GENESIS SAMPLE MATERIAL SUBDIVIDING PLANS.

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Introduction: Subdivision of Genesis collectors is necessary to maximize the science return of the mission and retain a representative subset of the collection to archive for the future. The ability to verify important scientific results and resolve experiment anomalies through experiment replication and independent experimental techniques requires a method for subdividing individual sample collectors so that equivalent samples are made available. In addition, subdividing the Genesis collectors is required to accommodate the size limitations of many analytical systems. Samples allocated for analysis are likely to be small sections of individual collectors.

Subdivision of returned Genesis solar wind samples must be performed in a manner having minimum impact on the accuracy and interpretation of analytical information obtained from these samples. It is also necessary that we ensure that consequences of any processing are well understood and documented.

Sample Subdivision: Subdivision of the Genesis collectors presents a uniquely complex technical challenge. Although many of the materials are similar to single crystal materials used in the electronics industry, their physical dimensions and cleanliness requirements prohibit the direct use of techniques traditionally used to divide these materials. For example, silicon semiconductor devices are produced on large single crystal wafers that are diced into chips using a mechanized diamond scribing system. This system operates with a wafer that is lapped to a thickness of approximately 100µm and then cuts are aligned with the known orientation of the single crystal. There are no strict cleanliness requirements for this process since the devices have already been passivated and protected, allowing cutting to be performed with a relatively dirty apparatus outside of the cleanroom. Genesis collectors, on the other hand, are much thicker (550-700µm) and cannot be lapped, passivated, or handled outside of a cleanroom. In addition, the collectors were machined into symmetric hexagons for mounting onto arrays in the Science Canister and the single crystal orientation information was not preserved.

The majority of collectors are hard, brittle materials, which do not lend themselves to simple subdividing techniques. In addition, the majority of the 15 types of collectors on the Genesis spacecraft are not single crystal materials, but combinations of amorphous, polycrystalline, and layered structures. Nearly half of the collectors are actually thin coatings on silicon and sapphire substrates. Most of these are merely physically deposited coatings with limited adhesion and hardness, complicating the handling of these collectors. Finally, the concentrator targets, which are to be evaluated to address the highest priority goals of the mission, represent all of these difficulties and require cutting to greater precision as a result of the extremely limited quantities available.

Significance: The most significant issues for processing and handling collector materials is that of contamination and solid-state diffusion of the implanted species. Uncontrolled diffusion will broaden the implant zone, effectively reducing the ion concentration available for detection[1], while contamination could lead to miscalculations of elemental and isotopic abundances. Yet, some method of subdivision is required for sample size compatibility with the majority of analytical instruments proposed for the examination of Genesis samples. Preserving sample integrity means insuring that techniques used for collector subdivision do not introduce contamination or alter the implant distribution within the samples. Since the majority of subdivision techniques involve thermal agitation, either intentionally or as a by-product, solid-state diffusion is the major concern.

Approach: Numerous options have been considered for sample collector subdivision including water jet cutting with ultra-pure water, various laser cutting systems, and mechanical techniques such as diamond sawing and "scribe and cleave" techniques. Trade studies of these techniques considered contamination, thermal agitation, material loss, and cut reliability and precision. The thickness of the collectors significantly hinders the application of mechanical scribing techniques, even for single crystal silicon. Mechanical scribe & cleave techniques result in unpredictable breakage and often shattering at thickness above 400 µm. More complex collectors, such sapphire or gold on sapphire that lack well-defined cleavage planes, have greater reliability and reproducibility problems [2]. Although it is possible to perform water-jet cutting using ultra-pure water as the working fluid, the excessive force used in this process may disrupt physically deposited layers, and there is added risk of surface contamination due to contact with the working fluid. Material losses and precision of the cut are unacceptable in water-jet cutting as well[3]. Finally, mechanical cutting with devices such as diamond saw blades require the use of cutting fluids, introduce contamination by contact, and cause local heating of the collectors, leading to implant diffusion[4].

The remaining alternatives are laser related cutting techniques using either thermal or ablative processes. Thermal systems, such as the CO_2 lasers, operate at longer wavelengths and rely on vibrational and rotational excitations to disrupt bonds, producing local melting. Ablative lasers, such as some Nd:YAG and UV lasers, operate at shorter wavelengths where thermal agitation is reduced and material is removed by direct ablation. However, even though thermal agitation concerns are reduced with the use of ablative lasers, there is still some cause for concern in terms of ion diffusion and fractionation effects. For coated collectors, there is the added complexity of potential coating and implant removal as well as ablative mixing, leading to the blurring of implant profiles.

Backside Laser Scribing. A new backside laser scribing technique is being developed which will minimize the thermal agitation of the implanted solar wind in Genesis collectors while maintaining cleanliness levels required for processing and archiving. The laser scribing is controlled to penetrate through only a partial thickness, from the backside, of the collectors. Depth control of the laser scribe will reduce thermal agitation, improve cleanliness, and prevent surface damage or ablation of fragile coatings, which are present on many of the collector surfaces and contain the implanted material of interest.

Thermal Imaging. Critical to developing this technique is the measurement of thermal profiles on the implanted collector surfaces during scribing in order to develop a suitable protocol, including appropriate laser power, pulse rate, and duty cycle, for cutting. Thermal profiles of the surface are sufficient since the implants lie at an average depth of less than 100nm from the surface. These thermal profiles will be used with literature diffusivities to calculate the maximum expected dispersion of the implants. Although thermal absorption is expected to be low for all collector materials at the wavelengths to be used, it is important to measure both maximum temperature excursion and temperature distribution on the implant side of the collectors. This allows for mapping temperature variations and calculating diffusion profiles over the prepared samples. This information is critical for the science community to understand edge effects and spatial limitations for the application of analytical techniques.

Diffusion Modeling & Verification. After determining the temperature distribution across the implant side of the collectors, diffusion rates for implanted species can be calculated with the assumptions that (1) literature values for diffusion coefficients are available, (2) binary diffusivity is valid and independent of concentration, and (3) one-dimensional Fickian diffusion provides a sufficient model over the implanted layer.

The first assumption is not entirely valid at this time, however, experimental work already underway promises to provide diffusion constants and activation energies required for this model within the next year [5]. Binary diffusivity is valid based on the extremely low two-year cumulative solar wind fluence implanted in the samples; it is so dilute that multi-component interactions are minimal. Finally, the implant thickness is extremely small relative to the lateral dimensions of the sample, justifying one-dimensional Fickian diffusion. Therefore, Fick's second law becomes:

$$\frac{\delta c_x}{\delta t} = D_{AB} \frac{\delta^2 c_x}{\delta x^2}$$

c_x: concentration of solar wind species A at depth x

x: distance from the implant interface

t: duration of temperature excursion during subdivision

D_{AB}: diffusivity of solar wind species, A, in collector material, B calculated as a function of temperature

The validity of this model will be verified by measuring implant profiles for select cases of experimental samples before and after subdivision. Implant profiles in these samples will be measured using ion probe analysis both before and after subdividing.

Clean Sample Division: The final requirement for Genesis collector subdivision is that it be performed in a clean environment. To this end, a cleanable stainless steel enclosure has been designed to isolate individual collectors during cutting operations. This enclosure is fitted with a 355nm transparent window to allow laser transmission to the backside of the collector and an IR transparent window to allow simultaneous thermal imaging of the front side of the collector. The use of this portable enclosure allows laser scribing operations to take place outside of the cleanroom environment without exposing collectors to a degraded environment

References:

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