Depth sensitive sampling of implanted species in Genesis Collectors using UV laser ablation and SIMS

Conference or Workshop Item

DEPTH SENSITIVE SAMPLING OF IMPLANTED SPECIES IN GENESIS COLLECTORS USING UV LASER ABLATION AND SIMS. I. A. Franchi[1], N. Suhaimi[2], R. J. Chater[2], D. S. McPhail[2], P. van Calsteren[3] and A. L. Butterworth[1,4] [1]Planetary and Space Sciences Research Institute, Open University, Milton Keynes MK7 6AA, UK (i.a.franchi@open.ac.uk), [2]Dept of Materials, Imperial College, London, SW7 2AZ, UK, Dept Earth Sciences, Open University, Milton Keynes MK7 6AA, UK, [4]Space Sciences Laboratory, University of California, Berkeley CA 94720, USA.

Introduction: The energy spectrum of the solar wind ions collected by the Genesis spacecraft is such that they are implanted into the ultra-pure collector materials to a depth where there should not be any significant diffusion loss. However, this depth is of the order of only 100nm [1] which presents two key problems when sampling the implanted solar material. Firstly, the collector materials generally contain some level of contamination, which given the low abundance of the solar material in the collectors, even after ≈2 years of collection, means it is necessary to ensure that only the implanted ions in the collector material are sampled. Second, and even more demanding, the analytical techniques have to remove or resolve surface contamination on the samples. Possible sources of contamination include absorbed atmospheric gases or volatiles degassed from other parts of the spacecraft, particulate debris from micrometeorite impacts or particles introduced during cutting, distribution and analysis.

Previously, we have demonstrated that UV laser ablation was well suited to sampling the implanted gases from diamond and diamond like material [2]. The use of a pulsed 193nm homogenized excimer laser offers exceptionally uniform power densities across the beam and therefore very flat bottomed pits. Here we examine the practicality of using such a laser system to remove a very shallow layer of surface material to eliminate the surface contamination without significantly affecting the implanted sample. We have used Secondary Ion Mass Spectrometry (SIMS) to determine the distribution with depth (depth profiles) of various species in the sample before and after laser ablation.

Sample and Technique: A polished sample of CVD diamond was irradiated with $1.5x10^{15}$ ^{18}O ions cm^{-2} at 36keV. Prior to irradiation the sample was coated with a thin film of Au to prevent charging of the target. A series of 215μm diameter shallow pits were then excavated in the sample by laser ablation using a New Wave 193 UP excimer laser system. 1, 3 and 5 pulses of about 30ns using two different energy outputs of approximately 2 and 4 Jcm^{-2} were used to generate a series of pits.

A series of SIMS profiles were then generated in 3 of the pits as well as in the surrounding areas unaffected by the laser ablation. The SIMS instrument was an Atomika DIDA 6500 retrofitted with a floating low energy ion gun (FLIG) to allow high depth resolution analysis. The samples were analysed with a 5 keV nitrogen beam scanned over an area 200 μm^2 across using electronic gating to collect secondary ions from the central 25% of the crater. The intensities of the negative secondary ions of ^{12}C, ^{16}O, ^{18}O and ^{35}Cl were determined from areas approximately 50μm across. ^{16}O and ^{35}Cl were monitored as measures of contamination levels.

Results and Discussion: Figure 1 shows the depth profile from a pristine area of the diamond. Two clear features are apparent – a clear ^{18}O implant profile and a large amount of surface contamination as indicated by the high concentrations of ^{16}O and ^{35}Cl. A TRIM calculation [3] simulating the implant is also shown, scaled to match the SIMS ^{18}O profile. A good match between the calculated and observed profiles indicates that the TRIM calculation can be used to scale the depth of the SIMS analysis – indicating that the peak of the implant occurs at around 45nm. Using the known implanted abundance of ^{18}O as a calibration the amount of ^{16}O surface contamination is estimated as $3.3x10^{15}$ atoms cm^{-2} - approximately 2 orders of magnitude greater than the anticipated solar flux in the concentrator target! This areal dose suggests that the surface is contaminated with a monolayer or two of oxide. The apparent distribution of the contaminants, which decay linearly (when the secondary ion intensity is plotted on a logarithmic scale) down to depths of ~ 40nm are probably due to SIMS beam induced mixing processes. The decay lengths of ~ 7.5nm/ decade are consistent with the range of nitrogen into diamond at this SIMS primary beam energy (~12 nm). Figure 2 shows the SIMS profile from the centre of a pit created with 5 pulses of 2 Jcm^{-2}. Most of the ^{18}O implant remains but there is an indication that the peak is now nearer to the surface. The level of surface contamination has been significantly reduced, with the amount of ^{16}O surface contamination now ≈$6x10^{14}$ atoms cm^{-2}. This is still double the expected solar flux in the concentrator target.

Figure 3 shows the ^{18}O SIMS profiles from Figures 1 and 2 linearly re-scaled such that the deeper half of the two profiles are best matched, compensating for any differences in the sputter rate between the two pits. It appears that some of the shallowest part of the ^{18}O implant has been lost during the laser ablation. Comparing the area of the implant curve of the profile from the laser pit with that of the pristine implant indicates that

approximately 10% of the implanted ^{18}O has been lost. Performing a similar exercise with a deeper pit generated with 5 pulses at twice the energy (4 Jcm^{-2}) reveals a 20% loss of the implant. In this case the amount of surface contamination was greater than that seen in the shallower pit but given the unclean handling experienced by the sample such irregularities may be expected. A shallower pit generated with 3 pulses at 2 J cm^{-2} revealed a loss of only 8% of the implant while maintaining a comparable level of surface contamination to that seen in Figure 2.

Conclusions: This initial study of the use of homogenized UV laser ablation as a means of performing surface clean up of the Genesis concentrator target material (CVD diamond) for oxygen isotope analysis has shown that sufficient depth resolution can be achieved to remove a shallow layer of surface material containing much of the surface contamination. The minimum levels of surface ^{16}O at $\approx 6 \times 10^{14}$ atom cm^{-2} observed in the ablation pits is mostly likely absorbed atmospheric oxygen (or moisture) as the samples were exposed to atmosphere for some considerable time between ablation and SIMS analysis. The higher levels of contamination seen in the pristine area may be related to the Au coat. A more realistic analysis protocol would of course eliminate atmospheric exposure between the surface clean-up ablation and the subsequent sampling of the solar wind-rich portion of the collector.

The conditions employed here appear to result in the loss of a small amount of implanted sample (at least 8%). Some of this may be due to some unevenness in the ablation process, possibly related to the granularity of the CVD diamond. However, scope exists to reduce the number of pulses and to reduce the power density of the laser beam to further limit the depth of ablation in this clean up stage. Thermal de-gassing of the samples prior to analysis would also be expected to help significantly reduce the surface contamination. The energy of the oxygen ions implanted in the Genesis concentrator target material will in fact be higher than the ions implanted in the test sample used here [4], with a peak implant depth of around perhaps 75nm as opposed to the 45nm shown in Figure 1. This offers better scope for resolving surface contamination from the solar wind sample, although it should be noted that there will be a considerable spread in energies from the solar wind which will see some of the tail at shallow levels.

References: [1] Burnett D. S. et al (2003) *Space Sci. Rev., 105,* 509-534. [2] Butterworth A. L. et al. (2003) *LPS XXXIV, Abstract* #1591. [3] Ziegler J. F. and Biersack J. P. (2003) SRIM.com. [4] Wiens et al. (2003) *Space Sci. Rev., 105,* 601-625.

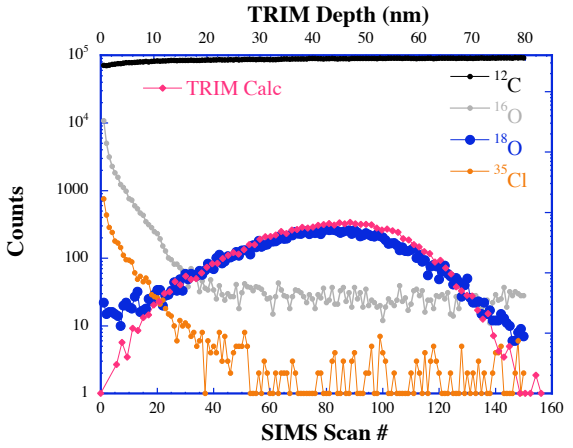

Figure 1 – SIMS profile from pristine area of CVD diamond. TRIM profile also shown scaled to match SIMS profiles (negative secondary ions).

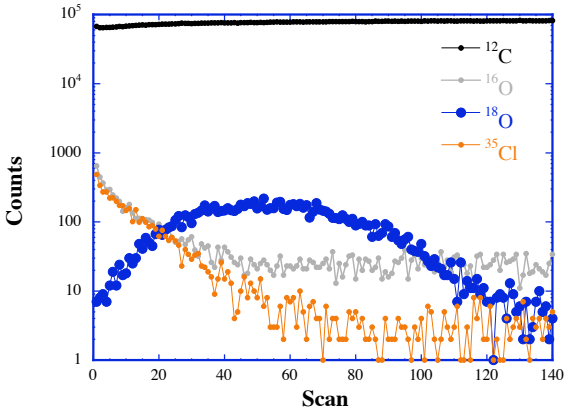

Figure 2 – SIMS profile from centre of laser pit created with 5 pulses of 2 Jcm^{-2}.

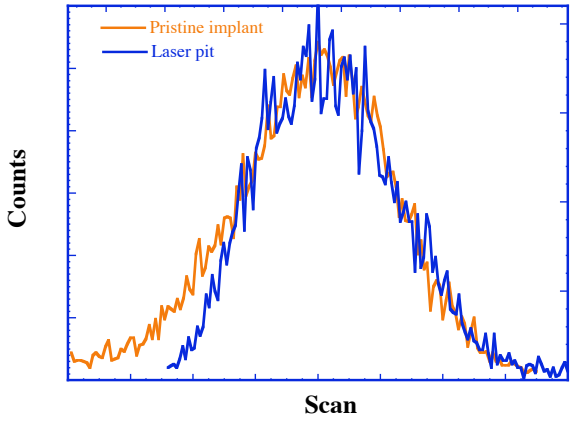

Figure 3 ^{18}O SIMS profiles from pristine area (Fig1) and laser pit (Fig2) re-scaled to match deeper half of profile to reveal differences close to surface.