APPLICATION OF CO_2 SNOW JET CLEANING IN CONJUNCTION WITH LABORATORY BASED TOTAL REFLECTION X-RAY FLUORESCENCE SPECTROMETRY FOR GENESIS SOLAR WIND SAMPLES. M. Schmeling[1], D.S. Burnett[2], J.H. Allton[3], M. Rodriguez[3], C.E. Tripa[4], and I.V. Veryovkin[4]
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Introduction: The Genesis mission was the first mission returning solar material to Earth since the Apollo program [1,2]. Unfortunately the return of the space craft on September 8, 2004 resulted in a crash landing, which shattered the samples into small fragments and exposed them to desert soil and other debris. Thus only small fragments of the original collectors are available, each having different degrees of surface contamination. Thorough surface cleaning is required to allow for subsequent analysis of solar wind material embedded within. An initial cleaning procedure was developed in coordination with Johnson Space Center which focused on removing larger sized particulates and a thin film organic contamination acquired during collection in space [3]. However, many of the samples have additional residues and more rigorous and/or innovative cleaning steps might be necessary. These cleaning steps must affect only the surface to avoid leaching and re-distribution of solar wind material from the bulk of the collectors. To aid in development and identification of the most appropriate cleaning procedures each sample has to be thoroughly inspected before and after each cleaning step. Laboratory based total reflection X-ray fluorescence (TXRF) spectrometry lends itself to this task as it is a non-destructive and surface sensitive analytical method permitting analysis of elements from aluminum onward present at and near the surface of a flat substrate [4]. The suitability of TXRF has been demonstrated for several Genesis solar wind samples before and after various cleaning methods including acid treatment, gas cluster ion beam, and CO_2 snow jet [5 – 7]. The latter one is non-invasive and did show some promise on one sample [5]. To investigate the feasibility of CO_2 snow jet cleaning further, several flown Genesis samples were selected to be characterized before and after CO_2 snow application with sample 61052 being discussed below.

Experimental: Genesis flight sample 61052 (silicon material) was chosen for CO_2 snow cleaning studies and initially inspected optically by using a Leica DM6000 microscope. Some selected areas were magnified 50 times to better visualize the contamination. After this first inspection the sample was characterized by TXRF (PicoFox, Bruker AXS, Madison, WI) before undergoing CO_2 snow jet cleaning (K4-10, Applied Surface Technologies, New Providence NJ) and the same area of the sample was re-analyzed by TXRF again.

Results and Conclusion: Figure 1 shows the micrographs of optical microscope inspection for sample 61052 before CO_2 snow treatment. The two inserts right and left of the sample picture are 50 times magnifications of the most distinctive features outlined in red. It is clear from this that some contamination should be present at the surface and detected by TXRF.

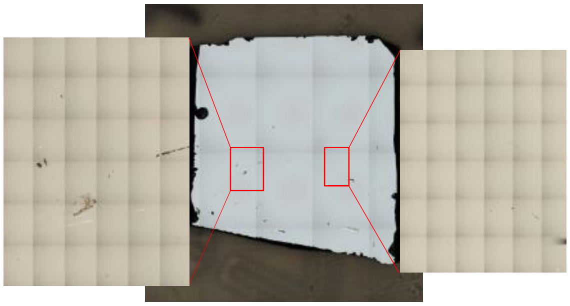

Figure 1: Micrographs of Genesis flight sample 61052.

Figure 2 shows the same micrograph as figure 1 highlighting the area probed by TXRF analysis. The analyzed section also includes the two spots magnified in figure 1.

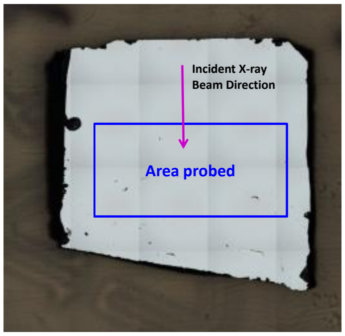

Figure 2: TXRF analyses area of Genesis flight sample 61052. The arrow indicates the direction of the incident X-ray beam.

Figure 3 displays the results obtained by TXRF analyses for the outlined region in figure 2. The black spectrum was acquired before application of CO_2 snow jet and the red spectrum after application of CO_2 snow jet. For better comparison figure 4 magnifies the spectra of figure 3 and shows only the energy range from 3 to 10keV corresponding to the major contaminants peaks.

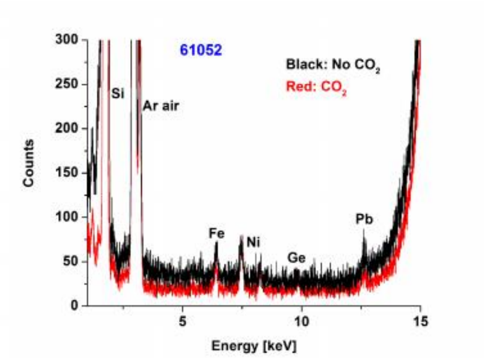

Figure 3: TXRF spectrum of Genesis flight sample 61052 before CO_2 snow (black) and after CO_2 snow (red) cleaning.

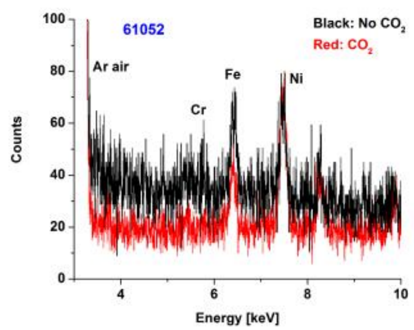

Figure 4: Rescaling of TXRF spectrum from figure 3 and magnifying the energy range 3 to 10keV. The spectra colors correspond to the ones in figure 3.

The results obtained indicate some reduction in contaminants, specifically Fe, after CO_2 snow application on the sample. The CO_2 treatment also appears to have a smoothing effect on the surface of the sample visible by the reduced background signal in the red spectrum. To gauge the type(s) of contamination present, an angle scan of the same area was performed. Angle variation of the incident X-ray beam not only identifies the optimal angle of incidence (alpha critical), where bulk substrate background signal is at its lowest, but it also aids in determining what type of contaminant is present (thin film versus particle) at or near the surface [4]. Figures 5a and 5b display the angle scan for sample 61052 before CO_2 cleaning, showing the count rate change for Si as bulk substrate (5a) and for Fe, Ni as main contaminants (5b). The critical angle at 0.1 degree corresponds to the lowest Si counts thus indicating the smallest penetration depth and highest reflection of the beam. Both Fe and Si follow a similar pattern indicating that Fe might be present as thin film type contaminant. The situation for Ni is less clear, but appears to be similar than Fe. It should be noted that this sample had on average very little metal contamination and the full effect of CO_2 snow cleaning might not be visible. To fully gauge the capability of a CO_2 snow cleaning step it is necessary to investigate more samples and perform angle scans before and after the cleaning application.

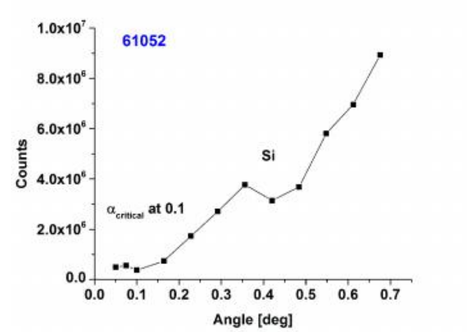

Figure 5a: Si TXRF angle scan of sample 61052.

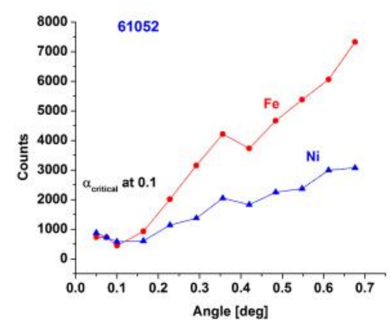

Figure 5b: Fe (red) and Ni (blue) TXRF angle scan of sample 61052.

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