

Polymers for Space Applications Processed by Plasma Immersion Ion Implantation

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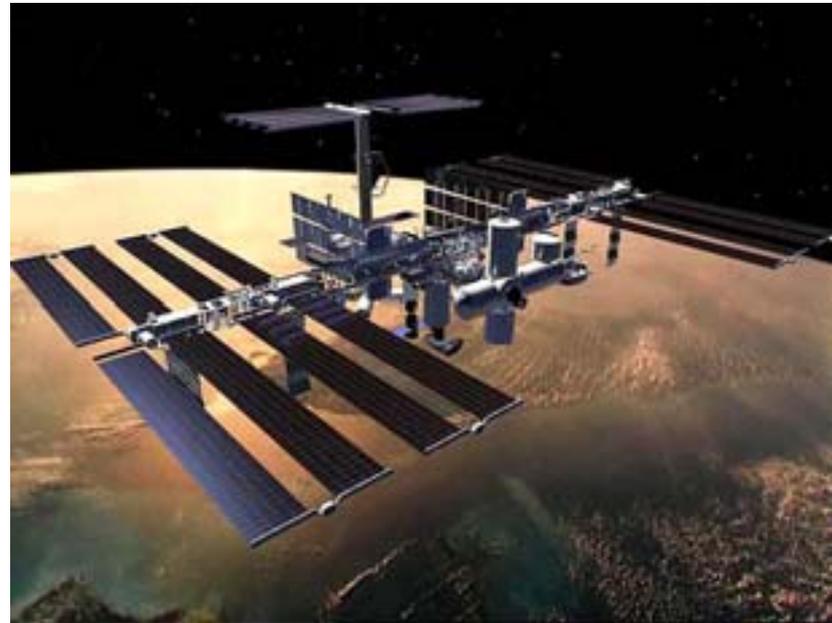
Protection of Components for Spacecrafts Orbiting LEO

Low Earth Orbit (LEO) environment (180-650 km) : rich in atomic oxygen, degrades polymeric materials (like Kapton, Mylar or Teflon) used in satellites.

May erode certain polymers
by over 2 μm in 90 days

Oxygen resistant polymers could improve the lifetime of satellites and space stations and could find many applications in space, including huge fold-up antennas, inflatable mirrors & lenses, solar sails...

Kapton is extensively used in thermal blankets



International Space Station
orbiting LEO region (~ 450 km)

Oxidation protection: Thin layers of several metal oxides such as Al_2O_3 , MgO , or SiO_2 are being studied as protective coatings for polymers in LEO

Thermal transients (-100°C to $+100^\circ\text{C}$): Superior adhesion of the thin film is required \emptyset **Plasma Immersion Ion Implantation and Deposition (PIII)** of metal ions is ideal.

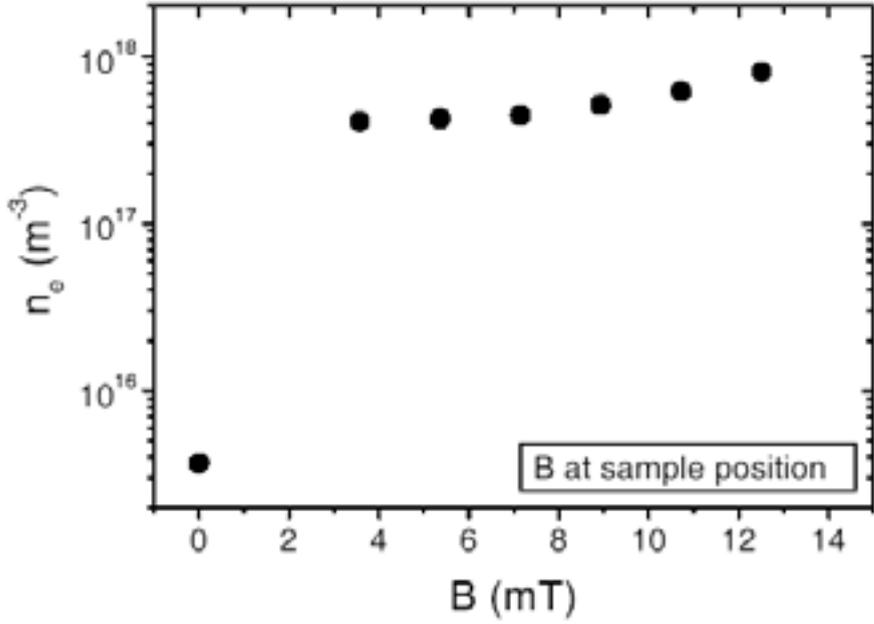
PIII in polymers: charging of the dielectric is proportional to plasma density

Typically for $\sim 20\ \mu\text{m}$ thick polymers:

$n \sim 10^{17}\ \text{m}^{-3}$ \emptyset $\Delta V \sim 7\text{kV}$ in $2\ \mu\text{s}$

$n \sim 10^{15}\ \text{m}^{-3}$ \emptyset $\Delta V \sim 700\text{V}$ in $60\ \mu\text{s}$

In metal plasmas generated by vacuum arcs: Magnetic field increases plasma density by two orders of magnitude



Objectives

Aluminum implantation in Kapton® by three different methods

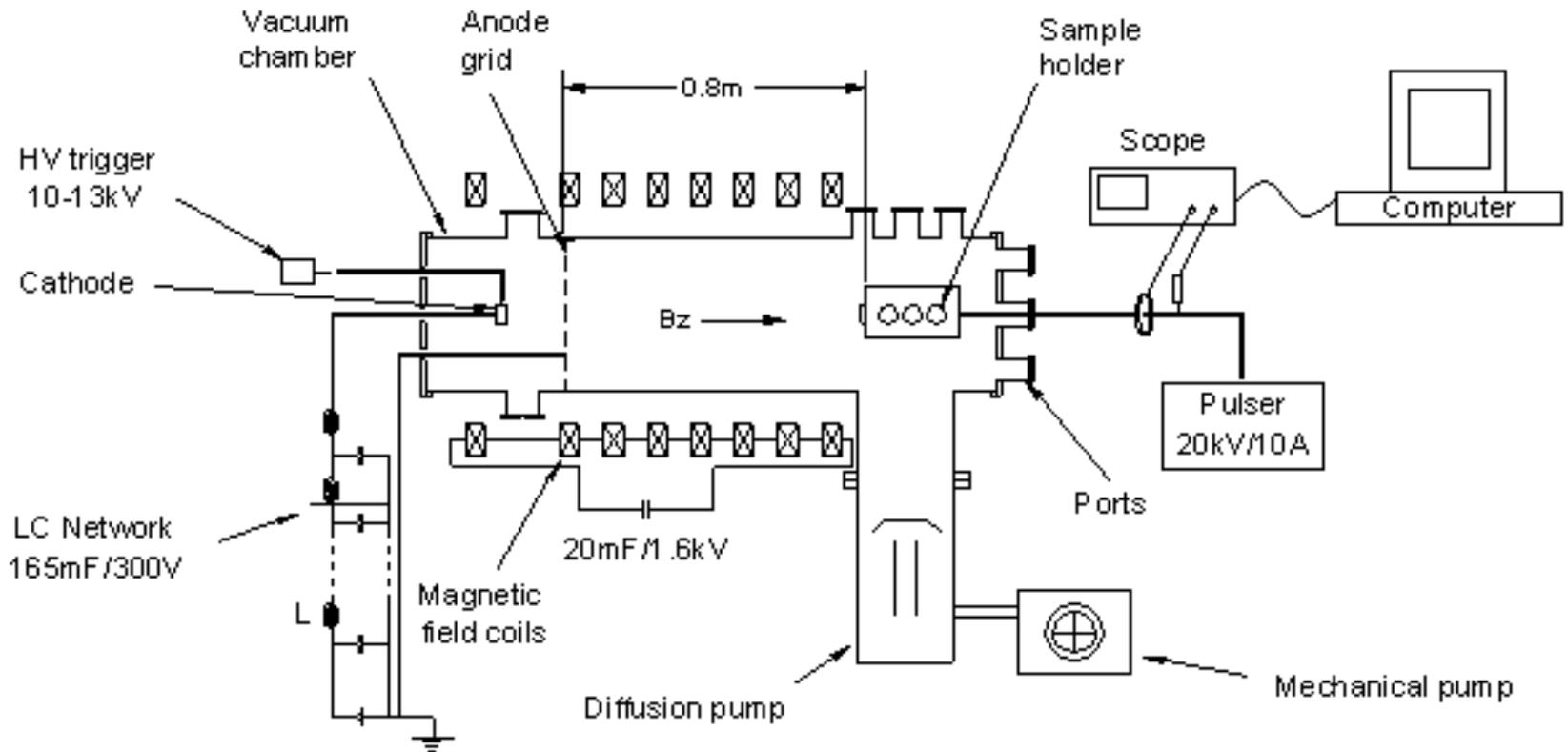
- Direct implantation in a magnetized Al plasma
- Direct implantation in an unmagnetized Al plasma
- Al deposition + implantation in nitrogen plasma (recoil implantation)

Resistance tests for space environment

- Oxygen degradation (oxygen plasmas)
- Thermal cycling
- Adhesion test

Direct Aluminum implantation

Experimental Set-up



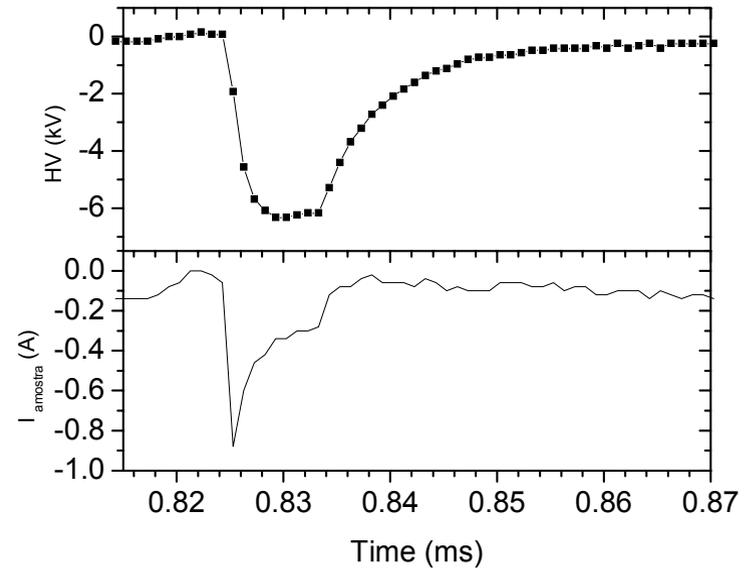
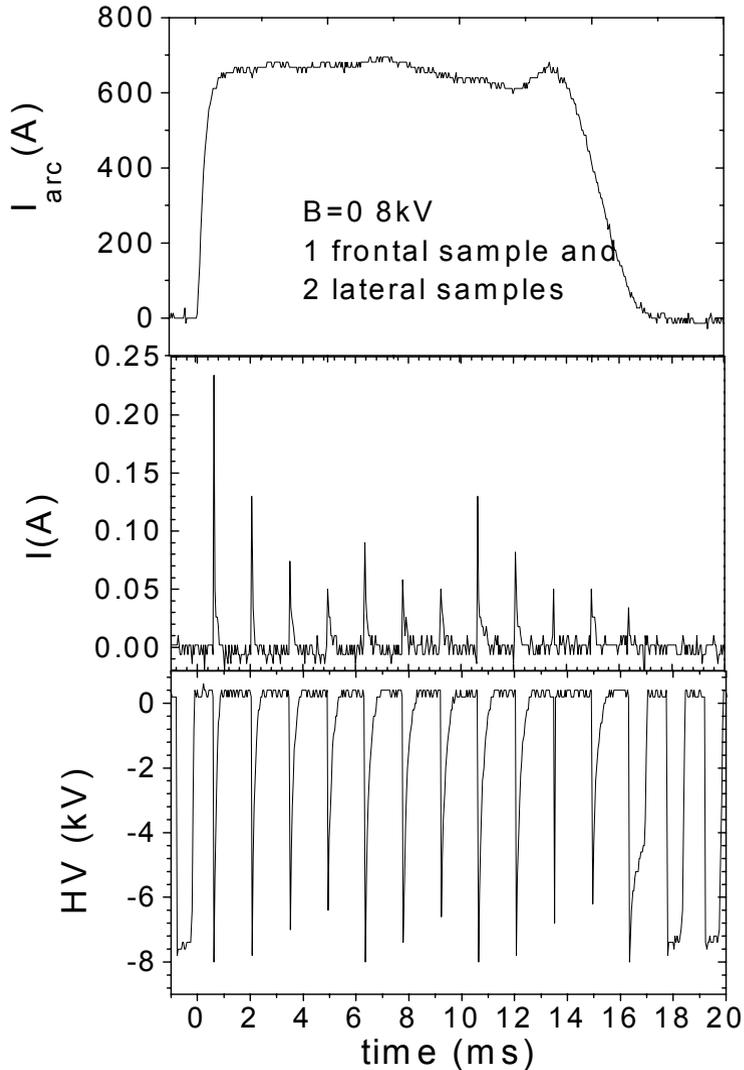
Vacuum Arc: Al cathode Tungsten grid anode
HV trigger : 10-13 kV

Vacuum chamber $\phi=0.22\text{m}$, $L=1.05\text{m}$
Base pressure $\sim 1 \times 10^{-4}$ Pa
B field : 150G-7kG

Sample holder: 85cm from cathode

Straight magnetic filter: not so good filtering but good plasma transport. Macroparticles avoided and deposition minimized by orienting samples **parallel to plasma stream**

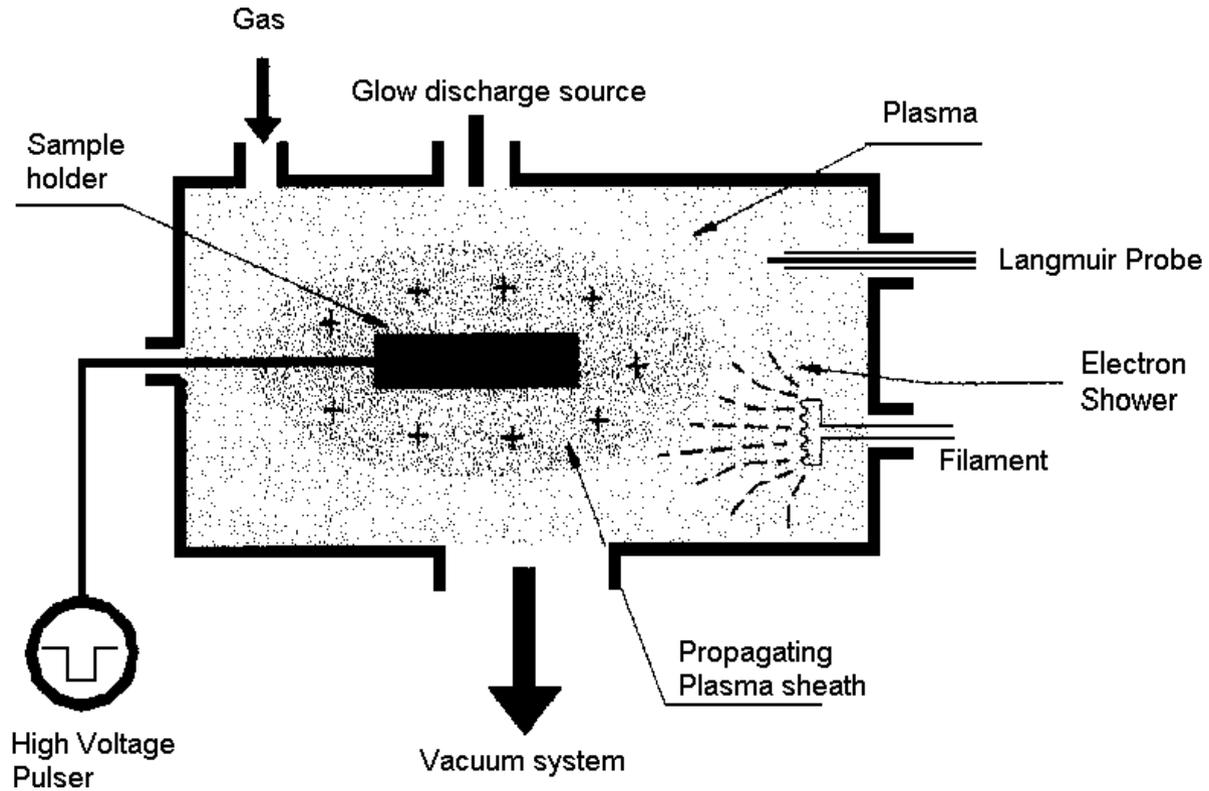
Implantation Conditions



With $B = 125$ G $I_{\text{arc}} = 1$ kA
7 μ s pulses, 2.5 kV,
900 Hz (13-14 pulses / discharge)
100 discharges

With $B = 0$ $I_{\text{arc}} = 1$ kA
7 μ s pulses, 6 kV, 900 Hz
800 discharges

Recoil Aluminum implantation



200 Å, 500 Å and 2000 Å aluminum films deposited by electron beam on Kapton samples followed by

Implantation in **Nitrogen** ($n \sim 10^{10} \text{ cm}^{-3}$, $T_e < 10 \text{ eV}$) and **argon** plasmas.

HV pulses: 5 μs , 100Hz, 5 kV

Treatment time of 30 minutes

Analysis

Elemental composition and morphology

- RBS
- XPS
- SEM , EDS

Oxygen degradation

- Oxygen plasma: 40kHz parallel plate capacitive reactor
- 200 mTorr, 200W (10^{10} cm⁻³, 1-2 eV), ~ one hour exposure

Transmittance and Reflectance (Hitachi U-3501 spectrophotometer)

Thermal cycling

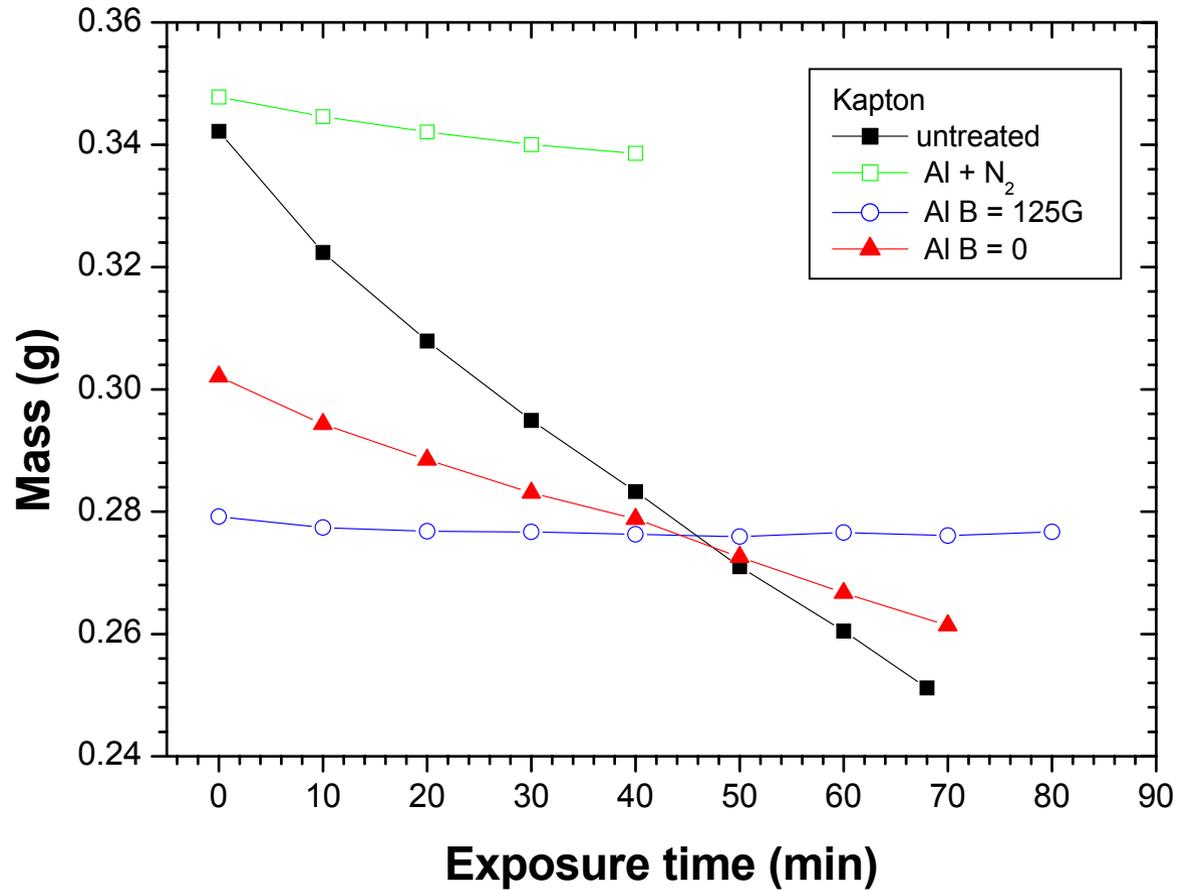
- 1 minute liquid nitrogen immersion (-196 °C)
- 1 minute pre-heated oven (100 °C)
- 15 cycles

Adhesion Test

- applying and removing a pressure sensitive tape + SEM

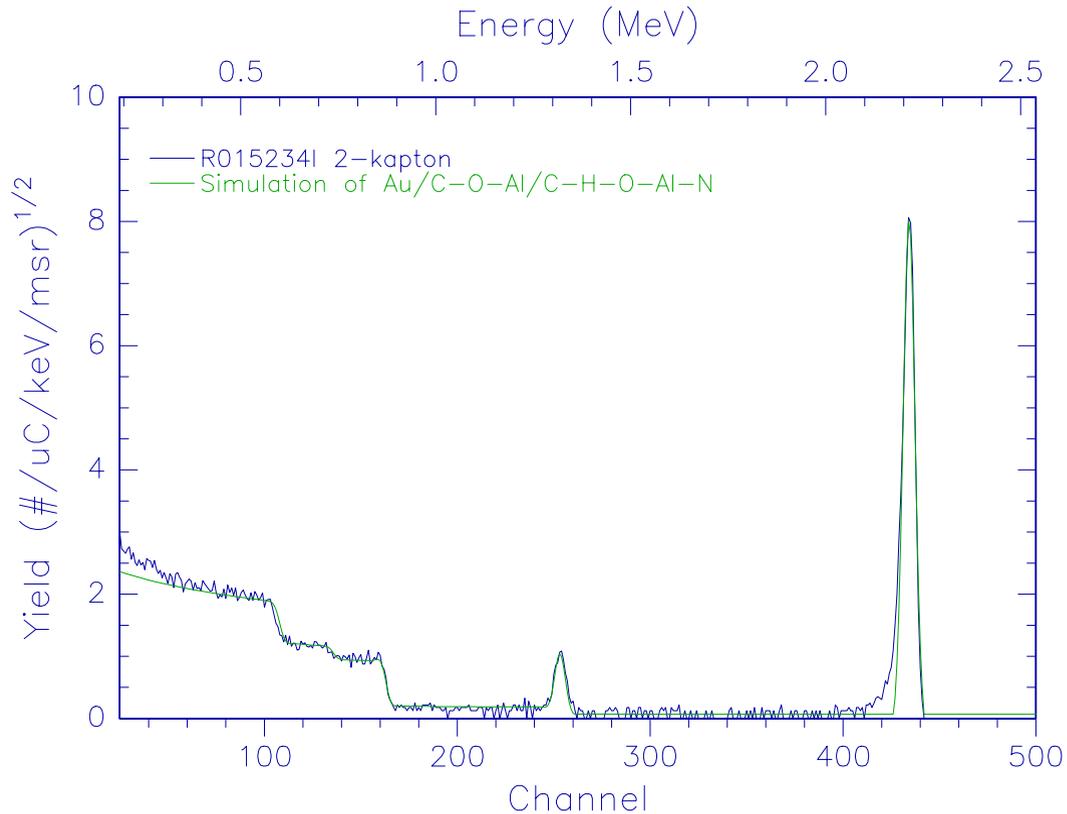
Results

Oxygen Degradation

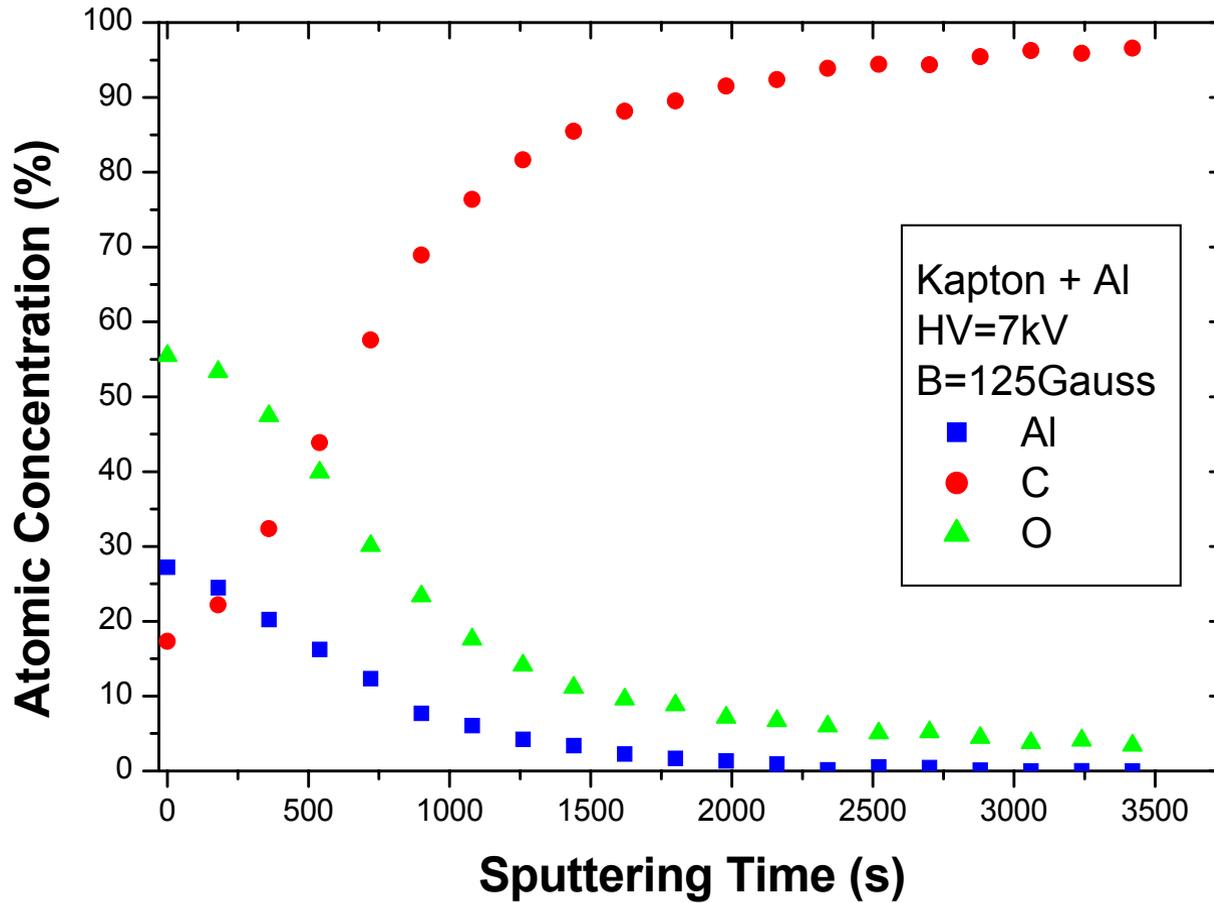


Direct Implantation in Magnetized Plasmas

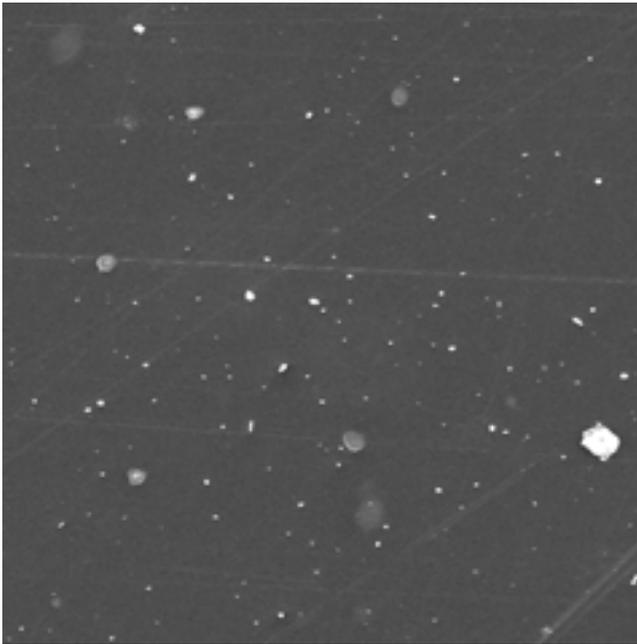
RBS \bar{A} retained doses of 10^{16} atoms/cm², but mostly at the surface



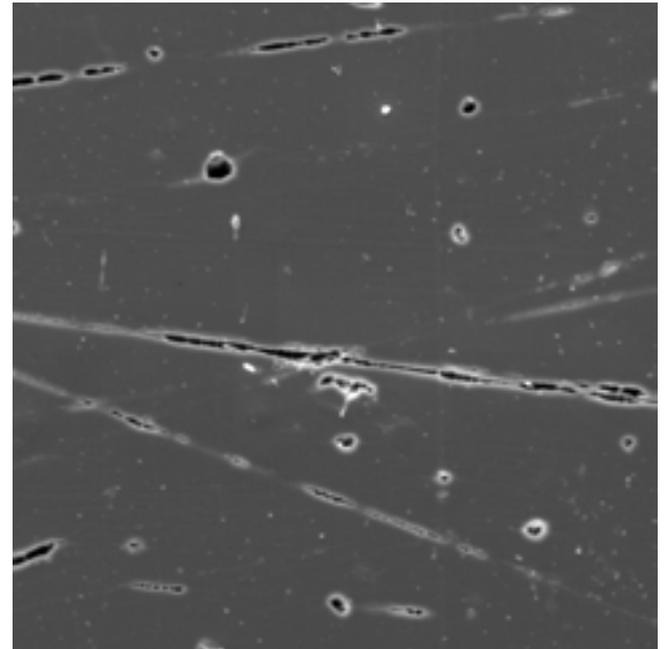
XPS Δ formation of an ion mixing layer



SEM : morphology conserved after oxygen degradation, thermal cycling and adhesion tests

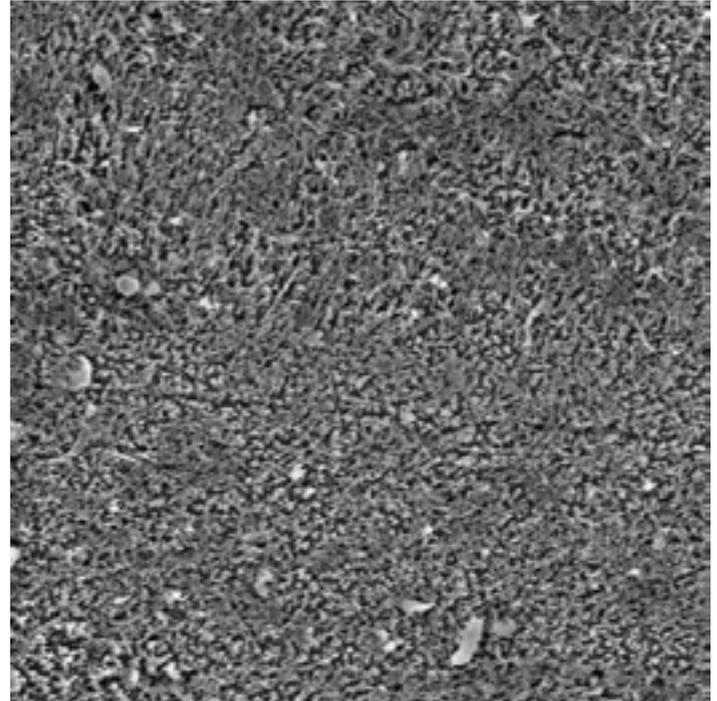


Pristine Kapton

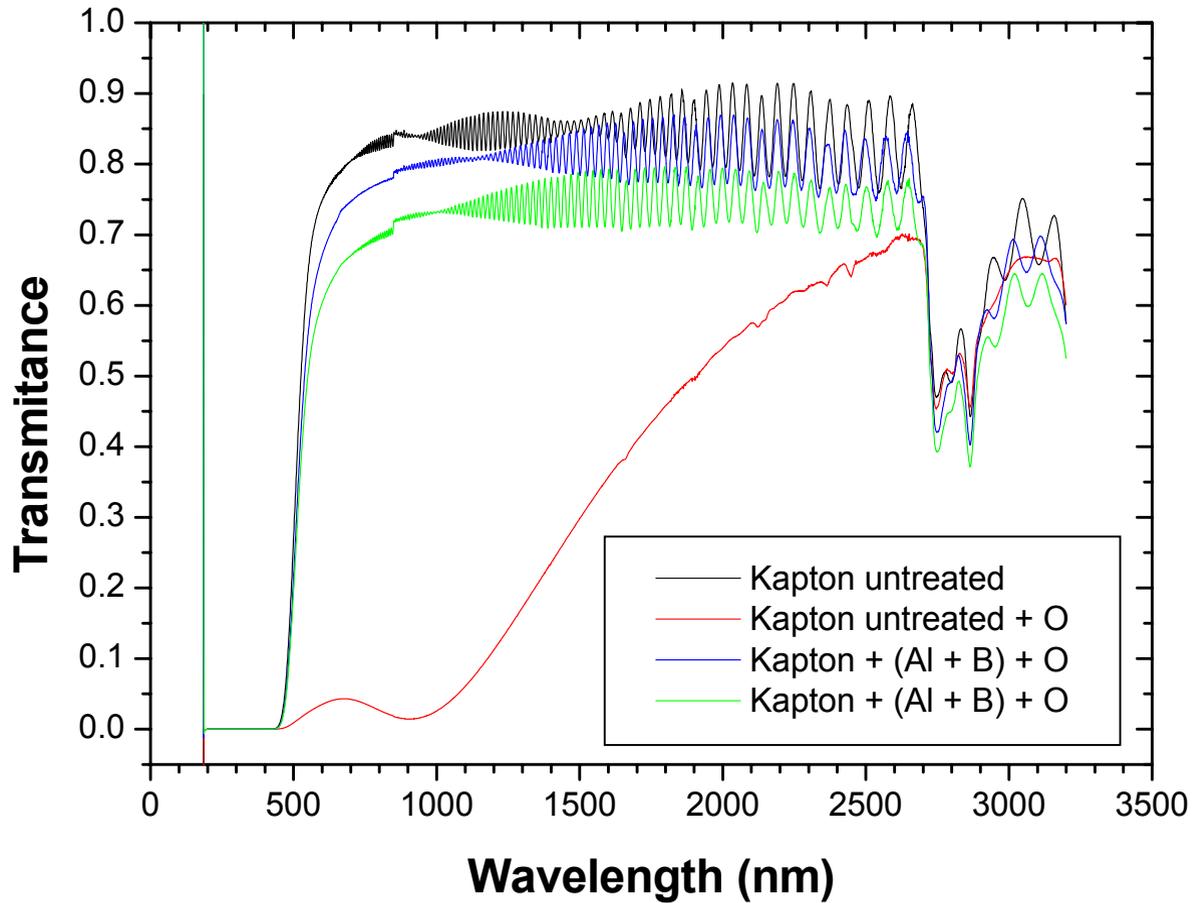


After tests

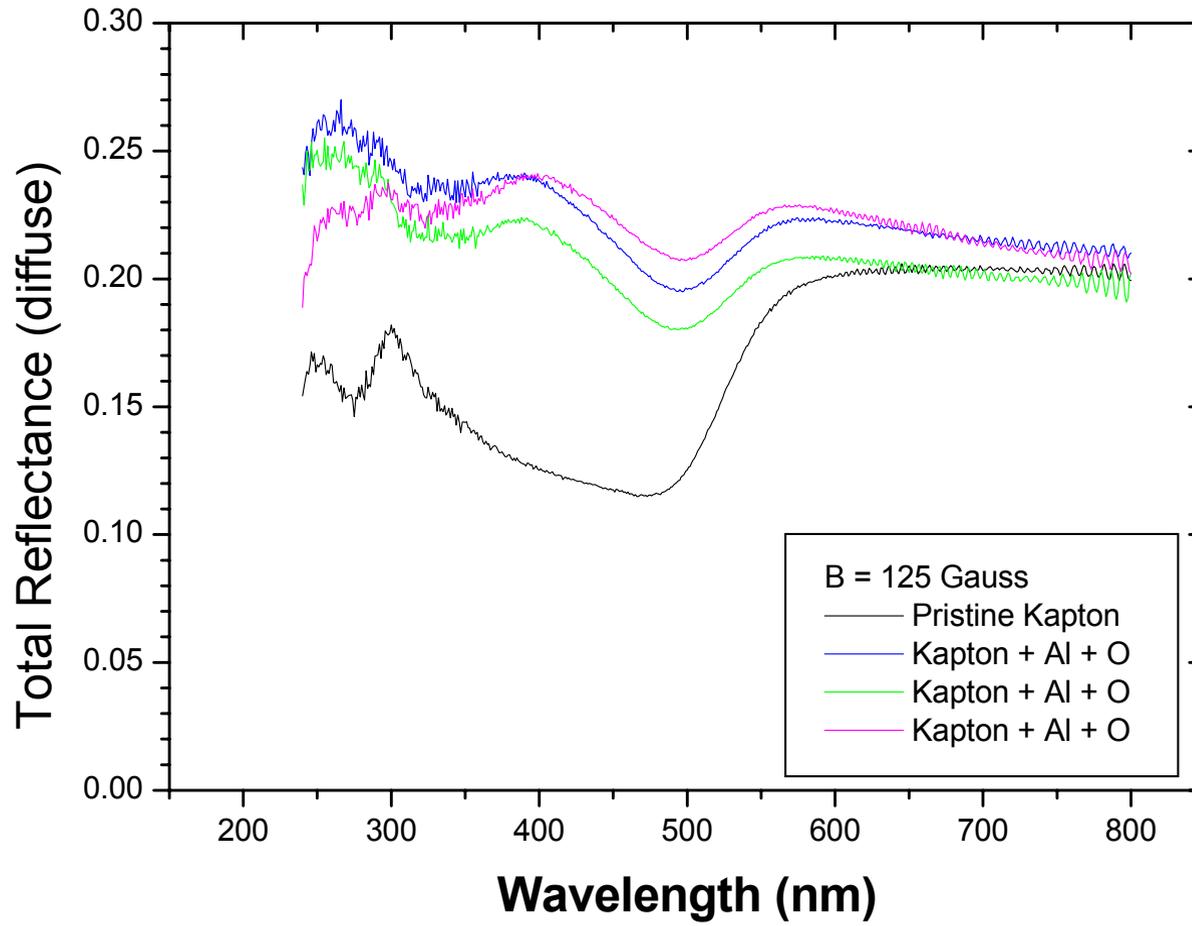
Without treatment,
after oxygen exposure



Transmittance decreases only by (5-15)%



Total reflectance increases

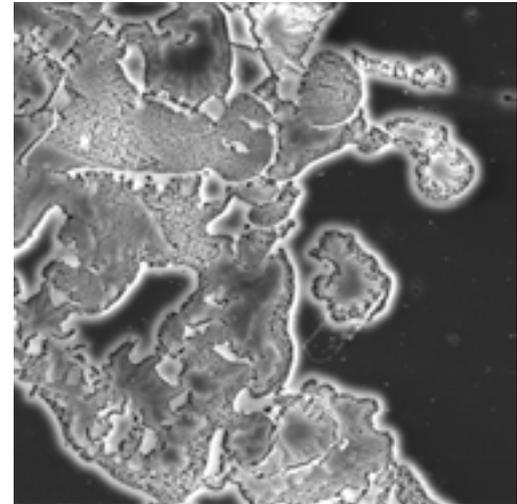
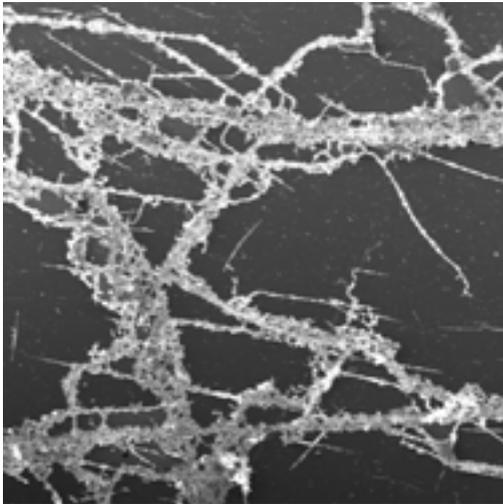


Direct Implantation in Unmagnetized Plasmas

- Treatment time increased eightfold was not enough to compensate the plasma density decreased by two orders of magnitude without magnetic field. This resulted in **lack of uniformity**
- Treated samples had “good” and “bad” parts.
- “Good” parts behaved in the same way as samples treated in magnetized plasmas: no oxygen degradation revealed by conserved transparency, and conserved morphology after oxygen exposure, thermal cycling and adhesion tests.
- “Bad” parts behaved like untreated samples: “carpet” like morphology after oxygen exposure, loss of transparency.

Deposition and recoil implantation

- For 200 Å and 500 Å depositions (but not for 2000 Å films), nitrogen and argon implantation results in a cracked film.



- Cracking of the deposited film is not caused by differences in thermal expansion since it does not occur after oven heating at 100 °C or even at 200 °C.
- Immersion of the deposited samples in nitrogen or argon plasmas does not result in cracking, which occurs as soon as the high voltage pulses are turned on.
- In direct implantation process, an aluminum film is also deposited without cracking, probably due to **ion induced stress relief**.
- Cracking in recoil implantation could be related to the formation of a stressed aluminum nitride (with nitrogen plasmas) and a stressed aluminum dioxide (which occurs in argon plasmas, even with very low oxygen contamination) films, although ion induced stress relief would be expected in this case as well.
- We have no conclusive explanation for the observed **ion induced cracking**.

Conclusions

- Kapton samples implanted with Al in a magnetized vacuum arc discharge resulted in excellent protection of the polymer against oxygen degradation.
- Retained doses of 10^{16} atoms/cm² was obtained, and although most of the atoms are concentrated on the surface, an intermediate ion mixing layer was formed.
- Adhesion test after thermal cycling shows good adhesion to the substrate.
- Implantation with Al in unmagnetized plasmas produces a protection layer as effective as in the magnetized case, but needs much longer treatment times, incompatible with present machine configuration.
- Al deposition by e-beam, followed by recoil implantation in nitrogen and argon plasmas resulted in a cracked film, induced by ion bombardment. No conclusive explanation has been found for this observation.