

A Study of the Self-Passivation of Space-Survivable POSS Kapton Polyimides



Dr. Sandra J. Tomczak
AFRL/PRSM Materials Application Branch
Space and Missile Propulsion Division
10 East Saturn Blvd., Bldg. 8451,
Edwards AFB, CA 93524-7680
Phone: (661) 275-5171
sandra.tomczak@edwards.af.mil

Mrs. Vandana Vij, Dr. Tim Haddad, ERC Inc.
Dr. Darrell Marchant, Dr. Joe Mabry (Group Lead)
AFRL/PRSM, Edwards AFB, CA.

Dr. Timothy K. Minton, Amy Brunsvold, Montana State University

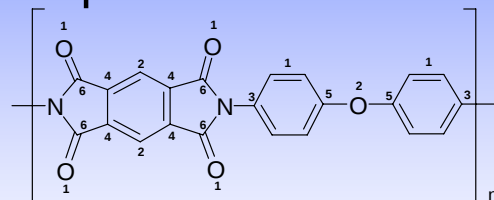
Report Documentation Page				Form Approved OMB No. 0704-0188	
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1. REPORT DATE JUL 2005		2. REPORT TYPE		3. DATES COVERED -	
4. TITLE AND SUBTITLE A Study of the Self-Passivation of Space-Survivable POSS Kapton Polyimides Poster Session				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S) Sandra Tomczak; Vendana Vij; Darrell Marchant; Timothy Haddad; Joseph Mabry				5d. PROJECT NUMBER DARP	
				5e. TASK NUMBER A443	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Air Force Research Laboratory (AFMC),AFRL/PRSM,10 E. Saturn Blvd.,Edwards AFB,CA,93524-7680				8. PERFORMING ORGANIZATION REPORT NUMBER	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited					
13. SUPPLEMENTARY NOTES					
14. ABSTRACT N/A					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES 16	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			



Goal

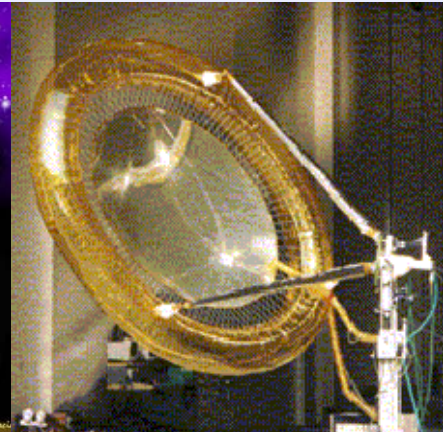
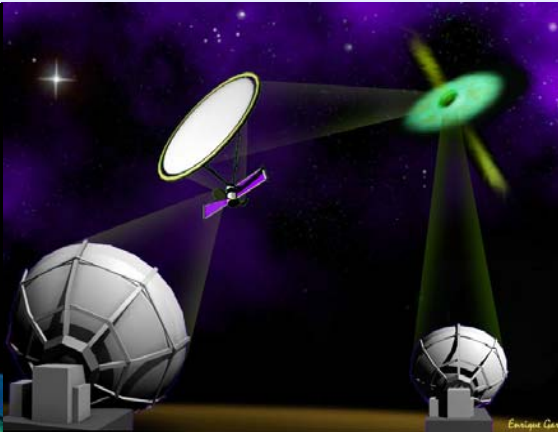
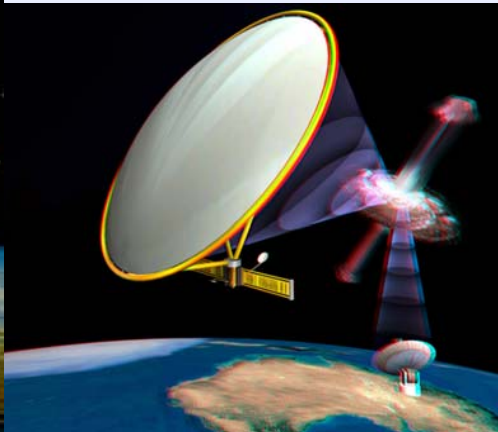
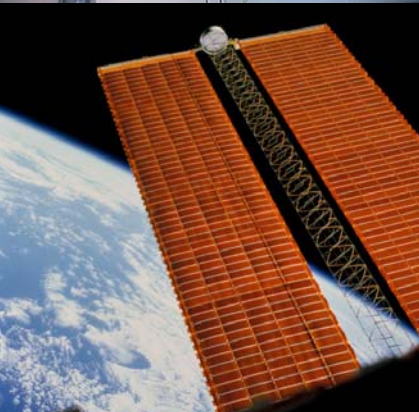
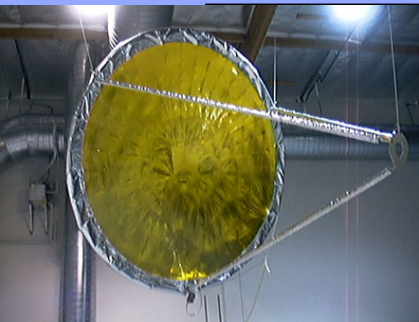


Kapton Structure



Our goal is to create an efficient drop-in replacement for Kapton that:

1. Has increased space survivability due to **resistance to atomic oxygen**, thermal cycling, solar UV and VUV radiation, protons and electrons.
2. Is **Self-Passivating** based on hybrid organic/ inorganic nanocomposite incorporation
3. Has superior optical properties, low solar absorptance, high thermal reflectance
4. Has excellent mechanical thermal properties.





Atomic Oxygen in Lower Earth Orbit



LEO Environment

(Altitudes of 200 to 1500 km)

- Atomic Oxygen (AO): $\sim 10^6 - 10^8$ atoms/cm³, up to 90 % of the atmosphere at 500km (typical altitude for international space station).
- Typical orbital speed of spacecraft is 7.8 km/sec
- Actual AO flux on spacecraft $\sim 10^{12} - 10^{14}$ atoms/cm²•s
- AO Collision energy ~ 5 eV (C-C bond energy ~ 4 eV, C-N ~ 3 eV, Si-O ~ 8.3 eV)
- Low-energy and high energy charged particles.
- Thermal cycling -50 to 150°C
- Solar VUV and UV radiation ($\sim 100 - 400$ nm)
- Bond scission and radical formation can lead to embrittlement.

Bond	Dissociation Energy (eV)	λ (nm)	Material
-C ₆ H ₄ -C(=O)-	3.9	320	Kapton®
C-N	3.2	390	Kapton®
Si-O	8.3	150	Nanocomposite

Satellites & Space Systems





AO Undercutting Study of Aluminized-Kapton MLI

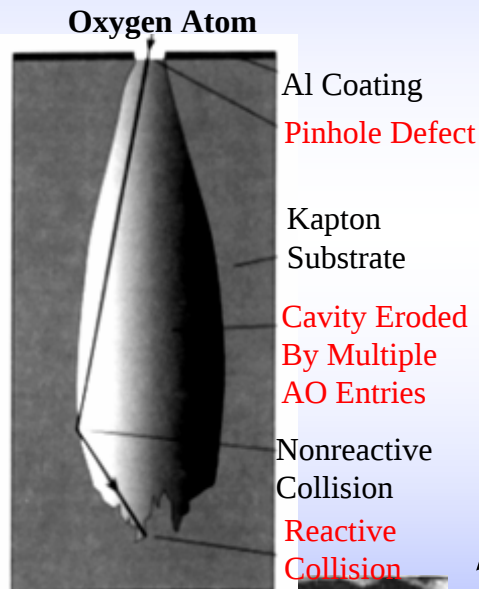
Kim K. de Groh and Bruce A. Banks
Spacecraft and Rockets, Vol. 31, No. 4, (1994)



MLI were flown 5.8 yrs in LEO on the **Long Duration Exposure Facility**.

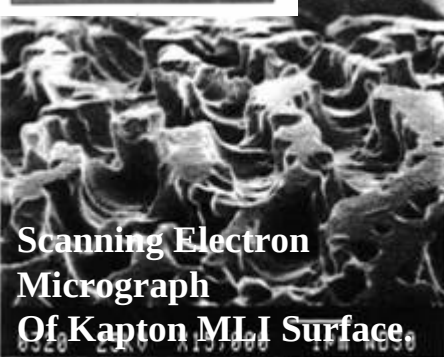
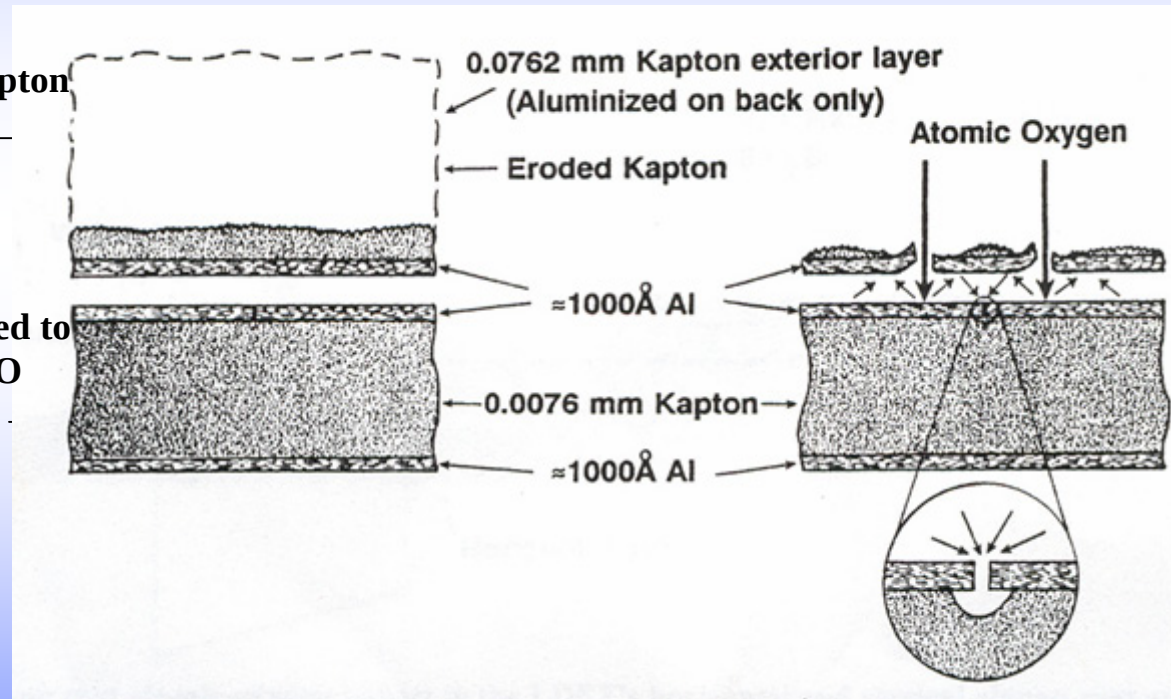
Total AO Exposure: 9×10^{21} atoms/cm²

95% of Al-Kapton underwent underpinning.



0.076mm Kapton eroded
 2.55×10^{21} O atoms/cm²

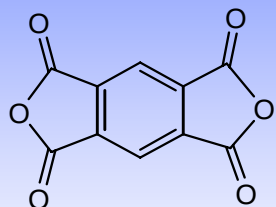
MLI exposed to
 6.44×10^{21} O atoms/cm²



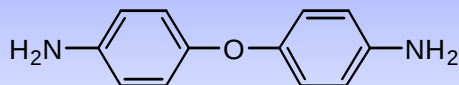
Fluence atoms/cm³ :
Eroded material divided by
Kapton erosion yield of 3×10^{21} cm³/atom.



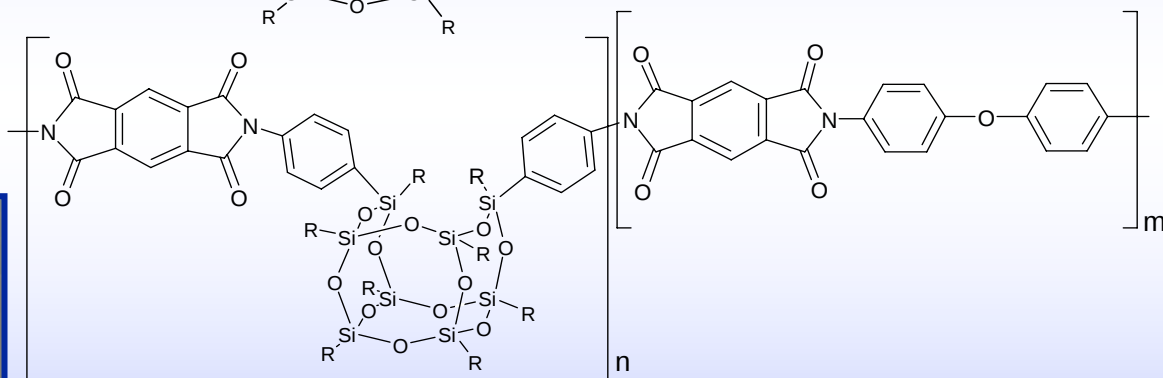
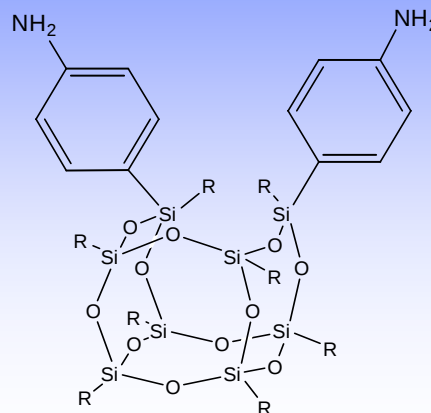
POSS - Kapton Polyimides



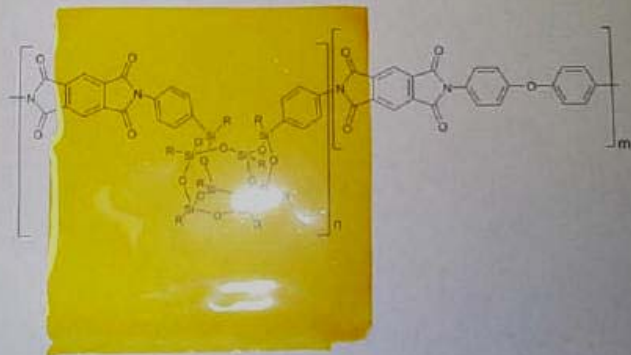
PMDA



ODA



- transparent films at 25 wt % POSS.



- POSS Polyimides do not lose rigidity above the glass transition temperature .
- Tg of POSS polyimides is 5 – 10 % lower than polyimides (414 °C).
- Room temperature modulus unaffected by POSS.
- High temperature modulus (above 430 °C) is increased with POSS content.

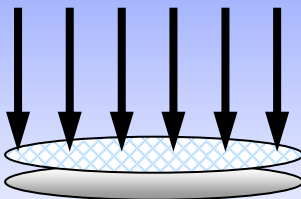


O-Atom Etching Experiment

Total AO fluence of 8.47×10^{20} atoms cm^{-2} (100,000 pulses)



Hyperthermal AO Beam (CO_2 laser, $\lambda = 4.93\text{-}8.42$ eV)



Screen
Sample

Kapton H Standard

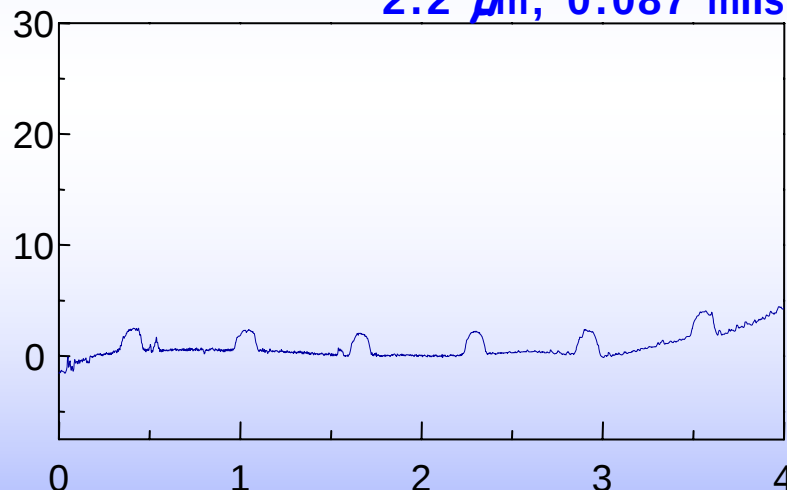
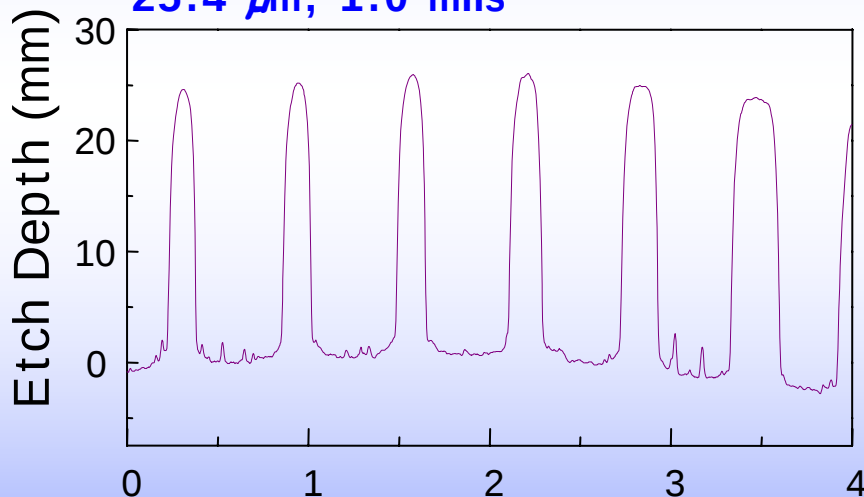
Average etch depth:

$25.4 \mu\text{m}$; 1.0 mils

Kapton 10 wt% (2 mole %) POSS

Average etch depth:

$2.2 \mu\text{m}$; 0.087 mils



Scanning Length (mm)

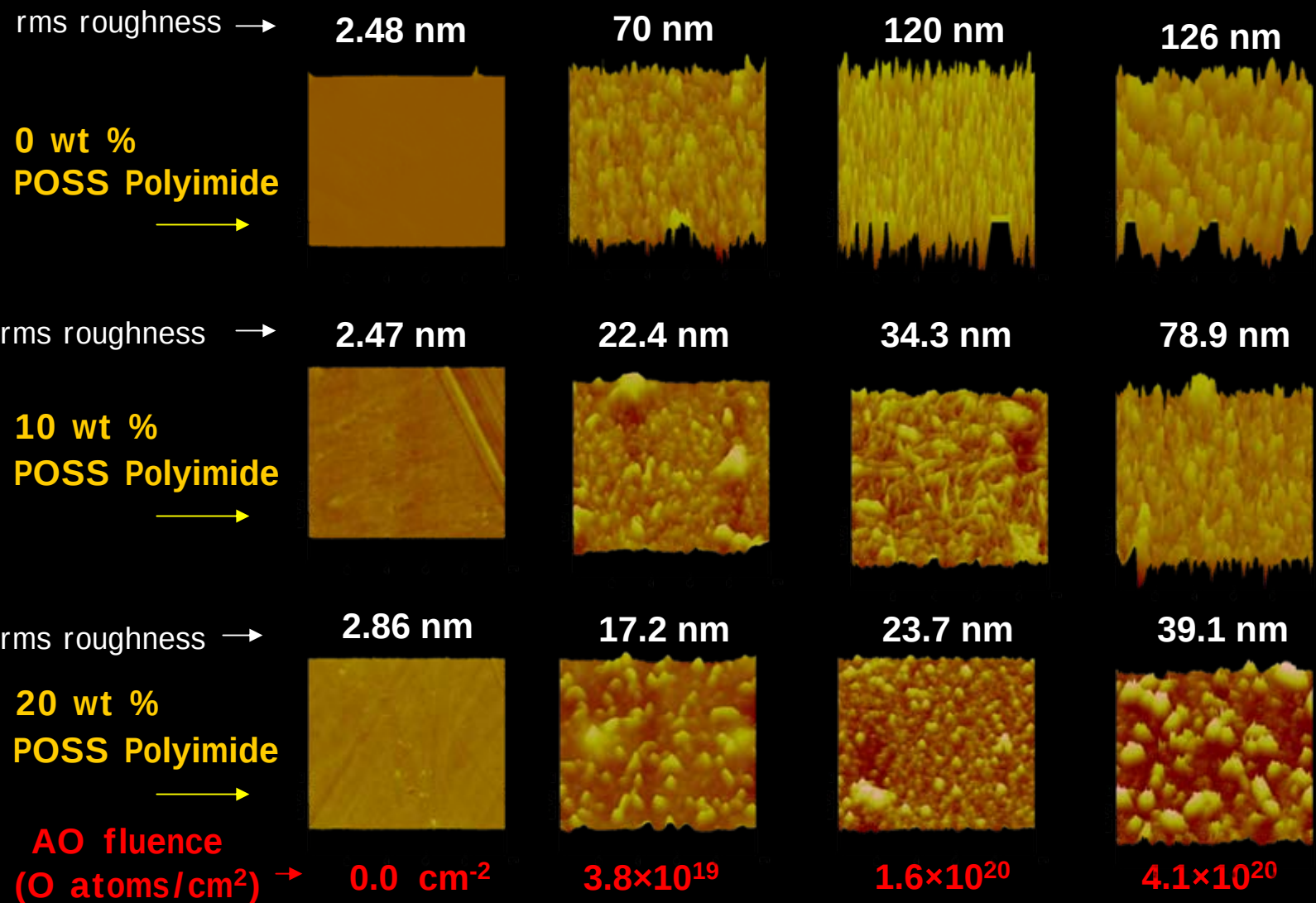
Significantly improved oxidation resistance due to a rapidly formed ceramic-like, passivating silica layer preventing further degradation of underlying virgin polymer.



AFM Images of POSS Polyimides With increasing AO Flux.



(10 × 10 μm; z scale = 500 nm)



Note:
1 × 10²⁰ O atoms/cm² is roughly equivalent to a spacecraft operating at 500 – 600 km orbit during nominal solar activity conditions for periods of at least 1 year.

Surface Atomic Concentrations (%) determined from XPS (X-ray Photoelectron Spectroscopy) Survey Scans before and after exposure to Atomic Oxygen.



Sample	Exposure (beam pulses)	Kapton-equivalent atomic oxygen fluence (10^{20} O atoms cm^{-2})	C	O	Si	N
0 wt% POSS polyimide	0	0	72	19.5	1	7
	6	~0.1	69	20	2	9
	100	1.63	69	24	1	6
	250	4.10	55	36	0	9
10 wt% POSS polyimide	0	0	77	16	2	5
	6	~0.1	73	18.5	5	3.5
	100	1.63	48	30	19	3
	250	4.10	20	56	23.5	0.5
20 wt% POSS polyimide	0	0	70	20	6	4
	6	~0.1	66	24	7	3
	100	1.63	20	54	25	0
	250	4.10	12	60	26	1

Calculated at%: 0 wt% POSS PI: C = 75.9, O = 17.2, Si = 0, N = 6.9

10 wt% POSS PI: C = 75, O = 17.2, Si = 1, N = 6.4.

20 wt% POSS PI: C = 75, O = 17.2, Si = 1.8, N = 6.0



Erosion of POSS Polyimides by a Beam of Hyperthermal (5eV) O Atoms



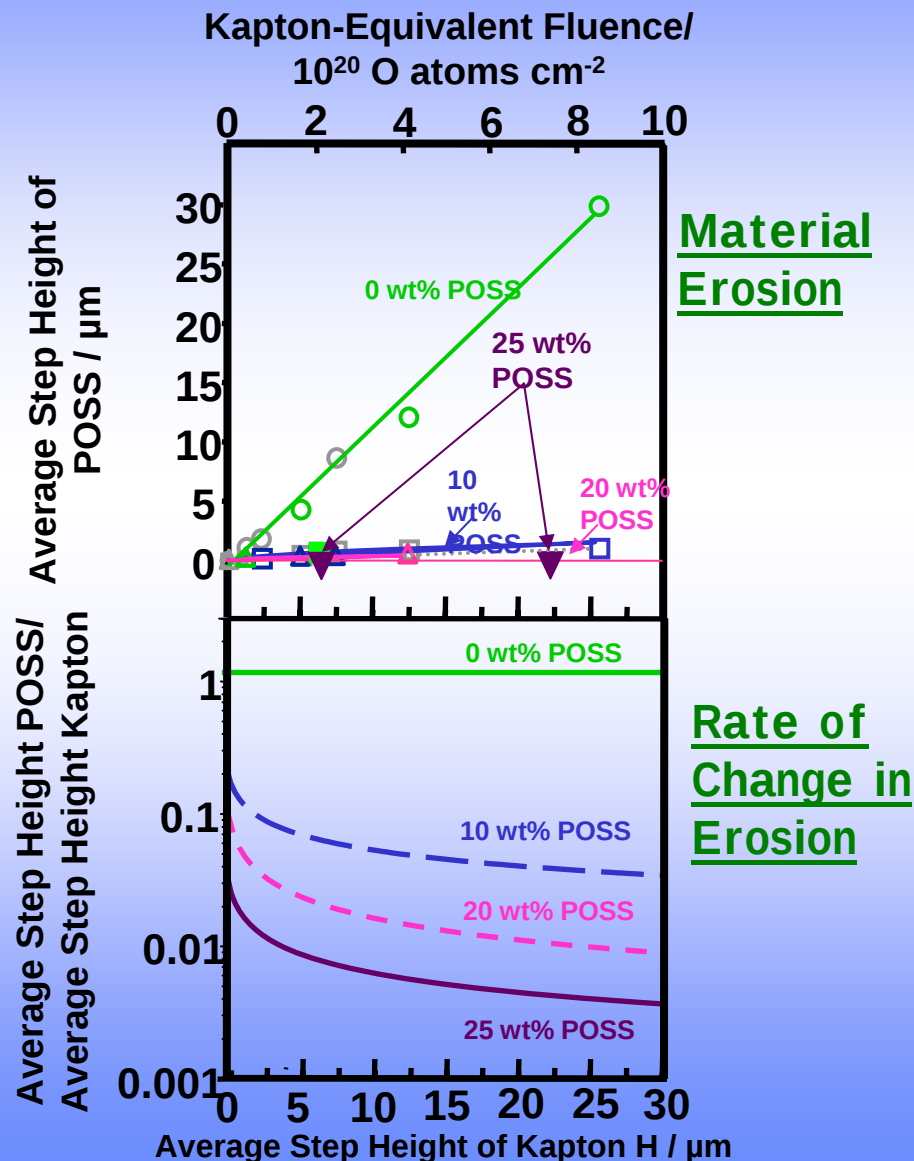
1st AO Erosion experiment:

The erosion rate of the 10 and 20 wt % POSS Polyimide samples were 3.7 and 0.98 percent, respectively, of the erosion rate for Kapton H at the highest fluence used in this experiment (8.5×10^{20} atoms cm^{-2}).

2nd AO Erosion Experiment:

The new 25 wt% POSS polyimide samples had an erosion rate that was 0.3 percent of the erosion rate for Kapton H at a fluence of 8.5×10^{20} atoms cm^{-2} .

This erosion rate is one third that of the previously synthesized 20 wt% POSS polyimide.





Self-passivation of POSS Polyimide Upon Exposure to 2.3×10^{20} O atoms cm^{-2} .

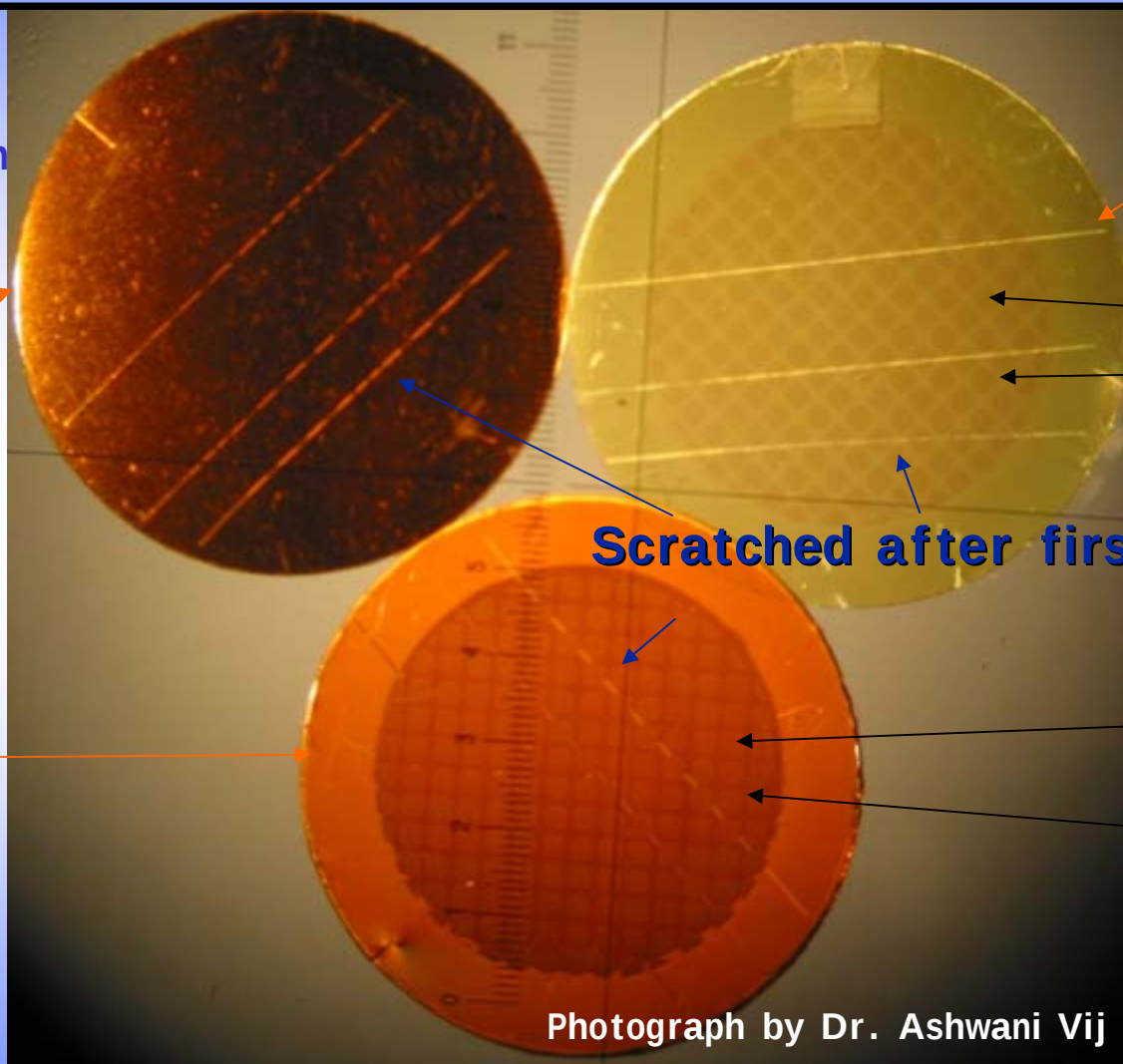


Dr. Timothy Minton
Amy Brunsvold

**SiO₂ (130 nm)
/ Kapton (2 mil)
/ Al (100nm)**

**Commercial
Kapton H**

Sample diameter
= ½ inch.



**25 wt %
POSS Polyimide**

**AO Exposed
Protected
from AO**

Scratched after first AO exposure

**AO Exposed
Protected
from AO**

Photograph by Dr. Ashwani Vij

Screen-protected samples were exposed to 2.3×10^{20} O atoms cm^{-2} , unprotected, scratched with a diamond scribe 1 μm deep, screen-protected, and re-exposed to 2.3×10^{20} O atoms cm^{-2} .



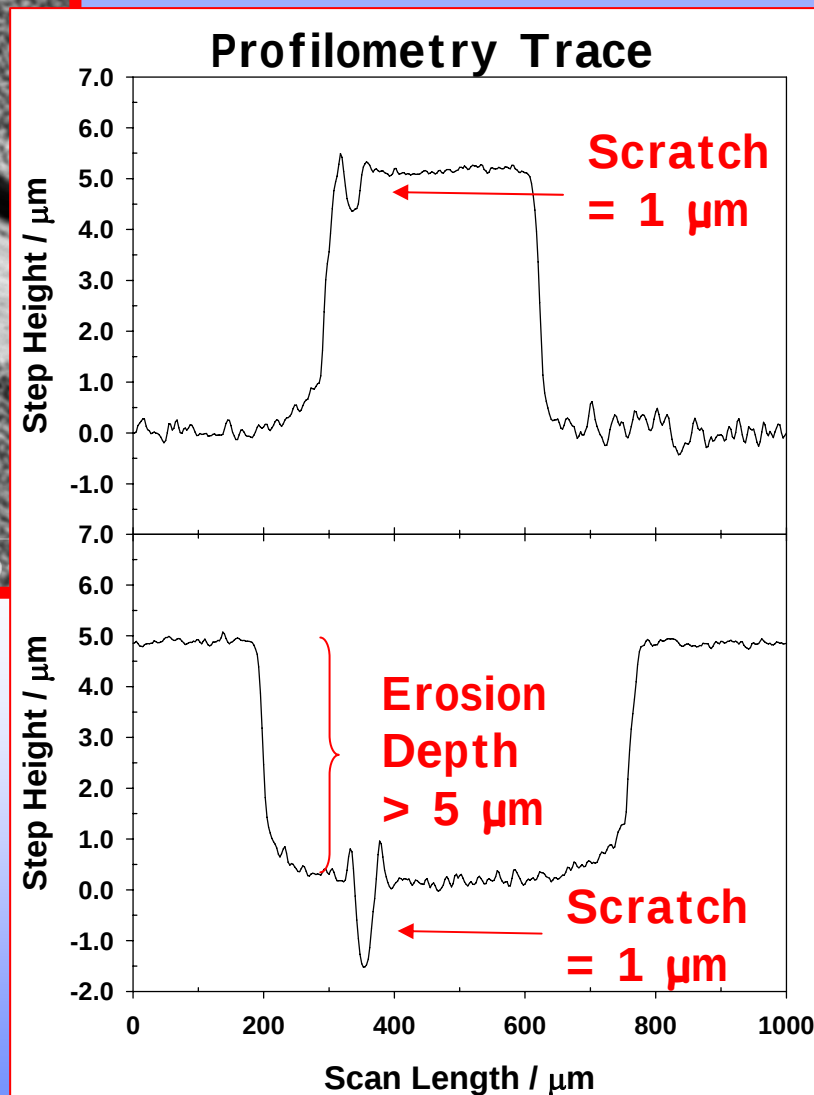
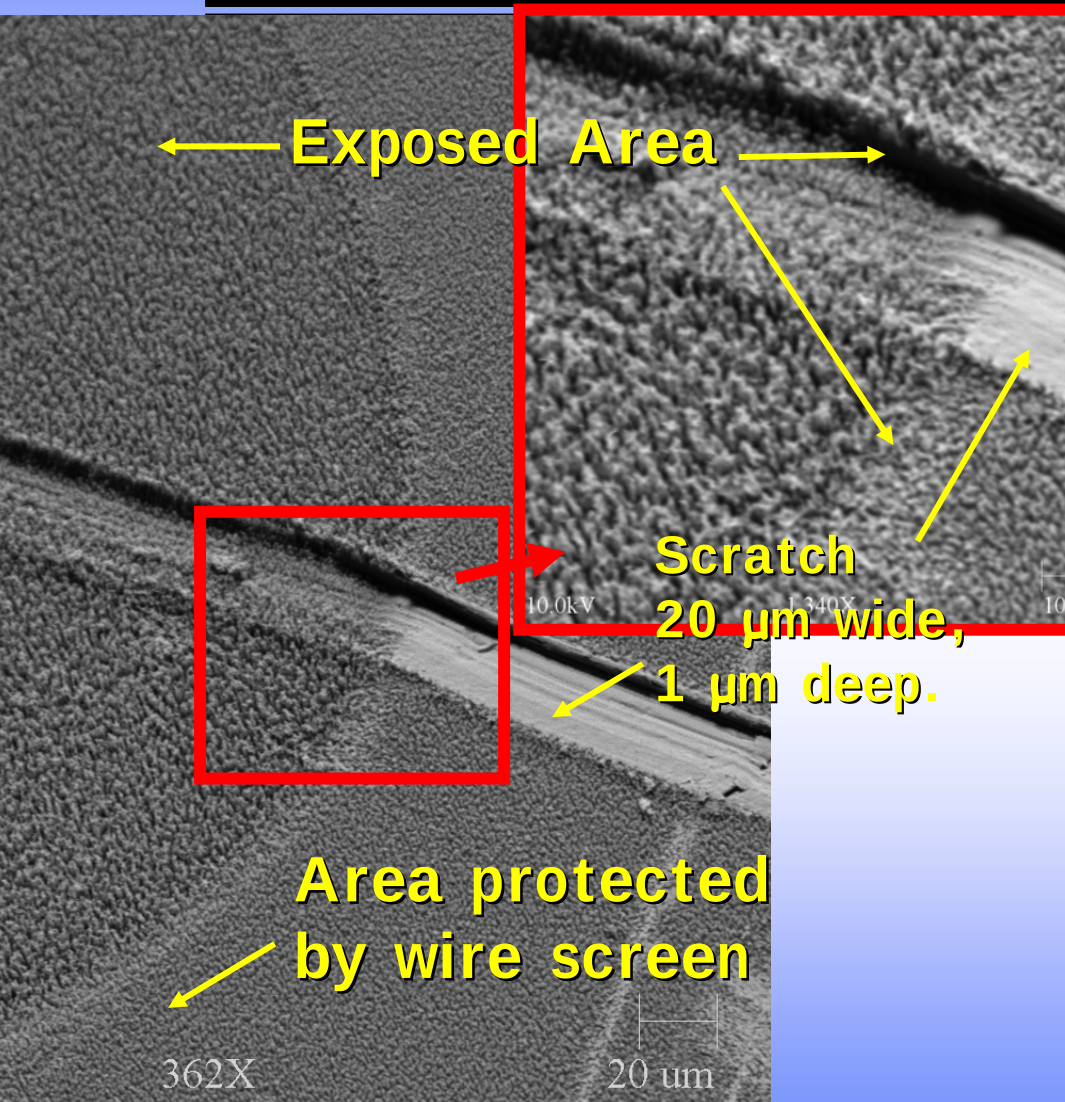
Summary of Self-Passivation Study



	SiO ₂ coated Kapton HN with Al under-coating	Kapton H	25 wt % POSS polyimide
Erosion depth after 1 st exposure to 2.3x10 ²⁰ O atoms cm ⁻² .	~ 0 μm	5 μm	0.200 μm
All samples were scratched 1 μm deep.			
Erosion depth outside of the scratch after 2 nd exposure to 2.3x10 ²⁰ O atoms cm ⁻² .	~ 0 μm	5 μm	~ 0 μm
Erosion depth inside of the scratch after 2 nd exposure to 2.3x10 ²⁰ O atoms cm ⁻²	7 μm	5 μm	0.200 μm



SEM of Kapton H surface after the Second Atomic Oxygen Exposure



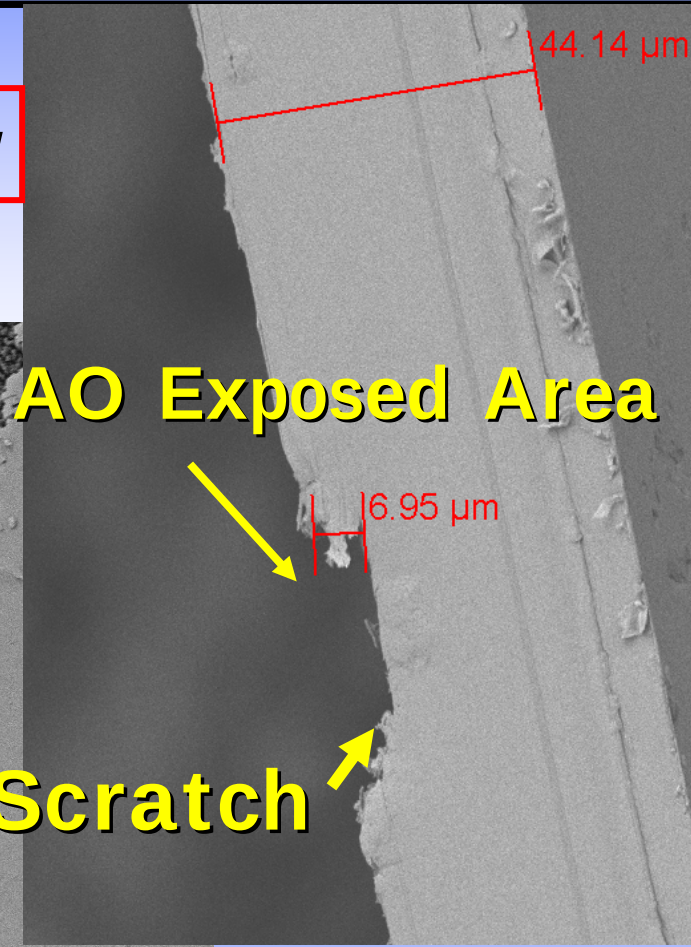
SEM by Marietta Fernandez,
Edwards AFRL



SEM Images of Scratch on SiO_x Coated Kapton After Second AO Exposure



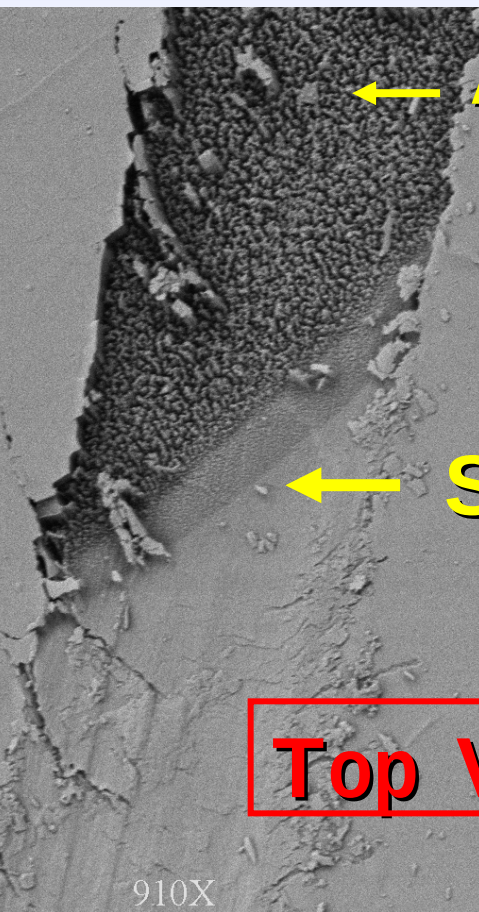
Side View



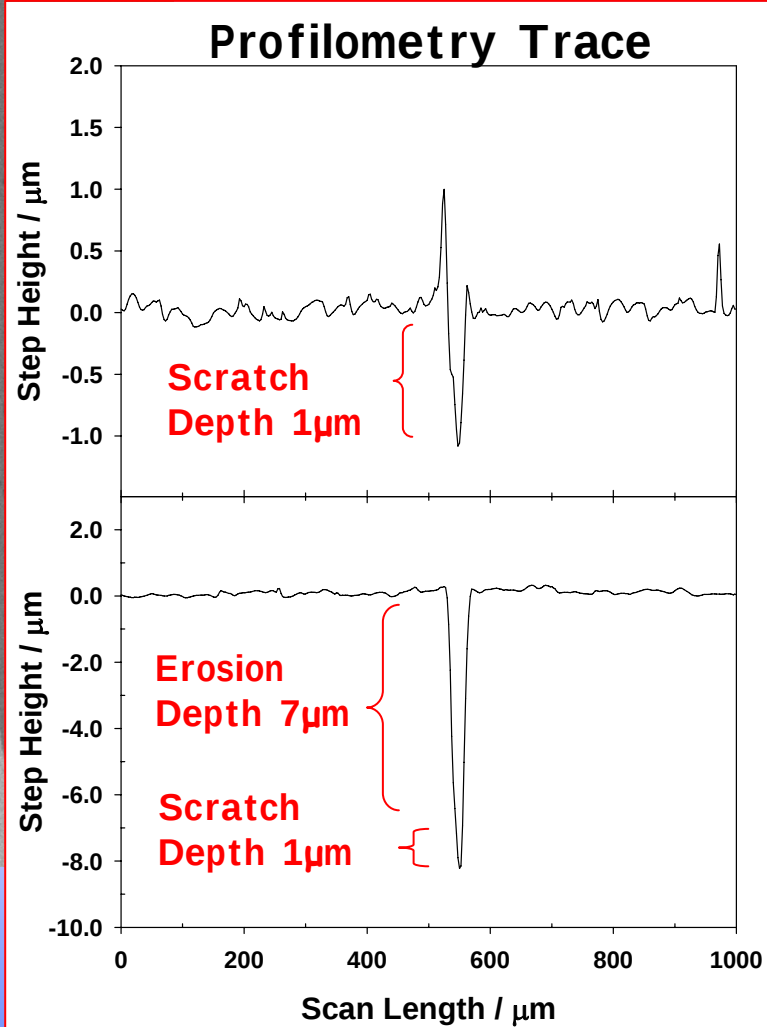
AO Exposed Area

Scratch

Top View

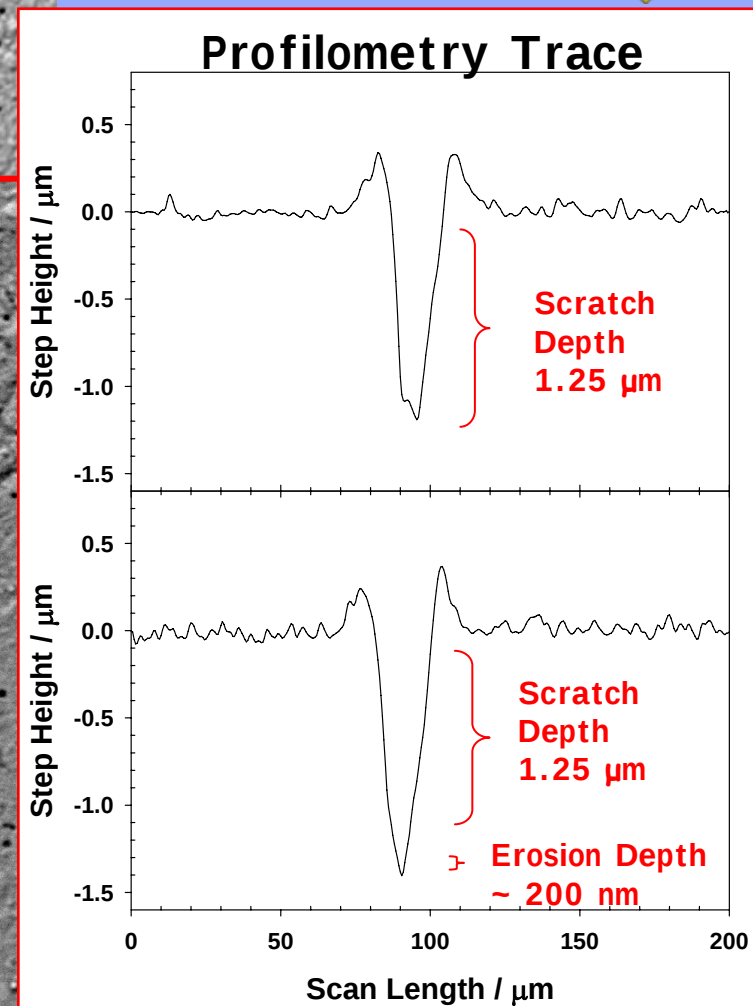
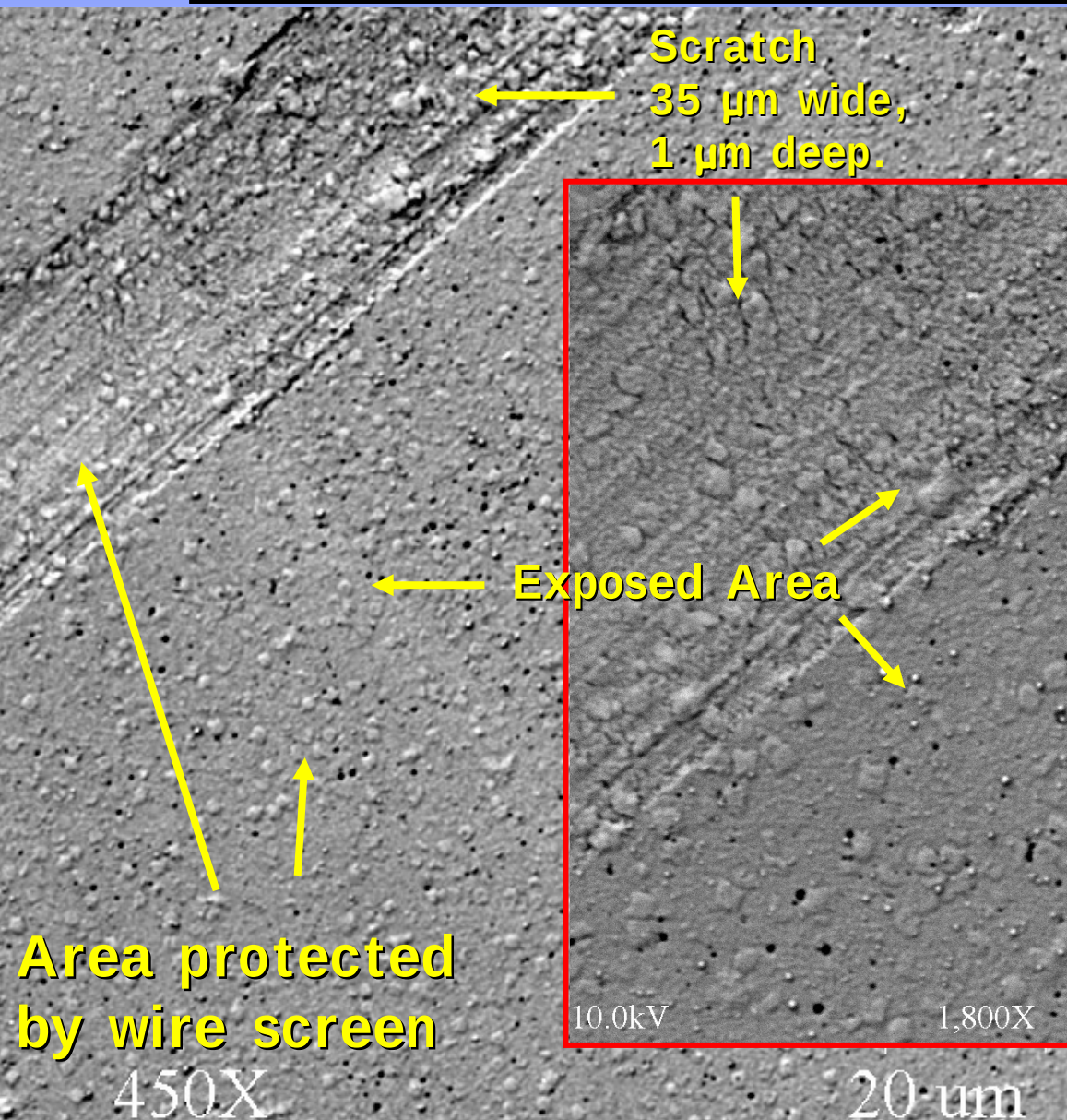


SEM by
Marietta Fernandez
Edwards AFRL





SEM of 25 wt % POSS Polyimide Surface After the Second Atomic Oxygen Exposure



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SEM by Marietta Fernandez,
Edwards AFRL



New POSS Monomer

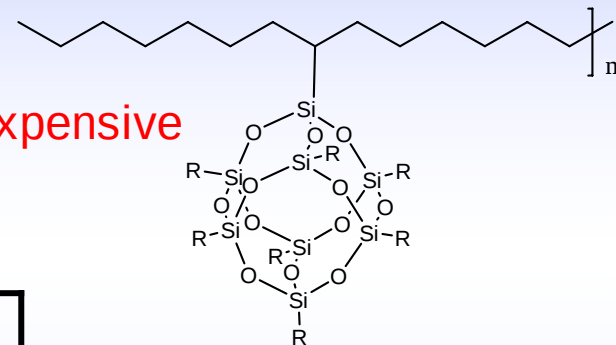


- China Lake Naval Air Warfare Center, Weapons Division collaborators:
Dr. Michael Wright, Dr. Brian Petteys, Dr. Andy Guenthner, Dr. Gregory Yandek.

Recently synthesized:

Side-chain POSS diamine monomer which is relatively **inexpensive**
and of **facile synthesis**.

Resultant POSS Polyimides are **transparent flexible films**.



Sample	Kapton-Equivalent Fluence/ 10^{20} O atoms cm^{-2} .	Erosion Yield
Kapton H	4.10	12.3
20 wt % main-chain POSS polyimide	4.10	0.47 Is 3.8 % of that of Kapton H.
Kapton H	3.53	10.6
7 Si ₈ O ₁₂ wt % side-chain POSS polyimide.*	3.53	0.35 Is 3.3 % of that of Kapton H.

**AO resistance is
similar between
main-chain and
side-chain POSS
polyimides.**

* equivalent
SiO content to 20 wt %
main-chain POSS PI



Acknowledgments



Polymer Working Group: Dr. Rusty Blanski, Mr. Pat Ruth,
Mrs. Sherly Largo, Ms. Sarah Mazzella, 2Lt. Amy Palecek, 2Lt. Laura Moody.

Previous Group Member: Capt. Rene Gonzalez, Ph. D.

Branch Chief: Dr. Steve Svejda

Collaborators:

Hybrid Plastics: Dr. Joesph Lichtenhan

China Lake Naval Weapons Center: Dr. Mike Wright, Dr. Andy Guenthner,
Dr. Brian Pettys, Dr. Gregory Yandek

Michigan State University: Dr. Andre Lee

Funding :

Air Force Office of Scientific Research, Dr. Charles Lee

Defense Advanced Research Projects Agency, Dr. Leo Christodolou

