Preparation and Characterization of Thin-Film Protective Coatings for Aerogels

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Background & Introduction

Aerogels [1]:
Light-weight super-insulating material that in the native form is 99.98% air made by replacing the liquid material of a gel with air prepared with the sol-gel process. Aerogels have been used extensively in aerospace applications (e.g. insulation of Mars rover batteries) and are more recently investigated for green energy applications (e.g. home insulation) also. Aerogels can be synthesized from a variety of precursors resulting in different types of physical and chemical properties [2,3,4]

Two types of aerogels were investigated in this study:

X-Silica Aerogel: Cross-linked aerogels have added mechanical strength while preserving at least 50% of the original gel (normally 90%) [2]. Also is optically translucent.

Polyimide Aerogel: Flexible and optically opaque. Important for insulation of cryogenic tanks used for NASA applications, as depicted in the picture.

http://www.techbriefs.com/component/content/article/18-ntb/techbriefs/materials/25170

Motivation: The focus of this study was to prepare, synthesize, and characterize thin film protective and antifouling coatings for two types of aerogel substrates. Our goal was to create a uniform and continuous thin film coating with 100% coverage of the surface underneath.

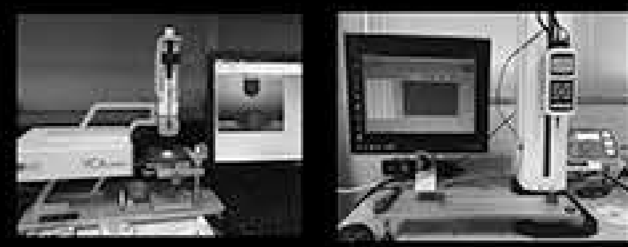
Methods & Procedures

Choice of coating: Dow Corning 3715 [5]: This compound was chosen for this study and its properties were tested. It is a highly viscous solventless polymer made by mixing the base and curing agent in a ratio of 7:3 and allowing time for curing. Due to time limitations only one type of coating was tested. Both types of aerogels were tested and characterized.

Coating techniques: Drop cast and spincoated, **Characterization techniques**: Mechanical agitation (testing bond strength) compression tests, surface coverage, and uniformity of coverage tested by light microscopy, **Contact Angle**: to test surface properties . **Coefficient of friction testing**: to assess shear force and "slip & stick" properties

Variations in Experimentation:
1. Drop-cast- drop 3715 onto aerogel and allowed it to spread and cure overnight
2. Method 1- drop 3715 on samples of polyimide before spinning at full speed (speeds: 4,000; 5,000; 6,000; 7,000; 8,000 RPM)
3. Method 2- dropped 3715 on samples of both types of aerogel while sample is spinning at full speed (speeds: every 1,000 from 1,000-8,000 RPM)
4. Outgassing- outgassed 3715 in oven until all bubbles were gone. Spincoated using new method at different RPMs (polyimide: 4,000; 6,000; 8,000 RPM; silica: 4,000; 8,000 RPM)

1. Mix Dow Corning 3715
2. Spincoat samples (made for us using previously published method) at different speeds depending on method and cure for 1 hour at 60°C on hot plate
3. Use light microscope to image each sample at 5X.
4. Sonicate for 15 minutes in IPA
5. Take contact angle measurements with AST VCA optima.
6. Complete friction tests with MARK-10 10N gauge with 200g steel plate; attach substrates on with double-sided tape and allows machine to run for aprox. 25 mm.
7. Calculate coefficient of friction with Excel.



(left) contact angle machine (AST VCA optima (right) friction testing machine (MARK-10 10N gauge including the steel plate and surface

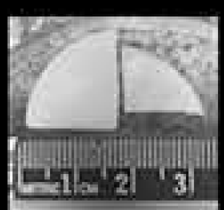
Results

X-Silica Aerogel

Frictional Studies of Control vs Drop-Cast

	Control	Drop-Cast
Static Friction	0.382449	0.319898
Kinetic Friction	0.279813	0.303244

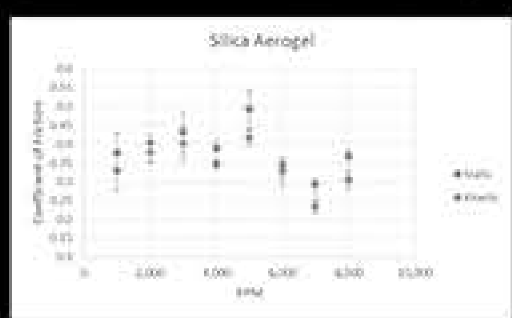
Friction measurements for the control and drop-cast samples



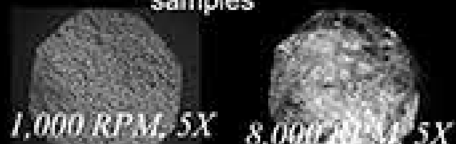
Control vs. Drop-cast

a) friction: very similar coefficients; can conclude that 3715 makes very little difference
b) Sonication/compression: 3715 adheres well to the surface

3715 + Silica Aerogel

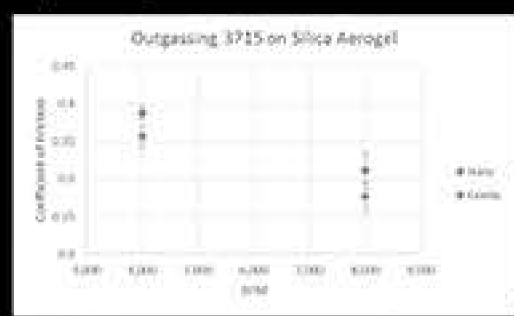


(top) Friction measurements for 3715 + aerogel samples



a) microscope: higher RPM displays more coverage but more striations
b) Friction: pretty consistent static and kinetic friction coefficients

Outgassed 3715 + Silica Aerogel



(top) Friction measurements for outgassed samples

Darker spots are places where the 3715 did not cover

Lighter spots are places that indicate coverage; fluctuation in darkness shows non-uniformity

a) microscope: outgassing results in more coverage and more consistent striations
b) Friction: very similar static and kinetic friction coefficients.

Contact Angle

	Left	Right
Control	72.7	72.675
3715 + Aerogel	109.545	110.898
Outgassed 3715 + Aerogel	97.25	98.125

(top) angle measurements for different methods (bottom) contact angle images for control, 3715 + aerogel and outgassed 3715

- Silica aerogel is originally moderately hydrophilic
- 3715 adheres well to this surface and creates a hydrophilic surface

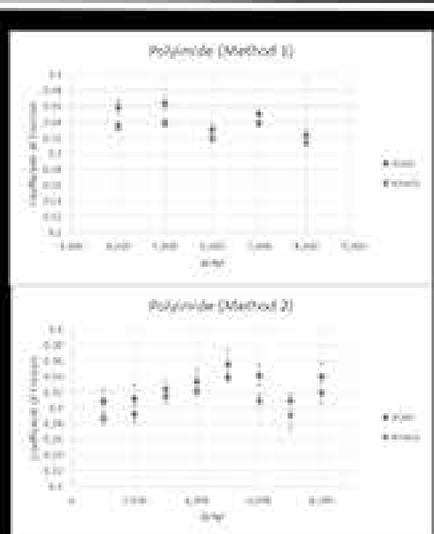
Polyimide Aerogel

	Control	Drop-Cast
Static Friction	0.691633	0.303061
Kinetic Friction	0.561302	0.295128

Friction measurements for the control and drop-cast samples

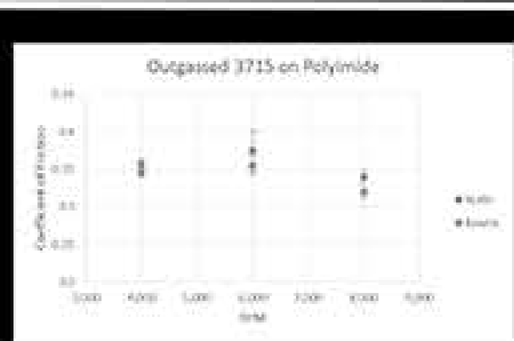
Control vs. Drop-cast

a) friction: drastically different coefficients; can conclude that 3715 lowers the coefficient of friction on polyimide
b) Sonication/compression: 3715 rubbed off easily like powder or paint.



(left) friction graphs for method 1 and method 2 (top) microscope imaging for method 1

a) microscope: little difference between all variations
b) Friction: very similar static and kinetic friction coefficients despite variables



(top) Friction measurements for outgassed samples

a) microscope: little difference between outgassed and not outgassed
b) Friction: very similar static and kinetic friction coefficients. A little fluctuation but overall consistent.

	Left	Right
Control	72.7	72.675
3715 + Aerogel	109.545	110.898
Outgassed 3715 + Aerogel	97.25	98.125

(top) angle measurements for different methods (left) technical hurdle

- Polyimide is originally very hydrophilic
- There were technical difficulties with the machine to get the water droplet to fall onto the polyimide

Conclusion & Summary

- 3715 spincoated top coat adheres well to surface of X-silica aerogels. It is hypothesized that the nanostructure of the aerogel substrate enhances the anchoring capability of the topcoat layer to the substrate
- Due to challenges associated with imaging aerogels, it was not possible to assess the degree of uniformity of the coating by means of light microscopy.
- 3715 topcoat layer resulted in a more hydrophobic surface compared to the control surface. The degree of reduction in accumulation of contamination was not assessed due to time limitations
- 3715 did not change the coefficients of friction at different RPMs for X-silica aerogels.
- The coating techniques used here did not lead to a successful binding and uniform coating of polyimide aerogels with 3715 polymer does
- Delaminating occurred at early stages of the testing, post curing
- Microscope had trouble differentiating the different RPMs due to opaqueness of the polyimide.
- 3715 topcoat did however drastically lower coefficients of friction in cases that it did adhere to the substrate
- Spincoating and out gassing of 3715 on aerogel substrates can lead to a uniform coating, however, preconditioning the surface of flexible aerogels will be necessary prior to deposition.

Future Works

- Improve the surface coverage by rendering the surfaces more hydrophilic, prior to coating of substrate
- Characterizing the spin conditions as a function of film thickness
- Finding a computer software to calculate the percent coverage of each of the samples
- Experimenting with different ratios of 3715 (base: curing agent) to decrease viscosity
- Test the coatings under cryogenic temperatures to see how the 3715 affects the insulation of aerogels

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References: [1] "The Blog." *Aerogelorg RSS.* N.p., n.d. Web. 11 July 2017 < http://www.aerogel.org/> [2] "Spectroscopic evaluation of polyurea crosslinked aerogels, as a substitute for RTV-based chromatic calibration targets for spacecraft." *Spectroscopic evaluation of polyurea crosslinked aerogels, as a substitute for RTV-based chromatic calibration targets for spacecraft - ScienceDirect.* N.p., n.d. Web. <http://www.sciencedirect.com/science/article/pii/S0273117710006319>.[3] **Parajuli, Pratikshya, Stephen W. Allison, and Firouzeh Sabri. "Spincoat-fabricated multilayer PDMS-phosphor composites for thermometry."** *Measurement Science and Technology* **28.6 (2017)** [4] Allison, S.W., Radiation Physics and Chemistry (2017) http://dx.doi.org/10.1016/j.radphyschem. 2017.01.045 [5] "DOW CORNING® 3715 TOPCOAT." *Silicones from Dow Corning.* Dow Corning Corporation, n.d. Web. <http://www.dowcorning.com/applications/search/default.aspx?R=1349EN>.