



# Mechanical and Tribological Behavior of Polymer-Derived Ceramics Constituted from $\text{SiC}_x\text{O}_y\text{N}_z$

---

**Tsali Cross, Somuri V. Prasad, David R. Tallant, Thomas E. Buchheit**

Materials Science & Engineering Center

Sandia National Laboratories

Albuquerque, NM

[svprasa@sandia.gov](mailto:svprasa@sandia.gov)

**Rishi Raj**

University of Colorado at Boulder, Boulder, CO

***Materials Science & Technology 2006***

***October 15-19, Cincinnati, Ohio***

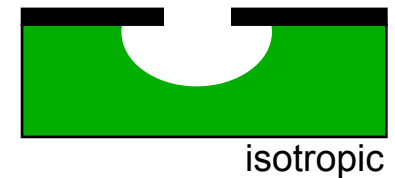
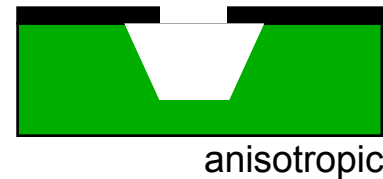


# A variety of fabrication methods can be used to construct microsystems

---

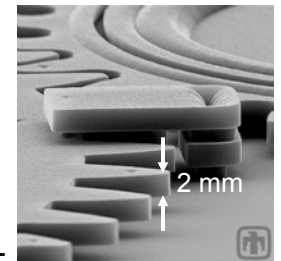
## Bulk Micromachining

- subtractive process; pattern and etch
- well established for Si; also done in GaAs



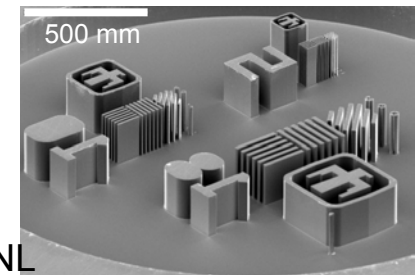
## Surface Micromachining

- additive process; structural (Si) and sacrificial ( $\text{SiO}_2$ ) layers
- dissolve sacrificial layers to free structures



## Lithographie, Galvanoformung und Abformung (LIGA)

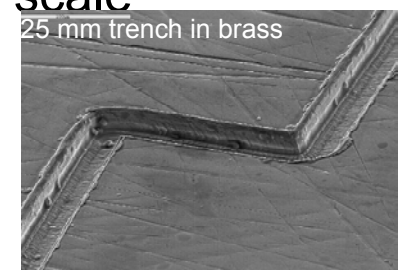
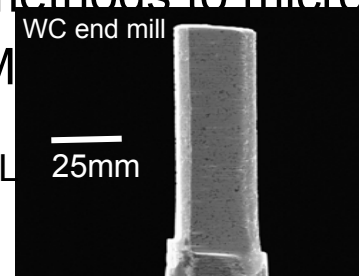
- plate or press into thick polymer mold
- micron to centimeter scale parts



## Meso-Machining

- subtractive process; push machining methods to micron scale
- micro-milling, micro-EDM, plunge EDM

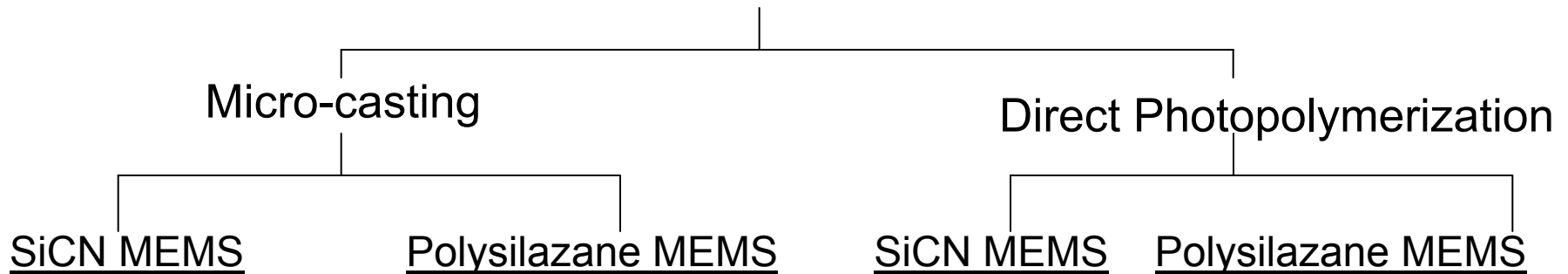
T. Christenson, SNL



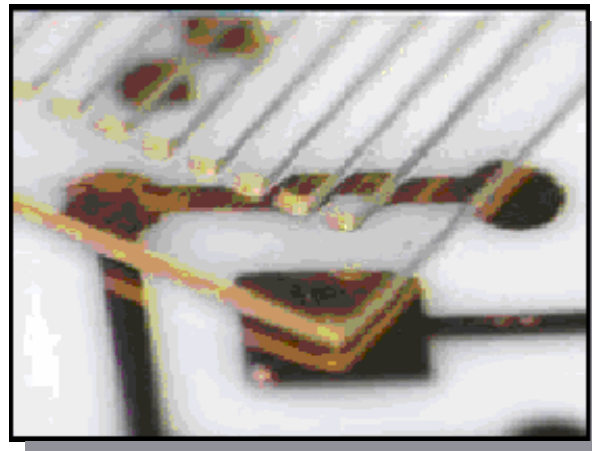
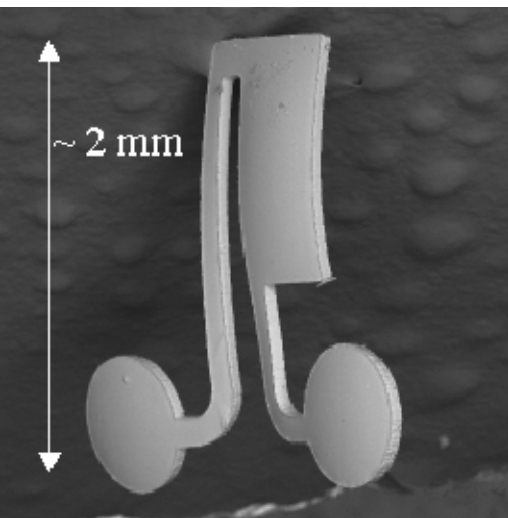
G. Benavides, SNL



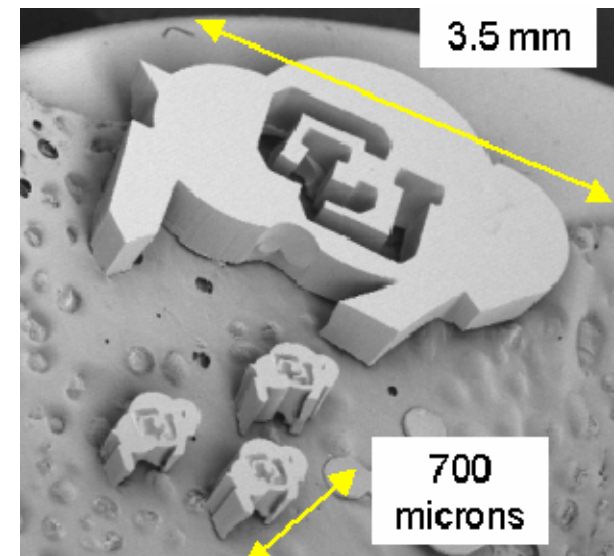
# Polymer Micromachining



Cast in SU8 molds, followed by de-molding

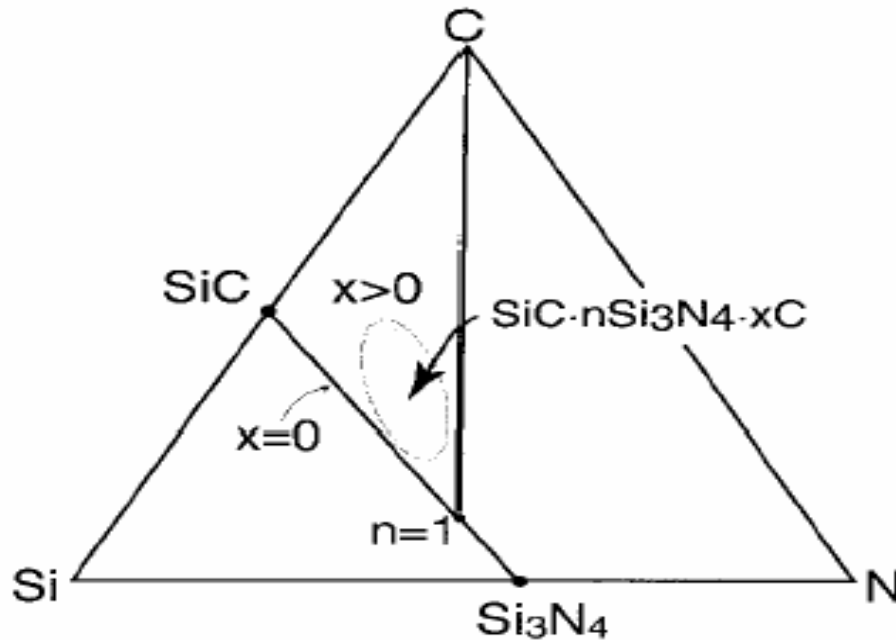


Treat precursor as a negative photoresist



Preceramic polymer buffaloes, 500 microns thick

# Composition diagram and the properties of SiCN PDC materials



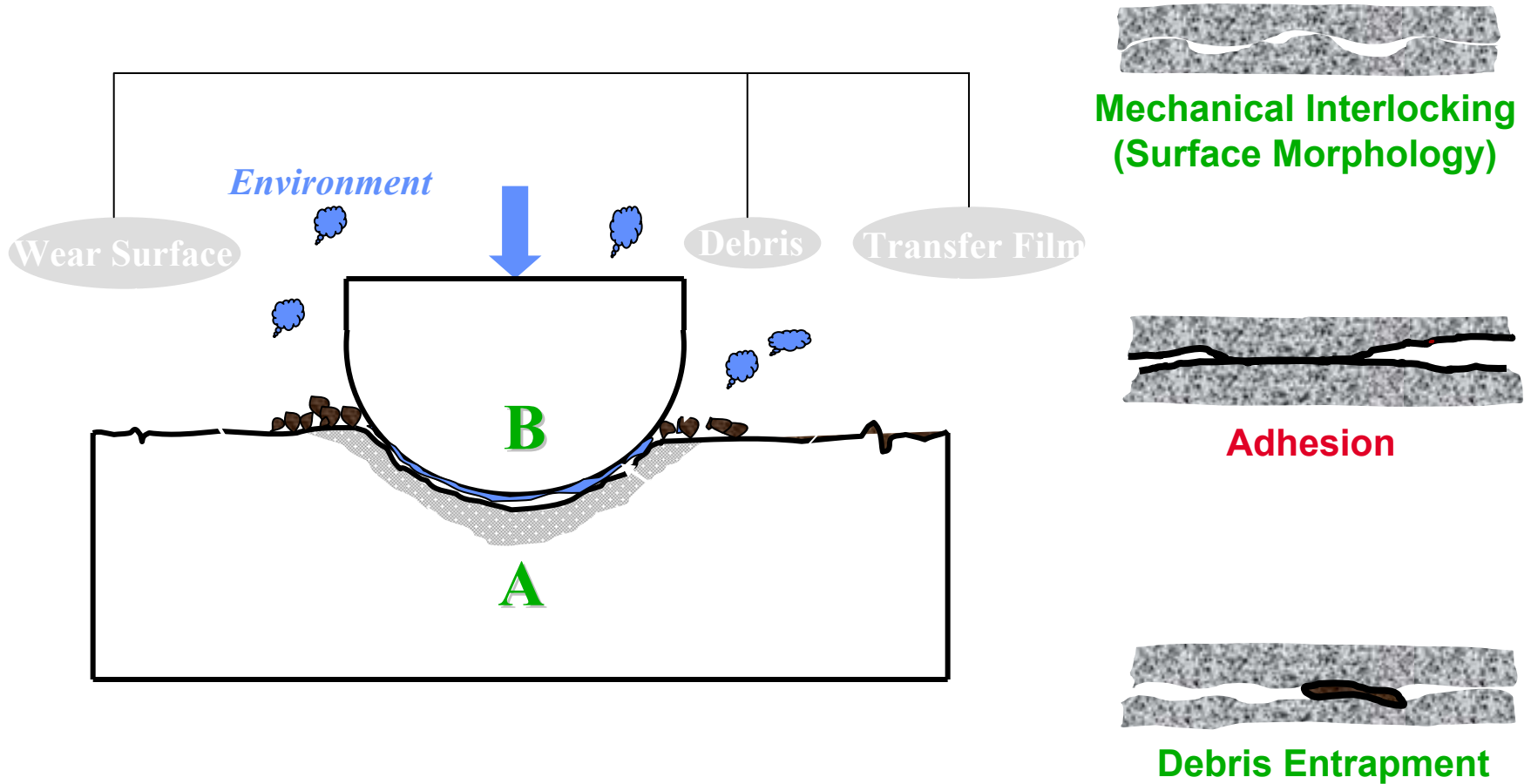
	SiCN	SiC	Si <sub>3</sub> N <sub>4</sub>
Density (g/cm <sup>3</sup> )	2.35	3.17	3.19
E Modulus (GPa)	150	405	314
Poisson's Ratio	0.17	0.14	0.24
CTE (x 10 <sup>-6</sup> /K)	~ 3	3.8	2.5
Hardness (GPa)	25	30	28
Strength (MPa)	1100	418	700
Toughness (MPa.m <sup>1/2</sup> )	3.5	4 - 6	5 - 8
Thermal Shock FOM*	2300	270	890
Creep rate at 1350 °C (s <sup>-1</sup> )	< 10 <sup>-8</sup>	~ 10 <sup>-9</sup>	~ 10 <sup>-9</sup>
Oxidation rate at 1350 °C (cm/hr <sup>1/2</sup> )	~ 10 <sup>-5</sup>	~ 10 <sup>-5</sup>	~ 10 <sup>-5</sup>

\* strength/(E-modulus.CTE)

Raj et. al.

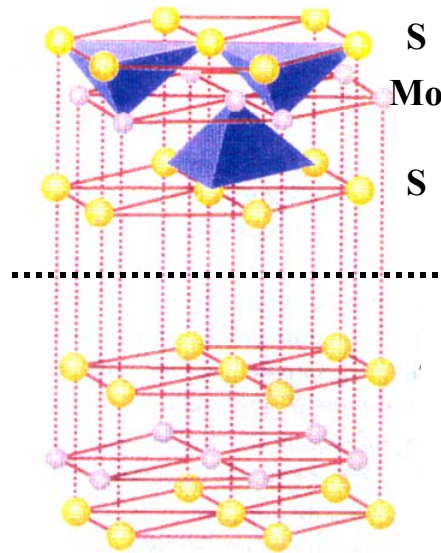
# Tribology @ Micro-level: a systems property

---



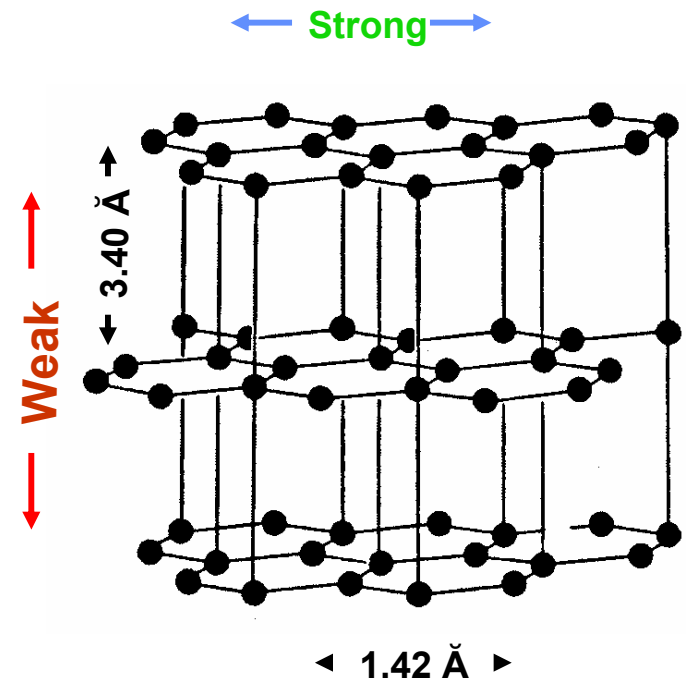
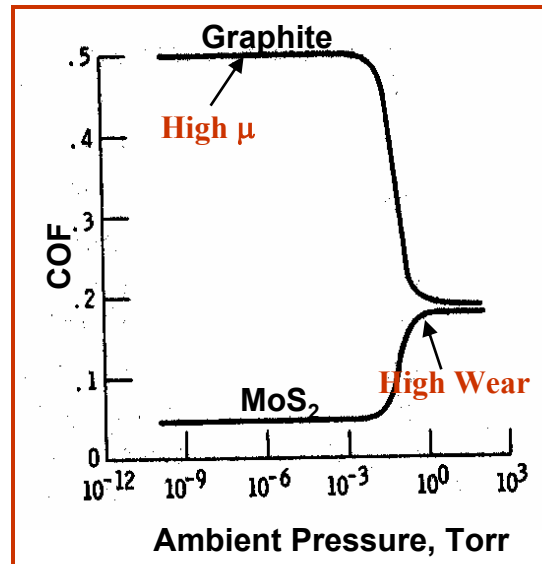
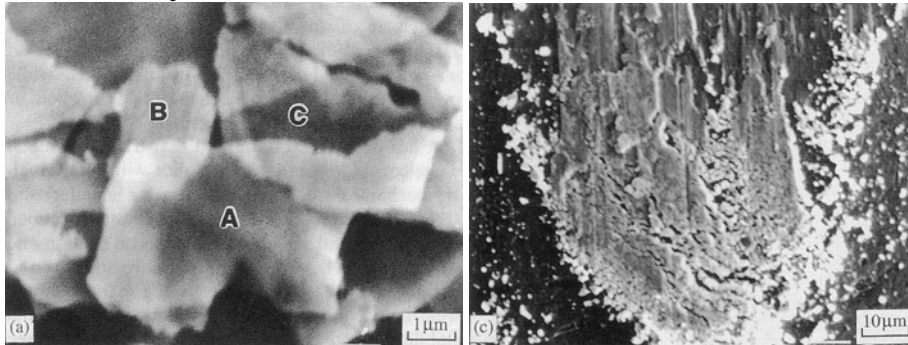
# No material can act as a solid lubricant in all environments and under all operating conditions

MoS<sub>2</sub>: Extremely low COF (0.01-0.05) and long wear life, **but only in dry environments.**



## Mo/W Disulfide

They form thin transfer films on the counterface

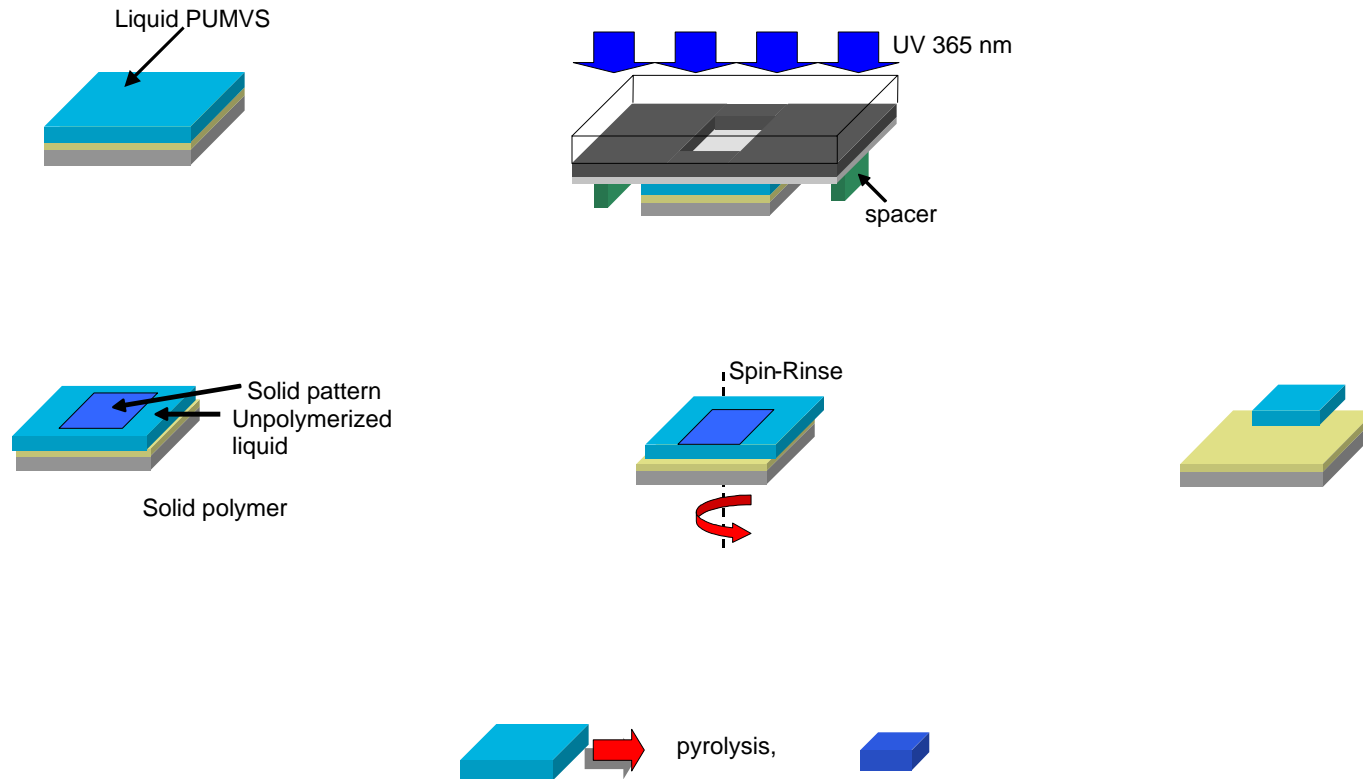


## Graphite

- Graphite needs moisture or adsorbed gases in the environment (>100 ppm) (they either act as intercalants, or passivate the dangling covalent bonds) to lubricate.
- In vacuum, graphite exhibits high friction and wear—a phenomenon known as “dusting”, first observed in the late 1930’s when graphite brushes in aircrafts experienced accelerated wear at high altitudes.

# A simplified process was used for making tribology test coupons (Courtesy of Sandeep Shah)

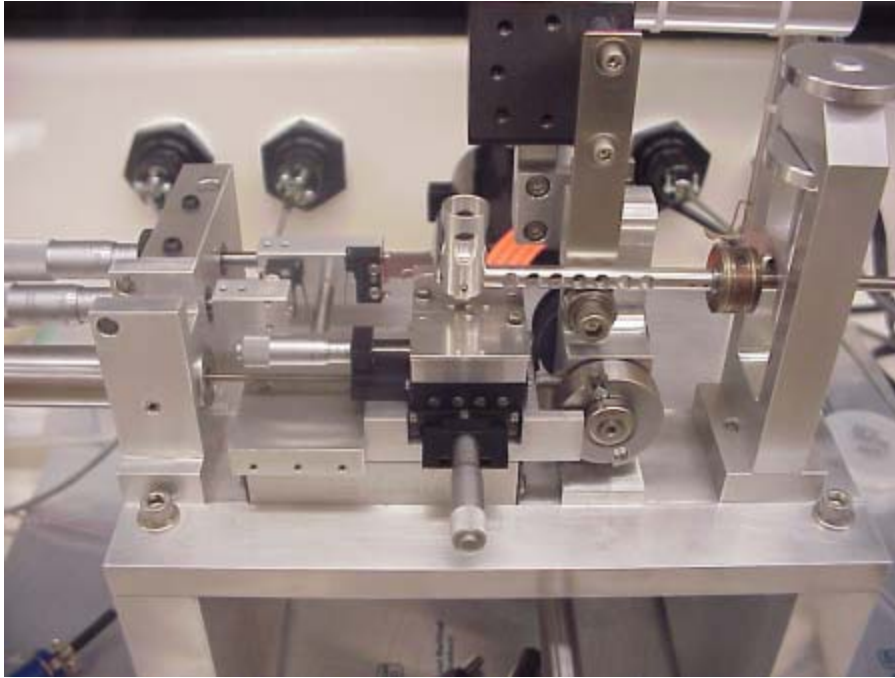
---



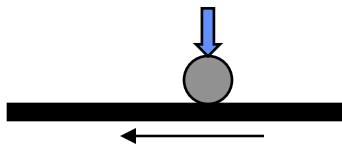


# Tribological measurements were made in controlled environments

---



Linear Wear Tester  
(Ball-on-Flat configuration)



Counterfaces:  $\text{Si}_3\text{N}_4$  and 440C Steel Balls (3.175 mm dia)

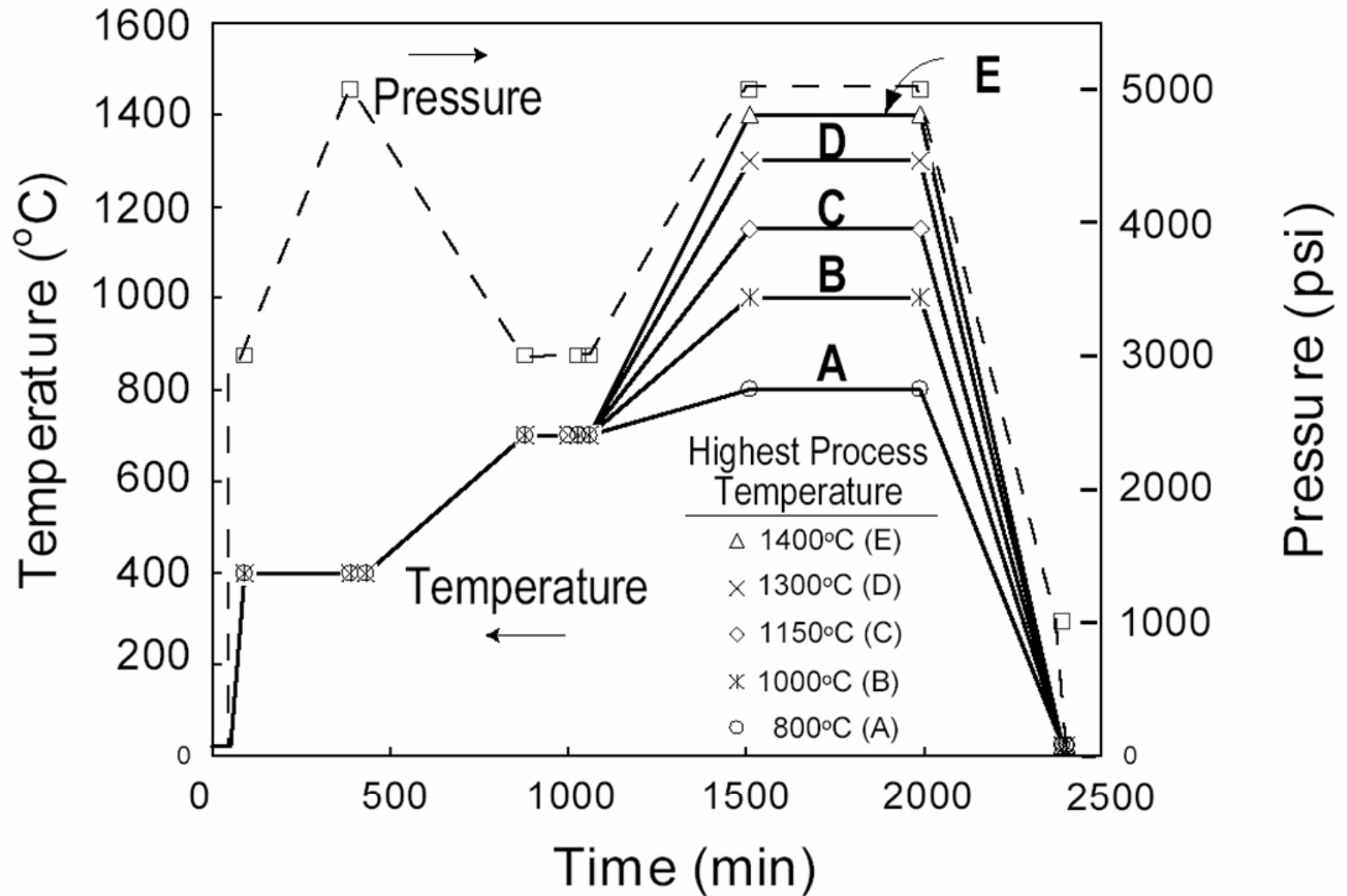
Normal Loads: 0.44 GPa, 0.75 GPa, 0.95 GPa

Environments: Dry Nitrogen, Dry Air, Air with 20, 40, 50 and 80% RH



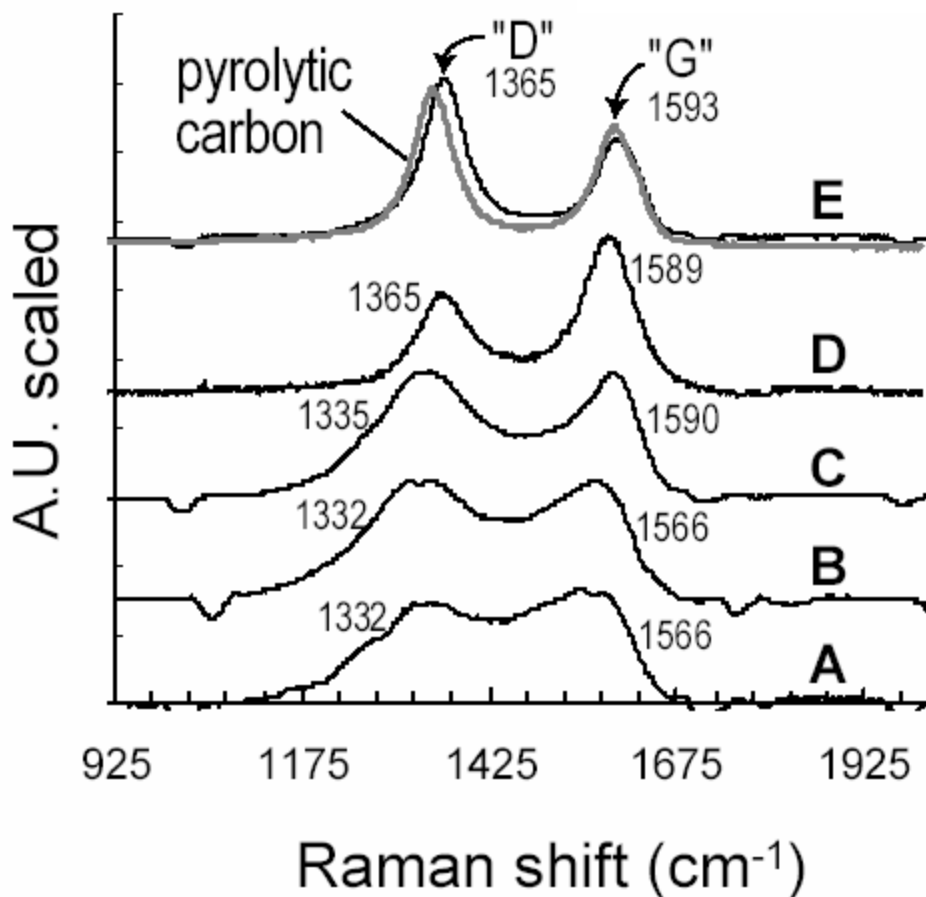
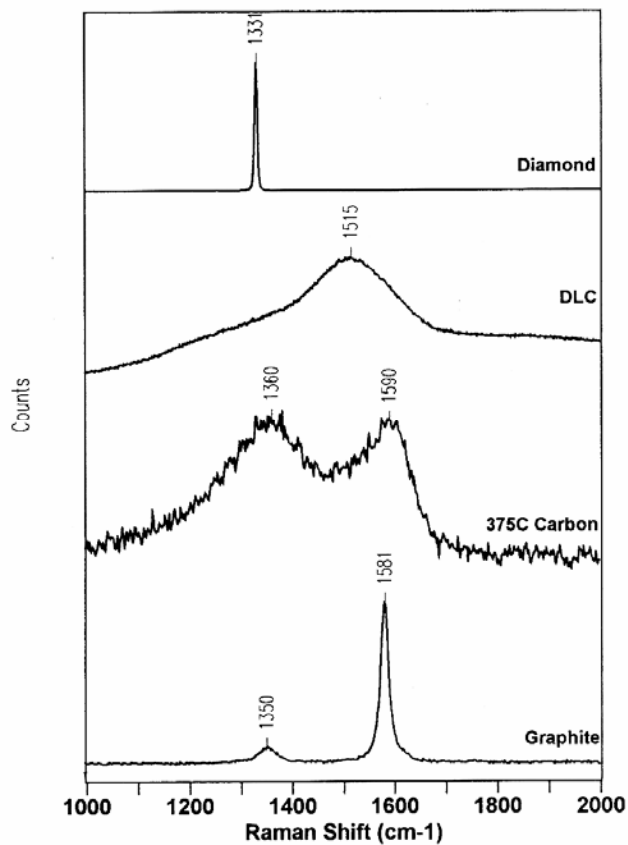
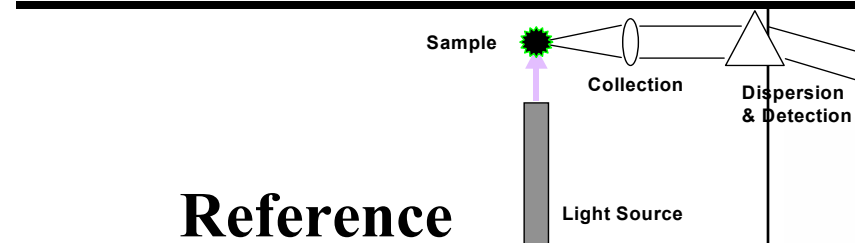
Environmental Control

# Pyrolysis cycles

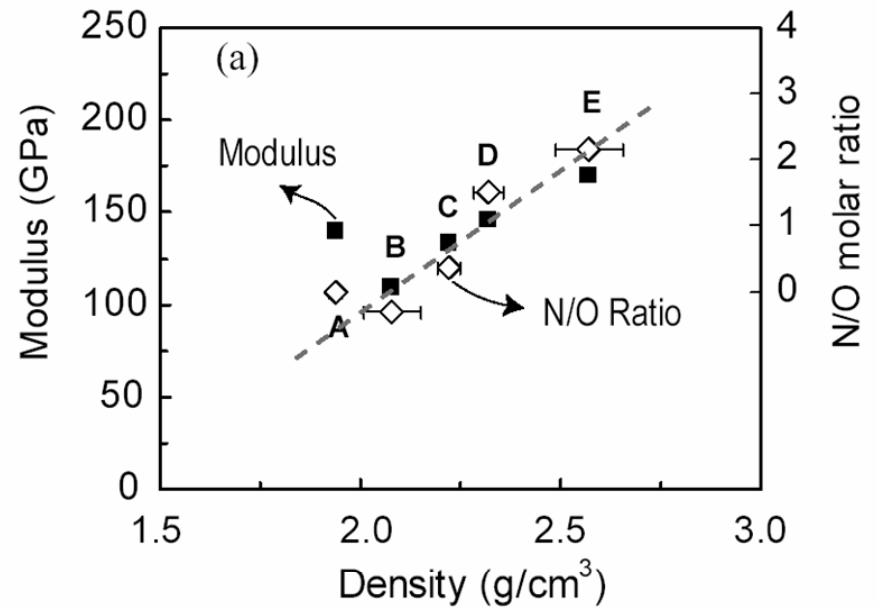
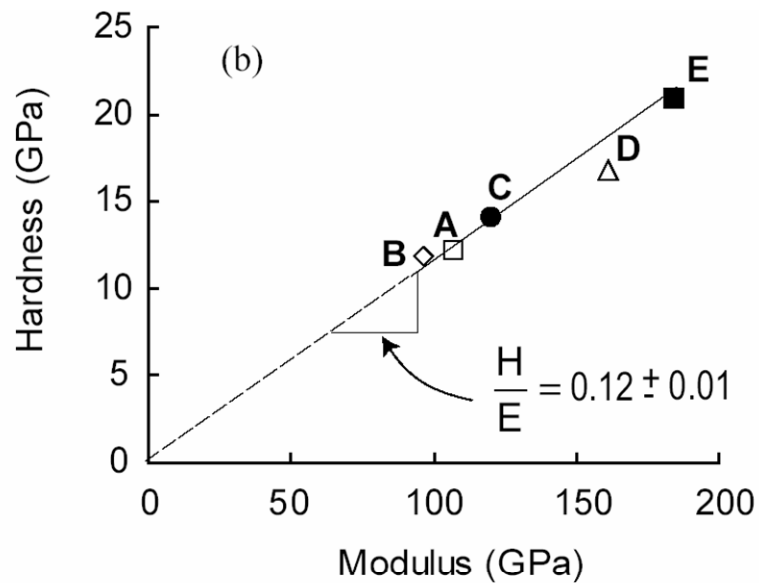


# Raman spectra (Argon laser: 458 nm wavelength, Spot Size: 1 $\mu\text{m}$ )

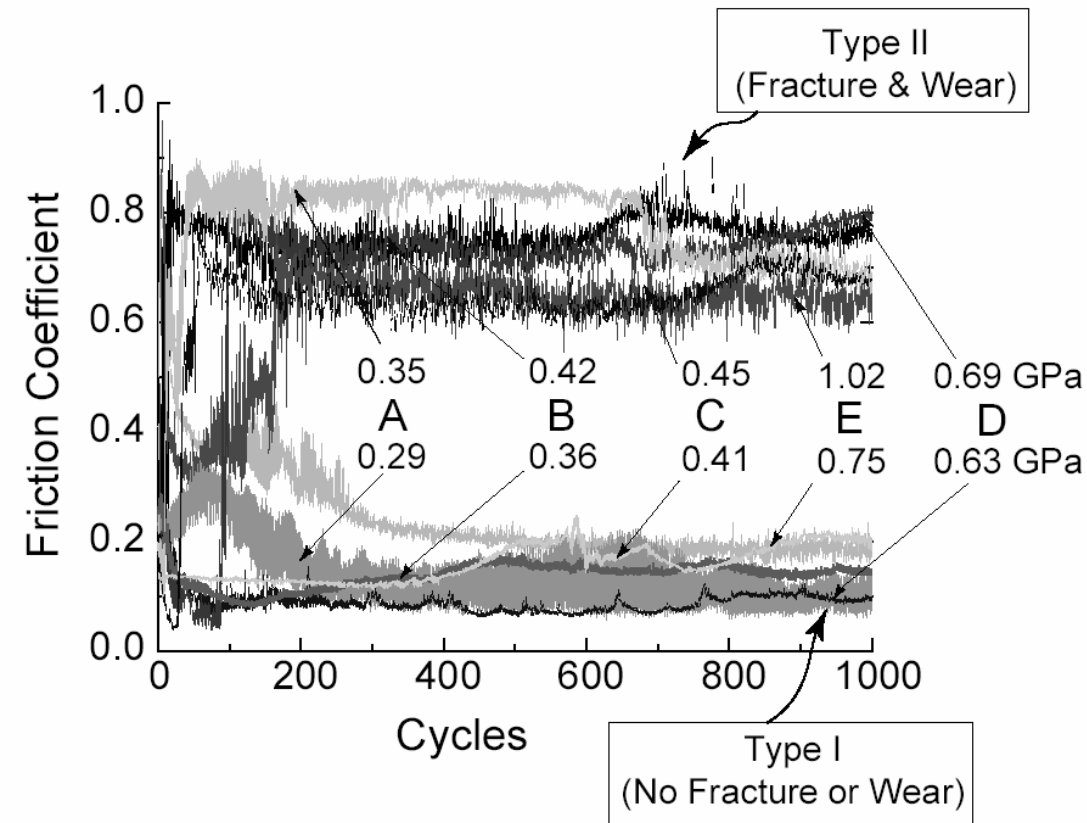
## Reference



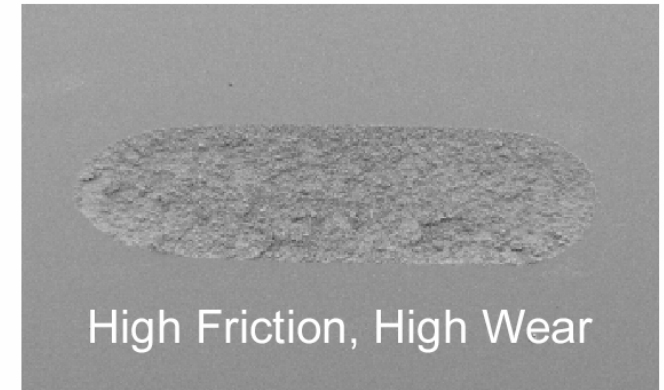
# Mechanical behavior and composition



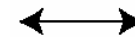
# Friction behavior in dry nitrogen



Type II: 0.97 GPa,  $\mu \sim 0.7$



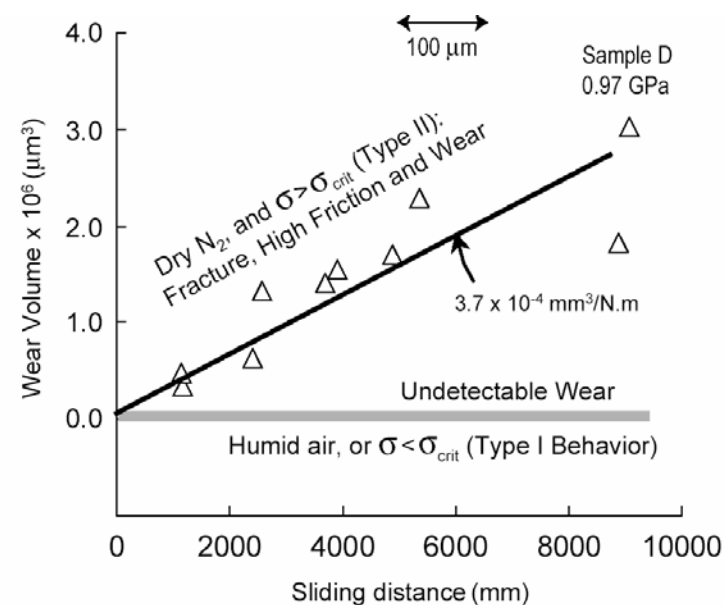
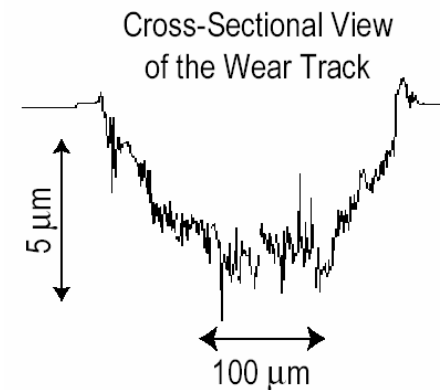
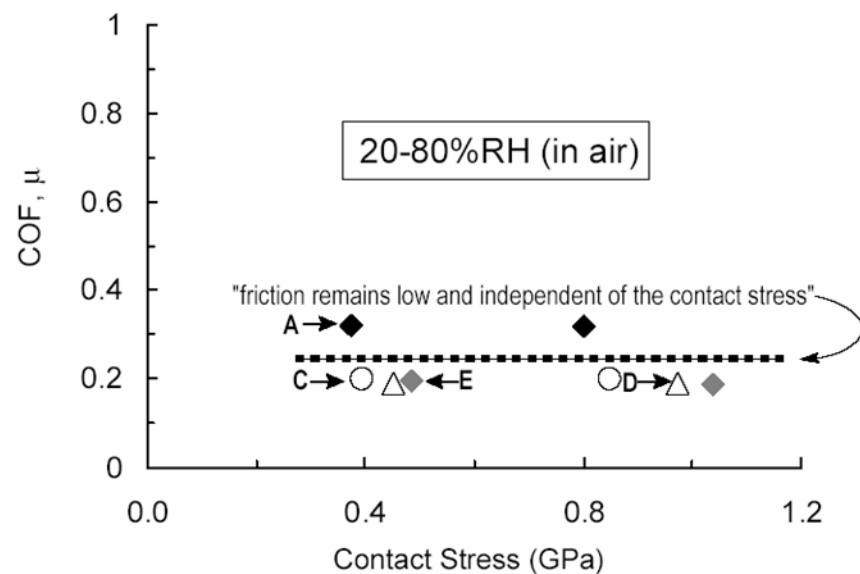
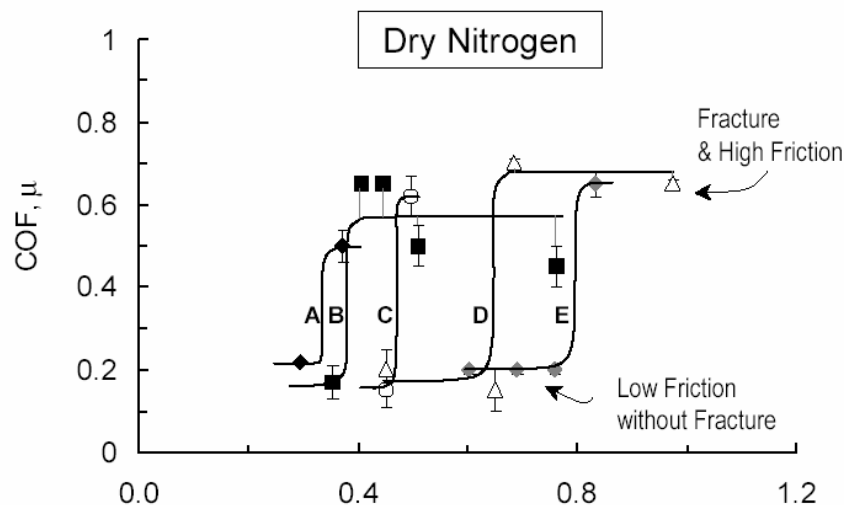
200  $\mu\text{m}$



Type I: 0.36 GPa,  $\mu \sim 0.2$

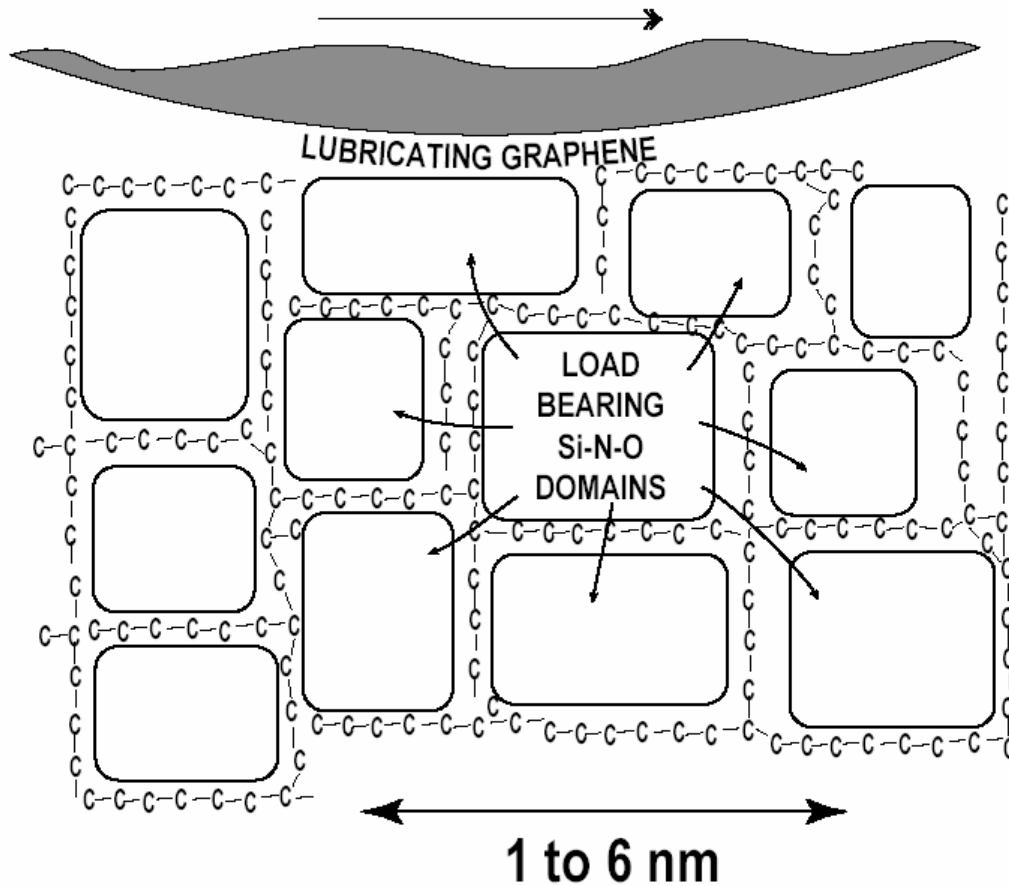


# Summary of tribological data showing environmental effects on friction and wear transitions



# Schematic depiction of dual-phase structure in SICNO

---



# Conclusions Remarks

---

- SiCNO showed two distinct regimes of tribological behavior: one identified by low  $\mu$ , and negligible wear, and the other by high friction and high wear.
- The **onset of fracture** under a critical value of the applied contact stress is believed to lead to the **transition** from low friction to high friction behavior. The transition apparently moved to contact stresses beyond the experimental regime in humid environments (where only the low friction behavior was seen).
- This result is thought to be related to an increase in the work of fracture of the PDC in the presence of water molecules at the crack tip when the crack propagation velocity is of the same order as the sliding friction velocity (that is, non-stress-corrosion type of fracture conditions).



# Acknowledgements

---

- **Regan Stinnett and Sandia's MESA Institute for financial assistance to Tsali Cross**
- **Rand Garfield for friction measurements**
- **Dave Tallant and Gina Simpson for Raman spectroscopy**
- **Bonnie Mckenzie and Dick Grant for scanning electron microscopy**