A BRIEF HISTORY OF THE CURATION OF GENESIS SAPPHIRE SOLAR WIND COLLECTORS. J. H. Allton[1], C. P. Gonzalez[2], K. K. Allums[2], A. J. G. Jurewicz[3], M. Schmeling[4], and R. A. Synowicki[5]. [1]ARES, Code XI2, NASA-JSC, 2101 NASA Parkway, Houston, TX 77058, USA (Judith.h.allton@nasa.gov), [2]JETS, NASA Johnson Space Center, Houston TX 77058, USA. [3] Arizona State University, Tempe, AZ, 85287, USA, [4]Loyola University Chicago, Chicago, IL 60660, [5] J.A. Woollam Co., Inc., 645 M Street, Suite 102, Lincoln, NE 68508 USA

Introduction: Commercial, high purity semiconductor sapphire (AKA corundum) wafers were used as one of the passive solar wind collector materials on the Genesis sample return mission because it exhibited lower diffusion rates for alkali elements than silicon and had sufficiently low alkali blanks. The 20 whole hexagons of sapphire flown on the Genesis spacecraft were distributed among the solar wind (SW) regimes as follows: 8 bulk solar wind (plus two half hexagons), 4 coronal mass ejection (CME), 4 coronal hole (CH), and 4 interstream (IS). Excellent overviews: a) collector material properties presented in [1] and b) solar wind environment experienced by collectors presented in [2].

Hexagon Description: The Genesis flight collectors were purchased from Kyocera Industrial Ceramics Corporation in 1999. Surface cleanliness assessments were performed by the Genesis science team. Description of Genesis sapphire hexagon is shown in Table. 1:

Table 1 Genesis hexagon description.

Dimension: 86 mm bare sapphire hexagonal	
Material: SA100	
Thicknesses: 550, 600, 650, 700 µm	
R-plane	
One side polished, 3 notches R 6 mm	
Maximum impurities acceptable (atoms/cc):	
Na: 1E17	C: 6E17
K: 7E15	Si: 2E18
Li: 1E14	

Genesis mission sapphire collectors were single crystal sapphire grown by the edge-defined film-fed growth method (i.e., they were grown as hexagonal boules and then sliced and polished). See Kyocera brochure for mechanical, thermal, and electrical characteristics of Kyocera single crystal sapphire at: https://global.kyocera.com/prdct/fc/product/pdf/s_c_sapphire.pdf. Reference density 3.97g/cm^3.

Solar Wind Exposure. Passive collectors were exposed to the solar wind at Earth-Sun L1 during the declining phase of solar cycle 23 from Dec 2001 to April 2004. Duration of exposure and estimated H fluence, is shown in Table 2. Fluence indicates amounts of solar atoms accumulated for each type of solar wind. Each type of SW was exposed to differing velocities/energies. Temperature of sapphire array collectors under solar exposure conditions, derived from JPL thermal-vacuum testing and modeling, is estimated at 56˚ C [1].

Table 2. Solar wind exposure (modified from [2]).

Collector	Duration, days	H fluence/cm^2
Bulk	854	2.06e16
IS (slow)	334	9.15e15
CH (fast)	313	6.40e15
CME	193	4.73e15

Sapphire Collector Condition after Recovery. Sapphire is stronger mechanically and harder to cleave than silicon. After the hard landing upon re-entry at Utah Test and Training Range September 8, 2004, it was apparent that sapphire and sapphire-based collector fragments survived in larger pieces than silicon collectors [3, 4, 5]. Although sapphire is a harder material than silicon, the sapphire fragments also sustained gouges, scratches and cracks with embedded fine debris particles (Ge and silicon dust from powdered collectors are examples) and splashes of molten material (Fig. 1). Some subsurface cracks, caused by impacting projectiles, could have opened and closed by flexure during the impact process trapping debris. Fortunately, transparency of the sapphire makes this debris optically visible. Post-recovery planning in 2005, for all passive collector materials, included physical and chemical cleaning methods such as solvent, replicate, CO2 snow, laser ablation and H plasma, but long-term all were not equally successful [6].

Initial Sapphire Collector Cleaning and Cleanliness Assessment. Fortunately, sapphire is not only mechanically sturdy but is chemically resistant; therefore, allowing a number of sample surface cleaning techniques to be tested on a few samples designated "cleaning matrix" samples. Results were assessed using optical imaging, SIMS and total reflection x-ray fluorescence, both synchrotron and lab-based. The Stanford Linear Accelerator and Advance Photon Source were early synchrotron resources using variation of incidence angle to perform "depth profiling" and enabling discrimination of surface contamination vs implanted solar wind [7,8,9]. Schmeling developed an efficient cleanliness assessment with a laboratory TXRF which is still in use, a method that is non-destructive and non-contaminating [10].

Curation provided sample cleaning, upon request, using megasonically energized ultrapure water (UPW) which is especially effective at removing the small debris particles which are not chemically bound [11]. UV ozone was used to remove molecular films [12]. For particulate contaminants adhering through stronger bonds to sample surfaces, various science team members tested cleaning with harsher reagents: HF, HCl, HNO_3, NH_4OH, H_2O_2, NaOH, KOH. Most of these solvents remove some contaminants, but eliminating solvent residue adds to the complexity. For analyses which cannot be performed by a profiling technique to discriminate surface contamination from subsurface solar wind, the surface must be cleaned and remaining contamination must be known. This issue is being addressed in a comprehensive, well-documented cleaning study on sapphire initiated by Welten *et al.* [13].

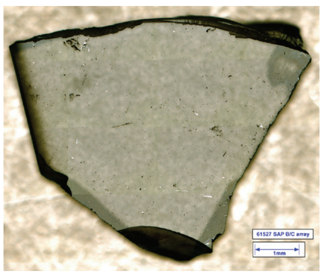

Fig. 1. Optical image of sapphire 61527, a bulk solar wind sample from which a FIB/TEM cross-section was later extracted. Keller *et al.* [21] report structural alteration in this sample, abstract #1196, this volume.

In addition to producing high resolution optical images (Fig. 1), early curation provided cleanliness assessment by ellipsometry, developed preflight for surface film assessment [14,15,16]. Applications of ellipsometry to investigate the physical state of the collectors are now being revisited with intent to develop a rapid screening method that can be correlated with known structural alteration determined by destructive methods [17]. Plots of wavelength vs refractive index for solar-wind-exposed and unexposed sapphire show conflicting results (Fig. 2). Other data show increased roughness for solar exposed specimens. This is tantalizing preliminary data requiring further investigation for correlation to less model dependent characterization.

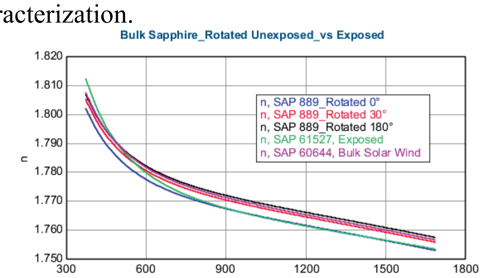

Fig. 2. Ellipsometry refractive index vs wavelength plot for non-flown sapphire reference material and exposed to bulk solar wind, 6157, 60644. 61527 appears to have changed, but 60644 has not.

Unlike the softer collector materials, the high hardness of sapphire makes it a better candidate for mechanical cleaning. Cleaning studies using polishing have been initiated using TXRF for cleanliness assessment [18,19]. Interestingly, a persistent contaminant easily identified by TXRF is fine powdered germanium. In this way Ge has use as a marker for cleanliness (O_2 etching should remove residual Ge).

Emerging sapphire characterization trends. Relatively recent forays into sophisticated sample characterization include TEM cross-sections of silicon showing physical alteration of the collector material [17,20]. "The solar wind affects the matrix of the silicon collectors, moreover the effect is different among the solar wind regimes." [20]. These TEM characterization studies began using silicon collectors, and now a sapphire TEM cross-section has been generated, also exhibiting alteration [21].

Experience by Genesis analysts has shown that Genesis-flown material behaves differently from non-flight reference materials, presumably due to solar wind exposure [22,23]. The TEM cross-sections help us visualize damage by solar wind and consider how substrate structural alteration affects both cleaning protocols and solar wind analytical results.

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