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HANSEN SOLUBILITY PARAMETERS FOR FLUOROALKYL SILICATES

October 17, 2012

Andrew Guenthner^{1*}, Gregory R. Yandek¹, Timothy S. Haddad²,
Kevin R. Lamison², Lisa M. Lubin³, Joseph M. Mabry¹

¹Aerospace Systems Directorate, Air Force Research Laboratory

²ERC Corporation

³ AFRL Summer Externship Program

andrew.guenthner@edwards.af.mil



Outline



- Background / Motivation
 - Fluoro-POSS Applications as Polymer Modifiers
 - Relationships Between Surface and Bulk Energy
 - Hansen Solubility Parameters for Bulk Energy
 - Girifalco-Good Parameters for Surface Energy
- Previous Work on Fluoro-POSS Surface Energy
- Current Work on Fluoro-POSS Bulk Energy
 - HSP of Fluoro-POSS and Related Silicate Compounds
 - Group Contribution Estimates
- Comparisons of Surface and Bulk Energy Values



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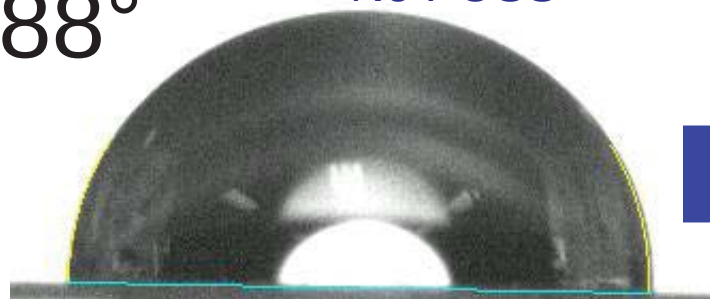


Effect of Fluorodecyl-POSS on Poly-(chlorotritluoroethylene) Surfaces



Water Contact Angles ...

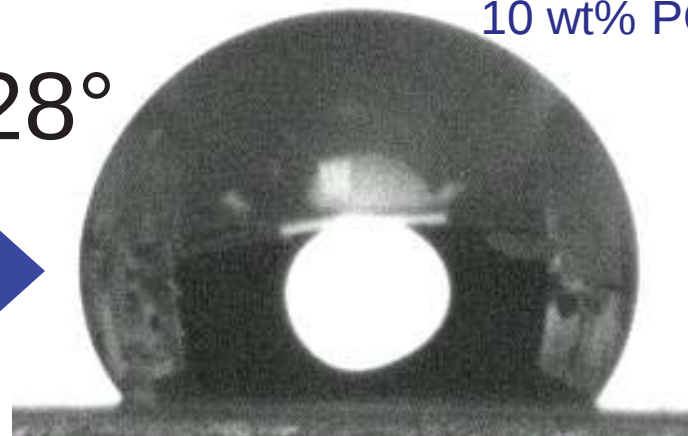
88° No POSS



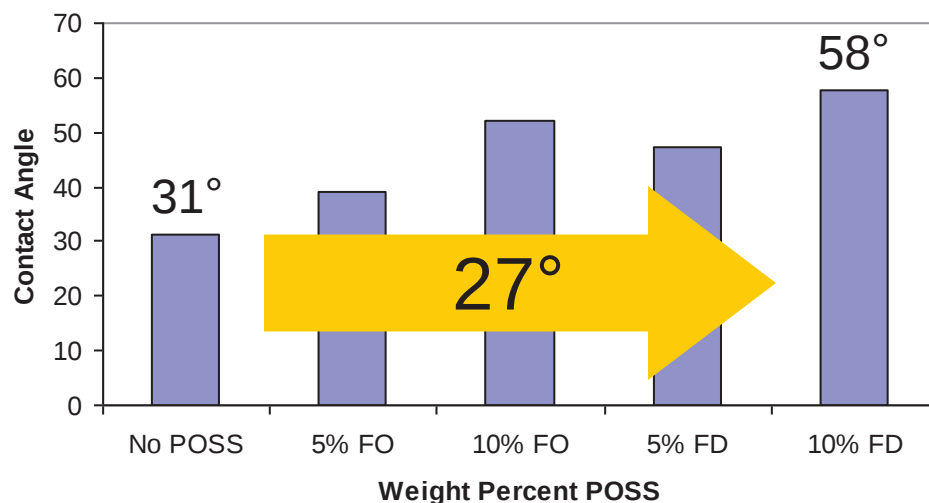
40°

128°

10 wt% POSS



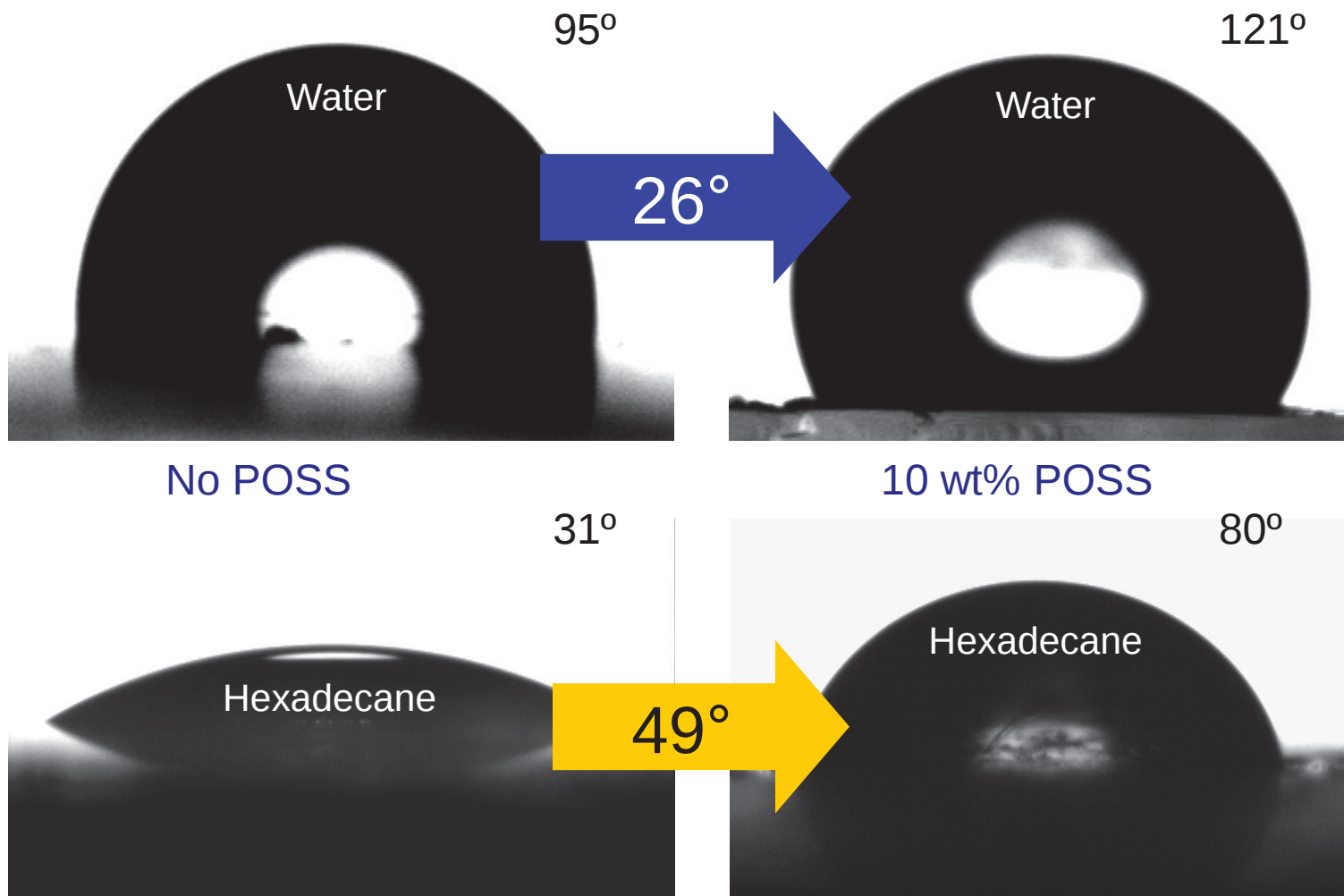
Hexadecane Contact Angles



Significant improvements in repellence of both water and oil are seen when fluoro-POSS is added to PCTFE



10% FD POSS in 6F PFCB polymer



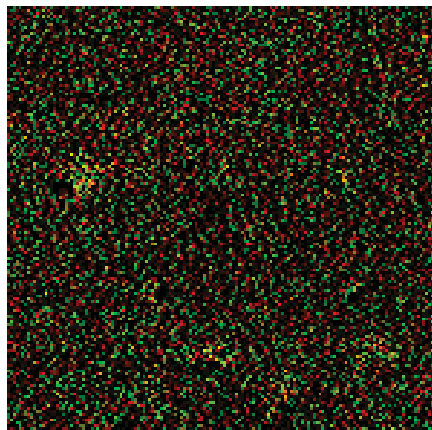
Another example of a fluoropolymer with liquid repellence improved by addition of fluoro-POSS, presumably due to the lower surface energy of Fluoro-POSS



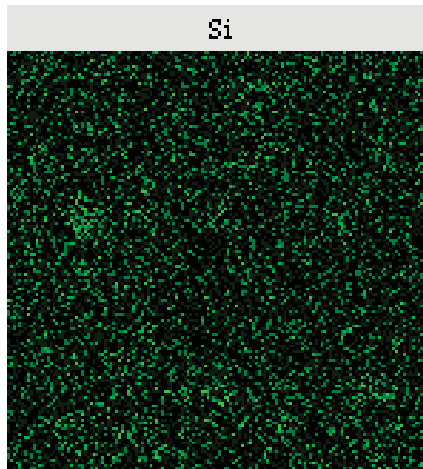
EDX/AFM of POSS/PFCB Surfaces



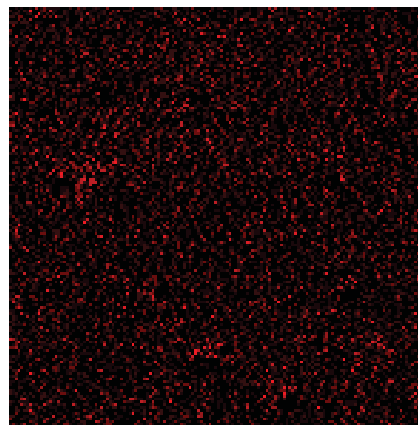
Composite Blend



Si



F



EDX shows that the surface becomes enriched in fluoro-POSS; AFM shows that surface migration alters the surface topography in addition to lowering surface energy.

6F PFCB

15 wt% FD_8T_8

0.5 nm
 μm
rms

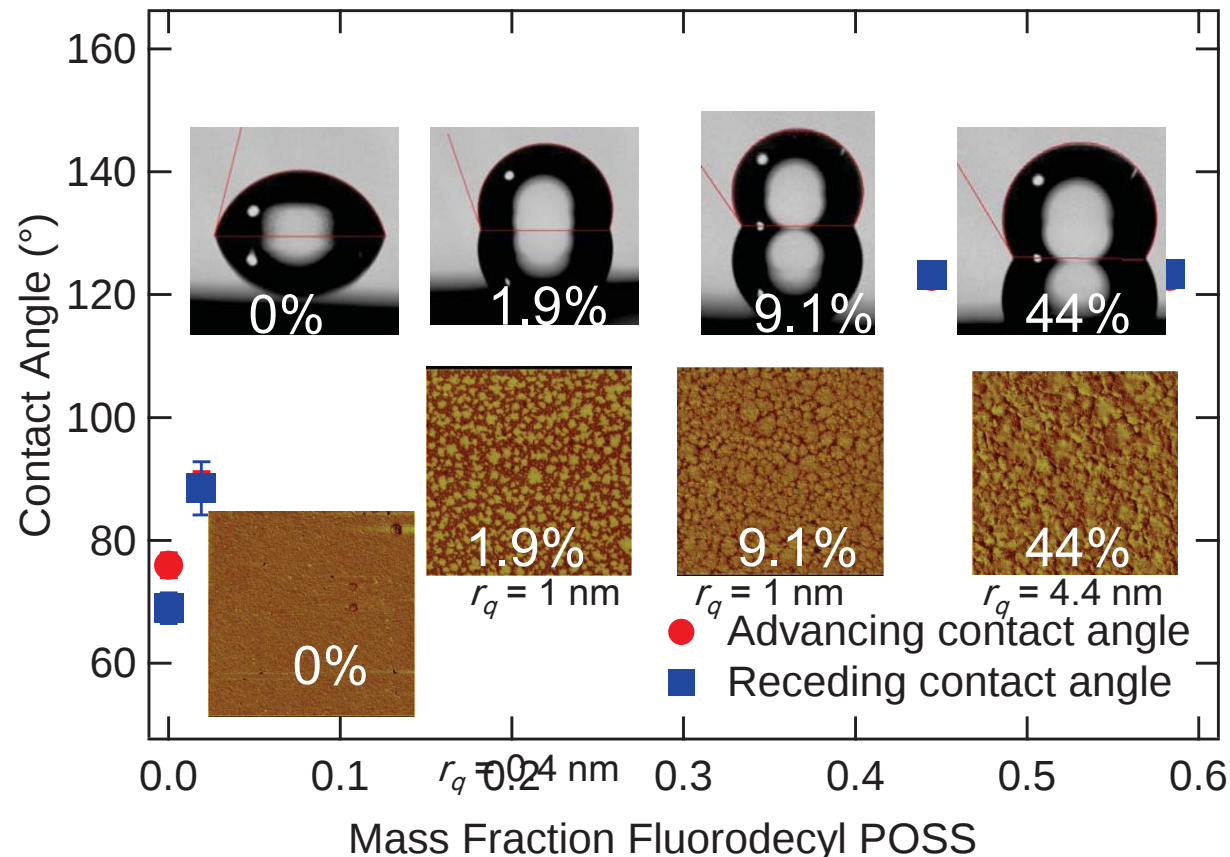
95° (H_2O)
31° (Oil)

121° (H_2O)
80° (Oil)

The performance of fluoropolymers with added fluoro-POSS depends on both bulk (phase separation) and surface (migration) energies.



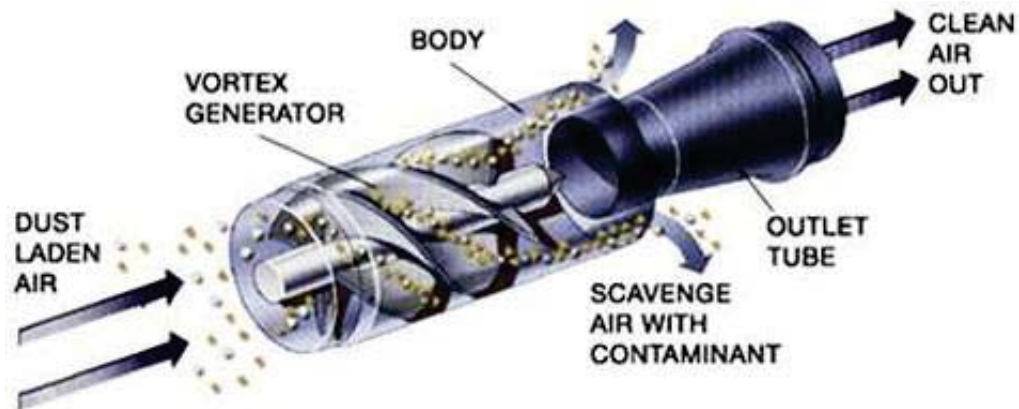
POSS/PMMA: Spin coating smooth surfaces



Pure PMMA is hydrophilic, yet phase separation and surface migration drastically alter water repellence. Understanding both bulk and surface interactions between fluoro-POSS and polymers is the key to controlling performance. At present, a quantitative understanding of these phenomena is lacking.



Controlled Surface Repellence Is an Important Enabler for AF Applications



Helicopter vortex tubes are used to exclude dust and contaminants from engine intakes. Requirements include light weight, ease of manufacturing, mechanical (structural / abrasion resistance), *fouling resistance*, and thermal performance.

The traditional material is a polypropylene that is highly filled to provide the required performance. The high filler loading makes manufacturing difficult due to the low thickness of part features (needed to save weight) that must be molded using a high viscosity material.

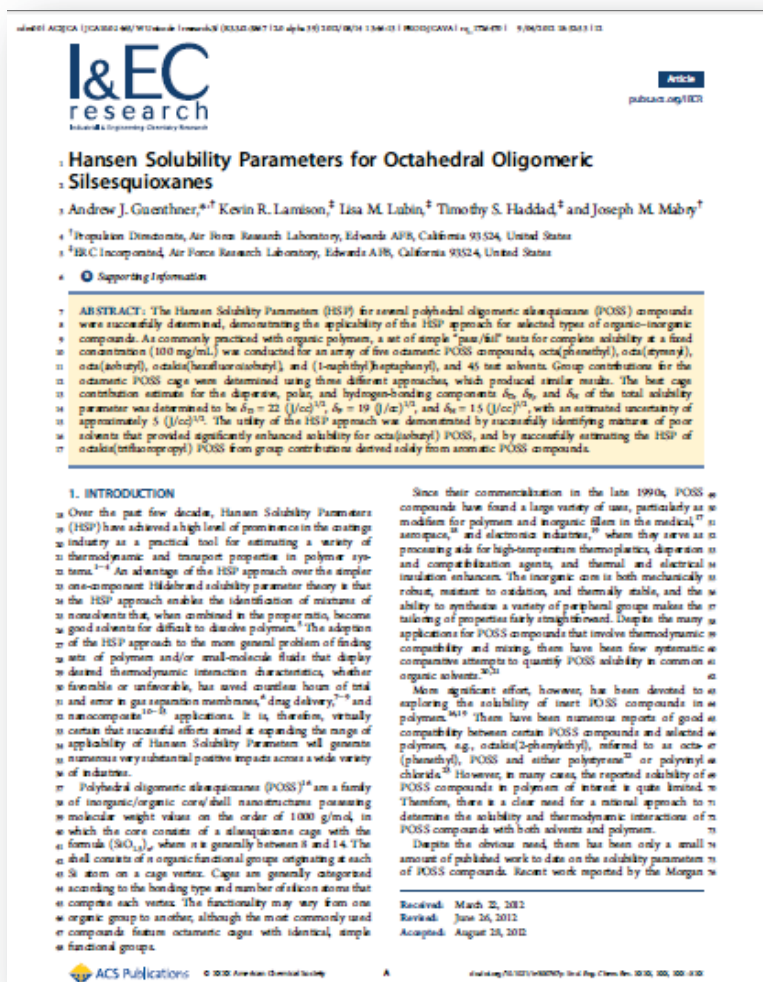
Inert POSS compounds, added as flow aids, allow more robust plastics such as cyclic olefin copolymers (COCs) to replace the polypropylene base material without sacrificing the ease of flow that facilitates ease of manufacturing. The high thermal stability and ~1 nm size of the POSS molecules provide the best available flow-enhancing characteristics and could also aid fouling resistance.



Bulk Interactions Can Be Quantified through Hansen Solubility Parameters



- Hansen parameters developed in late 1960s / early 1970s enabled development of a solvent mixture (n-butanol / nitroethane) for removal of “insoluble” epoxy primers from metal surfaces
- Hansen Solubility Parameters became widely used in the coatings industry, including for systems containing inorganic pigments – they remain the only successful approach for achieving miscibility via mixed solvents
- Solubility parameters for POSS compounds studied since ~2010 by numerous groups (Morgan-USM, Schiraldi-CWRU, AFRL)





Relation Between HSP and Phase Separation Dynamics



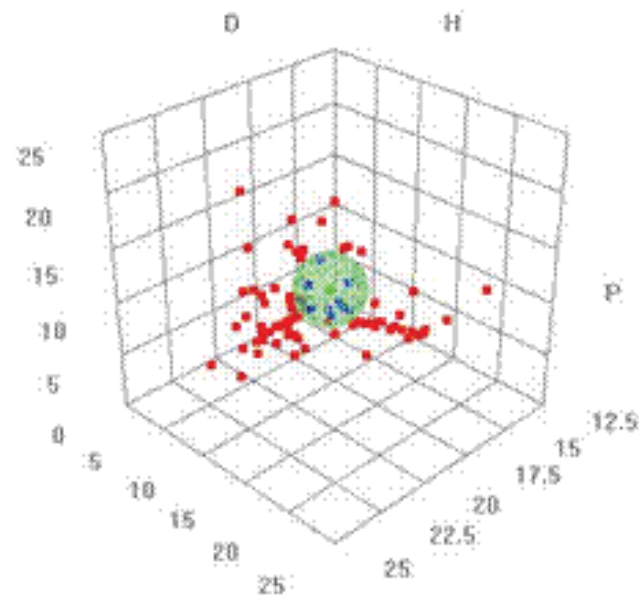
Solubility parameter
Enthalpy of mixing
Molar enthalpy of vaporization
Molar volume
Dispersive, polar, and H-bonding components

$$\delta^2 = (\Delta H_{\text{vap}} - RT) / V$$
$$\delta^2 = \delta_D^2 + \delta_P^2 + \delta_H^2$$
$$\Delta H_{\text{mix}} = \phi_1 \phi_2 V_{\text{ref}} [(\delta_{D1} - \delta_{D2})^2 + (1/4)(\delta_{P1} - \delta_{P2})^2 + (1/4)(\delta_{H1} - \delta_{H2})^2]$$

Volume fractions

Knowledge of Hansen Solubility Parameters enables the computation of the enthalpy of mixing between two components. For polymers, the value of the enthalpy of mixing controls the rate and extent of phase separation.

When plotted in a 3-dimensional “solubility parameter space”, good solvents (an indication of low enthalpy of mixing) tend to lie within a “sphere of solubility” centered on co-ordinates that correspond to the HSP of the solute. This “spherical rule” results from the similarity of the above equation to a geometric distance formula.



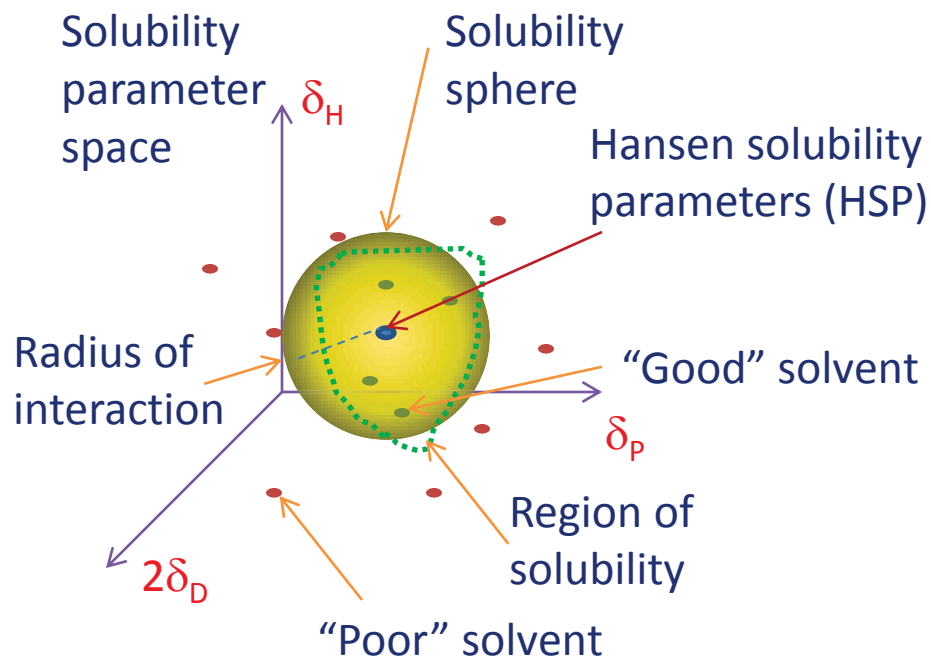
Hansen Solubility Parameter diagram for CO₂



Hansen Solubility Parameters for Polymer Systems



Experimental method for determining HSP



The traditional approach to estimating Hansen solubility parameters requires trials of typically 30 or 50 different solvents, but is straightforward to carry out. "Good" typically is indicated by >5% or 10% solubility, or by swelling, ESC, etc.

1. Determine solubility (or affinity) in a large set of trial solvents
2. Plot the HSP of "good" and "poor" solvents in a "solubility parameter space" as shown.
3. "Good" solvents normally lie near one another in a "region of solubility"
4. When $2\delta_D$ is used as an axis, the region of solubility is typically bounded by a sphere.
5. The center coordinates of the sphere mark the newly determined HSP.
6. With knowledge of the HSP and "radius of interaction" (test dependent), test results for any subsequent solvent (or mixture) are reliably predicted.

Source: Hansen Solubility Parameters: A User's Handbook, 2nd ed., CRC Press, 2007



Quantitative Interaction Parameters for Surfaces: Girifalco-Good Approach



Work of adhesion between solid and liquid

Dispersive components

$$W_{sl}^a = \gamma_{lv}(1 + \cos \theta_E) = 2[\sqrt{\gamma_{sv}^d \gamma_{lv}^d} + \sqrt{\gamma_{sv}^+ \gamma_{lv}^-} + \sqrt{\gamma_{sv}^- \gamma_{lv}^+}]$$

Equilibrium contact angle

Acid (+) / base (-)
component interaction
terms

- Measurement of contact angles for several probe liquids is used to determine the component values using linear regression.

ARTICLE

Fluoroalkylated Silicon-Containing Surfaces—Estimation of Solid-Surface Energy

Shreerang S. Chhatre,[†] Jesus O. Guardado,[‡] Brian M. Moore,[§] Timothy S. Haddad,^{||} Joseph M. Mabry,[§] Gareth H. McKinley,^{*,†,‡} and Robert E. Cohen^{*,†}

Department of Chemical Engineering, Department of Mechanical Engineering, and Department of Materials Science and Engineering, Massachusetts Institute of Technology, Cambridge, Massachusetts 02139, United States, Space and Missile Propulsion Division and ERIC Incorporated, Air Force Research Laboratory, Edwards Air Force Base, California 93524, United States

ABSTRACT The design of robust omniphobic surfaces, which are not wetted by low surface tension liquids such as octane ($\gamma_s = 21.6$ mN/m) and methanol ($\gamma_s = 22.7$ mN/m), requires an appropriately chosen surface microstructure in addition to a low solid surface energy (γ_s). 1H,1H,2H,2H-heptafluorooctadecyl polyhedral oligomeric silsesquioxane (fluorododecyl POSS) offers one of the lowest solid surface energy values ever reported ($\gamma_s \approx 10$ mN/m) and has become the molecule of choice for coating textured surfaces. In this work, we synthesize and evaluate a series of related molecules that either retain the POSS cage and differ in fluoromethyl chain length or that retain the fluorododecyl chains surrounding a linear or cyclic molecular structure. The solid surface energy (γ_s) of these molecules was estimated using contact angle measurements on flat spin-coated silicon wafer surfaces. Zisman analysis was performed using a homologous series of *n*-alkanes ($15.5 \leq \gamma_s \leq 27.5$ mN/m), whereas Girifalco–Good analysis was performed using a set of polar and nonpolar liquids with a wider range of liquid surface tension ($15.5 \leq \gamma_s \leq 72.1$ mN/m). The hydrogen bond donating, hydrogen bond accepting, polar, and nonpolar (dispersion) contributions to the solid surface energy of each compound were determined by probing the surfaces using a set of three liquid droplets of either acetone, chloroform, and dodecane or diiodomethane, dimethyl sulfoxide, and water.

KEYWORDS: superhydrophobicity • oleophobicity • solid surface energy • Zisman analysis • Girifalco–Good method

INTRODUCTION

In the recent past, there have been a number of reports on surfaces that are not wetted by liquid droplets, i.e., superhydrophobic (1–4), oleophobic (5–15), hydrophobic (16), and omniphobic (7, 12) surfaces. These surfaces have potential applications in oil–water separation, non-wettable textiles (2, 3, 6, 8, 9, 14, 15), and fingerprint/smudge resistant touch-screen devices. Here we use the term omniphobicity to refer to surfaces that are not wetted by a broad set of liquids, including water, alkanes, alcohols, acids, bases, and other organic liquids. The design of omniphobic surfaces involves selection of a suitable surface chemistry to minimize the solid surface energy and optimal choice of the surface texture.

In our previous work, we emphasized re-entrant topography as a necessary condition for the design of surfaces that are not wetted by low surface tension liquids (7–9, 11–13). Liquids such as octane ($\gamma_s = 21.6$ mN/m) and methanol (γ_s

$= 22.7$ mN/m) will partially wet a flat untextured surface (equilibrium contact angle, $\theta_E < 90^\circ$) of any surface chemistry. Using a combination of surface chemistry and re-entrant texture, surfaces that exhibit substantially enhanced nonwettability to such liquids (apparent contact angle, $\theta^* > 90^\circ$) can be created. On such nonwetting surfaces, liquid droplets sit partially on the solid texture and partially on the air trapped between the asperities of the solid texture. The Cassie–Baxter (CB) relation can be used to understand variations in the apparent contact angles (θ^*) for liquid droplets with solid–liquid–air composite interfaces. The CB relation shows that the apparent contact angle (θ^*) increases as the equilibrium contact angle (θ_E) increases and as the relative amount of trapped air increases (17). We have also developed an expression for the breakthrough pressure (P_b) required for the disruption of this solid–liquid–air composite interface for “CB state” (12). Both the apparent contact angle (θ^*) and the breakthrough pressure (P_b) increase monotonically with increasing equilibrium contact angle (θ_E) (7–9, 12). Therefore, maximizing θ_E is one objective in the optimal design of omniphobic surfaces with robust composite interfaces.

We have used fluorododecyl POSS-based coatings to design a range of robust nonwettability surfaces (7–9, 11–13). A fluorododecyl POSS molecule consists of a silicon–oxygen cage surrounded by eight 1H,1H,2H,2H-heptafluorooctadecyl chains (18). A flat silicon wafer spin-coated with a uniform coating of this molecule has one of the highest reported values of equilibrium contact angle for water

* Corresponding author. Tel.: 617/253-3777 (R.E.C.); 617/258-0754 (G.H.M.). Fax: 617/258-8224 (R.E.C.); 617/258-8629 (G.H.M.). E-mail: mcohen@mit.edu (R.E.C.); gcmh@mit.edu (G.H.M.).

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[†] Department of Chemical Engineering, Massachusetts Institute of Technology

[‡] Department of Materials Science and Engineering, Massachusetts Institute of Technology

[§] Space and Missile Propulsion Division, Air Force Research Laboratory

^{||} ERIC Incorporated, Air Force Research Laboratory

^{*} Department of Mechanical Engineering, Massachusetts Institute of Technology

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2010 American Chemical Society



Relationships Between Surface and Bulk Properties



$$\gamma = 0.75 \delta^{4/3}$$

D. W. Van Krevelen, *Properties of Polymers*, 3rd ed., (Elsevier, Amsterdam, 1990)

$$\gamma = [P_s / V(T)]^4; P_s \sim V_w$$

M. Bicerano, *Prediction of Polymer Properties*, 3rd ed. (Marcel Dekker, New York, 2002)

$$\gamma_D = 4.79 \delta_D - 51.87$$

A. Carré and J. Vial, *J. Adhesion*, **1995**, 42, 265.

- These relationships predict the properties of liquid surfaces, however, most fluorosilicates are solids at room temperature
- Even in liquids, molecular order at the surface is not taken into account by the predicted values
- To date, no widely known correlations exist for polar and hydrogen bonding components
- In general, hydrogen bonding is much stronger in bulk than at the surface (perhaps due to fewer constraints on interlocking in the bulk)

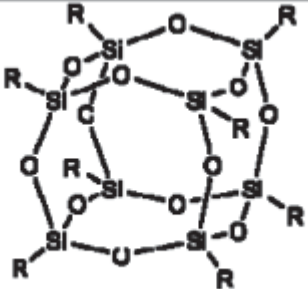
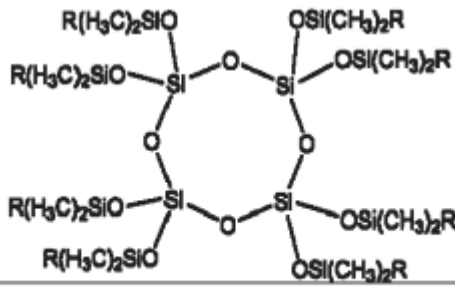


Fluoro-POSS and Related Fluoroalkyl Silicate Compounds



HSP

GG

	Fluorodecyl T_8 , $R = -(CH_2)_2-(CF_2)_7-CF_3$		
	Fluorooctyl T_8 , $R = -(CH_2)_2-(CF_2)_5-CF_3$		
	Fluorohexyl T_8 , $R = -(CH_2)_2-(CF_2)_3-CF_3$	✓	✓
	Fluoropropyl T_8 , $R = -(CH_2)_2-CF_3$	✓	✓
	Hexafluoro- <i>i</i> -butyl T_8 , $R = -CH_2-CH(CF_3)_2$	✓	✓
	Fluorodecyl Q_4 , $R = -(CH_2)_2-(CF_2)_7-CF_3$		✓
$F_3C(F_2C)_7(H_2C)_2(H_3C)_2Si-O-Si(CH_3)_2(CH_2)_2(CF_2)_7CF_3$	Fluorodecyl M2, $R = -(CH_2)_2-(CF_2)_7-CF_3$	✓	✓

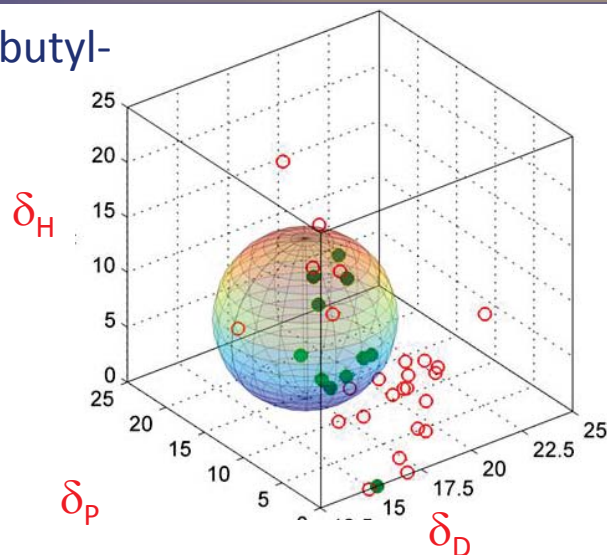
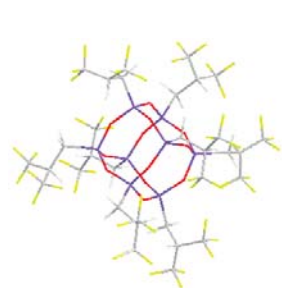
- T and Q compounds are crystalline at room temperature
- M compound is liquid at room temperature
- Q compound can be thought of as roughly half a POSS cage; M compound as roughly one-fourth a POSS cage



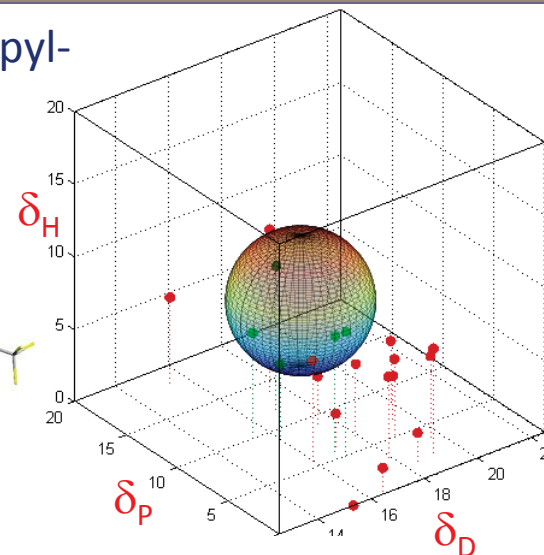
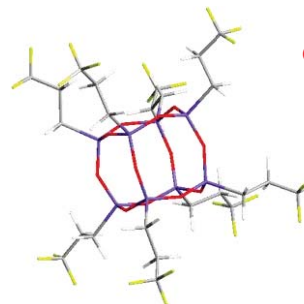
HSP Data for Fluoroalkyl Silicate Compounds



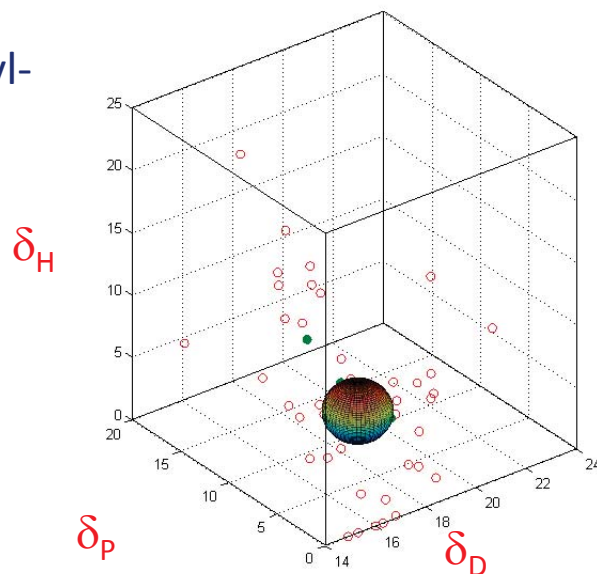
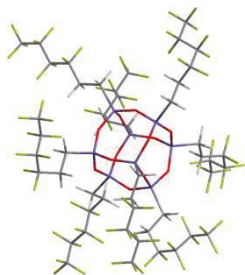
Octa-hexafluoroisobutyl-
POSS



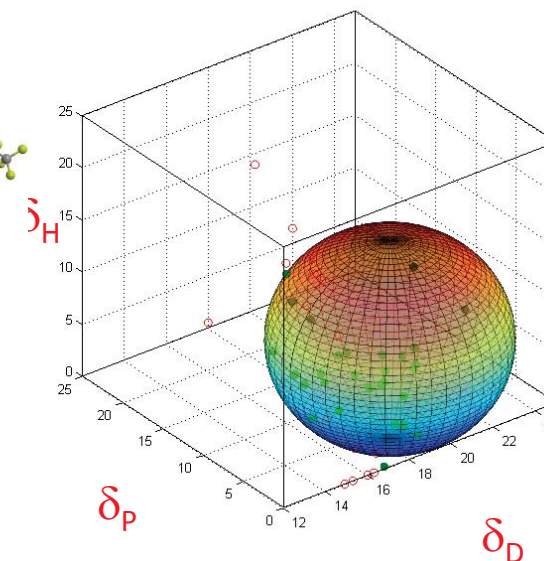
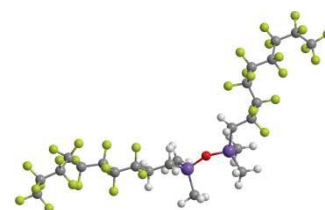
Octa-trifluoropropyl-
POSS



Octa-fluorohexyl-
POSS



Fluorodecyl-M2





Comparison of Hansen Solubility Parameters for POSS Compounds

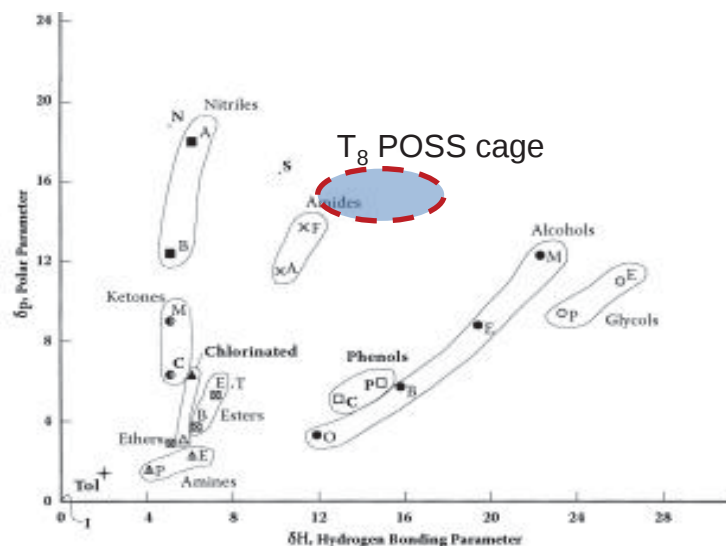


FIGURE 1.4 δ_p vs. δ_H plot showing the location of various common solvents. The glycols are ethylene glycol and propylene glycol. The alcohols include methanol (M), ethanol (E), 1-butanol (B), and 1-octanol (O). The amides include dimethyl formamide (F) and dimethyl acetamide (A). The nitriles are acetonitrile (A) and butyronitrile (B). The esters are ethyl acetate (E) and n-butyl acetate (B). The amines are ethyl amine (E) and

HSP trends do reflect the qualitative features expected on the basis on the peripheral chemical structure, such as:

- Smaller δ_D for fluorocarbons
- Larger δ_p for fluorocarbons

HSP for multiple POSS types provide an estimate of the HSP for a T8 cage

POSS / Silicate Type	δ_D	δ_p	δ_H	R_0	#Exceptions / #Good
Octa-isobutyl	18.0	2.1	2.7	4.5	6 / 8
Octa-hexafluoroisobutyl	15.3	9.3	11.0	7.3	2 / 10
Octa-fluorohexyl	16.3*	6.8*	6.7*	2*	n/a
Octa-trifluoropropyl	16.8	9.1	8.9	4.5	0 / 5
Fluorodecyl-M2	16.0*	5.4*	5.3*	5*	n/a

All units (J/cc)^{1/2}

*estimated



Comparison of Surface Energy Parameters for POSS Compounds



Table 3. Computed Values of the Dispersion (γ_{sv}^d), Acidic (γ_{sv}^+), and Basic (γ_{sv}^-) Components of Solid-Surface Energy (mN/m) for Various Fluoroalkylated Silicon-Containing Moieties

	alkanes (Zisman analysis)	all liquids ^b (eq 1 with $\varphi_{sl} = 1$)	diiodomethane, dimethyl sulfoxide and water (eq 5)				
	γ_c	γ_{sv}	γ_{sv}	dispersion (γ_{sv}^d)	polar (γ_{sv}^p)	acidic (γ_{sv}^+)	basic (γ_{sv}^-)
fluorodecyl T ₈	5.5	9.3	8.8	8.7	0.1	0.04	0.1
fluorooctyl T ₈	7.4	10.6	10.9	10.6	0.3	0.2	0.1
fluorohexyl T ₈	8.5	11.6	47.4	11.4	36.0	20.8	15.6
fluoropropyl T ₈	19.7	18.7	38.4	19.1	19.3	11.8	7.9
hexafluoro-i-butyl T ₈	17.7	19.1	26.9	26.8	0.1	0.002	0.8
fluorodecyl T ₈	5.5	9.3	8.8	8.7	0.1	0.04	0.1
fluorodecyl Q ₄	14.5	14.3	14.9	14.5	0.8	0.0	0.2
fluorodecyl M ₂	19.6	26.8	39.7	30.9	8.8	2.0	9.7

Predicted values based on Hansen Solubility Parameters (for “liquid” surfaces)

	γ_{lv} (dyn / cm)	γ_{lv}^d (dyn / cm)
Fluorodecyl T8	34.6	24.8
Fluorohexyl T8	37.7	26.2
Fluoropropyl T8	43.7	28.6
Hexafluoro-i-butyl T8	43.5	21.4
Fluorodecyl M2	34.6	24.8

- For perfluoroheptane, the predicted value of γ_{lv} of 21 dyn/cm is close to expectations
- Agreement for the dispersive component is better, but $\gamma_{lv}^d < \gamma_{lv}$ without rearrangement



Application to Miscibility and Phase Separation



$$\frac{\partial \phi(r,t)}{\partial t} = -\nabla J(r,t) + \eta(r,t)$$

$$J(r) = -M \nabla \mu(r)$$

$$\mu = \frac{\delta G}{\delta \phi} = \frac{\partial G}{\partial \phi} - \nabla \frac{\partial G}{\partial \nabla \phi} \quad \kappa = \frac{1}{36} \left(\frac{a_1^2}{\phi} + \frac{a_2^2}{1-\phi} \right)$$

$$\frac{\Delta G}{k_B T} = \int_V \left(f[\phi(r)] + \kappa [\nabla \phi(r)]^2 \right) dV$$

$$\frac{\partial \phi(r,t)}{\partial t} = M \nabla^2 \frac{\delta G}{\delta \phi} + \eta(r,t)$$

$$\frac{\partial \phi(r,t)}{\partial t} = M \nabla^2 \left\{ \begin{array}{l} \frac{\ln \phi + 1}{N_1} - \frac{\ln(1-\phi) + 1}{N_2} + \chi(1-2\phi) \\ -\frac{1}{18} \left(\frac{a_1^2}{\phi} + \frac{a_2^2}{1-\phi} \right) \nabla^2 \phi + \frac{1}{36} \left(\frac{a_1^2}{\phi^2} - \frac{a_2^2}{(1-\phi)^2} \right) (\nabla \phi)^2 \end{array} \right\}$$

The Time-Dependent Ginzburg-Landau equation (linearized to Cahn-Hilliard equation) represents the established mathematical framework for predicting phase separation, if you know the “ χ ” parameter

$$\chi = V_{\text{ref}} [(\delta_{D1} - \delta_{D2})^2 + (1/4)(\delta_{P1} - \delta_{P2})^2 + (1/4)(\delta_{H1} - \delta_{H2})^2]$$

As an example, for octa-phenethyl-POSS in PEI:

Phenethyl-POSS: (J/cc)^{1/2}

$$\delta_D = 19.7; \delta_P^2 = 8.0 \quad \delta_H = 5.6;$$

PEI: (J/cc)^{1/2}

$$\delta_D = 19.6; \delta_P^2 = 7.6 \quad \delta_H = 9.0;$$

$$V_{\text{ref}} = 100 \text{ cc/mol} \therefore \chi = 0.12$$

$$N_1 = 10; N_2 = 100$$

Predicted miscibility: 10-20%

Actual miscibility: 2.6%

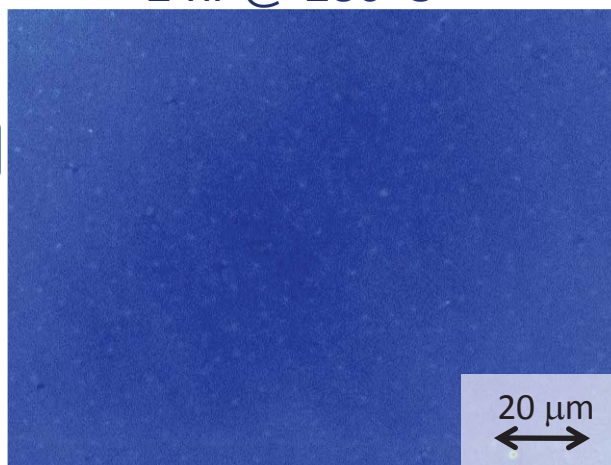


Surface Migration of 5 wt% Octa-Phenethyl POSS in PEI

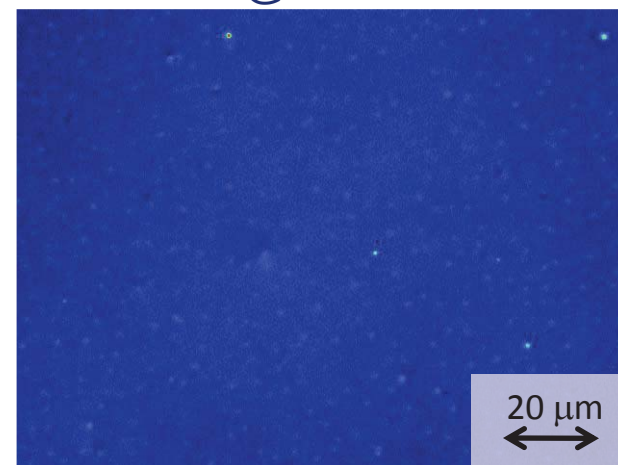


Films annealed at 180°C show effects that are not significantly different from those seen in pure PEI. At 210°C, fine aggregates and, later, a phase separated texture appear. The insets show the autocovariance (same length scale as main figure, red = high) of the pattern for annealing at 210°C, indicating periodicity.

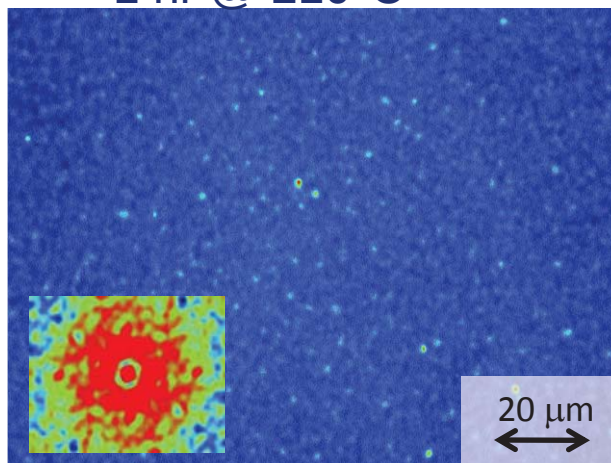
1 hr @ 180°C



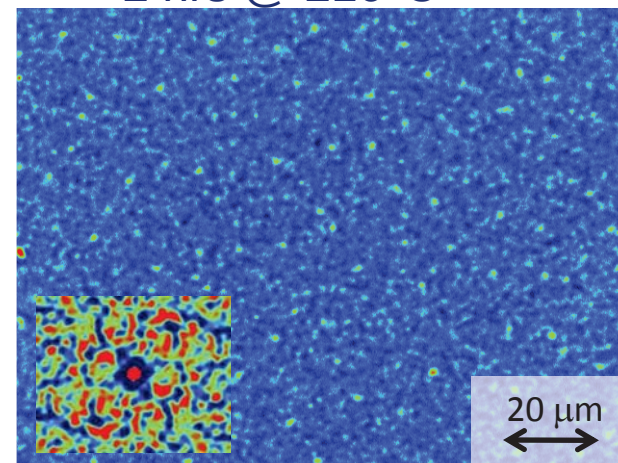
2 hrs @ 180°C



1 hr @ 210°C



2 hrs @ 210°C





Future Work

- Determine Hansen solubility parameters for additional silicate types (“D” is available from polysiloxanes, “T” from POSS, more “M” and “Q” examples needed)
- Improve precision of group contribution values for POSS cages and other silicate structures, as well as for fluorocarbons
- Generate additional predictions of miscibility and develop a spatio-temporal model for surface migration of silicate additives
- Use controlled migration to design organic / inorganic hybrids with optimal liquid repellence and other desirable properties

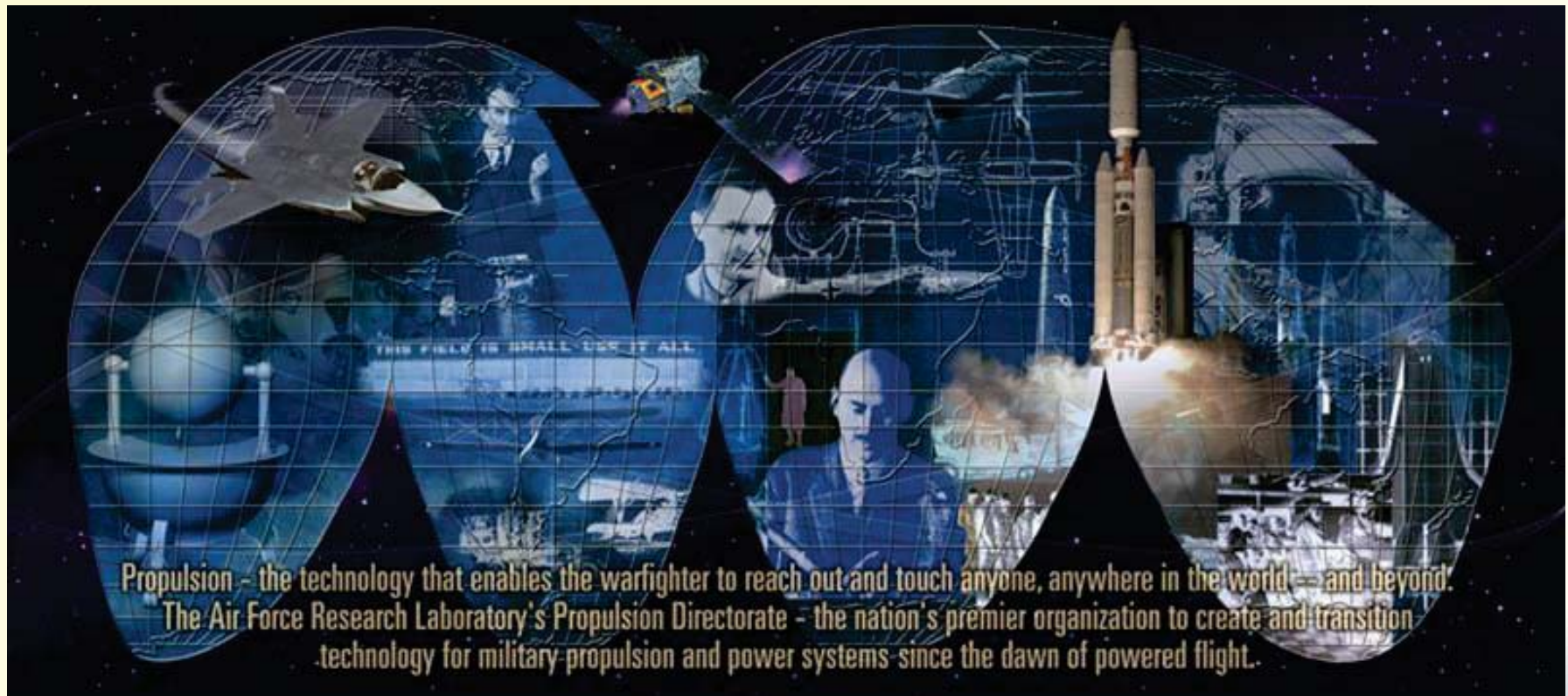


Summary



- Controlling the liquid repellence characteristics of polymer / silicate nanocomposites requires quantitative knowledge of both bulk and surface thermodynamic parameters.
- Quantitative surface and bulk thermodynamic data for polymer / silicate nanocomposites (including fluorinated polymers and fluoro-POSS) has recently become available.
- Comparisons of surface and bulk thermodynamic parameters provide insight into the nature of the silicate / polymer nanocomposite surface
- Application of the thermodynamic data to investigate miscibility and phase separation in polymer / silicate nanocomposites is underway





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