Unravelling the systematics in IBSD

C. Bundesmann, R. Feder, T. Lautenschläger, L. Pietzonka, D. Spemann, H. Neumann
Leibniz Institute of Surface Engineering (IOM), Leipzig, Germany
Outline

- Introduction
  - Leibniz institute of Surface Engineering
  - Motivation
  - Fundamentals
- Particle properties
  - Sputtered target particles
  - Scattered primary particles
- Film properties
  - Ion beam sputter deposition of Ag and TiO₂
  - Dual ion beam sputter deposition of SiO₂ and TiO₂
- Summary and outlook
Leibniz Institute of Surface Engineering (IOM)

- Founded: 01.01.1992
- Director: Prof. Dr. A. Anders (formerly LBNL)
- Funding:
  - Federal government and Saxony
  - Research grants and industry
- Employees: >150 (ca. 70 scientists)

Application-oriented basic research related to engineering surfaces

Tools: Ions, electrons, plasmas and photons

Combining physical and chemical approaches
Introduction

Ion Beam Activities at IOM: Applications

- Ion beam etching, sputtering and implantation
  (Figuring, structuring, planarization, smoothing, coating)

![Graph showing hardness vs. temperature and layer thickness](image)

**Layer thickness [μm]**

- Temperature 300 - 400 °C
- LEI 15 min, PIII 60 min
Introduction

Ion Beam Activities at IOM: Developments

- Ion beam sources
- Components
- Diagnostic tools and chambers
- Simulation tools

IOM has unique positions: From tools and processes to application
Introduction

Ion Beam Activities at IOM: Developments

IOM has unique positions: From tools and processes to application

Almost 50 years of expertise with ion beam technologies
Motivation

- Technological background: Increasing demand for films with tailored properties

- Film properties depend on properties of film-forming particles

Deposition technique and process parameters

Energy and flux of film-forming particles

Thin film properties
**Motivation**

- Ion beam sputter deposition
- PVD technique with very good film properties
- Offers unique opportunity to tailor particle and film properties
- Used for decades, but full potential not investigated and used yet

**Our goal:**
Systematic investigation of IBSD process

**Ion beam and geometrical parameters**

**Energy and angular distribution of film-forming particles**

**Thin film properties**
Introduction

Ion-solid interaction

Primary particles

Scattered particles

Sputtered particles

Collision cascade

Implanted particles

Target particles

(1) sputtered from a collision cascade
(2) directly sputtered (single knock-on)

Primary particles

(1) directly scattered at target particle
(2) directly scattered at implanted primary particles
(3) scattered after multiple collisions
(4) sputtered after implantation
**Direct Scattering and Sputtering**

### Direct Scattering

\[ E_P = E_{ion} \left( \frac{m_P \cos(\gamma) \pm \sqrt{m_T^2 - m_P^2 \sin^2(\gamma)}}{m_P + m_T} \right)^2 \]

For \( m_P > m_T \): \( \gamma \leq \arcsin\left( \frac{m_T}{m_P} \right) < 90^\circ \)

### Direct Sputtering

\[ E_T = E_{ion} \frac{4m_P m_T}{(m_P + m_T)^2} \cos^2(\gamma) \]

\( \gamma < 90^\circ \)
Direct Scattering and Sputtering

Directly scattered and sputtered particles can gain energy up to primary ion energy.

Energy depends strongly on scattering angle and mass ratio.

- Ar $\rightarrow$ Ag
- Xe $\rightarrow$ Ag
- Ar $\rightarrow$ Ti
- Xe $\rightarrow$ Ti
- Ar $\rightarrow$ Si
- Xe $\rightarrow$ Si
- Ar $\rightarrow$ O
- Xe $\rightarrow$ O
- Ar $\rightarrow$ Ar

$E_{ion} = 1000$ eV

Direct scattering

Direct sputtering

Xe $\rightarrow$ 131.3 amu
Ag $\rightarrow$ 107.9 amu
Ti $\rightarrow$ 47.9 amu
Ar $\rightarrow$ 39.4 amu
Si $\rightarrow$ 28.1 amu
O $\rightarrow$ 16.0 amu
Ion Beam Techniques

Ion beam etching
- Etch source
- Workpiece

Ion beam sputter deposition
- Target
- Substrate
- Sputter source

Dual ion beam sputter deposition
- Assist source
- Target
- Sputter source
- Substrate
Introduction

**Ion Beam Techniques**

- **Ion beam etching**
  - Etch source
  - Workpiece

- **Ion beam sputter deposition**
  - Sputter source
  - Target
  - Substrate

- **Dual ion beam sputter deposition**
  - Assist source
  - Sputter source
  - Target
  - Substrate
**Experimental Setup and Process Parameters**

- Particle characterization and film deposition
- Systematic variation of ion beam and geometrical parameters

- **Ion energy:** 500 eV – 1500 eV
- **Ion species:** \( O^+, Ar^+, Xe^+ \)
- **Incidence angle:** 0°, 30°, 60°
- **Emission angle:** -40° ... 80°
- **Target material:** Ti, TiO₂, Ag (Ge, Si, SiO₂)
- **O₂-inlet**
**Experimental Setup and Process Parameters**

- Particle characterization and film deposition
- Systematic variation of ion beam and geometrical parameters

**Ion energy:** 500 eV – 1500 eV

**Ion species:** O\(^+\), Ar\(^+\), Xe\(^+\)

**Incidence angle:** 0°, 30°, 60°

**Emission angle:** -40° ... 80°

**Target material:** Ti, TiO\(_2\), Ag (Ge, Si, SiO\(_2\))

**Ion beam source:** Magnetron sputtering

**Geometry fixed**

\[ (\alpha \sim 0^\circ, \beta \sim 0^\circ) \]

\[ \gamma \sim 180^\circ \]
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Energy of sputtered ions

\( E_{\text{ion}} = 1.0 \text{ keV}; \alpha = 30^\circ \)

- \( \beta < 60^\circ \), i.e. \( \gamma > 90^\circ \):
  - Follows Thompson-Model
  - \( \frac{dY}{dE} \propto \frac{E}{(E+U)^3} \)

- \( \beta \geq 60^\circ \), i.e. \( \gamma \leq 90^\circ \):
  - Contributions by directly sputtered particles

Calculations (simple binary elastic collision):

\[
E_T = E_{\text{ion}} \frac{4m_pm_T}{(m_p + m_T)^2} \cos^2(\gamma)
\]

<table>
<thead>
<tr>
<th>( \beta )</th>
<th>Ag (Ar-Ag)</th>
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<tr>
<td>[°]</td>
<td>[eV]</td>
</tr>
<tr>
<td>60</td>
<td>-</td>
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Energy of sputtered ions: Ion energy

- Principal shape of curves stays the same (maximum, shoulder)
- Additional features shifted to higher energies (curves are "stretched")

Energy of sputtered ions: Ion species

- Shape of energy distribution similar for both ion species
- Slightly higher energies for sputtering with Xe
Energy of sputtered ions: Ion incidence angle

\[ E_{\text{ion}} = 1.0 \text{ keV}; \alpha = 30^\circ \]

\[ \beta = 30^\circ, 40^\circ, 50^\circ, 60^\circ, 70^\circ, 80^\circ \]

\[ Y [\text{s}^{-1}] \]

Shape of curves the same when comparing curves with the same \( \gamma \);
e.g. \( \alpha / \beta = 30^\circ / 80^\circ \) and \( 60^\circ / 50^\circ \)

C. Bundesmann, OCLA Symposium, Buchs SG, April 12, 2018
Energy of sputtered ions: Target material

Ge shows no (or less) contributions due to direct sputtering events; appears only for smaller $\gamma$

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**Particle properties (IBS)**

**Energy of scattered ions**

Maxima at low energies (sputtering or charge exchange)

Shoulder \((E < 250 \text{ eV})\): Ar-Ar scattering

Peaks \((E > 250 \text{ eV})\): Ar-Ag scattering

Shift to higher energies with increasing angle

Calculations (simple binary elastic collision):

\[
E_P = E_{ion} \left( \frac{m_p \cos(\gamma) \pm \sqrt{m_T^2 - m_p^2 \sin^2(\gamma)}}{m_p + m_T} \right)^2
\]

<table>
<thead>
<tr>
<th>(\beta) [(^\circ)]</th>
<th>(\text{Ar} (\text{Ar-Ar})) [eV]</th>
<th>(\text{Ar} (\text{Ar-Ag})) [eV]</th>
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<tbody>
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<td>-</td>
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<tr>
<td>80</td>
<td>117</td>
<td>603</td>
</tr>
</tbody>
</table>
Energy of scattered ions: Ion energy

- Principal shape of curves stays the same (maxima, shoulder)
- Additional features shifted to higher energies (curves are "stretched")

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Energy of scattered ions: Ion species

Shoulder due to Xe-Xe scattering the same as for Ar-Ar scattering

Maxima due to Xe-Ag scattering is missing; appears only if $\gamma < 125^\circ$
Particle properties (IBS)

Energy of scattered ions: Ion incidence angle

- Principal shape of curves stays the same when comparing curves with the same $\gamma$; e.g. $\alpha / \beta = 30^\circ / 70^\circ$ and $60^\circ / 40^\circ$
Energy of scattered ions: Target material

shoulder due to Ar-Ar scattering similar
maxima due to Ar-Ge scattering appear at smaller energy than for Ar-Ag scattering

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**Ag films: Electrical resistivity (4PPM)**

Ion incidence angle varied

- $\text{Ar}_1 (60^\circ, 1000 \text{ eV})$
- $\text{Ar}_2 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_4 (30^\circ, 500 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

Ion energy varied

- $\text{Ar}_2 (30^\circ, 1500 \text{ eV})$
- $\text{Ar}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_4 (30^\circ, 500 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

- $\text{Xe}_1 (60^\circ, 1000 \text{ eV})$
- $\text{Xe}_2 (30^\circ, 1500 \text{ eV})$
- $\text{Xe}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Xe}_4 (30^\circ, 500 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

**Electrical resistivity**

- Smaller for films grown by sputtering with Xe ions
- Increases with increasing emission angle
- Mainly influenced by scattering geometry

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*Film properties (IBSD)*

**Ag films: Electrical resistivity (4PPM)**

Ion incidence angle varied

- $\text{Ar}_1 (60^\circ, 1000 \text{ eV})$
- $\text{Ar}_2 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_5 (0^\circ, 1000 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

Ion energy varied

- $\text{Ar}_2 (30^\circ, 1500 \text{ eV})$
- $\text{Ar}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Ar}_4 (30^\circ, 500 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

- $\text{Xe}_1 (60^\circ, 1000 \text{ eV})$
- $\text{Xe}_2 (30^\circ, 1500 \text{ eV})$
- $\text{Xe}_3 (30^\circ, 1000 \text{ eV})$
- $\text{Xe}_4 (30^\circ, 500 \text{ eV})$

$\rho_{\text{Bulk}}$ vs. $\gamma$ [°]

**Electrical resistivity**

- Smaller for films grown by sputtering with Xe ions
- Increases with increasing emission angle
- Mainly influenced by scattering geometry

**Ag films: Electrical resistivity vs. crystallinity (XRD)**

- Films are polycrystalline
- Average grain size correlates with electrical resistivity
- Differences between sputtering with Ar and Xe indicate different scattering mechanisms (grain boundaries and additional defects)

**TiO$_2$ films: Growth rate**

- Growth rate increases with increasing ion energy and ion incidence angle, larger for sputtering with Xe than for sputtering with Ar (sputter yield)
- Tilted in forward direction
- Caused by anisotropy effects in the evolution of collision cascade inside the target

**TiO$_2$ films: Surface roughness (AFM)**

- Films are very smooth
- RMS roughness ($\sigma$) increases with increasing scattering angle
- $\sigma$ depends on ion species: $\sigma$(O) $\leq \sigma$(Ar) $\leq \sigma$(Xe)
- Higher surface mobility due to higher energy of secondary particles

Films are stoichiometric

Implantation of backscattered primary particles (Ar, Xe)

Mainly influenced by scattering geometry

Concentration larger for Ar than for Xe (more directly backscattered Ar ions)

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**TiO₂ films: Optical properties (SE)**

- Index of refraction $n$ shows strong variations
  $\Delta n \sim 0.4$
- $n$ decreases with decreasing scattering angle (higher particle energy)
- $n$ depends on ion species: $n(O) \leq n(Ar) \leq n(Xe)$

*Film properties (IBSD)*
Energy distribution of secondary particles

Additional high-energy contributions due to direct sputtering or scattering events
- Maximum particle energy can gain several 100 eV
- Energy (and signal intensity) depends on ion energy, scattering angle, ion species

\[ \gamma = 180^\circ - \alpha - \beta \]

**TiO₂ films: Optical properties vs. mass density**

Index of refraction $n$ and mass density $\rho$ show strong correlation.

**TiO$_2$ films: Optical properties vs. mass density**

Index of refraction $n$ and mass density $\rho$ show strong correlation.

Systematics fits to data from other PVD techniques (related to different particle energy).

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DIBSD: Stress engineering of SiO$_2$ and TiO$_2$

Reducing intrinsic stress by assisting ion bombardment

Film stress vs. energy of assisting ions

DIBSD: Optical properties of SiO$_2$ and TiO$_2$

- Index of refraction in dependence on energy of assisting ions

Index of refraction gets smaller with increasing ion energy (except bombardment of SiO$_2$ with Xe ions)

Index of refraction is smaller for bombardment with Ar ions

Summary

- Investigations show that IBSD is a technologically interesting deposition technique.
- Process parameters, properties of film-forming particles and film properties reveal strong systematic correlations.
- IBSD can be used to tailor thin film properties over a large range: crystallinity, grain size, porosity, surface roughness, mass density, optical properties, stress, electrical resistivity.
- Most important parameters: scattering geometry and process gas.
- Strong impact of backscattered primary particles.
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