DECONTAMINATION OF GENESIS ARRAY MATERIALS BY UV OZONE CLEANING. M.J. Calaway[1], D.S. Burnett[2], M.C. Rodriguez[3], S. Sestak[4], J.H. Allton[5], and E.K. Stansbery[5]; (1) Jacobs (ESCG) at NASA Johnson Space Center, Houston, TX; (2) California Institute of Technology, Pasadena, CA; (3) GeoControl (ESCG) at NASA Johnson Space Center, Houston, TX; (4) Open University, Milton Keynes, UK; (5) NASA at Johnson Space Center, Houston, TX; michael.calaway-1@nasa.gov.

Introduction: Shortly after the NASA Genesis Mission sample return capsule returned to earth on September 8, 2004, the science team discovered that all nine ultra-pure semiconductor materials were contaminated with a thin molecular organic film approximately 0 to 100 Å thick [1, 2]. The organic contaminate layer, possibly a silicone, situated on the surface of the materials is speculated to have formed by condensation of organic matter from spacecraft off-gassing at the Lagrange 1 halo orbit during times of solar exposure [2]. While the valuable solar wind atoms are safely secured directly below this organic contamination and/or native oxide layer in approximately the first 1000 Å of the ultra-pure material substrate, some analytical techniques that precisely measure solar wind elemental abundances require the removal of this organic contaminate.

In 2005, Genesis science team laboratories began to develop various methods for removing the organic thin film without removing the precious material substrate that contained the solar wind atoms. Stephen Sestak and colleagues at Open University first experimented with ultraviolet radiation – ozone (UV/O_3) cleaning of several non-flight and flown Genesis silicon wafer fragments under a pure flowing oxygen environment [3]. The UV/O_3 technique was able to successfully remove organic contamination without etching into the bulk material substrate [3]. At NASA Johnson Space Center Genesis Curation Laboratory, we have installed an UV/O_3 cleaning devise in an ambient air environment to further experimentally test the removal of the organic contamination on Genesis wafer materials. Preliminary results from XPS analysis show that the UV/O_3 cleaning instrument is a good non-destructive method for removing carbon contamination from flown Genesis array samples. However, spectroscopic ellipsometry results show little change in the thickness of the surface film.

Method: The UV/O_3 cleaning method uses a photo-sensitized oxidation process to excite and/or dissociate organic contaminate molecules by UV radiation absorption. Molecular oxygen at 184.9 nm wavelengths and ozone at 253.7 nm wavelengths are dissociated simultaneously producing atomic oxygen. UV radiation at 253.7 nm is then absorbed by ozone and hydrocarbons. Excited contaminate molecules such as silicone or other types of organic contamination will react with the generated atomic oxygen forming volatile molecules that desorbs from the surface of the material or native oxide. Therefore, any organic contamination thin-film would theoretically be consumed and removed via the UV/O_3 cleaning process.

Jelight Model 342 UV – ozone cleaner with a suprasil synthetic fused quartz (ozone producing) lamp was installed on a class 100 laminar flow clean bench in the Genesis Curation Laboratory. The lamp operates 90% at 184.9 nm and 10% at 253.7 nm and was chosen for faster cleaning time over the normal fused quartz low pressure mercury lamp that produces 65% 184.9 nm and 35% at 253.7 nm. The lamp also produces 28,000 microwatts/cm^2 at 254 nm wavelength and can reach maximum operating temperatures of ~ 95 º C. The UV/O_3 cleaning method also requires that the samples be ~ 5 mm away from the lamp for effective interaction with the generated atomic oxygen.

Experiment: The most recent experiments were derived to evaluate the amount of carbon removed from flown Genesis array materials as well as the minimal time needed for exposure to the UV/O_3 cleaning process. Spectroscopic ellipsomtery was performed on 20 samples of silicon and sapphire flown materials to assess the thickness of the contamination film before and after UV/O_3 exposure. XPS analysis was used to evaluate the change in the amount of carbon and oxygen present on the surface of the samples. Subsequent samples were then slowly exposed to UV/O_3 cleaning process at various times and ellipsometry was used to evaluate the thickness at every 10 minutes of exposure for 4.5 hours. The results provide detailed observations of carbon loss and change in thin film thickness under various UV/O_3 exposure times.

Results: Within the first 10 minutes of exposure, the surface film showed a 2 to 5 Å reduction in thickness and then stabilizes for the duration of the 4.5 hour experiment. However, the sapphire samples grew 2 to 5 Å. The UV/O_3 cleaning process does not reduce the thickness of the thin film to preflight condition. For example, non-flight Genesis silicon wafers have a native oxide SiO_2 thin film of ~ 18 – 22 Å.

XPS results show that a 10 to 30 minute exposure dramatically reduces the carbon on the surface of the samples by 70 to 85% (highlighted results are shown below). The enriched atomic oxygen environment of the UV/O_3 cleaning process also saturates the surface material with oxygen. A comparison of atmospheric oxygen absorption in the non-flight sample and UV/O_3 cleaned sample of Si 60260 or sapphire 60275 show that the surface of the material have been enriched in oxygen and may have reached full saturation. This may possibly explain the lack of change in the thin-film thickness.

Sample	Material	Thickness (Å)	Carbon (cps)	Oxygen (cps)	SiO_2/Si
Before UV-ozone Exposure					
60266	Si - Control	38	62890	150404	1.27
60260	Si	41	63101	165821	1.33
Non-flight	Si	22	21675	304718	0.40
After 30 minutes UV-ozone Exposure					
60266	Si- Control	50	70749	150994	1.25
60260	Si	38	10667	338459	2.18
Non-flight	Si	18	6101	357864	0.53

Highlighted Silicon Results

Sample	Material	Thickness (Å)	Carbon (cps)	Oxygen (cps)
Before UV-ozone Exposure				
60271	SAP - Control	25	37817	218711
60275	SAP	30	62797	155943
After 30 minutes UV-ozone Exposure				
60271	SAP - Control	41	61246	205612
60275	SAP	32	14635	329015

Highlighted Sapphire Results

Summary: All experiments to date have shown UV/O_3 cleaning method to be the best non-destructive method for removing organic contamination from the surface of the Genesis materials. The UV/O_3 cleaning process can also clean carbon contamination to levels below non-flight standards. This can be seen by comparing sample 60260's carbon 10667 cps with non-flight Si carbon 21675 cps. Therefore, surface carbon contamination should not hinder the analysis of solar wind.

References: [1] Burnett, D.B. et al. (2005) LPSC XXXVI, Abstract # 2405; [2] Calaway, M.J. et al. (2006) LPSC XXXVII, Abstract # 1420; [3] Sestak, S. et al. (2006) LPSC XXXVII, Abstract # 1878.