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Introduction: “Nothing tends so much to the advancement of knowledge as the application of a new instrument,” noted the 19th century English chemist Sir David Humphrey. The Laboratory for Space Sciences at Washington University in St. Louis has a strong history in the acquisition and development of micro-analytical instrumentation for the advancement of research in the planetary and space sciences.

Our record dates back to the early 1980s when, through the vision of the late Robert M. Walker, one of the first Cameca ims 3f SIMS (Secondary Ion Mass Spectrometry) instruments was installed in our laboratory. Under the leadership of the late Ernst Zinner, groundbreaking work carried out by our group contributed significantly to the establishment of SIMS as a standard tool in geological and space science laboratories around the world [1].

In the decades since, we have remained at the forefront of micro-analytical innovation. Advances such as the development (together with Cameca) of the NanoSIMS, a new type of SIMS instrument [2], and acquisition of the scanning Auger Nanoprobe for nanometer-scale compositional information [3], have led to significant scientific advances in the fields of cosmochemistry, astrophysics and planetary science.

An important aspect of our success over the years has been the ‘open door’ policy we have maintained for external collaborations. We strongly believe that advanced analytical equipment and operational know-how should be shared within the scientific community with outside colleagues, and that such access is the best approach for enabling progress in diverse scientific fields.

Below we describe the capabilities of the major instrumentation present in our lab and some of the key significant scientific advances to result from application of these instruments. An integral part of our Laboratory for Space Sciences group is the noble gas laboratory run by Alex Meshik and Olga Pravdivtseva. Although our group functions as a synergistic whole, due to space limitations the unique capabilities of this laboratory are described in a separate abstract [4].

Cameca NanoSIMS 50: The NanoSIMS is a novel type of ion microprobe that was specifically designed to optimize both sensitivity and spatial resolution. Its most significant features are a very small Cs⁺ primary beam (~100 nm), a high secondary ion yield even at high mass resolution, and the ability to simultaneously measure up to five masses using moveable detectors

located in the focal plane of the spectrometer. In addition, the NanoSIMS offers raster imaging, secondary electron detection and normal-incidence electron-gun charge compensation. Additional in-house development work allows automated particle definition in which discrete grains can be identified from ion images by image processing algorithms and then analyzed individually for precise isotopic determination, enabling rapid isotopic characterization of large populations of grains.

PHI 700 Scanning Auger Nanoprobe: Our state-of-the-art scanning Auger spectrometer operates by measuring the characteristic energies of Auger electrons produced by interaction of an electron beam with a sample. Elemental compositions are determined from a surface layer only a few nanometers in depth, allowing for accurate in situ compositional measurements of submicron grains. The PHI 700 features a field emission electron source with a beam size of less than 10 nm and a cylindrical mirror analyzer resulting in coaxial electron gun and Auger electron analyzer geometry. This eliminates shadowing effects and ensures that NanoSIMS and Auger analyses measure the same sample areas regardless of topological variations. The instrument is equipped with a secondary electron detector and an Ar⁺ ion sputter gun for specimen cleaning and depth profiling.

FEI Quanta 3-D FEG Dual-Beam FIB-SEM: Our dual-beam FIB-SEM is equipped with a NG Schottky FEG emitter electron source for high-resolution imaging and a liquid Ga ion emitter for fast and precise specimen milling. The instrument is also equipped with a Pt gas injection system, and an Omniprobe AutoProbe 200.2 micromanipulator for the preparation and lift-out of electron-transparent thin sections ready for TEM analysis.

Other Instrumentation: We also have a JEOL 840a SEM equipped with a LaB₆ emitter and a Noran Si(Li) light element detector. This instrument was upgraded in 2008 with a Deben SPRITE 3-axis stage controller and with a Noran System Seven spectral imaging data acquisition computer system.

In addition, the Institute of Materials Science & Engineering has a state-of-the-art field emission TEM to which we have access on an as-needed basis. The instrument is equipped with a Schottky FEG emitter for micro-diffraction, convergent-beam, and electron spectroscopic studies, a high-resolution pole piece (C_s=1.0 mm), allowing point to point resolution of 0.23 nm, and lattice resolution of 0.1 nm, an analytical

scanning imaging device (ASID) for scanning (S)-TEM mode of operation and a Bruker Xflash 5060T solid state (Si drift) x-ray detector with 60 mm² active area, which allows rapid spectral acquisition and mapping even at low beam currents. The TEM is ideally suited for determination of the microstructures and crystallinities of small isotopically anomalous presolar grains.

We also have access to a state-of-the-art Cameca ims 7f-geo ion microprobe, housed in the Earth and Planetary Sciences Department. The ims 7f features two separate ion sources, continuously adjustable acceleration and extraction voltages, microscope and microprobe imaging modes, dynamic transfer optics for imaging at high mass resolution, electron charge compensation, oxygen flooding for yield enhancement, a spatial resolution of ~1 μm , different detection options (FC, EM, channel plate), adjustable mass resolution up to $m/\Delta m = 20,000$, and the necessary control software for various analysis modes.

Other general research equipment available in our laboratory includes optical microscopes, clean benches, polishing and sawing equipment, balances, ovens, ultrasonic cleaner, evaporators, microbalances, a clean room, a convertible Au/Pt sputter coater with digital thickness monitor, an argon ion mill for sample cleaning, a Nikon E600 binocular microscope with a large working distance (1.5 cm) at high magnification (1000x) and equipped with a Patchman NP micromanipulator for grain picking and mounting, and a Reichert-Jung ultra-microtome.

Discussion: The development of the NanoSIMS, with its unique features for the study of presolar grains has allowed us to make important scientific contributions to the isotopic study of extraterrestrial materials. Among the highlights of this work are the discovery of presolar silicates in interplanetary dust particles (IDPs) [5], primitive meteorites [6] and Antarctic micrometeorites [7], the determination of presolar SiC polytypes [8], the discovery of isotopically anomalous carbonaceous phases [9], as well as SiC and corundum, in IDPs [10], the first observation of presolar grains in matter from comet Wild 2 [11-13], the correlated TEM-NanoSIMS studies of presolar grains within presolar grains [14], the determination of interstellar exposure ages of presolar SiC grains [15], and the measurement of Fe isotopes in presolar SiC and silicate grains [16,17].

Following their initial discovery with the NanoSIMS, the study of presolar silicate grains has benefited largely from our acquisition of the Auger Nanoprobe, which allows for the routine elemental characterization of these submicron grains, leading to the discovery that most are ferromagnesian silicates

with unexpectedly high Fe contents [18-21]. Combined NanoSIMS, Auger Nanoprobe and FIB-TEM studies have also led to the discovery of other new presolar phases, including magnesiowüstite, magnetite and silica [22-24], as well as the identification of contemporary interstellar dust collected by the Stardust spacecraft [25,26]. It's worth noting that many of these discoveries are the result of collaborations with external colleagues who bring their own scientific expertise, interests and samples, leading to mutually beneficial interactions and enhanced scientific advancement.

In addition to collaborations within our field of cosmochemistry, we have also sought to share our technical expertise and instrumentation with colleagues in other fields, including biogeochemistry [27], microbiology [28,29], condensed matter physics [30] and aerosol science [31]. In addition to providing unique research opportunities to colleagues from diverse scientific and institutional backgrounds, we benefit from these collaborations through the acquisition of expertise in new sample preparation methods and the development of protocols for diverse analytical needs, ultimately benefiting the larger scientific community.

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