly $^{16}$O-rich with average $\Delta^{17}\text{O}$ values ranging from $–23.3$ to $–24.4_{oo}$ and $–22.6$ to $–24.5_{oo}$ for diopside and olivine, respectively (Bodénan et al. 2014). Thus, IntiF1 has chemical, textural, mineralogical, and O isotopic similarities to WLRs. The slightly lower $\Delta^{17}\text{O}$ values measured in IntiF1 compared to the WLRs may, in part, reflect differences in the analyzed materials, that is, single minerals in the WLRs versus bulk measurements in IntiF1.

Because pyroxenes in refractory objects are often Ti-rich, measurements of their $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios can provide insights into the oxidation environments in which they formed. In Allende, Simon et al. (2007) studied Al,Ti diopsides in WLRs by XANES spectroscopy and found a wide range in $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios (~0.0–2.45), which were comparable to pyroxenes in their host CAI interiors. In a study of fassaites using electron energy loss spectroscopy (EELS) measurements in the TEM on four refractory Wild 2 and GCP grains (P3-4, P6-14, P1-1, and IntiF1/F15), Joswiak and Brownlee (2015) demonstrated a similar wide variation in $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios (0.02–3.8) within single nodules and also between nodules. Three EELS measurements from two nodules in P6-14 showed variations in $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios from 0.12 to 2.7. This surprisingly wide range was interpreted to mean that individual nodules reacted with nebular gas in local environments with variable oxidizing conditions prior to aggregation. The Ti valence states in pyroxenes in WLRs in the Simon et al. (2007) study and the measured Wild 2 and GCP samples have similar wide ranges. In a different WLR study, however, Simon et al. (2005) used calculated $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios from microprobe data of fassaites in Leoville and Allende to show that the WLR pyroxene $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios were somewhat more restricted ($\text{Ti}^{3+}/\text{Ti}^{4+} = –0.50$ to 0.82) and distinct from pyroxenes in their host CAI interiors. This latter study was reaffirmed in a more complete study of pyroxenes in Leoville in the same WLRs (Dyl et al. 2011), thus these studies show relatively wide ranges in $\text{Ti}^{3+}/\text{Ti}^{4+}$ ratios in pyroxenes in WLRs. Similar wide variations in oxidation states occur in Ti-rich pyroxenes in the Wild 2 and GCP samples.

Similar mineral sequences, mineral textures, $^{16}$O-rich compositions (CAI nodule IntiF1), and widely varying Ti valence states in Al,Ti clinopyroxenes demonstrate that at least some of the refractory nodules in Wild 2 and GCP may have formed in similar environments as WLRs. If WLRs were formed by condensation in the nebula, then perhaps a portion of the condensing gases nucleated on small pre-existing grains, rather than the much larger CAIs, to form the FGI-like objects that are observed in the comet samples. Alternatively, precursor grains could have been altered by metasomatism via solid–gas reactions followed by diffusive mineral exchange (Ruzicka 1997).
refractory materials from which these objects may have formed. Bulk compositions of the nine refractory cometary fragments are plotted in the Mg$_2$SiO$_4$ - Al$_2$O$_3$ - Ca$_2$SiO$_4$ phase diagram (Fig. 17), which was first proposed by Huss et al. (2001) and later expanded upon in MacPherson and Huss (2005) and others. Included in the plot are two additional bulk compositions from TuleF4 and CokiF2, calculated by the addition of 10$\times$ and 5$\times$, respectively, their average rim pyroxene compositions. These bulk compositions were calculated because textural evidence strongly indicated that significant rim abrasion occurred during capture in aerogel (see Table 7). In the diagram, petrogenetic relationships between many diverse objects in chondrites including CAIs, FGIs, WLRs, chondrules, and AOAs are shown (references provided in figure caption). Oval regions show that the more refractory objects, Type A and B CAIs, plot in the melilite + liquid field, while the less refractory objects, Type C CAIs, FGIs, and ARCs plot in the anorthite and forsterite (+liquid) phase fields and together define a different trend extending from anorthite toward forsterite. AOAs and Mg-rich ferromagnesian chondrules occupy the forsterite primary phase volume closer to the forsterite apex of the ternary diagram. Also shown in the figure (green arrows) is the calculated condensation trend of bulk solids from solar gas at 10$^{-6}$ bar (MacPherson et al. 2004). Bulk WLRs (Ruzicka 1996) parallel the anorthite-forsterite trend but are more Ca-rich. The possible significance of the CAI versus the chondrule + AOA trends and their convergence near anorthite was discussed in MacPherson and Huss (2000, 2005) and Krot et al. (2004b) who pointed out that Type C CAIs and their likely precursors, FGIs, may have formed, at least in part, from nebular alteration of the more refractory Type A CAIs. However, a similar condensation + alteration scenario is unlikely to account for ARCs which probably were derived from more Mg-rich precursors with evaporation a likely important process. Because the most refractory Wild 2 and GCP fragments (IntiFx, WF216, TuleF4, CokiF2, P3-4, and P6-14) have bulk compositions that plot near anorthite and are in or close to the anorthite + liquid primary phase volume, these objects fall near the convergence point of the CAI–ARC + AOA trends and therefore could have their origins from either trend.

Because the Wild 2 and GCP samples have comparable mineral assemblages and textures as FGIs in chondrites, which Krot et al. (2004a) showed are likely to be altered nebular condensates, the Wild 2 and GCP objects are also likely to be condensation products that formed by nebular alteration from more refractory objects and therefore most likely represent the lower temperature alteration products of the CAI trend. The minor and trace quantities of melilitie and perovskite in IntiFx and IntiF1 and melilitie in WF216 and the spinel inclusions in anorthite in many of the fragments are evidence of this. GCP fragment P1-1, however, does not have a nodular texture nor contain spinel like the other CAI comet fragments (Fig. 11), but does have a bulk composition similar to the nodular GCP aggregate P6-14 (Table 7, Fig. 17), thus an origin specific to the CAI or ARC trend is less clear.

An additional means to compare these objects is to look at the degree of their refractory character by comparing refractory to more volatile elements. A plot of the refractory oxides Al$_2$O$_3$ + CaO versus the more volatile oxides MgO + SiO$_2$ for the nine Wild 2 and GCP fragments is shown in Fig. 18 (after Grossman 2010) with bulk CAIs, FGIs, ARCs, AOAs, and WLRs from chondrites (Wark and Boynton 1987; Ruzicka 1996; Komatsu et al. 2001; Krot et al. 2004a, 2004b; MacPherson and Huss 2005; Grossman et al. 2008). The plot shows that all the fragments in Wild 2 and GCP have (Al$_2$O$_3$ + CaO)/(MgO + SiO$_2$) ratios below 20$\times$ solar values and therefore are more volatile-rich than Type A and B CAIs whose corresponding ratios are mostly >20$\times$ solar. The most refractory Wild 2 and GCP fragments are most similar to Type C CAIs, FGIs, and the most refractory ARCs from chondrites and fall along the central portion of the (Al$_2$O$_3$ + CaO)/(MgO + SiO$_2$) = −1 line corresponding to the more moderately refractory objects in chondrites. The two least refractory fragments, the ARC BidiF1 and LT1, a probable AOA, plot in ARC or AOA regions corresponding to their chondrite counterparts. The plot also shows which objects have higher abundances of elements more volatile than Mg and Si such as Na and Fe. Symbols representing these objects plot below the (Al$_2$O$_3$ + CaO)/(MgO + SiO$_2$) = −1 line and are largely represented by WLRs and the least refractory AOAs and ARCs.

**Oxygen Isotopes: CAI WF216 (Track 172)**

The O isotopic composition obtained on the Wild 2 CAI WF216 plots on the CCAM line (Fig. 13) at intermediate $\delta^{18}$O and $\delta^{17}$O values ($\Delta^{17}$O = −15$^{\circ}$). This compares to the Wild 2 CAI IntiFx fragment which also falls on the CCAM line but is significantly more $^{16}$O-rich ($\Delta^{17}$O = −18.9 to −20.4$^{\circ}$) (McKeegan et al. 2006) indicating that the O isotopic histories of the two Wild 2 fragments are different. This indicates that WF216 either formed in a different nebular environment or was $^{16}$O-rich like Intif1 and later exposed to relatively $^{16}$O-poor nebular gas. Examination of numerous fragments in track 80, the capture track of WF216, produced no other CAI nodules (Joswiak et al. 2012; Stodolna et al. 2012); therefore, WF216 was likely
formed and transported as a single nodule in the nebula and therefore would have been directly exposed to altering nebular gas. It is possible that the $^{16}$O-rich fragment IntiF1 was present in the interior of its aggregate and was largely isolated from direct exposure to $^{16}$O-poor nebular gas. It is also possible that IntiF1 and the other nodules in the aggregate were never exposed to an $^{16}$O-poor gas. This could be tested by analyzing additional CAI nodules from track 25 to determine whether they are more $^{16}$O-poor like WF216. We note that the nodules within fine-grained, spinel-rich inclusions in CV chondrites, which are close analogs to the Wild 2 CAIs, do not have uniform O isotopic compositions (Fig. 13) (Aléon et al. 2005). These nodules formed in $^{16}$O-rich environments but many were exposed to reservoirs with $^{16}$O-poor gas as shown by their large oxygen isotopic variations along the CCAM line.

Oxygen isotopes were obtained on CAI WF216 from an anorthite + Al clinopyroxene + spinel (minor) mixture. As noted previously, this mineral assemblage is very similar to nodules in FGIs from CV chondrites (Krot et al. 2004a). FGI precursors were spinel + perovskite + melilite + hibonite nebular condensates, which were variously altered by low-temperature gas to produce Al,Ti clinopyroxene and anorthite in $^{16}$O-poor nebular environments (Krot et al. 2004a; Aléon et al. 2005) as indicated by the large spread in O isotopic compositions (Fig. 13). In general, most spinels and Al, Ti diopside in the FGIs are $^{16}$O-rich, while anorthites and melilites vary significantly in O isotopic composition due to their higher susceptibility to O exchange. The presence of significant amounts of anorthite and Al clinopyroxene and smaller quantities of spinel and melilite in WF216 and its intermediate $^{16}$O composition suggests that this CAI may have formed from a more refractory precursor in a $^{16}$O-poor environment.

Absence of Low Temperature Alteration Phases

Fine-grained, spinel-rich inclusions in the oxidized CV chondrite Allende contain alkali- and Fe-rich phases including nepheline, sodalite, hedenbergite, andradite, FeO-rich olivine, and other minerals that often are observed in interstices between refractory minerals (Krot et al. [2004a] and references within). FGIs in Vigarano, a reduced CV chondrite, also contain low-temperature alteration phases which likely formed in a parent body; however, other reduced CV3s such as Leoville and Efremovka largely lack these minerals. Similar low-temperature minerals are absent in the Wild 2 and GCP fragments, although LT1 has a small amount of a Na,Al silicate glass or albite of uncertain origin. Thus, the Wild 2 and GCP refractory fragments largely escaped the low-temperature parent body alterations which affected FGIs in some CV and other chondrites. This indicates that prior to accretion into the Wild 2 or the GCP comet bodies, the fragments did not reside in large parent bodies where low-temperature alteration most likely modified many FGIs in chondrites.

Abundances of Refractory Materials in Comets

The abundances of CAIs in chondrites vary between different groups ranging from <0.01% for CI and ordinary chondrites to nearly 3% for the CV chondrites (Hezel et al. 2008). Although not quantified like CAIs, Al-rich chondrules are present in all chondrite groups, while AOAs are common in most carbonaceous chondrites but absent in enstatite and most ordinary chondrites (Itoh et al. 2004; Scott and Krot 2005). The types of refractory inclusions within specific chondrites can show significant variations. For instance, in Acfer 094, Simon and Grossman (2011) found at least 27 different CAI lithologies. CAIs, in Acfer 094, composed of mixtures most like those observed in Wild 2 and GCP—spinel + anorthite + diopside or spinel + melilite + diopside + anorthite mineral assemblages—constitute only 5.9% and 0.1% of the 289 CAIs observed, respectively.

Our compilation of the numbers of refractory materials from Wild 2 and GCP is shown in Table 8. Based on observations from 205 Wild 2 fragments (from 19 tracks) and 50 fragments from GCP, we estimate that ~2–3%, by number, is CAIs. In this estimate, all 23 CAI fragments in track 25 were grouped as a single fragment as it is likely that all the individual CAI fragments in the track were derived from a single aggregate impactor. CAIs were found in four different tracks and all were bulbous. In our study of comet Wild 2 samples, 21% of all tracks and 44% of all bulbous tracks have CAIs (four of nine). This is a somewhat surprising result and suggests that future systematic studies of fragments in bulbous tracks should have a

| Source          | CAI   | ARC | AOA   |
|-----------------|-------|-----|-------|
| Comet Wild 2    | 4/205 | 1/205 | 0/205 |
| GCP             | 2/50  | (0–1)/50 | (1–2)/50 |
| Wild 2 + GCP    | 6/255 | (1–2)/255 | (1–2)/255 |
| Wild 2          | 2%    | 0.5% | 0.0%  |
| Wild 2 + GCP    | 4%    | 0–2% | 2–4%  |
| Wild 2 + GCP    | 2.4%  | 0.4–0.8% | 0.4–0.8% |

CAI = calcium-aluminum-rich inclusion; ARC = Al-rich chondrule; AOA = amoeboid olivine aggregate; GCP = giant cluster IDP.
nearly 50% chance of finding a CAI if all large fragments from within a track are studied. From the three ARC and AOA fragments (BidiF1, P1-1, and LT1), less than 1%, by number, of each of these object types is present in the comet samples.

In order to provide a more direct comparison to CAI abundances in chondrites, we estimated the volume percentage of total CAI material from the Wild 2 tracks and GCP. For the Wild 2 samples, this was done using calculated impactor sizes for each track and measured CAI sizes from either microtome slices or potted butt images. Impactor sizes from type A tracks were taken from Joswiak et al. (2012) (Table 1), while sizes from type B tracks were estimated from fig. 20 in Burchell et al. (2008). We estimate that all Wild 2 CAI material that we studied summed to about 1805 μm³, while the total volume calculated from the 19 impactors is ~356,000 μm³. Thus, we calculate that ~0.5 volume% of material from comet Wild 2 was present in CAIs. We emphasize that this is only a rough approximation because of the small number of CAIs observed and the uncertainties of measuring sample sizes. For GCP, we calculated the volumes of all 50 fragments to determine a CAI abundance of ~1 volume%. Like Wild 2, the abundance estimate is considered only an approximation.

Paucity of the Most Refractory Objects in Comet Samples

We have shown that all the refractory materials that we observed in comet samples are composed of mineral assemblages equivalent to the lowest temperature refractory objects in chondrites including fine-grained, spinel-rich inclusions, amoeboid olivine aggregates, and Al-rich chondrules. The most refractory objects such as Type A CAIs, spinel-hibonite, and grossite-rich inclusions are conspicuously absent. Widely varying Fe/Mn ratios of FeO-rich olivines from comet Wild 2 and the giant cluster IDP provide evidence that comets accreted materials that formed in multiple nebular environments (Brownlee and Joswiak 2017), thus a fundamental question is why are the more refractory-rich objects absent in comet samples? Several explanations may account for this discrepancy. (1) The more refractory objects are present in comets but the number of fragments studied (>250) is statistically insufficient to account for the full range of materials present in the comet samples. An argument in support of this idea is that SD tracks are mineralogically diverse and no two tracks contain the same combinations of mineral and rock grains, thus enough tracks simply have not been examined. If the types of refractory inclusions in comets have Poisson distributions like they do in chondrites (Hezel et al. 2008) then larger sample sizes are needed to observe the full range of materials in comets. (2) The most refractory objects in chondrites are significantly larger than the sampled sizes of the comet grains. CAI sizes span a large range varying from microns to several millimeters or more. Modal and size abundance studies show that CAI sizes peak at >20 μm in most chondrites (except Acfer 094) (Hezel et al. 2008), sizes which exceed those available for study in the comet samples. It might be expected that CAI fragments (broken CAIs) would exist in the comet samples similar to the chondrule fragments (broken chondrules) which are commonly observed in Wild 2 samples (Nakamura et al. 2008; Brownlee et al. 2012; Joswiak et al. 2012; Nakashima et al. 2012, 2015; Ogliore et al. 2012, 2015; Frank et al. 2014; Gainsforth et al. 2015). Although we have occasionally observed minerals (i.e., spinel, anorthite, and Al,Ti clinopyroxene) in some tracks which could have been associated with moderately refractory objects like those observed in the comet samples, more refractory phases such as corundum, hibonite, grossite, and perovskite (except for the single occurrence in IntFi1) have not been observed that we are aware of, suggesting that fragments from the most refractory conventional CAIs are not present. (3) The most refractory objects in chondrites whose sizes are comparable to the refractory objects observed in the comet samples were not produced in large numbers in the nebula. Small refractory corundum-bearing CAIs (micro CAIs) have been observed in some primitive carbonaceous chondrites and were first discussed by Bland et al. (2007) and Nakamura et al. (2007). In a recent study of small corundum ± hibonite ± spinel-bearing CAIs (micro CAIs) from Murchison and ALHA 77307, Needham et al. (2017) estimated an abundance of ~0.3–3 cm⁻², abundances which are comparable to those reported by Makide et al. (2013) from Murchison, Murray, and Adelaide. These results indicate that small highly refractory CAIs are uncommon in even the most primitive chondrites suggesting that they were either not produced in large numbers in the nebula or were largely destroyed or altered after their formation. In a size distribution study of >3000 CAIs from CV and CK chondrites including numerical simulations, Charnoz et al. (2015) suggested that more refractory CAIs are expected to be smaller than less refractory counterparts due to their growth at higher temperatures where longer growth times are required because of lower dust/gas ratios. However, because only CAIs >30 μm were studied, it is unclear whether the trend can be extrapolated toward CAIs with sizes comparable to those found in the comet samples. In general, small, highly refractory micron-scale CAIs are not commonly reported in refractory inclusion studies in chondrites. This may be
due to a lack of studies or that they are simply not present. (4) The most common type of CAI produced in chondrites are nodules that comprise fine-grained, spinel-rich inclusions, the closest analog objects in chondrites to the Wild 2 and GCP refractory fragments. As whole objects, FGIs are large (typically \( > 1 \text{ mm} \), Lin and Kimura 1998; Krot et al. 2004a); however, FGIs are composed of enormous numbers of much smaller objects—nODULES—whose smallest members approach the sizes of the largest CAIs in Wild 2 and GCP. In the Ningqiang carbonaceous chondrite, concentric spinel + anorthite + clinopyroxene (in sequence, center to rim) nodules range in diameter from 6 to 15 \( \mu \text{m} \) (Lin and Kimura 1998) and in a \( ~4 \times 6 \text{ mm} \) FGI (E42) in Efremovka, \( ~10-40 \mu \text{m} \) spinel + anorthite + pyroxene nodules were reported (see fig. 1, MacPherson et al. 2004). We calculate that this FGI is composed of a total of \( \sim 10^7 \) individual nodules assuming an average nodule diameter of 25 \( \mu \text{m} \). This indicates that enormous numbers of small CAI nodules with moderately refractory compositions were produced in the nebula, many of which aggregated into larger objects (FGIs). The nodules observed in Wild 2 and GCP, however, suggest that some of these small nodular CAIs escaped aggregation into chondrites and were instead transported to the outer solar system either individually (WF216, TuleF4, and CokiF2) or in small number aggregates (IntiFx, P3-4, and P6-14) and incorporated into comets. Because only small sample sizes (\(< 20 \mu \text{m} \) for Wild 2 and \(< 40 \mu \text{m} \) for GCP) are represented in the Wild 2 and GCP collections, the observed cometary CAIs are heavily weighted toward the large number of these moderately refractory inclusions that the nebula produced, which may account for the preponderance of FGI-like objects in Wild 2 and GCP. (5) The highest temperature materials were not as efficiently transported to the edge of the solar system as they were to the regions where carbonaceous chondrites formed.

**Origin of Refractory Materials in Comets**

The O isotopic compositions measured on the terminal particle IntiF1 from track 25 and the fragment WF216 from track 172, which fall on the CCAM line, demonstrate that the Wild 2 CAIs likely formed in solar nebula environments that were similar or identical to those where many pristine CAIs in chondrites formed. Compared to IntiF1 with is \( ^{16}\text{O}-\text{rich} \) in composition (\( \Delta^{17}\text{O} = -18.9 \) to \( -20.4_{\text{oo}} \)), the O isotopic composition of CAI WF216 (\( \Delta^{17}\text{O} = -15_{\text{oo}} \)) indicates that it either formed in a more \( ^{16}\text{O}-\text{poor} \) region of the nebula or that it partially re-equilibrated with \( ^{16}\text{O}-\text{poor} \) gas after formation similar to some fine- and coarse-grained CAIs in chondrites (Yurimoto et al. 2008). Oxygen isotopes have not been measured on the other comet CAI fragments (TuleF4, CokiF2, P3-4, and P6-14) but all of these CAIs are mineralogically and texturally similar to the Wild 2 fragments IntiFx and WF216. As previously discussed, all the Wild 2 and giant cluster IDP CAI fragments are composed of only moderately refractory minerals and all are mineralogically zoned with spinel typically occupying the cores, which are then directly overlain by anorthite and \( \text{Al} \pm \text{Ti} \) clinopyroxene. The physical and chemical properties of the Wild 2 GCP nodules closely match nodules that comprise fine-grained, spinel-rich inclusions, which are found in CV and other chondrites. This suggests that the comet CAIs and meteoritic FGIs have common source regions.

CokiF2, whose measured \( ^{26}\text{Al}-^{26}\text{Mg} \) isotopes showed no excess \( ^{26}\text{Mg} \), may have formed (1) before \( ^{26}\text{Al} \) was injected into the solar nebula, (2) in a region where \( ^{26}\text{Al} \) was not present, or (3) \( > 1.7 \) Myr after the onset of CAI formation similar to some Type C CAIs and Al-rich chondrules (Matzel et al. 2010). Krot et al. (2004a) showed that the bulk compositions of FGIs were similar to Type C CAIs and suggested that the FGIs, and by inference, the nodules that they are comprised of, were possible precursors to these CAIs. The similarity of CokiF2 with some nodules in FGIs and its low \( ^{26}\text{Al} / ^{27}\text{Al} \) ratio, then, is directly linked to Type C CAIs and thus CokiF2 may have formed from low temperature alteration of more refractory precursors as originally suggested for fine-grained CAIs by Beckett and Grossman (1988).

MacPherson et al. (2004) demonstrated that at low total pressures, the trajectory of bulk solids condensing from a solar nebula gas will more closely overlap the bulk compositions of the moderately refractory FGIs and ARCs (shown by the green arrow in the larnite-corundum-forsterite diagram in Fig. 17). Thus, some or all of the nine refractory fragments in Wild 2 and GCP likely formed in the nebula by alteration of more refractory objects where lower pressures and moderate temperatures prevailed. The minor amount of observed melilitite would likely be relict along with the trace perovskite found in IntiF1. Because Cr is a moderately volatile element and has a condensation temperature lower than Al and Mg (Lodders 2003), its elevated presence in spinel in the Wild 2 and GCP fragments compared to spinels in Type A and B CAIs and some FGIs (Fig. 15) is also consistent with equilibration in nebular gas at more moderate temperatures. The enstatite + forsterite accretionary rims present on two of the GCP CAIs could have formed from further reactions with SiO-rich nebular gas similar to AOAs and FGIs (Krot et al. 2004a, 2004b). Because little to no alkali- and Fe-rich minerals are present in these
Refractory materials in comet samples

fragments (with the exception of the AOA LT1), they were not subjected to low-temperature alteration reactions in the presence of these more volatile elements. This suggests that prior to incorporation into Wild 2 or the GCP comet, the fragments were not present in previous large parent bodies or present for long periods in the nebula, which could have enriched them in these more volatile elements.

CONCLUSIONS

This study of six CAIs and three Al-rich chondrule or AOA fragments from comet Wild 2 and a giant cluster IDP, of probable cometary origin, has produced important, if limited, information on refractory materials that existed in the outer solar system during comet formation. The comet CAIs consist of one or a small number of nodules similar to fine-grained, spinel-rich inclusions found in CV3 and other carbonaceous chondrites and like the FGIs have similar concentric textures and primary minerals including spinel, anorthite, and Al,Ti clinopyroxene ± melilite. The O isotopic composition of one measured CAI from Wild 2 has an intermediate O isotopic composition (Δ17O = −15‰) and lies on the CCAM line where some FGIs fall. One fragment from the giant cluster IDP is either an amoeboid olivine aggregate, Al-rich chondrule, or a hybrid AOA/ARC object. A second fragment (LT1) from GCP is likely an altered AOA. Many of the nodular comet objects are composed of layered minerals that resemble Wark-Lovering rims commonly observed on CAIs in chondrites.

The bulk compositions and mineral assemblages show that all the refractory objects found in Wild 2 and the giant cluster IDP are of only moderate refractory character. With the notable exceptions of the tiny osbornite inclusions, they largely lack the most refractory minerals found in the most refractory objects in chondrites. The lack of the most refractory objects in the comet samples may reflect selective transport of refractory grains from the inner solar system to the comet-forming regions; may be a result of the large number of intermediate small nodules (FGI components) that were produced in the nebula compared to more refractory objects; or a result of the small sizes and the low number of comet samples observed, which may not reflect representative abundances of nebula-produced refractory materials.

Like some fine-grained, spinel-rich inclusions in chondrites, the Wild 2 and giant cluster IDP CAIs may have formed from previously condensed more refractory objects that were altered by nebular gas prior to incorporation into their respective comet bodies. The general lack of alkali- and Fe-rich minerals in most of the refractory comet samples indicates they were not subjected to the lowest temperature alterations that can produce aqueously altered minerals and thus they probably did not reside in larger parent bodies prior to accretion into Wild 2 or the giant cluster IDP comet, nor did the comet bodies experience significant aqueous alteration themselves.

Approximately, 2–3% of the 255 one micron and larger cometary fragments studied, by number, are CAIs while ARCs and AOA fragments comprise less than 1% of grains. From these abundances, a rough estimate suggests that comet samples are composed of ~1 volume% CAIs, estimates that are comparable to some carbonaceous chondrite groups but higher than enstatite and ordinary chondrite groups. The observed low fraction of AOA fragments might be an intrinsic property of comet samples. With respect to Wild 2, however, low AOA abundances may be related to difficulty in identification of AOA having resulted from fragmentation and abrasive material loss during collection in silica aerogel.

In both meteorites and comets, the refractory components appear to have been formed in different environments than the bulk of the components in their host bodies and their presence required migration. It is nearly certain that both carbonaceous chondrites and comets formed exterior to the regions where ordinary and enstatite chondrites formed. The higher abundances of CAIs in the outer solar system bodies provide a profound clue to the origin and transport of refractory materials. Major questions on the comet refractories are where they came from, how they were transported, and when they were transported. Continued study should show if the comet refractories are from the same reservoirs as those in chondrites or whether they just formed by similar processes. The findings reported here provide improved insight into the abundances and nature of refractory materials that were present in cold regions of the protosolar nebula at the time that Wild 2 and other comets accreted.

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