



Status and Science Results D. S Burnett June 12, 2008



# Contents

	Slides
Background/Review Material	3-10
O isotopes	12-27
N isotopes	28-35
Noble Gases	36-45
C Isotopes	46
Elemental Abundances FIP fractionations SIMS Mg/Fe RIMS ICPMS SIMS C, N TRXRF	47-73 47-50 51-60 61-62 63 64-69 70-73
SW-Sun Isotope Fractionation	74-79
Mg Isotopes	80-81
Higher Energy Solar Particles	82-90
Solar Surface Nuclear Proceses	91-99
Mission Status/Summary	100-104



# What: Mission in a Nutshell

 Placed a spacecraft outside the terrestrial magnetosphere

### Exposed Materials

- Solar wind ions (keV/amu) implant and stick
- Exposed for 27 months
- Fluences low, so materials must be ultrapure.
- Returned materials to Earth for analysis in terrestrial laboratories.



# **Why: Genesis Science Objectives**

- Provide solar isotopic abundances to level of precision required for planetary science purposes.
- Provide greatly improved knowledge of solar elemental abundances.
- Provide a reservoir of solar matter to meet the needs of 21st century planetary science.
- Provide elemental and isotopic data for the 3 different types ("regimes") of solar wind.



# **Solar Wind Regimes**

- Three different kinds ("regimes") of solar wind:
  - High speed (coronal hole)
  - Low speed ("interstream")
  - Coronal Mass Ejections
- Genesis separately sampled each of 3 solar wind regimes as well as bulk solar wind:
  - Allows correction for differences in composition between sun and solar wind
  - Agreement in derived solar composition from different regimes validates correction procedures





### **Canister and Collector Materials pre launch**





# **Analysis Overview**

- Genesis sample analysis/testing is proceeding on a broad front in 28 laboratories worldwide.
- Rates vary, but progress is being made.
- The goal of Genesis is quantitative data; great emphasis on getting numbers right.
- A major advantage of sample return missions is that important data can be verified, and in most cases, replicated with different techniques.
- A major effort has been to make accurate, replicated measurements of the fluences of Mg and Ne. Most techniques can analyze one of these elements, which will then constitute primary quantitative reference fluences for other elements.



# Analysis Overview, con't

**Two distinct requirements:** 

- Extract implanted solar wind from collector materials.
- Analyze extracted solar wind.

Can mix and match approaches for extraction and analysis. Mass spectrometry is the most widely-used analysis technique.



# **Science Team Analysis Methods**

- Secondary Ion *Mass Spectrometry (SIMS)* 
  - Solar wind extracted by ion beam sputtering
- Gas Source Mass Spectrometry
  - Extraction by laser ablation or chemical etching (HNO<sub>3</sub>, Hg)
- Resonance Ionization Mass Spectrometry (RIMS)
  - Extraction by ion beam sputtering
- Total Reflection X-ray Fluorescence
  - in-situ analysis; unique in not requiring extraction.
  - essentially non-destructive.
- Inductively-coupled Plasma Mass Spectrometry
  - Extraction by differential chemical etching
- Accelerator Mass Spectrometry
  - Extraction by differential chemical etching.
- Radiochemical Neutron Activation Analysis
  - Extraction by differential chemical etching.



# Work published

**2008 Lunar Planetary Science Conference** 

- 14 abstracts.
- 9 Oral presentations.

In addition to the conference abstracts/presentations, there were 20 presentations to the Genesis Science Team meeting on Monday March 9. These contributions are the basis for what is in this quarterly report.



# **Solar Wind O Isotopic Composition**

- This is our highest priority science objective.
- Three of the four quadrants in the concentrator target were designed to support O isotopic analyses.
  - SiC (2)
  - 13C CVD diamond (1)
  - All of these quadrants were unbroken in crash.



Plan A for O isotopic analysis:

- Ability to replicate important results by independent techniques is a major advantage of sample return missions.
- Three major O isotope efforts have been supported by the Project.
  - Secondary ion mass spectrometry (UCLA MegaSIMS): SiC target
  - Laser ablation extraction; isotope analysis of CO (Open U): <sup>13</sup>C CVD target.
  - Laser Fluorination extraction (UCSD); isotope analysis of O<sub>2</sub>. SiC target.



#### Solar Wind O profile in target SiC. UCLA MegaSIMS. (McKeegan, Mao, et al. LPSC 08 abstract)



The MegaSIMS is a Genesisdedicated instrument. The front end is a standard SIMS instrument in which 15 keV Oions are produced by sputtering of the SiC sample. However, instead of just measuring the low E secondary ions, they are instead accelerated to 0.5 MeV. The acceleration destroys all interfering molecular ions, in particular the 16OH interference at mass 17. After acceleration, the ions are mass-analyzed and counted with a unique multicollector detector system.

Xylene cleaning produced 150 micron areas free of particulate contamination.

Residual molecular surface contamination was removed by sputter cleaning with low energy (5 keV) Cs. This produces an acceptable loss of about 20 nm, but results in the clean SW profile shown. Instrumental background was reduced to an acceptable level by a cryo-pumped vacuum of about 2e-11 torr. Paper in press in Applied Surface Science.





implications from MegaSIMS preliminary result

We have a clear solar wind signal, background corrections are tolerable

### The Sun appears to be enriched in $^{16}$ O by ~60 ‰ relative to Earth and bulk meteorites

- $\triangleright$ Potentially large systematic errors have yet to be properly evaluated, however all should be mass-dependent.
- $\triangleright$ Genesis data well resolved from terrestrial mass fractionation line.
- Sun is different from Earth
- Sources of mass fractionation:
  - MegaSIMS
  - concentrator optics
  - backscatter from target
  - AAAA solar wind acceleration
  - gravitational settling in photosphere



# **MegaSIMS: What next?**

#### MegaSIMS mass fractionation.

- Present instrumental mass fractionation corrections based on terrestrial magnetite of known isotopic composition. This is not exactly correct, but uncertainties hard to estimate.
- The ideal mass fractionation standard is an O implant into SiC with an independent, accurately-known <sup>18</sup>O/<sup>16</sup>O.
- An implant of the <sup>16</sup>O<sup>18</sup>O (mass 34) molecule is being carried out.
  - The nominal ratio is 1 set by the mass number.
  - Small contributions from the tails of <sup>16</sup>O<sup>16</sup>O from mass 32 in the ion implanter will be monitored by measuring the shape of the mass 32 peak and calculating the contribution at mass 34.
- An alternative approach is to use a SiC implant with the <sup>18</sup>O/<sup>16</sup>O ratio measured by laser fluorination at UCSD.



# **Concentrator Mass Fractionation**

- The spatial distributions of O isotopes are not the same in the concentrator which introduces a mass-dependent fractionation.
- From the beginning, the plan to handle this calculate the amount of fractionation based on the quantitatively-well-understood ion optics properties of the Concentrator (see LANL GPMC report), but verify the calculations by measured <sup>20</sup>Ne<sup>/22</sup>Ne on small areas on the frame of the target holder (Au cross).



In-flight Concentrator Performance from analysis of Au cross. (V. Heber, ETH, Zurich)

#### **Recovered Targets and Holder**



Au cross held targets in place

Ne can be measured on mmsize spots by laser ablation.

Agreement of 12 and 9:00 arms very important; no azimuthal SW inhomogeneities in targets





Model Ne profiles compared to measured on Au cross

- Measured and calculated Ne profiles (previous slide) are close but not in good agreement.
- Refined modeling underway (Wiens, Reisenfeld); calculated distributions may depend critically on solar wind angular distribution.
- But corrections are also required for backscattering on Ne by Au, and these must also allow for the distribution of non-normal incidence angles along the Au cross.
- The electroplated surface of the Au cross is relatively rough. An implant experiment is underway to directly measure the effects in both Ne fluence and <sup>20</sup>Ne/<sup>22</sup>Ne of non-normal incidence and surface roughness.
- Beyond these effects there is also reason to be concerned about the accuracy of the backscatter corrections (following page)



### Heber et al (Zurich) 2008 LPSC Abstract 2327



He, Ne, and <sup>36</sup>Ar were compared on 6 different collector materials. He data are subject to diffusion loss so only <sup>20</sup>Ne/<sup>36</sup>Ar shown here. In light materials, backscattering is negligible and good agreeement is obtained. For Ge and the BMG alloy, backscattercorrected ratios agree with light materials, but Au is overcorrected.

Open squares, measured; solid blue, backscatter corrected.



#### **Ne on Flight SiC Quadrants**

- Given the apparent unreliability of the Au backscatter corrections (previous slide), it appears necessary to sacrifice very small (mmsized) areas on flight SiC quadrant 60001 for laser ablation Ne analysis at Zurich.
- Once the MegaSIMS analysis of 60001 is complete, the quadrant will be transported to Zurich for the Ne fluence and <sup>20</sup>Ne/<sup>22</sup>Ne analysis.
- The backscatter corrections for the SiC are quite small, and there is no surface roughness issue. The effects of non-normal incidence will be directly measured in an implantation experiment.
- The SiC data will provide an accurate set of measurements to refine the Concentrator fractionation model which in turn will lead to an accurate correction for the target O mass fractionation factor.



### **O** isotopic analysis of CVD diamond (Open U)

- UV(193nm) Laser Ablation  $\rightarrow$  Continuous Flow He  $\rightarrow$  Gas Source Mass Spec analysis as CO.
- Surface cleanup step with less than 200 A removed followed by extraction step (2500-3000 A) with all of solar wind.
- <sup>13</sup>C<sup>17</sup>O analysis; higher precision because of lower background.
- Cryogenic trapping and Gas Chromotography separation of CO

#### Analysis Hurdles

- Integrated blank from 1hour of LA
- Losses due to active surfaces with 1ng of analyte species
- Ability to control depth resolution with laser system over large areas
- SW O recoveries based on Implant analysis
- Minimizing extracted interferences (CO rather than OH)



#### Laser Ablation Extraction Efficiency and Blanks

- Based on <sup>18</sup>O implants of known fluence.
- Simulated flight implant samples (LTI) available:
  - H loaded at flight fluences and energy distribution
  - Heated for 28 months at flight temperatures (160 deg C)
  - High fluence implants are contaminated with hydrocarbons and overlying SiO<sub>2</sub> layer:
    - Require HF treatment followed by uv-ozone to clean up before realistic <sup>18</sup>O experiment possible.
    - XPS studies shows that this works, but many monolayers of O, presumably bonded to C, remain.
    - Attempt will be made to treat with H<sub>2</sub> to remove surface O.
  - <sup>16</sup>O/<sup>18</sup>O in implant low, so LTI samples provide good blank along with extraction efficiency measurement.
- Control samples, including <sup>13</sup>C flight spare controls available for blanks.
- Comparison of <sup>16</sup>O blank from HF-etched LTI sample with unetched implant provides some check on possibility of deep O uptake due to H radiation damage.
  - Such uptake appears not to happen with SiC, but CVD could differ.
  - XPS on appropriately cleaned LTI sample shows no increase in O due to acid etching, but XPS may not look deep enough.



# **O** isotopes by Laser Fluorination (UCSD)

Extraction of solar wind oxygen as O<sub>2</sub>

- Ablate solar wind collector substrate by a excimer laser
- In presence of ultra high purity fluorine gas (F<sub>2</sub>)
- Subsequent cleaning of analyte gas oxygen
- Determination of oxygen isotopic ratio by IRMS

#### **Required Steps**

- 1) Building up a ultra low oxygen background fluorination system
- 2) Generating ultra low background F2
- 3) Setting lasing parameters and lase (artificially) implanted samples
- **4)** Develop surface cleanup step
  - **• GC** monitoring of SiF<sub>4</sub> and CF<sub>4</sub> enables control.
- 5) Blank analyses.
- 6) Lase flight samples



#### Laser Fluorination Extraction Efficiency and Blanks

- Almost identical to plan for CVD given earlier.
- Based on <sup>18</sup>O implants of known fluence.
- Simulated flight implant samples (LTI) available:
  - H loaded at flight fluences and energy distribution
  - Heated for 28 months at flight temperatures (160 deg C)
  - High fluence implants are contaminated with hydrocarbons + overlying SiO<sub>2</sub> layer:
    - Require HF treatment followed by uv-ozone to clean up before realistic <sup>18</sup>O experiment possible.
    - XPS studies shows that this works, but many monolayers of O remain ("oxycarbides").
    - Attempt will be made to treat with H<sub>2</sub> to remove/exchange surface O.
  - <sup>16</sup>O/<sup>18</sup>O in implant low, so LTI samples provide good blank along with extraction efficiency measurement.
- Comparison of <sup>16</sup>O blank from HF-etched LTI sample with unetched implant provides check on possibility of deep O uptake due to H radiation damage.
  - SIMS studies show that uptake doesn't happen with SiC, so acid etching can be used to remove particulate contamination.
- Additional SiC control samples, cleaned of inorganic constituents at XPS levels, available for blanks.



#### **Laser Fluorination Status**

- All hardware in place and functioning.
  - Cleanup and GC systems deliver pure  $O_2$  to mass spec.
- Laser parameters have been optimized.
- no F<sub>2</sub>, no laser system blank: no detectable O<sub>2</sub>
- F<sub>2</sub> (but no laser) system blank about 1e14 atoms O
  - Concentrator average O fluence estimated as 3e14/cm<sup>2</sup>.



### Surface cleanup; brown stain removal

#### **Options**

- Fluorination (F<sub>2</sub> reacts rapidly with hydrocarbons)
- Laser fluorination
- Laser ablation.
- Various combinations of these have been tried on small flight diamond-like-C (DOS) collector array samples
  - Assumes brown stain same material on DOS array and SiC target samples.
  - Verify brown stain levels by XPS prior to treatment.
  - Assess brown stain removal by XPS after treatment.

All samples pick up F, presumably as fluorocarbon deposit.

Hard to assess brown stain removal because flurocarbons may bury brown stain.

May have to use uv-ozone removal of brown stain on flight sample

- XPS studies verify that this works.
- HF treatment necessary to remove residual SiO2
  - but acid treatment removal of particulate contaminants necessary in any case.
- Cleanup step after ozone/HF treatment.



The isotopic composition of N shows wide variations in solar system materials. Focus here on Earth-Jupiter difference.





### N isotope analysis status

- This is our #2 measurement objective.
- Samples of Au-on-sapphire (AuoS) have been allocated to two laboratories:
  - U. Minn (release and analysis of N<sub>2</sub> by room temperature amalgamation)
  - CRPG Nancy, France (laser depth profiling with analysis of N<sub>2</sub>)
  - more details on following slides.
- Nancy also allocated "Au cross" frame from Concentrator target holder.
- Distinctly different analysis techniques are being used, so if consistent results can be obtained, great confidence can be attached to the results.



### Minnesota (Pepin, Schlutter, Becker, Palma)

- Analysis based on room temperature amalgamation of Au with release of N<sub>2</sub> gas for mass spec analysis. Ne and Ar measured as control elements.
- Technique is inherently low blank and insensitive to particulate contamination.
- Brown stain interferes, but removed by O plasma cleaning..
- Blank from cleanup line has been too high, but now improved and blank runs with flight AuoS and in progress.
- Problem has been incomplete N recovery from flight samples relative to Ne or Ar.
  - Ne/Ar recovery 60-70%, but based on estimated SW N fluence, recovered amounts of N range from 0 to 60% (1 sample).
  - plasma cleaning may leave SiO2 residue, but removal of residue with HF etching doesn't improve yield.



#### Nancy (B. Marty) measurement of <sup>15</sup>N/<sup>14</sup>N

- Approach is laser ablation of AuoS or Au cross from concentrator target holder (see Nov 07 and Feb 08 Science Team GPMC).
- Implants show release of N as N<sub>2</sub> with 100% yield.
- Ne measured as control element in flight samples; allows partitioning of measured N in terms of terrestrial contamination and solar wind.
- Multi step extraction: laser ablation depth profiling.
  - 1st steps should remove any N from particulate contamination.
  - Depth profiling is carried out varying the number of laser pulses in a controlled manner.
  - Maxmize SW recovery relative to background contamination by following release of solar wind 20Ne.
  - Ablation stopped prior to reaching Au-sapphire interface avoiding this major souce of contamination N.
- Brown stain removed by uv ozone in collaboration with Open U (S. Sestak).
- Procedural blanks at 10% solar wind levels, which is good.
- Impurity level of N in both materials is a serious problem.
  - Au cross has more solar wind N but also more impurity N, break even on signal to noise.
- Error bars are large, but result is surprising.



# The isotopic composition of N shows wide variations in solar system materials. Sun like Earth, not like Jupiter.





# **Nancy Preliminary Results**

- For both AuoS and Concentrator Au Cross the Au has a large amount of terrestrial impurities.
- The Au cross has more solar wind N because of the Concentrator, but it also has higher levels of impurities.
- For both materials, the maximum observed N/Ne ratio is about 23% of that expected for the solar wind.
- Based on the measured amount of solar wind Ne, the measured <sup>15</sup>N/<sup>14</sup>N can be partitioned into solar wind and contamination components.
- For AuoS: solar wind <sup>15</sup>N/<sup>14</sup>N = (3.44±0.73) x 10<sup>-3</sup>
- For the Au cross: <sup>15</sup>N/<sup>14</sup>N = (3.69 ± 0.24) x 10<sup>-3</sup>
- This agrees with the terrestrial atmosphere: <sup>15</sup>N/<sup>14</sup>N = 3.67 x 10<sup>-3</sup>
- Although errors are large, the solar wind <sup>15</sup>N/<sup>14</sup>N is very different from that observed for the Jovian atmosphere (2.3±0.3) x 10<sup>-3</sup>, a very surprising result.
- Some of high background in Au cross may be surface contamination, and better data possible with ozone-cleaned Au cross sample; analyses in progress.



#### N Interpretations

- Preliminary data by entirely different technique from U. Minn. are consistent with Nancy isotopic composition.
- Based on Ne and Ar (see later slides or Meshik et al Science paper), widely believed that terrestrial atmospheric <sup>15</sup>N/<sup>14</sup>N was increased by atmospheric escape. Sun like Jupiter.
  - No support for this in preliminary Genesis N result !
  - If NeAr fractionations are from atmospheric escape, may have occurred very early with N still bound in solid Earth.
- N isotopes may indicate fundamental differences in materials for inner and outer solar system !
  - This opens up the potential of a major synergy with Stardust, where no evidence to date of low <sup>15</sup>N/<sup>14</sup>N in Stardust data; instead high <sup>15</sup>N/<sup>14</sup>N found, consistent with coma molecular species.
  - Some Stardust grains come from inner solar system but not necessarily all
  - Is low <sup>15</sup>N/<sup>14</sup>N unique to Jupiter?.
- Inner/outer difference may be in gas/dust.
  - Inner solar system materials may be preferentially dust, but surprising to find dust/gas ratio higher in Sun than Jupiter.



# Plan B for N

- Analyses at both Nancy and Minnesota have been limited by high amounts of background N in Au collector materials.
- Given the surprising, and important, result of the Au samples, it is important to confirm with more precise data and to see if differences between terrestrial and solar N exist.
- This possibility was anticipated in mission planning, and 1/4 quadrants in Concentrator target contained a Sandia diamond-like-C (DOS) sample from which N should be analyzable by stepwise combustion or possibly by SIMS.
  - This quadrant was broken in the crash, but most pieces have been recovered.
- Stepwise combustion efforts are underway at Open U (England) and U. Minnesota.
  - Prelaunch tests at Minnesota had shown ≈ 50% recovery on <sup>15</sup>N implants , but 2008 OU experiments did not recover <sup>15</sup>N until the Si substrate of the DOS was dissolved away. This is a discrepancy to be resolved.
- The possibility of SIMS analysis will be evaluated based on attempts to measure N fluence in collector array Si samples (see later slides).



Noble Gases in Bulk Solar Wind.

This is our #3 measurement objective.

Data on Ne and Ar have been published (Meshik et al, *Science* 318, 443, 2007).


#### Regime Ne, Ar isotopic analyses, Wash U Meshik et al. Science Oct 19, 2007

Sample, collection time	<sup>20</sup> Ne/ <sup>22</sup> Ne	<sup>36</sup> Ar/ <sup>38</sup> Ar	<sup>20</sup> Ne/ <sup>36</sup> Ar
Bulk, 852.88 days	13.972±.025	$5.501 \pm .005$	59(5)
High speed,313.01 days	13.956±.041	$5.502 \pm .010$	66(6)
CME, 193.2 days	13.979±.031	5.467±.017	59(5)
Low speed,333.67 days	13.990±.031	5.508±.010	46(4)
Apollo SWC (7)	$13.7 \pm 0.3$	$5.4 \pm 0.3$	$49\pm7$

Less than 0.6% differences among regimes based on 1 sigma errors.

Strong limits set on SW Ne, Ar isotopic fractionation.



### Regime <sup>20</sup>Ne/<sup>22</sup>Ne Wash U

Regime		
	<sup>20</sup> Ne/ <sup>22</sup> Ne	error
Bulk	13.86	0.06
High Speed (Coronal Hole)	13.93	.08
CME	13.99	.08
Low Speed	13.89	.04
Apollo SWC foils	13.7	0.4
Terrestrial Atmosphere	9.8	

Big Ne isotopic differences (known from SWC) between Earth and SW. Genesis data much more precise, but given large difference with atmosphere, this isn't important.

Ar in regimes same to within 0.2%; see next slide.



Differences in SW and terrestrial Ar precisely defined. from Meshik et al (2007)



Ne and Ar imply Early loss of terr. atmosphere? Shouldn't there be variations in N as well?

Major issue requiring further study



- Resonance ionization mass spectrometer for measuring Xe isotope ratios
- Best blank ~ 1000 atoms <sup>132</sup>Xe
- Detection limit ~ 950 atoms <sup>132</sup>Xe
- Samples restricted to < ~ 10<sup>6</sup> atoms
- Xe extracted by IR laser step heating.
- Sum results from large number of ≈ 3 mm Si samples
  - crash has supplied many of these.
- Replicate analyses of samples of 2e4 to 4e4 <sup>132</sup>Xe atoms from individual extraction steps of Genesis samples show a 28 permil standard deviation for <sup>132</sup>Xe/<sup>136</sup>Xe.



### RELAX



Isotopic compositions of largest extractions agree with solar wind Xe from lunar ilmenite samples.

Complication is that amounts of Xe in some control samples as large as flight samples.

Analyses were made of FZ Si which was made in Ar atmosphere. New samples of CZ Si which should have lower impurity levels are being analyzed.



#### Heavy noble gas analyses on the Polished Al Kidney (PAC) Wash U



Fig. 1. "Kidney" collector after initial subdivision. Cube is 1 cm in size.

This large piece of polished AI was added to the part of the canister surface exposed to the solar wind once the collector arrays were deployed. (See slides 6 and 7). The anticipated use was to provide large area samples for heavy noble gas (Ar, Kr, Xe) analysis. Prelaunch blanks of bulk samples indicated that if a thickness of less than 0.5 micron were analyzed, the levels of ArKrXe contamination were not significant compared to the solar wind.



#### Xe background from PAC

- Test experiments on flight spare control samples indicate that there are relatively large amounts of surface contamination associated with the polishing process.
- Most of the background appears to be hydrocarbons, but some could be Xe.
- This contamination occurred during the polishing of the kidney.
- It is not known how deep the contamination has been worked into the surface during the polishing.
- uv-ozone cleaning of flight spare control samples is underway which could remove much of the hydrocarbon background.
- In any case, using the PAC for heavy noble gas analysis requires being able to do noble gas depth profiling.
  - This has been demonstrated for Ar from the PAC



#### Laser Ablation Ar depth profile from PAC



From Meshik et al LPSC 2008 abstract. The power level of a uv ablation laser was adjusted to remove only a small amount from the surface, and the solar wind Ar isotopic composition measured with each step in the release. The SW Ar isotopic composition is known from previous studies and can be distinguished from atmospheric Ar based on the measured amount of <sup>40</sup>Ar. The decreasing <sup>36</sup>Ar/<sup>38</sup>Ar ratio results from isotopic fractionation during implantation and is expected.

The successful SW Ar depth profiling points the way for similar experiments on Kr and Xe which will minimize the effects of surface contamination.

Procedures for multicollector heavy noble gas isotopic analyses have been worked out. This will maximize the precision that can be obtained from a given gas sample.



Preliminary results on heavy noble gas fluences and elemental ratios (PAC Wash U)

### 1.1E+6 <sup>132</sup>Xe atoms/cm<sup>2</sup>

Upper limit because of hydrocarbon background, but close to predicted 9e5/cm<sup>2</sup> <sup>132</sup>Xe fluence

~ 1.2E+7 <sup>84</sup>Kr atoms/cm<sup>2</sup> Predicted = 1.8 e7/cm<sup>2</sup>

 $^{36}Ar/^{84}Kr \approx 2200$ 

 $^{84}$ Kr/ $^{132}$ Xe  $\approx 11.4$ 

### **Carbon Isotope Analysis (Open U)**

#### Approach

- Wet chemical and UV/Ozone pre-clean
- Offline vacuum furnace bake (~400°C)
- Stepped combustion; special low blank silica glass tube.
  - 400-800°C terrestrial carbon oxidised
  - 800-1400°C implanted SW carbon released (based on <sup>13</sup>C implants)
  - Use of Pt catalyst significantly improves CO<sub>2</sub> yield,
  - but still C loss to evaporated Si on tube walls, even in presence of O2. changing tubes between combustion can minimize this.
- UHV gas cleanup
- IRMS analysis of CO<sub>2</sub>
  - Custom static vacuum MS
  - Sub-ng sensitivity
- Stepped combustion very good at removing terrestrial contamination, especially on scratched, pitted surfaces where laser ablation may be less effective.

Blanks, not mass spec is limiting factor

Based on system blanks, solar wind C isotopic analyses possible now on 10 cm<sup>2</sup> of B/C array FZ Si. This much sample available.

Blanks combusting cleaned flight spare control samples now in progress.

With a factor of 3-5 blank reduction, smaller areas possible.

Laser heating of sample now being installed should give lower blanks.



# Science Issue: Do Sun and solar wind have same *elemental* composition?

- Spacecraft data have shown that high first ionization potential (FIP) elements are depleted in solar wind compared to solar surface (photosphere).
  - e.g. Fe/He is higher in SW than in photosphere.
- Data for most easily-ionized elements (FIP < 9eV) appear unfractionated.
  - Most of elements in terrestrial planets have FIP<9eV</p>
- Genesis will provide a better test, but never will escape need to know a few photospheric elemental ratios accurately.
- If fractionations due only to first ionization potentials, solar wind and photosphere isotope ratios expected to be same.



### **Fractionation Factor**

# F = (X/Mg)<sub>SW</sub> / (X/Mg)<sub>photosphere</sub>



### **FIP Plot from spacecraft data**





#### FIT (first ionization time) plot from spacecraft data

FIT is an estimate of the time required to ionize a neutral atom upon transport from the lower temperature photosphere into the solar corona, from where the ion will be accelerated and incorporated into the solar wind. FIT is more physical than FIP, but is model-dependent. Data plots using FIT are cleaner than those with FIP with the 9eV fractionation cutoff (translated to about 20 sec ionization time) showing clear depletions of high FIP/FIT elements. Note that, although Fe and Mg have the same FIP, they differ considerably in FIT





Mg Secondary Ion Mass Spectrometry (SIMS) (Jurewicz et 2008 LPSC abstract)

- Sample sputtered with O2+ ions, sometimes in presence of O<sub>2</sub> flood gas for Si.
- Analyses at ASU (Jurewicz, Guan, Hervig), UCLA (McKeegan), and CIW (Nittler)
  - All SIMS instruments, but different design.
    - replicate analyses by different instruments; unique feature of sample return missions:
  - Mg+ ions produced and analyzed with mass spectrometer.
- Measure Mg relative to matrix ion:
  - Si or C from Amorphous diamond-like-carbon (Sandia).
  - Verify accuracy by replicating results on different materials
    - better quality data is a major feature of sample return missions.
- Data from 30-50 micron-size areas.
  - Many analyses possible even for small samples.
- Depth profiles acquired in 5-10 minutes, (after hours of tuning).
- Quantitation simple in principle; relative to lab implant standard.



Beautiful depth profiles for Mg in Sandia diamond-like C and Si collector materials (Jurewicz et al, ASU)





#### Mg fluence data ordered by run date.

see following slides for interpretations.





#### Interpretation: Mg fluences in Si and Sandia diamond-like-C

• Data points in previous slide from individual profiles. "Error" bars represent % range of implants run on same day as sample.

- Beautiful depth profiles, but more scatter in derived fluences for Sandia than for Si.
- Blue and black points "externally" standardized with separate implant sample.
- Almost no overlap of Si and Sandia fluences, standardized externally.
- Implant variability much worse for Sandia than Si. However, some of the variability comes from deliberate variations in analytical conditions.
- Three SIMS instruments with two different designs (ASU, UCLA, CIW) give same Sandia/Si Mg fluence difference. This is a SIMS effect, not a specific instrument effect.

• The Sandia/Si fluence difference for Mg has been a major unresolved discrepancy, but was resolved by implanting <sup>25</sup>Mg *into flight samples* as internal standard, as opposed to external standardization on a separate implant sample (more details in August Science Team GPMC).

• Using internal standard (red points in previous slide) good reproducibility for both materials, and for first time, Mg fluences for Si and Sandia agree.

• ANL data by resonance ionization mass spectrometry (RIMS) also show no difference for Mg between Si and Sandia.



#### Mg fluence interpretation, con't

- Previous factor of 2-3 Si-Sandia difference reduced to 5% !
- To increase precision, all samples analyzed in the same center position of sample mount. Mg/Si or Mg/C ratios vary with mount position, especially Mg/C, which may account for some of scatter in previous Sandia data.
- Flight Implant Sample fluences are in the higher part of the previous Si range, much lower than most previous Sandia analyses.
- Reason for original discrepancy may be effect of solar wind H on Mg sensitivity in Sandia. H not in implant standards.
- Adding H to implant standards will check this; major series of H implants into all previous implant standards now being carried out.
  - May need to have H in implants for SIMS analysis from here out. Painful, but feasible.
- We adopt 2.15e12/cm<sup>2</sup> as our present best estimate of Mg fluence.
- We adopt ±5% precision at present. Converting precision to accuracy requires independent verification of implant standards. This should be possible without significant decrease in final uncertainities.



### Fe fluences, ordered by run date





### **Fe fluence interpretation**

- Good depth profiles; surface contamination well resolved.
- Derived fluence from each profile plotted in previous slide.
- Prelaunch predictions based on literature diffusion coefficients for Fe in Si indicated that Fe would be highly diffused at flight temperatures. However, in the SoS collectors, where Si is only a thin (1800A) layer on a sapphire substrate, the Fe has no place to go and should be analyzable. This was confirmed, but in fact typical SW Fe depth profiles were obtained indicating that significant Fe diffusion in Si did *not* occur and that Fe can be analyzed in Si collectors. This was done
- Unlike Mg, 3 different materials, Sandia, Si, Si on sapphire (SoS), give consistent Fe fluences.
  - Two different SIMS instruments (ASU, UCLA) used.
  - NanoSIMS analysis of Sandia sample 60062 by Nittler et al. (CIW) gives 2.39±0.15 e12/cm2, at the high end of the range on the previous slide.
- Earlier data discrepant for unknown reasons but data from four most recent runs are consistent with an Fe fluence of 1.41±.07 x 10<sup>12</sup>/cm<sup>2</sup>.
- Most recent run done with sample in center hole position in sample mount; should give highest precision.



#### **Comparison with photosphere and spacecraft Fe/Mg**



All data sets agree within errors of other data. No evidence for FIP (FIT) fractionations. Both Fe and Mg have FIP < 9 eV.



#### **Compare with CI chondrites**



Most compilations of "solar" elemental abundances based on analyses of CI chondritic meteorites. Justification for this is agreement with photospheric abundances. Genesis Fe/Mg, at present, distinct from CI ratio, but systematic errors in implant fluences must be assessed before final conclusions drawn. Precision will remain as on figure, but value of Fe/Mg could change.



### Plan to verify Mg and Fe implant fluences

- One set of Fe implants are available (Kroko 2005).
  - 3 separate implants: 56Fe at 5e15/cm2, 56Fe at 4e13/cm2, 54Fe at 2e13/cm2.
  - SW fluence data on Sandia (diamond-like-C), SoS, and Si.
  - Fluence can be independently analyzed accurately on the 5e15/cm2 Si implant by isotopic dilution using ICPMS (FSU).
  - Accurate measurement of high fluence implant in one material applies to all materials in same implant.
  - Sandia data based on 4e13/cm2 implant but accurate measurement of *relative* fluence measurement by SIMS of 5e15 and 4e13 Sandia implants is possible (ASU, CIW).
  - High fluence implant will also be calibrated by RBS (ASU) and SXRF (SSRL)
- Three sets of 25Mg implants are available
  - HRL 2002, Kroko 2006, Kroko 2007.
  - SW data based on Si and Sandia using HRL and K06 implants as standards.
  - 2007 implant has high fluence 25Mg implant in Si which can be analyzed accurately by isotopic dilution.
  - Independent calibrations by TIMS (JPL) and ICPMS (FSU) in progress.
  - *relative* fluences for other implants can be measured precisely by SIMS
    - Replicate measurements at ASU and CIW in progress.



**Resonance Ionization Mass Spectrometry (RIMS) essentials.** 

- RIMS analysis begins with sputtering with a primary ion beam, like SIMS.
  - Analyses on 100x200 micron spots.
- However, only roughly 1/1000 of the atoms sputtered are the ions utilized by SIMS.
- RIMS ionizes the sputtered neutral species by timing ionizing laser pulses with an ion beam pulse. Mass analysis by time-of-flight mass spectrometry.
  - Laser duty cycle limits acquisition time, but improvements have doubled this from 1 to 2 kHz
- A very large fraction (>10%) of the neutrals can be ionized and counted, producing very high sensitivity.
  - About 20% has been demonstrated for Mg.
- The photoionization takes place in two steps.
  - One laser frequency is highly tuned to excite the selected atom into an excited state. This
    provides high selectivity of the element being analyzed from any molecular ions of the same
    mass.
  - A second laser ionizes the excited atom which is detected by the time of flight mass spectrometer.
- A RIMS instrument designed specifically for Genesis samples is operating at ANL.
  - Optical imaging system allows particles down to micron size to be avoided.
- At present, both SIMS and RIMS are useful for Genesis samples, but eventually only RIMS will be able to analyze elements of low abundance.
  - Present detection limit is below 50 ppt.



#### RIMS Mg Analysis (ANL; Veryovkin et al LPSC 2008 abstract)



Figure shows both SW Mg depth profiles and that of 25Mg implant standard at 2 KeV/amu, along with a calculated profile for Mg at 1 KeV/amu. The solar wind has a distribution of energies.

The superb RIMS depth resolution is illustrated with the use of a logarithmic depth scale. Surface contamination (below 10 nm) and solar wind are clearly resolved.

The Mg fluence derived for Si  $(2.0 \pm 0.1 \text{ e}12 \text{ atoms/cm}2 \text{ agrees with the SIMS value.}$ 



### Fe and Mg fluences from ICPMS (FSU)

- see Huang and Humayun LPSC 2008 abstract
- This approach involves removal of Si layer on cm-size pieces of Si-onsapphire by HF/HNO3 dissolution.
- See discussion of surface cleaning in separate Genesis Sample Cleaning ppt file.
- Number of atoms measured by isotopic dilution with <sup>26</sup>Mg and <sup>54</sup>Fe tracers.
- Fluence calcuated by measuring the area etched on sample.
- Mg fluence = 2.0 ± 0.1 e12/cm2 which agrees with the ASU SIMS and ANL RIMS results.
- Fe fluence = 2.0 ± 0.1 e12/cm2 which is about 30% higher than the ASU SIMS or APS TRXRF result.
  - Some flight spare control samples analyzed as blanks show high amounts of apparent Fe impurities
  - These probably represent occasional Fe particulate contamination of the Sisapphire interface.
  - Future analyses will focus on Si collector materials.



#### Solar wind C and N fluences (Y. Guan, A. Jurewicz)

- The elemental abundances of C, N, and O are important because they define the transition in FIP/FIT plots between high FIP elements (H, He) that are clearly depleted and low FIP elements which appear to be unfractionated relative to the photosphere.
- FZ Si collectors are sufficiently pure to provide a measurement of these elements; however, previous attempts at SIMS analyses were unsuccessful because of the combined effects of surface contamination and instrumental background.
- As shown by the MegaSIMS work on O, good vacuum conditions can bring the instrumental background down to acceptable levels.
- A new Cameca 7f SIMS instrument at CalTech is available with pressures in the 10<sup>-11</sup> torr range, which has produced acceptably low instrumental backgrounds.
- For unknown reasons, the low energy Cs sputtering for surface cleaning of O from SiC does not work for C from Si, so several stages of cleaning with uv-ozone (JSC, Open U) were used to minimize the surface contamination background.



#### C depth profiles in FZ Si (Y. Guan, Caltech)





#### **Background in SIMS C analysis**

- Examples of <sup>12</sup>C depth profiles are shown in the preceding slide, illustrating two background components:
  - surface background reflecting either adsorbed gases or a distribution of small (10-1000 A) aerosol particles.
  - an "asympototic" instrumental background.
    - the cps C associated with the instrumental background is independent of sputtering rate, showing that the C signal is not due to C impurities in the Si.

Profiles are shown for flight sample 60342, a FZ control sample, OU 199, and the predicted solar wind profile (Chad). The data plotted are "Apparent" C concentrations because at the shallowest and deepest depths the measured signal is not from C in the sample. For the control, none of the C is from the sample.

As hoped, the achieved asymptotic background in the CalTech instrument was very good, the best we have ever done in many such tries

The surface background is the major problem. Mixing by the primary Cs ion beam causes significant surface contamination contributions down to depths of as much as 1000A.



#### **Background in SIMS C analysis, con't**

- The samples analyzed had all been uv-ozone cleaned by S. Sestak (Open U), which very effectively removes surface hydrocarbons.
  - XPS studies of Sestak-cleaned samples show very small residual C peaks.
  - But the XPS data are consistent with an amount of surface C equivalent to about 10<sup>14</sup> C atoms/cm<sup>2</sup>. Although this much C is much less than a monolayer, it can account for the SIMS surface C component.



#### Background in SIMS C analysis, con't

- We do not believe that the observed surface component is due to particulate contamination.
- During the SIMS sputtering process a surface adsorbed layer is mixed into deeper depths ("gardened"). This can account for the observed surface component in the preceding slide.
  - An exponential depth dependence is expected for gardening. The surface component depth profiles are good exponentials beyond 150-200 A
    - The very shallow peak at around 100 A represents sensitivity changes associated with the acquisition of a steady state Cs layer on the surface.
  - Particles would not necessarily give an exponential depth distribution for the surface component.
  - As illustrated by the two profiles shown, 5 different samples showed the same shape for the surface component. This is easier to explain by an adsorbed gas layer than by particles. Particulate contamination is characterized by extreme variability..

Similar behaviour is observed for N and O, although data processing has not been completed.



Interpretations: C surface contamination.

- Despite identical cleaning history, the amount of surface contamination is variable, actually lower on the flight sample in the cases shown.
- The surface contamination is not crash-related, but presumably represents a tightly bound surface adsorbed component.
- The surface contamination was reduced by uv-ozone cleaning sufficient to measure primarily solar wind C at intermediate depths (1000-1500 A),but additional ozone treatment did not produce significant reductions .
- The good agreement between the intermediate depth solar wind C and the calculated SW profile shows that our pre-launch estimate of the solar wind C fluence is fairly good.
- Although corrections of around 50% will be required an estimate of the C wind fluence will be possible, but will have rather large errors.
- Similar-looking data were obtained for solar wind N.
- A technique other than SIMS may be required for C and N fluences of acceptable accuracy, although "backside depth profiling" which measures SW before hitting surface contamination is under study.
  - Backside depth profiling is a relatively well-established approach in the semiconductor analysis world.



### TRXRF

- Analytical Application of Snell's Law.
- Intense collimated beam of high energy synchrotron radiation photons incident at critical angle.
- While being reflected, photons induce fluorescent X-rays from elements in near surface regions (see following page).
- Small variations (0.x degrees) in tilt angle probe below surface to solar wind depths, resolving surface contamination from solar wind.
- Minimal penetration surpresses continuous X-ray background giving high signal/noise.
- Two groups (SSRL and APS) working collaboratively
- Sapphire and SoS identified as optimum materials.
- Depth resolution allows separation of surface contamination, but cleaning still required; tests in progress.





## GENESIS APS (Kitts et al)








### **APS TRXRF Status and Fe fluence**

Implementation and commissioning of new instrumentation (a multi-channel Si detector) to increase our sensitivity, reduce measurement time and lower our minimum detection limit.

**Improvement of data reduction via the development of deconvolution routines** 

- Development of forward modeling algorithms for reflectivity and fluorescence yield analysis in order to determine element specific depth profiles
- **Extraction of absolute concentrations from the depth profiles.**
- **Derived Fe fluence from sapphire collector:** 1.48e12/cm<sup>2</sup>
- This is in good agreement with ASU SIMS result.



### Photosphere/SW *Isotope* Fractionation?

- More an issue to Genesis than elemental fractionation since isotopes are our highest priority objective.
- Spacecraft data: <5%/amu. Too large for planetary science.
- FIP/FIT are atomic properties, wouldn't expect isotopic fractionation.
- "Coulomb Drag" effects associated with acceleration of solar wind from corona would be mass dependent; specific model by Bochsler (2000); relatively large fractionations predicted.
- Regimes are different kinds of solar wind, so if isotopes fractionated, expect to see differences.
- Test with He, Ne and Ar extracted by laser ablation at Zurich (Sandia) and Wash U St. Louis(AloS) from 5 mm-sized fragments.
  - no cleaning required.



#### Inter-regime He isotopic variations readily observable.



<sup>3</sup>He/<sup>4</sup>He higher in low speed than high. Right direction for Coulomb Drag effect, and agrees quantitatively with Bochsler (2004).



Heber et al., data. Bochsler (2000) Coulomb Drag model



He isotopic data very precise. Error bars are size of points. Genesis He/H data from LANL monitors. Bochsler model is parameterized in terms of He/H. Variations in He/H are assumed to be due to Coulomb Drag, then variations in any other isotopic or elemental ratio can be predicted.



#### **Genesis He isotope variations: Interpretation**

- Data for H and L arrays agree well with Bochsler (2000) Coulomb Drag model.
- E array [coronal mass ejection (CME) regime] does not.
- Bochsler (2000) extrapolation to the helioseismology He/H gives a solar <sup>4</sup>He/<sup>3</sup>He ratio of about 3300.
  - This is significantly higher than other estimates of solar <sup>4</sup>He/<sup>3</sup>He from the solar wind literature. E.g. Bochsler (2007) gives 2700.
  - Based on the Galileo probe measurement of Jovian <sup>4</sup>He/<sup>3</sup>He and D/H, a solar <sup>4</sup>He/<sup>3</sup>He of 2100 ± 600 is predicted. This is only compatible with the Bochsler (2000) estimate at 2 sigma.
  - All of the Genesis regime data are greater than <sup>4</sup>He/<sup>3</sup>He=2100, so Galileo probe estimate does seem low. Is this a Jupiter/Sun difference, like N?
- Bochsler (2000) model is semi-empirical; no explicit treatment of solar wind source regions. Assumes all He/H fractionation due to Coulomb Drag, i.e FIP fractionation negligible.
- Either Bochsler (2000) model too general or Genesis CME sample in some way "doesn't count"
- Alternatively, monitor He/H data are not sufficiently precise.
- H fluence measurements possible from Genesis samples and these are in progress (G. Huss, U. Hawaii).



#### Regime Ne, Ar isotopic analyses, Wash U Meshik et al. Science Oct 19, 2007

Sample, collection time	<sup>20</sup> Ne/ <sup>22</sup> Ne	<sup>36</sup> Ar/ <sup>38</sup> Ar	<sup>20</sup> Ne/ <sup>36</sup> Ar
Bulk, 852.8 days	13.972±.025	$5.501 \pm .005$	59(5)
High speed,313.01 days	13.956±.041	$5.502 \pm .010$	66(6)
CME, 193.2 days	13.979±.031	$5.467 \pm .017$	59(5)
Low speed, 333.67 days	13.990±.031	$5.508 \pm .010$	46(4)
Apollo SWC (7)	$13.7 \pm 0.3$	$5.4 \pm 0.3$	$49\pm7$

Less than 0.6% Ne differences among regimes based on 1 sigma errors.

Strong limits set on SW Ne, Ar isotopic fractionation.

H-L limit consistent with Bochsler (2004) Coulomb Drag model.

Better data for solar physics world:

Do regimes represent small variations in large solar/SW differences? compare previous 2 slides.

### Ne and Ar isotopic variations in regime samples.



Heber et al., ETH Zurich LPSC 2008 abstract.
Laser ablation mass spec analyses of regime samples.
Resolvable differences between L and H arrays.
Consistent with upper limits from published data of Meshik et al.
Sense and magnitude of variations between H and I array samples consistent with Bochsler (2000) Coulomb Drag Model



### The importance of Mg isotopes

- The Bochsler (2000) model concludes that inter-regime isotopic variations represent a relatively small fraction of the differences between the photosphere and *any* solar wind sample.
- Measurements of the Mg isotopic composition in Genesis bulk samples provide an alternative way to test solar wind/Sun isotopic differences.
- Excluding CAIs, isotopic differences for most nonvolatile elements among different inner solar system materials, specifically <sup>25</sup>Mg/<sup>24</sup>Mg, are small. For non-volatile elements in general, there may be differences at the ppm level, but this is controversial.
- Thus, a reasonable assumption is that <sup>25</sup>Mg/<sup>24</sup>Mg is the same in the Earth and in the Sun, thus a solar wind/Earth comparison is the same as a direct solar wind/Sun comparison, independent of any other solar data or model.
- Coulomb drag or other generic isotopic fractionations between the photosphere and solar wind can be safely assumed to be mass dependent in the usual geochemical sense,.
- The measurement of <sup>25</sup>Mg/<sup>24</sup>Mg must be made with external corrections for instrumental mass fractionation.
  - The loss of the ability to use <sup>26</sup>Mg to correct for instrumental mass fractionation because of the possibility of <sup>26</sup>Mg from <sup>26</sup>Al decay is not a show-stopper.
- The ability to test for differences in radiogenic <sup>26</sup>Mg between the Earth and the Sun is an important science bonus in the measurement of isotopic variations in Genesis samples.



#### The importance of Mg isotopes, con't

- The Bochsler (2000) model predicts 10-20 per mil differences in <sup>25</sup>Mg/<sup>24</sup>Mg between the Sun and the solar wind. This should be readily measurable.
- SIMS measurements on Genesis bulk Si samples are in progress at Nancy (G. Srnivasan, Toronto).
  - A <sup>24</sup>Mg/<sup>26</sup>Mg implant standard has been prepared to calibrate instrumental mass fractionation.
  - A precise isotopic composition measurement of the standard is being measured by ICPMS (Wadhwa, ASU).
- More precise <sup>25</sup>Mg/<sup>24</sup>Mg should be obtainable by ICPMS (Wadhwa or Humayun, FSU) or by TIMS (Papanassatassiou, JPL).
- These require extraction of Mg from large (cm-sized) areas by preferential dissolution of the implanted surface, which in turn requires improved surface cleaning techniques (e.g. Huang and Humayun, LPSC 2008)



Compare slide 6

BMG = 5 component metallic gla

Synthesized by C Hays, CalTech



#### Genesis Studies of higher energy (SEP) solar noble gases

• Closed-system etching studies of mineral separates from lunar soils indicated higher energy solar noble gases (SEP) with distinct isotopic and elemental composition than the solar wind, e.g. <sup>20</sup>Ne/<sup>22</sup>Ne about 11 as opposed to 13.8 for the solar wind. (see next page).

• The bulk metallic glass was added to the Genesis payload specifically to better understand the origin of the SEP.

• The bulk metallic glass was shown to etch uniformly and to retain He and Ne.

• Subsequent studies (Heber et al; LPSC abstract; Grimberg et al, Geochimica,Cosmochimica, in press) have shown that He diffusion is significant.



### SEP from Lunar Soils; data by closed system acid etching.



# CSSE – Depth Profiling



- Removal of contaminating filr
  - Cleaning with O<sub>2</sub> & SF<sub>6</sub>-Plasma

### Repeated etching with HNO3

- Stepwise noble gas release
- Measurement of He and Ne released at each step

#### BMG chosen for uniform etching.

Progression towards higher ion energy and SW-velocity respectively

# Genesis BMG: CSSE - Release



- BMG CSSE 1
- BMG CSSE 2

The 3 isotope release plot is similar to what observed on lunar samples! Here there is nothing special about SEP; continued etching gives lower 20/22 ratios than 11. Unlike the lunar sample data, Initial ratios are as high as 16, much higher than the average SW ratio of

0.09

0.10





# **BMG Conclusions**

- The BMG Ne isotopic variations during etching can be quantitatively described by the differences in implantation depth of the higher energy <sup>22</sup>Ne in the solar wind.
- The lunar soil Ne-isotope patterns can be explained as a mixture of SW fractionated during implantation and GCR-products distributed in the material.
- "SEP-component" is in all probability an artifact of different implantation depths of single isotopes, not independent component.

"SEP-component" does not represent suprathermal particle composition



Why wasn't true nature of "SEP" recognized before?

Lunar data are better fit with three components:
First lunar extraction steps for Ne agree with Apollo foils (20/22 = 13.7)

 First extraction steps from Genesis BMG: <sup>20</sup>Ne/<sup>22</sup>Ne approaching 16! Never seen in lunar sample data.

- Parts of lunar samples with higher <sup>20</sup>Ne/<sup>22</sup>Ne aren't there!
- All lunar samples have had significant erosion of surfaces !
- No lunar (meteoritic?) sample has an unaffected record of solar wind!



### **Erosion Mechanisms?**

- Sputtering steady state?
  - Requires 1500-2000 A of erosion; too much??
- Minimum erosion about 200-300 A.
  - Still may be too much for sputtering.
- Impact Erosion??.



### Radioactive nuclei in the solar wind.

- The composition of the solar wind contains a record of 4.56x10<sup>9</sup> years of nuclear processing associated with solar flare activity.
  - the abundance of D is entirely the result of solar surface nuclear processing.
  - the abundance of F could be significantly enhanced by proton reactions on <sup>20</sup>Ne.
     etc.
- Long lived nuclei with lifetimes in the 10<sup>5</sup>-10<sup>7</sup> year range have a record of charged particle over a comparable time range.
- Mo-coated Pt foils were put in the lid of the Sample Return Capsule to provide a large collector area to measure the amounts of such nuclei by accelerator mass spectrometry.
  - Prelaunch estimates indicated that this would be difficult, but feasible.
  - Dedicated experiment carried out by K. Nishiizumi (UC Berkeley SSL)
- Special attention was given to <sup>10</sup>Be because measurements in lunar soil samples of surface <sup>10</sup>Be indicated an amount far larger that could be accounted for by present models of solar surface nuclear activity.



### Genesis Mo-Pt Foils (as recovered Sept. 2004)



Although large areas of the foils were recovered from the crash, they were highly crumpled, as shown in this figure and heavily contaminated with Utah dirt.

Foils have to be decompressed in order to be cleaned.

#### Array of guitar tuners successful for smaller foils; should work for larger pieces.

#### Kuni

Removing crumpling: solved.



#### **Lid Foil Status Summary**

- Sufficient area survived the crash, but is in bad shape in terms of crumpling and soil contamination.
- Technique for decompression has been worked out; see previous page.
- Based on measured 10Be concentration of Utah crash site soils, large amount of decontamination required: < 0.1 micrograms soil/ cm<sup>2</sup> foil.
- Large area Mo vapor deposition was accompanied by significant amounts of oxidation.
- This makes the Mo coating highly soluble, even with plain water. Solvent cleaning limited to non-aqueous solvents.

• A large number of tests made with different solvents on flight spare control foils coated with Utah dirt.

- Weight loss measures dirt removal.
- Chemical measurement of amount of Mo loss.
- 70 reagents; 600 test samples
- Solvent only tests of flight foil samples show that several have acceptable amounts of Mo loss (< 10 nm).
  - no large difference between room temperature and 50 deg C in subset of tests.
- Crown ethers dissolved in organic solvents do an efficient job of dirt removal.
- Bubble agitation during tests was inneffective, despite varying conditions of bubbling.



# Vacuum Cavitational Streaming (VCS)

- Vacuum cavitation (low pressure boiling of organic solvents) is a commercial approach to particulate cleaning.
- In addition to bubble cleaning action, ill-defined (at least to us) surface tension effects aid in particle removal.
- Many different solvents have been tested; see following slide.
- Good result: large pink bar, small blue bar.
  - 1:1 ether/water system appears promising.





## H<sub>2</sub> Hydrogenation



Oxidation of Mo foil makes it soluble. If foil can be reduced, then more vigorous solvent cleaning should be possible.

Test samples were soaked in 1500 psi  $H_2$  for 1 hour with significan decresase in Mo solubility.

This approach is promising.



#### **Lid Foil Summary**

- Considerable progress has been made on a very difficult problem.
- A method of decompressing highly crumpled foils has been devised.
- Exposure to H<sub>2</sub> appears to significantly reduce the solubility of the Mo.
- A large number of Utah soil decontamination processes have been tested, and the field of possibilities considerably narrowed.
  - Best results with vacuum cavitation using 1:1 ether: water.
- Tests are in progress on supercritical CO2.

• We are close to beginning decontamination testing cm-sized pieces of the flight foils.



### **Genesis Mission: Top Level Status Summary**

- The bar has been raised considerably by crash, but not giving up on any of our measurement objectives.
- Particulate contamination is our biggest challenge, but
- Optimism is justified by fact that contamination is on the surface,
- And solar wind is *below* the surface.
- The separation between dirt and signal is small (typically 100A).
- But, being a sample return mission, all of contemporary science and technology is available to clean the surfaces without disturbing the implanted solar wind.
- With some luck, major effect will be delay in results.



### GENESIS **Specific Measurement Objectives** (prioritized). Prelaunch.

- (1) **O** isotopes.
- N isotopes in bulk solar wind. (2)
- (3) (4)
- Noble gas elements and isotopes. Noble gas elements and isotopes; regimes.
- (5) C isotopes.
- (6) C isotopes in different solar wind regimes.
- Mg,Ca,Ti,Cr,Ba isotopes. (7)
- **Key First Ionization Potential Elements** (8)
- (9) Mass 80-100 and 120-140 elemental abundance patterns.
- (10) Survey of solar-terrestrial isotopic differences.
- Noble gas and N, elements and isotopes for higher energy solar particles. (11)
- (12) Li/Be/B elemental and isotopic abundances.
- Radioactive nuclei in the solar wind. (13)
- (14) F abundance.
- Pt-group elemental abundances. (15)
- Key s-process heavy elements. (16)
- Heavy-light element comparisons. (17)
- Solar rare earth elements abundance pattern. (18)
- Comparison of solar and chondritic elemental abundances. (19)

Measurement of bulk solar wind except when noted.



**Color-coded Science Assessment; updated 5/08** 

- Measurement can definitely be made
- Should be Possible
- Challenging
- Very Challenging
- Not Possible

The number of green elements continues to grow. There is no red.



### <u>GENESIS</u> **Specific Measurement Objectives**

### (prioritized)

- O isotopes. (1)
- (2) N lsotopes in bulk solar wind.
- (3) (4)
- Noble gas Elements and Isotopes. (He, Ne, Ar Kr, Xe) Noble gas Elements and Isotopes; regimes (He Ne Ar, Kr, Xe).
- (5) **C** Isotopes
- (6) C lsotopes in different solar wind regimes.
- Mg,Ca,Ti,Cr,Ba Isotopes. (7)
- Key First Ionization Potential Elements (Na, Mg, Fe, Si, Ca, Cr, C, N, etc) (8)
- (9) Mass 80-100 and 120-140 Elemental abundance patterns.
- (10) Survey of solar-terrestrial lsotopic differences.
- Noble gas Elements and Isotopes: higher energy solar particles. (11)
- (12) Li/Be/B Elemental and Isotopic abundances.
- Radioactive nuclei in the solar wind. (13)
- (14) F abundance.
- (15) **Pt-group Elemental abundances**.
- (16) Key s-process heavy Elements.
- (17) Heavy-light Element comparisons.
- Solar rare earth Elements abundance pattern. (18)
- (19) **Comparison of solar and chondritic elemental abundances.**



# **Summary**

- The bar has been raised considerably, but nothing is red even after 3 years.
- The amount of green is growing.
  - O and N isotopes.
  - Xe and Kr fluences and isotopes.
  - SIMS feasibility of Ca, Cr, Na has been demonstrated.
  - Good near term prospects for (Cr, Mn, Ni) from TRXRF and AI from RIMS/ SIMS or ICPMS. There is a significant amount of blue. We are optimistic that blue will turn to green.
- Significant progress since hitting bottom on 9/8/04, but pushing forward on a broad front.
- With some luck, the major effect will only be a delay in science.