Black Molecular Adsorber Coatings for Spaceflight Applications

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OVERVIEW

■ Introduction
  ■ Molecular Outgassing, Molecular Adsorber Coating, Stray Light, Black Thermal Control Coatings, Black Molecular Adsorber Coating

■ Approach
  ■ Three Stage Test Plan

■ Development
  ■ Black Pigments, Adhesion Performance

■ Characterization
  ■ Thermal and Optical Property, Surface Morphology

■ Testing
  ■ Thermal Survivability and Vacuum Stability, Molecular Capacitance

■ Conclusions
  ■ Accomplishments, Future Work, Acknowledgements
INTRODUCTION

What are Molecular Adsorber Coatings and why do we need them for spaceflight applications?
INTRODUCTION

- **Molecular Outgassing**

  - *For spaceflight applications:*

  - Significant threat to the spacecraft and the lifetime of NASA missions

  - Originates from materials that outgas or release molecules during orbit inside of the spacecraft

    - **Examples:** plastics, adhesives, lubricants, epoxies, tapes, potting compounds, solvents, and other similar sources

  - On-orbit molecular contaminants from outgassed materials can deposit onto hardware and instrument components, and thereby, degrade the performance of highly sensitive surfaces

    - **Examples:** optics, electronics, laser systems, detectors, solar arrays, and thermal control coatings
INTRODUCTION

- Molecular Adsorber Coating (MAC)
  - Developed by NASA Goddard Space Flight Center (GSFC) as a practical *low mass* and *cost effective* solution to address on-orbit molecular contamination from outgassing.
  - Sprayable, novel paint technology comprised of processed *white pigments* made from:
    - *Highly permeable, porous zeolite minerals*
    - *Inorganic colloidal silica based binders*
  - Successfully demonstrated its technology in relevant space environments.
  - Ready for infusion into spaceflight projects and commercial markets that need to protect surfaces against the damaging effects of molecular contamination.
  - Other applications include reducing pressures inside cavities with high voltage electronics, or in vacuum chambers for thermal bake-outs.
INTRODUCTION

PROBLEM

- **Stray light**

**Stray light** is responsible for:

- Decrease in contrast and image quality
- Increase of background noise for optical systems

**Low reflectivity** properties

- Desired for some spaceflight applications that require specific *stray light control* in the near infrared range and possibly, deeper into mid to far infrared for cryogenic applications

SOLUTION

- **Black Thermal Control Coatings**

Typically used to *reduce stray light* and meet specific thermal control properties

- **Examples**: Polyurethane Coatings, such as Aeroglaze® Z306 and Z307

Applied on interior surfaces

- **Examples**: instrument baffles, detectors, and high voltage electronics boxes

Used within light paths between sensitive optical cavities to *absorb light*

- **Examples**: cameras, telescopes, mirrors, and laser systems
INTRODUCTION

- Black Molecular Adsorber Coating (MAC)
  - Unfortunately, the existing MAC is **NOT SUITABLE** for these spaceflight applications due to its **white color**
  - Therefore, a **black version** of MAC was developed:
    - To provide similar **adsorptive capabilities**
    - To provide **low reflectivity** for thermal control properties
    - To reduce the effects of **optical path degradation**

<table>
<thead>
<tr>
<th></th>
<th>BLACK THERMAL CONTROL COATING</th>
<th>WHITE MAC</th>
<th>BLACK MAC</th>
</tr>
</thead>
<tbody>
<tr>
<td>Molecular Contamination Control</td>
<td>NO</td>
<td>YES</td>
<td>YES</td>
</tr>
<tr>
<td>Low Reflectivity Thermal Control</td>
<td>YES</td>
<td>NO</td>
<td>YES</td>
</tr>
<tr>
<td>Optical Stray Light Control</td>
<td>YES</td>
<td>NO</td>
<td>YES</td>
</tr>
</tbody>
</table>
APPROACH

What is the primary objective and proposed test plan for this study?
### APPROACH

#### Primary Objective
- Transform the existing White MAC into a black coating with similar adsorptive properties

#### Three Stage Test Plan
- Evaluate the performance of the developed Black MAC formulas under ground handling environments and simulated spaceflight conditions
- Compare the performance against the current White MAC

<table>
<thead>
<tr>
<th>STAGE 1: DEVELOPMENT</th>
<th>STAGE 2: CHARACTERIZATION</th>
<th>STAGE 3: TESTING</th>
</tr>
</thead>
<tbody>
<tr>
<td>Modify the current formula by incorporating black pigments</td>
<td>Characterize the thermal and optical properties of the new coatings</td>
<td>Evaluate the thermal survivability and vacuum stability of the new coatings</td>
</tr>
<tr>
<td>Evaluate the changes in adhesion performance of the new coating formulas</td>
<td>Characterize the surface morphology with microscopic imaging techniques</td>
<td>Determine the molecular capacitance of the new coatings</td>
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</table>
DEVELOPMENT

*Stage 1:* How was the coating formulation modified and how did these changes affect the adhesion performance?
DEVELOPMENT

Black Pigments

- Modified the current formula by incorporating black pigments
- Explored several parameters to optimize adhesion performance and compatibility of coating materials

<table>
<thead>
<tr>
<th>PIGMENT IDENTIFICATION</th>
<th>FORMULA VARIATIONS</th>
<th>SAMPLE SUBSTRATES</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-MH</td>
<td>3</td>
<td>9</td>
</tr>
<tr>
<td>2-ML</td>
<td>12</td>
<td>36</td>
</tr>
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<td>3-CV</td>
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<td>18</td>
</tr>
<tr>
<td>4-CB</td>
<td>3</td>
<td>9</td>
</tr>
<tr>
<td>5-CR</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Totals</strong></td>
<td><strong>24</strong></td>
<td><strong>72</strong></td>
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- **Type of Pigments** included commercially available inorganic black powders, such as specialty grade carbon black, bone charcoal, or carbon infused silica type materials
  - Avoided use of *polymeric* based black pigments due to possibility of outgassing or blocking available adsorption pore sites

- **Amount of Pigment** varied between 40 and 80 percent

- **Binder to Pigment Ratio** varied between 0.6 and 4.8

- **Methods of Pigment Processing Techniques** from previous studies were executed
**Adhesion Performance**

- Measured in accordance with ASTM* D3359-02 (Method A) Adhesion Tape Test
  - Evaluated for any signs of delamination or separation of coating
  - Some factors affecting adhesion include formulation, thickness, and spray application method

- Several of the black pigment infused formulas exhibited cohesive failures
  - Failures occurred on the top layer rather than at the primer/substrate interface, suggesting slightly weak cohesive bonding properties between the top and underlying coating layer
  - Consequently, this made it difficult to appropriately determine adhesive bonding strength at the coating to primer/substrate interface

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*American Society for Testing and Materials (ASTM)*
**DEVELOPMENT**

### Adhesion Performance

- **Pigment:** 2-ML  
  - Demonstrated most promising adhesion results

- **Formula:** BLACK MAC 2-ML-H75  
  - Selected as potential candidate for characterization and testing purposes

Although results demonstrated good adhesion, additional optimization of selected coating may be required in the future to advance its adhesion performance.

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CHARACTERIZATION

**Stage 2:** What are the thermal/optical properties and surface morphology of black molecular adsorber coatings and how does it compare to its white counterpart?
## Characterization

### Thermal and Optical Properties

<table>
<thead>
<tr>
<th>SOLAR ABSORPTANCE (α)</th>
<th>NORMAL EMITTANCE (εN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>• The measure of the proportion of solar radiation the coating absorbs</td>
<td>• The measure of the relative ability of the coating to radiate absorbed radiation</td>
</tr>
<tr>
<td>• <em>Instrument:</em> AZ Technology LPSR-300 Spectral Reflectometer</td>
<td>• <em>Instrument:</em> Gier-Dünkle DB-100 Infrared Reflectometer</td>
</tr>
<tr>
<td>• Reflectance measured from 0.25 to 2.8 microns at a 15° angle of incidence (ASTM E903-82)</td>
<td>• Reflectance measured from 5 to 40 microns at room temperature (ASTM E408-71)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>COATING TYPE</th>
<th>COATING DESCRIPTION</th>
<th>COATING THICKNESS</th>
<th>SOLAR ABSORPTANCE</th>
<th>NORMAL EMITTANCE</th>
</tr>
</thead>
<tbody>
<tr>
<td>White Silicate</td>
<td>Z-93P</td>
<td>4.0-5.0 mils</td>
<td>0.16</td>
<td>0.92</td>
</tr>
<tr>
<td>White MAC</td>
<td>B-TRL6</td>
<td>4.0-5.0 mils</td>
<td>0.30</td>
<td>0.93</td>
</tr>
<tr>
<td>Black MAC</td>
<td>2-ML-H75</td>
<td>2.5-8.5 mils</td>
<td>0.97</td>
<td>0.92</td>
</tr>
<tr>
<td>Black Polyurethane</td>
<td>Aeroglaze® Z306</td>
<td>2.0-3.0 mils</td>
<td>0.96</td>
<td>0.91</td>
</tr>
<tr>
<td>Black Polyurethane</td>
<td>Aeroglaze® Z307</td>
<td>2.0-3.0 mils</td>
<td>0.97</td>
<td>0.88</td>
</tr>
</tbody>
</table>
Reflectance Curve for Black MAC at a thickness of 2.5 mils

- Black MAC exhibits low reflectivity properties in the near infrared, as well as high solar absorptance within the solar spectrum.
**Effect of Coating Thickness on Thermal/Optical Properties for Black MAC**

- Solar absorptance and normal emittance do not significantly differ with various coating thicknesses.
CHARACTERIZATION

- **Surface Morphology**
  - Confocal Imaging Microscope (CIM) Analysis
    - Olympus LEXT confocal laser scanning microscope
      - 3D imaging of coating surface at 20X magnification

- **Black MAC** appears to be flatter with *less surface roughness* than White MAC
  - This may be a result of the smaller particle size distribution of the pigments in Black MAC

- Further optimization of formula will help increase the surface area or texture of the coating
  - Enlarging the particle size of the pigments
CHARACTERIZATION

- **Surface Morphology**
  - Scanning Electron Microscope (SEM) Analysis
    - SEM images reveal *highly porous structure* of zeolite materials in molecular adsorber coatings
      - Black MAC surface shows presence of pores that are desired for sufficient adsorptive performance. However, these pores appear to be *slighter larger, more randomly dispersed* when compared to White MAC, which exhibit *smaller, more uniformly dispersed* pores.

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**BLACK MAC**

**WHITE MAC**

*Formula 2-ML-H75, 456X Magnification at 8 kilovolts (kV)*

*Formula B-TRL6, 636X Magnification at 5 kilovolts (kV)*
TESTING

**Stage 3:** What are the thermal survivability/vacuum stability and molecular capacitance of black molecular adsorber coatings and how does it compare to its white counterpart?
TESTING

- Thermal Cycle Testing

**PURPOSE**

- Evaluate the *thermal survivability* and *vacuum stability* of Black MAC
  - Based on coating adhesion performance before and after exposure to test conditions

**TEST CONDITIONS**

<table>
<thead>
<tr>
<th>LOCATION</th>
<th>DURATION</th>
<th>TEMPERATURE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Veeco bell jar vacuum chamber</td>
<td>~100 cycles</td>
<td>-60 °C to 90 °C</td>
</tr>
</tbody>
</table>

In general, MAC is anticipated to operate at temperatures that are representative of electronics boxes and other interior surfaces, which typically reach temperatures between -10 °C to 40 °C.

*Test has a 50 °C margin for both hot and cold cases*
TESTING

- Thermal Cycle Testing

- SAMPLE DESCRIPTION
  - 21 - 2” by 2” Aluminum Samples
    - Coated with selected formula, BLACK MAC 2-ML-H75
    - Coated with some other formulas using 2-ML pigment

- TEST RESULTS
  - BLACK MAC 2-ML-H75
    - Samples coated with this formula demonstrated the *most favorable results* for thermal and vacuum durability
      - Minimal amount of coating removal after testing when compared against other 2-ML variations
    - This formula is *recommended to undergo further testing* under vacuum conditions for repeatability purposes, as well as after additional formula optimization
TESTING

Molecular Capacitance Testing

PURPOSE

- Measure the molecular capacitance, or the capability to adsorb outgassed materials, of Black MAC and compare to its White MAC counterpart

TEST CONDITIONS

<table>
<thead>
<tr>
<th>CONTAMINANT SOURCE</th>
<th>TEMPERATURE</th>
<th>EXPOSURE TIME</th>
</tr>
</thead>
<tbody>
<tr>
<td>Stearyl Alcohol *</td>
<td>45°C</td>
<td>88 to 160 hours</td>
</tr>
</tbody>
</table>

* Contaminant source is representative of commonly outgassed hydrocarbons for spaceflight applications

SAMPLE DESCRIPTION

- 18 samples of BLACK MAC 2-ML-H75
- 21 samples of WHITE MAC B-TRL6
Effect of Coating Thickness on Molecular Capacitance for MAC

- Molecular capacitance is a function of coating thickness.
- Ex: White MAC at 6 mils is projected to have a molecular capacitance three times greater than a 3 mil sample.
- Although Black MAC showed less surface area based on coating morphology, preliminary data exhibit similar trends for adsorption when compared to White MAC.
- Black MAC adsorbed 0.5 to 4.2 mg/cm² for thicknesses between 1.8 and 12.0 mils.
- Similarly, White MAC captured about 0.6 to 4.8 mg/cm² of contaminants for thicknesses between 2.3 and 10.7 mils.
CONCLUSIONS

What are the accomplishments, future work, and final conclusions for this study?
ACCOMPLISHMENTS

**DEVELOPMENT**
- Modified current White MAC formula by incorporating a variety of inorganic black powders
- Optimized formulation process by investigating different types and quantities of black pigments, binder to pigment ratios, and methods for treating pigments
- Evaluated adhesion performance for Black MAC at ambient conditions

**CHARACTERIZATION**
- Characterized thermal and optical property measurements, such as solar absorptance and normal emittance, as a function of coating thickness
- Verified low reflectivity characteristics of Black MAC for thermal and stray light control
- Established surface morphology with microscopic imaging techniques, such as CIM and SEM

**TESTING**
- Investigated adhesion performance, thermal survivability, and vacuum stability of Black MAC after thermal cycling at temperatures between -60 °C and 90 °C
- Performed preliminary molecular capacitance testing on Black MAC to establish its adsorption capability

**OVERALL**
- Compared Black MAC characteristics and determined its similarities and differences with White MAC, and other commonly used black spaceflight coatings
- Selected a potential Black MAC formula for further development and testing on repeatability and qualification studies
FUTURE WORK

- Optimize the coating with other inexpensive black carbon materials
- Evaluate particulation shedding effects of the coating
- Advance the low reflectivity properties into the mid to far infrared wavelengths for use in cryogenic applications
- Improve adhesion performance and increase surface area
- Repeat measurements and tests for reproducibility
- Perform a qualification effort of the final, optimized product for spaceflight and commercial use on surfaces to reduce both outgassing and optical path degradation
- Develop electrically dissipative and conductive versions of the coating to prevent charge build up and minimize voltage gradients on sensitive spacecraft surfaces
CONCLUSION

In conclusion, this project has successfully demonstrated the development of black molecular adsorber coatings.

- Preliminary coating formulations were characterized and tested for thermal and optical properties, surface morphology, adhesion performance, thermal survivability, vacuum stability, and molecular adsorption to protect sensitive surfaces from the damaging effects of outgassed materials.

- These results are promising and have shown that black molecular adsorber coatings have great potential to both reduce contamination levels and address thermal and stray light control issues for spaceflight applications.
ACKNOWLEDGEMENTS

- Testing and development efforts for black molecular adsorber coatings was funded through the Internal Research and Development (IRAD) Program at NASA GSFC.

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  - Alfred Wong (SGT, Code 546)
  - Kenneth O’Connor (SGT, Code 546)
  - Mollie Grossman (NASA GSFC, Code 541)
REFERENCES


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