

PARTICLE ANALYSIS SOFTWARE APPLIED TO QUANTIFYING AND MAPPING CONTAMINATION ON GENESIS FOILS AND OTHER COLLECTOR MATERIALS. A. J. Bixler¹, K. C. Welten¹, K. Nishizumi¹, M. W. Caffee², A. J. G. Jurewicz³, D. S. Woolum⁴, and D. S. Burnett⁵, ¹Space Sciences Laboratory, University of California, Berkeley, CA 94720, USA (ajbixler@berkeley.edu; kcwelten@berkeley.edu), ²PRIME Laboratory, Purdue University, West Lafayette, IN 47907, USA; ³School of Earth & Space Exploration, Arizona State University, Tempe, AZ 85287, USA; ⁴Dept. of Physics, California State University, Fullerton, CA 92834, USA; ⁵Div. Geological & Planetary Sciences, California Institute of Technology, Pasadena, CA 91125, USA.

Introduction: The Genesis mission exposed a variety of high-purity collector materials to capture the solar wind (SW) and return these SW samples to Earth for detailed elemental and isotopic analysis [1]. Upon return, the sample return capsule made an unexpected hard landing in the Utah desert, shattering many of the collector materials and contaminating them with Utah dirt (e.g., NaCl, Mg,Ca-carbonates silicates) and debris from spacecraft and SW collectors. Our main goals are to analyze: (1) SW radionuclides in a large piece (1000-4000 cm²) of Mo-coated Pt foil [2], and (2) SW Cl, Co and Ir in a small piece (~1 cm²) of Genesis Si or sapphire [3,4]. These measurements require the dissolution of the top layer of the foil/fragment that contains the SW ions, so it is important that we identify and remove (if necessary) any surface contamination that interferes with our measurements.

Previously, we used the Scanning Electron Microscope (SEM) scanning to collect backscattered electron (BSE) images of ~5000 cm² of the Mo-Pt foil. The flight foil – on average – contains ~15 µg of contamination/cm², but not all of this is Utah dirt. Moreover, some types of contamination (such as Utah dirt for SW radionuclides and SW Cl; and stainless steel for SW Co) are more problematic than others. If it was Utah dirt, then contamination is a factor of ~150 higher than our required upper limit (~100 ng of dirt/cm² [2]), but it is not all dirt. Thus, it is imperative for us to distinguish the different types of contamination; e.g., Utah dirt vs. collector materials (e.g., Si, sapphire, Ge, etc.) vs. spacecraft materials (C, Al, stainless steel, Ga,Zn-spinel paint).

Our Tescan Vega SEM instrument was already equipped with Oxford Instrument X-ray detectors for chemical analysis in Energy Dispersive Spectroscopy (EDS) mode. With our existing SEM software, we could only analyze small areas (a few mm²) in EDS mode, while scanning larger areas would require an excessive amount of time (many years for the Mo-Pt foils) and would require a lot of user intervention to both acquire and save the data. Because manually identifying and mapping contaminants for future removal (if necessary) is so labor intensive, here we present an automated alternative, the Aztec particle analysis software package for our SEM instrument.

Here we report the first results of applying this software to a piece of Genesis Mo-Pt foil and discuss some of the challenges we are still facing to optimize this software package for our purpose.

Method. The Aztec particle analysis software allows us to first scan each field of the sample in BSE mode and identify contaminant particles based on the grayscale, which represent the average Z of the surface. These particles are subsequently analyzed in EDS mode and are tagged into different chemical classes. For our first test, we scanned an area of ~4 cm² of the center Mo-Pt foil 50053,0212. This area was scanned in 144 fields of 2x2 mm² with ~10% overlap between individual fields. After BSE scanning, each feature with a minimum size of ~150 µm² is analyzed in EDS mode for 5 s, with a maximum of 15 min. (or 180 particles) for each field, yielding a total run time of ~40 hours. Here we discuss some of the preliminary results and challenges.

Results. We identified and analyzed more than 2500 features with a total area of ~1 mm², equivalent to ~0.3% of the total surface. About 500 particles are classified as Utah dirt, 500 as paint, and ~600 as Si-metal or Si-rich particles. Only 18 particles were classified as stainless steel (9) and Ge (9). We encountered several issues that need improvement: (1) The feature detection from the BSE image relies on the gray-level thresholds that we set at the start of the run. The microscope electron beam varied significantly in brightness during the run, causing variations in brightness of the BSE images acquired for each field (Fig. 1). This probably caused some variation in how many features were identified in each image. Although we applied an in-run threshold adjustment every 30 minutes, the brightness variations led to some overcorrections in the gray-level threshold. For 3 fields, the threshold was adjusted so high that the entire field was counted as a single feature (Fig. 1). These fields were omitted from the analysis. We hope that changing the filament and the annual maintenance will reduce the brightness variations. (2) The particle analysis routine timed out for 51 of the 144 fields, before it was able to analyze all the particles in that field, so effectively only 326 mm² (~80% of the surface) was analyzed by EDS. Increasing the minimum feature size

for EDS analysis by a factor of ~ 2 (in area) would reduce the total number of particles by a factor of 3, resulting in a more complete analysis of the total surface. We will have to find the right balance between speed of analysis, minimum particle size and completeness of the analysis (in terms of total surface area).

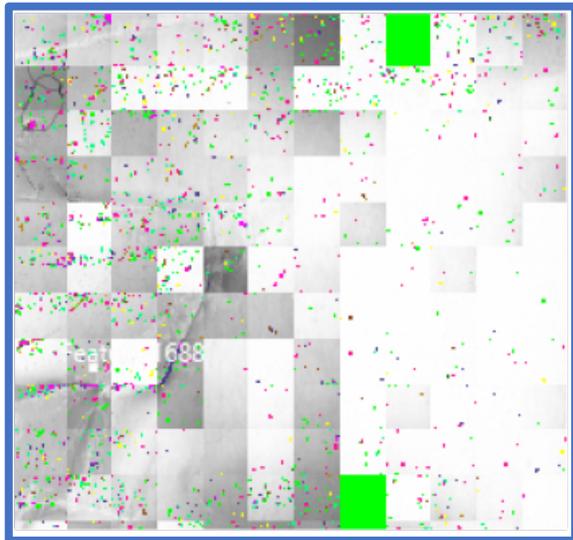


Fig. 1. Aztec Particle Analysis results of ~ 4 cm² of Genesis Mo-Pt foil 50053,0212 before cleaning. The chemical characterization of 2500 particles into 9 groups is based on Na, Mg, Al, Si, Cl, Ca, Ge, Ga and Zn. Green fields suffered from bias in threshold adjustment due to variations in image brightness.

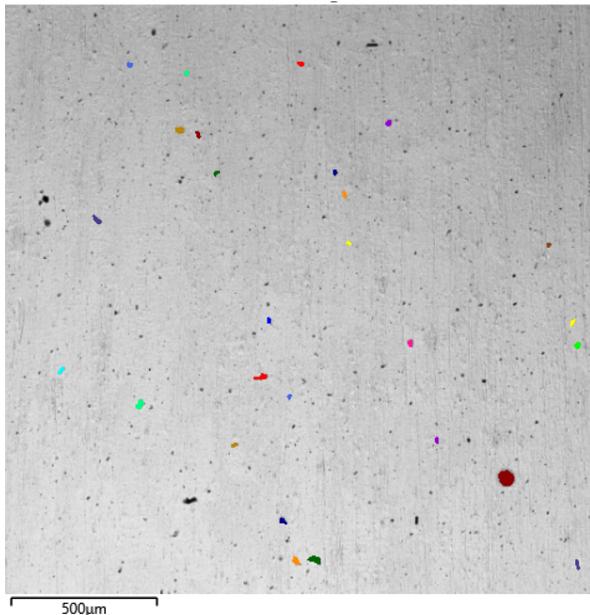


Fig. 2. Aztec Particle Analysis results of 2×2 mm² field of Genesis Mo-Pt foil 50053,0212 before cleaning. Color of particles same as in Fig. 1.

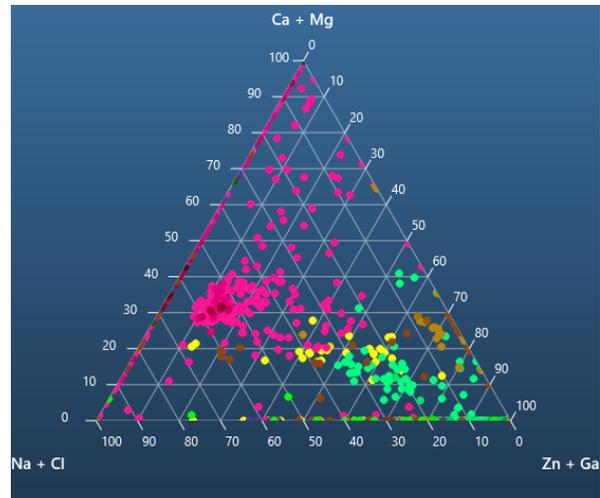


Fig. 3. Ternary diagram of Na+Cl (salt), Mg+Ca (carbonates) and Ga+Zn (paint) in features identified on Mo-Pt foil 50053,0212. Color of particles same as in Fig. 1.

(3) The X-ray spectra of many features are dominated by C, O, Mo and Pt signals, so these elements were excluded from the chemical characterization of the particles. This may cause some of the areas with MoO to be misidentified as something else if traces of other elements are also detected. (4) Many of the features identified are mixtures of dirt and paint, dirt and Al, or others, so it is difficult to classify each particle correctly. We checked the compositions of some of the Si-containing particles after the run was finished, and conclude that some should probably be classified as dirt, whereas other may be mixtures, as is also evident in Figure 3. Since the Utah dirt is the most threatening contaminant for our SW radionuclide analysis, we will further refine our classification scheme to include mixtures of dirt with other contaminants.

Conclusions: The Aztec particle analysis software on our SEM instrument allows efficient chemical characterization of the particles based on their X-ray spectra. This software makes obtaining a quantitative estimate of the remaining contamination possible within a grant period for a large area like the foil. Not only are contaminants identified by type and quantified, but their location is mapped, so problematic areas can be identified, and those regions can either be re-cleaned or removed before the final SW extraction and analysis.

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References: [1] Burnett D. S. (2013) *MAPS*, 48, 2351–2370. [2] Welten K. C. et al. (2019) *LPSC 50*, Abstract #2718. [3] Welten K. C. et al. (2018) *LPSC 49*, Abstract #2660. [4] Welten K. C. et al. (2019) *LPSC 50*, Abstract #2626.