National Aeronautics and Space Administration

Application of the Molecular Adsorber Coating technology on the Ionospheric Connection Explorer Program

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Abstract

The Molecular Adsorber Coating (MAC) is a zeolite based highly porous coating technology that was developed by NASA Goddard Space Flight Center (GSFC) to capture outgassed contaminants, such as plastics, adhesives, lubricants, silicones, epoxies, potting compounds, and other similar materials. This paper describes the use of the MAC technology to address molecular contamination concerns on NASA's Ionospheric Connection Explorer (ICON) program led by the University of California (UC) Berkeley's Space Sciences Laboratory. The sprayable paint technology was applied onto plates that were installed within the instrument cavity of ICON's Far Ultraviolet Imaging Spectrograph (FUV). However, due to the instrument's particulate sensitivity, the coating surface was vibrationally cleaned through simulated acoustics to reduce the risk of particle fall-out contamination. This paper summarizes the coating application efforts on the FUV adsorber plates, the simulated laboratory acoustic level cleaning test methods, particulation characteristics, and future plans for the MAC technology.

- ***Keywords:*** Molecular Adsorber Coating, MAC, zeolite, molecular adsorber, adsorber, adsorption, getter, outgassing, molecular contamination, particulate contamination, sprayable paint technology, coatings, spaceflight applications, Ionospheric Connection Explorer, ICON, far ultraviolet instrument, FUV, acoustic cleaning

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Introduction

- *Background*
- *Contamination Control Requirements*
- *Molecular Adsorber Coating*

Background

Ionospheric Connection Explorer (ICON)

- NASA Explorers Program
  - *Newest addition to its fleet of heliophysics satellites*
- Led by University of California (UC) Berkeley's Space Sciences Laboratory
- Launch scheduled for late 2017
  - *Launch Vehicle: Pegasus XL Rocket*

Image Credit: NASA/UC Berkeley

- Comprised of ***four main instruments***
  - **IVM:** Ion Velocity Meter
  - **MIGHTI:** Michaelson Interferometer for Global High resolution imaging of the Thermosphere and Ionosphere
  - **EUV:** Extreme Ultraviolet Spectrograph
  - **FUV:** Far Ultra Imaging Spectrograph

Background

Ionospheric Connection Explorer (ICON)

- Will investigate changes in the boundary between Earth and the space environment, and study the continuous interactions of ***space weather*** and ***Earth weather***, and the sources of ***ionospheric variations***
  - *These findings will provide a better understanding of the disturbances that are responsible for signal interferences to space based technologies, such as communication and navigation systems*

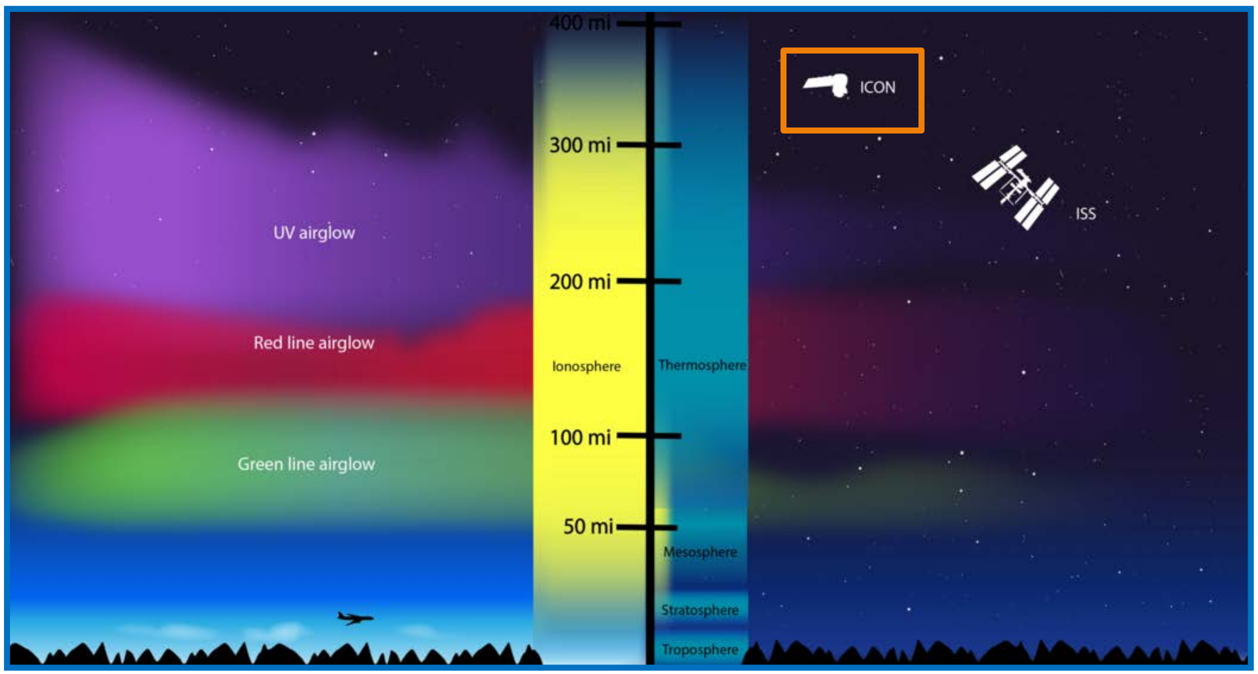


Image Credit: NASA/UC Berkeley

"NASA's ICON mission will orbit above the upper atmosphere, through the bottom edge of near-Earth space. From this vantage point, ICON will be able to observe both the upper atmosphere made of neutral particles and a layer of charged particles called the ionosphere, which extends from about 50 to 360 miles above the surface of Earth.

Processes in the ionosphere also create bright swaths of color in the sky, known as **airglow.** ICON will observe how interactions between terrestrial weather and the ionosphere create such shimmering airglow as well as other changes in the space environment."

Text Source: *http://www.nasa.gov/image-feature/goddard/2016/info-graphic-icon-and-the-edge-of-the-atmosphere*

Contamination Requirements

FUV Instrument

- Identified as one of the most sensitive to contamination of the four instruments
- Responsible for measuring the density of ionized gas in the ionosphere and imaging the upper atmosphere in the far ultraviolet range
- Effects of ***material outgassing*** within the instrument cavity posed significant challenges to meeting the molecular Cleanliness Levels (CL)
  - *Outgassing from materials, such as plastics, adhesives, lubricants, silicones, epoxies, potting compounds, and other similar materials, may result in the degradation of the instrument's performance*

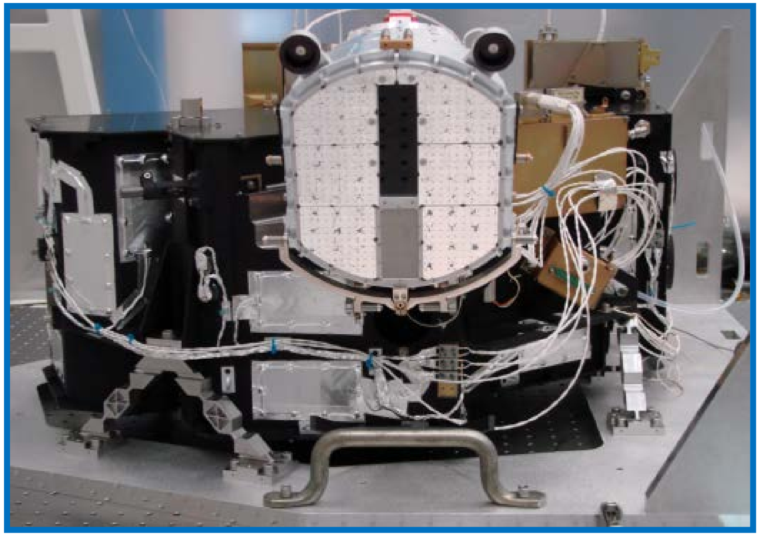


Image Credit: NASA/UC Berkeley

ICON FUV Instrument Contamination Cleanliness Level Requirements *

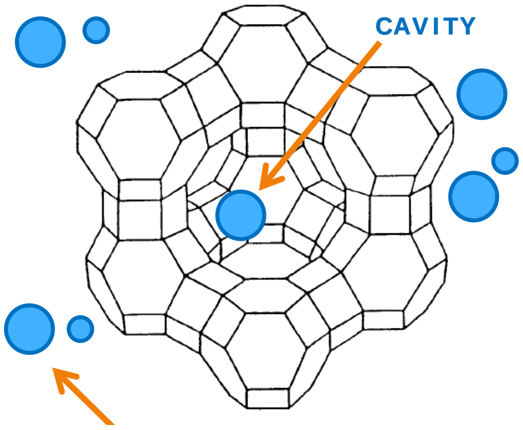
Mission Phase	Particulate CL	Molecular CL	
Pre-Integration	100	A/2	0.5 μg/cm^2
Beginning of Life (BOL)	500	A	1.0 μg/cm^2
End of Life (EOL)	500	C	3.0 μg/cm^2

* *The listed particulate and molecular CL are taken from the ICON Contamination Control Plan per IEST-STD-CC1246D*

Molecular Adsorber Coating

Molecular Adsorber Coating (MAC)

- Proposed as a mitigation method to address the FUV instrument's material outgassing concerns
- Developed by NASA Goddard Space Flight Center (GSFC)
- Sprayable, zeolite based and highly porous coating technology that was designed to ***passively capture*** outgassed contaminants
  - *Thereby, reduce the risk of on-orbit molecular contamination from degrading the performance of sensitive interior surfaces on spaceflight hardware and instruments*



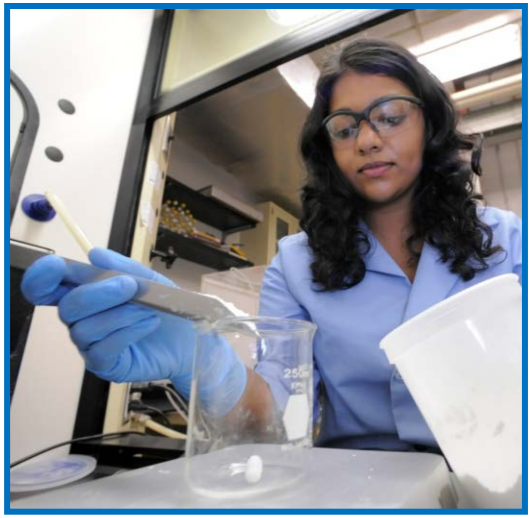


Photo Credit: NASA/Pat Izzo

- Available in both ***white*** and ***black*** coating variations
  - *White Molecular Adsorber Coating, GSFC MAC-W*
  - *Black Molecular Adsorber Coating, GSFC MAC-B*
- Through GSFC's Internal Research and Development (IRAD) program, significant testing and demonstration efforts were performed in relevant environments (i.e. vacuum) for use on spaceflight applications
  - *Testing includes: molecular capacitance, thermal/optical property, adhesion performance, thermal cycle, thermal shock, and particle fall-out tests*

Molecular Adsorber Coating

MAC's Textured Surface Morphology

- Improves ***molecular adsorption*** properties of the coating
  - *High Surface Area*
  - *Surface Roughness*
  - *Highly Porous Structure*
- To reduce the likelihood of damage that may occur due to handling, contact with the coating should be ***limited***
  - *Treat as "no touch" surface*
- Rubbing or touching the coating may damage its high surface area, and thus generate particles
- Particle generation may also occur from acoustic or vibration related spacecraft activities, such as:
  - *Integration and Testing (I&T) Phase*
  - *Launch Environment*

Confocal Imaging Microscope (CIM) Analysis of MAC at 20X Magnification

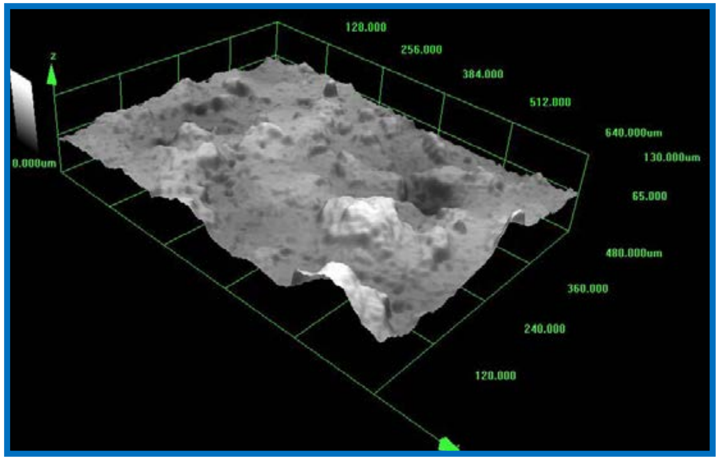


Image Credit: NASA

Scanning Electron Microscope (SEM) Analysis of MAC at 636X Magnification

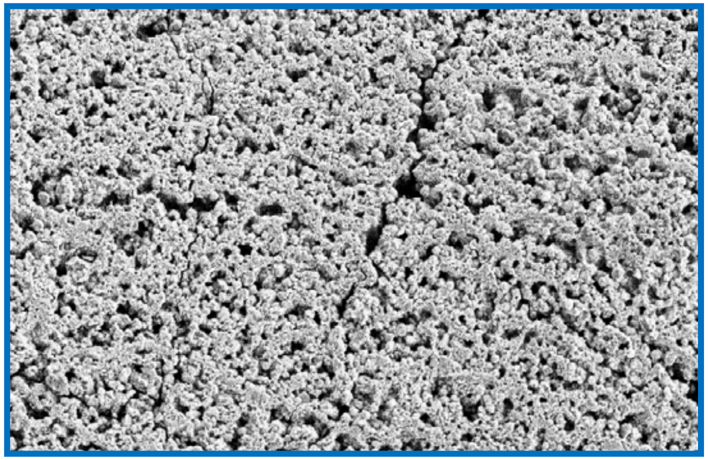


Image Credit: NASA

Molecular Adsorber Coating

CONCERN

Difficult to meet FUV's highly sensitive molecular contamination requirements due to material outgassing within instrument

SOLUTION

Implement MAC technology within instrument to help meet molecular contamination requirements

ANOTHER CONCERN

Due to FUV's particulate sensitivity, there is a need to address the potential risk of additional contamination that may occur due to particle fall-out from the coating itself

INVESTIGATION

Evaluate MAC's particulation characteristics through various trade studies

SOLUTION

Mitigate risk by vibrationally cleaning the coating surface through simulated acoustic methods prior to spaceflight to help meet the mission specific particulate cleanliness levels

Flight Application Efforts

- *Coating Application*
- *Coating Thickness*
- *Molecular Adsorption Capacity*
- *Thermal Properties*
- *Adhesion Performance*
- *Molecular Outgassing*

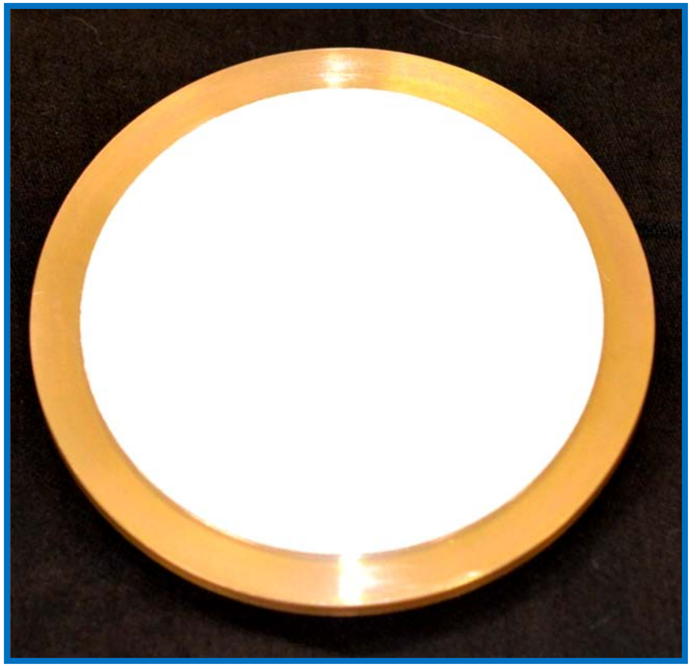
Coating Application

- ***Fabrication:*** UC Berkeley fabricated six FUV adsorber plates (3 in diameter)

Location	Quantity
Spectrograph Bench	2
Image Bench	1
Flight Spares	3
Total	**6**

Image Credit: NASA

- ***Design Features:***
  - Helical coil insert on back side for easy installation within the instrument cavity
  - Rigid substrate to limit hardware flexure



- ***Application:*** NASA GSFC applied MAC to adsorber plates on July 2015
  - An uncoated 0.25 in border was strategically implemented to reduce coating damage during handling and installation activities

Coating Thickness

- On average, the coated FUV adsorber plates measured a total coating thickness of 5.5 mils
  - *Thickness measurement includes both the applied primer and the multiple MAC layers*

Summary of Coating Thickness

Flight Hardware Sample ID	Average Coating Thickness *(mils)*
MAC-FUV-A1	6.1
MAC-FUV-A2	5.7
MAC-FUV-A3	5.5
MAC-FUV-A4	4.8
MAC-FUV-A5	5.3
MAC-FUV-A6	5.5
Total Average Coating Thickness	**5.5**

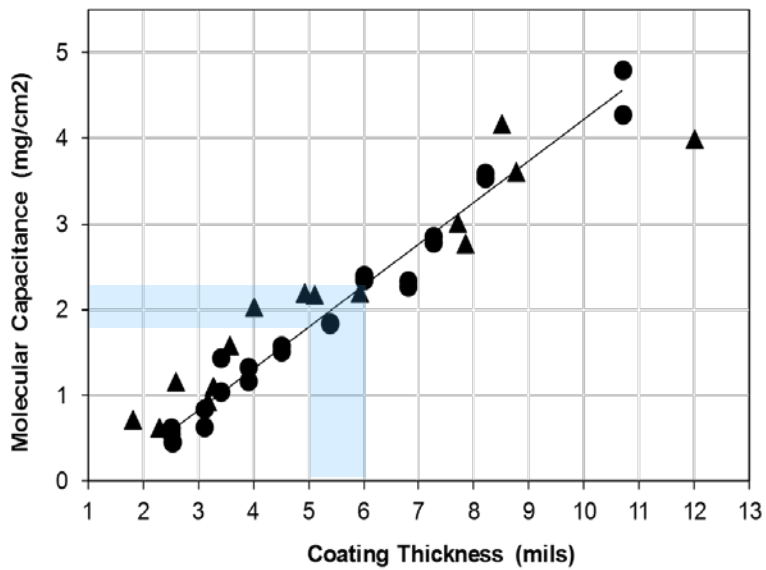
Coating thickness on the flight hardware was evaluated using the Vector TX1 thickness gage manufactured by NDT Instruments. The instrument uses an eddy current to measure the thickness of an applied coating on a metal substrate. Measurements were calibrated with standards of known thicknesses.

Molecular Adsorption Capacity

- Also referred to as "molecular capacitance"
- Defined as the measure of the coating's capability to ***adsorb or entrap outgassed materials*** (i.e. molecular contaminants)
- Experimental data has shown that the molecular adsorption capacity of the MAC technology is directly proportional to total coating thickness
  - The test data used **stearyl alcohol** at 45 °C as a model contaminant source at exposures between 88 and 160 hours

Stearyl alcohol is an 18 chain hydrocarbon contaminant that is representative of the commonly outgassed materials found in spaceflight applications

Effect of Coating Thickness on MAC Adsorption Capabilities

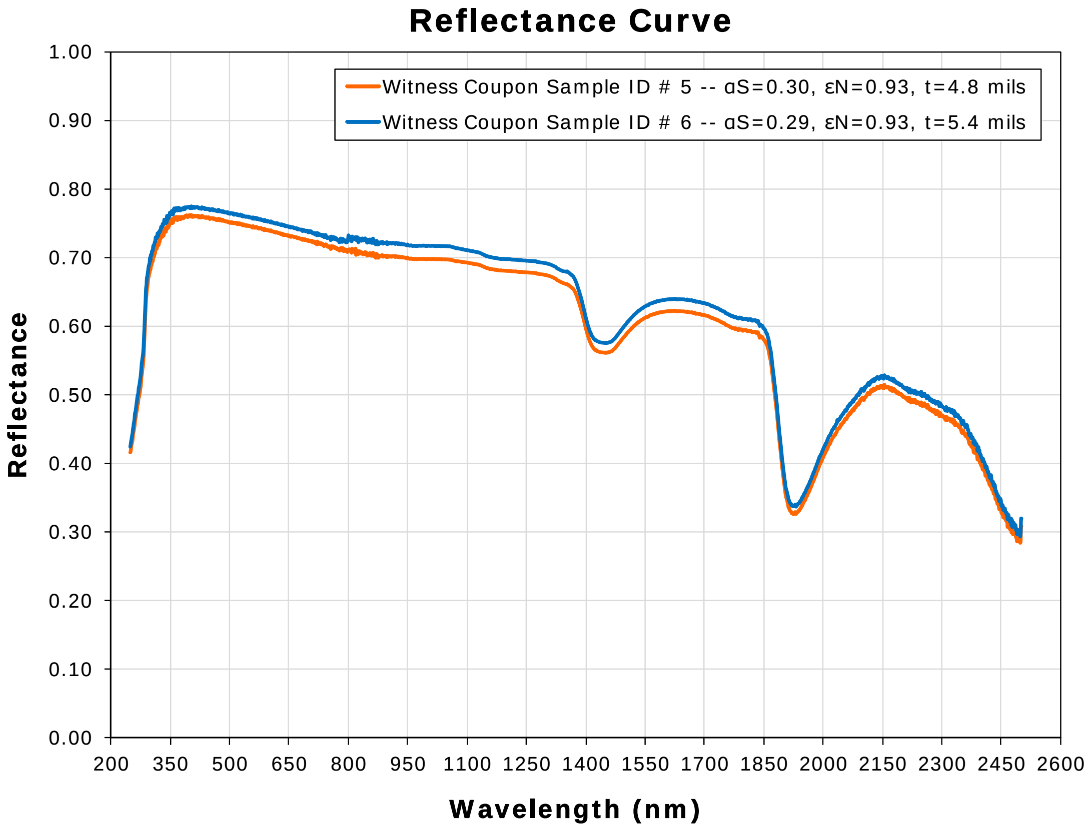


- Based on this set of data, the molecular adsorption capacity for white MAC between 5 to 6 mils thick is approximately ~2 mg/cm^2

Estimated Molecular Adsorption Capacity Based on Experimental Data

Coating Diameter per FUV Adsorber Plate	Coating Area per FUV Adsorber Plate	Molecular Capacitance for 5 – 6 mils MAC	Estimated Adsorption per FUV Adsorber Plate
6.35 *cm*	31.7 cm^2	~2 mg/cm^2	~63 *mg*

Thermal Properties



- MAC will <u>not</u> be used for thermal control purposes within the FUV instrument
  - *However, for reference purposes, thermal property measurements were evaluated on two 2 in by 2 in aluminum witness coupons that were sprayed alongside the FUV flight hardware with white MAC*

Solar Absorptance: Calculated using the Varian Cary 5000 spectral reflectometer, which measures the reflectance from 250 to 2500 nm at an 8° angle of incidence using the ASTM E903-82 standard test method with a measurement accuracy of ±0.02.

Normal Emittance: Calculated using the Gier-Dünkle DB-100 infrared reflectometer, which measures the normal emittance from 5 to 40 µm at room temperature using the ASTM E408-71 standard test method with a measurement accuracy of ±0.02.

Summary of Thermal Property Measurements

Witness Coupon Sample ID	Solar Absorptance (α_S)	Normal Emittance (ε_N)	Total Coating Thickness (*mils*)
Sample # 5	0.30	0.93	4.8
Sample # 6	0.29	0.93	5.4
Average	**0.29**	**0.93**	**5.1**

Adhesion Performance

- Two 2 in by 2 in witness coupons were used to perform adhesion performance tape tests per Test Method A of ASTM D3359-09
  - Test results validate the **structural integrity** of the coating and the good workmanship process for the coated flight parts

Summary of Adhesion Performance Results

Witness Coupon Sample ID	Average Total Coating Thickness *(mils)*	ASTM D3359-09 Adhesion Rating	NASA GSFC Adhesion Performance Criteria
Sample # 1	5.3	4A	Pass
Sample # 3	5.0	4A	Pass

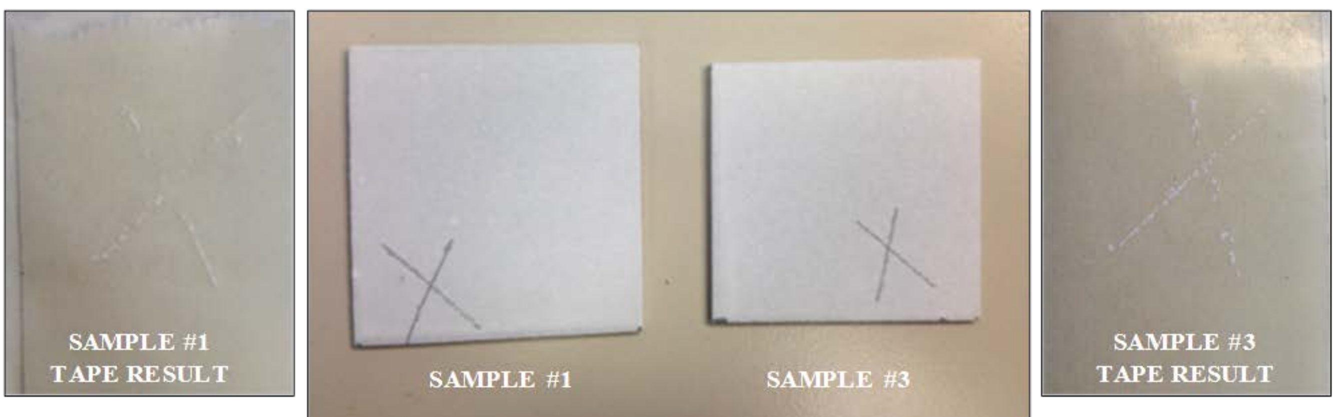


Image Credit: NASA

Material Outgassing

- ASTM E-595 test method was performed to confirm that the coating meets spaceflight material outgassing criteria in a vacuum environment

Typical Screening Criteria for Spaceflight Materials

- *Collected Volatile Condensable Material (CVCM) ≤ 0.10 %*
- *Total Mass Loss (TML) ≤ 1.0 %*

Material Outgassing Properties of White MAC

TML	WVR *at 50 % RH*	CVCM
11.19 %	11.03 %	0.01 %

- MAC exhibits the following properties due its chemical composition which is comprised of inorganic materials, such as zeolite
  - **Low outgassing** properties in vacuum conditions
    - *CVCM is 0.01 %*
  - **Hygroscopic** properties at ambient conditions
    - *Water Vapor Release (WVR) at 50 % Relative Humidity (RH) is 11.03 %*

    The relatively high TML is a result of water moisture loss in a vacuum environment, and not due to material outgassing!

Simulated Acoustic Cleaning Efforts

- *Simulated Acoustic Cleaning*
- *Test Configuration*
- *Test Characterization*
- *Test Method*
- *Test Results*
- *Post Testing*

Simulated Acoustic Cleaning

Launch Environment

- During launch, a spacecraft is exposed to ***extreme acoustic conditions*** that produce high levels of vibrations
  - Consequently, it is important for the payload structure and its components to endure such an intense environment

Acoustic Test Chamber

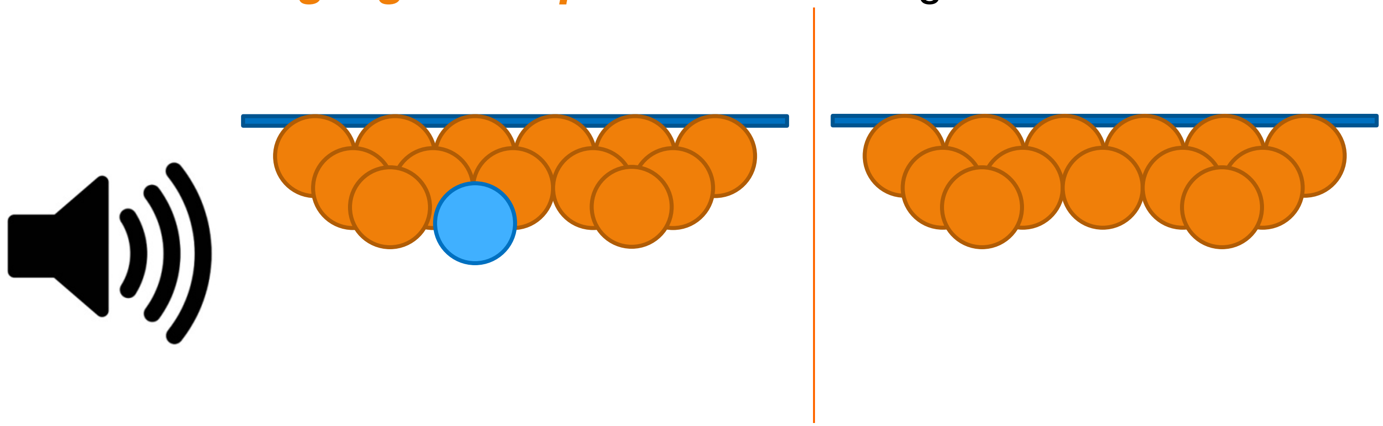
- At NASA GSFC, these acoustic induced vibrations are simulated in a 42 foot tall acoustic test chamber for ground testing purposes
  - Houses 6 foot wide horns that generate acoustic noise as high as 150 decibels
  - Sound produced by alternating flow of gaseous nitrogen
  - Payload exposed to this "simulated" acoustic environment for typically two minutes during testing

Image Credit: NASA

Simulated Acoustic Cleaning

Simulated Acoustic Cleaning Test

- Designed a simulated laboratory level acoustic cleaning test
  - *To mitigate risk of particle fall-out from coating due acoustic level vibrations related to spacecraft activities and launch environments*
  - *Similar concept to NASA's acoustic test chamber used for ground testing*
- In industry, acoustic cleaning is a common maintenance practice that involves ***dislodging solid particles*** through sound transmission



- Similarly, this acoustic cleaning method will be used to ***vibrationally shake*** or ***remove particles loose*** from the coating's textured surface that may at a later time come off due to vibroacoustic impacts of a launch environment

Simulated Acoustic Cleaning

Trade Study

- In 2014, NASA GSFC conducted simulated acoustic cleaning trade studies on flat aluminum substrates coated with MAC
- ***Purpose:***
  - To evaluate particulation characteristics of MAC due to acoustic induced vibrations
- ***Results:***
  - Demonstrated correlation between coating thickness and particulate shedding effects
  - Showed significant reduction of particle fall out when coating is acoustically cleaned or experiences a ***simulated "shake"***

Image Credit: NASA

- This simulated acoustic cleaning technique will be performed on ICON's flight hardware prior to spaceflight use to help meet the mission's particulate cleanliness levels, and considerably ***reduce the threat of excess particulate contamination*** within the FUV instrument

Test Configuration

Acoustic Cleaning Test Configuration

- Laboratory bench scale simulation of vibration forces that are applied in the NASA GSFC acoustic test chamber

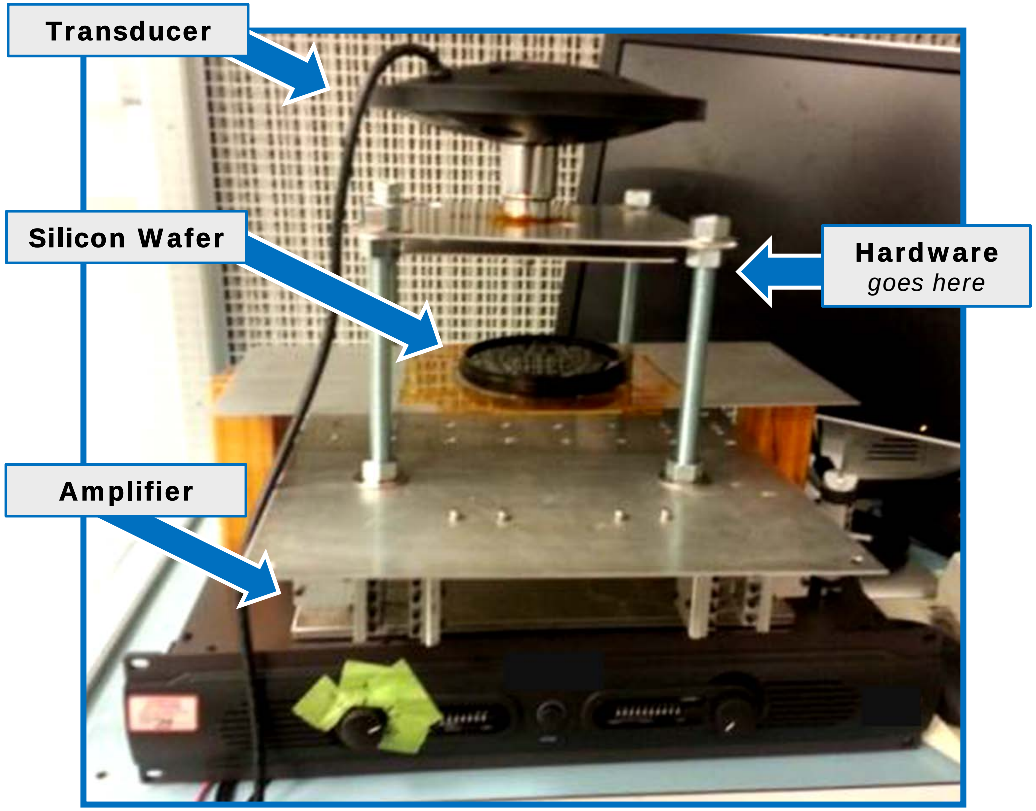


Image Credit: NASA

COMPONENT FLOW DIAGRAM

1. Brown Noise Audio File
- *From connected computer*
- *Provides input signal*

2. Audio Power Amplifier
- *Increases power of audio to produce speaker level noise*
- *Provides output signal*

3. Tactile Sound Transducer
- *Transfers energy of output signal*
- *Capable of transmitting low frequency vibrations onto surfaces*
- *Mechanically transfers acoustic energy to mounted hardware*

4. Coated Flight Hardware
- *Experiences the transferred acoustic energy's vibrations and shaking*

5. Silicon Wafer
- *Collects loose particles removed due to these vibrations/shaking*
- *Placed below hardware (where coating is facing wafer)*

Test Characterization

Amplifier Settings

- Determine appropriate amp setting that produces levels similar to flight conditions
  - Expected overall random vibration response level for the FUV instrument is **~14 G_{rms}**
- Uncoated non-flight FUV adsorber plates
  - Mounted to the test apparatus using a custom designed adaptor fitting attachment, which screws into the helical coil insert on the backside of the hardware
  - Two accelerometers were attached to the hardware on two opposite sides
  - Subjected to various amp settings to evaluate acoustic induced vibration response via accelerometer signals
  - Signal analyzer was used to record responses of acoustic cleaning activity on the hardware at various amp settings

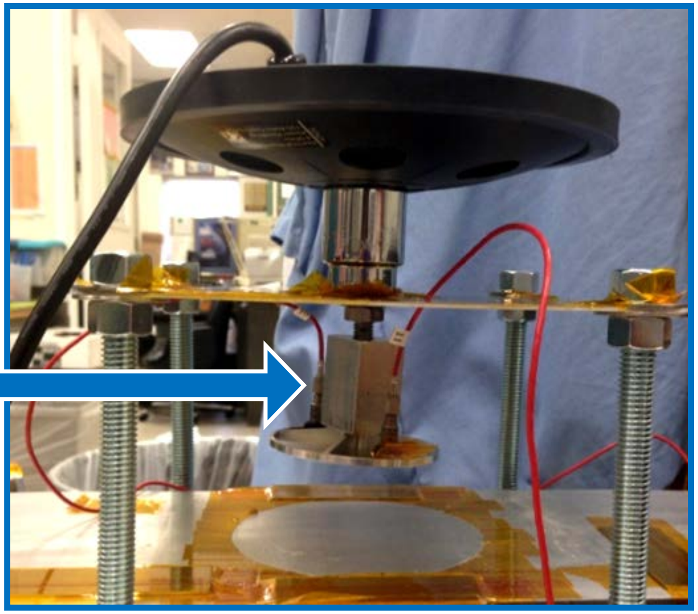
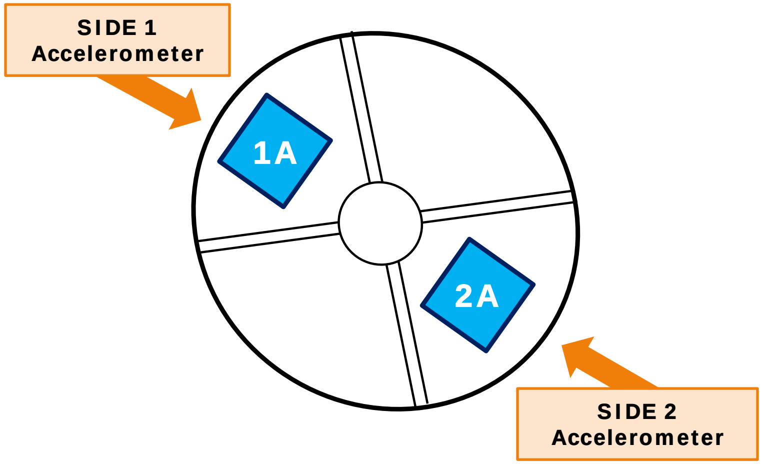
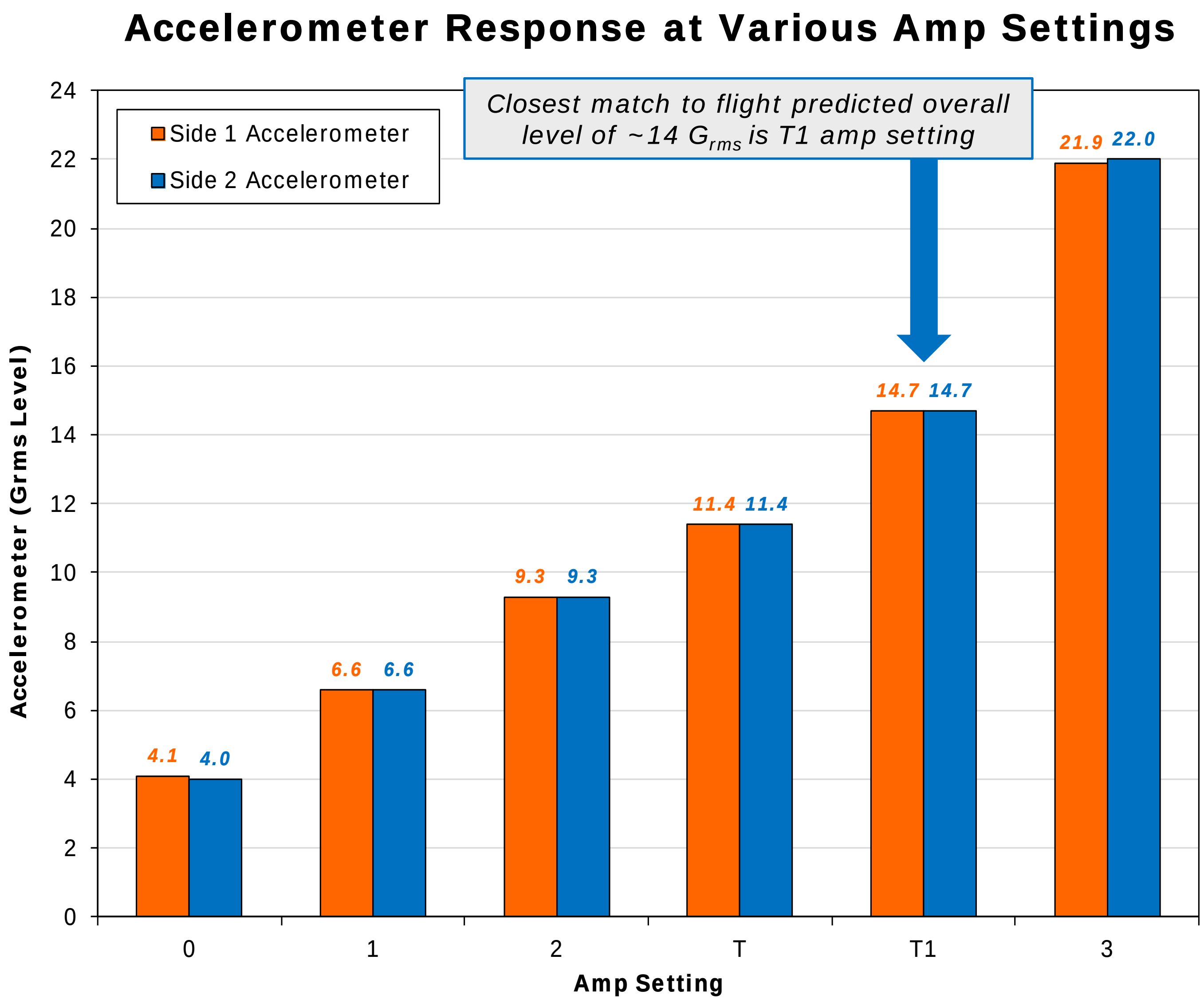


Image Credit: NASA



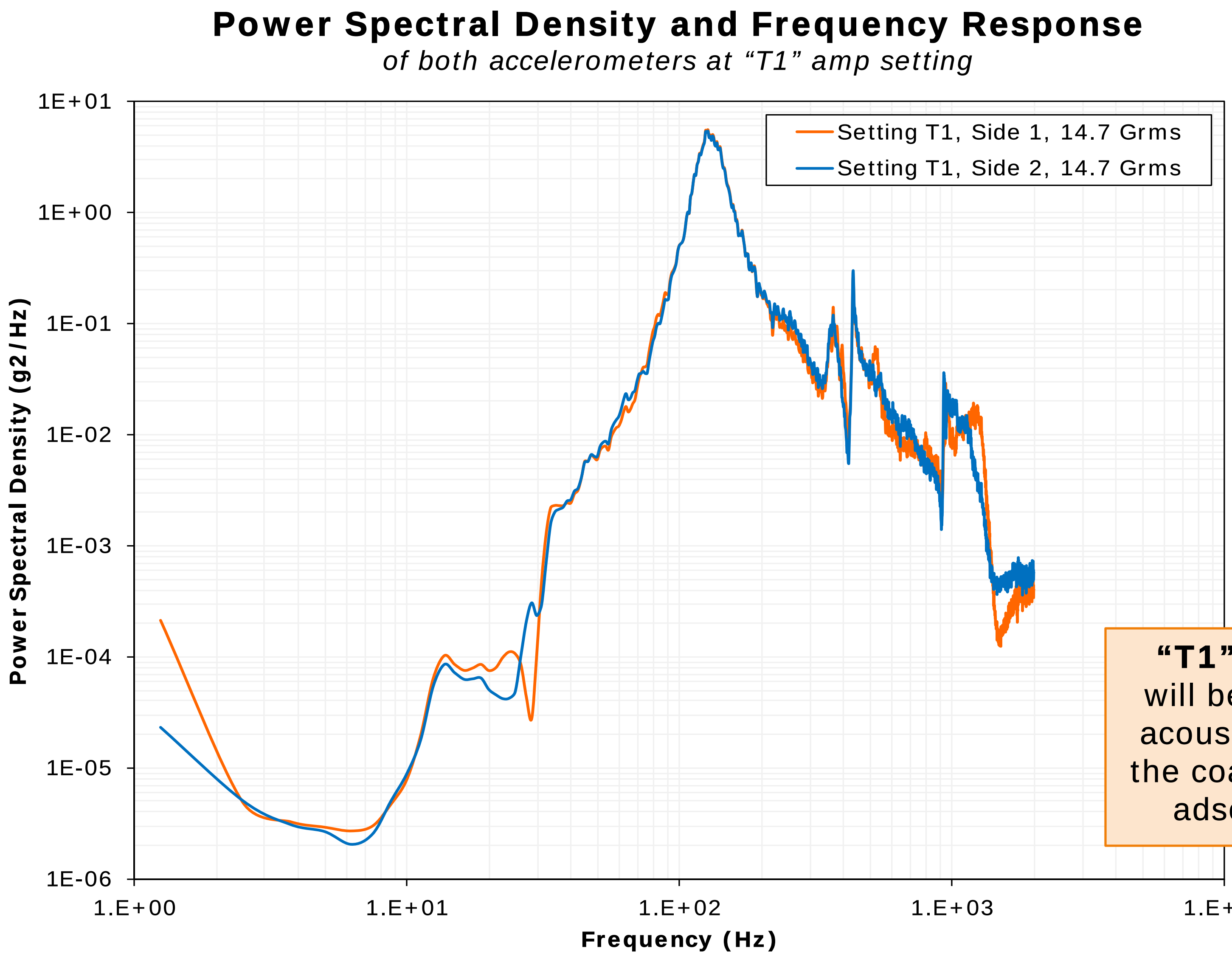
Test Characterization



Both accelerometers showed the same G_{rms} response for each different setting.

This indicates that the mounted hardware is subjected to ***even distribution*** of the acoustic induced vibrations.

Test Characterization



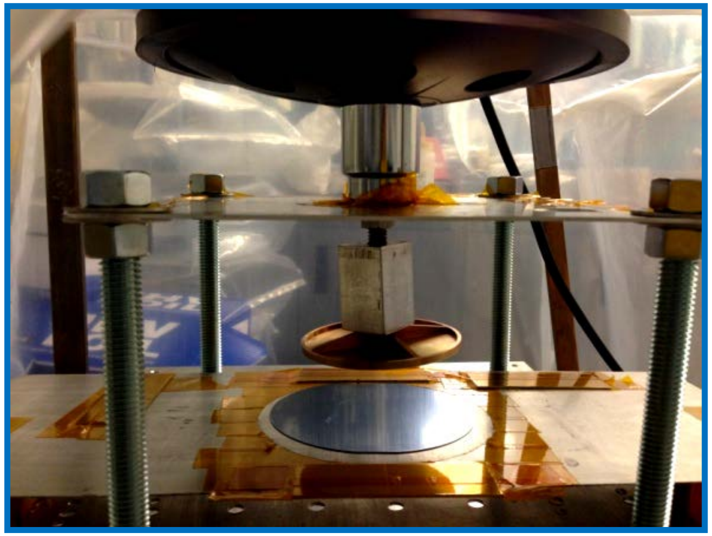
"T1" amp setting will be used for the acoustic cleaning of the coated flight FUV adsorber plates

Test Method

- Silicon wafers are scanned using an automated ***Image Analysis (IA)*** system comprised of a microscope and analyzer software
  - *IA scan provides particle count data, which is used to determine:*
    - **Percent Area Coverage (PAC)**
      - Based on total area covered by particles over total area scanned by IA microscope
    - **Cleanliness Level (CL)**
      - Based on surface particle obscuration

        Based on empirical relations from the paper "Surface particle obscuration and BRDF predictions" by Ma, Fong, and Lee. CL empirical relations represent spherical shaped particles and cylindrical-hemispherical shaped particles.
- Coated flight hardware is securely mounted on test apparatus underneath transducer
  - *Coated side of hardware is facing down towards where the silicon wafer will be placed on the test apparatus*

Image Credit: NASA



Test Method

- Summary of test method used to perform a simulated acoustic cleaning:

BASELINE RUN

- ***Step 1:*** Perform IA scan of clean wafer as a background reference
- ***Step 2:*** Mount coated hardware to test set-up
- ***Step 3:*** Place scanned clean wafer under flight hardware

RUN 1

- ***Step 4:*** Run amplifier setting at "T1" for 2 minutes
- ***Step 5:*** Remove and transfer contaminated wafer to IA microscope
- ***Step 6:*** Perform scan of particle fall-out on wafer
- ***Step 7:*** Return same wafer under hardware

RUN 2

- ***Step 8:*** Run amplifier setting at "T1" for 2 minutes
- ***Step 9:*** Remove and transfer contaminated wafer to IA microscope
- ***Step 10:*** Perform scan of particle fall-out again on same wafer
- ***Step 11:*** Remove coated hardware from test set-up

Test Results

- Performed a total of six simulated acoustic cleaning tests
  - ***One wafer*** was used for three runs per coated FUV adsorber plate
    - *Three runs include:*

  - Coated flight hardware were designated as A1 through A6

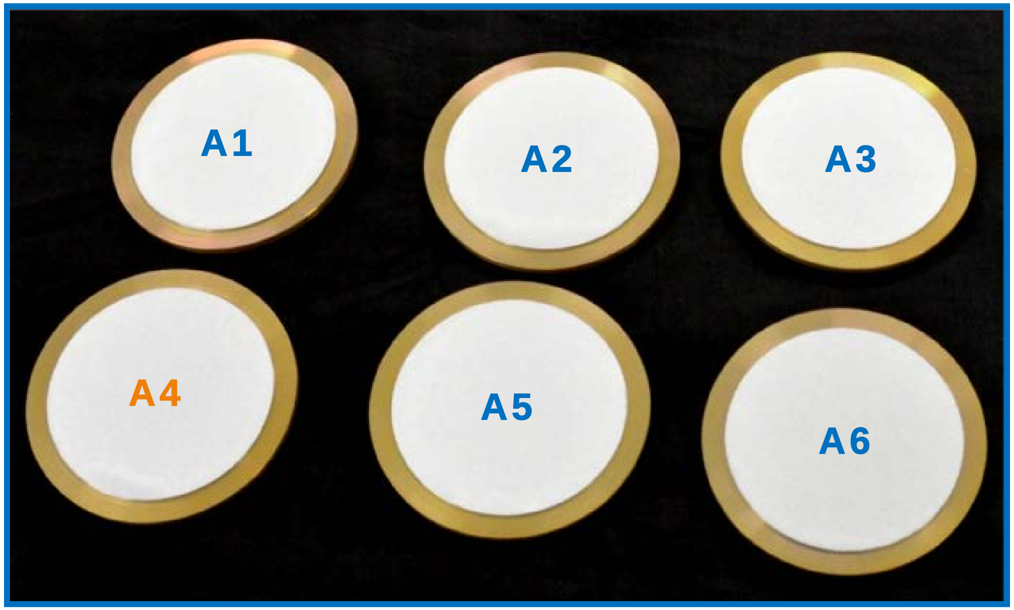


Image Credit: NASA

- Test efforts for **A4** experienced particulate contamination from the supplied wafer case
  - *This error resulted in an inconclusive analysis of data*
  - *As a result, fall-out data for A4 was removed from the findings presented here*

Test Results

Percent Area Coverage (PAC)

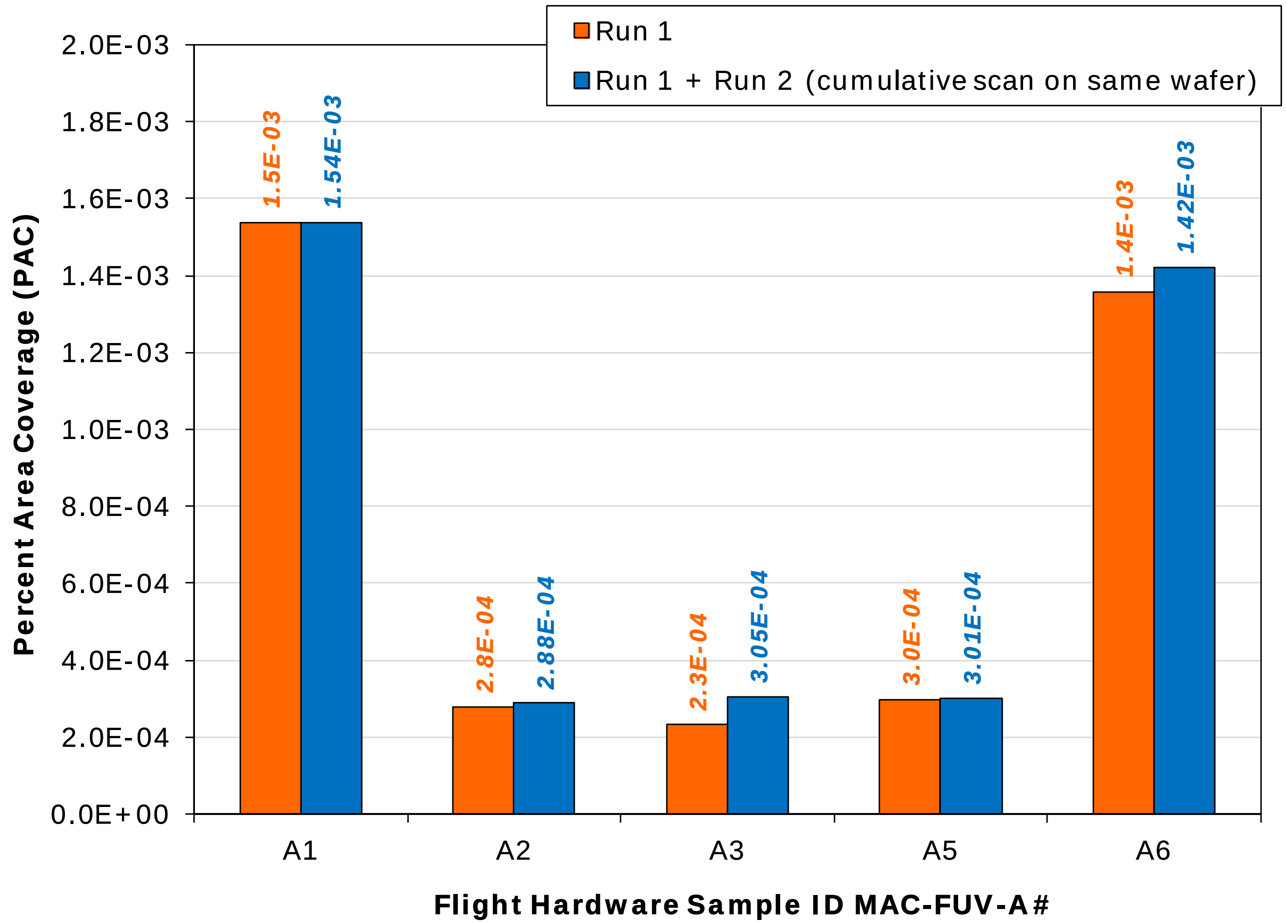
- Based on total area covered by particles over total area scanned by IA microscope

PAC = $\frac{\text{Particle Obscured Area}}{\text{Wafer Scan Area}} * 100$

RUN 1

One Acoustic "Shake"

- Reflects the amount of loose MAC particles that were removed during the initial excitation experienced from the first simulated acoustic vibrations
- Initial PAC varies per sample due to the variable surface texture of the applied coating



Test Results

Percent Area Coverage (PAC)

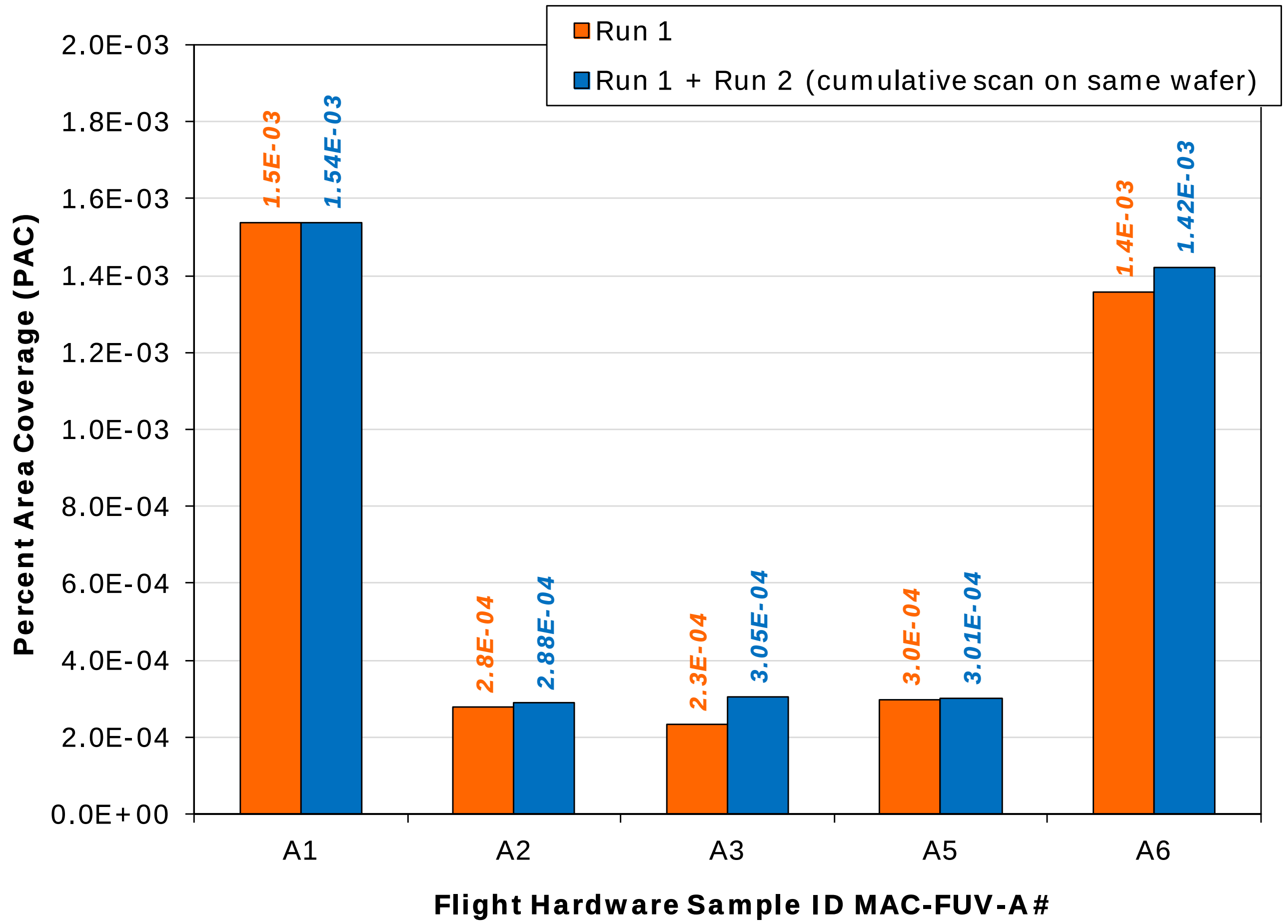
- Based on total area covered by particles over total area scanned by IA microscope

$$PAC = \frac{\text{Particle Obscured Area}}{\text{Wafer Scan Area}} * 100$$

RUN 1 + RUN 2

Two Acoustic "Shakes"

- Reflects the cumulative PAC from the two simulated acoustic vibrations
- Cumulative PAC remained about the same as Run 1 PAC
- Suggests that no additional particles (or very little in some cases) were additional removed from the coating surface during the second run



Test Results

Percent Area Coverage (PAC)

- *Based on total area covered by particles over total area scanned by IA microscope*

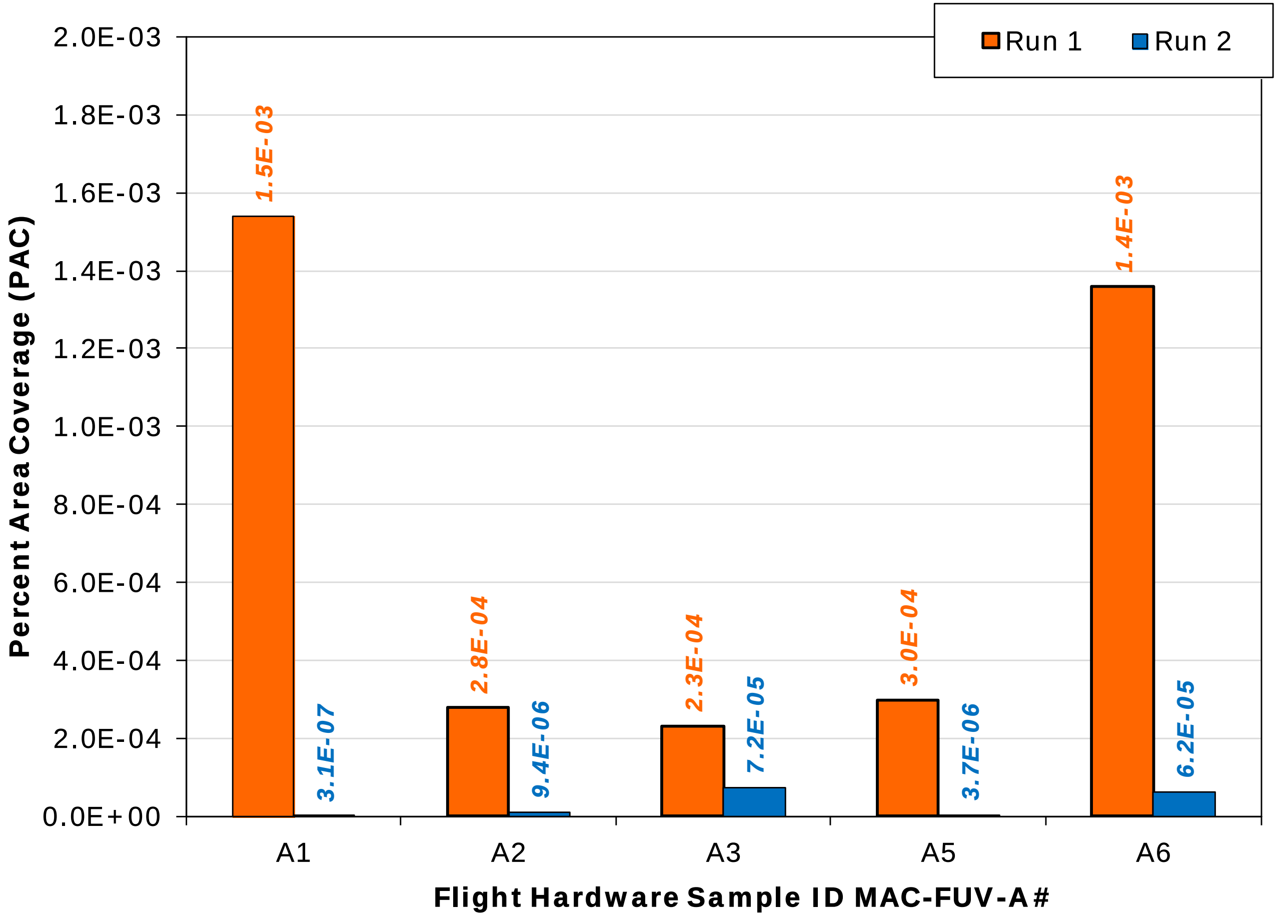
$$PAC = \frac{\text{Particle Obscured Area}}{\text{Wafer Scan Area}} * 100$$

RUN 2

Two Acoustic "Shakes"

- Reflects the particle fall-out from the second simulated acoustic vibration only
- Shows at least 95 percent particulate fall-out reduction
  - *Outlier: A3*

Flight Hardware Sample ID	Particulate Fall-out Reduction
A1	100 %
A2	97 %
A3	*69 %*
A5	99 %
A6	95 %



Test Results

Percent Area Coverage (PAC)

- *Based on total area covered by particles over total area scanned by IA microscope*

  - Trend suggests that after an initial exposure to "simulated" acoustic effects, the likelihood of the coating resulting in additional particle fall-out due to vibroacoustic anomalies is ***very minimal***
  - Majority of the loose particles were removed during the first "shake" event
  - These results reduce the concern for further particulate contamination

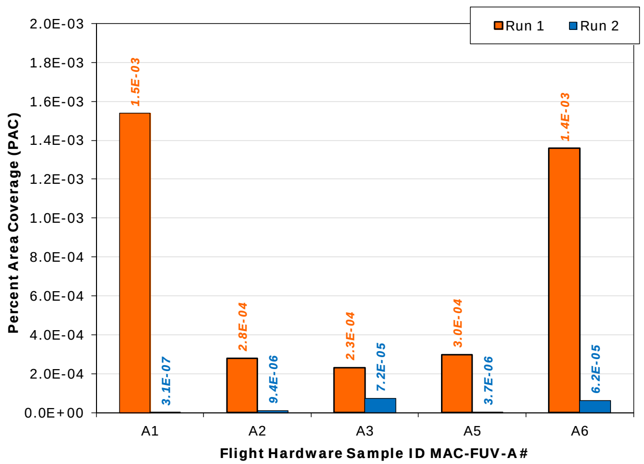


Image Credit: NASA

Test Results

Cleanliness Level (CL)

- Based on empirical relations for surface particle obscuration from the paper "*Surface particle obscuration and BRDF predictions*" by Ma, Fong, & Lee (1989)
  - CL remained about the same after completing the second run
  - This again suggests that a ***significant reduction*** of particle fall-out from the coating occurred as a result of the second acoustic induced vibrations on the hardware

CL Assumptions

SPHERICAL SHAPED PARTICLES

CYLINDRICAL HEMISPHERICAL SHAPED PARTICLES

	Cleanliness Level (sphere)		Cleanliness Level (cylinder)	
Flight Hardware Sample ID	**Run 1** *1 Acoustic Shake*	**Run 1 + Run 2** *2 Acoustic Shakes*	**Run 1** *1 Acoustic Shake*	**Run 1 + Run 2** *2 Acoustic Shakes*
A1	154	154	165	165
A2	99	100	107	108
A3	94	102	102	109
A5	101	101	109	109
A6	149	151	160	162

Run 1 + Run 2 reports a cumulative scan of both runs on the same silicon wafer

Post Testing

Prepare for Shipment

- The acoustically cleaned MAC coated flight hardware was securely packaged in shipping containers that were provided by UC Berkley
  - *Shipping containers were custom designed to* **limit contact** *with the MAC surface; and thereby reduce particle generation during transportation activities*
- Special handing instructions for the ***"no touch"*** MAC surfaces were supplied as precaution to further reduce the risk of particulation

Arrival at UC Berkeley

- Inspection upon arrival showed almost no additional particles due to shipping and handling related activities
- Three of the six flight plates were installed inside the continuously purged FUV instrument cavity prior to instrument Thermal Vacuum (TVAC) testing
  - *As planned, the remaining three FUV adsorber plates were kept as flight spares*
- Post vibration inspection reported no significant particulation related anomalies within the FUV cavity

Conclusions

- *Future Work*
- *Acknowledgements*
- *References*
- *Contact Information*

Future Plans

- Further investigations are expected to better understand particle fall-out characteristics of MAC
- Future plans include:
  - Exploring improved (or alternative) methods of mitigating coating fall-out due to acoustic induced vibrations
    - **For example, on larger spaceflight hardware with particulate sensitivity requirements**
  - Evaluating other parameters that may introduce additional stresses within the coating structure that may contribute to particle fall-out
    - **For example, these parameters may include but are not limited to:**
      - Thermal Cycling
      - Vacuum Exposure
      - Shock Events
      - Extended TVAC Bake-Outs
      - Substrate Flexures

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