Development and Application of Reflection High-Energy Positron Diffraction

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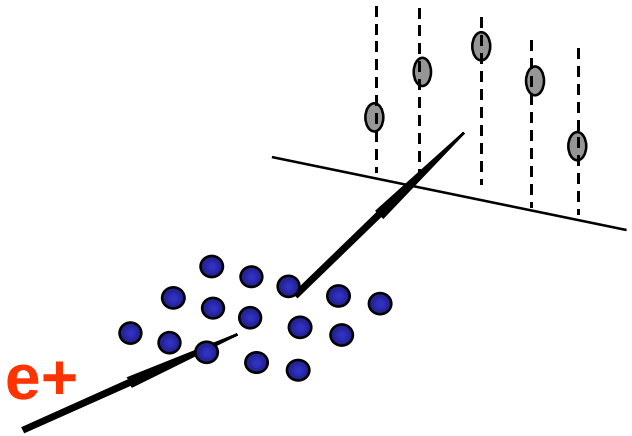
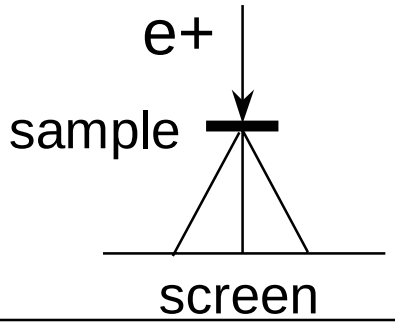
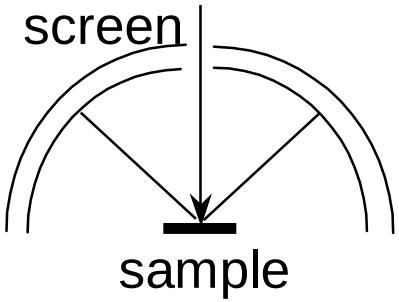
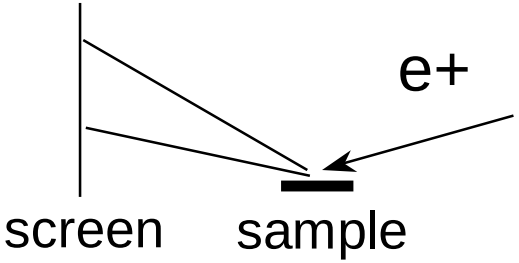


1. What is RHEPD?
2. Establishment of RHEPD
3. Application of RHEPD
   - *H-terminated Si and SiC
   - *Surface dipole of metals
4. Summary and Feature Plan

1. What is RHEPD?

Positron Diffraction Experiments

		
TPD **T**ransmission **P**ositron **D**iffraction	LEPD **L**ow **E**nergy **P**ositron **D**iffraction	**RHEPD** **R**eflection **H**igh **E**nergy **P**ositron **D**iffraction
100keV ▪ 1MeV	10 ▪ 500eV	10 ▪ 100keV
Bulk ▪ Study	▪ **Surface** ▪ **Study** ▪	

LEPD ▪ ▪ 1980 USA

▪ Coulomb Repulsion from ion core

▪ No exchange interaction

RHEED/RHEPD experiments

•Exchange-correlation interactions…**negligible**
•Inelastic process (e.g., plasmon excitation)…**reduced**

RHEPD

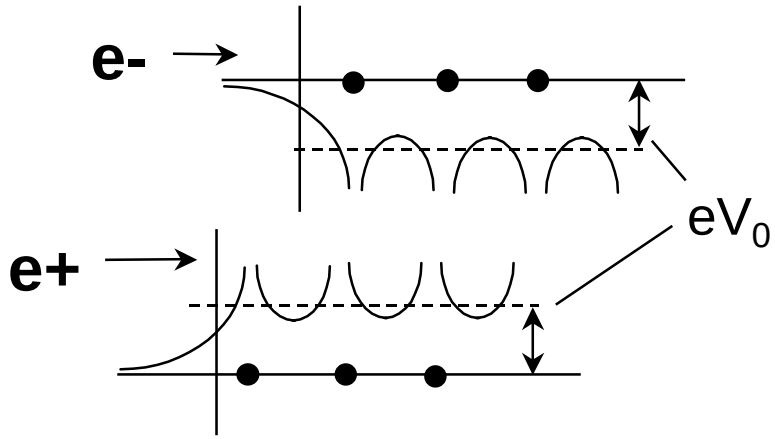
Ichimiya, A., Sol. Stat. Phen. 28&29 (1992/93)143.

- **Primary Bragg Reflection**
- **Total Reflection**

Negative Inner Potential

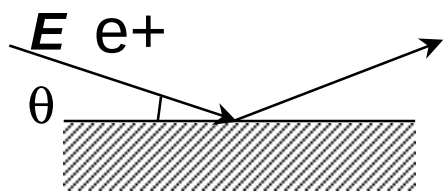
□

Positive Inner Potential



Bragg condition: $E\sin^2\theta = 37.5\, n^2/d^2 + eV_0$

Primary Bragg peak



Si(111) ▪ : d=3.14 A

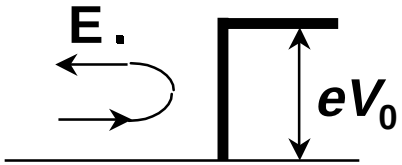
$eV_0 = \begin{cases} -12 \text{ eV for } e^- \\ +12 \text{ eV for } \mathbf{e^+} \end{cases}$ $\quad E\sin^2\theta \begin{cases} <0 \\ >0 \end{cases}$ for n=1

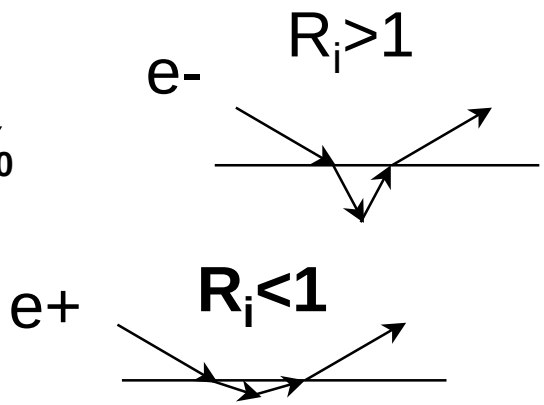
Total reflection

$\mathbf{E}\,.\,(E\sin^2\theta) < eV_0$

$\Downarrow$

$\theta = \sin^{-1}[eV_0/E]^{1/2}$

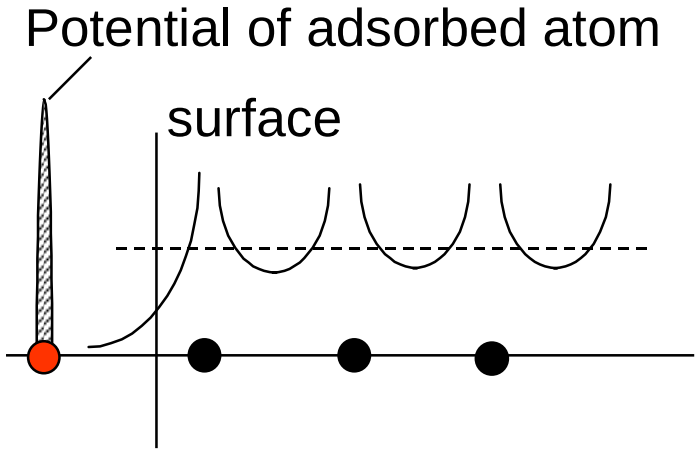


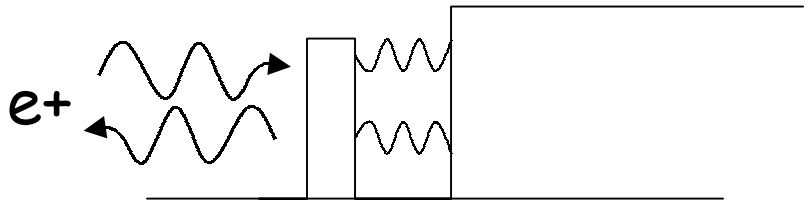


Possible Application of RHEPD

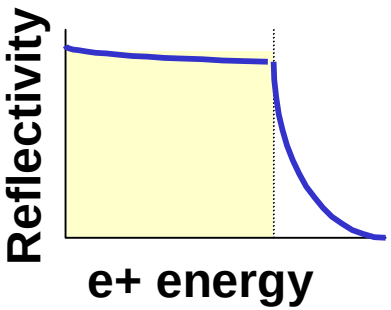
Diffraction pattern
Rocking curve
(+Dynamical theory)

- Adsorbed layer
- Surface roughness
- Surface Debye temperature
- Surface dipole barrier of metals

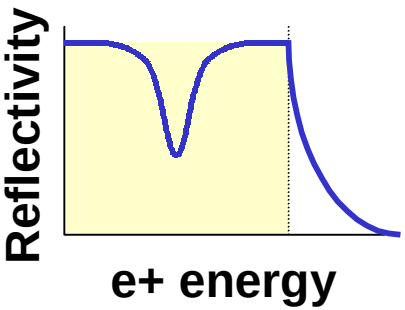




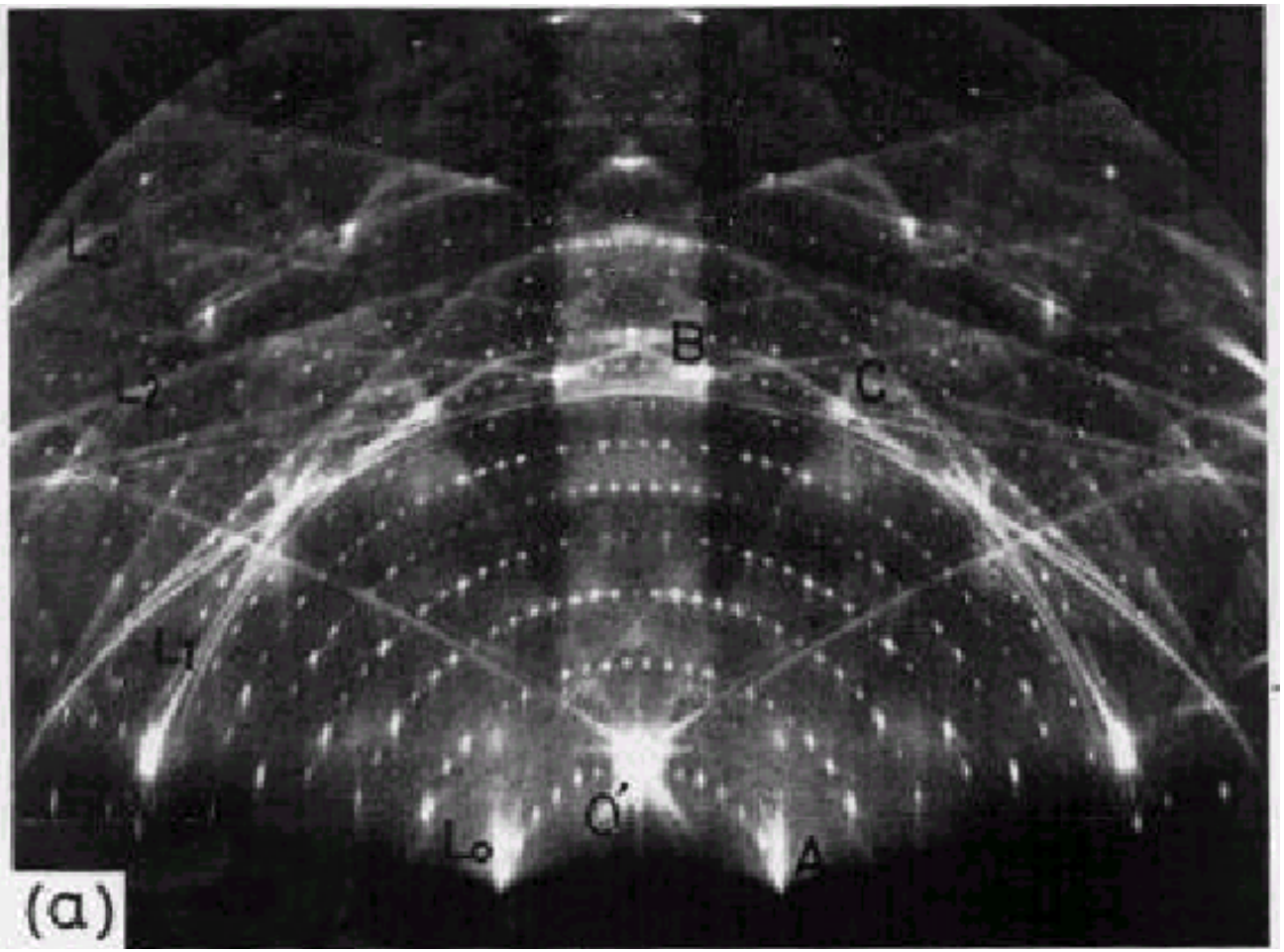
Flat surface



Irregular surface



Si(111)7x7 Ino (JJAP)



2. Establishment of RHEPD

History

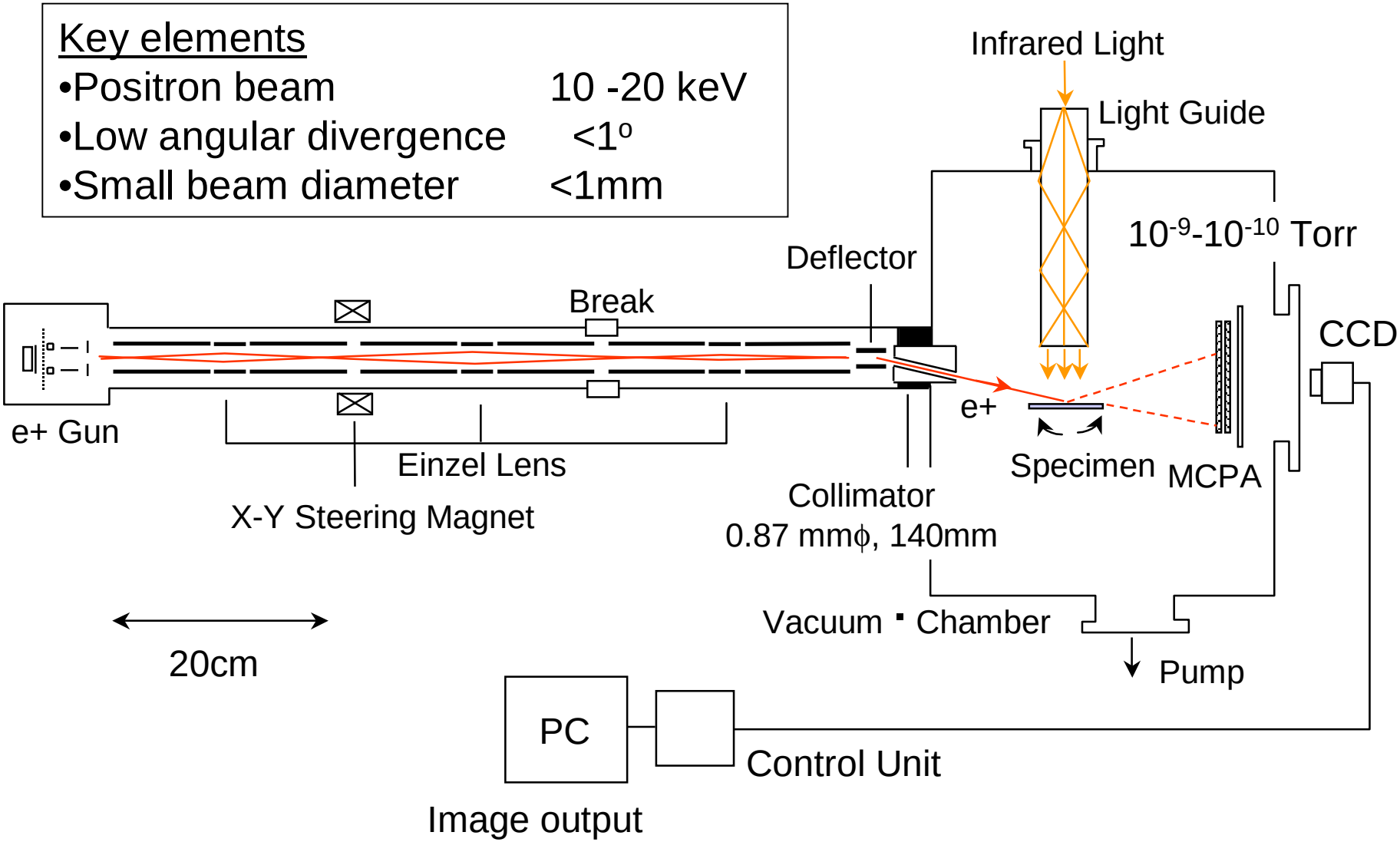
- **Positron reflection at metal surface**
  Oliva (PhD. thesis, UC San Diego, 1979)

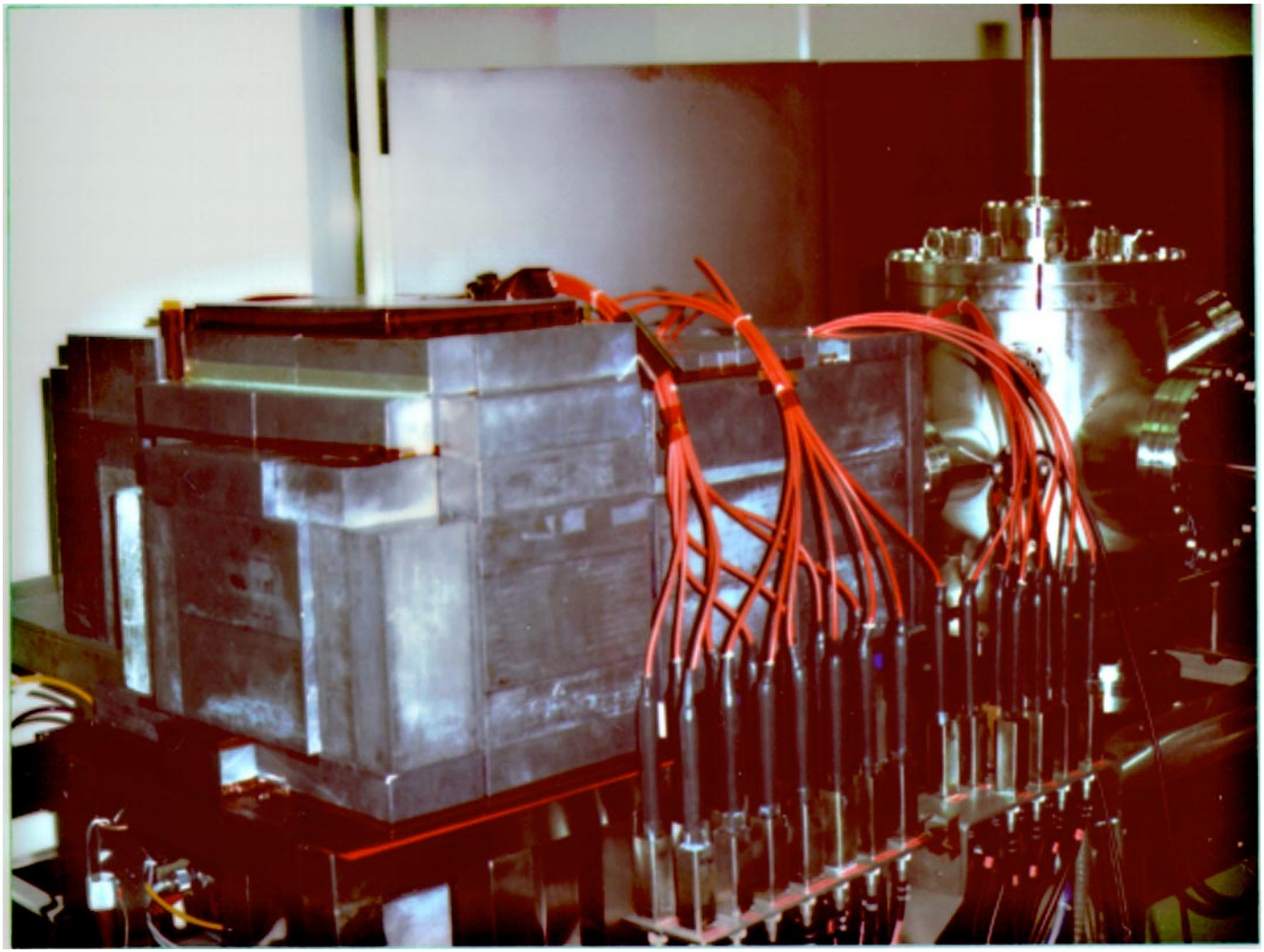
- **Theoretical description of RHEPD**
  Ichimiya (Sol. Stat. Phen. **28&29** (1992/93)143.)

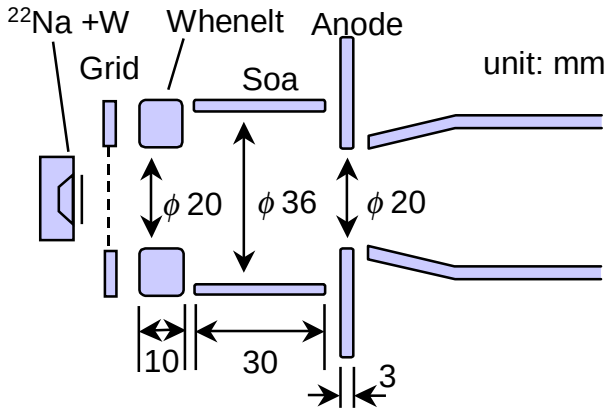
<u>What we did to establish RHEPD thechnique</u>

•e+ beam development
•Confirmation of e+ diffraction phenomenon
•Determination of rocking curve

Present RHEPD apparatus at JAERI



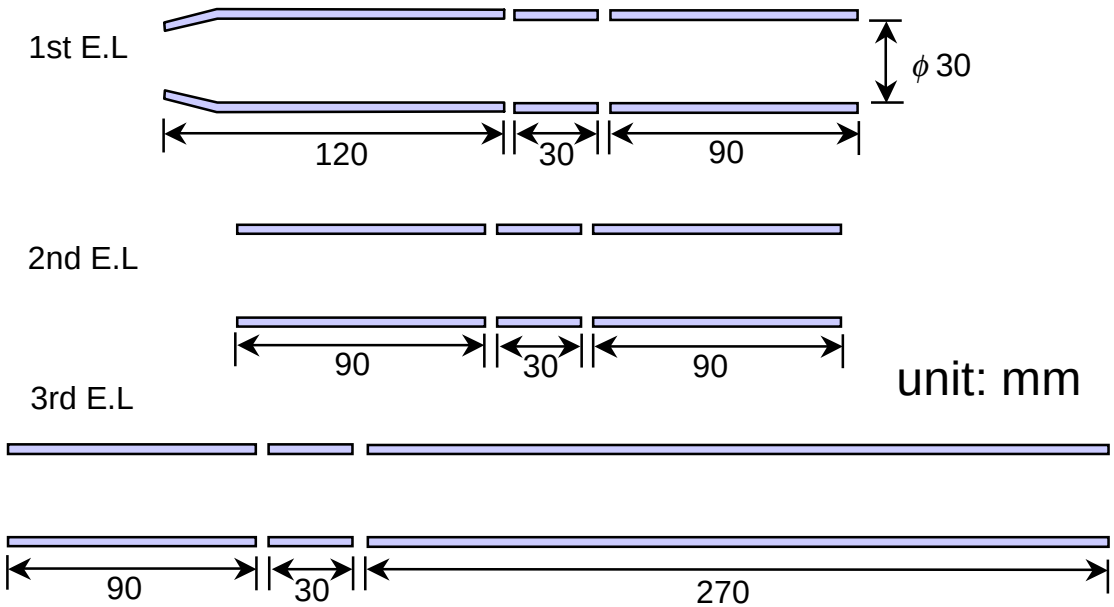




K. F. Canter et al in:
Positron Studies of Solids, Surfaces, and Atoms, eds. A. P. Mills, Jr., W. S. Crane and K. F. Canter (World Scientific, Singapore, 1986) p.199.

e+ gun

Einzel lenses



E. Harting and F.H. Read, “Electrostatic lenses”, (Elsevier scientific publishing company, Amsterdam, 1976).

$\sin\theta_d \times d \times E^{1/2}$ **=constant**

θ_d: angular divergence
d: diameter
E: energy

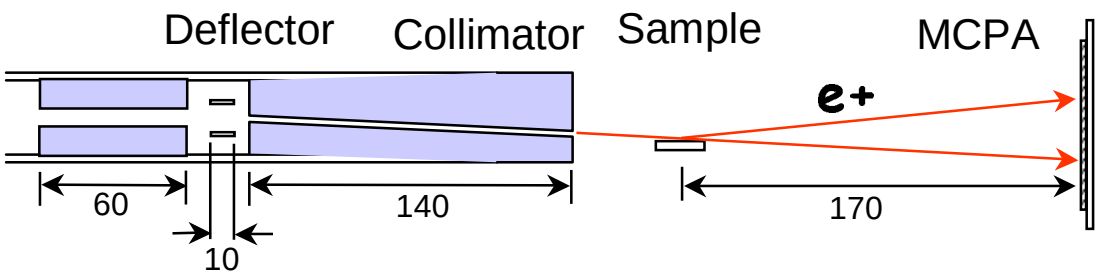
Acceleration up to 20 keV

Sufficient reduction of θ_d ▪ d

c.f.

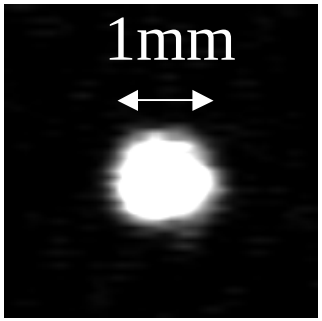
E_{ini}=3 eV, d_{ini}=7 mm, θ_d=5°
E_{fin}=20keV, d_{fin}=5 mm, θ_d<0.1°

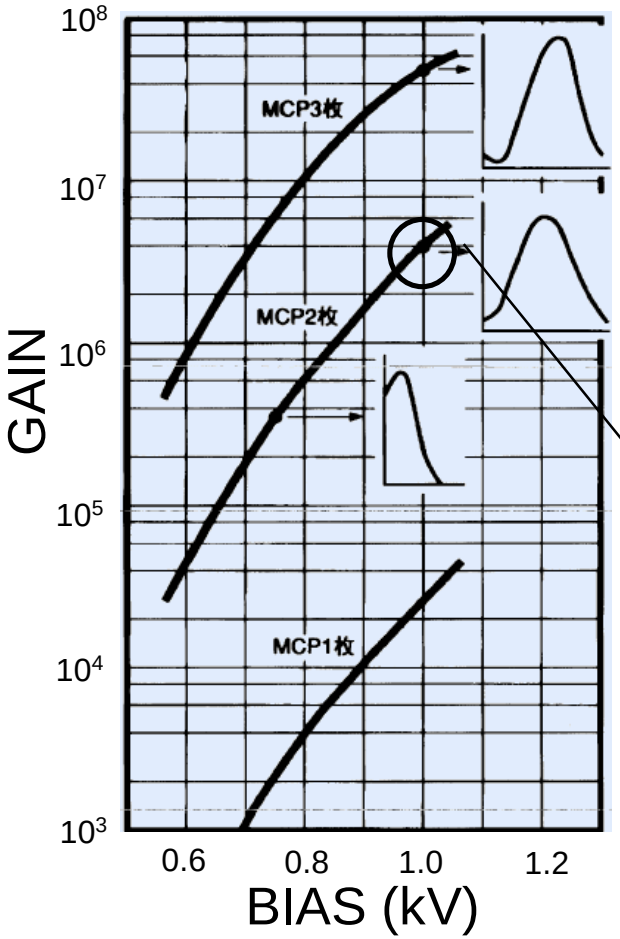
- Beam operation in "Defocus mode"
- Long-length collimator → "Axial beam"

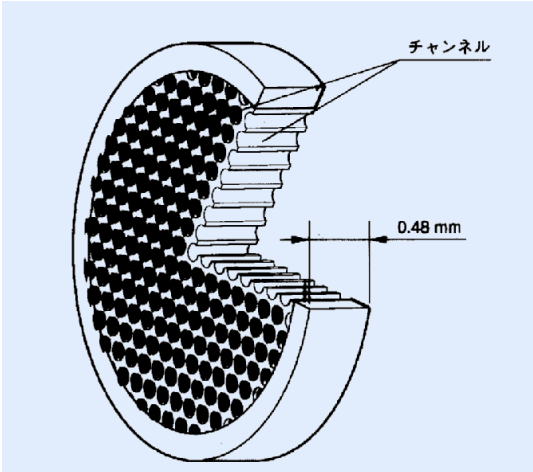


Characteristics of Positron Beam

Beam Energy	20 keV fixed
Final e+ flux	▪ 3000 e+/sec
Beam diameter	0.9 mm
Angular divergence	< 1 deg.







Max gain 4×10^6

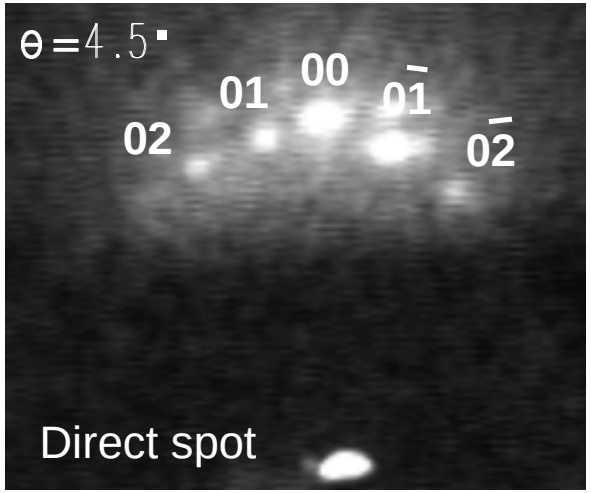
One RHEPD pattern :1-10 hrs.

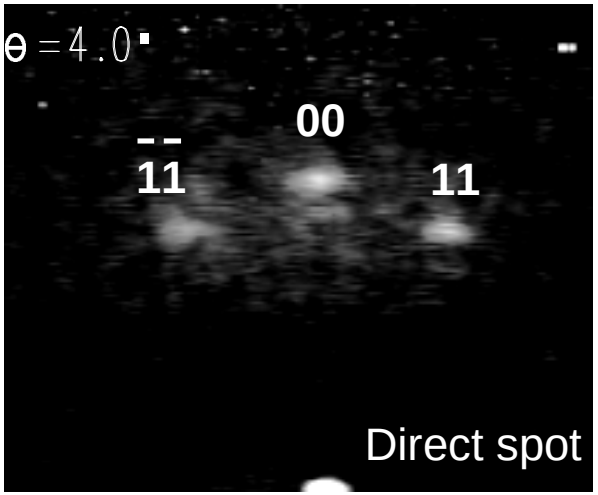
First RHEPD pattern from Si(111)H

[11$\bar{2}$] incidence

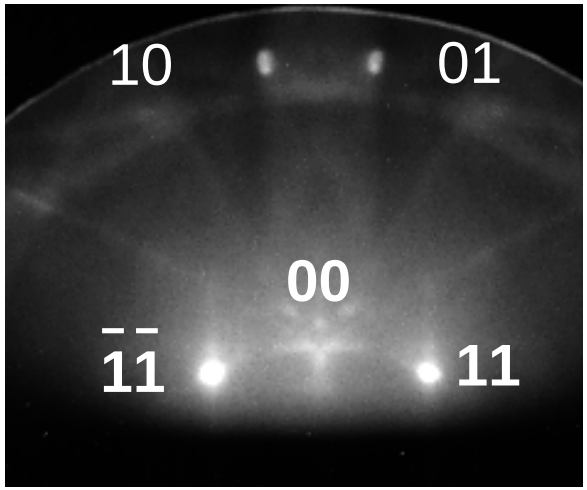
[1$\bar{1}$0] incidence

RHEPD





RHEED

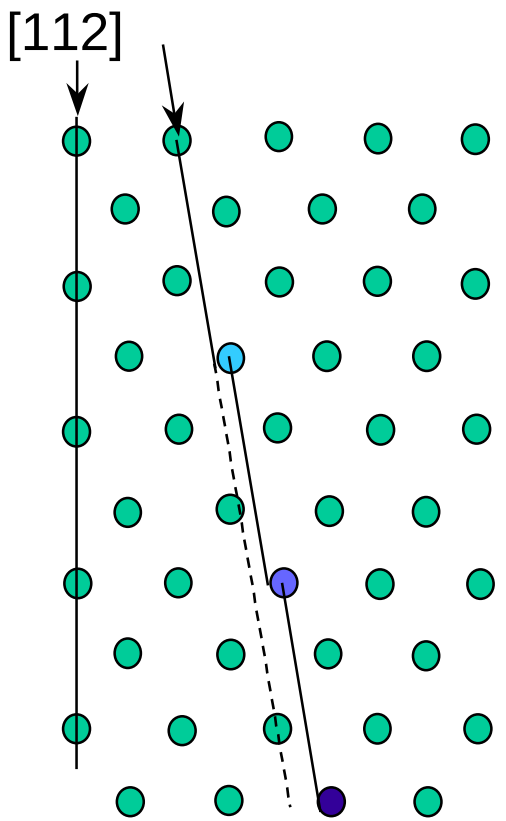


Confirmation of One-Beam Condition

Only specular spot in an asymmetric incidence 7.5°-off to [112], Si(111)

c.f. Symmetrical axis, [110] ▪ [112] for Si(111)

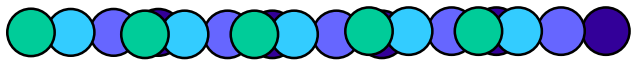
Specular spot ▪ Diffraction spots (Reciprocal lattice)



Si(111) 1x1 surface

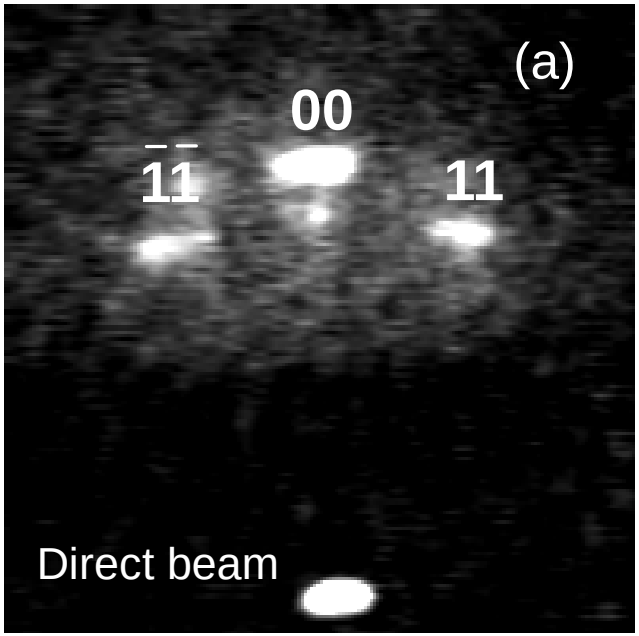
View from [112]

View from 7.5°-off ▪ [112]

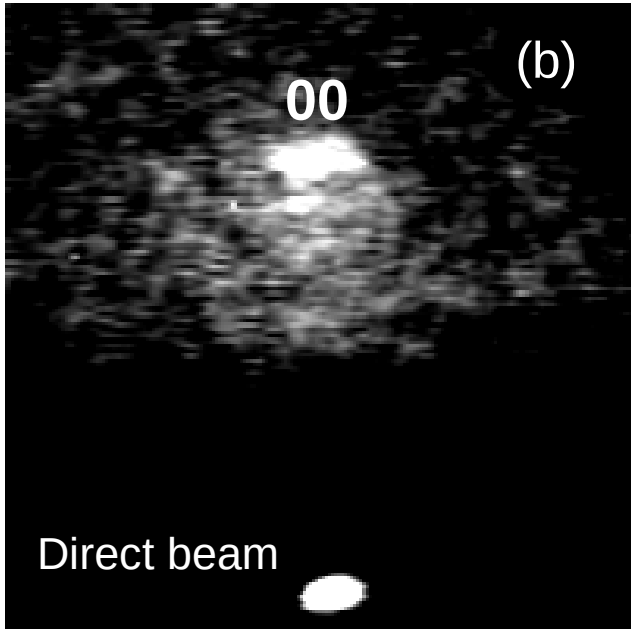


Transvers symmetry vanishes in one-beam condition

$[11\bar{2}]$ incidence

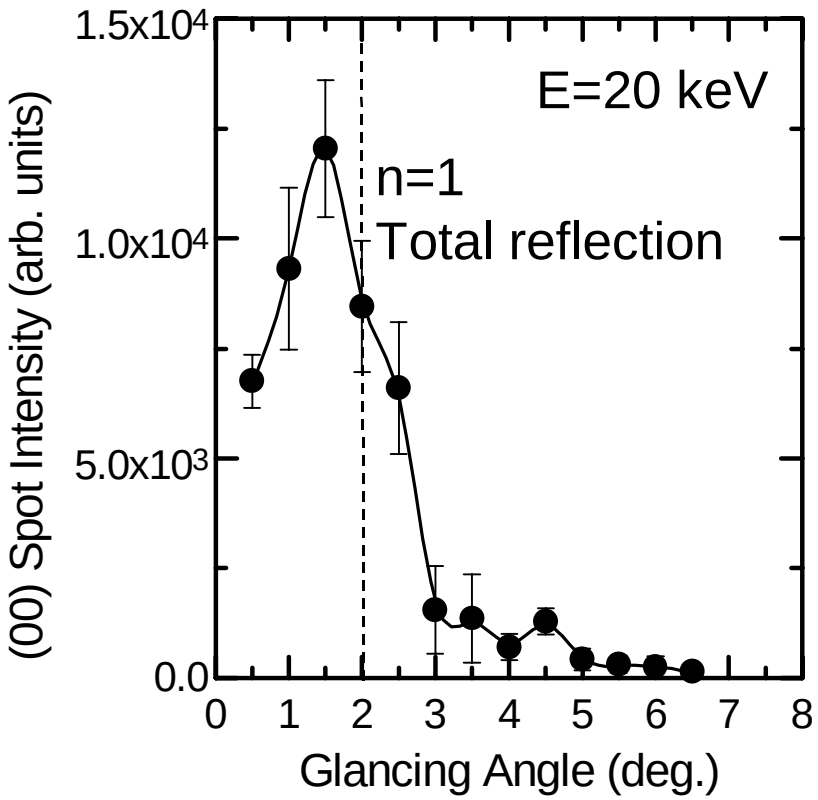


One beam condition
7.5° off from $[1\bar{1}2]$

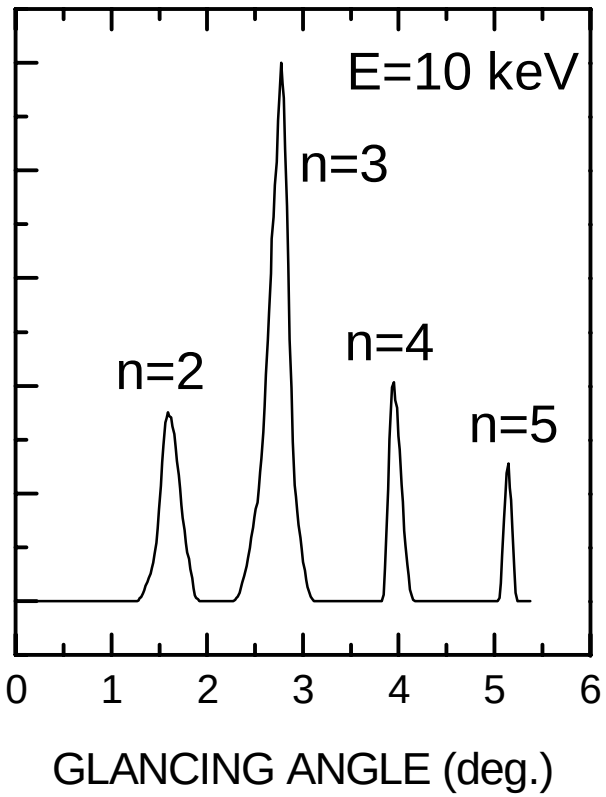


First RHEPD Rocking Curves

Positron



Electron



3. Applications of RHEPD

- **Characterization of H-terminated Si(111)**
- **Hydrogen-etching effect on SiC(0001) morphology**
- **Surface dipole barrier of metals**

H-termination of Si(111)

x3

Boiling in HNO_3 . 10min

⇩

Dip in 1%-HF

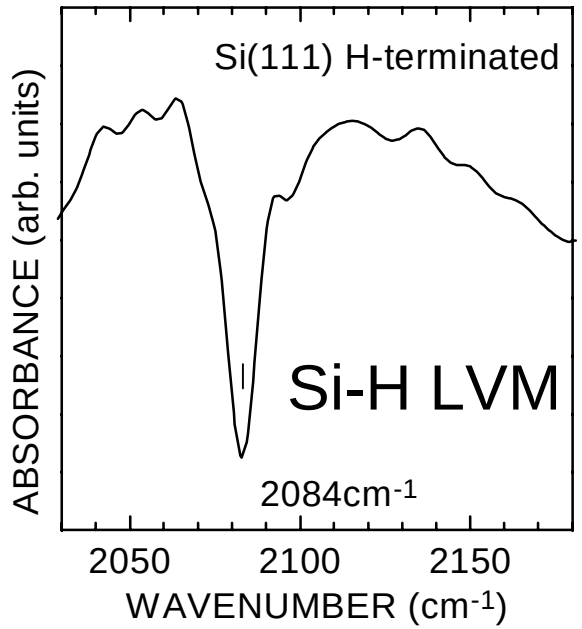
⇩

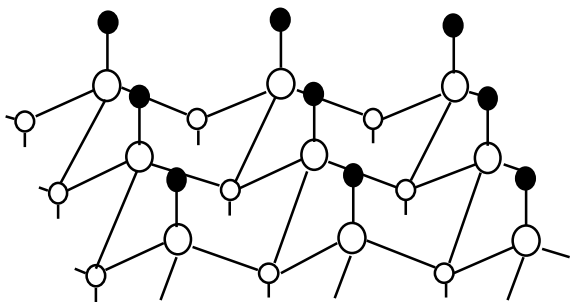
Rinse in H_2O

⇩

Dip in NH_4F 20min

IR spectrum

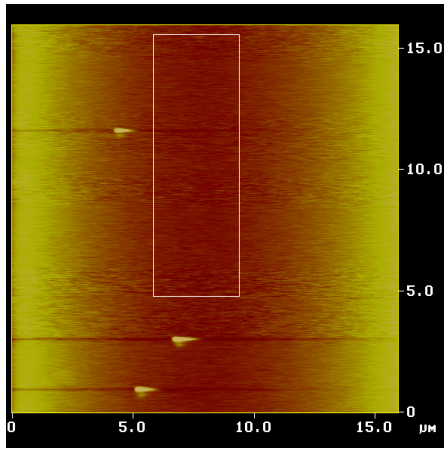




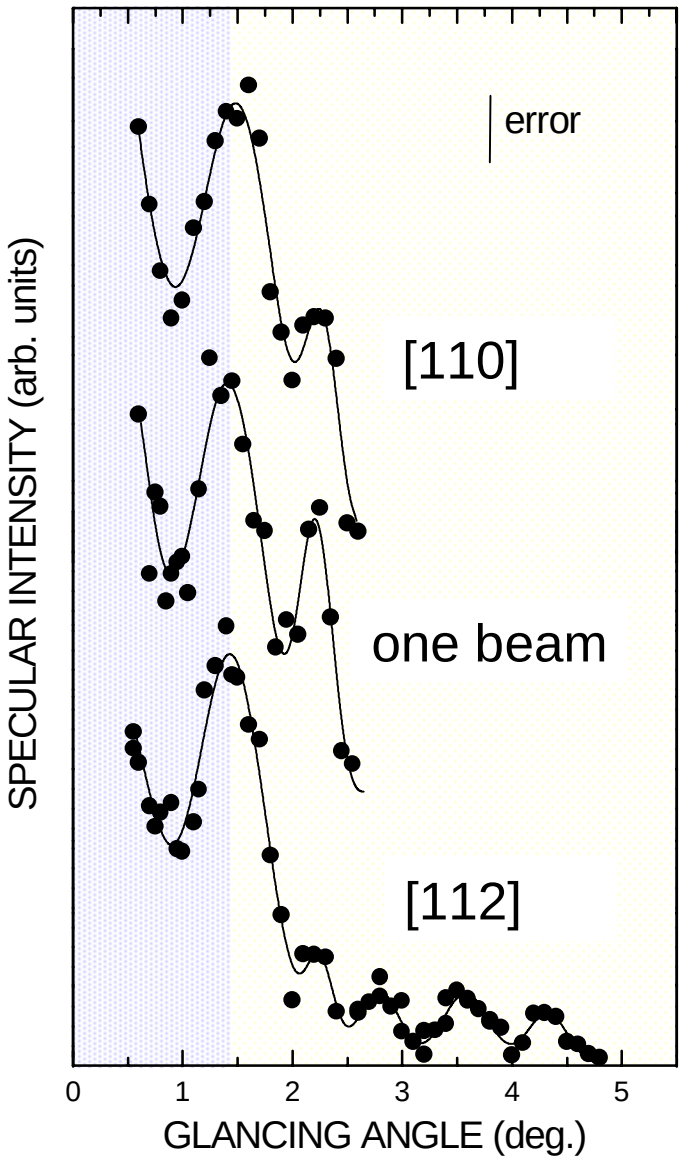
AFM

Roughness

2 ▪ 3 ▪

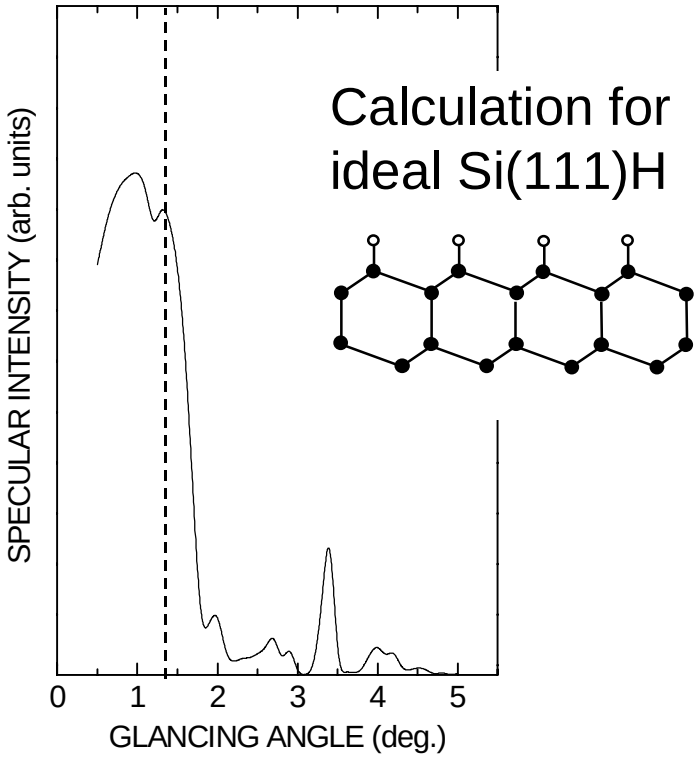


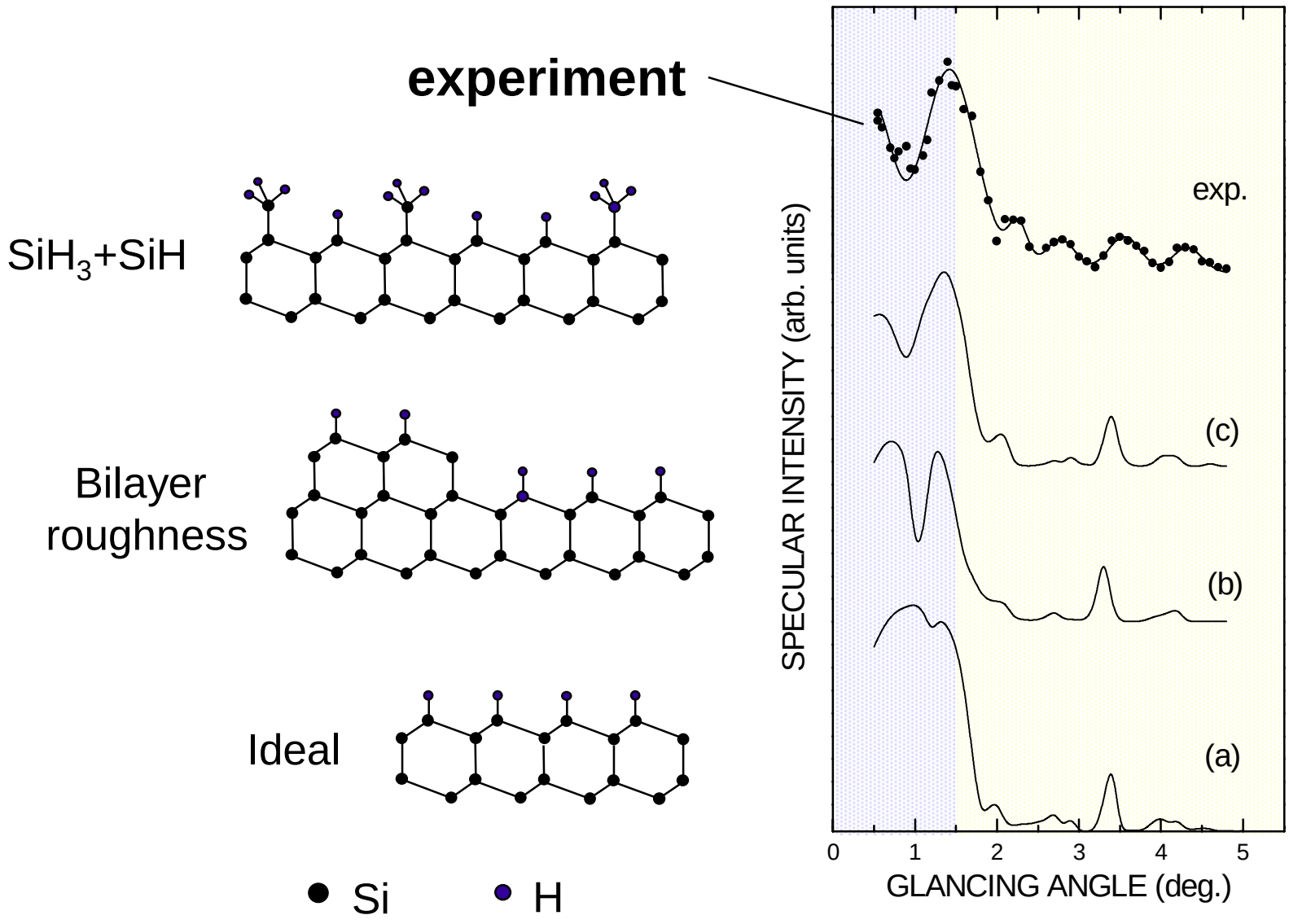
RHEPD rocking curve

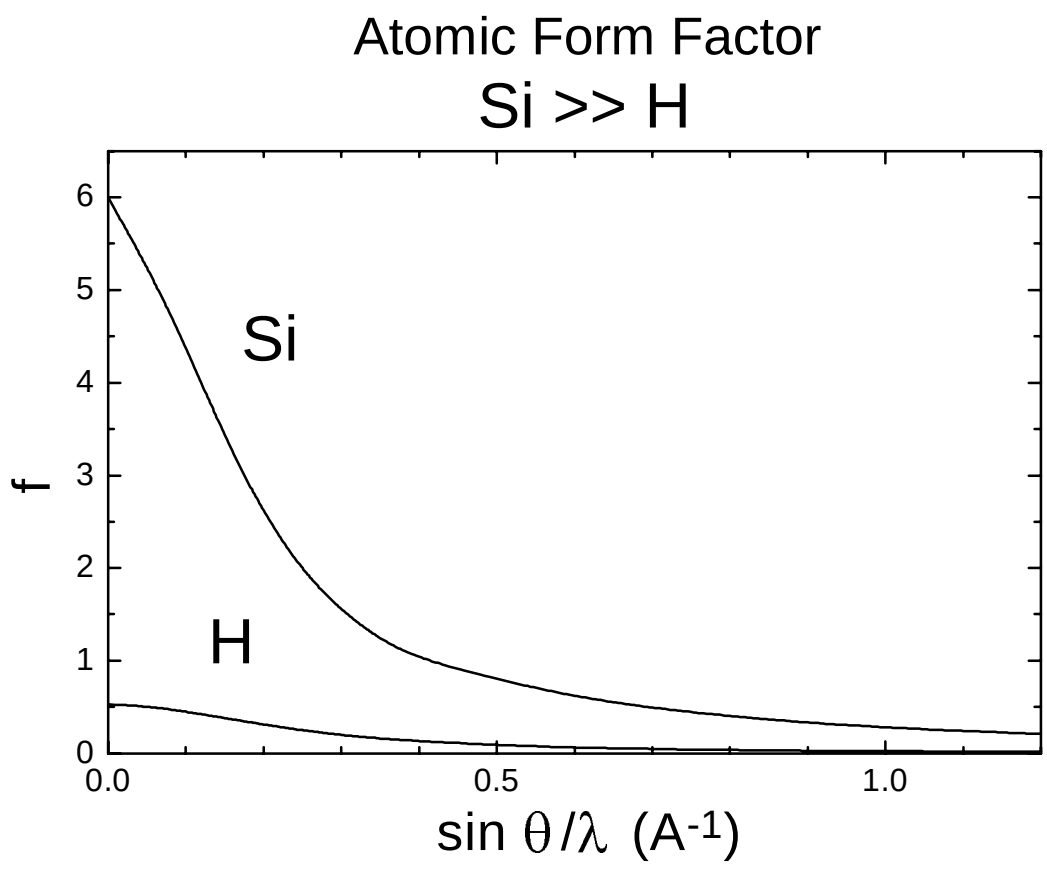


n=1-5 Bragg peaks

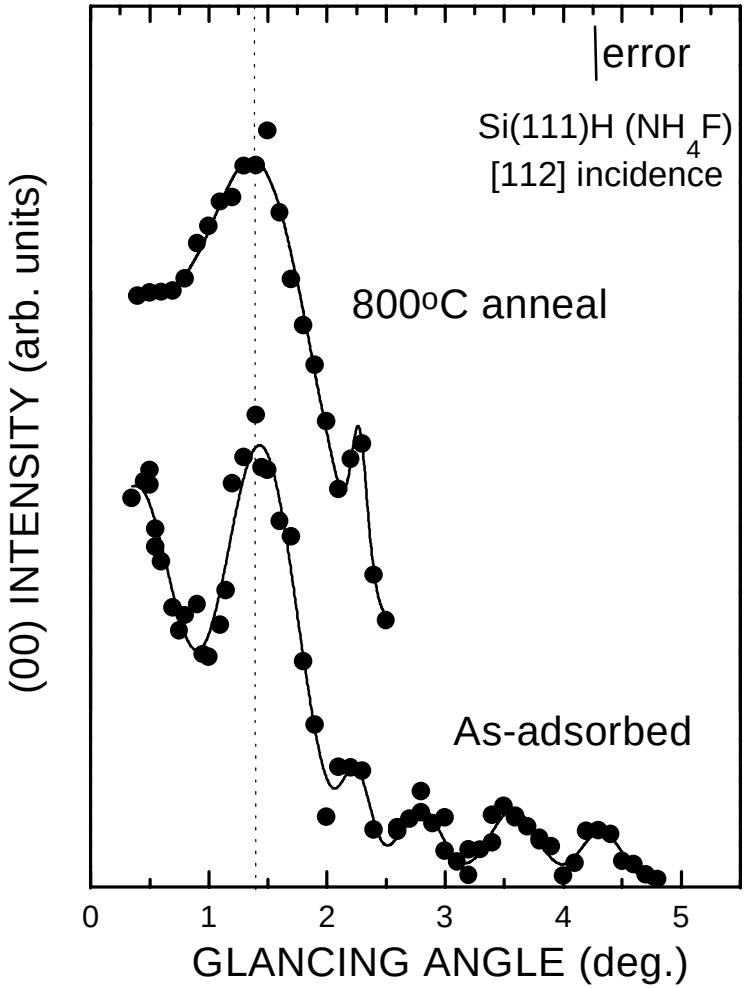
Total Reflection Region
(θ ▪ 1.4º)
"Dip structure"
independent of azimuth




experiment
SiH3+SiH
Bilayer roughness
Ideal
Si
H
exp.
(c)
(b)
(a)
SPECULAR INTENSITY (arb. units)
0
1
2
3
4
5
GLANCING ANGLE (deg.)

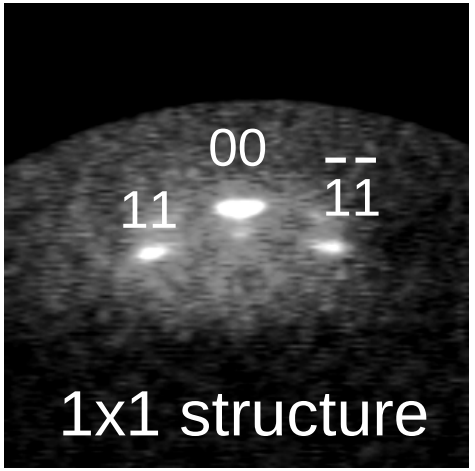

Atomic Form Factor
Si >> H
Si
H
f
0
1
2
3
4
5
6
0.0
0.5
1.0
sin θ/λ (A-1)

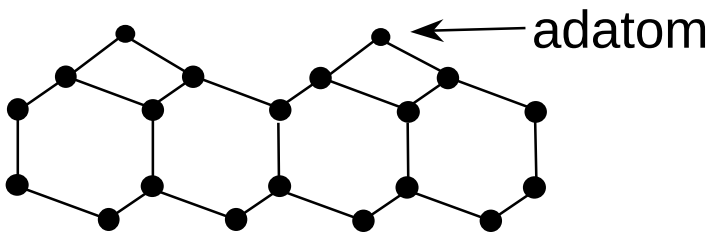
Effect of heat treatment



Disapperance of structure

800 ▪ anneal





Why hydrogen etching of SiC(0001) ?

From SiC technology…

- Elimination of surface roughness
- Hydrogen passivation

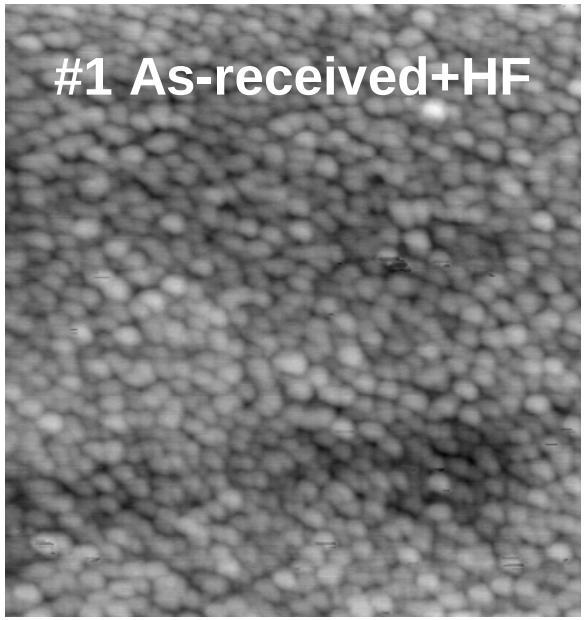
Chemical HF etching is ineffective for SiC
□High temperature H_2 annealing

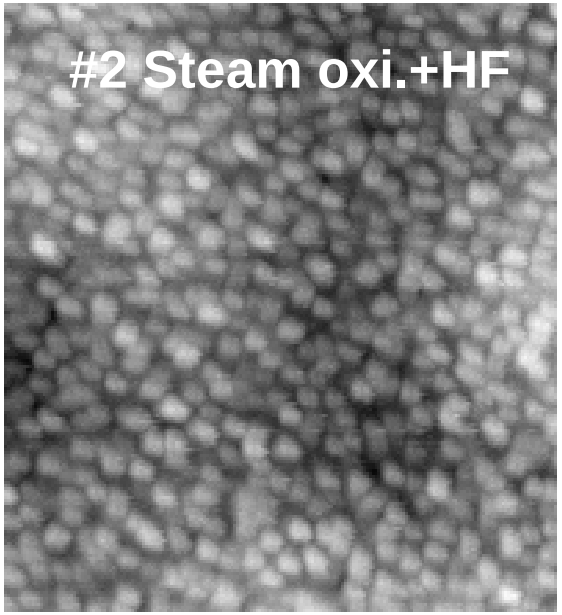
6H SiC (0001) surface

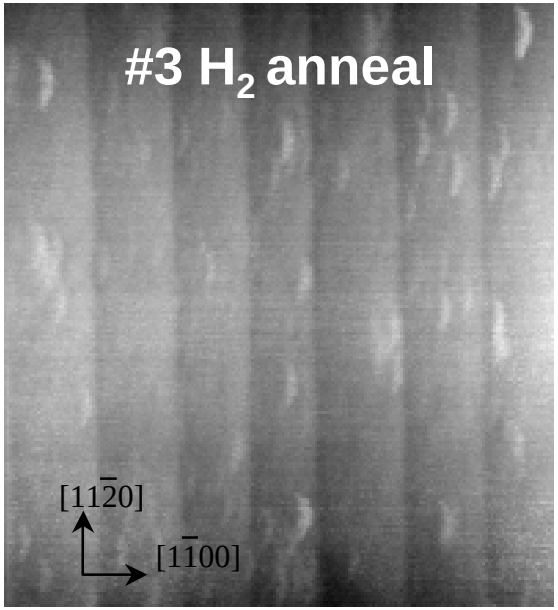
#1 As-received+HF
#2 Steam oxidation ▪ 1100 ▪ x2h ▪ +HF
#3 H_2anneal(1400 ▪ x8h, 100Torr, 2slm)
#4 H_2anneal+oxidation(800 ▪ x4h)+HF

AFM observation

500 nm

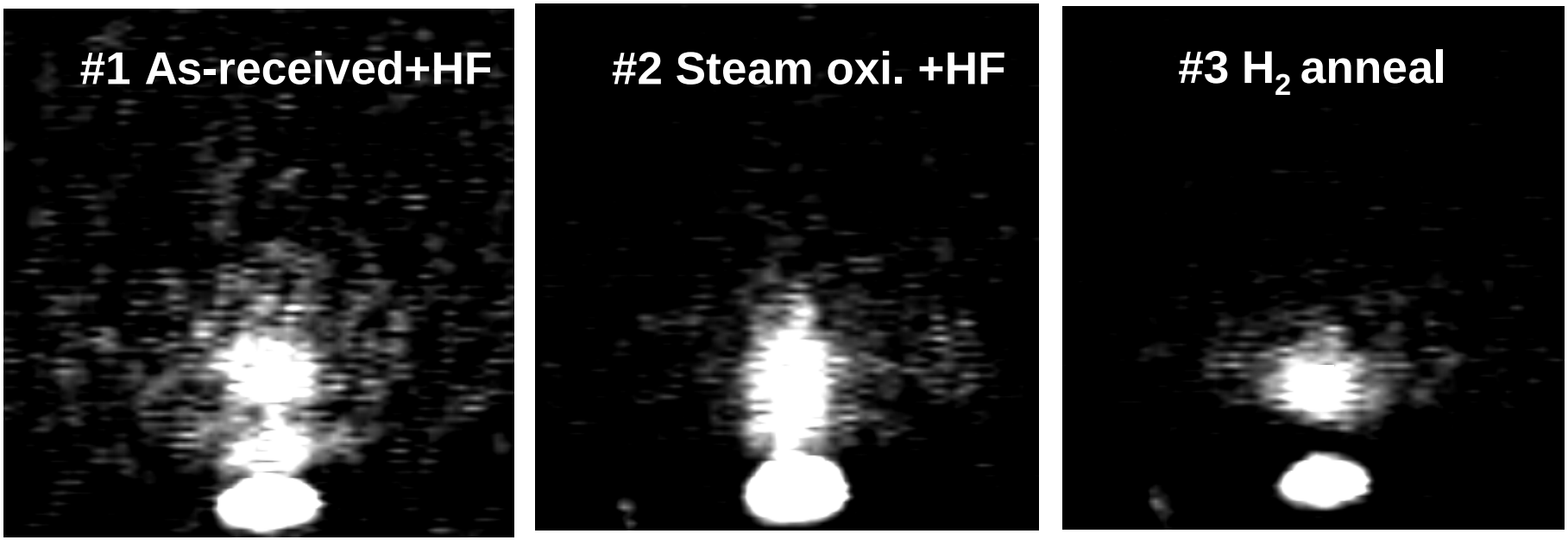


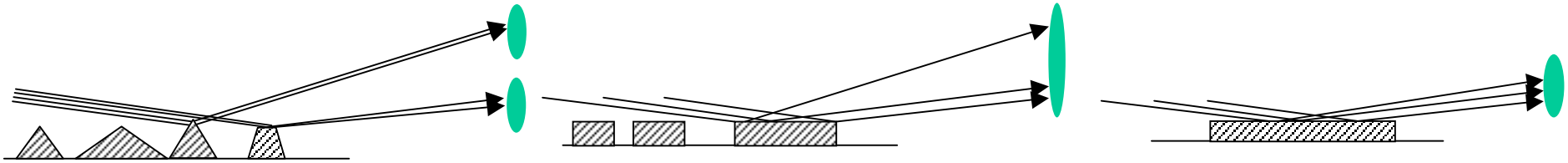




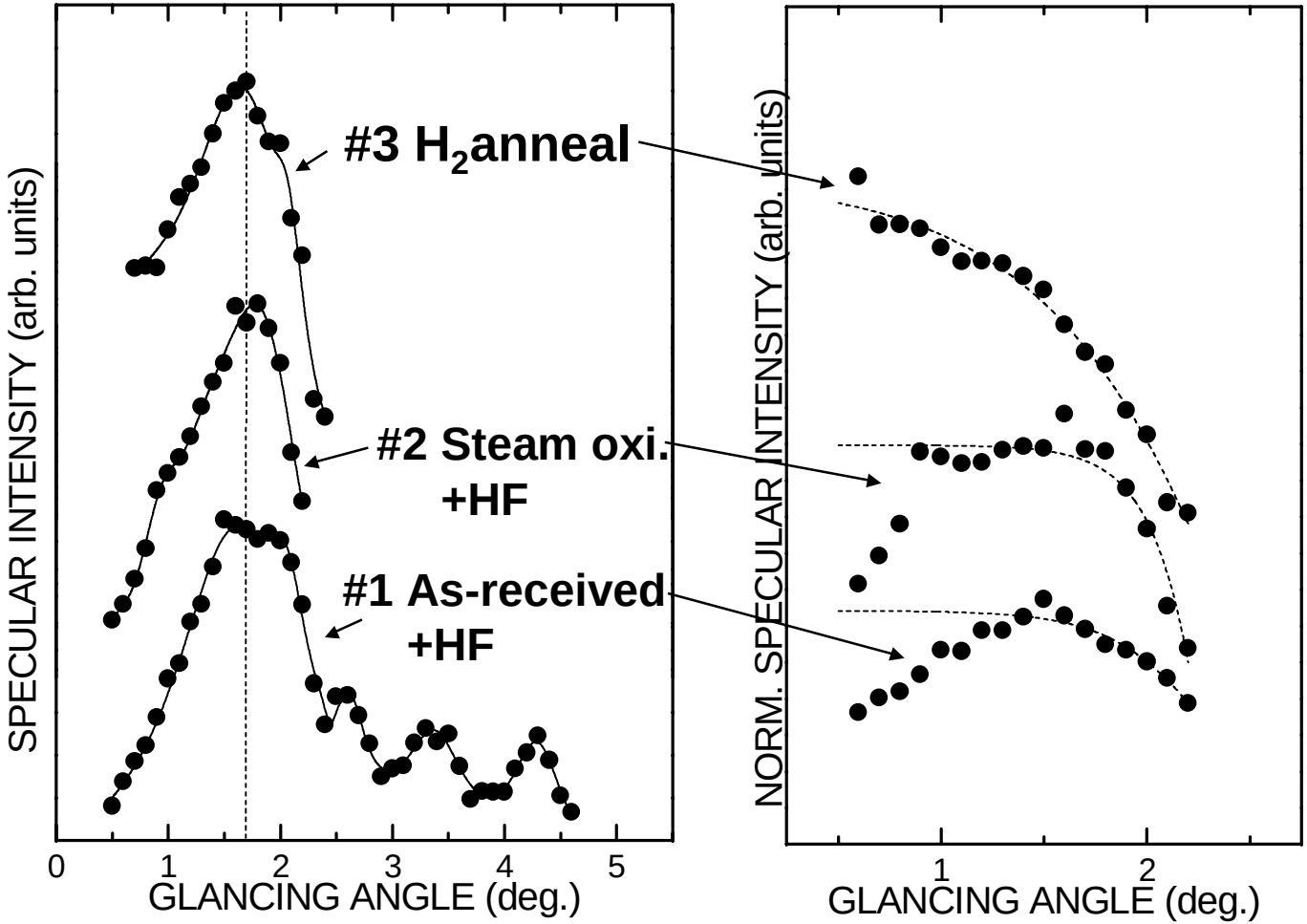
R*q*>10Å **R*q* ~10Å** **R*q*<1Å**

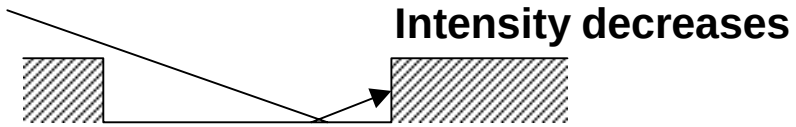
RHEPD Total Reflection Pattern



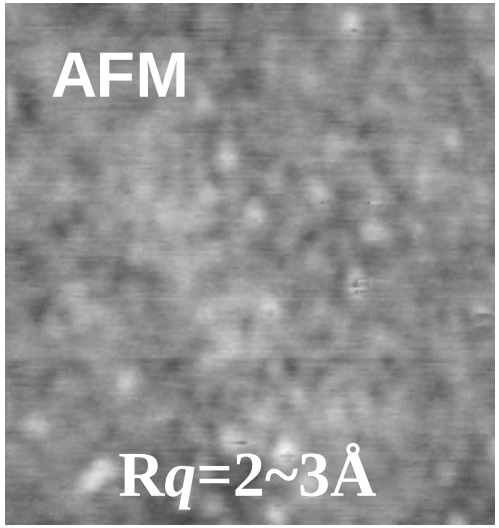


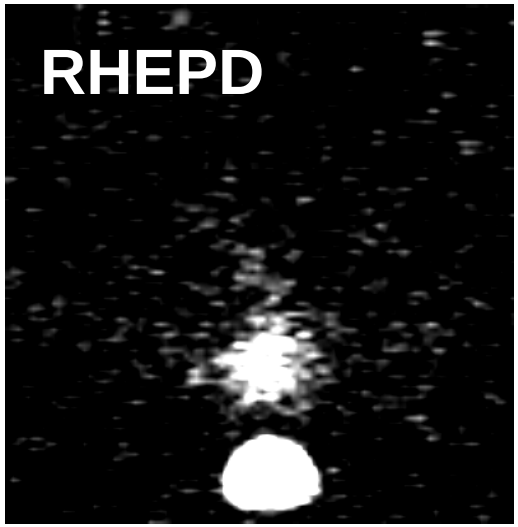
Rocking curves

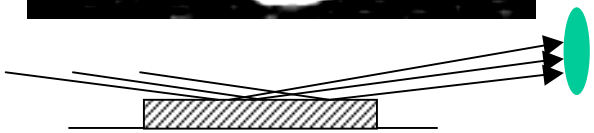


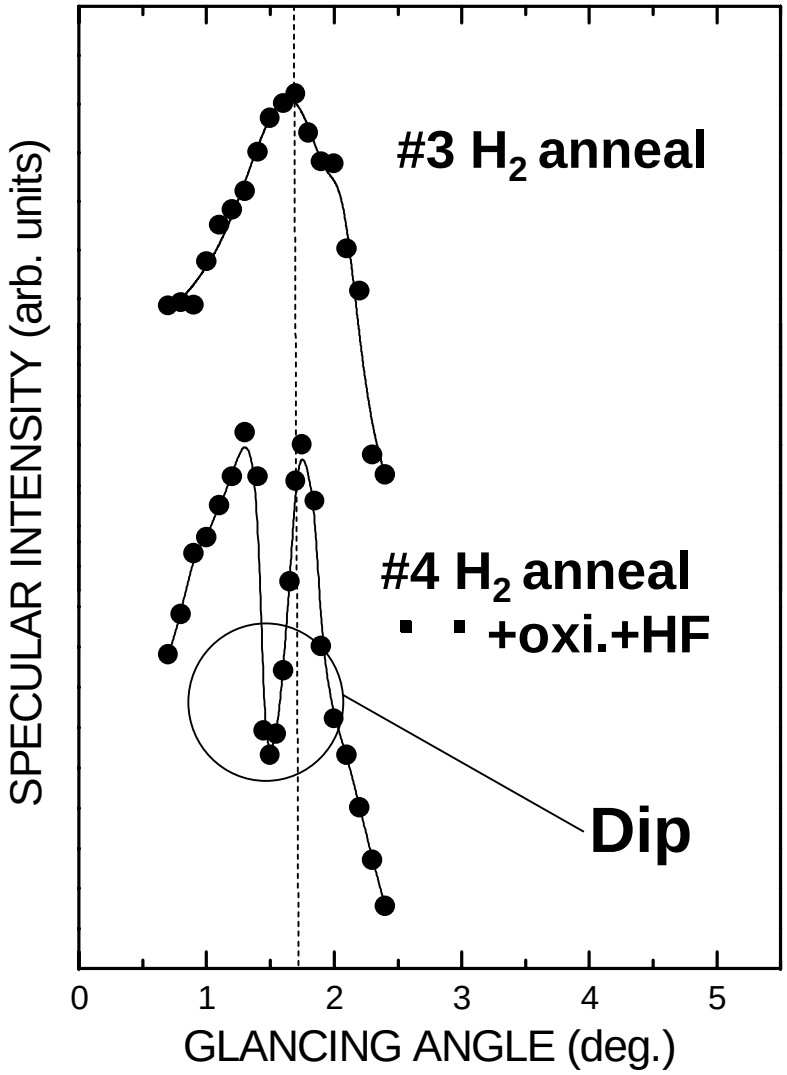


Effect of oxidation after H_2 annealing

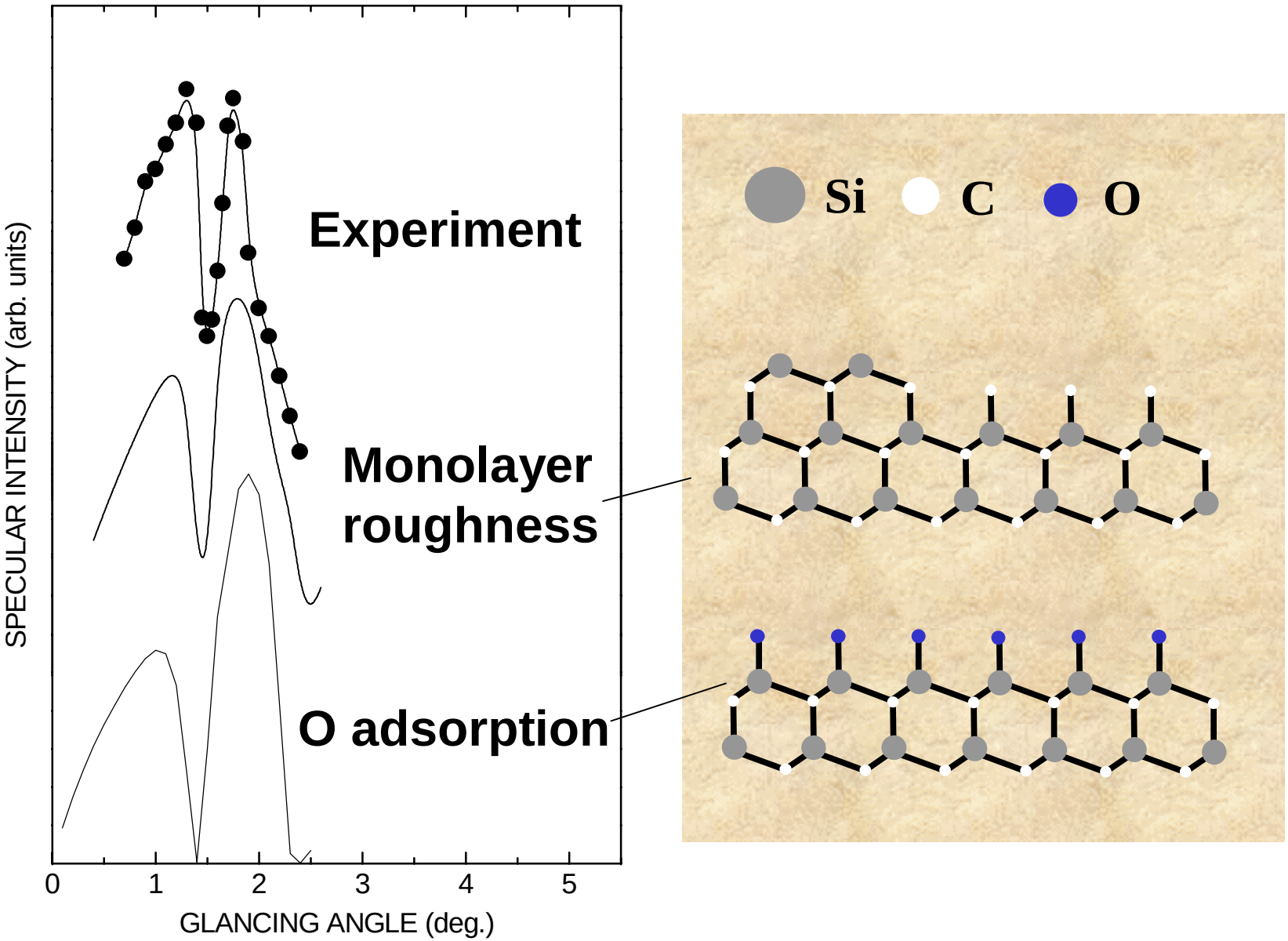




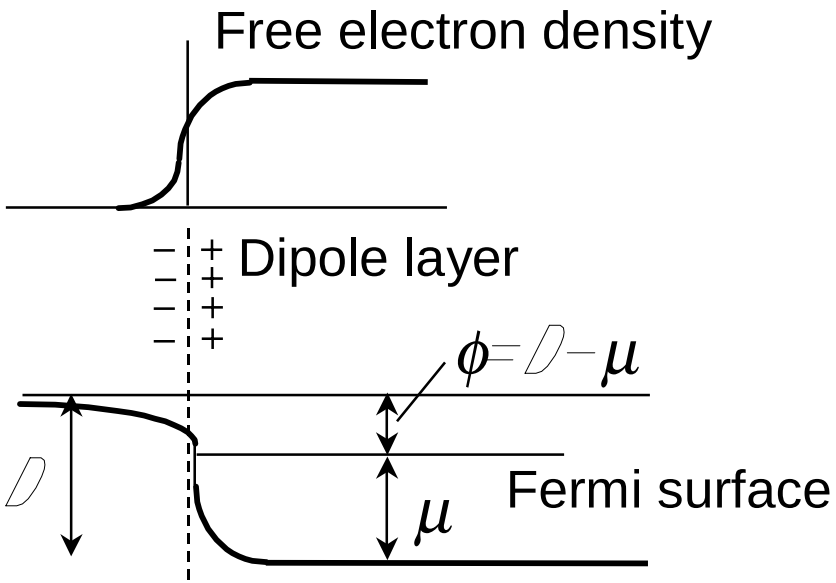




Dynamical Calculation with “Roughness”



Oliva's idea for surface dipole barrier

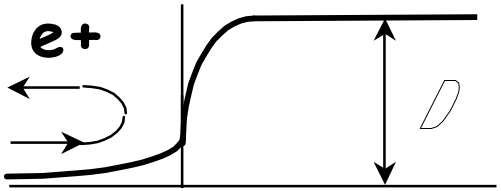


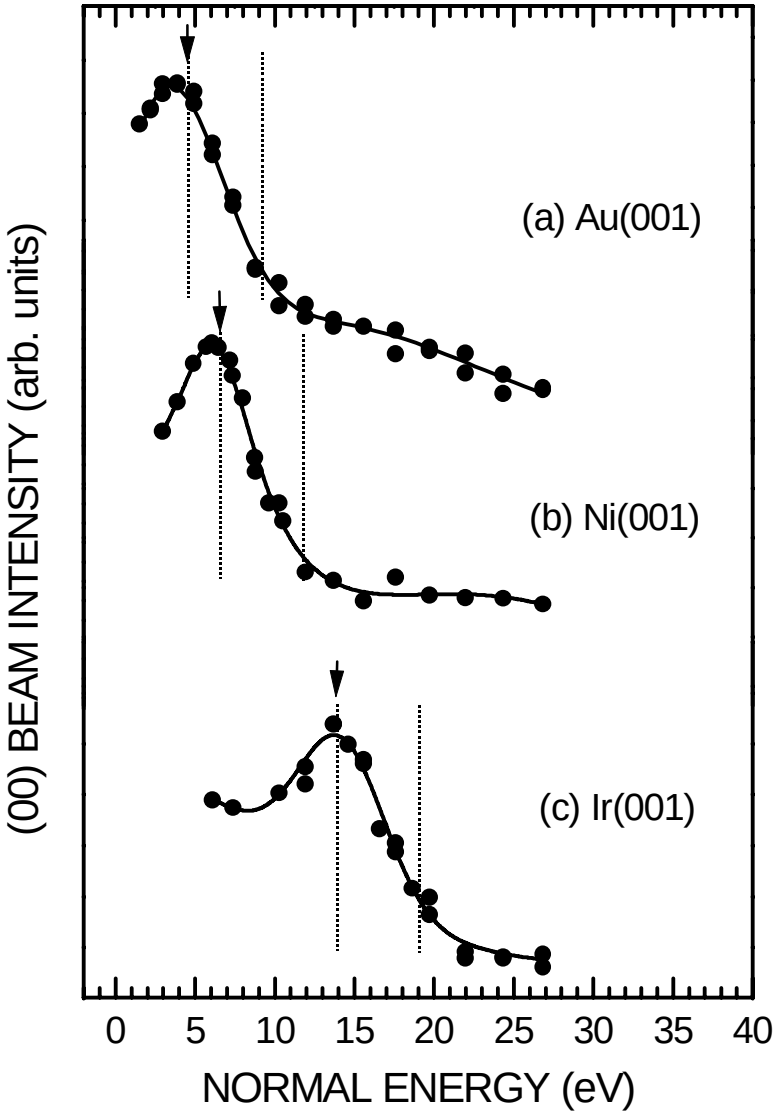
Φ: measurable

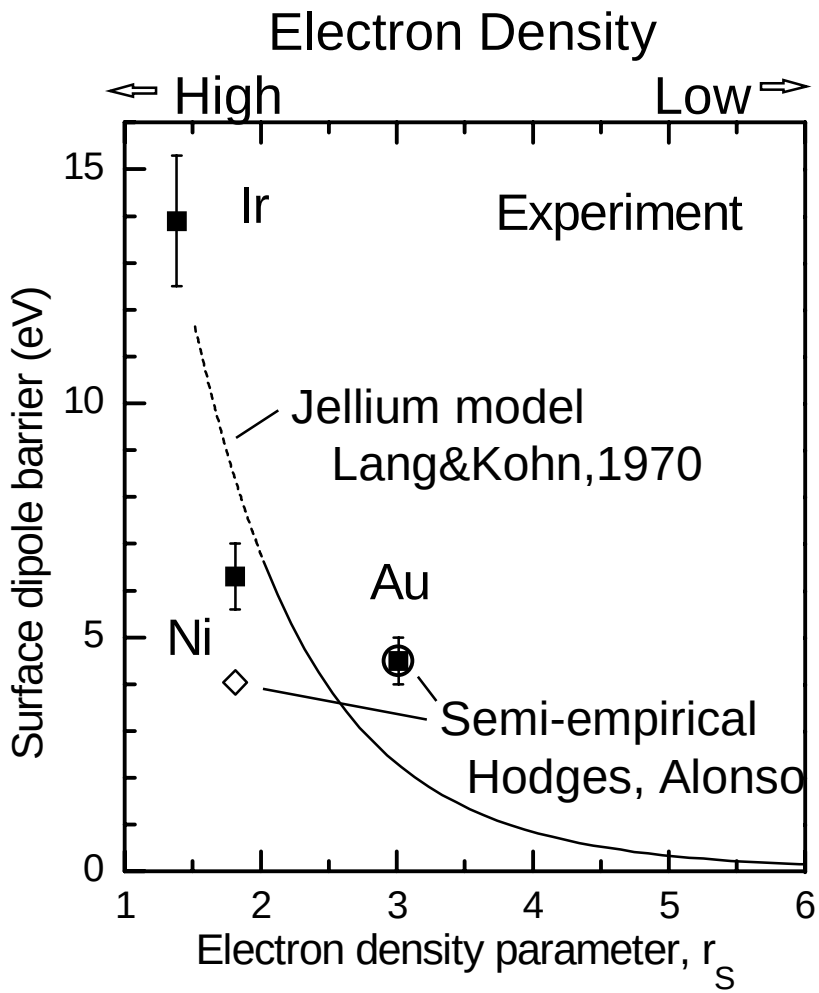
No direct methods for *D*

RHEPD should give determines *D*

since e+ reflectivity drops down at E .> D




(00) BEAM INTENSITY (arb. units)
(a) Au(001)
(b) Ni(001)
(c) Ir(001)
0
5
10
15
20
25
30
35
40
NORMAL ENERGY (eV)


Electron Density
High
Low
Ir
Experiment
Jellium model
Lang&Kohn,1970
Au
Ni
Semi-empirical
Hodges, Alonso
Surface dipole barrier (eV)
0
5
10
15
1
2
3
4
5
6
Electron density parameter, r_S

4. Summary

- **Establishment of RHEPD technique**
  - Diffraction Pattern
  - Total reflection & 1st Bragg peak etc.
- **Application of RHEPD**
  - Residual roughness on Si(111)H surface
  - Ideally flat SiC(0001) surface by H-etching
  - Surface dipole barrier of some metals
- **Feature plan**
  - Adsorbed layer structure analysis
  - Precise surface Debye parameter