

# ***Treatment of Polymers by Plasma Immersion Ion Implantation for Space Applications***

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# Outline

- **Motivation**
- **Objectives**
- **Direct AI implantation**
- **Recoil AI implantation**
- **Analysis**
- **Results**
- **Conclusions and Future Work**

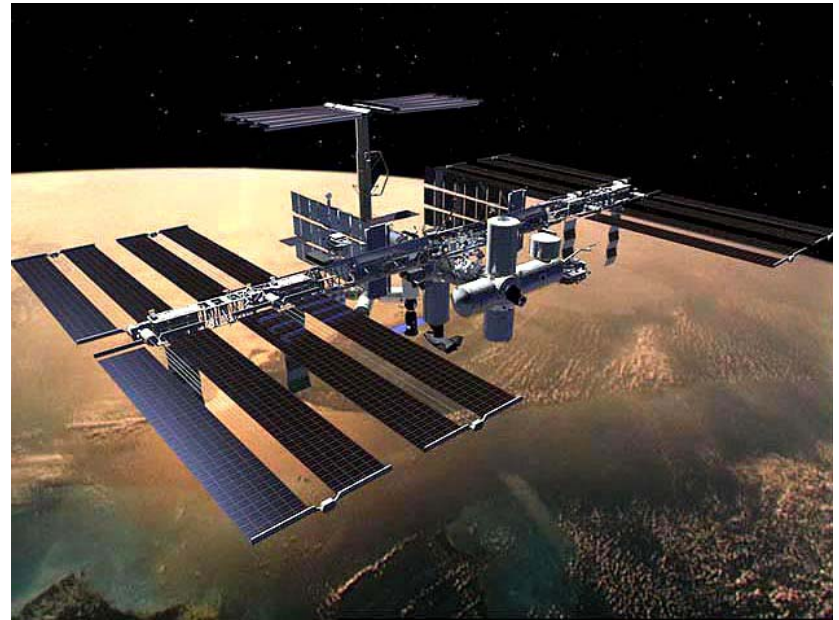
# Motivation

## Protection of Components for Spacecrafts Orbiting LEO

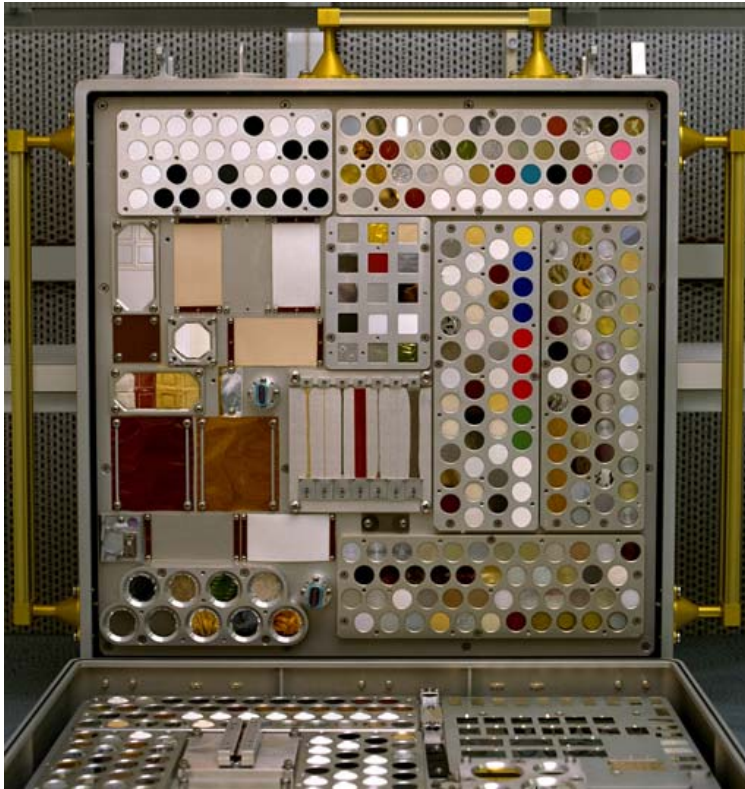
**Low Earth Orbit (LEO)** environment (180-650 km ): rich in atomic oxygen, which degrades polymeric materials (such as Kapton, Mylar or Teflon) used in satellites.

May erode certain polymers  
by over 2  $\mu\text{m}$  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...



International Space Station  
orbiting LEO region (450 km)



## Materials International Space Station Experiment – MISSE project

Trays of materials samples will expose 750 materials to LEO environment, for 18 months.

**Oxidation protection:** Thin layers of several metal oxides such as  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ , or  $\text{SiO}_2$  are being studied as protective coatings for polymers in LEO

**Thermal transients** ( $-100^\circ\text{C}$  to  $+100^\circ\text{C}$ ): Superior adhesion of the thin film is required  
→ Metal Plasma Immersion **Ion Implantation and Deposition**

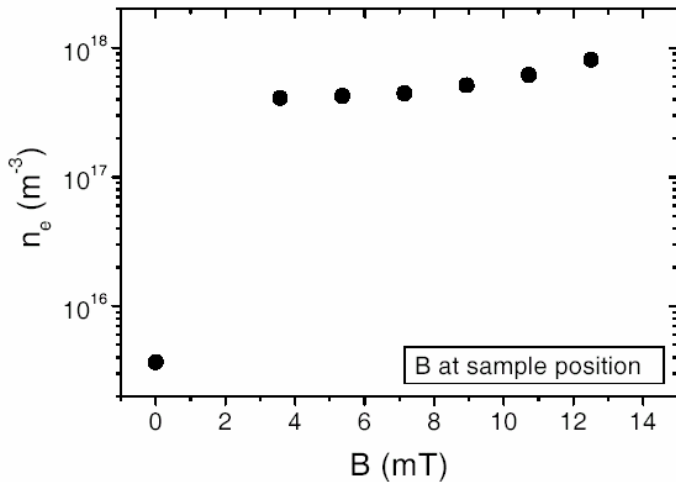
**PIII in polymers:** charging of the dielectric is proportional to plasma density

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

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

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

**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**

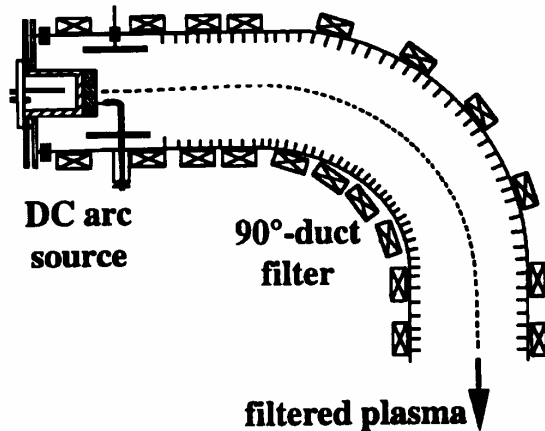
- Oxygen degradation (oxygen plasmas)
- Thermal cycling
- Adhesion test

# Direct Aluminum implantation

**Vacuum Arc Plasmas:** HV trigger: arc initiation  
Plasma **drift velocity**  $\sim 10^4\text{m/s}$

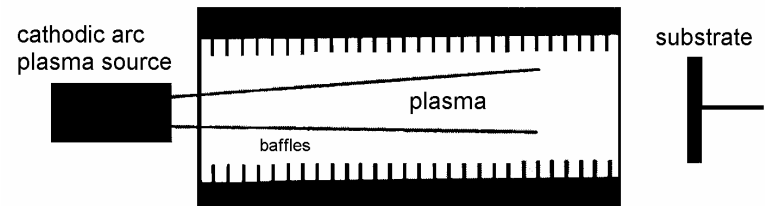
$I_{\text{arc}} \sim (100-1\text{k})\text{A}/50\text{V}$   
 $I_{\text{ion}} \sim \text{up to } (5 - 10)\% I_{\text{arc}}$

**Macroparticle Filtering systems:**



**Curved magnetic filter**

good filtering  
not so good plasma transport

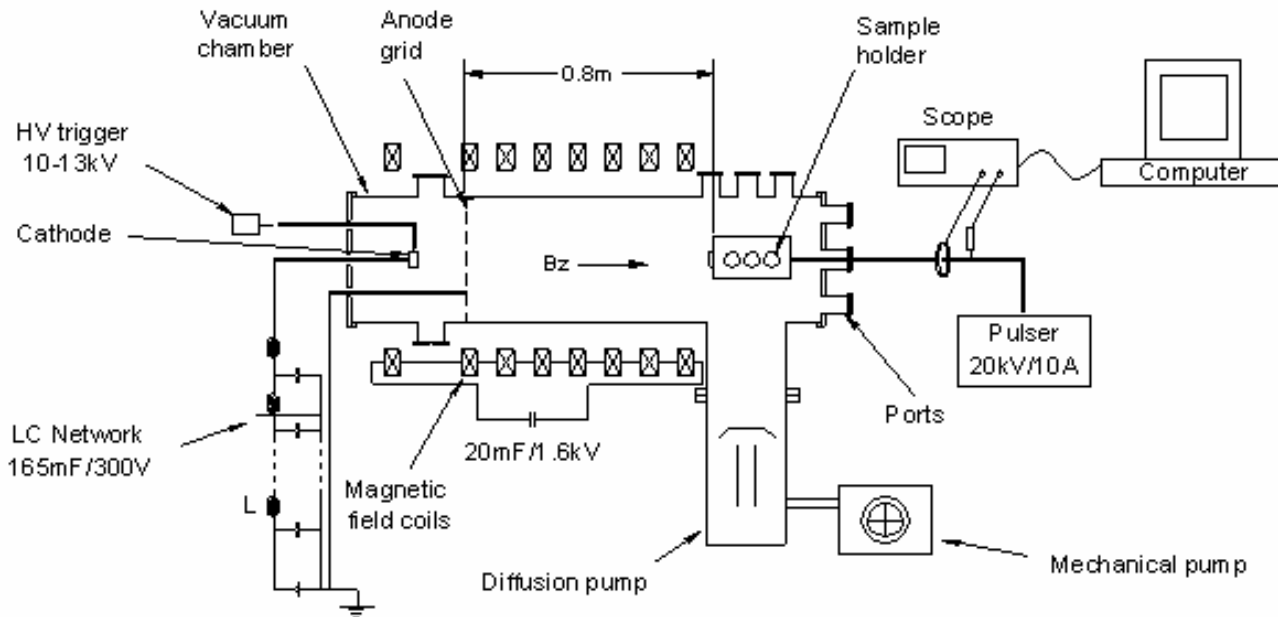


**Straight magnetic filter**

not so good filtering  
good plasma transport

Macroparticles avoided and deposition minimized by orienting samples **parallel to plasma stream**

# Experimental Set-up



**Vacuum arc:**

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

**Vacuum chamber**

$\phi=0.22\text{m}$ ,  $L=1.05\text{m}$

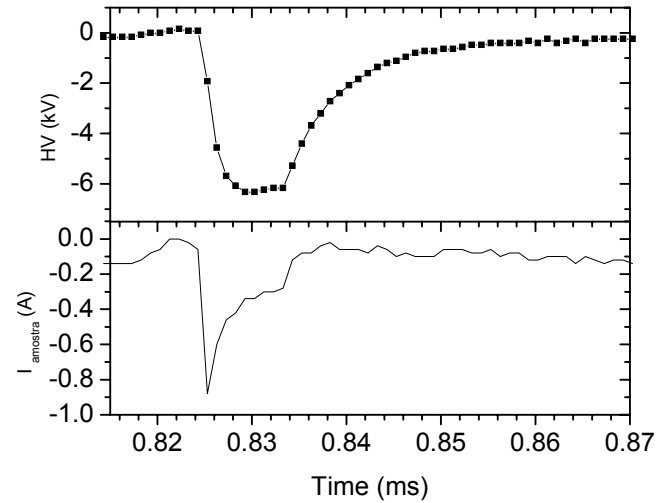
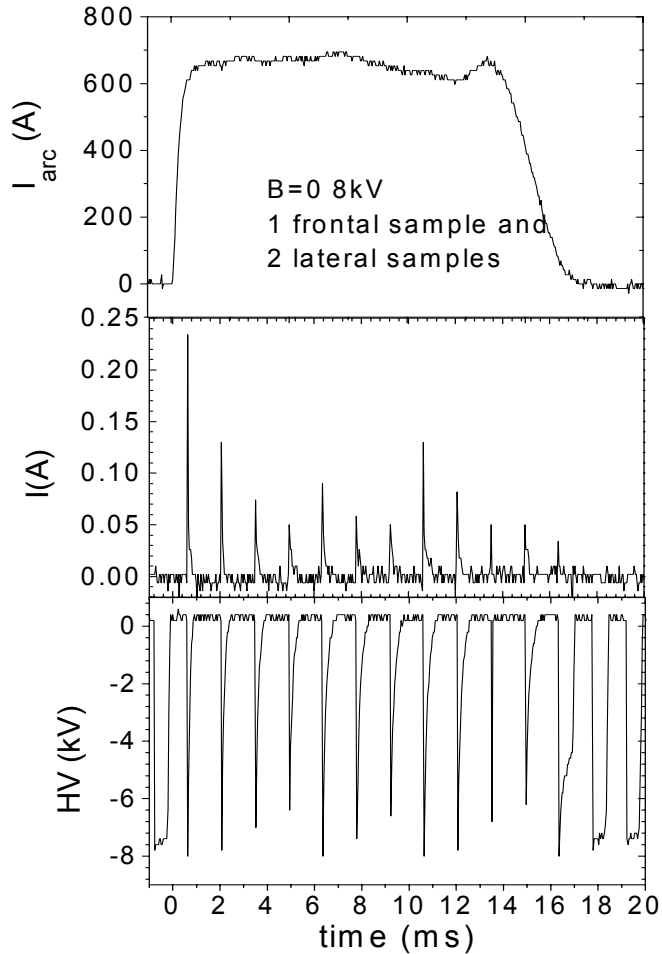
Base pressure  $\sim 1 \times 10^{-4}$  Pa B field: 150G-7kG

**Sample holder:**

85cm from cathode



# • Implantation Conditions



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

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

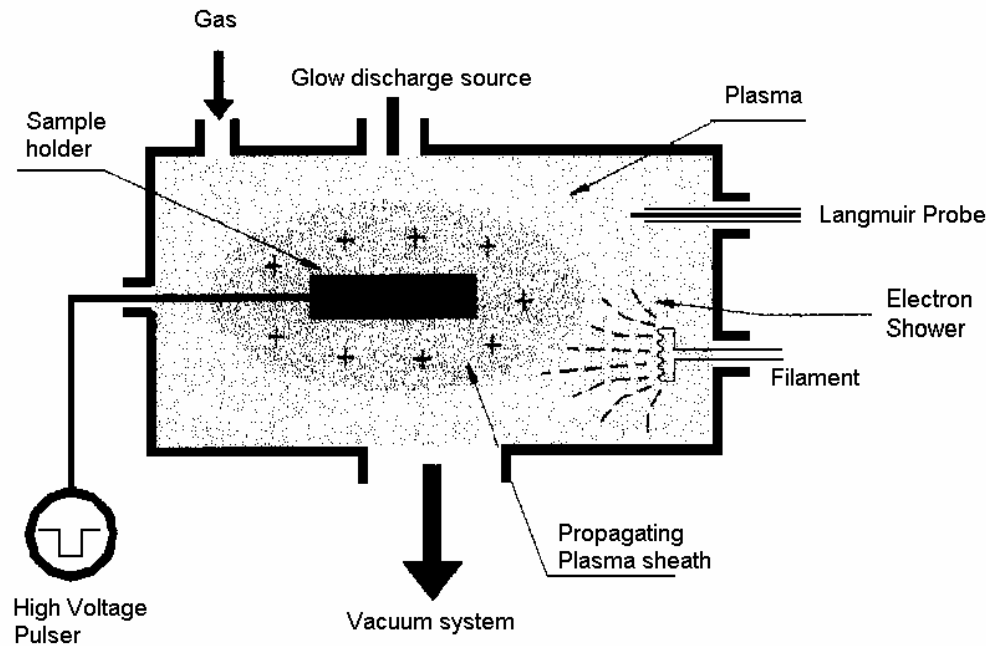
# Recoil Aluminum implantation

**200 Å aluminum film deposited** by electron beam on a Kapton sample (8.5cm×12cm) wound around sample holder. HV in contact with deposited film.

Implantation in **Nitrogen plasmas**:  $n \sim 10^{10} \text{ cm}^{-3}$ ,  $T_e < 10 \text{ eV}$

HV pulses: 5  $\mu\text{s}$ , 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  $\rightarrow n \sim 10^{10} \text{ cm}^{-3}$  ,  $T_e \sim (1-2) \text{ eV}$   
~ one hour exposure

## Thermal cycling

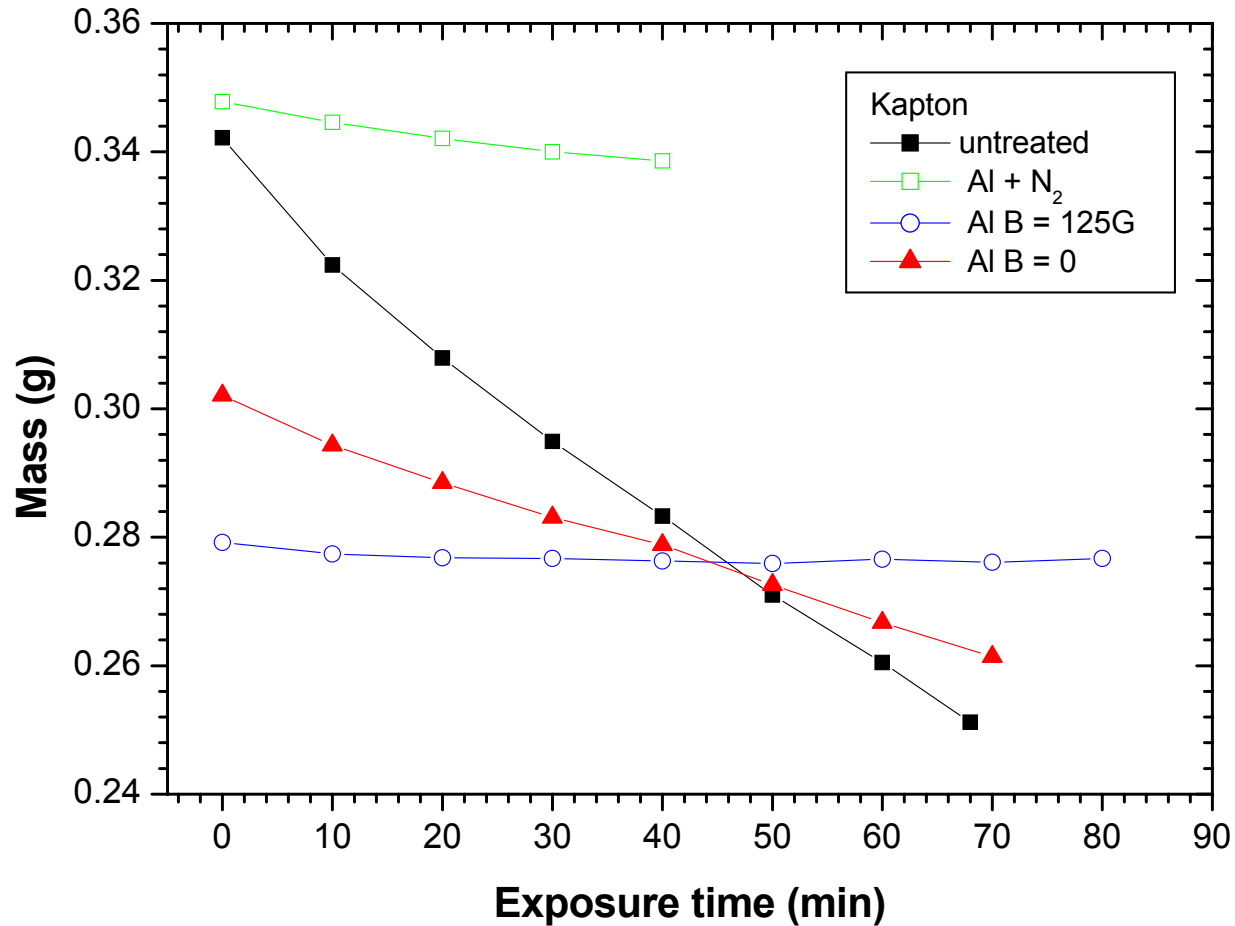
- 1 minute liquid nitrogen immersion (-196 °C )
- 1 minute pre-heated oven (100 °C)
- 15 cycles

## Adhesion Test

- applying and removing a pressure sensitive tape + SEM

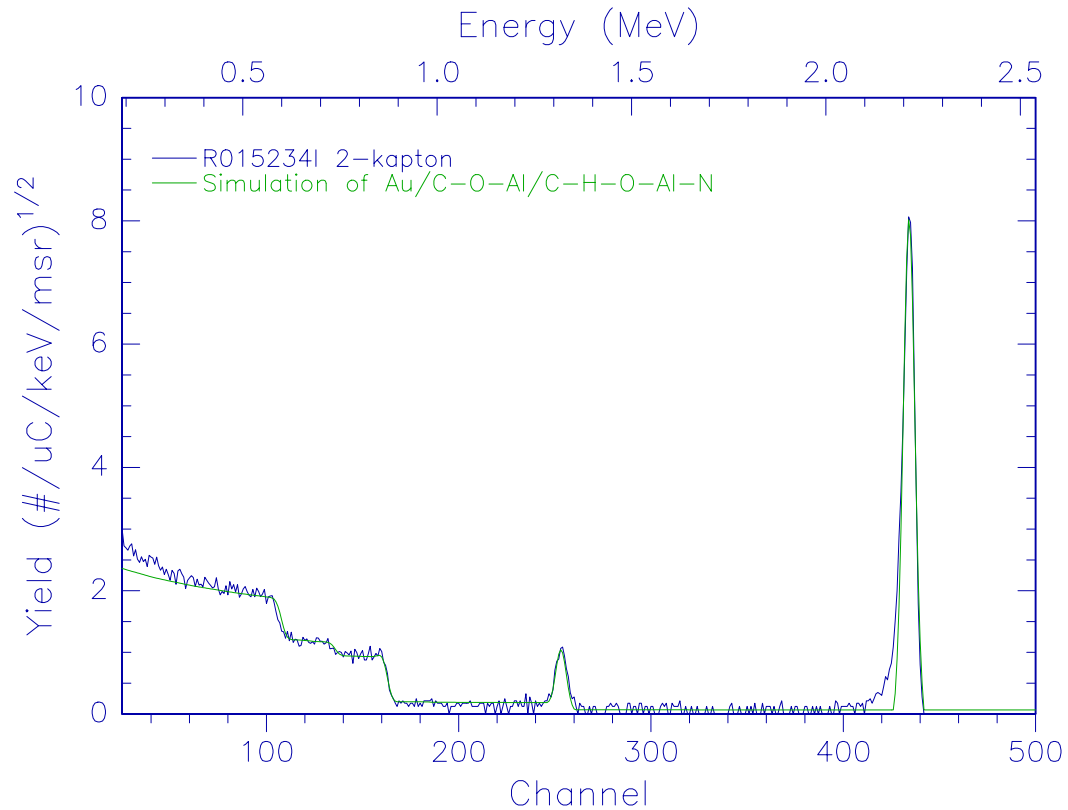
# Results

## Oxygen Degradation

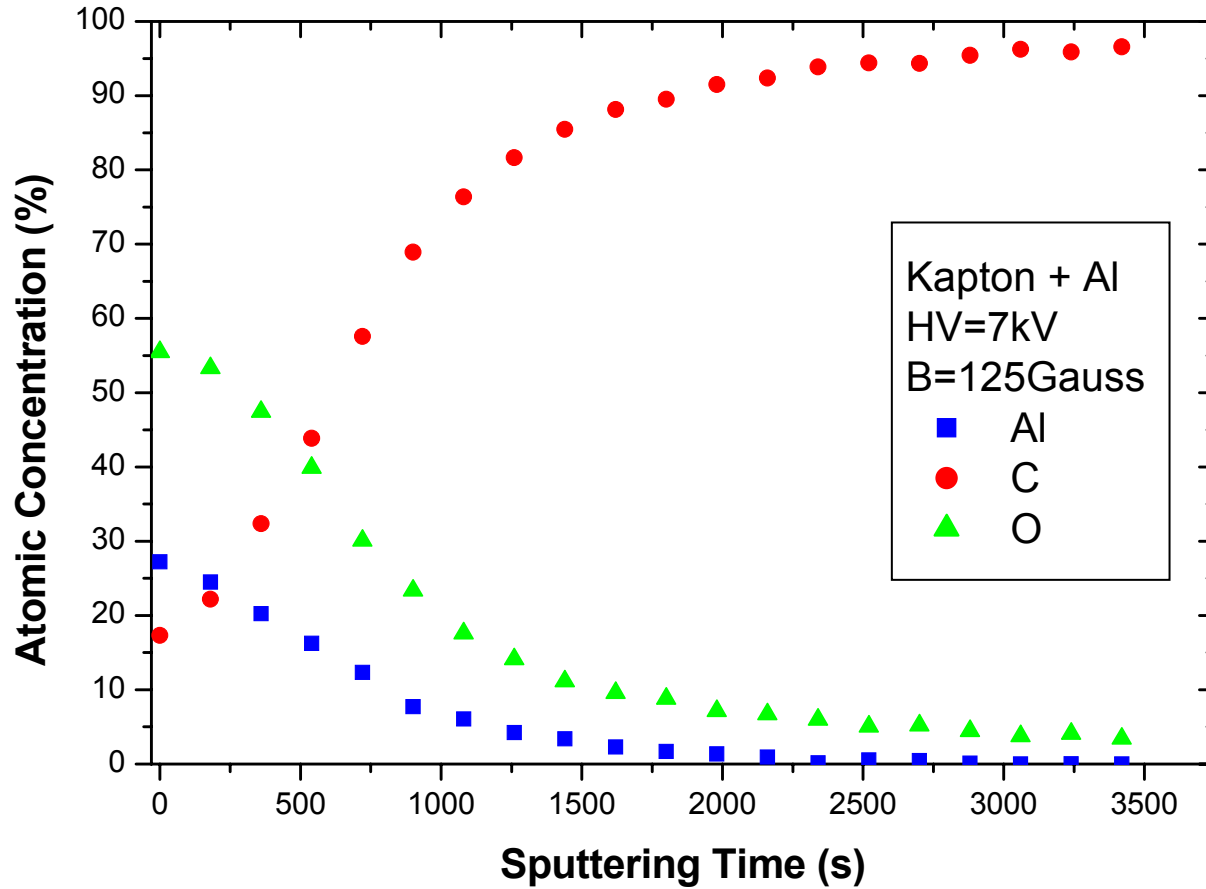


## Kapton sample implanted with Al in a magnetized plasma

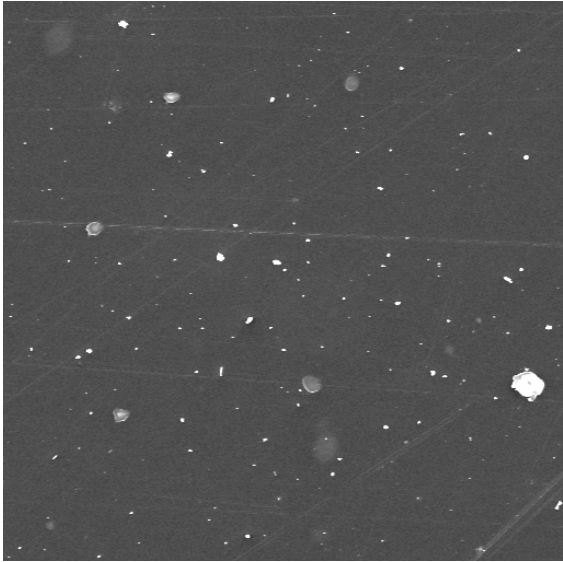
- **RBS** → retained doses of  $10^{16}$  atoms/cm<sup>2</sup>, but mostly at the surface



- **XPS** → formation of an ion mixing layer

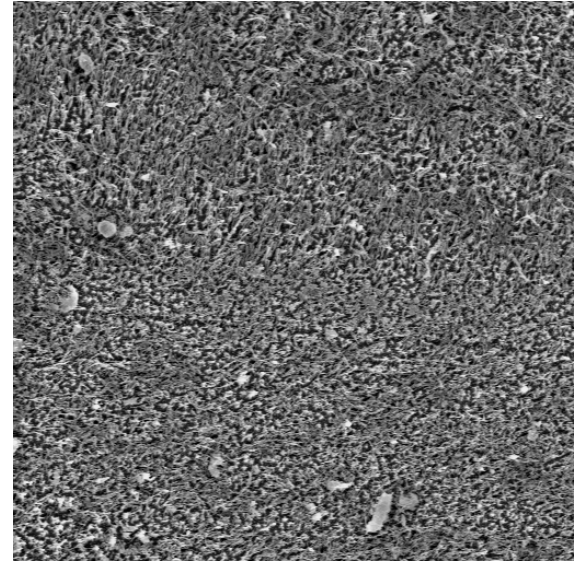


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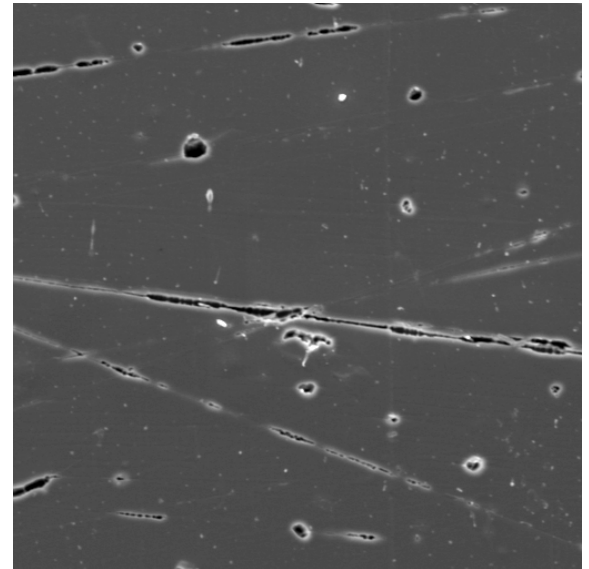


Without treatment

Without treatment  
after oxygen  
exposure →



Treated sample  
after oxygen  
exposure +  
thermal cycling →  
+ adhesion  
tests



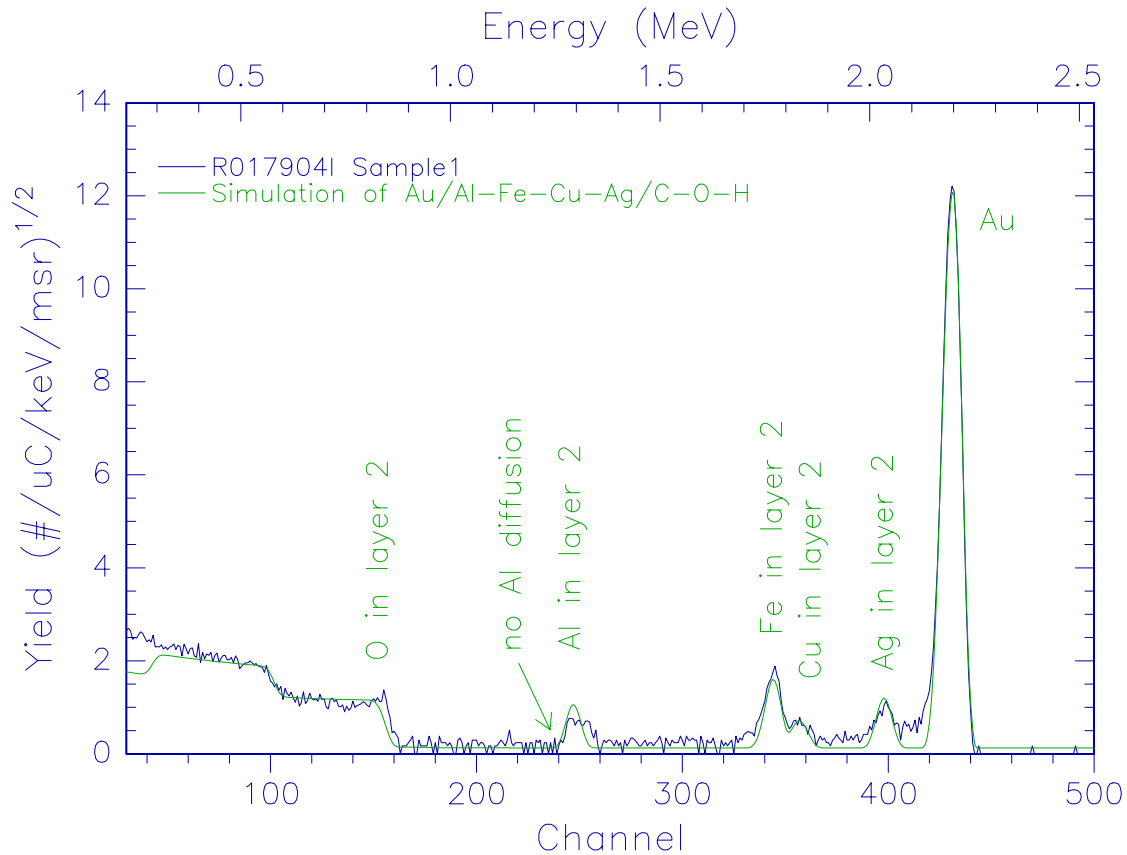
## Kapton sample implanted with Al in an unmagnetized plasma

- Not uniform : good parts behave like magnetized case after exposure to O plasmas  
bad parts behave like untreated case after exposure to O plasmas
- Possible causes: Insufficient dose  
misalignment with  
plasma stream



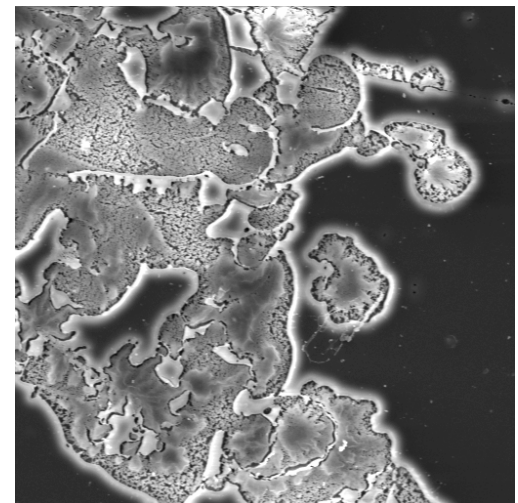
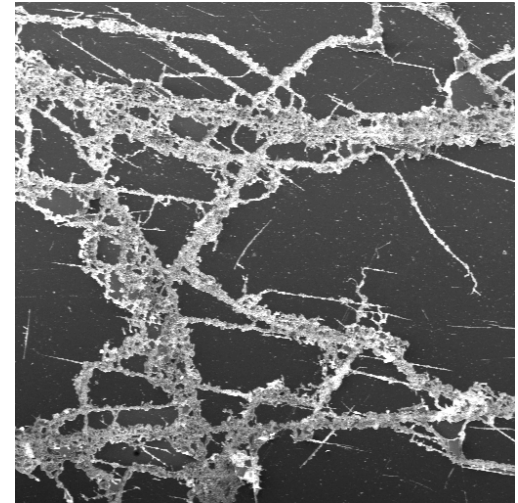
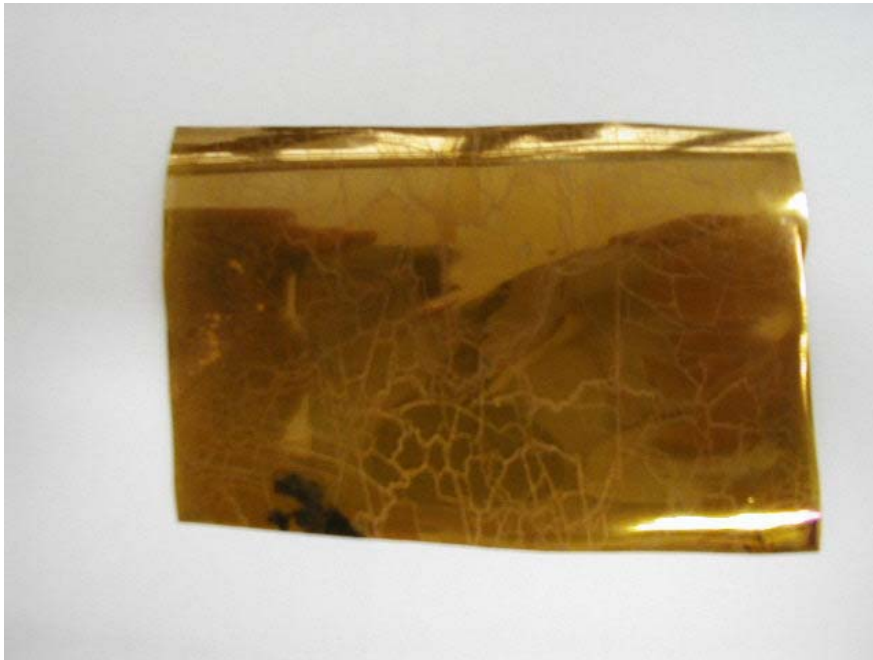


- **RBS** – “good” parts – about 100Å Al deposition mostly at surface

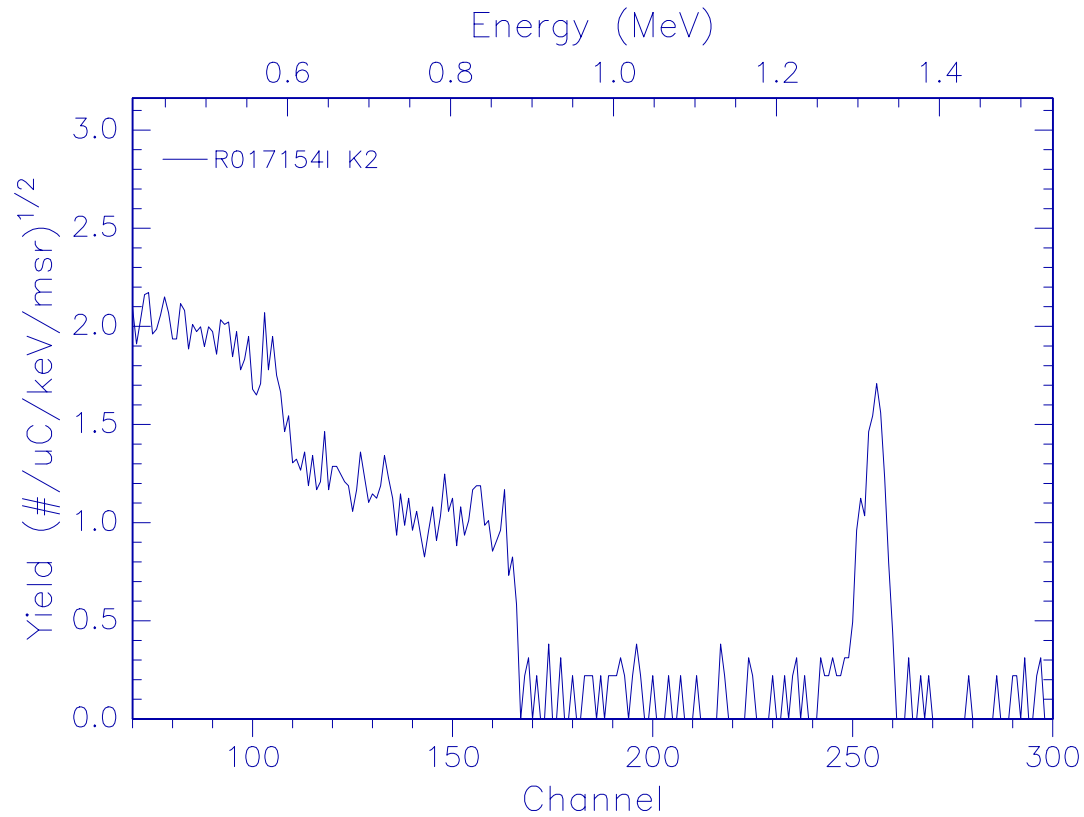


- **Adhesion tests after thermal cycling showed no delamination**

- **Kapton sample with Al deposition (200Å) + nitrogen plasma implantation**
- Formation of cracked film → probably stressed aluminum nitride
- Loss of transparency



- **RBS** – Al deposited mostly at surface



- **Adhesion tests after thermal cycling showed no delamination**

# 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/cm<sup>2</sup> were 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 non-magnetized plasmas needs much longer treatment times, incompatible with present machine configuration.
- Al deposition by e-beam, followed by recoil implantation in a nitrogen plasma resulted in a cracked film, probably due to the formation of a stressed aluminum nitride film. Recoil implantation in an argon plasma is underway.

## **Acknowledgements:**

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