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**

<table>
<thead>
<tr>
<th></th>
<th>TPD</th>
<th>LEPD</th>
<th>RHEPD</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Type</strong></td>
<td>Transmission</td>
<td>Low Energy</td>
<td>Reflection High</td>
</tr>
<tr>
<td></td>
<td>Positron</td>
<td>Positron</td>
<td>Energy Positron</td>
</tr>
<tr>
<td></td>
<td>Diffraction</td>
<td>Diffraction</td>
<td>Diffraction</td>
</tr>
<tr>
<td><strong>Energy</strong></td>
<td>100keV - 1MeV</td>
<td>0 - 500eV</td>
<td>0 - 100keV</td>
</tr>
<tr>
<td><strong>Study</strong></td>
<td>Bulk Study</td>
<td>Surface Study</td>
<td></td>
</tr>
</tbody>
</table>

**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**


- Primary Bragg Reflection
- Total Reflection

![Diagram showing primary and total reflection with negative and positive inner potentials](image-url)
Bragg condition: \( E \sin^2 \theta = 37.5 \, n^2/d^2 + eV_0 \)

**Primary Bragg peak**

Si(111) \( \cdot \) : \( d = 3.14 \, \text{Å} \)

\[ eV_0 = \begin{cases} 
-12 \, \text{eV} \text{ for } e^- \\
+12 \, \text{eV} \text{ for } e^+ 
\end{cases} \]

\[ E \sin^2 \theta \begin{cases} 
<0 \text{ for } n=1 \\
>0 
\end{cases} \]

**Total reflection**

\[ E \cdot (E \sin^2 \theta) < eV_0 \]

\[ \theta = \sin^{-1}[eV_0/E]^{1/2} \]
Possible Application of RHEPD

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

Diffraction pattern
Rocking curve
(+Dynamical theory)

Potential of adsorbed atom
surface

Flat surface
Irregular surface

Reflectivity
e+ energy

Reflectivity
e+ energy
2. Establishment of RHEPD

History

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

- **Theoretical description of RHEPD**

What we did to establish RHEPD technique

- e+ beam development
- Confirmation of e+ diffraction phenomenon
- Determination of rocking curve
Present RHEPD apparatus at JAERI

Key elements
• Positron beam 10 -20 keV
• Low angular divergence <1°
• Small beam diameter <1 mm

\[ \sin \theta_d \times d \times E^{1/2} = \text{constant} \]

- \( \theta_d \): angular divergence
- \( d \): diameter
- \( E \): energy

Acceleration up to 20 keV

Sufficient reduction of \( \theta_d \times d \)

c.f.

\( E_{\text{ini}} = 3 \text{ eV}, \quad d_{\text{ini}} = 7 \text{ mm}, \quad \theta_d = 5^\circ \)

\( E_{\text{fin}} = 20 \text{keV}, \quad d_{\text{fin}} = 5 \text{ mm}, \quad \theta_d < 0.1^\circ \)

- Beam operation in "Defocus mode"
- Long-length collimator \( \rightarrow \) "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.

![Graph showing gain vs. bias (kV)]

Max gain $4 \times 10^6$

One RHEPD pattern: 1-10 hrs.
First RHEPD pattern from Si(111)H

[1\overline{1}2] 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] \( \equiv \) [112] for Si(111)
Specular spot \( \equiv \) Diffraction spots (Reciprocal lattice)

Transvers symmetry vanishes in one-beam condition
[112] • incidence

One beam condition
7.5° • off • from [112]
First RHEPD Rocking Curves

**Positron**

- E = 20 keV
- n = 1
- Total reflection

**Electron**

- E = 10 keV
- n = 2
- n = 3
- n = 4
- n = 5

(00) Spot Intensity (arb. units) vs. Glancing Angle (deg.)
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)

- Boiling in HNO₃, 10 min
- Dip in 1% HF
- Rinse in H₂O
- Dip in NH₄F, 20 min

IR spectrum

- Si-H LVM
- Absorbance (arb. units)

Wavenumber (cm⁻¹)

2050
2100
2150

2084 cm⁻¹

AFM

Roughness
2 - 3
RHEPD rocking curve

n=1-5 Bragg peaks

Total Reflection Region
(θ ≈ 1.4°)
“Dip structure”
independent of azimuth

Calculation for
ideal Si(111)H

One beam

[110]

[112]

SPECULAR INTENSITY (arb. units)

GLANCING ANGLE (deg.)

0 1 2 3 4 5

0 1 2 3 4 5
SiH₃+SiH

Bilayer roughness

Ideal

\[ \text{SPECULAR INTENSITY (arb. units)} \]

GLANCING ANGLE (deg.)

experiment

exp.

(a)

(b)

(c)
Atomic Form Factor

Si >> H
Effect of heat treatment

Disappearance of structure

800°C anneal

1x1 structure

adatom
Why hydrogen etching of SiC(0001) ?

From SiC technology…
- Elimination of surface roughness
- Hydrogen passivation

Chemical HF etching is ineffective for SiC
- High temperature H₂ annealing

6H SiC (0001) surface
#1 As-received+HF
#2 Steam oxidation • 1100 • x2h • +HF
#3 H₂anneal(1400 • x8h, 100Torr, 2slm)
#4 H₂anneal+oxidation(800 • x4h)+HF
AFM observation

#1 As-received+HF

#2 Steam oxi.+HF

#3 H₂ anneal

Rq > 10 Å

Rq ~ 10 Å

Rq < 1 Å
RHEPD Total Reflection Pattern

#1 As-received + HF
#2 Steam oxi. + HF
#3 H₂ anneal
012345

SPECULAR INTENSITY (arb. units)

GLANCING ANGLE (deg.)

NORM. SPECULAR INTENSITY (arb. units)

GLANCING ANGLE (deg.)

#1 As-received +HF

#2 Steam oxi. +HF

#3 H₂ anneal

Intensity decreases

Rocking curves
Effect of oxidation after H$_2$ annealing

AFM

R$_g$=2–3Å

RHEPD

SPECULAR INTENSITY (arb. units)

GLANCING ANGLE (deg.)

#3 H$_2$ anneal

#4 H$_2$ anneal

+oxi.+HF

Dip
Dynamical Calculation with “Roughness”

- Experiment
- Monolayer roughness
- O adsorption

SPECULAR INTENSITY (arb. units)
GLANCING ANGLE (deg.)

- Si
- C
- O
Oliva’s idea for surface dipole barrier

$\Phi$: measurable

No direct methods for $D$

RHEPD should give determines $D$

since $e^+$ reflectivity drops down at $E \rightarrow D$
Surface dipole barrier (eV)

Electron density parameter, $r_S$

Experiments

(a) Au(001)
(b) Ni(001)
(c) Ir(001)

Jellium model
Lang & Kohn, 1970

Semi-empirical
Hodges, Alonso
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