DISCRIMINATION AND QUANTIFICATION OF CONTAMINATION AND IMPLANTED SOLAR WIND IN GENESIS COLLECTOR SHARDS USING GRAZING INCIDENCE SYNCHROTRON X-RAY TECHNIQUES: INITIAL RESULTS K. Kitts[1], S. Sutton[2, 3], P. Eng[3], S. Ghose[3], D. Burnett[4], [1]Department of Geology & Environ. Geosciences, Northern Illinois University (Davis Hall 312, Normal Rd, DeKalb, IL 60115; kkitts@niu.edu), [2]Department of Geophysical Sciences and [3]Consortium for Advanced Radiation Sources (University of Chicago, Chicago, IL 60637), [4]Geology & Planetary Sciences, Caltech (MS 100-23, Pasadena, CA 91125).

Introduction: Accurate knowledge of the composition of the Sun provides a baseline, which allows an understanding of how the solar system has evolved over time and how solar processes and solar wind mechanics behave. Unfortunately, the errors in photospheric abundances are too large for many planetary science problems and this hampers our understanding of these different processes. Analyses of solar wind implanted in meteorites or lunar soils have provided more precise data [e.g. 1] but alteration processes on these bodies may complicate such information.

In response to this need for pristine solar wind samples, NASA developed and launched the Genesis Probe. Unfortunately, the probe smashed into the Utah desert shattering the 300 collector plates into 15,000+ pieces all of which are now coated in a both a fine terrestrial dust and Si and Ge powder from the disrupted collectors themselves. The solar wind penetration depth is 100-200 nm [2] and the superposed contamination layers are typically 40-50 nm [3]. Stringent cleaning regimes have the potential of removing the solar wind itself. The best solution is to have sufficient spatial resolution to separately analyze the surface contamination and penetrated solar wind.

To that end, three Genesis collector array shards and their appropriate flight spares were characterized via grazing incidence x-ray fluorescence and x-ray reflectivity. The goals were (1) to evaluate the various cleaning methods used to eliminate contamination, (2) to identify the collector substrates most suited for this technique, (3) to determine whether the solar wind signature could be deconvolved from the collector background signature, and (4) to measure the relative abundances of Ca to Ge in the embedded solar wind.

Materials and Methods: The Curatorial Staff at Johnson Space Center provided two flown shards, one single crystal silicon (60171) measuring 1 cm by 0.4 cm and one single crystal sapphire (2x1 cm) that was subdivided with one section undergoing a solvent cleaning method (D30554c) [4] and one remaining uncleaned (D30554d).

The Genesis Team provided seven flight spares including samples of vapor deposited Al on single crystal sapphire (AlOS-00776), vapor deposited Au on single crystal sapphire (AuOS-00828), single crystal sapphire (SAP 650-4A) and amorphous diamond like carbon on silicon (ADO 71000-c, d).

The flight samples and spares were analyzed using x-ray reflectivity and grazing incidence x-ray fluorescence at APS (GSECARS sector 13 Newport General Purpose Diffractometer). The x-ray beam was derived from an APS undulator with the gap set to supply 11.5 keV photons at the undulator fundamental. A cryogenic Si (111) double-crystal monochromator was used to narrow the energy bandwidth of the beam. A combination of focusing mirrors in a Kirkpatrick-Baez geometry and slits resulted in a 20 x 20 μm x-ray beam containing ~ 10^{12} photons/sec.

The samples and spares were mounted vertically on the diffractometer enclosed in a helium-flow sample chamber containing a thin (5 μm) Mylar window. A scintillation detector was used to measure the intensity of the reflected x-ray beam and a Vortex silicon-drift energy-dispersive detector mounted in direct contact with the Mylar window was used to collect XRF spectra. To determine the critical angle (CA), reflectivity curves were obtained using θ-2θ trajectory scans with an angular sampling interval of 4 millidegrees (θ). Full XRF spectra were collected at each angle and were subsequently processed via a peak fitting routine to produce fluorescence yield profiles (peak intensity vs. angle) for each detected XRF peak between 1 and 11 keV [5].

Results: Figure 1 plots reflectivity, and K-edge fluorescent yields of Ge, Zn, Ga and Fe vs. x-ray incident angle for the flown sapphire shards D30554c (cleaned), D30554d (uncleaned) and flight spare (SAP 650-4A). The dashed line marks the CA for the substrate. The fluorescent yield axis shows relative intensity. No Ge or Ga was detected in the flight spare. The Zn yield curve of the spare has a similar profile to the flown samples but a fluence two to three orders of magnitude lower (not shown).

The source of the Ge is contamination from the disrupted Ge detectors themselves and has proven quite useful as a tracer aiding in the deconvolution of the surface contaminant signal from embedded solar wind. The Ga and Zn yield curves have the same shape as the Ge curve and all typify surface contamination profiles with a relatively flat profile below the CA, a sharp drop at the CA and a very low signal above the CA. In contrast, the peaked yield profile of the Fe suggests a near surface and/or shallow-implanted component with the yield peaked near the CA. A bulk substrate impurity profile would be approximately a mirror image of a surface contaminant profile. The silicon shard profiles are similar to those of the sapphire.

Cleaning Method Evaluation: Figure 1 shows that the solvent cleaning method is efficacious for removing Ga and Zn with Zn functioning as an indicator of terrestrial contamination. However, the Ge from the disrupted plates is particularly resistant to organic solvent cleaning methods. The Fe yield profile shows evidence of particulate Fe (plateau at low angle) on the surface of D30554c, which retained a visible "scuffmark" after cleaning. Therefore, it is unknown whether solvent cleaning is beneficial.

Collector Substrate Evaluation: The substrates that produce the highest quality spectra with the greatest potential for obtaining a unique solar wind signal are the vapor deposited Al on single crystal sapphire, the single crystal sapphire and the single crystal silicon. The spectra produced by the vapor deposited Au on single crystal sapphire substrate is complicated by the Au-M fluorescence lines and the amorphous diamond like C film on silicon is non-uniform resulting in a loss of depth control.

Solar Wind Deconvolution: By comparing the shape of the profiles and the yield abundances in Figure 1, an implanted (solar wind) component can be differentiated from both surface contamination and collector impurities. This is a particularly important result given the extensive contamination issues associated with the Genesis collection.

Inferred Solar Wind Fe Abundance: Because the surface of D30554c shows evidence of particulate Fe, the uncleaned portion (D30554d) was used for the Fe abundance determination. The Fe bulk abundance is $9.6 \times 10^{12}/cm^2$ calculated using the highest angle yield value and normalizing to that measured for a Ge-implanted silica standard (1 keV/nucleon; $5 \times 10^{14}/cm^2$). Assuming that our measured spare represents the pre-flight contamination of our flown sapphire collector, the Fe abundance of the spare can be subtracted off and the solar wind fluence inferred to be $8 \times 10^{12}/cm^2$. This is slightly higher than the predicted Fe fluence of $2 \times 10^{12}/cm^2$ [6]. Note that in Fig. 1c, the background is 20% of the signal and that if this number should vary by 50% then the inferred fluence would vary by 10%. Thus, it is necessary to analyze other samples and spares to determine the variability in pre-flight contamination/impurity levels.

Conclusions: Grazing incidence x-ray fluorescence is an efficient and non-destructive technique that can be used to differentiate the embedded solar wind component from surface contamination and collector background signature in the Genesis shards. Initial solar Fe abundance in D30554 is $8 \times 10^{12}/cm^2$ assuming the collector background contribution is the same between the flight spare and flown sample. Further study of other flown samples and flight spares is required to establish the variability in pre-flight contamination/impurity levels.

References: [1] Kitts et al., (2003) *Geochim. et Cosmochim. Acta*, **67**, 4881-4893. [2] Burnett et al., (2003) *Space Sci. Rev.*, **105**, 509-534. [3] Lauer et al. (2005) *LPSC* Abstr., **36**, #2407. [4] Procedural details for the solvent cleaning technique available from A. Jurewicz, ASU, jurewicz@gps.caltech.edu. [5] Trainor et al., (2006) *J. Electron Spectrosc. Relat. Phenom.*, 150, 66-85. [6] Genesis Curation http://curator.jsc.nasa.gov/genesis/Requests.htm.

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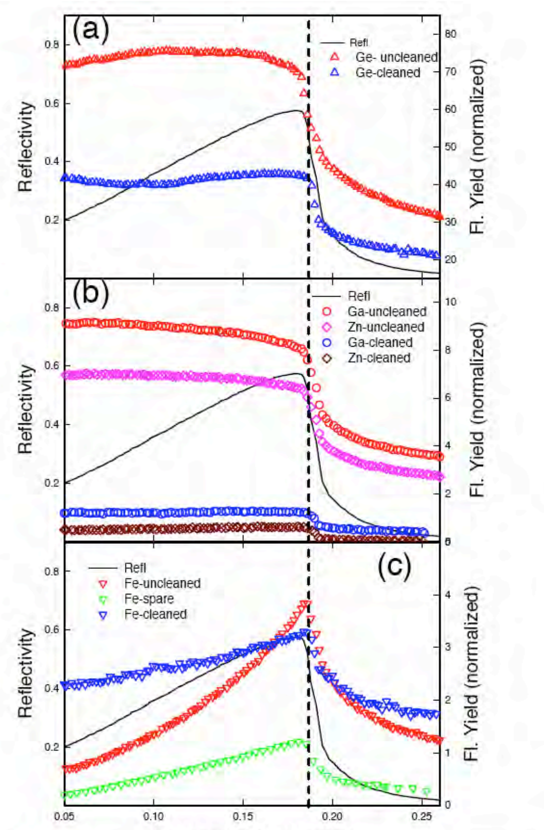

Figure 1: The reflectivity (solid line and left hand axis) and various elements k-edge fluorescent yield (symbols and right hand axis) plotted with respect to the incidence x-ray angle for flight GENESIS sapphire uncleaned (30554d) (symbol in Red/Pink), flight GENESIS sapphire cleaned (30554c) (symbol in Blue/Brown) and GENESIS sapphire spare (650-4A) (symbol in Green) substrates. The dashed line marks the critical angle for the substrate. The fluorescent yield axis shows relative concentration.