

SOLAR WIND SAMPLE RETURN FROM GENESIS: A TECHNIQUE FOR THE EXTRACTION OF CARBON IMPLANTED INTO SILICON-ON-SAPPHIRE WAFERS. A. L. Butterworth, I. A. Franchi and C. T. Pillinger, Planetary Sciences Research Institute, The Open University, Walton Hall, Milton Keynes MK7 6AA, UK.

Introduction: The objective of the Discovery mission Genesis is to return samples of the solar wind to Earth for determination of the solar abundances and isotopic composition of a large number of elements. The aim is to provide a precise data set to which all other planetary compositional data can be compared as well as studying processes within the Sun [1]. The spacecraft is to be inserted into a Lissajous orbit at L1, the libration point, with an array of collectors facing the sun. Following a two year collection period, the spacecraft will be returned to Earth and samples of solar wind material will be provided for analysis in suitable laboratories.

The solar wind ions implanted into the collector material will have accumulated with a maximum concentration at a depth of several hundred Ångströms. Part of the pre-flight development is focused on the selection of material to be used in the collector array. Part of the rationale for collector material selection is based on the limitation of contamination to the elements of interest. The materials must be ultra-pure, so that an upper limit equivalent to 1% of the expected solar wind concentration has been set for the bulk purity. Of equal importance, the collector materials must render the implanted ions for analysis in the laboratory. The extraction efficiency of analytical techniques is therefore part of collector development.

This work is concerned with the choice of collector material for, and subsequent extraction of, implanted carbon. Silicon has been proposed as a suitable collector material, because it has been shown to be produced with exceptionally high purity with regard to carbon – as low as ppb levels. Carbon may be extracted from silicon by heating in oxygen to obtain the carbon as CO₂.

Apart from bulk purity, surface contamination for light elements such as carbon is unavoidable. Any proposed extraction technique for carbon must offer ‘depth sensitivity’ or a means of separating surface contamination from the extracted solar wind carbon. In routine analysis of extra terrestrial samples, such as meteorites, terrestrial contamination is removed by employing a stepped heating procedure [2]. All surface carbon species, primarily organic, but also carbonate, are removed at temperatures $\leq 600^\circ\text{C}$.

Fig. 1 shows a stepped combustion experiment of float zone silicon implanted with nanogram quantities of ¹²C. The large amount of surface contamination carbon on this sample was virtually all removed by 600°C, but implanted carbon was not released until

temperatures above 1000°C. Silicon itself will not combust until heated to 1100°C, thus providing a means of depth resolution, separating surface contamination from implanted carbon.

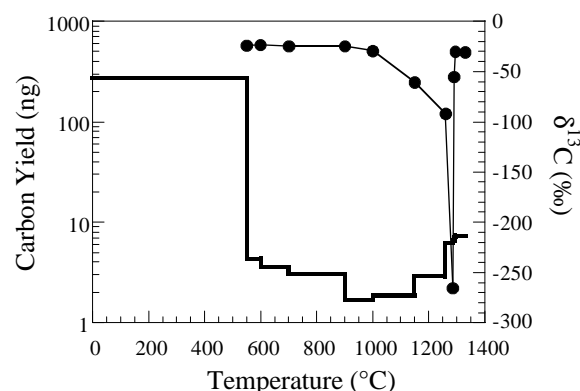


Fig. 1: Stepped combustion experiment of float zone silicon containing implanted ¹²C.

However, the combustion of thick ($\approx 700 \mu\text{m}$) wafers of silicon is rather crude as it is difficult to control the oxidation of Si to an even depth. Therefore, a refinement of the combustion technique is to use a thin layer of silicon grown onto a sapphire substrate. A sub-micron layer (typically $0.4 \mu\text{m}$) is deep enough to trap all the implanted carbon, but minimises the volume of silicon employed, hence reducing bulk carbon content. The whole Si layer may be ablated from the sapphire beneath to release the implanted carbon by heating with a UV laser. The laser beam couples only with the Si and passes straight through the sapphire. Pre-heating of the silicon on sapphire wafer is possible to effect a stepped combustion approach.

Method: A preliminary investigation of a laser extraction technique has been undertaken for recovering implanted carbon from silicon-on-sapphire (SOS). Samples of sapphire wafer ($\sim 1 \text{ mm}$ thick) with a $0.4 \mu\text{m}$ layer of silicon grown onto to the surface were provided for testing. One such sample was implanted with ¹³C with a nominal fluence of 3×10^{14} atoms/ mm^2 ($6.5 \text{ ng } ^{13}\text{C per mm}^2$).

A static mass spectrometer was used to determine the stable isotope composition of CO₂ analytic gas. The technique is capable of determining $\delta^{13}\text{C}$ of $\sim 3 \text{ ng C as CO}_2$ with a precision of $\pm 0.2\text{‰}$ [3]. The mass spectrometer employed in this initial investigation is less precise, but it was straightforward to differentiate between implanted ¹³C and contamination by (mostly) ¹²C.

An oil free vacuum extraction line made from Pyrex was built for sample preparation, collection and transfer of CO₂ to the mass spectrometer source. The laser extraction apparatus comprised of a stainless steel chamber of approximately 50 cm³ internal volume attached to the vacuum line. Two quartz-UV grade windows with a view diameter of 36 mm allowed optical passage for a pulsed beam of 266 nm produced by a quadrupled Nd-YAG laser, mounted above the chamber.

The sample SOS wafers (irregular sizes, usually with surface area of ~2 cm²) were roasted in air at 450°C for 2 hours before being loaded into the stainless steel chamber in a class 100 clean-room. The chamber was then attached to the vacuum line and evacuated to a pressure of 10⁻⁶ mbar. The whole chamber was heated to 150°C while pumping in an effort to eliminate some carbon contamination. The bake-out temperature was limited by the commercially acquired viewports.

The extraction of carbon was accomplished by laser heating of SOS samples in the presence of an atmosphere of oxygen to produce CO₂. A pressure of 5 mbar gave an excess of oxygen – in the region of 2,000 parts O to 1 part C. Areas of sample were ablated by moving the sample chamber relative to the fixed laser by means of an x-y stage and viewing the area on a video screen. A joystick was used to raster the stage back and forth in order to ablate areas of 1mm² of Si from the sapphire substrate.

The CO₂ produced was separated from the oxygen present by trapping on a cold finger immersed in liquid nitrogen (-196°C) for 20 minutes. The oxygen was then trapped onto molecular sieve (13X) and the remainder pumped away. A second clean-up stage of cryo-separation was used to remove any water and residual oxygen. The CO₂ analyte could then be admitted to the mass spectrometer source for isotopic analysis.

Results: Preliminary results showed that the silicon layer was indeed removed from the sapphire substrate. The lased tracks were clearly visible. In addition it was noted that some of the silicon condensed into metallic droplets concentrated at the edges of the tracks. Elemental analysis (EDS analysis using SEM with Kevex Quantum detector) confirmed that the droplets were of pure silicon, and not contaminated by Al or O from the sapphire beneath. It could safely be assumed that the 266 nm laser beam coupled with silicon but the sapphire was unaffected.

The carbon contamination levels and extraction efficiency were compared for an un-implanted and a ¹³C implanted SOS sample, and the results are shown in Fig. 2.

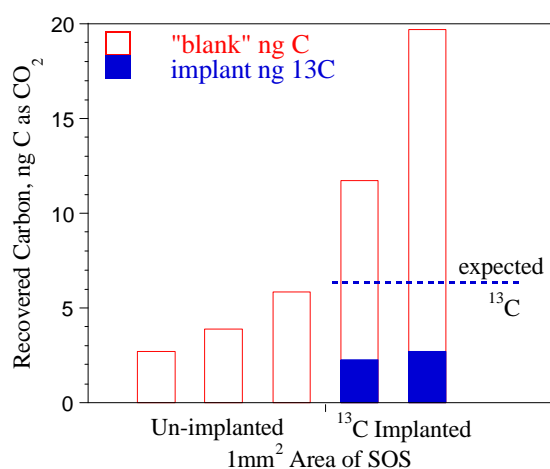


Fig. 2: CO₂ extracted from roasted (at 450°C) silicon-on-sapphire (SOS) wafers. CO₂ extracted from a sample implanted with ¹³C is compared with an untreated SOS sample.

The laboratory implantation is essentially a dirty process and this is reflected in the contamination levels of up to 20ng ¹²C per mm², which is equivalent to 10¹⁵ atoms/mm². The “clean” un-implanted SOS yielded <6 ng/mm². “Blank” experiments carried out using similar amounts of oxygen, but without lasing of samples, resulted in <1 ng C as CO₂ being recovered. It is important to note here that no stepped heating experiments were possible with the apparatus available at this preliminary stage, although stepped combustion is known to be essential for removal of prevalent surface contamination. It has not been ruled out that the sample chamber contributes some carbon during use of the laser, since stainless steel may not be the preferred material for very low level carbon detection.

Despite the high levels of contamination in these experiments, the use of ¹³C implants permits the discrimination of implant and contamination carbon with a precision of a few percent in order to estimate the efficiency of the extraction. The highest yield of ¹³C obtained was 42 %, which may have been due to incomplete recovery of CO₂. Previous experience with the implantor used has indicated a level of uncertainty in the flux comparable to that seen in these extractions. Further work is continuing to confirm a low yield, or increase the extraction efficiency if required. However, these initial results are encouraging for the use of SOS as the carbon collector on GENESIS.

References: [1] Burnett D. S. et al. (1997) GENESIS Feasibility Study. [2] Swart P. K. et al. (1983) *Meteoritics*, 18, 137-154. [3] Prosser S. J. et al. (1990) *Chem. Geol.*, 83, 71-88