Laboratory Analysis of Returned Samples
Abstracts of selected proposals.
(2NNH10ZDA001N-LARS)

Below are the abstracts of proposals selected for funding for the Laboratory Analysis of Returned Samples program. Principal Investigator (PI) name, institution, and proposal title are also included. 20 proposals were received in response to this opportunity, and 10 were selected for funding.

Gary Huss/University of Hawaii at Manoa
Isotopic Analysis of Stardust Samples by Ion Microprobe

This proposal requests funding to four years to study the mineralogy and isotopic compositions of samples returned by the Stardust Mission to comet 81P/Wild 2. This work is part of a consortium study with Andrew Westphal’s group at UC Berkeley that will carry out complete chemical, mineralogical/petrological, and isotopic studies of Stardust cometary particles. Dr. Westphal’s group will carry out coordinated micro-XAS/XSRF/SXRD/XANES/STXM/TEM work on Stardust tracks and particles prior to sending them to us. Our part of the work will be to carry out Raman spectroscopic measurements of mineral grains and foil-crater residues to determine or confirm their mineralogy and to measure isotopic compositions of grains and crater residues. We will concentrate initially on oxygen isotopes and expect to be able to measure grains as small as 2-3 microns with a precision of ~2 permil. We have developed the experimental protocol to make these measurements and have demonstrated this precision on analog grains. Isotope ratio imaging using the SCAPS detector will be part of this work. We will also investigate the 26Al-26Mg systematics of suitable grains. Other elements, such as sulfur, can also be targets for isotopic studies. In addition to measuring individual particles from Stardust tracks, we will measure the isotopic compositions or residues in comet-side foils. Our consortium has already been allocated five tracks and three foils, and work has been proceeding to get them ready for isotopic measurements. Several grains and craters are ready for isotopic measurements.

Hope Ishii/Lawrence Livermore National Laboratory
Deciphering Comet Wild 2 Dust: Refractory Component Formation, Analogue Capture Effects and Coordinated Analysis on New Stardust Particles

The primary objective of this proposal is increased understanding of comet 81P/Wild 2 in relationship to other small solar system bodies. In particular, we will explore the formation environments of its refractory particles and time scales over which they formed and the degree of alteration experienced during capture via analogue light gas gun shots. In addition, we will expand the subset of Stardust impact tracks for which basic mineralogical and petrographic information is available by preparing and studying additional, as-yet-unexplored impact tracks. We propose to accomplish these goals by applying the new sample preparation developments and multi-technique, round-robin approaches that we have developed in our collaboration to enable study of the
mineralogy, petrography, trace element chemistry, organics and isotope compositions of particles often with nanometer to micron grains sizes. The results of this research will offer insight into the variability and transport of refractory objects prior to incorporation in comet Wild 2 and how the refractory components compare to their meteoritic cousins, the survivability and recognizability of products of aqueous alteration in the captured Wild 2 sample for comparisons to Tempel 1, chemical intermixing effects of hypervelocity impact by aggregate particles, and fundamental mineralogical and petrographic information on new comet Wild 2 dust particles, any one of which could be key to our understanding of early solar system processes effecting comet accretion. This work addresses the NASA LARS Program goals of maximizing scientific return from Stardust samples by analysis through combined analytical instrumentation and of investigating the details of the capture process.

Scott Messenger/NASA Johnson Space Center

Multianalytical Studies of Stardust Mission Samples

We propose to study the early history of the Solar System and the origins of cometary materials through coordinated studies of the organic, mineralogical, and isotopic properties of Stardust mission samples. Our primary objectives are to (1) determine the origins and histories of organic matter in comets and thereby delineate the lifecycle of organic matter in the Solar System, (2) identify the origins of cometary crystalline silicates and their relationship with meteoritic materials (3) measure the abundance and nature of presolar materials in comet Wild 2, (4) characterize cometary amorphous silicates and establish their potential relationship with amorphous silicates in cometary interplanetary dust particles and (5) determine whether any materials formed by planetary processes in comet Wild 2, including hydrothermal and shock processes.

We will perform these studies with a uniquely powerful analytical arsenal, including a one-of-a kind resonance laser ionization mass spectrometer for measuring organic molecules with high sensitivity and selectivity, a field emission transmission electron microscope for determining mineralogy and chemical compositions at nanometer spatial scales, and a NanoSIMS ion microprobe for measuring isotopic compositions on submicrometer scales. We will analyze Stardust samples in a carefully coordinated analytical sequence that will maximize the information and therefore the science that can be obtained from these precious samples. Our approach will be the comprehensive analysis of materials in entire Stardust tracks; we will perform all sample documentation and processing from start to finish in house. Ultimately, we will return to the NASA curatorial office the great majority of our samples together with a complete description of the samples and an extensive database of our findings for use by the greater community.

The proposed research is relevant to the overarching NASA goal of advancing the scientific knowledge of the origin and history of the Solar System. Furthermore it is directly responsive to the goals of the Laboratory Analysis of Returned Samples Program (LARS) by broadening the types of studies applied to the returned Stardust samples which in turn will: (1) Enhance the scientific return of the Stardust Mission. (2) Improve our understanding of the evolution of the outer Solar System. (3) Determine state of preservation of presolar and nebula materials in cometary bodies. (4) Provide insight into dynamical processes operating in the early outer Solar System. Finally this work will
also have direct overlap with the goals and objectives of NASA Cosmochemistry, Origins, and Exobiology programs, which also seek to understand the origin of the Solar System and the nature of the materials that gave rise to it.

Robert Pepin/University of Minnesota
The Composition of Solar Wind Nitrogen Implanted in Genesis Collector Materials

The issue of the isotopic composition of solar wind (SW) nitrogen, the second highest priority of the Genesis mission, is still not completely resolved. Three sets of Genesis measurements currently exist. Two of these, from laboratories at CRPG-Nancy/Canada and UCLA using very different experimental techniques, are on nitrogen extracted from Genesis concentrator materials. The SW-15N/14N ratios deduced from these two data sets agree within error with each other and with nitrogen composition in the Jupiter atmosphere. This would appear to settle the question. However measurements at Minnesota, in this case on nitrogen implanted in non-concentrator gold-on-sapphire (AuOS) collectors exposed to the bulk solar wind, give a substantially different ratio. Nitrogen data from five AuOS samples, extracted by a technique not used in the other laboratories, yield 15N/14N ratios attributed to SW-N that cluster closely around a value more than a factor of two higher than the CRPG/Nancy and UCLA ratios. Thorough reviews of the Minnesota analytic and data reduction procedures on calibrations, blanks, unflown AuOS reference material, and SW-exposed AuOS samples flown on Genesis reveal no evidence for experimental uncertainties that could plausibly account for this large discrepancy. Under these circumstances it is important to continue to investigate the possibility that we are dealing with fundamental differences in the way the solar wind N signature is recorded in collectors of varying physics and chemical properties or subject to different SW irradiation conditions, or, alternatively, that one or more of the data sets or interpretations is flawed in some way. We therefore propose to continue and extend our current Genesis nitrogen study, now with an experimental focus on analysis of concentrator target material to see if the Minnesota analytical technique reproduces the CRPG/Nancy-UCLA results. Since that technique involves N release by amalgamation, its application is restricted to gold-coated collectors. In the concentrator the only SW-irradiated material suitable for amalgamation is the gold-plated cross assembly used to support the concentrator targets. This was the material analyzed for nitrogen by the CRPG/Nancy group, and so a direct comparison of results obtained by two different extraction techniques is possible. A request for initial samples of the cross assembly has been approved by the Genesis allocation committee for amalgamation analyses at Minnesota.

Eric Silver/Smithsonian Astrophysical Observatory
Advanced Chemical Analysis of Cometary Material and Interstellar Dust Using a Microcalorimeter and A Low Vacuum Scanning Electron Microscope

Without question one of the most exciting developments in cosmochemistry was the delivery to Earth in 2006 of dust grains collected from the coma of comet Wild-2 by NASA’s STARDUST spacecraft. Comets likely formed in the outermost reaches of our solar system and thus consist of the most primitive (largely unprocessed) early solar
system material. Preliminary examination of the STARDUST material revealed many surprises, such as the almost complete (at least, so far) absence of water and the presence of a calcium-aluminum-rich inclusion and other kinds of presumably high-temperature grains (e.g. Ca-rich olivine). The existence of these particles has already had a major implication for solar nebula models, namely that there appears to be a very efficient mechanism for delivering high-temperature materials to the outer solar system region where comets accreted. The STARDUST cometary material and interstellar dust collections were collected in a low-density silica aerogel. They are the most technically challenging of all of NASA's extraterrestrial collections. Cometary impactors are fine-grained and fragile, so they disintegrate on impact with the aerogel. The largest and most robust particles penetrate deep into the aerogel, but the smallest fragments are distributed non-uniformly along the lengths of carrot-shaped impact tracks. Tracks are now routinely extracted from aerogel tiles in wedge-shaped keystones and preparing them for analysis is tedious and very time consuming. In February, 2008, NASA awarded us funds for two years to develop an instrument to advance the chemical analysis of cometary particles and interstellar dust returned by the STARDUST spacecraft. The objective has been to marry two, state-of-the art technologies. One is a cryogenic x-ray microcalorimeter spectrometer built at the Smithsonian Astrophysical Observatory and the other is a high resolution, environmental scanning electron microscope (ESEM) modified to permit material-selective, gas-mediated, electron beam-induced etching (EBIE). As an integrated system, the microcalorimeter-ESEM capabilities are unique -- it can remove in-situ the aerogel encapsulating the cometary particles without damaging or modifying the sample material. Once the particles are exposed, broad band, high resolution x-ray analysis using the microcalorimeter is performed. EBIE promises to enable finer machining of aerogel to expose individual small particles. This may be particularly useful in the bulbs of Stardust tracks where it is currently difficult to identify cometary particles optically. The ability to collect and monitor broad band, high resolution x-ray spectra during EBIE etching will minimize or entirely prevent etching of cometary particles during exposure. With the aerogel removed by EBIE, major elemental abundances in particles can be characterized by the microcalorimeter. Its increased spectral resolution (2.5 - 6 eV) over the energy range of 0.2 keV to 10 keV compared with present generation EDS (Si(Li), germanium or silicon drift) detectors provides better element-to-element discrimination and the added ability to observe shifts in characteristic emission lines due to chemical bonds. In this renewal proposal, we report that all of our most important technical goals have been accomplished. These achievements are reviewed and serve as a progress report and foundation for the few additional tasks that are required to finish the instrument. Funds are requested to complete these construction tasks and for analyzing the STARDUST particles.

Christine Floss/Washington University
Advancement of Microanalytical Techniques for the Characterization of Returned Samples

A comprehensive microanalytical characterization of returned space samples is critical for maximizing the scientific return from such missions. Although our previous development efforts in secondary ion mass spectrometry and Auger spectroscopy have
been highly successful in providing the necessary technological framework for a detailed microcharacterization of returned samples, we have also encountered new analytical challenges in our work with materials from both the Stardust and Genesis missions. In several cases we have identified new analytical approaches that, after some development effort, will lead to significant advancements in the characterization of returned samples and our understanding of the solar system or pre-solar history of these materials. Here we propose several development projects aimed at improving the microanalytical capabilities for the isotopic, elemental, and structural characterization of extraterrestrial materials from space return missions.

Rhonda Stroud/Naval Research Laboratory
Acquisition of PRISM: a Picometer-Resolution Imaging and Spectroscopy Microscope

We request funding for the acquisition of an aberration-corrected, cold-field emission, dedicated scanning transmission electron microscope with electron-energy loss and energy dispersive X-ray spectrometers. The proposed microscope, which we call PRISM, for Picometer-Resolution Imaging and Spectroscopy Microscope, will enable imaging and chemical speciation of returned samples and related planetary materials at scales down to the single atom. The critical capabilities this instrument will bring to the Cosmochemistry community are: (1) atomic-resolution imaging at an operating voltage range of 60kV to 200 kV, which at the low end is below the 80 kV displacement damage threshold for C-C bonds and thus optimized for analysis of organics, nanodiamonds and other carbonaceous materials, and at the high end of 200 kV, is optimized for the thicker specimens required for coordinated-structure isotope studies on the same specimen; (2) ability to image single atoms of Z > 4, i.e., B and heavier, at low voltage; (3) simultaneous high energy-resolution (<0.4eV) with high probe-current (> 1 nA) and (4) a spot size as small as 80 pm for electron energy loss detection and oxidation state analysis of trace elements.

The Naval Research Laboratory has made a substantial commitment to success of the proposed facility in the form of existing, high-quality laboratory space, and $1.2M in matching funds. PRISM will be housed in the Nanoscience Institute at the Naval Research Laboratory, a special purpose facility built to meet the environmental control specifications of aberration-corrected electron microscopes and other sensitive nanoscience equipment.

Our planned studies of Stardust, interplanetary dust and meteoritic organics and minerals using PRISM will help further our understanding of early solar system processes, including the formation and alteration of prebiotic organic compounds, and evolution of the chemical composition of the solar system in line with the goals of NASA Strategic objective 3c: “advance scientific knowledge of the origin and history of the solar system, the potential for life elsewhere, and the hazards and resources present as humans explore space.”
Rhonda Stroud/Naval Research Laboratory
Structure-Isotope Studies of Extrasolar and Nebular Processing Signatures in Stardust Crater and Track Samples

We propose a multifaceted, electron beam, ion beam and x-ray microscopy-based effort to better understand the mineralogy, chemistry and isotopic composition of STARDUST samples, both cometary and interstellar, captured in aerogel and in Al foil. Our specific research goals for this proposal fall into four general task areas: (1) microanalysis of impact craters on cometary and interstellar foil (2) analysis of the chemistry, microstructure and isotopic compositions of organic-rich cometary particles (3) mineralogical studies of cometary particles captured in aerogel; and (4) development of FIB techniques for rapid mounting and bisection of particles. The knowledge gained from these studies will improve our understanding of the formation of minerals and solid organics in the early solar system and the interstellar medium, and their incorporation into planetary bodies, such as comets and asteroids. This research is in line with the goals of NASA Strategic objective 3c: “advance scientific knowledge of the origin and history of the solar system, the potential for life elsewhere, and the hazards and resources present as humans explore space.”

Mark Thiemens/University of California, San Diego
Nanometer Scale Infrared Spectral Mapping of Crystalline and Amorphous SiO2, SiC, and Nitrile Functional Groups in Returned Samples

Using a recently developed infrared (IR) nanoscope (funded by SRLIDAP 2007), we have demonstrated the ability to make IR spectral maps of SiC, various forms of carbon including graphene and graphite, SiO_2 and Si with 10 nm spatial resolutions in laboratory standards. Using a broadband IR wavelength (4-10 microns) approach, we have recently demonstrated the ability of our novel instrument to distinguish between crystalline and amorphous SiO_2. Here, we propose to extend this novel analytical technique to map cosmochemically significant functional groups such as SiO_2 (crystalline and amorphous), SiC, graphene and graphite, and nitrile (CN) at the 10 nanometer scale in natural samples. After the preparation and characterization of "natural standards" to validate the response of the instrument, we propose to measure and map individual cometary dust grains from the Stardust Mission. We emphasize that the sample preparation and standardization techniques developed as part of this proposal are expected to be widely applicable for the characterization of returned samples and, as such, directly address the main goals of the Laboratory Analysis of Returned Samples Program. In addition, this instrument, which includes an atomic force microscope, is capable of detecting and mapping silicate dust contamination on the surface of the Genesis SiC wafers and funding for this purpose has also been requested.
Andrew Westphal/U. C. Berkeley
Coordinated Synchrotron X-Ray Microprobe, TEM, and Ion Microprobe Analysis
of Stardust Cometary Samples

We propose to carry out coordinated analyses of Stardust cometary samples, using
synchrotron x-ray microprobes at the Advanced Light Source, Transmission Electron
Microscopes at the National Center for Electron Microscopy, and an Cameca 1280
secondary ion microprobe at the University of Hawai'i.