Plasma-surface interactions: diagnostics

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Surface processes and reactive plasmas

Plasma controls surface processes: *deposition, etching, surface modification*

Surface processes influence plasma properties:

Ar/O₂ plasma cleaning of stainless steel reactor with hydrocarbon film on the wall:

Reactions in plasma

\[ \text{O}_2 + e^- \rightarrow 2\text{O} + e^- \]

Surface (with film)

\[ \text{O} + \text{a-C:H film} \rightarrow \text{CO (CO}_2 \text{)} \]

H and H₂ is also released

Surface (clean)

\[ \text{O(gas) + O(wall)} \rightarrow \text{O}_2\text{(gas)} \]

\[ \text{O(wall) + O(wall)} \rightarrow \text{O}_2\text{(gas)} \]
Surface processes and hydrogen plasmas

Plasma controls surface processes: deposition, sputtering, surface modification

Surface processes influence plasma properties:

W impurity accumulation in the JET tokamak, UK

Plasma composition is determined by surface reactions
Impurity accumulation plays a detrimental role on the performance

\[ P_{br} \propto Z^2 \quad P_{line} \propto Z^4 \]
How to address surface processes relevant to plasma deposition?

Our goal
- deposition of thin film
- or
- etching of wall material
  - almost always a combination of deposition and etching

Which plasma species arrive at the wall? Which energy do they have?
How do they react at the wall? How does the material form?
Which species leave the surface? What is their energy?
How do they influence the plasma?

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Key challenges

- energy distributions (ions, fast neutrals)
- molecular radicals
- synergistic effects
- not well-defined surfaces
- heterogeneous surface reactions

The unreconstructed surface of nickel
Scanning tunneling microscopy image

IBM Research, Almaden Research Center

Cross-section of a-C:H film
Molecular dynamics simulation


M. Zeuner et al., JAP 81 (1997) 2985
Outline

Plasma-surface interactions – short summary

Diagnostics of plasma surface processes

• \textit{ex-situ} and \textit{in-situ} plasma diagnostics

• beam experiments and growth models
Low energy particles at the surface

Gas temperature in low pressure plasmas

\[ T_h \sim 300 - 2000 \text{ K} \]

\[ E_k \sim 0.026 - 0.17 \text{ eV} \]

Lower than the binding energy between atoms in the material

\[ r + \gamma + s = 1 \]

Overall surface reaction probability: \( \beta = \gamma + s = 1 - r \)

Surface reactions often depend on surface coverage

\[ \theta = \frac{n_{\text{surface, occupied}}}{n_{\text{surface, all}}} \in \langle 0,1 \rangle \]
Interaction particle-substrate

**Physisorption**

weak van der Waals dipole-dipole interaction

$E_{\text{pot}} \sim \frac{1}{r^6}$

For metals: interaction between particle and its virtual image

$E_{\text{pot}}(z) \sim \frac{1}{z^3}$

Electron exchange – chemical bond

By metals: electron donated from conduction band

By insulators: e.g. reaction at unpaired electron → radical site/dangling bond or radical insertion into existing bond

Binding energy > 0.5 eV
Minimum much closer to the surface

**Chemisorption**

Pauli repulsion

Binding energy ~ few meV

When is a particle reflected and when captured at the surface?

Particle loses part $p_{\text{loss}}$ of its kinetic energy in the impact

It is captured when:

$$E_{\text{after}} = (1-p_{\text{loss}}) \times E_{\text{before}} < E_{a}$$

energy loss in collision with the surface $\rightarrow m$ and $M$ dependent

max. energy transfer: $\sim 4 \frac{mM}{(m+M)^{2}}$

$\sim 100\%$ for $m=M$

$\sim 4m/M$ for $m<<M$

The sticking coefficient is higher if:

- Kinetic energy ($E_i$) is small.
- The depth of the potential well ($E_a$) is large.
- The $M$ and $m$ are similar $\rightarrow$ effective energy transfer.
Surface diffusion

Surface potential is **corrugated** → particles at the surface do not move freely. 

\[ E_{\text{diff}} \] depends on the type of bonding (e.g. larger for chemisorption).

Steps or defect sites have a higher energy barrier. 
Surface diffusion can be promoted e.g. by ion bombardment.
Surface reactions

Two basic mechanisms:

1) Eley-Rideal

- Direct reaction upon impact
- Only barrier-less and exothermic reactions
- Usually involves radicals

Reaction rate: \( R = k p_A \Theta_B \)

Example: \( H(g) + H(s) \rightarrow H_2(g) \)

hydrogen abstraction

2) Langmuir-Hinshelwood

- Reaction of adsorbates at the surface
- Most common surface reaction mechanism
- Allows reactions between molecules

Reaction rate: \( R = k \Theta_A \Theta_B \)

\( T_{\text{prod}} \approx T_{\text{substrate}} \)

Example: \( \text{CO}(s) + \text{O}(s) \rightarrow \text{CO}_2 \)
Typical ion energies in low pressure plasmas: eV - keV

Possible interaction at the surface:

a) Reflection: used for surface diagnostic
   “Ion Scattering Spectroscopy” – ISS
b) Secondary electron emission – important for
   a plasma ignition and operation: $\gamma$-coefficient
c) Ion implantation: e.g. Plasma Immersion
   Ion Implantation (PIII); $E_{\text{ion}} > 10$ keV
d) Structural changes in the material:
   enhanced cross-linking $\rightarrow$ ion assisted growth
e) Sputtering of material
   Dependent on ion energy
Ion-surface interaction: stopping power

Incoming ion $E_0, m_1, Z_1$

Surface

Structural changes in the material

Displaced atom

Implanted atom

Binary collision cascade

Ion interaction in the material:

Low energy (plasma)

High energy

(binary) elastic collisions with target atoms

Inelastic collisions with electron gas

Ion-surface interaction: stopping power, example

Stopping power: carbon on carbon

Data from: http://www.exphys.uni-linz.ac.at/stopping/
Ion-surface interaction: binary collision cascade

At the ion energy range of few eV to few keV: Binary Collision Approximation (BCA) can be used

TRIM code (TRansport of Ions in Matter)

W. Jacob, Thin Solid Films 326 (1998) 1

ion assisted growth
Physical sputtering of target atoms

Sputtering yield: \[ Y = \frac{\Gamma_{\text{sputtered}}}{\Gamma_{\text{ions}}} \]

Threshold for physical sputtering

\[ E_{\text{threshold}} = E_{SB} \frac{1}{\gamma(1 - \gamma)} \]

\[ \frac{E_1}{E_0} = 4 \frac{m_1 m_2}{(m_1 + m_2)^2} = \gamma \]

Surface binding energy \( E_{SB} \) is not identical with sublimation energy = fitting parameter for the TRIM calculation.
Sputtering of graphite

TRIM is also valid for low energies ($\sim E_{SB}$) but chemical effects can dominate the results.

- Sputtering yield is dependent on $M_{\text{projectile}}$
- More effective E transfer at higher M
- Drops at high energies
- Energy deposited more into the volume
- Chemical reactions can enhance it
- Chemical sputtering → see later

$E_{\text{ion}} \sim 100–1000$ eV: sputter yield can be estimated by Sigmund-formula (1969):

$$Y = \frac{\Gamma_{\text{sputtered}}}{\Gamma_{\text{ions}}} \sim \frac{E_{\text{ion}}}{E_{SB}} \frac{4 m_i m_t}{(m_i + m_t)^2} \frac{3 \alpha}{4 \pi^2}$$

$$\alpha = \alpha \left( \frac{m_t}{m_i} \right) \in (0, 1/2)$$

empirical factor
Outline

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Surface reactivity $\beta$: imaging of radicals interacting with surfaces (IRIS)

LIF signal of CH radical:

$\beta = 0.95 \pm 0.07$

$\beta = 0.32 \pm 0.09$

### Surface Reactivity β: Imaging of Radicals Interacting with Surfaces (IRIS)

<table>
<thead>
<tr>
<th>Species</th>
<th>Plasma sources</th>
<th>Excited transition</th>
<th>$\lambda$(nm)$^a$</th>
<th>Radiative lifetime (ns)</th>
<th>Dipole moment (D)</th>
<th>Relative surface reactivity$^b$</th>
<th>Reference(s)</th>
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</thead>
<tbody>
<tr>
<td>C₂</td>
<td>C₂H₂</td>
<td>$A^1\Pi \leftarrow X^1\Sigma^+$</td>
<td>691</td>
<td>$1.85 \times 10^4$</td>
<td>—</td>
<td>—</td>
<td>143</td>
</tr>
<tr>
<td>C₃</td>
<td>C₂H₄</td>
<td>$A^1\Pi \leftarrow X^1\Sigma^+$</td>
<td>440</td>
<td>200</td>
<td>0.44</td>
<td>low/moderate</td>
<td>126, 139</td>
</tr>
<tr>
<td>CH</td>
<td>C₂H₅, CH₂OH</td>
<td>$A^2\Delta \leftarrow X^2\Pi$</td>
<td>430</td>
<td>537</td>
<td>0.55</td>
<td>high</td>
<td>117, 139</td>
</tr>
<tr>
<td>CHF</td>
<td>CH₂F₃₅</td>
<td>$A^1\Lambda' \leftarrow X^1\Lambda'$</td>
<td>571</td>
<td>$2.45 \times 10^3$</td>
<td>1.30</td>
<td>low/moderate</td>
<td>144</td>
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<tr>
<td>CF₂</td>
<td>C₂F₄</td>
<td>$A^2\Sigma^+ \leftarrow X^2\Pi$</td>
<td>224</td>
<td>26.7</td>
<td>0.64</td>
<td>low/moderate</td>
<td>65</td>
</tr>
<tr>
<td>CF₃</td>
<td>C₉F₈</td>
<td>$A^1\Sigma_1 \leftarrow X^1\Sigma_1$</td>
<td>226</td>
<td>61</td>
<td>0.44</td>
<td>low</td>
<td>65, 84</td>
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<tr>
<td>CCl₂</td>
<td>CCl₄, CH₄/Cl₂</td>
<td>$A^2\Delta \leftarrow X^2\Pi$</td>
<td>279</td>
<td>105</td>
<td>—</td>
<td>—</td>
<td>145</td>
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<tr>
<td>CN</td>
<td>CH₃CN, CH₄/N₂</td>
<td>$B^2\Sigma^+ \leftarrow X^2\Sigma^+$</td>
<td>387</td>
<td>65</td>
<td>0.50</td>
<td>high</td>
<td>140, 146</td>
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<tr>
<td>NH</td>
<td>NH₃, N₂/H₂</td>
<td>$A^3\Pi \leftarrow X^3\Sigma^-$</td>
<td>336</td>
<td>440</td>
<td>1.39</td>
<td>low/moderate</td>
<td>130</td>
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<tr>
<td>NH₂</td>
<td>NH₃, N₂/H₂</td>
<td>$A^2\Pi_1 \leftarrow X^2\Pi_1$</td>
<td>598</td>
<td>$10 \times 10^3$</td>
<td>1.82</td>
<td>moderate</td>
<td>130</td>
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<tr>
<td>NO</td>
<td>NO, N₂/O₂</td>
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<td>226</td>
<td>205</td>
<td>0.16</td>
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<td>147, 148</td>
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<td>OH</td>
<td>H₂O, H₂/O₂</td>
<td>$A^2\Delta \leftarrow X^2\Pi$</td>
<td>308</td>
<td>686</td>
<td>1.80</td>
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<td>28, 149</td>
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<tr>
<td>SiCl₄</td>
<td>SiCl₄, Cl₂$^c$</td>
<td>$B^2\Sigma^+ \leftarrow X^2\Pi$</td>
<td>297</td>
<td>10</td>
<td>—</td>
<td>—</td>
<td>150</td>
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<tr>
<td>SiCl₃</td>
<td>SiCl₄, Cl₂$^c$</td>
<td>$A^1\Pi \leftarrow X^1\Pi$</td>
<td>320</td>
<td>$4.5 \times 10^3$</td>
<td>1.46</td>
<td>low</td>
<td>135</td>
</tr>
<tr>
<td>SiF₄</td>
<td>SiF₄, CF₄$^c$, SF₆$^c$</td>
<td>$A^2\Sigma \leftarrow X^2\Pi$</td>
<td>437</td>
<td>230</td>
<td>1.07</td>
<td>moderate</td>
<td>24, 101</td>
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<tr>
<td>SiF₂</td>
<td>SiF₄, CF₄$^c$, SF₆$^c$</td>
<td>$A^1\Pi \leftarrow X^1\Pi$</td>
<td>225</td>
<td>62</td>
<td>1.23</td>
<td>low</td>
<td>24, 98</td>
</tr>
<tr>
<td>SiH₄</td>
<td>SiH₄, Si₂H₄</td>
<td>$A^2\Delta \leftarrow X^2\Pi$</td>
<td>413</td>
<td>534</td>
<td>0.14</td>
<td>high</td>
<td>20, 107</td>
</tr>
<tr>
<td>SiH₂</td>
<td>SiH₄, Si₂H₄</td>
<td>$A^1\Pi \leftarrow X^1\Pi$</td>
<td>580</td>
<td>111</td>
<td>0.16</td>
<td>moderate</td>
<td>109</td>
</tr>
<tr>
<td>SO</td>
<td>SO₂, SF₆/O₂</td>
<td>$B^3\Sigma \leftarrow X^3\Sigma$</td>
<td>235</td>
<td>16.2</td>
<td>1.55</td>
<td>—</td>
<td>151</td>
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<tr>
<td>SO₂</td>
<td>SO₂, SF₆/O₂</td>
<td>$A^1\Pi \leftarrow X^1\Pi$</td>
<td>300</td>
<td>$10 \times 10^3$</td>
<td>1.63</td>
<td>—</td>
<td>151</td>
</tr>
</tbody>
</table>

$^a$Excitation wavelength for listed transition.

$^b$Relative reactivity scale: low = < 0.1, low/moderate = ~0.1–0.3; moderate = ~0.3–0.7; high = ~0.7–1.0.

$^c$Species of interest is produced during Si processing.
Surface reactivity $\beta$: well / cavity Experiments

Measurement of $\beta$ in plasmas
Density of reactive species in front of a reactor wall

**Confinement time of a given species in the reactor**

$$\frac{1}{\tau_{\text{confinment}}} = \frac{1}{\tau_{\text{gas}}} + \frac{1}{\tau_{\text{surface}}} + \frac{1}{\tau_{\text{pump}}} = \sum_x k_r n_x + \frac{D}{\Lambda^2} + \frac{\text{Speed}}{V_{\text{reactor}}}$$

- loss in gas-phase reactions
- diffusion to the wall and surface loss
- loss due to gas pumping

→ density gradient at the wall!

**Flux lost at the surface**

$$j_{\text{lost}} = \frac{1}{4} n_{\text{wall}} v \frac{\beta}{1 - \beta/2}$$

**Special case (e.g. CH$_3$ radical)**

for $\beta \to 0, k_r = 0$

$$\frac{1}{\tau_{\text{diff}}} = \frac{v}{4 \frac{\text{volume}}{\text{Surface area}}} \beta$$

**Effective diffusion length (empirical)**

$$\Lambda^2 = \Lambda_0^2 + \lambda \frac{\text{volume}}{\text{surface area}}$$

$$\lambda = \frac{4}{3} \lambda_m \left(1 - \frac{\beta}{2}\right)$$

linear extrapolation length

mean-free-path

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Determination of surface reactivity $\beta$: decay in plasma afterglow

A. von Keudell et al. unpublished

Surface changes after switching off the plasma!
Determination of surface reactivity $\beta$: decay in modulated plasmas

$\rightarrow$ surface does not change significantly

Hoefnagels et al., CPL 360 (2002) 189
Determination of surface reactivity $\beta$: decay in modulated plasmas

- Si lost in gas phase reactions with SiH$_4$

  \[ k_{\text{Si-SiH}_4} = 2.6 \pm 1.0 \times 10^{-10} \text{ cm}^3\text{s}^{-1} \]

- SiH$_3$ does not react in the gas phase!

SiH$_3$ reacts at the wall.

$\beta$ independent of surface temperature.

Hoefnagels et al., CPL 360 (2002) 189
Measurement of surface reactions: spinning wall experiment

O recombination at spinning surface

Donnelly et al., e.g.:
L. Stafford et al., Pure Appl. Chem. 82, 1301 (2010)
Measurement of surface reactions: spinning wall experiment

Recombination of **atomic oxygen**:  

Recombination of **atomic chlorine**:  

Cl recombination depends on Cl₂ surface coverage → has to be considered in e.g plasma models
Measurement of surface reactions: spinning wall–effect of impurities

Spinning wall combined with Auger electron spectroscopy and evaporation sources.

**Effect of controlled "contamination" on the surface on O atom recombination coefficient:**

![Graph showing recombination coefficients](image)

Small contamination can have large effect!
The surface is not as clean as you think!


Outline

Plasma-surface interactions – short summary

Diagnostics of plasma surface processes

• *ex-situ* and *in-situ* plasma diagnostic

• beam experiments and growth models
**Beam experiment to study surface reactions of "plasma" particles**

Beam sources of radicals or ions are used to simulate in low pressure chamber the conditions in a plasma experiment → with well-defined particle fluxes.


Sticking of CH$_3$ varies with H flux to the surface.

\[ H \text{ flux (x} 10^{16} \text{ cm}^{-2}\text{s}^{-1}) \]

\[ \text{CH}_3 \text{ flux (norm.)} \]

growth

erosion

\[ s = 10^{-2} \]

\[ s = 10^4 \]

\[ \sim 4 \times 10^{15} \text{ cm}^{-2}\text{s}^{-1} \]

\[ \sim 3 \times 10^{14} \text{ cm}^{-2}\text{s}^{-1} \]
Beam experiments: $\text{CH}_3|\text{H}$ synergism, simple vs. extended model

simple model

\[
\frac{d\Theta_{\text{db}}}{dt} = (j_H \sigma_{\text{abstraction, H}} + j_{\text{CH}_3} \sigma_{\text{abstraction, CH}_3})(1 - \Theta_{\text{db}})
\]

\[-(j_H \sigma_{\text{addition, H}} + j_{\text{CH}_3} \sigma_{\text{addition, CH}_3}) + k_{\text{release, CH}_3(T)} \Theta_{\text{db}}.\]

\[\Gamma = j_{\text{CH}_3} \sigma_{\text{addition, CH}_3} n_0 \Theta_{\text{db}} - k_{\text{release, CH}_3} n_0 \Theta_{\text{db}}\]

extended model including cross linking

\[
\frac{d\Theta_{\text{db}}}{dt} = (j_H \sigma_{\text{abstraction, H}} + j_{\text{CH}_3} \sigma_{\text{abstraction, CH}_3})(1 - \Theta_{\text{db}})
\]

\[-(j_H \sigma_{\text{addition, H}} + j_{\text{CH}_3} \sigma_{\text{addition, CH}_3}) + k_{\text{release, CH}_3(T)} \Theta_{\text{db}}.\]

\[\Gamma = j_{\text{CH}_3} \sigma_{\text{addition, CH}_3} n_0 \Theta_{\text{db}} - k_{\text{release, CH}_3} n_0 \Theta_{\text{db}}\]

M. Meier, A. von Keudell JAP 90, 3585 (2001)
Measurement of surface reactions: infrared absorption spectroscopy

FTIR absorption spectra of an a-C:H film

Evolution of an FTIR spectrum after turning off the CH$_3$ radical source

FTIR can also be used in combination with isotopes (e.g. flux of D or D$_2$)

M. Meier, A. von Keudell JAP 90, 3585 (2001)

J. Ristein et al., JAP 84, 3836 (1998)

FTIR can also be used in combination with isotopes (e.g. flux of D or D$_2$)
"Simple" test of a growth mechanism

Analysis of a T-dependent growth of a-C:H films from electron cyclotron resonance CH$_4$ discharge

T-dependent film growth/erosion rate

*in CH$_4$ plasma*

T-dependent film erosion rate

*in H$_2$ plasma*

growth rate measured by in-situ single wavelength ellipsometry

→ combination of constant deposition rate with T-dependent film erosion by hydrogen

Beam experiments: ion-assisted film growth: energy dependence of $s(\text{CH}_3| \text{H}_2^+)$

- **Data**: CH$_3$ + H$_2^+$
- **Model**: TRIM.SP displaced H in the first ML
  - $c_H = 0.3$, $E_{dp}^H = 2.5$ eV

Beam experiments: ion-assisted film growth: film properties

H incorporation at the surface
Subsurface
H$_2$ release

Beam experiments in study of physical and chemical sputtering

Sputtering of graphite

Beam experiments allow:
- measurements of absolute sputtering yields
- determination of angular dependence
- study of different chemistries

K. Krieger in 'lectures on plasma physics'
Summer university for plasma physics
MPI für Plasmaphysik (1993)
Angular dependence of sputtering yield: physical sputtering

Formation of „grass“ due to micromasking

Dependence of etching yield on ion incident angles

Lecture Notes on Principles of Plasma Processing
F.F. Chen, J.P. Chang
http://www.ee.ucla.edu/~ffchen/Publs/Chen208i.pdf
Angular dependence of physical sputtering yield: TRIM calculation

$Y \sim 0.13$

$Y \sim 1.79$ !!!
Si (polysilicon) etching with 100 eV Ar\(^+\) ions and Cl atoms

reactive sputtering \[1\]
\[
\text{Ar}^+ (100 \text{eV}) + \text{Cl} \\
(\text{Cl}/\text{Ar}^+ = 600)
\]

Physical sputtering (TRIM)

Ar\(^+\) (100 eV) on Si

Etching Yield \(\frac{\text{Si}}{\text{Ar}^+}\)

Depends on material:
Cl can be easily implanted into poly-Si \(\rightarrow\) chem. sput.
but not into SiO\(_2\) \(\rightarrow\) physical sputtering

Proper angular dependence for each material has to be incorporated into profile simulators

Reactive sputtering: molecular dynamic simulation

![Graph showing etching yield vs. square root of energy for Cl⁺ and Ar⁺ ions.]

Fig. 11. Sputtering yield of polysilicon by Cl⁺ in the low energy regime, in comparison to molecular dynamic simulation results and low energy sputtering yield by Ar⁺.

![Diagrams showing molecular dynamic simulations at different ion fluences: 1.39 Monolayers, 7.9 ML, 13.9 ML, 20.8 ML.]

Fig. 13. Molecular dynamic simulation of Cl⁺ interacting with Si [Barone and Graves]. JAP 78 (1995) 6604

Cl-rich surface layer – reduces the surface binding energy – lower threshold, higher yield...

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Beam experiments: chemical sputtering

The famous plasma surface interaction experiment by Coburn and Winters JAP 50, 3189 (1979)

Si + F \rightarrow Si:F \quad \text{surface polymer}

Si:F + ions \rightarrow \text{volatile SiF}_4

\begin{align*}
\text{SiF}_x &\quad \text{c-Si} \\
\text{XeF}_2 &\quad \text{SiF}_4 \\
\text{Ar}^+ &\quad \text{Ar}^+
\end{align*}
Key features in the success of chemical sputtering
Anisotropy, selectivity, removal of etch products

Chemical etching:
selective, fast, good removal of etch products, but no anisotropy

Physical sputtering:
anisotropic, but not very selective, slow and problems with removal of etch products

Chemical sputtering (ideal case):
Selective (mask not etched)
Fast (neutral chemistry + ion damage)
Good removal of etch products (volatile)
Anisotropy (directed ions):
effect only at the bottom of the trench, side walls passivated → (almost) no etching
Conclusions

Plasma is a unique tool for surface modification
- provides reactive radical species with high reactivity at the surface
- provides energetic ions → essential for selective and anisotropic etching
- allows film growth at low substrate temperatures

Surface processes are determined by
- the fluxes of incoming species (including their energy and angular distribution)
- the state of the surface (temperature, composition roughness, passivation…)
- synergistic mechanisms between different species at the surface

Surface processes can be analyzed in
- particle beam experiments
- time resolved experiments (modulation of plasma, afterglow decay)
- time resolved measurement of surface properties (IR absorption, isotopic studies)
- spinning wall experiments, rotating substrate experiments
- well/cavity experiments
- molecular dynamic simulations, TRIM simulations
- …
Further reading


A. Zangwill, ‘Physics at surfaces’, Cambridge Univ. Press, 1988


http://www.ee.ucla.edu/~ffchen/Publs/Chen208i.pdf


J.W. Coburn, H. Winters, JVSTA16, 391 (1979)