ANALYSIS OF SOLAR WIND DAMAGE IN GENESIS SAPPHIRE SAMPLE 61527.

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Introduction: The Genesis mission collected samples of the solar wind (SW) by passive implantation into a variety of semiconductor (and other) materials and returned them to Earth in 2004 [1]. Subsets of these collector materials were exposed to either bulk solar wind, high-speed (coronal hole) solar wind, low speed (interstream) solar wind, or coronal mass ejections (CMEs) [2,3]. Although of short duration relative to naturally space-weathered materials, these exposures to SW caused structural damage (and related chemical changes) in silicon (e.g., [4]). Here we explore the structural consequences of bulk SW collection into Genesis single crystal sapphire.

Materials and Methods. Genesis sample 61527, an irregular fragment of sapphire ~6.85 x 5.59 mm, sampled the bulk solar wind regime for over 2 years (~853 days). Genesis flew Kyocera wafers of single crystal R-plane ($1\bar{1}02$) sapphire. Chip 61527 was cleaned of adhering crash debris with ultrapure water in the JSC Curatorial facility.

We used a JEOL 7900F scanning electron microscope (SEM) equipped with an Oxford Symmetry electron backscatter diffraction (EBSD) detector to orient the uncoated chip for focused ion beam (FIB) and transmission electron microscope (TEM) study. Prior to the FIB/TEM analyses, we coated the sample with ~5 nm of evaporated platinum to mark the uppermost surface the chip, then applied an evaporated carbon coating. In the FIB, we used e-beam assisted carbon deposition to protect the surface from ion beam damage. The EBSD crystallographic data was used to select the orientation of the FIB section. A JEOL 2500SE field-emission TEM was used to obtain high-resolution images, electron diffraction data, and energy-dispersive X-ray (EDX) analyses from the FIB section. With this process, we extracted a FIB section containing the major ($1\bar{1}02$) and (1120) planes that required minimal tilting in the TEM.

Results and Discussion. The 61527 sapphire shows evidence for damage that likely resulted from solar wind irradiation. The upper 10 nm of the sapphire surface is highly damaged and is partly amorphized, but still retains short range atomic order based on the presence of faint coherent diffraction spots in Fourier transforms of the high-resolution TEM images (Fig. 1). The damaged layer is not fully amorphous; rather, it is nanocrystalline with numerous defects and dislocations. The highly-damaged layer exhibits a sharp interface with the underlying bulk sapphire, which is fully crystalline but contains a high density of dislocations and other strain-inducing defects in a layer extending an additional 30-50 nm into the bulk crystal (Fig. 2). Other defects identified in the sample include a 10-15 nm-wide zone of ~5 nm-diameter vesicles that straddles the interface between the highly-damaged and crystalline-but-defective regions of the sample. We obtained quantitative EDX maps from the FIB section and did not observe any changes in the Al/O ratio in a transect from the undamaged crystal through the damaged region up to the surface.

Using the ion collision Monte Carlo code SRIM [5], we calculated curves for the collisional atomic displacement damage in units of displacements-per-atom (DPA) and the implanted (assumed 1 keV) H^+ ion concentration as a function of depth in the sample (Fig. 3). The curves correspond to the DPA damage and implantation concentrations expected for Al_2O_3 sapphire receiving the measured Genesis bulk solar wind fluences for H and He (energies assumed 1 keV and 4 keV, respectively) in the DPA damage case, and H only for the implantation concentration [2]. The boundary between the 10 nm-wide highly-damaged layer and the less-damaged defective bulk crystal falls close to the maxima in both the DPA damage and implanted H concentration curves. The entire width of the DPA damage curve is in good agreement with the ~50 nm width of the entire ion-processed region of the sample. The results strongly support the interpretation that the microstructure of the sample observed by TEM is, in fact, due to solar wind irradiation and not some other process.

Preflight thermal-vacuum tests showed that Genesis sapphire reached ~56°C during flight. Ion irradiation studies of the Al_2O_3-corundum structure for various ions over a range of conditions find the structure resists amorphization up to atomic displacement damage levels of 100 DPA or more at room temperature [6]. Amorphization does occur, however, if irradiation is performed at cryogenic temperatures around ~77°K [6]. Although room temperature samples resist amorphization, they do characteristically develop high densities of dislocations and other extended defects, very similar to those observed in the current sample [6]. Both the tendency to form extended defects, and the observed temperature effect, have supported a "dynamic defect annealing" model in which the room temperature Al_2O_3

(corundum) structure kinetically removes point defects that destabilize the crystalline structure [6]. Recent H irradiation experiments using 40 kV H ions to a total fluence of ~1 x 10^{18} ions cm^{-2}, did not amorphize single crystal corundum, but only resulted in the formation of dense dislocations, nanoscale vesicles and cracks [7].

Although we do not observe complete amorphization in any of the ion-damaged regions of the current sample, the close-to-amorphization level of damage that the upper 10 nm of the Genesis sample shows at only ~0.20 DPA (Fig. 3) does not seem consistent with the previous laboratory irradiation findings. Given that the chemical effects of implanted species have been suspected to play a role in promoting Al_2O_3 amorphization [6], we will be evaluating a similar chemical role for implanted solar wind species such as H in modifying the ion radiation damage response of the Genesis sapphire sample and possible artifacts.

Acknowledgements: This study was supported by NASA ISFM funding to the JSC Coordinated Analysis Work Package. Electron microsopy was performed in the Electron Beam Analysis Labs at JSC. We thank the JSC Curatorial facility for providing the sapphire sample for this study.

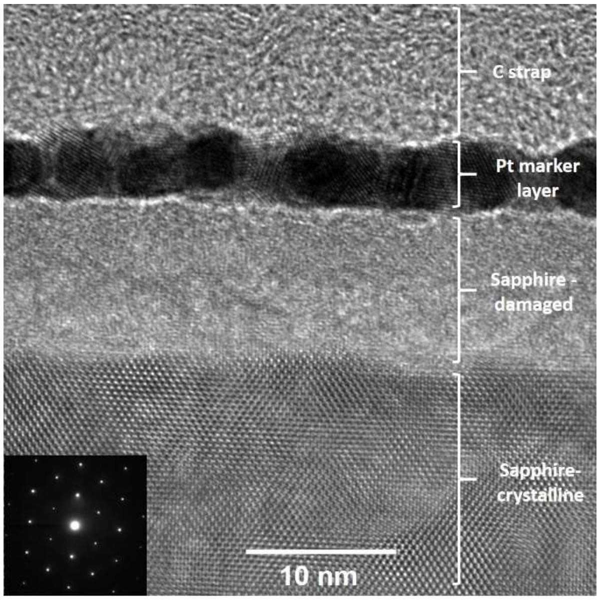

Figure 1. A high-resolution TEM image from a FIB section of 61527 showing a partly-amorphized, nanocrystalline damaged layer ~10 nm wide. The inset is a selected area electron diffraction pattern from the $[1\bar{1}\bar{1}]$.zone of sapphire.

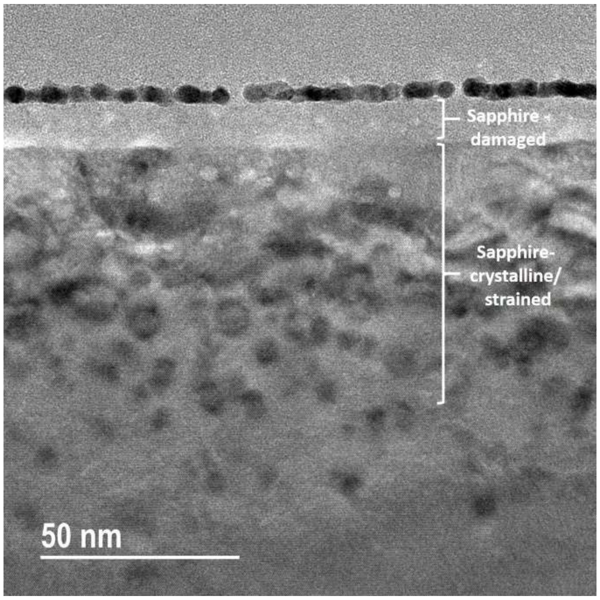

Figure 2. TEM image showing the strained region (spotty dark contrast) in the crystalline sapphire below the interface with the damaged layer.

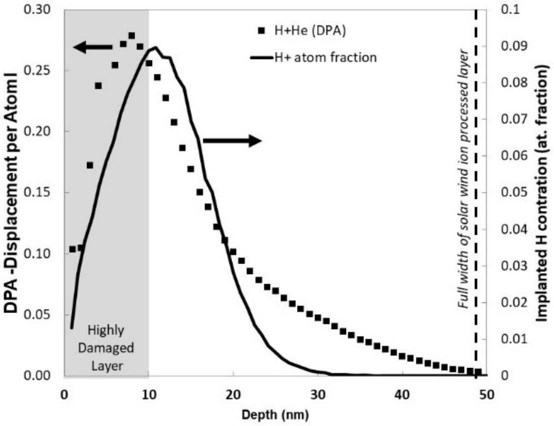

Figure 3. SRIM [5] calculations of DPA atomic displacement damage from combined solar wind H+He ions (dotted curve) and implanted H ion concentration (solid curve) for Al_2O_3 sapphire exposed to the Genesis bulk solar wind fluences reported in [2].

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