GAS CLUSTER ION BEAM CLEANING OF GENESIS SOLAR WIND SAMPLES: FURTHER STEPS IN THE METHOD EVALUATION. I. V. Veryovkin[1], M. Schmeling[2], N. Toyoda[3], T. Mashita[3], I. Yamada[3], A. J. G. Jurewicz[4], S. V. Baryshev[1], C. E. Tripa[1], A. V. Zinovev[1], and D. S. Burnett[5], [1]Materials Science Division, Argonne National Laboratory, Argonne, IL 60439, verigo@anl.gov, [2]Department of Chemistry, Loyola University Chicago, Chicago, IL 60660, [3]Graduate School of Engineering, University of Hyogo, Himeji, Hyogo, 671-2280, Japan, [4]Center for Meteorite Studies, Arizona State University, Tempe AZ 85287, [5] Division of Geological and Planetary Sciences, California Institute of Technology, Pasadena, CA 91125

Introduction: Solar Wind (SW) samples collected by the NASA Genesis Mission are hard to analyze accurately because their surfaces were contaminated during both the SW collection and the hard impact landing and breach of the Genesis sample return capsule. In the surviving pieces of SW collectors, the SW matter is implanted at shallow depths under a “blanket” of terrestrial contamination consisting of the same elements as SW but with orders of magnitude higher abundances. To date, trials of various physical and chemical cleaning methods have been used on sample surfaces. In the past few years [1, 2], we started special studies to evaluate the potential of the Gas Cluster Ion Beam (GCIB) bombardment technique [3] to remove the surface contamination quasi-non-destructively and gently from the Genesis samples. In this work we will present results of the most recent trials of this method applied to Genesis Si collectors.

Experimental: In order to characterize the GCIB-processed surfaces of Genesis samples, we use a laboratory-based Total Reflection X-Ray Spectrometry (TXRF) [4] and Resonance Ionization Mass Spectrometry (RIMS) in the *gentle dual beam* depth profiling regime [5]. The TXRF technique is non-destructive and thus permits characterization of the cleanliness of sample’s surfaces both prior to and after the GCIB irradiation. On the other hand, the RIMS method is one of the “customers” which needs the cleaned samples to per-

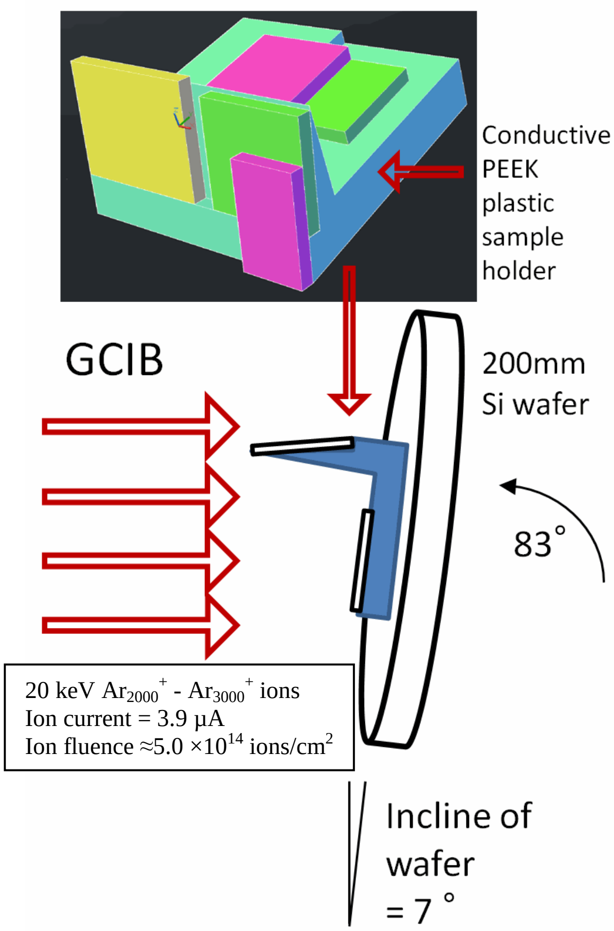

Figure 1. GCIB irradiation geometry

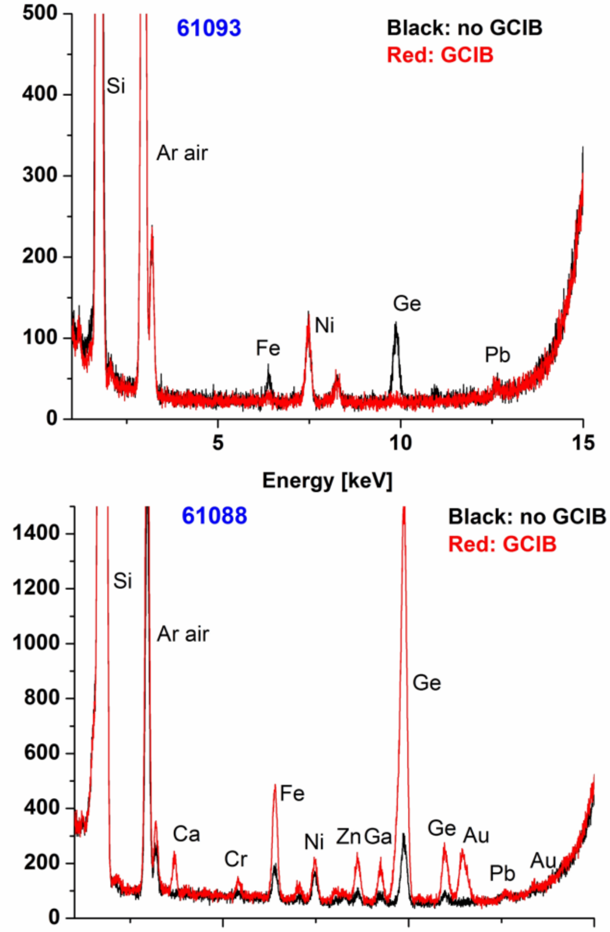

Figure 2. TXRF spectra of surface composition of Genesis samples prior to and after GCIB irradiation

form measurements of SW fluences by a destructive ion beam sputtering process. At the 43rd LPS Conference, we reported results of the GCIB cleaning evaluation done on the Genesis Si sample #60428 [2]. That sample was irradiated in orthogonal incidence geometry and one half of it was shielded from the gas cluster ion beam by a blank Si coupon in order to determine how much sample material this beam removed from the surface. Our optical profilometry measurements indicated that this number was 12 nm. As a follow-up of experiments reported for this sample in Ref. [2], we performed two more *gentle Dual Beam* depth profiling measurements with RIMS on it, and the results were inconclusive: while one spot showed reduced Cr contamination (and easily the best depth profile of SW Cr we have measured with RIMS to date), there was too much Ca contamination on it, and the other spot showed completely opposite situation - more Cr and reduced Ca contamination. Genesis 60428 sample was too small from the beginning ($\approx 3\times 5$ mm^2), and irradiating just one half of it with GCIB did not make the interpretation of analyses easier, either by TXRF or by RIMS. It became clear that more GCIB experiments are needed - in a new irradiation arrangement, which would be easier to interpret and thus obtain more conclusive results.

We hypothesized that if the surface contamination on Genesis samples, after all the cleaning procedures applied at the curatorial facility, consists mainly of nanometer-scale particles that are very hard to detect and remove, then the angle of the GCIB impact will not have much effect on the particle removal rate. Even at sample surface grazing incidence, the impact angle of GCIB onto the particles should remain near-orthogonal. Under such conditions, we can anticipate minimal ion beam damage to the Genesis sample and about the same particle destruction rate. We could even expect somewhat better cleaning because the fragments of the particles destroyed by GCIB would obtain kinetic energies in directions mostly parallel to the surface so that their re-deposition is minimized. To test these hypotheses, we designed a new sample holder for GCIB irradiation (Fig.1), which allowed to irradiate simultaneously four samples in one experiment under different conditions: (1) full surface of a Genesis Si sample at near-orthogonal impact angle, (2) part of the surface of a blank Si "witness" sample at the same near-orthogonal angle (the other part is shielded by a piece of blank Si in order to enable determination of the material removal rate by optical or contact profilometry); (3) full surface of a Genesis Si sample at grazing impact angle; (4) part of the surface of a blank Si "witness" sample at the same grazing angle (the other part is also shielded by a piece of blank Si in order to enable surface profilometry). This arrangement exposes the full surfaces of Genesis samples to the GCIB, and still allows us to quantify the amount of material removed from the SW collectors. The sample holder was manufactured from conductive PEEK plastic to reduce the potential of cross contamination. Two Genesis samples were assigned to this cleaning study in December 2012: #61093 and #61088. Chemical compositions of sample surfaces were measured with TXRF at Loyola University Chicago, and then their GCIB processing was conducted at the University of Hyogo. After receiving the samples back, we measured their surface compositions by TXRF again. The preliminary results of these measurements are presented in Fig.2. One can see that for the grazing incidence irradiation (#61093), there was indeed some contamination reduction, namely for Ge and Fe. However, for the near-orthogonal irradiation (#61088), *we observe an increase in TXRF signals from many species*. At this very moment, it is hard to explain the cause of this change. For this case, we expect that the GCIB beam has removed about 10 nm of surface material. We will soon verify this by optical profilometry on the corresponding "witness" sample. Moreover, in the nearest future, we will perform more elaborate TXRF measurements involving full angular scans so that it will be easier to say where these signals are coming from. Such a full scan was performed on this particular sample (#61088) prior to GCIB irradiation. Thus the comparison "before vs after" can be done for different X-ray incidence angles. These extra TXRF measurements will be followed with gentle dual beam RIMS measurements that potentially can give the ultimate answer, whether the GCIB sample processing makes it easier to determine the SW fluence in Genesis samples or not.

Conclusions: We have designed and conducted new experiments with cleaning Genesis samples by the Gas Cluster Ion Beam method and performed preliminary sample characterization. These experiments will be followed with an all-round samples characterization, and at the LPS Conference, we will present all these results, their interpretation and our conclusions.

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