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Survey of ‘Low-Z’ Particles from the Interplanetary Dust Collection in Search of IDPs

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ABSTRACT

Micrometeorites are one of the most common types of extraterrestrial material that fall to the Earth, beside meteorites and interplanetary dust particles (IDPs). All of these materials are known to contain presolar grains that, when studied in the laboratory, provide information about the origin of the solar system and help to constrain models of nucleosynthesis in stars. Recent studies of micrometeorites revealed the presence of a group of particles with very high C contents. These ultra-carbonaceous Antarctic micrometeorites (UCAMMs) are fine-grained, fluffy particles dominated by highly disordered carbonaceous matter. The presence of H and N isotopic anomalies, abundant presolar grains, and other minerals suggest similarities to IDPs. The presence of these ultra-carbonaceous particles led us to question if similar particles may be present in the NASA interplanetary dust collection. These particles undergo preliminary SEM-EDX analysis after being collected in the upper-stratosphere to separate IDPs from terrestrial contaminants. Some of the particles classified as terrestrial contaminants have spectra suggesting that they are dominated by low atomic number (low-Z) elements that are typically not detectable by EDX analysis. Although classified as terrestrial contaminants, some of these particles may in fact be extraterrestrial ‘low-Z’ IDPs with high C contents, similar to UCAMMs. In this study, we examined a number of low-Z particles to investigate this possibility. Using more sensitive EDX equipment, Raman analysis, and NanoSIMS isotopic imaging we showed that it is unlikely any of these low-Z particles contain presolar material, making it unclear that these particles have an extraterrestrial origin.

1. INTRODUCTION

Interplanetary dust particles (IDPs) are a form of primitive, extraterrestrial material believed to originate from comets and asteroids\(^1\). These particles are typically only 5-20µm in size, and are collected by NASA in the upper stratosphere before they burn up in the Earth’s atmosphere or are otherwise significantly altered by the atmosphere. Most of these particles are characterized as having fine-grained, or fluffy textures, helping to identify them\(^1,2\). Past studies on IDPs have yielded a great deal of information about the make-up and characterization of these particles. IDPs often contain anhydrous minerals, including phyllosilicates and GEMS (glass with embedded metals and sulfides), or insoluble organic matter (IOM)\(^1,2\). Due to the large concentration of presolar material found inside these particles, IDPs are an important source of extraterrestrial material, alongside meteorites, that can be studied to learn more about our solar system\(^1\).

Most presolar material available to study was incorporated into materials that formed at the same time as the solar system and have since fallen to the Earth, such as
meteorites and IDPs. When studied, these materials can give us an insight into the formation of our solar system and nucleosynthetic processes in stars. Presolar grains are marked by their unusual isotopic compositions compared to the bulk isotopic ratios observed in the rest of the parent body and the solar system. Such compositions suggest that these particles survived mixing and homogenization processes during the formation of the solar system. This presolar material would have come from the outflows of old stars or the remnants left behind by supernovae explosions and, as such, can be studied to help refine models of stellar evolution.

In particular, for IDPs, large areas of these particles have displayed high D/H ratios (enrichments in deuterium) and enrichments in $^{15}$N. These enrichments are correlated with the abundance of presolar grains found. The high D/H ratios, high C contents, and mineral inclusions are reminiscent of cometary compositions, aligning them with a cometary origin.

Micrometeorites are another common type of extraterrestrial material. Most micrometeorites are recovered from the Antarctic snow and ice after reaching the Earth’s surface. These particles are often much larger than IDPs, but otherwise share similar characteristics, including high C contents and some similar mineral inclusions. Recently, some micrometeorites have been found that have extremely high concentrations of carbon, giving them the name ultra-carbonaceous Antarctic micrometeorites (UCAMMs). These particles are very similar to anhydrous IDPs, displaying inclusions of GEMS, IOM, and large D/H ratios and N anomalies.

The discovery of UCAMMs led us to question if IDPs with similarly high amounts of carbonaceous matter also existed. Particles collected by NASA undergo preliminary analysis to categorize each particle as either IDPs or terrestrial contaminants. Many of the particles classified as contaminants suggest they are dominated by low-Z elements (including C), which cannot be measured using traditional energy-dispersive x-ray detectors. It is possible that some of these low-Z particles are actually IDPs with very high C contents similar to UCAMMs. We conducted a survey of low-Z particles to investigate this possibility.

2. EXPERIMENT

Twenty-six particles were obtained from the NASA cosmic dust collection, ranging in size from 8-70µm. These particles were chosen based on their preliminary energy-dispersive x-ray (EDX) spectra, which suggested large concentration of low atomic number (low-Z) elements. Using preliminary secondary electron (SE) images, our particles were sorted by their physical characteristics into groups of particles that most resembled IDPs.

In order to prepare the particles for analysis, they first needed to be mounted on gold foil. The gold serves as a conducting background that prevents the particles from charging, the build-up of electrons on a sample, under SE imaging. A grid was pressed into each of the gold foil mounts in order to help keep track of the particles once placed. Particles were mounted for analysis using standard procedures for grain-picking (see Appendix A2). A micromanipulator arm is controlled under a high-magnification optical microscope to guide a needle over each particle. The needle tip is sharpened in NaOH to a point of a few micrometers in diameter, and uses static electricity to lift the particles off
of glass slides. Particles are then maneuvered onto the gold foil mounts where their locations are noted on the foil grid. Once all the particles had been placed, each foil mount was flattened beneath a quartz disk (1 in. diameter x ¼ in. thickness), pressing the particles into the foil. This was done to ensure the particles were secure in the foil and also lay flat against the foil. In addition to helping prevent charging, it is especially important that the particles lay flat for analysis in instruments that are very sensitive over small distances, or else the data may be distorted by topographical effects. Area maps of each foil grid were made both before and after particles were moved onto the foil using a reflected light microscope (Fig. 1).

![Figure 1. Example map of a gold foil mount made from images taken with a reflected light microscope, before the particles were pressed into the foil. Particles can be seen as specks in a few of the grid areas.](image)

Next, the foil mounts were placed in a JEOL 840A Scanning Electron Microscope (SEM) for imaging and elemental analysis. Another map of the foil area was first made from SE images using an automated mapping program in the SEM software. High-magnification SE images of each individual particle were acquired to help confirm each particle’s size and better judge its physical characteristics. Elemental spectra of each particle were taken in the SEM using EDX spectroscopy. Most EDX detectors have trouble distinguishing between low-Z elements; however, our detector is fitted with a light element detector allowing for a more detailed spectrum to be acquired. This information was then used to help classify particles as potential IDPs or terrestrial contaminants based on their elemental spectra.

SEM-EDX spectra were sorted through to determine the most likely IDP candidates for further analysis. The gold foil mounts containing these candidates were placed in the Cameca NanoSIMS 50 to undergo analysis through secondary ion mass spectrometry (SIMS) (See Appendix A1). Raster ion imaging was carried out using a Cs⁺ primary beam on two different occasions; we first imaged isotopes of C and O, then, after Raman spectra were taken, C, O, and N (as CN⁻) isotopes were measured (see below). The data were examined for isotopically anomalous regions that would signify the presence of a presolar grain.
Raman spectra were taken for each particle that had undergone NanoSIMS analysis using a Renishaw inVia Raman microscope and a 633nm wavelength laser. Raman spectroscopy works by detecting the laser light scattered off of the molecules in a sample. The observed shift in wavelength can give us information about the vibrational modes of each molecule, helping to characterize the individual components of a sample. These spectra were taken to determine the mineralogical make-up of each particle, assisting us in classifying particles as terrestrial or extraterrestrial.

3. RESULTS & DISCUSSION

Of the 26 particles we received, 22 were successfully mounted across three gold foil mounts using the Micromanipulator. The remaining four particles were lost in transit to the mount after being picked up by the Micromanipulator needle. Some difficulties were encountered in pressing the particles into the foil. One quartz disk shattered over a foil mount during the pressing procedure, causing shards of quartz to be scattered on the foil. These quartz particles were easily recognizable in the SEM with EDX detection and could be removed with the Micromanipulator or avoided in later analysis. While pressing another foil mount, the quartz disk picked up and relocated the particles on the mount. These particles needed to be re-identified before SEM analysis could proceed.

<table>
<thead>
<tr>
<th>Particle</th>
<th>Size ($\mu$m$^2$)</th>
<th>Elements detected (SEM-EDX)</th>
</tr>
</thead>
<tbody>
<tr>
<td>L2008 C7</td>
<td>10x12</td>
<td>C, O, Na, Mg, Al, Si, S, Fe, Ca</td>
</tr>
<tr>
<td>L2047 E36</td>
<td>40x30, 30x20</td>
<td>C, N, O</td>
</tr>
<tr>
<td>U2097 B5</td>
<td>40x50</td>
<td>C, N, O, Na</td>
</tr>
<tr>
<td>L2036 M11</td>
<td>20, 15, 17</td>
<td>C, N, O, Na, (Al, Si, Ca, Cd, K)</td>
</tr>
<tr>
<td>W7190 G33</td>
<td>12, 8</td>
<td>C, N, O, Na, Mg, Ca, Al</td>
</tr>
<tr>
<td>U2098 A5</td>
<td>15x10, 20</td>
<td>C, O, Fe, Na, Mg, Al, K</td>
</tr>
<tr>
<td>L2047 B12</td>
<td>30, 36x20</td>
<td>C</td>
</tr>
<tr>
<td>L2047 B5</td>
<td>20x45, 15x35</td>
<td>C, N, O</td>
</tr>
<tr>
<td>L2047 D27</td>
<td>10x20, 30</td>
<td>C</td>
</tr>
<tr>
<td>L2021 E39</td>
<td>10, 20, 15x20, 30x20</td>
<td>C, O, Al, Mg, Fe (N, K, Ca)</td>
</tr>
<tr>
<td>L2047 B19</td>
<td>40x30</td>
<td>C</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Particle</th>
<th>Size ($\mu$m$^2$)</th>
<th>Elements detected (SEM-EDX)</th>
</tr>
</thead>
<tbody>
<tr>
<td>W7190 D40</td>
<td>18x30</td>
<td>C, N, O, Na, Ca</td>
</tr>
<tr>
<td>U2097 A5</td>
<td>40x30, 10x15</td>
<td>C, O, Si, Br, Na</td>
</tr>
<tr>
<td>L2021 C16</td>
<td>9, 5x7</td>
<td>O, Cr, C, Fe</td>
</tr>
<tr>
<td>L2036 L5</td>
<td>10, 6x10</td>
<td>C, N, O, Na</td>
</tr>
<tr>
<td>L2008 J16</td>
<td>10</td>
<td>C</td>
</tr>
<tr>
<td>U2097 B19</td>
<td>30</td>
<td>C, N, O</td>
</tr>
<tr>
<td>L2047 B10</td>
<td>10x22, 12x20</td>
<td>C, N, O, Na</td>
</tr>
<tr>
<td>L2047 B1</td>
<td>30</td>
<td>C, N, O, Si</td>
</tr>
<tr>
<td>L2009 D6</td>
<td>20x12</td>
<td>C, (Si, Al)</td>
</tr>
<tr>
<td>L2021 E15</td>
<td>22x10, 10, 30</td>
<td>C, N, O, Na</td>
</tr>
<tr>
<td>L2008 H2</td>
<td>8</td>
<td>C, O, Mg, Al, Si, S, Fe</td>
</tr>
</tbody>
</table>

Table 1. Elements compositions and particle sizes from SEM-EDX analysis
Figure 2. Three of the particles chosen for further analysis. [(a) L2008 C7. (b) L2008 H2. (c) L2021 E39] (d) The EDX spectra for these particles all showed elements common to IDPs.

Figure 3. Four of the particles chosen for further analysis. [(a) L2008 J16. (b) L2009 D6. (c) L2047 B12. (d) L2021 C16] (e) The EDX spectra for these particles all showed high contents of C.
SEM-EDX data for each particle can be seen in Table 1. The particles we were interested in had fine-grained textures and spectra most similar to those of known IDPs; these include elemental signatures of Al, C, Fe, Mg, O, S, Si. Particles with high C signatures were also of interest, as they may signify an ultra-carbonaceous extraterrestrial particle. Of these 22 particles, seven were chosen to be studied further in the NanoSIMS: particles L2008 H2, L2008 C7, L2047 E39, L2008 J16, L2009 D6, L2021 C16, L2047 B12 (Fig. 2, 3). The first three of these particles all had spectra similar to the spectra of typical IDPs (Fig. 2), while the last four particles had spectra dominated by C (Fig. 3). All the remaining particles had spectra containing elements more common to terrestrial contaminants and were, therefore, not of interest for further analysis.

The first round of NanoSIMS analysis provided isotopic maps of C and O. Images were created comparing isotopes of each element to search for isotopic anomalies in the seven particles. After preliminary analysis, NanoSIMS data seemed to reveal a few hotspots in each of the particles that possibly indicated presolar material. However, closer examination suggested that it is more likely that these anomalies were, instead, caused by topographical effects during ion collection in the NanoSIMS.

In order to further narrow down our selection of particles, we took Raman spectra for each of the seven particles that had undergone NanoSIMS analysis (Fig. 4). All spectra showed large D and G-band peaks for C at 1334 cm\(^{-1}\) and 1582 cm\(^{-1}\). The first four Raman spectra in Figure 4 correspond to those particles that displayed large carbon peaks in their EDX spectra (Fig. 3). The sharpness of their D and G-band peaks indicates that these particles likely consist of disordered graphite. This, along with the lack of any other obvious minerals, suggests that these particles are terrestrial contaminants. The other three particles’ D and G-band peaks are smoother and resemble peaks belonging to IOM. The L2008 H2 and L2008 C7 spectra contain peaks at 669 cm\(^{-1}\) that suggests the presence of phyllosilicates, though the peak is much less apparent in L2008 C7. There is also a peak at 2139 cm\(^{-1}\) in one of the L2008 H2 spectra, which may be indicative of a – CN functional group.

![Figure 4. The Raman spectra for each of the seven particles analyzed. Two spectra were taken for L2008 H2.](image-url)
With our selection of particles narrowed down to L2008 H2, L2008 C7, and L2021 E39, we returned to NanoSIMS imaging for a more extensive analysis of C, N, and O isotopes. Once again, no areas indicative of presolar grains were observed in the data. Any anomalous regions we observed were once again due to topographical effects.

4. CONCLUSION

Through a more detailed SEM-EDX analysis, our survey showed that many of these ‘low-Z’ particles do appear to be terrestrial contaminants. Analysis using Raman spectroscopy further narrowed down our selection of particles, classifying those particles with high C content as contaminants as well. The remaining particles displayed chondritic elemental spectra, inclusions of IOM, and fine-grained textures, all traits common to IDPs. Nevertheless, each of these particles lacked presolar grains or other isotopically anomalous components. This prevents us from making a definitive categorization of these particles as IDPs. However, after additional NanoSIMS ion imaging of C, N, and O, and the continued absence of isotopic anomalies, it seems unlikely that these particles have an extraterrestrial origin.

5. REFERENCES

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A1. APPENDIX I: Secondary Ion Mass Spectrometry

Mass spectrometry is a technique used to determine the isotopic composition of a sample. In general, ions are accelerated through an electric field, so all particles are traveling with a uniform energy. Magnets are used along the path of the ion beam to focus it through sets of lenses and apertures, so it will impact the detectors appropriately. Eventually, the ions pass through a large magnetic field arranged perpendicularly to the beam. This causes all of the isotopes in a sample to curve towards a set of detectors at different radii dependant upon their momentum (or masses). This procedure is most easily applied to gasses, which can be more readily be ionized than solids.

When analyzing a solid sample, this process requires an extra step in order to ionize the sample. A primary beam of ions is directed toward the sample knocking off particles as secondary ions and implanting themselves in the sample. This process, known as secondary ion mass spectrometry (SIMS), allows for depth profiling as a solid sample is rastered over, and more of the primary ions become implanted in the sample. However, this method of sample analysis is a destructive procedure, as the primary ion beam knocks off material from the sample. In addition, only a small fraction of the atoms are knocked off in a charged/ionized state that can be sent through the rest of the detector, while the remaining atoms are simply sputtered away. A sample can therefore be damaged, preventing any further characterization.

The NanoSIMS instrument allows for the analysis of particles at a high resolution down to the size of 50nm and is thereby one of the most sensitive forms of mass spectrometry. The instrument has the capability to use a primary ion beam of Cs$^+$, O$_2^+$, O$^-$, and O$_2^-$ depending on the isotopes that are being measured, or the type of measurement being performed. In this experiment a Cs$^+$ beam was used for all measurements. Up to five isotopes can be detected using electron multipliers; once an ion impacts a multiplier, a cascade of electrons is set off, amplifying the signal, allowing the collection of isotopic data. Our lab group also uses software to help quantify the data taken with the NanoSIMS, allowing for statistical calculations between isotopes of an element; these include isotopic ratios, delta values (calculated from the inverse ratios compared to the terrestrial standard ratios), and sigma variations (calculated from the average or bulk ratios of a sample).
A2. APPENDIX II: Grain Picking Procedures

Outlined here are the procedures for sample preparation through grain-picking:

Figure 1. A piece of gold foil is placed on a puck (the inner disk) for analysis before grain-picking begins. This puck is placed on an aluminum stand to make it easier to handle during the procedure. The grid pressed into the foil appears as a lighter area in the center of the foil in this image.

Figure 2. The foil is placed under a high magnification reflected light microscope before grain-picking begins. Particles are transported on glass slides, as seen in front of the foil puck in this image. The micromanipulator arm (pictured on the left) holds a needle in place that is sharpened to a few micrometers at its point, which is used to transport particles from the glass slides to the foil.
Figure 3. These images were taken with a ccd camera attached to the microscope. (a) The particle of interest on the glass slide. The needle is slightly above the plane of focus, so it may be maneuvered down to the particle. Particles are held in place in silicon-oil droplets, which can make it difficult to pick-up each particle. (b) The particle can be seen on the end of the needle after having been successfully picked up. The particles are held to the needle by electrostatic forces. After being picked up, the microscope stage is lowered and moved, so the needle is positioned above the gold foil, where the particle may be placed down.

Figure 4. The stage is slowly raised into focus as the micromanipulator arm is moved back and forth to set the particle on the gold foil. (a) The particle on the needle, before the foil is brought into focus. This image also displays another setback that may occur, where the particle flips onto the opposite side of the needle, requiring the needle to be flipped over. (b) The grid on the foil can be seen coming into focus. (c) The particle from figure 3 can be seen placed on the foil in front of the end of the needle. Its location on the grid is noted on a overview map of the foil made previously.
Figure 5. With all of the particles in place, the gold foil mount is placed inside the pressing equipment (pictured on the left). A quartz disk is cleaned and inserted in the press (pictured on the right).

Figure 6. Each half of the pressing equipment is put together and held in place by metal rods. The top disk is turned lowering the quartz disk onto the gold foil, and pressing the particles into the foil. The particles may be shifted around by this procedure or lifted up by the quartz disk due to static electricity.
Figure 7. Overview maps of a gold foil mount (a) before and (b) after being pressed. After being pressed, the particles are ready for further analysis.