

**FINAL REPORTS OF THE STARDUST ISPE: SEVEN PROBABLE INTERSTELLAR DUST PARTICLES.** Andrew J. Westphal,<sup>1\*</sup> Rhonda M. Stroud,<sup>2</sup> Hans A. Bechtel,<sup>3</sup> Frank E. Brenker,<sup>4</sup> Anna L. Butterworth,<sup>1</sup> George J. Flynn,<sup>5</sup> David R. Frank,<sup>6</sup> Zack Gainsforth,<sup>1</sup> Jon K. Hillier,<sup>7</sup> Frank Postberg,<sup>7</sup> Alexandre S. Simionovici,<sup>8</sup> Veerle J. Sterken,<sup>9</sup> Carlton Allen,<sup>10</sup> David Anderson,<sup>1</sup> Asna Ansari,<sup>11</sup> Saša Bajt,<sup>12</sup> Ron K. Bastien,<sup>6</sup> Nabil Bassim,<sup>2</sup> John Bridges,<sup>13</sup> Donald E. Brownlee,<sup>14</sup> Mark Burchell,<sup>15</sup> Manfred Burghammer,<sup>16</sup> Hitesh Changela,<sup>17</sup> Peter Cloetens,<sup>18</sup> Andrew M. Davis,<sup>19</sup> Ryan Doll,<sup>20</sup> Christine Floss,<sup>20</sup> Eberhard Grün,<sup>21</sup> Philipp R. Heck,<sup>11</sup> Peter Hoppe,<sup>22</sup> Bruce Hudson,<sup>23</sup> Joachim Huth,<sup>22</sup> Anton Kearsley,<sup>24</sup> Ashley J. King,<sup>19</sup> Barry Lai,<sup>25</sup> Jan Leitner,<sup>22</sup> Laurence Lemelle,<sup>26</sup> Ariel Leonard,<sup>20</sup> Hugues Leroux,<sup>27</sup> Robert Lettieri,<sup>1</sup> William Marchant,<sup>1</sup> Larry R. Nittler,<sup>28</sup> Ryan Ogliore,<sup>29</sup> Wei Jia Ong,<sup>20</sup> Mark C. Price,<sup>15</sup> Scott A. Sandford,<sup>30</sup> Juan-Angel Sans Tresseras,<sup>18</sup> Sylvia Schmitz,<sup>4</sup> Tom Schoonjans,<sup>16</sup> Kate Schreiber,<sup>20</sup> Geert Silversmit,<sup>16</sup> Vicente A. Solé,<sup>18</sup> Ralf Srama,<sup>31</sup> Frank J. Stadermann,<sup>20</sup> Thomas Stephan,<sup>19</sup> Julien Stodolna,<sup>1</sup> Stephen Sutton,<sup>25</sup> Mario Trieloff,<sup>7</sup> Peter Tsou,<sup>32</sup> Tolek Tyliczszak,<sup>3</sup> Bart Vekemans,<sup>16</sup> Laszlo Vincze,<sup>16</sup> Joshua Von Korff,<sup>1</sup> Naomi Wordsworth,<sup>33</sup> Daniel Zevin,<sup>1</sup> Michael E. Zolensky,<sup>10</sup> 30714 Stardust@home dusters<sup>34</sup>, <sup>1</sup>*Space Sciences Laboratory, U. C. Berkeley, Berkeley, CA USA*, <sup>2</sup>*Materials Science and Technology Division, Naval Research Laboratory, Washington, DC USA*, <sup>3</sup>*Advanced Light Source, Lawrence Berkeley Laboratory, Berkeley, CA USA*, <sup>4</sup>*Geoscience Institute, Goethe University Frankfurt, Frankfurt, Germany*, <sup>5</sup>*SUNY Plattsburgh, Plattsburgh, NY USA*, <sup>6</sup>*ESCG, NASA JSC, Houston, TX USA*, <sup>7</sup>*Institut für Geowissenschaften, University of Heidelberg, Germany*, <sup>8</sup>*Institut des Sciences de la Terre, Observatoire des Sciences de l'Univers de Grenoble, Grenoble, France*, <sup>9</sup>*IRS, University Stuttgart, Stuttgart, IGEP, TU Braunschweig, Braunschweig, Germany and MPIK, Heidelberg, Germany*, <sup>10</sup>*ARES, NASA JSC, Houston, TX USA*, <sup>11</sup>*Field Museum of Natural History, Chicago, IL USA*, <sup>12</sup>*DESY, Hamburg, Germany*, <sup>13</sup>*Space Research Centre, University of Leicester, Leicester, UK*, <sup>14</sup>*Department of Astronomy, University of Washington, Seattle, WA USA*, <sup>15</sup>*University of Kent, Canterbury, Kent, UK*, <sup>16</sup>*University of Ghent, Ghent, Belgium*, <sup>17</sup>*University of New Mexico*, <sup>18</sup>*European Synchrotron Radiation Facility, Grenoble, France*, <sup>19</sup>*University of Chicago, Chicago, IL USA*, <sup>20</sup>*Washington University, St. Louis, MO USA*, <sup>21</sup>*Max-Planck-Institut für Kernphysik, Heidelberg, Germany*, <sup>22</sup>*Max-Planck-Institut für Chemie, Mainz, Germany*, <sup>23</sup>*615 William St., Apt 405, Midland, Ontario, Canada*, <sup>24</sup>*Natural History Museum, London, UK*, <sup>25</sup>*Advanced Photon Source, Argonne National Laboratory, Lemont, IL USA*, <sup>26</sup>*Ecole Normale Supérieure de Lyon, Lyon, France*, <sup>27</sup>*University Lille 1, France*, <sup>28</sup>*Carnegie Institution of Washington, Washington, DC USA*, <sup>29</sup>*University of Hawai'i at Manoa, Honolulu, HI USA*, <sup>30</sup>*NASA Ames Research Center, Moffett Field, CA USA*, <sup>31</sup>*IRS, University Stuttgart, Stuttgart, Germany*, <sup>32</sup>*Jet Propulsion Laboratory, Pasadena, CA USA*, <sup>33</sup>*Wexbury, Farthing Green Lane, Stoke Poges, South Buckinghamshire, UK*, <sup>34</sup>*Worldwide*.

## Introduction:

The Stardust spacecraft carried the first space-borne collector specifically designed to capture and return a sample of contemporary interstellar dust to terrestrial laboratories for analysis [1]. The collector was exposed to the interstellar dust stream in two periods in 2000 and 2002 with a total exposure of  $\sim 1.8 \times 10^6 \text{ m}^2 \text{ sec}$ . Approximately 85% of the collector consisted of aerogel, and the remainder consisted of Al foils. The Stardust Interstellar Preliminary Examination (ISPE) was a consortium-based effort to characterize the collection in sufficient detail to enable future investigators to make informed sample requests. Among the questions to be answered were these: How many impacts are consistent in their characteristics with interstellar dust, with interplanetary dust, and with secondary ejecta from impacts on the spacecraft? Are the materials amorphous or crystalline? Are organics detectable? An additional goal of the ISPE was to develop or refine the techniques for preparation, analysis, and curation of these tiny samples, expected to be  $\sim 1$  picogram or smaller, roughly three orders of magnitude smaller in mass than the samples in other small particle collections in NASA's collections — the cometary samples returned by Stardust, and the collection of Interplanetary Dust Particles collected in the stratosphere.

## Methods:

The ISPE consisted of several interdependent projects. More than 30,000 volunteers carried out **track identification in aerogel** [2] by searching stacks of digital optical images of the aerogel collectors, using an online virtual microscope.

**Sample preparation** [3] consisted of extraction of candidate tracks from aerogel in “picokeystones” using techniques developed specifically for Stardust, and extraction and mounting of Al foils on foil “stretchers” for subsequent SEM scanning. Seven laboratories participated in **crater identification in foils** [12], using automated SEM imaging and a combination of automated and visual identification. We carried out **Fourier Transform Infrared (FTIR) Spectroscopy** [4] on two diffraction-limited, synchrotron FTIR beamlines at the Advanced Light Source (ALS) and the National Synchrotron Light Source (NSLS). **Scanning Transmission X-ray Microscopy (STXM)** [5] was carried out at  $\sim 30 \text{ nm}$  spatial resolution on STXM beamlines at the ALS. **Synchrotron-based X-ray Fluorescence (XRF) Spectroscopy** [6,7,8] was carried out at 100–400 nm spatial resolution on two XRF beamlines at the European Synchrotron Radiation Facility (ESRF) and an XRF beamline at the Advanced Photon Source (APS). **Synchrotron-based X-ray Diffraction (XRD)** [9] was carried out at ESRF in parallel with XRF analysis. **Laboratory-based interstellar dust capture analog experiments** [10] were conducted at the Heidelberg Dust Accelerator. We did **numerical modeling of interstellar dust propagation and kinetics** [3,11] to support the interpretation of track and crater observations. We did **elemental and isotopic composition measurements of craters** [12] by SEM/EDS, Auger spectroscopy, and NanoSIMS. Crater cross-sections were extracted with focused ion beam (FIB) microscopy with a FEI Nova 600 FIB-SEM at the Naval Research Laboratory (NRL), and subsequently ana-

lyzed with a JEOL 2200FS TEM at NRL. We carried out O-isotopic analysis on sections of two of interstellar dust impact candidates, using the NanoSIMS at the Carnegie Institution of Washington.

#### Results:

In a scanned area of  $\sim 250 \text{ cm}^2$  of aerogel, we identified 71 tracks. Most of the tracks had trajectories that were consistent with an origin as ejecta from impacts on the solar panels. XRF and STXM analyses of a subset of the tracks showed the presence of Ce, which is consistent with the Ce-rich cover glass of the solar panels, confirming this origin. Twenty five tracks showed trajectories consistent with an origin either in the interstellar dust stream, or as secondary ejecta from impacts on the deck of the Sample Return Capsule (SRC). Because of the extremely limited amount of sample, we chose to analyze 13 of these tracks. Most were rejected as interstellar candidates through X-ray Absorption Near-Edge Spectroscopy (XANES) at the Al K-edge, which showed the presence of Al metal, or by the detection of F, consistent with measurements of the anodization layer in samples removed from the Stardust SRC at the National Air and Space Museum. We identified three particles that have a likely interstellar origin. Two particles, Orion and Hylabrook, were captured at low speed: comparison of track morphology with experiments carried out at Heidelberg indicate capture speeds  $\ll 10 \text{ km sec}^{-1}$ . These particles at least partially survived capture in the aerogel. XRF and XRD analyses show the presence of crystalline olivine in both, and an additional spinel component in one. Both particles contain significant Fe-bearing phases that may consist of reduced iron nanoparticles. Both particles have low overall densities,  $< 1 \text{ g cm}^{-3}$ . The third particle, Sorok, was apparently captured at very high speed ( $\gg 10 \text{ km sec}^{-1}$ ), based on a comparison of track morphology with laboratory experiments. No residual particle survived impact. While no organic matter was identified in any of the three particles identified as likely of interstellar origin, infrared spectroscopy did identify organic matter in a contaminant particle of similar size, demonstrating the necessary sensitivity.

We identified 25 impacts in  $\sim 5 \text{ cm}^2$  of the Al foils. Four impacts contained projectile residues consistent with impacts of extraterrestrial projectiles. These particles show a diversity of composition and structural complexity. EDS analysis shows the presence of Mg-rich silicates, sulfides and Fe, Ni metal, in varying proportions. The morphology of one crater is indicative of a particle with two centers of mass.

#### Discussion:

The observed low densities of Orion and Hylabrook, their capture speeds, and their specific trajectories are all consistent with the hypothesis that a large fraction of interstellar dust particles in the picogram size range consist of low-density particles, perhaps agglomerates with a fractal-like structure [11], that are efficiently repelled by solar photon pressure as they enter the heliosphere. Such particles would be slowed and diverted from the nominal interstellar dust radiant in a direction that is consistent with our observations. This would also be consistent with our observation that the flux of particles in this

size range is much lower than expected. Particles with a ratio of solar radiation force to gravitation force  $\beta > 1.6$  would not have penetrated the solar system to the Stardust spacecraft orbit, so could not have been collected. However, as shown by the high-speed particle Sorok, it appears that not all particles have such characteristics. Therefore, from the point of view of future mission planning for an interstellar dust collector, the news is mixed: the flux is lower than expected, but a significant fraction of the particles have characteristics that allow them to be captured nearly intact. Missions would maximize particle statistics by carrying out exposures during solar minimum.

It is known from astronomical observations that most silicates in the ISM are amorphous. While the presence of crystalline materials in Orion and Hylabrook was unexpected, it is not inconsistent with the astronomical observations, because particles in this size range and larger compose  $\ll 1\%$  of the mass of ISM dust, and self-shielding in such particles can protect crystalline materials from the effects of amorphizing radiation.

On the other hand, the flux of very small ( $\sim 200\text{--}300 \text{ nm}$ ) particles in the foils was larger than expected. Although the trajectories of these particles are not well-constrained, it is thought that these are statistically likely to be interstellar in origin. We carried out O-isotopic analysis on FIB sections of two candidates, using the NanoSIMS at the Carnegie Institution of Washington. Astronomical observations show that local galactic oxygen is  $\sim 25\%$  richer in  $^{17}\text{O}$ , on average, relative to the Solar System [12]. NanoSIMS measurements of FIB cross-sections of two interstellar candidates revealed O-isotopic compositions consistent with solar values, within errors. While a deviation from solar values would have been an indication of interstellar origin, the converse is not true.

We conclude that the number of  $\sim 1 \text{ pg}$  particles that were likely captured in the aerogel is extremely limited, perhaps on the order of 12. Because no future interstellar dust collection missions are even in the planning stage, the Stardust interstellar dust collection is likely to be the unique for at least two decades, and perhaps longer. It follows that the collection must be treated with extreme care. No isotopic measurements have been carried out for the particles captured in aerogel. Before any destructive analyses (isotopic analysis, ultramicrotomy for TEM analysis) can be considered, it will be necessary to carry out a major effort to validate end-to-end sample preparation and analytical techniques with sufficiently high statistics that high confidence in the protocols can be established.

**References:** [1] Tsou, P., *et al.* (2003) JGR, 108.SRD3-1:21 [2] Westphal, A. J., *et al.* (2014) MAPS, in press. [3] Frank, D., *et al.* (2014) MAPS, in press. [4] Bechtel, H., *et al.* (2014) MAPS, in press. [5] Butterworth, A., *et al.* (2014) MAPS, in press. [6] Brenker, F., *et al.* (2014) MAPS, in press. [7] Simionovici, A., *et al.* (2014) MAPS, in press. [8] Flynn, G., *et al.* (2014) MAPS, in press. [9] Gainsforth, Z., *et al.* (2014) MAPS, in press. [10] Postberg, F., *et al.* (2014) MAPS, in press. [11] Sterken, V., *et al.* (2014) MAPS, in press. [12] Stroud, R., *et al.* (2014) MAPS, in press. [13] Young, E. D., *et al.* (2011) ApJ 729, 43.