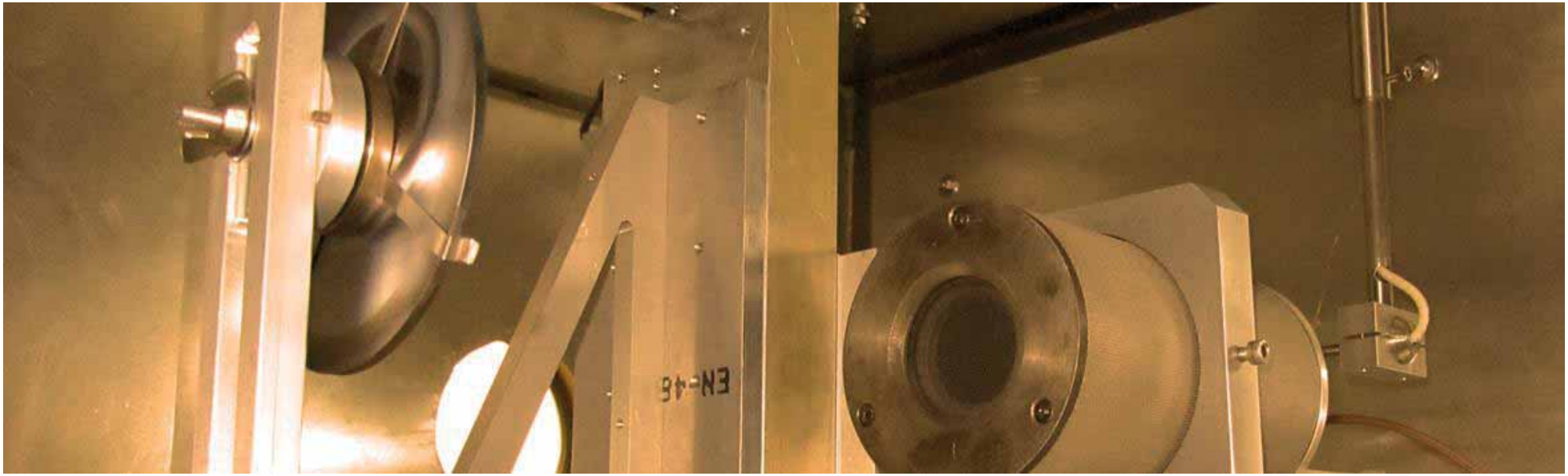


Unravelling the systematics in IBSD

C. Bundesmann, R. Feder, T. Lautenschläger, L. Pietzonka, D. Spemann, H. Neumann

Leibniz Institute of Surface Engineering (IOM), Leipzig, Germany

OCLA Symposium

Buchs SG, Switzerland, April 12, *2018*

IOM

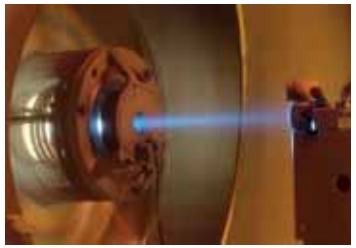
Leibniz Institute of Surface Engineering

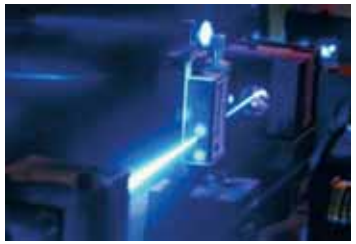
Outline

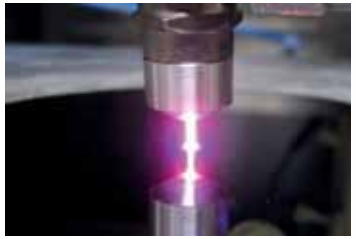
- Introduction
  - Leibniz institute of Surface Engineering
  - Motivation
  - Fundamentals
- Particle properties
  - Sputtered target particles
  - Scattered primary particles
- Film properties
  - Ion beam sputter deposition of Ag and TiO_2
  - Dual ion beam sputter deposition of SiO_2 and TiO_2
- Summary and outlook

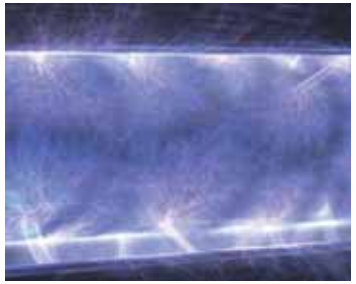
Leibniz Institute of Surface Engineering (IOM)

- 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)





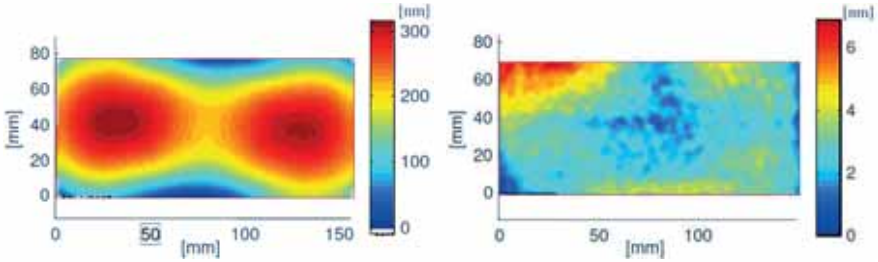


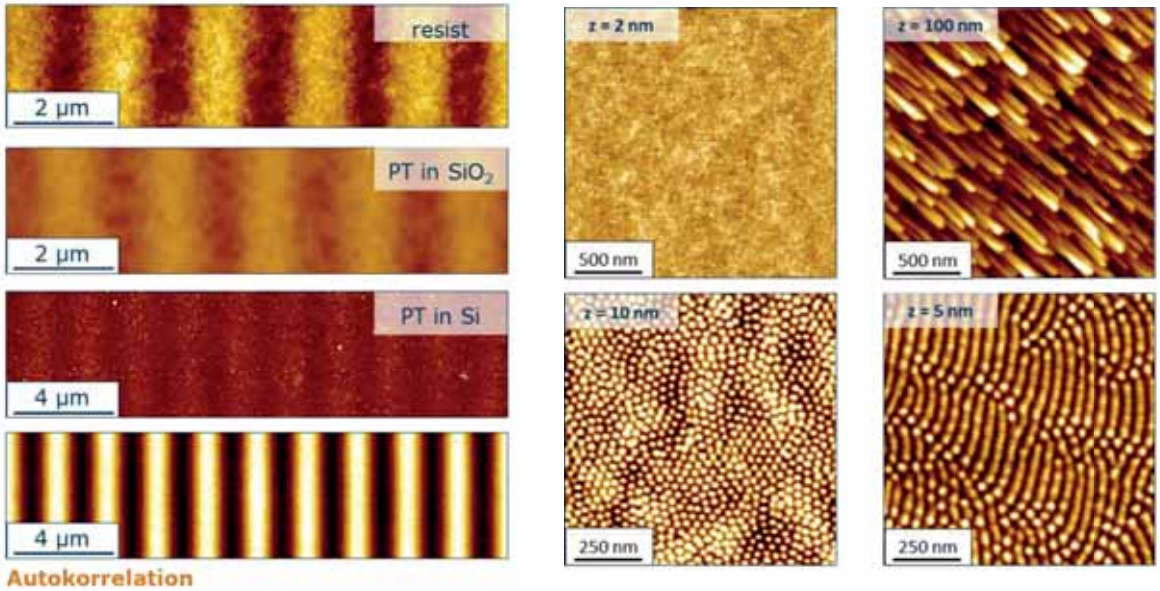


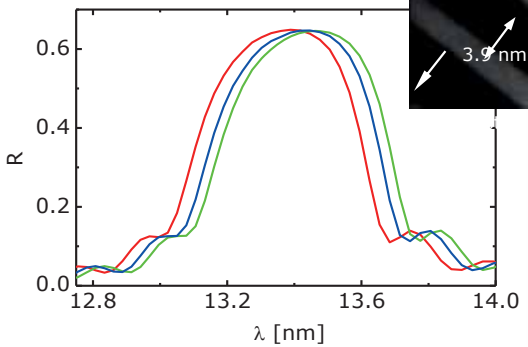
- Application-oriented basic research related to engineering surfaces
- Tools: Ions, electrons, plasmas and photons
- Combining physical and chemical approaches

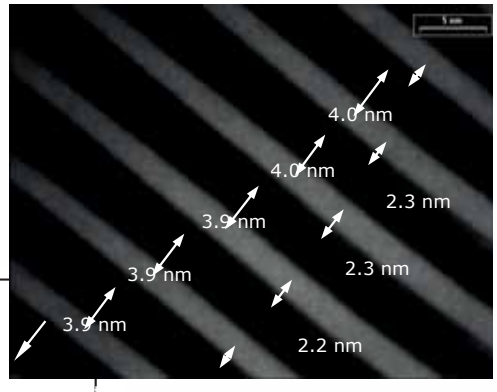
Ion Beam Activities at IOM: Applications

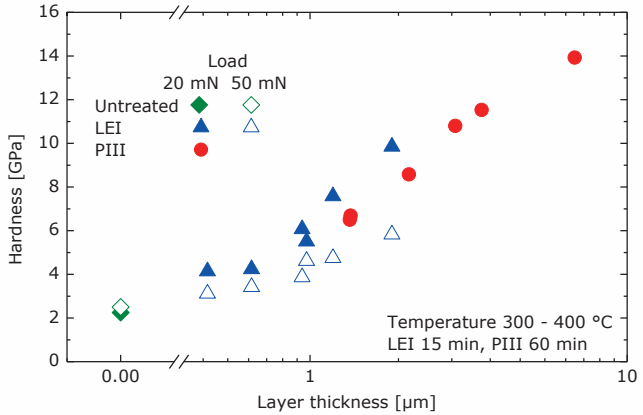
- Ion beam etching, sputtering and implantation
  (Figuring, structuring, planarization, smoothing, coating)





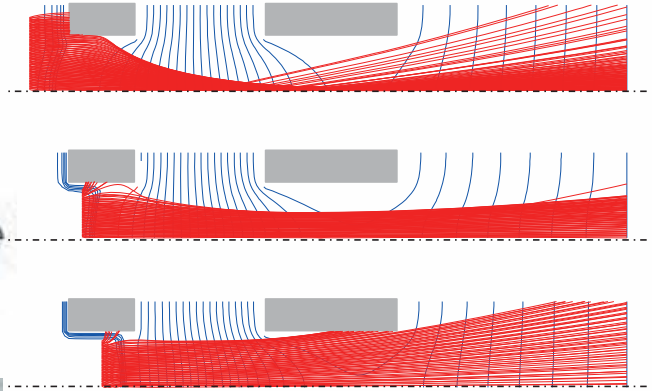


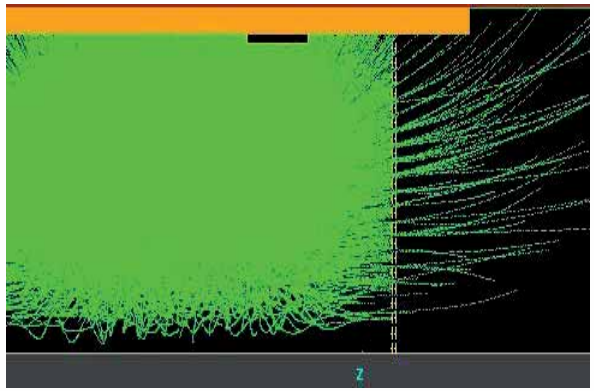




Ion Beam Activities at IOM: Developments

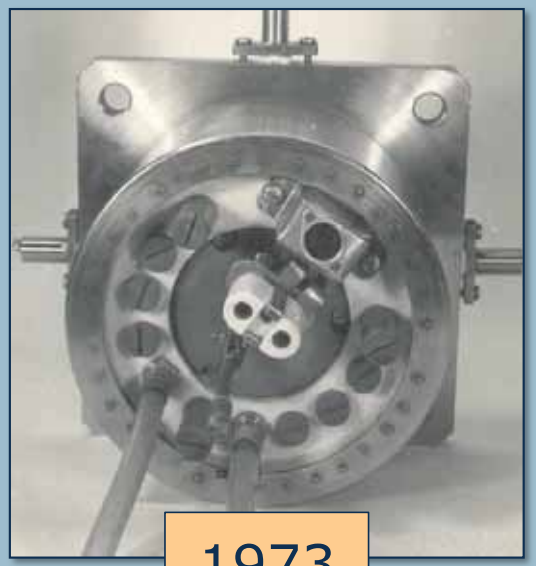
- Ion beam sources
- Components
- Diagnostic tools and chambers
- Simulation tools





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

Ion Beam Activities at IOM: Developments



1973

Рис. 1. Схема изготовления малоугловых структур методом низкотемпературного плазменного травления: 1 – источник ионов (0,3–3,0 кэВ); 2 – маска; 3 – привод; 4 – вакуумная система; 5 – держатель; 6 – образец; 7 – диафрагма

1978

vakuum information

Fields of application

Ion Beam Etching Plant ISA 150

1984

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

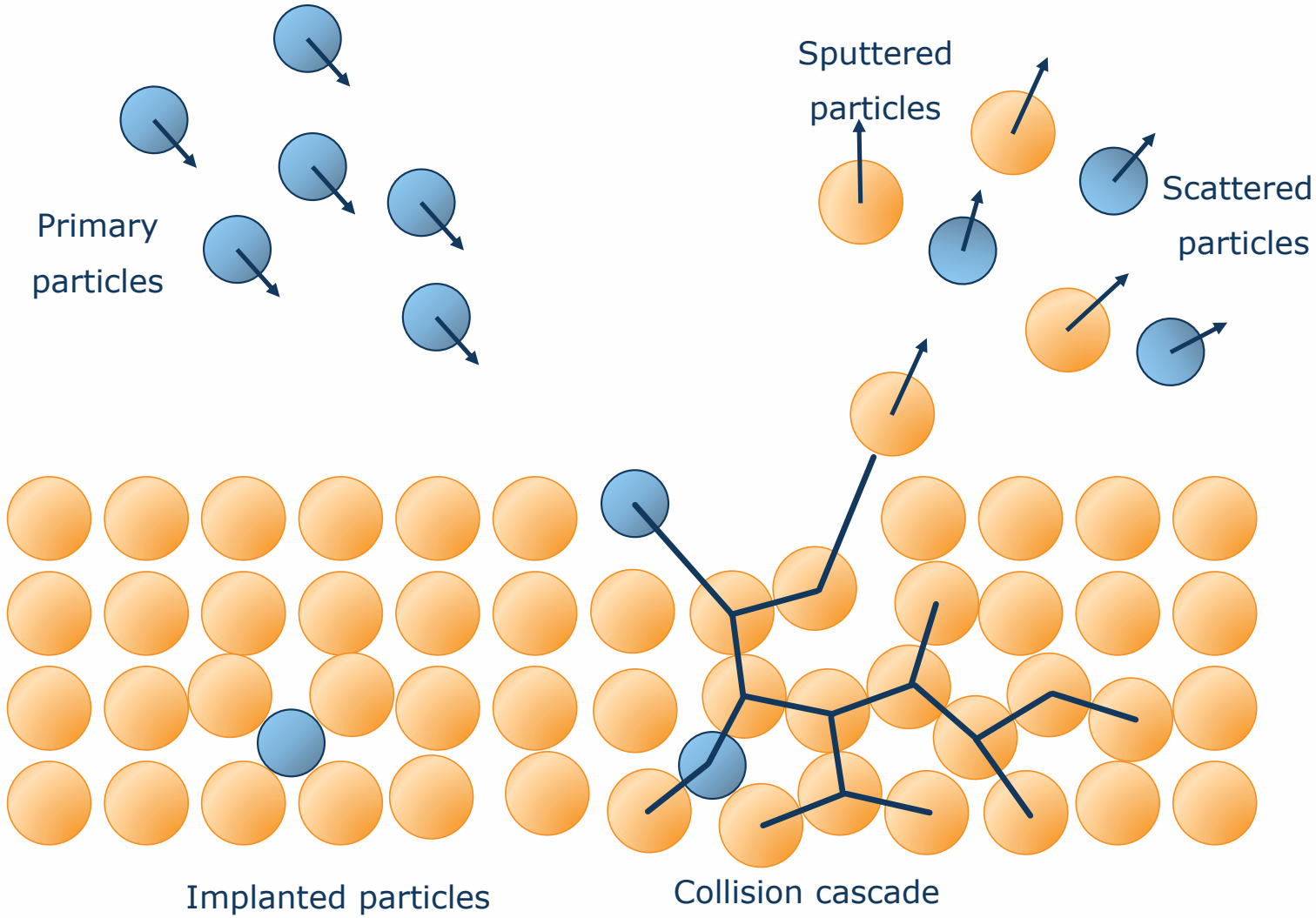
↓

Energy and angular distribution of film-forming particles

↓

Thin film properties

Ion-solid interaction



Target particles

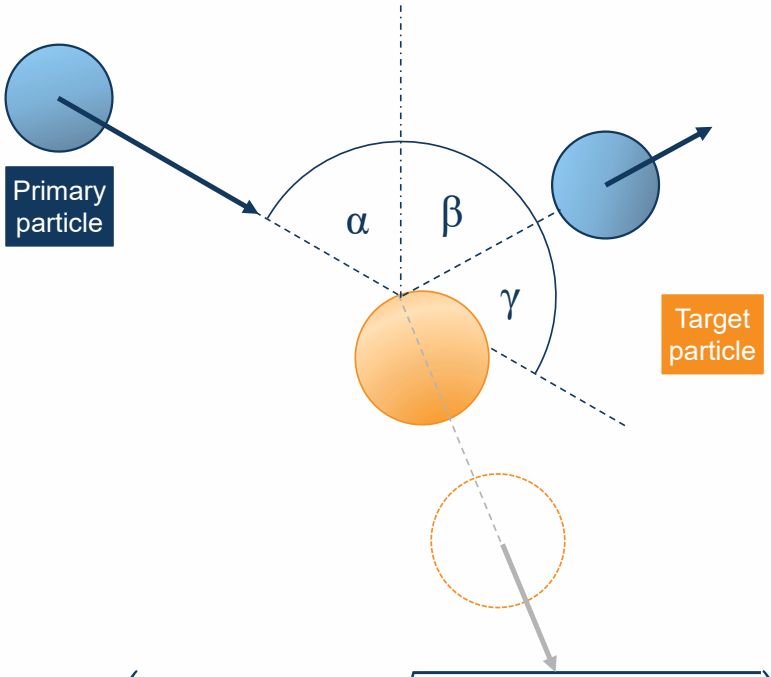
(1) sputtered from a collision cascade
(2) directly sputtered (single knock-on)

Primary particles

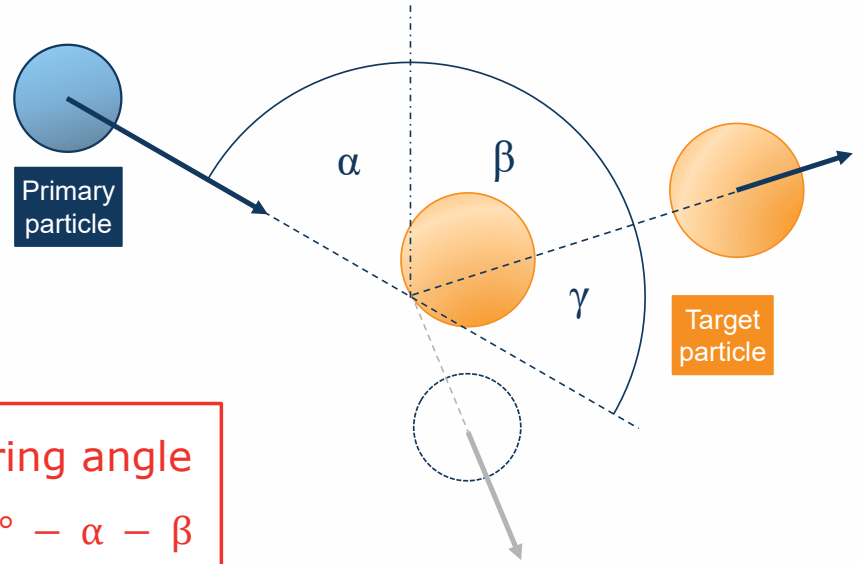
(1) directly scattered at target particle
(2) directly scattered at implanted primary particles
(3) scattered after multiple collisions
(4) sputtered after implantation

Direct Scattering and Sputtering

Direct scattering



Direct sputtering



Scattering angle

$$\gamma = 180° - \alpha - \beta$$

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

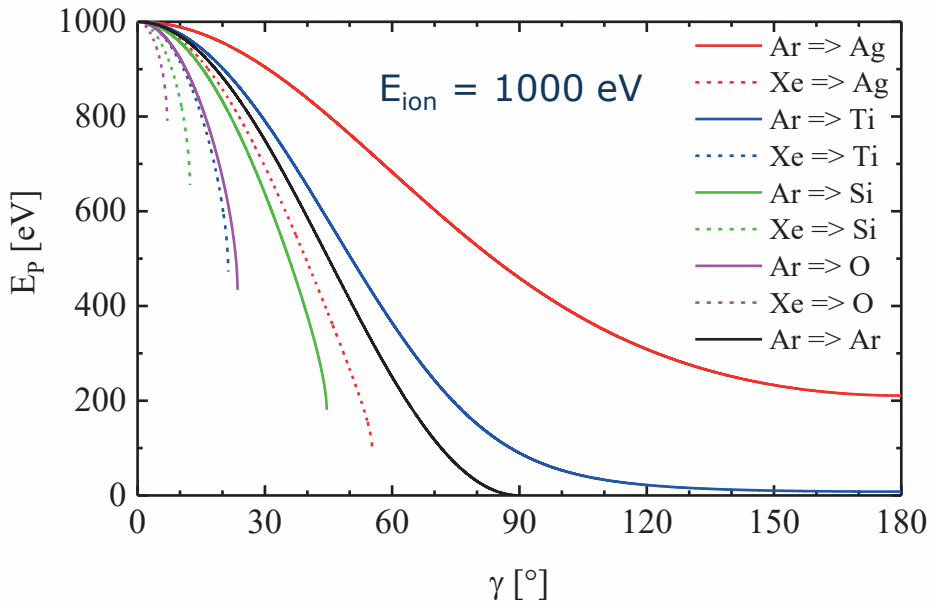
$$m_P > m_T: \quad \gamma \leq \arcsin\left(\frac{m_T}{m_P}\right) < 90°$$

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

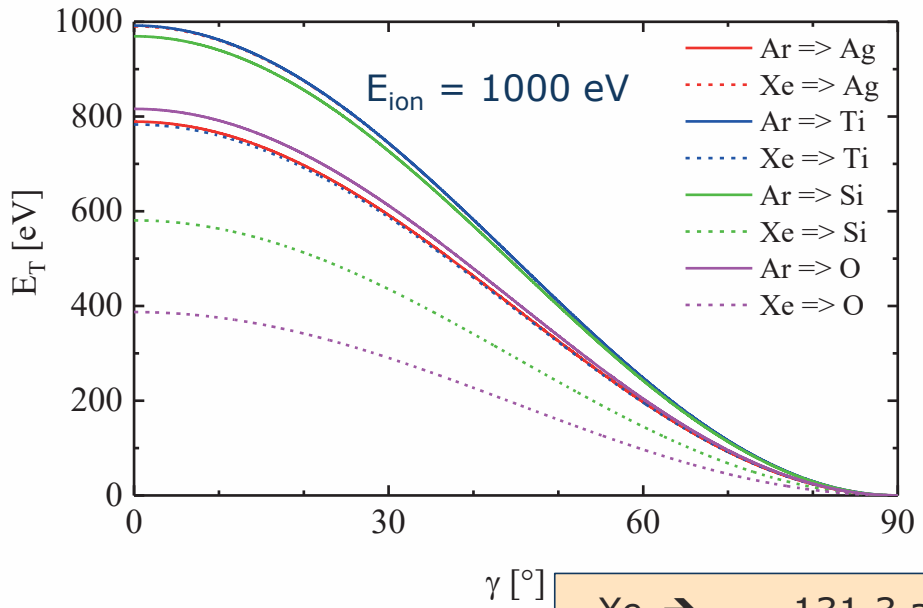
$$\gamma < 90°$$

Direct Scattering and Sputtering

- Direct scattering



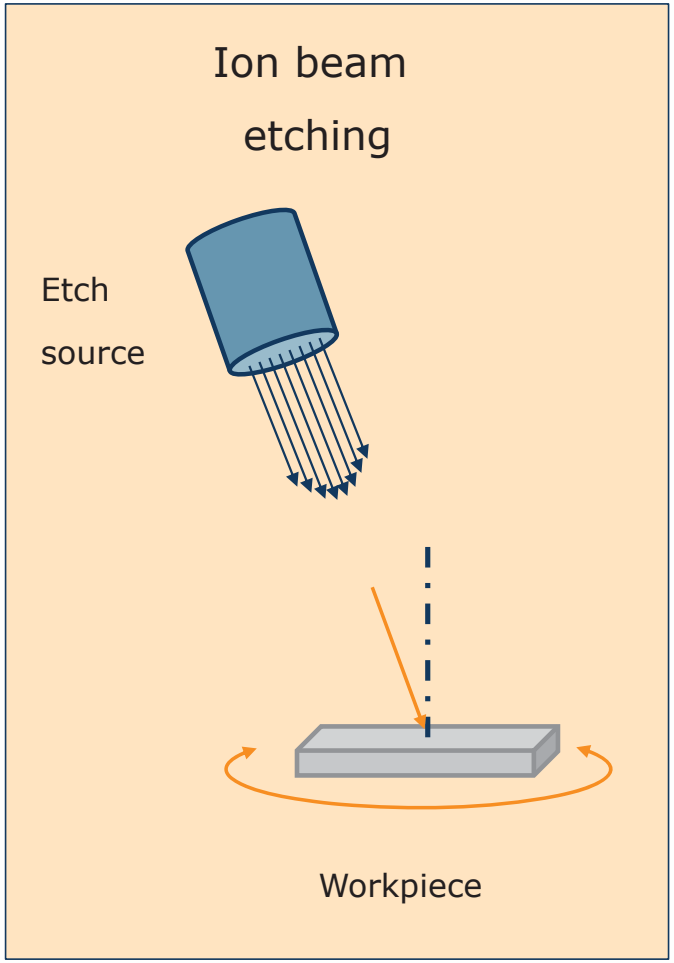
- Direct sputtering

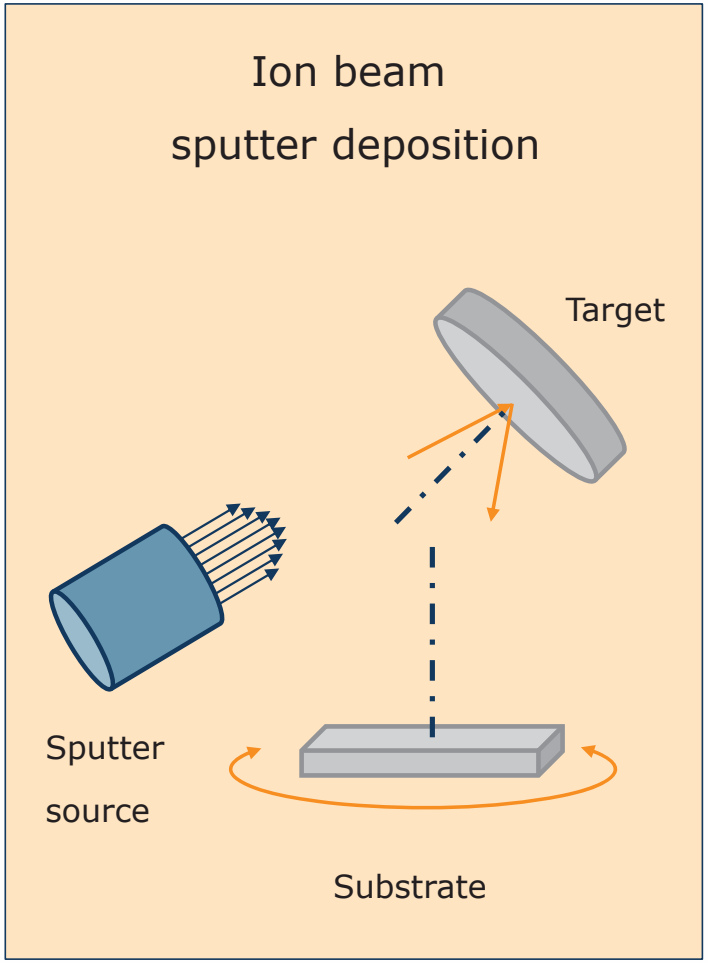


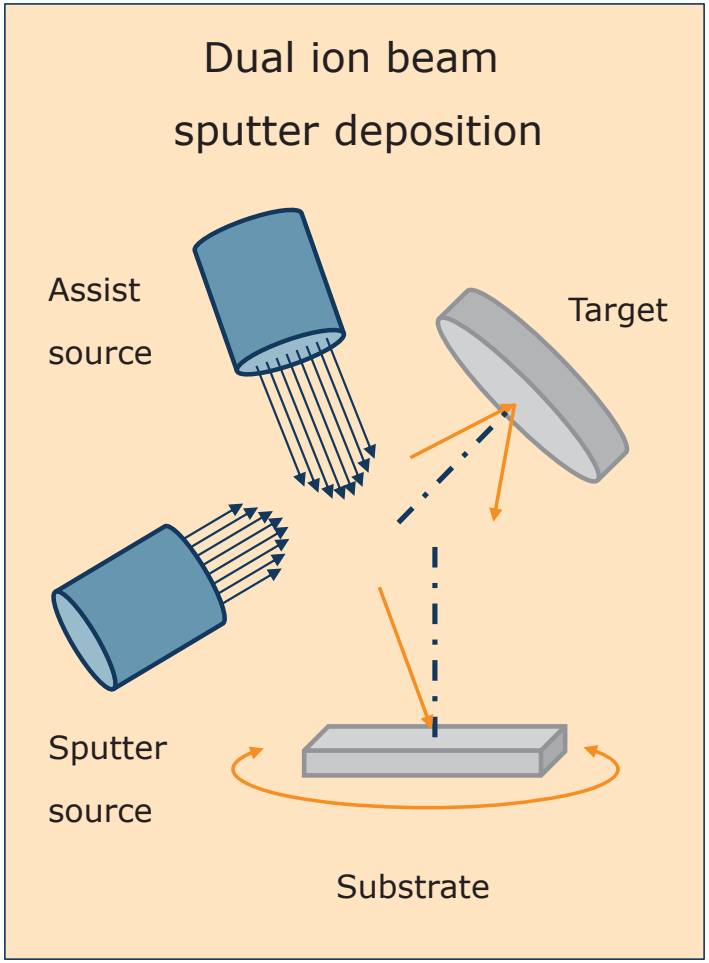
Xe →		131.3 amu
	→ Ag	107.9 amu
	→ Ti	47.9 amu
Ar →		39.4 amu
	→ Si	28.1 amu
	→ O	16.0 amu

- Directly scattered and sputtered particles can gain energy up to primary ion energy
- Energy depends strongly on scattering angle and mass ratio

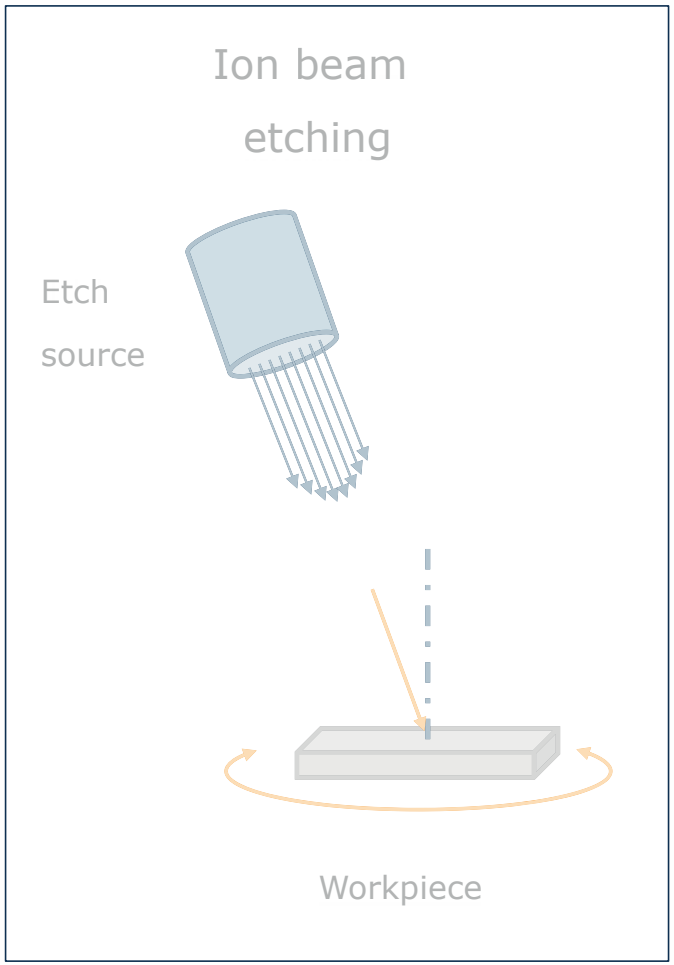
Ion Beam Techniques

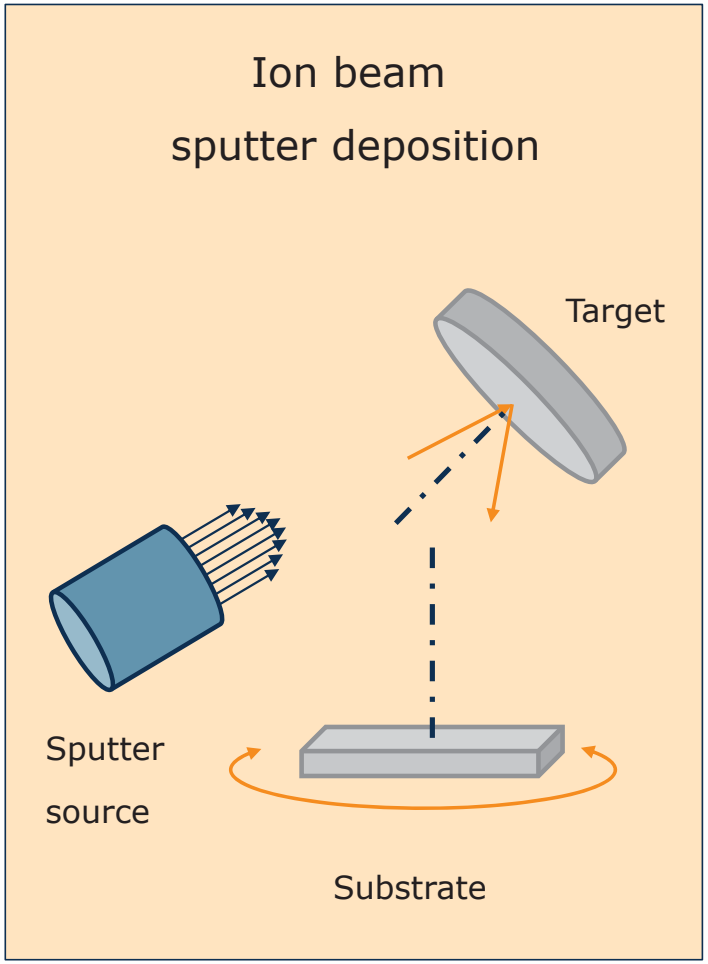


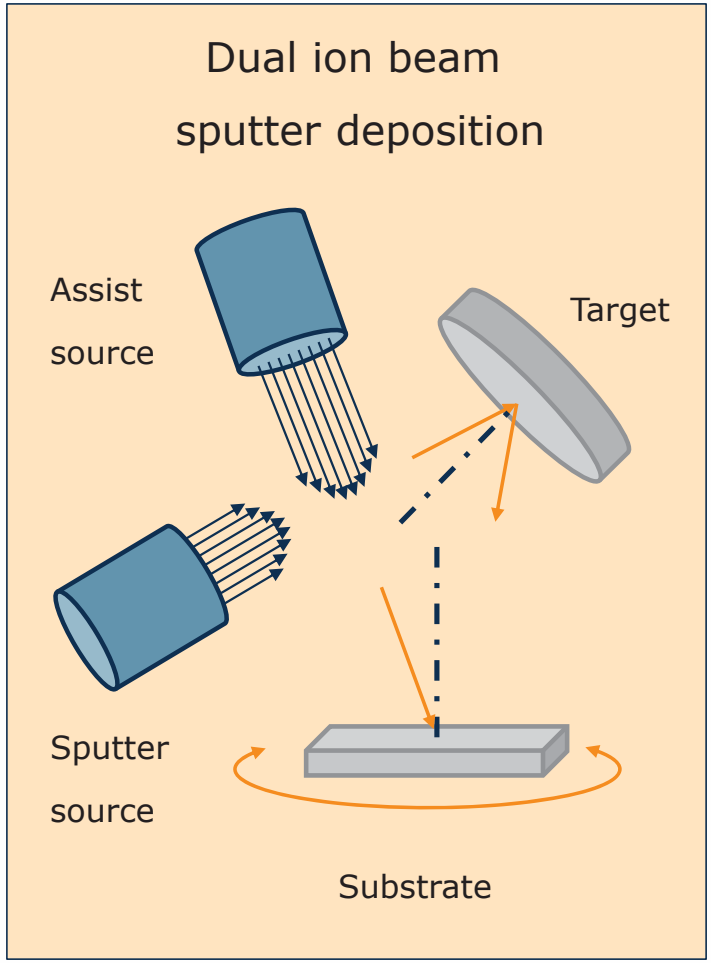




Ion Beam Techniques

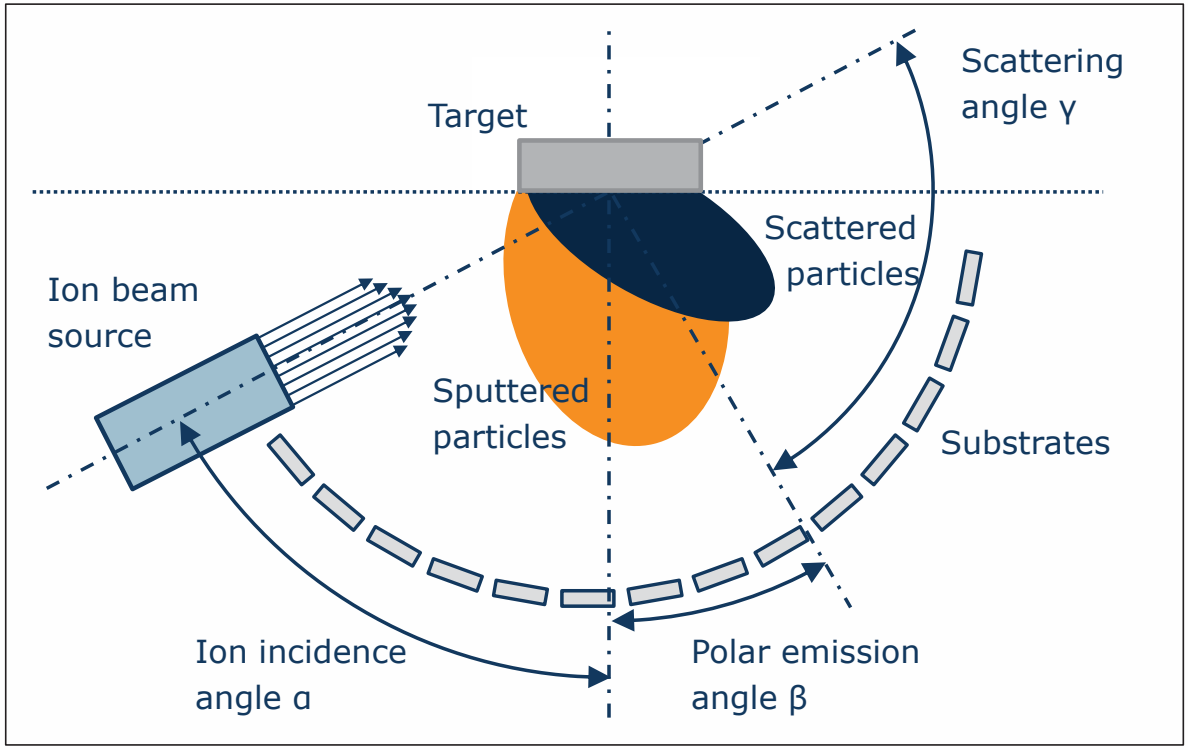






Experimental Setup and Process Parameters

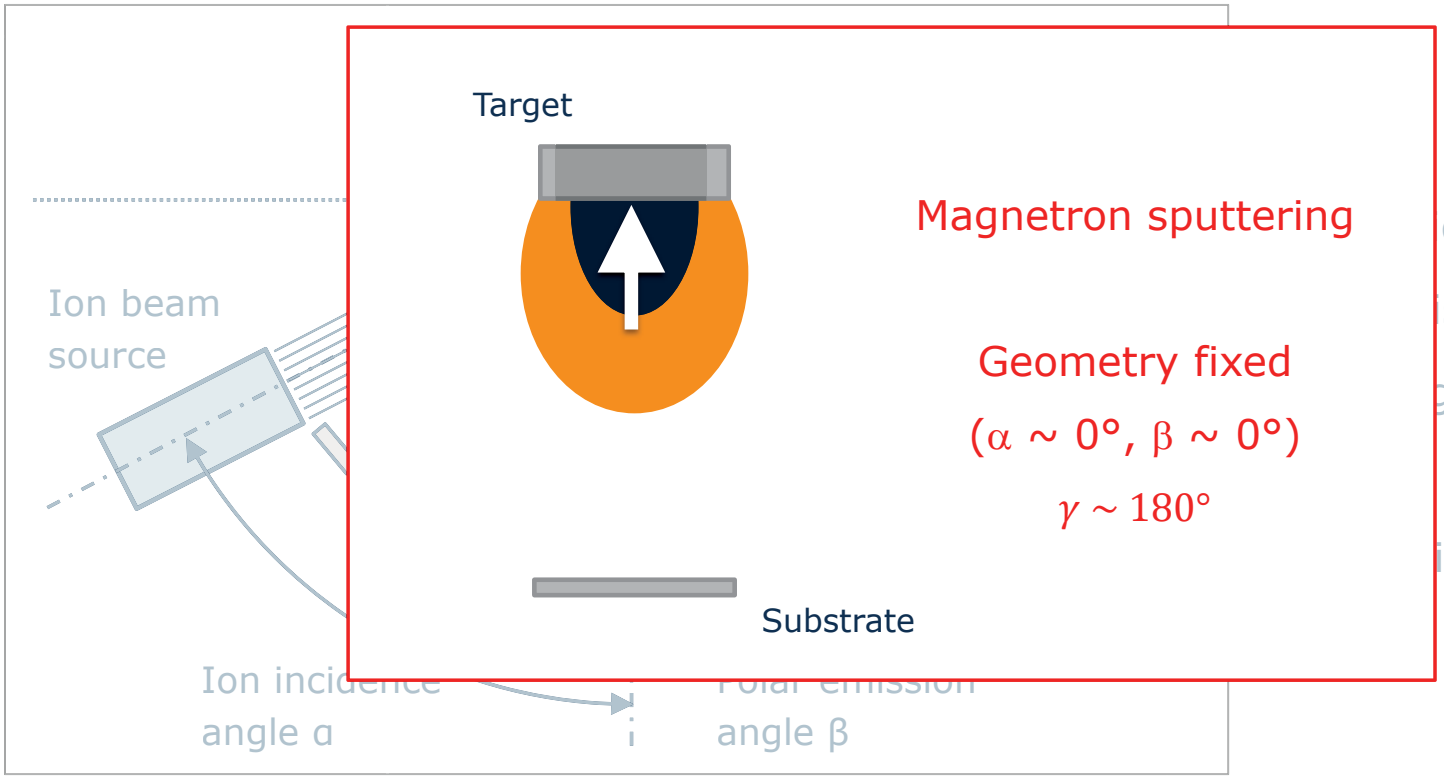
- Particle characterization and film deposition
- Systematic variation of ion beam and geometrical parameters



- 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)
- O_2-inlet

Experimental Setup and Process Parameters

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



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inlet	

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  - Fundamentals
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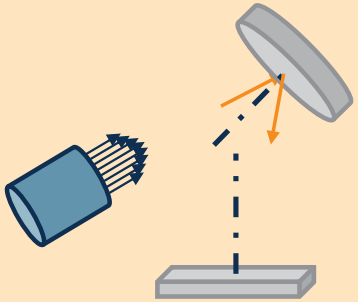
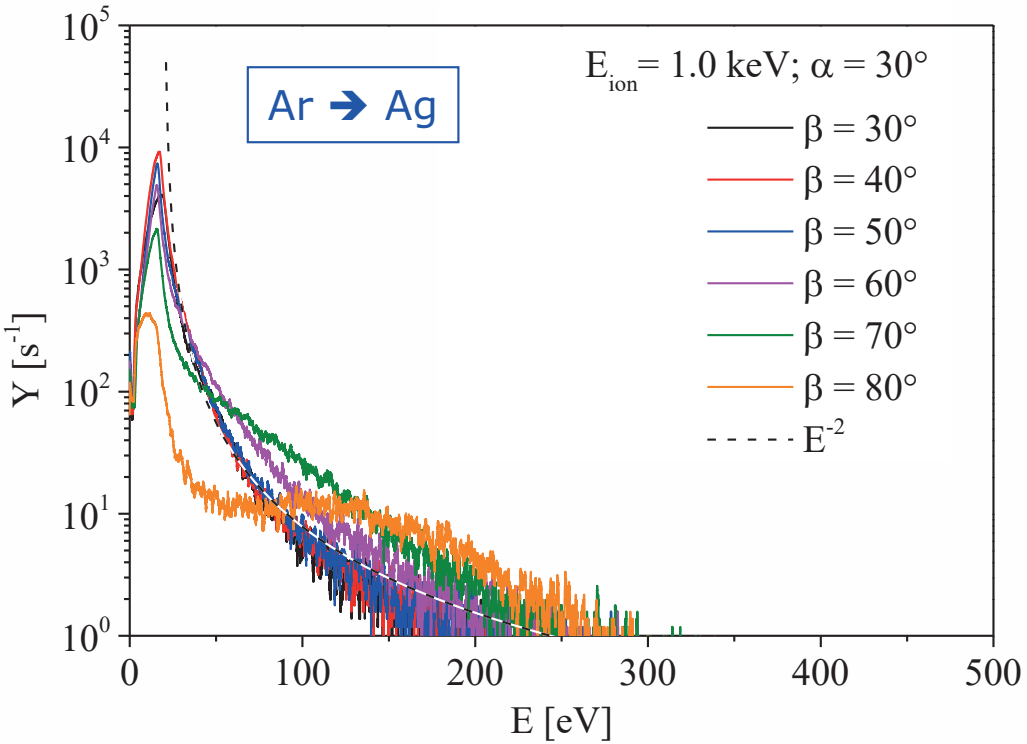
IBS

Particle Properties



Energy of sputtered ions



- β < 60°, i.e. γ > 90°:
  Follows Thompson-Model

$$\frac{dY}{dE} \propto \frac{E}{(E+U)^3}$$

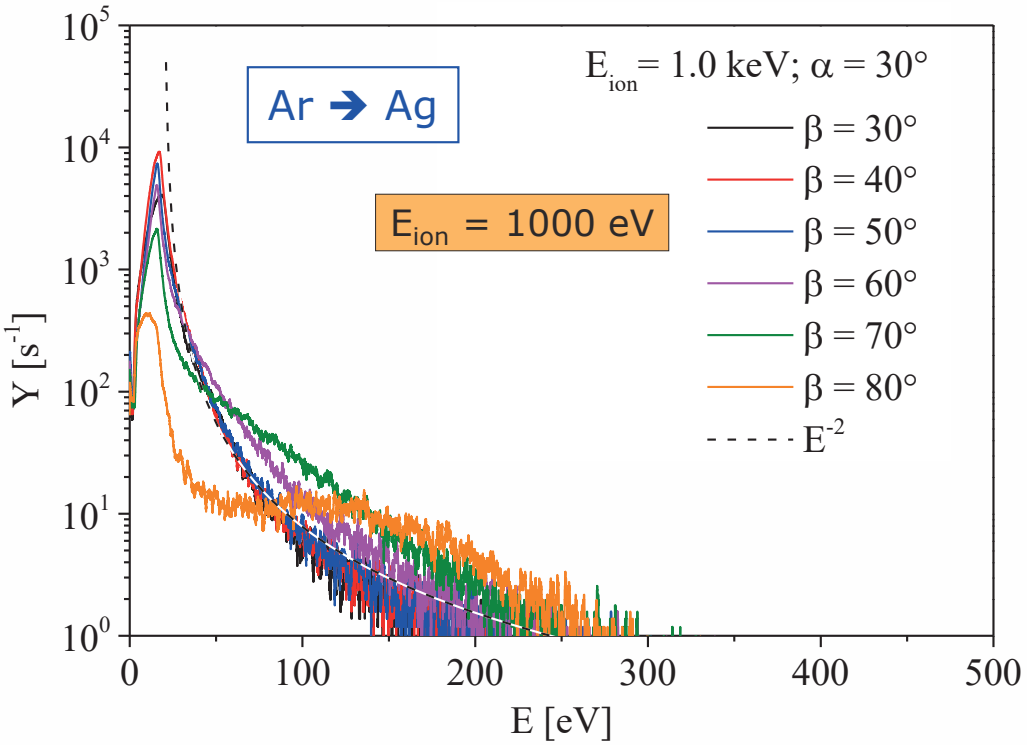
- β ≥ 60°, i.e. γ ≤ 90°:
  Contributions by directly sputtered particles

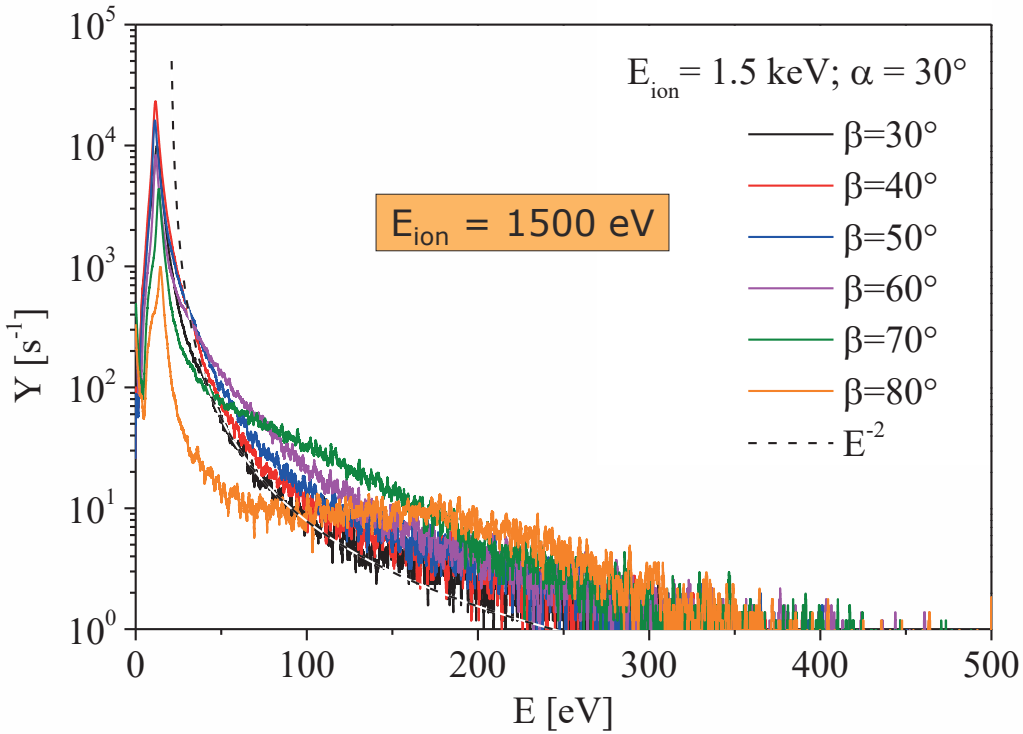
- Calculations (simple binary elastic collision):

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

β	Ag (Ar-Ag)
[°]	[eV]
60	-
70	24
80	92
90	197

Energy of sputtered ions: Ion energy

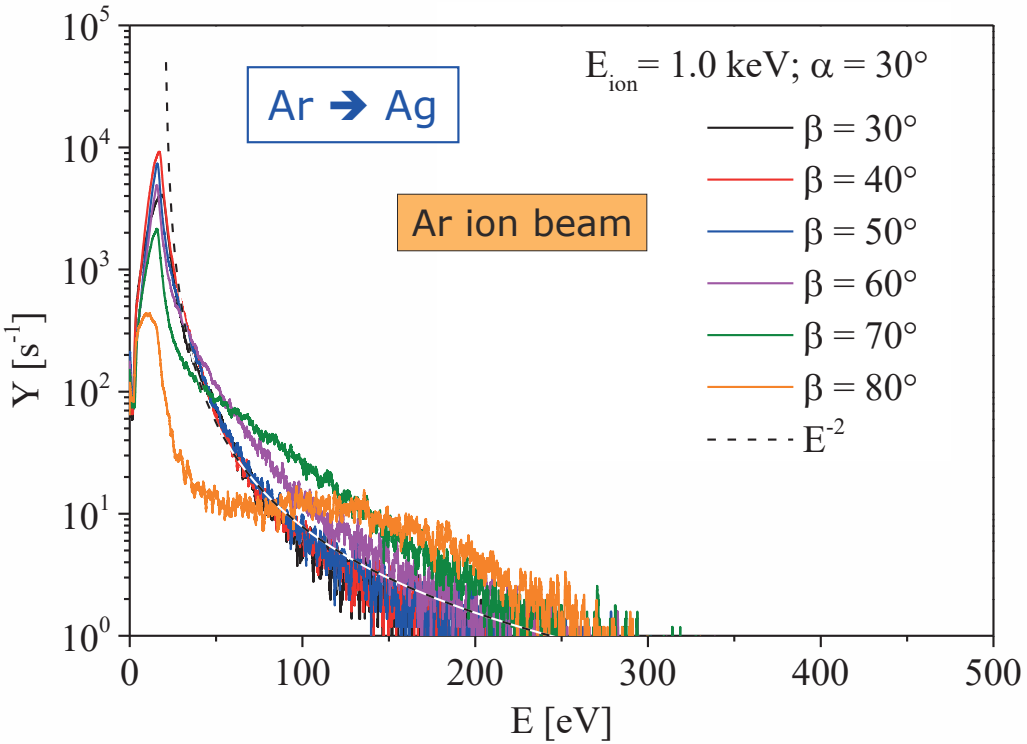


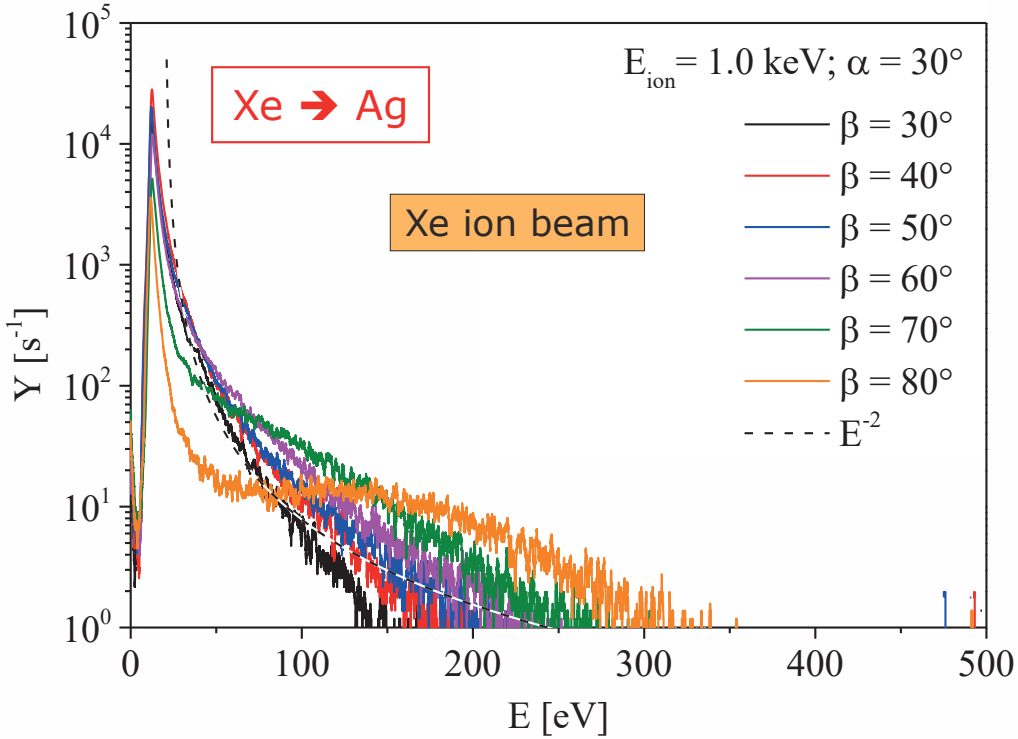


- Principal shape of curves stays the same (maximum, shoulder)
- Additional features shifted to higher energies (curves are "stretched")

R. Feder, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 316 (2013) 198.
C. Bundesmann, et al., Contrib. Plasma Phys. 55 (2015) 737.

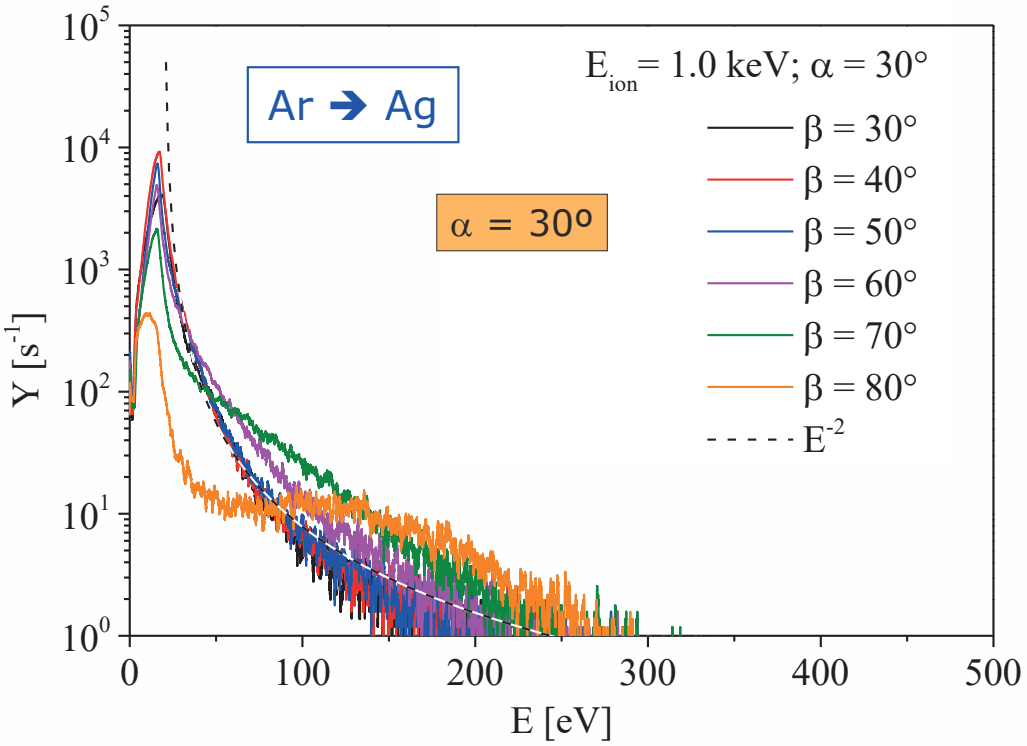
Energy of sputtered ions: Ion species

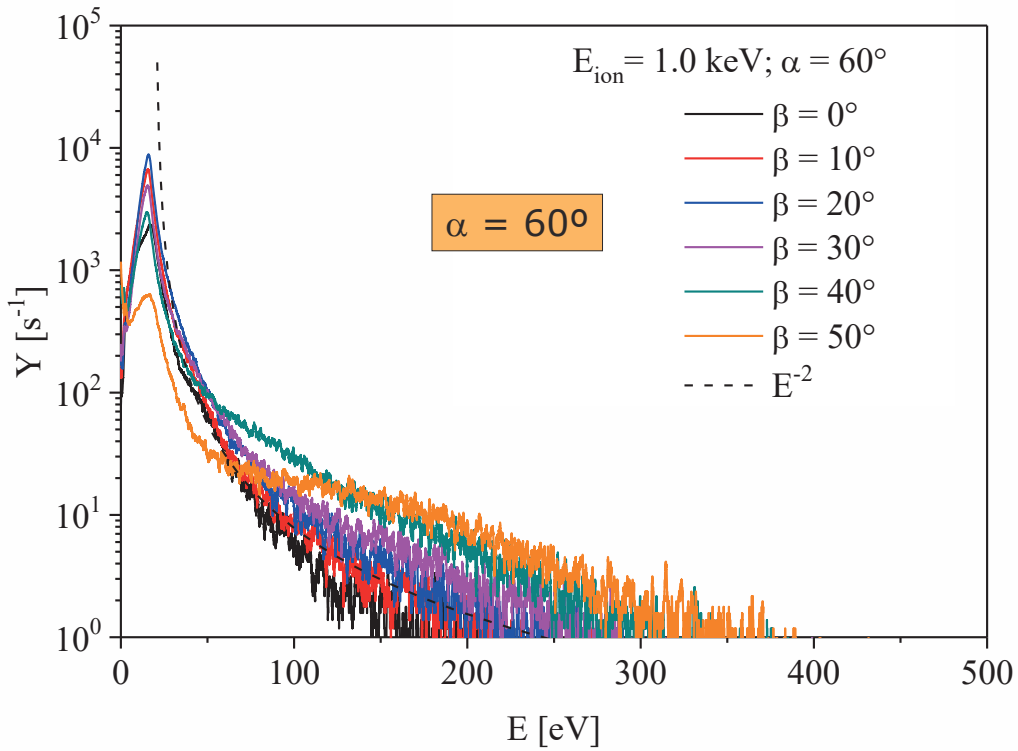




- Shape of energy distribution similar for both ion species
- Slightly higher energies for sputtering with Xe

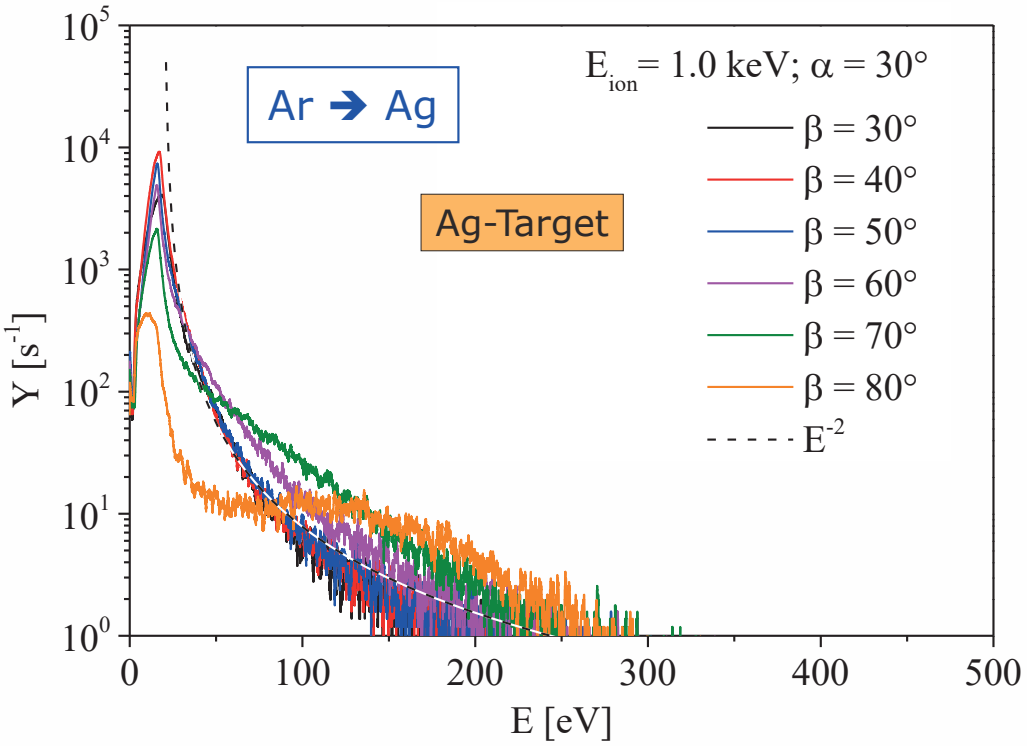
Energy of sputtered ions: Ion incidence angle

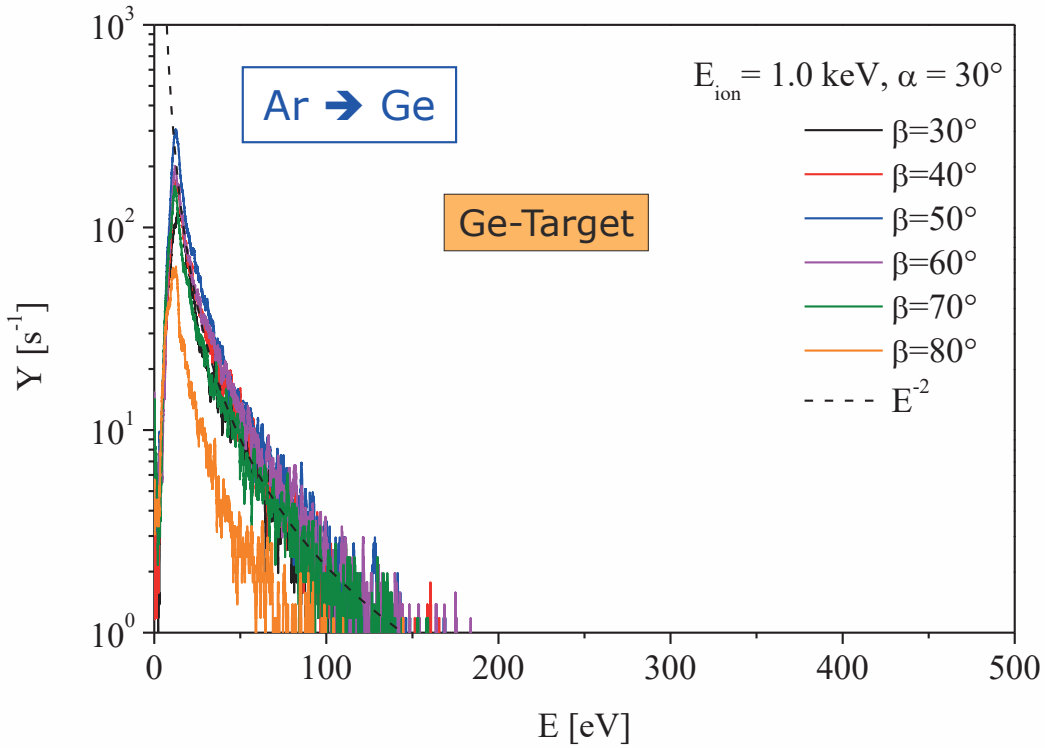




- Shape of curves the same when comparing curves with the same γ;
  e.g. α / β = 30° /80° and 60° /50°

Energy of sputtered ions: Target material

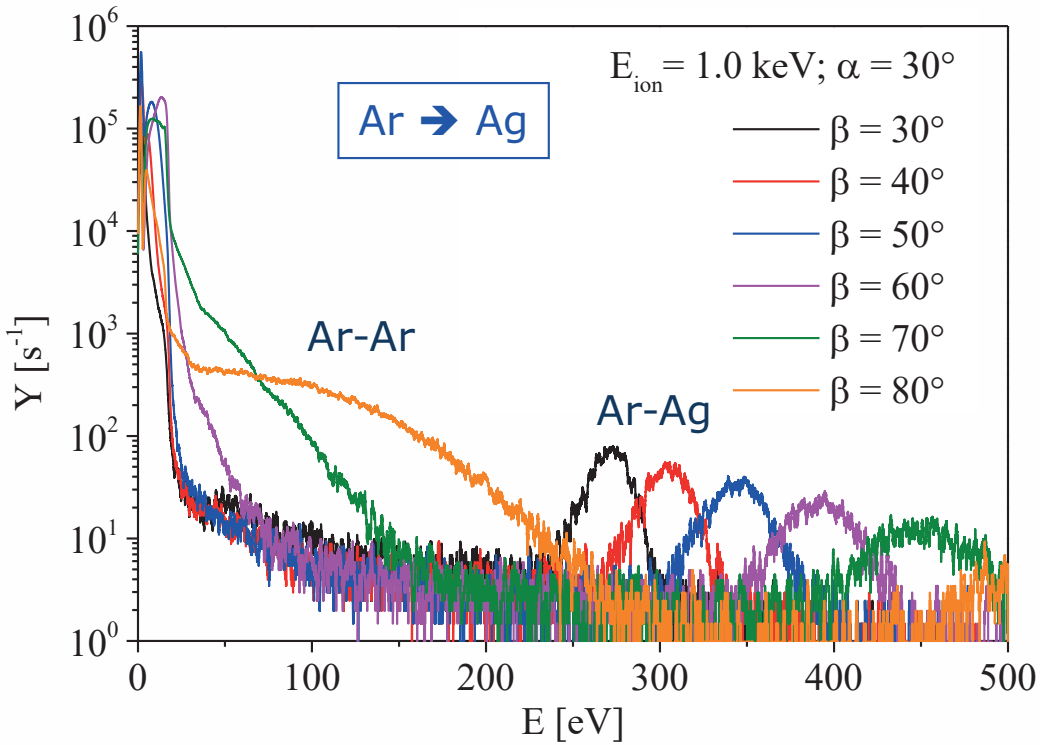




- Ge shows no (or less) contributions due to direct sputtering events; appears only for smaller γ

R. Feder, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 316 (2013) 198.
R. Feder, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 334 (2014) 88.

Energy of scattered ions



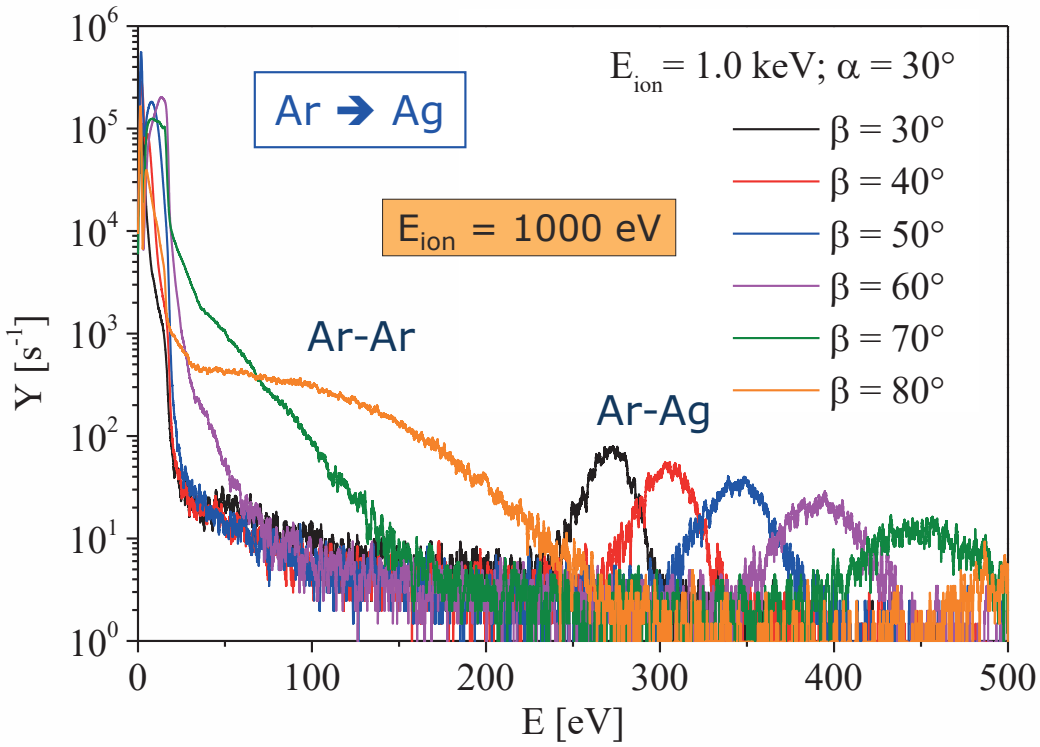
- Maxima at low energies (sputtering or charge exchange)
- Shoulder (E < 250 eV): Ar-Ar scattering
- Peaks (E > 250 eV): Ar-Ag scattering
- Shift to higher energies with increasing angle

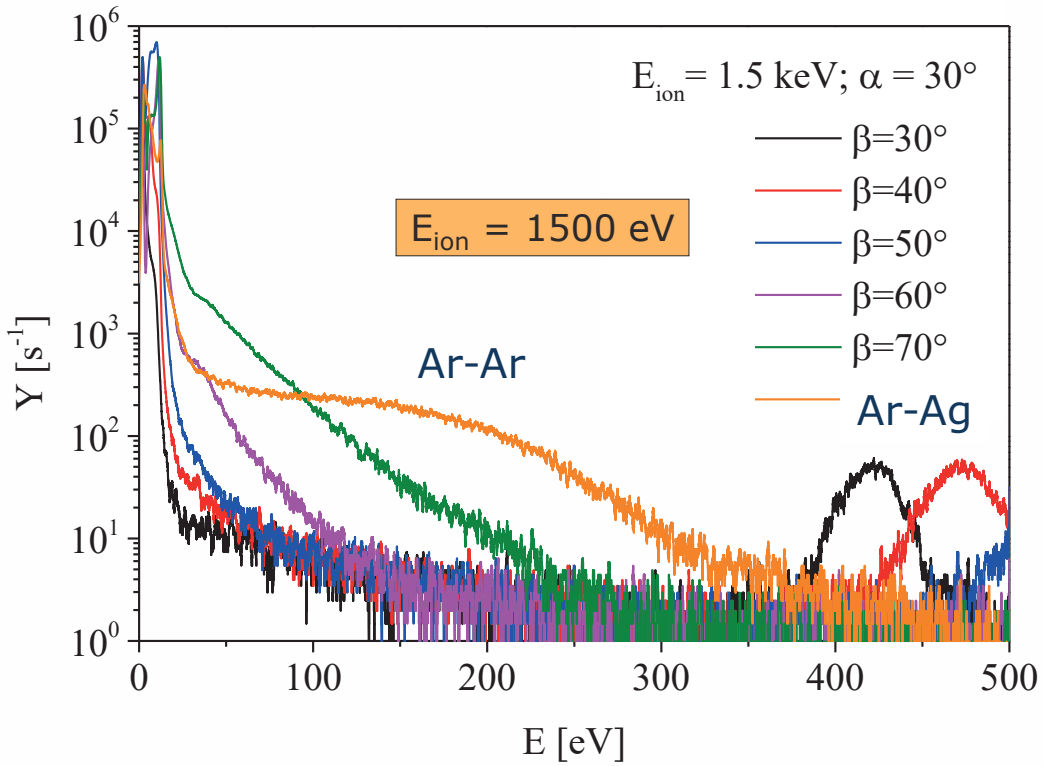
β	Ar (Ar-Ar)	Ar (Ar-Ag)
[°]	[eV]	[eV]
30	-	309
40	-	350
50	-	400
60	-	459
70	30	528
80	117	603

- Calculations (simple binary elastic collision):

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

Energy of scattered ions: Ion energy





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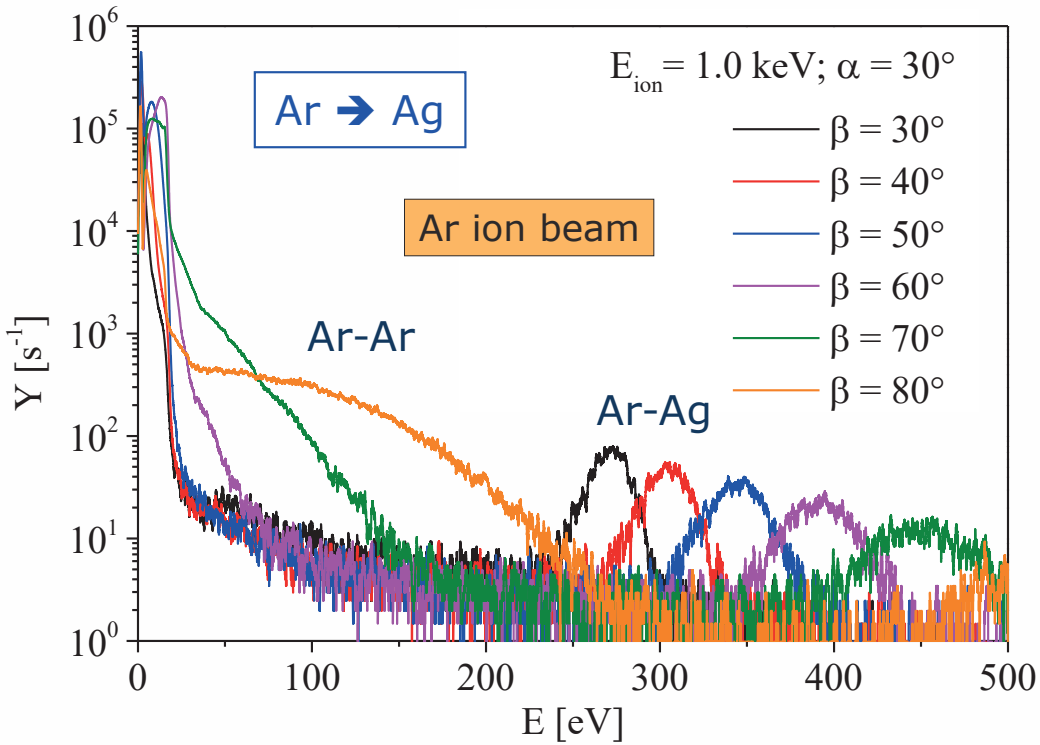
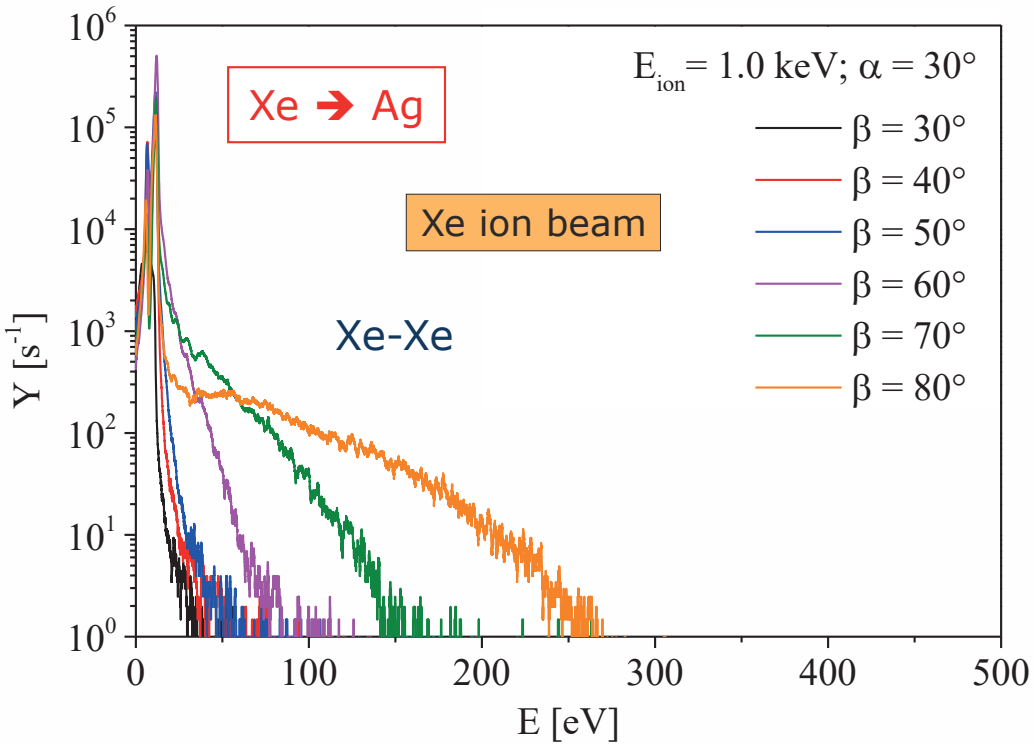
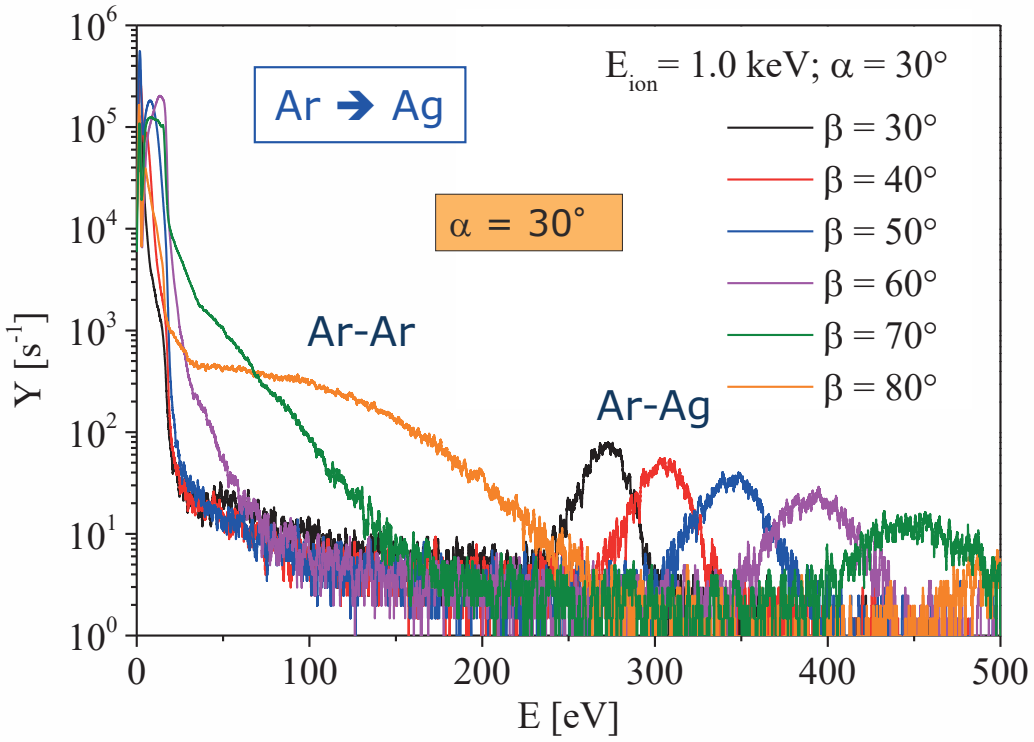
Energy of scattered ions: Ion species

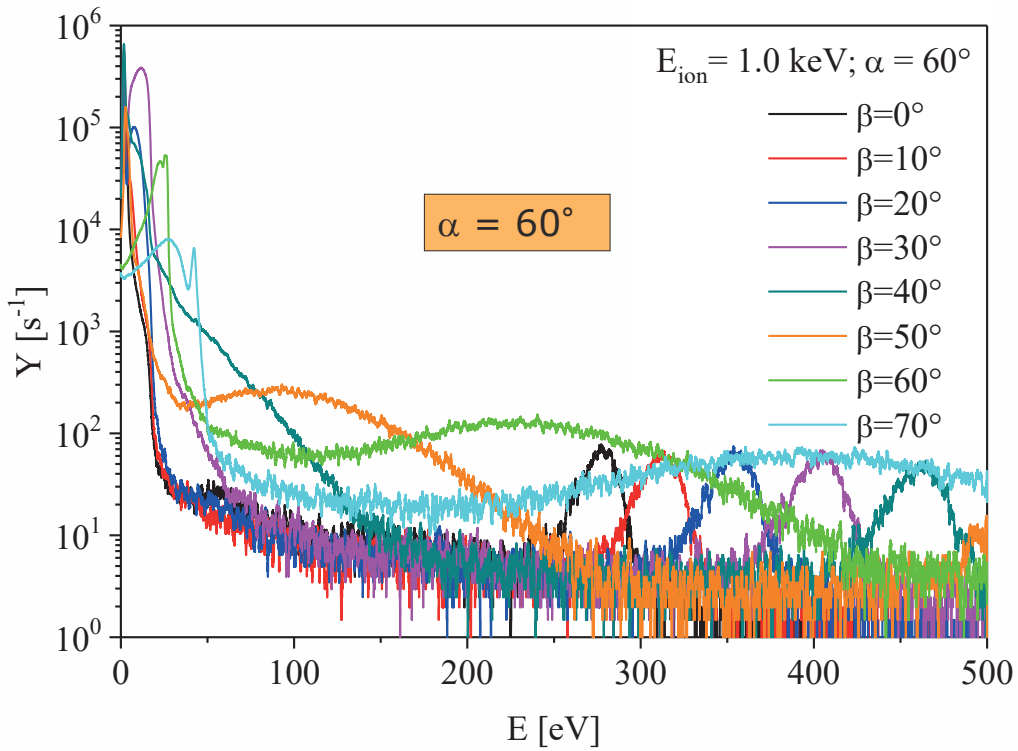




- Shoulder due to Xe-Xe scattering the same as for Ar-Ar scattering
- Maxima due to Xe-Ag scattering is missing; appears only if $\gamma < 125°$

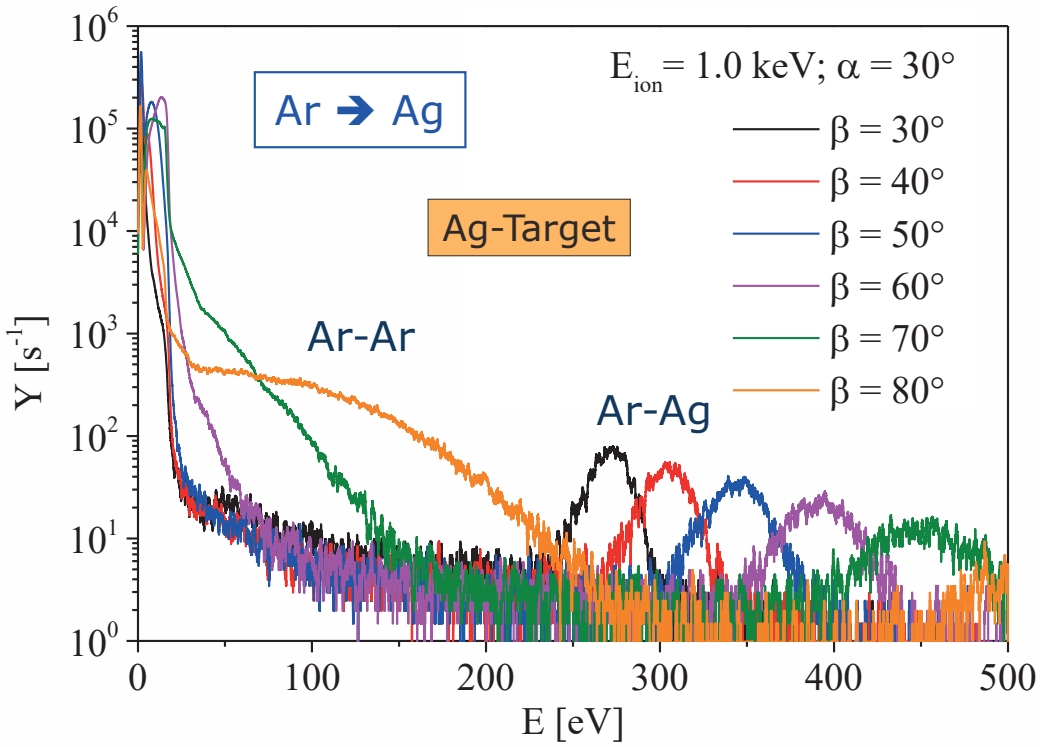
Energy of scattered ions: Ion incidence angle

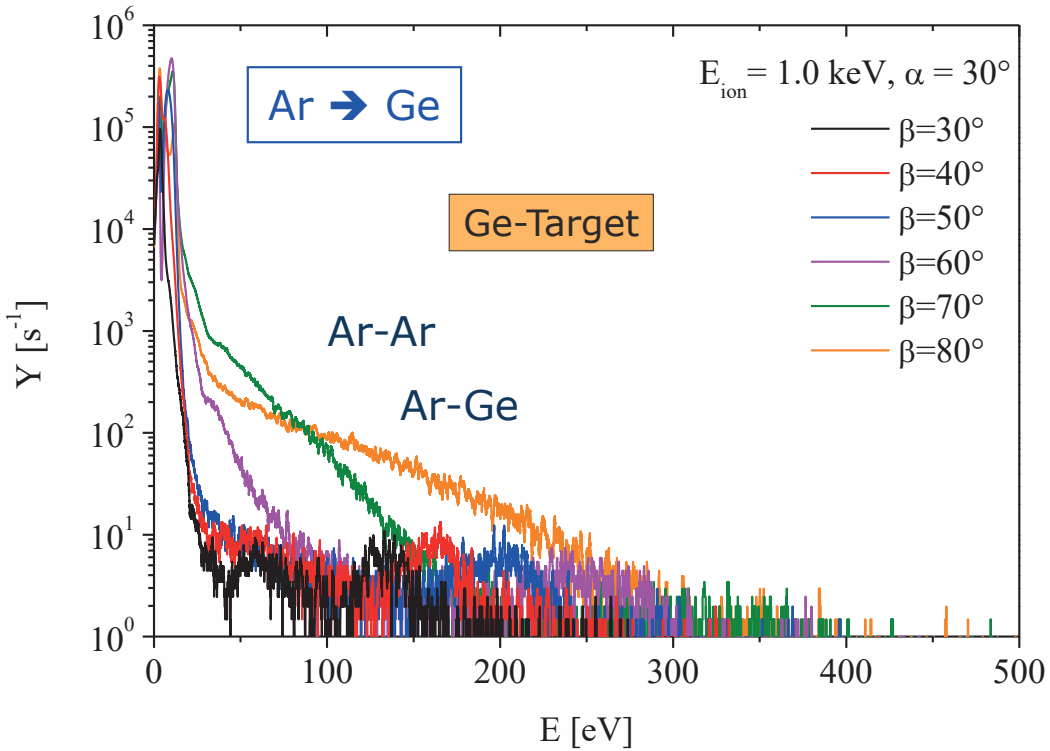




- Principal shape of curves stays the same when comparing curves with the same γ; e.g. α / β = 30º/70º and 60º/40º

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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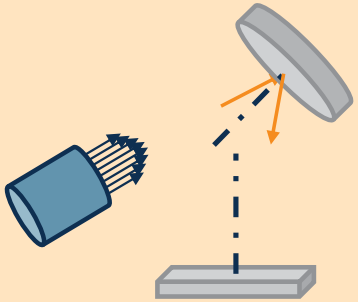
  - Leibniz institute of Surface Engineering
  - Motivation
  - Fundamentals
- Particle properties
  - Sputtered target particles
  - Scattered primary particle
- Film properties
  - Ion beam sputter deposition of Ag and TiO_2
  - Dual ion beam sputter deposition of SiO_2 and TiO_2
- Summary and outlook

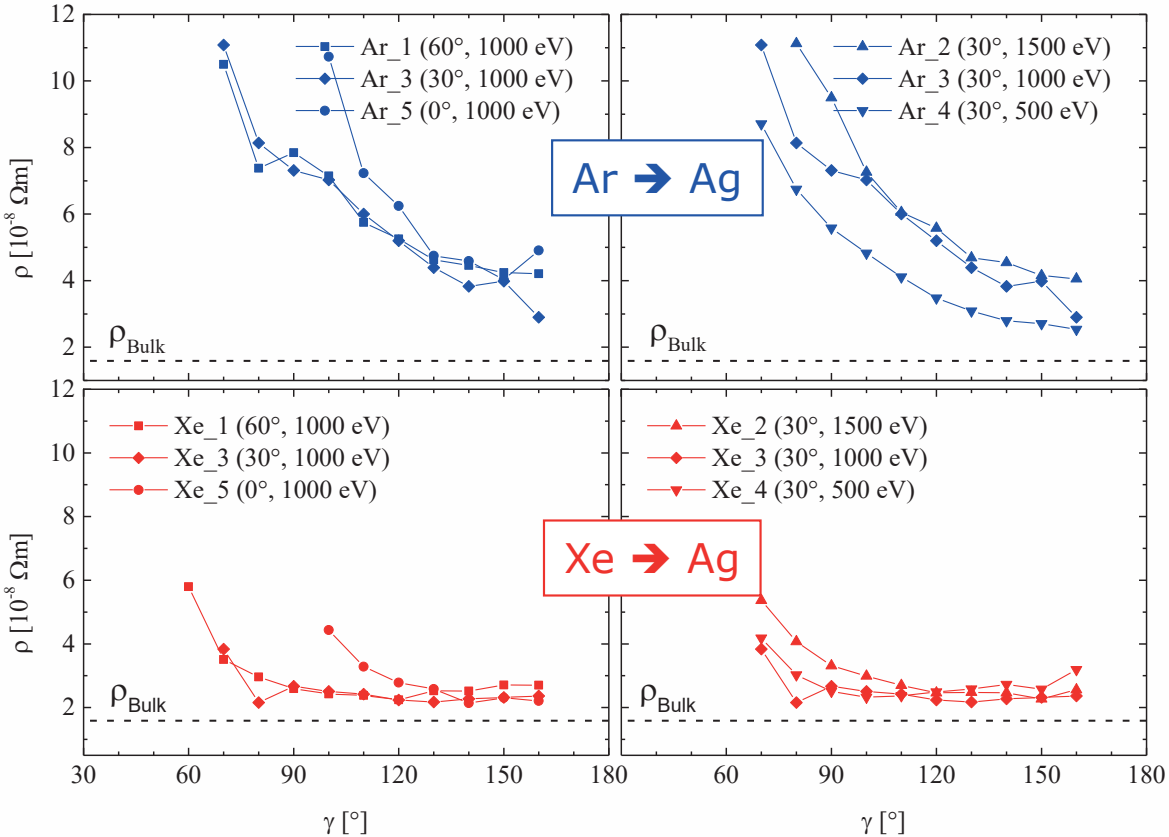
IBSD

Thin Film Properties



Ag films: Electrical resistivity (4PPM)

Ion incidence angle varied | Ion energy varied

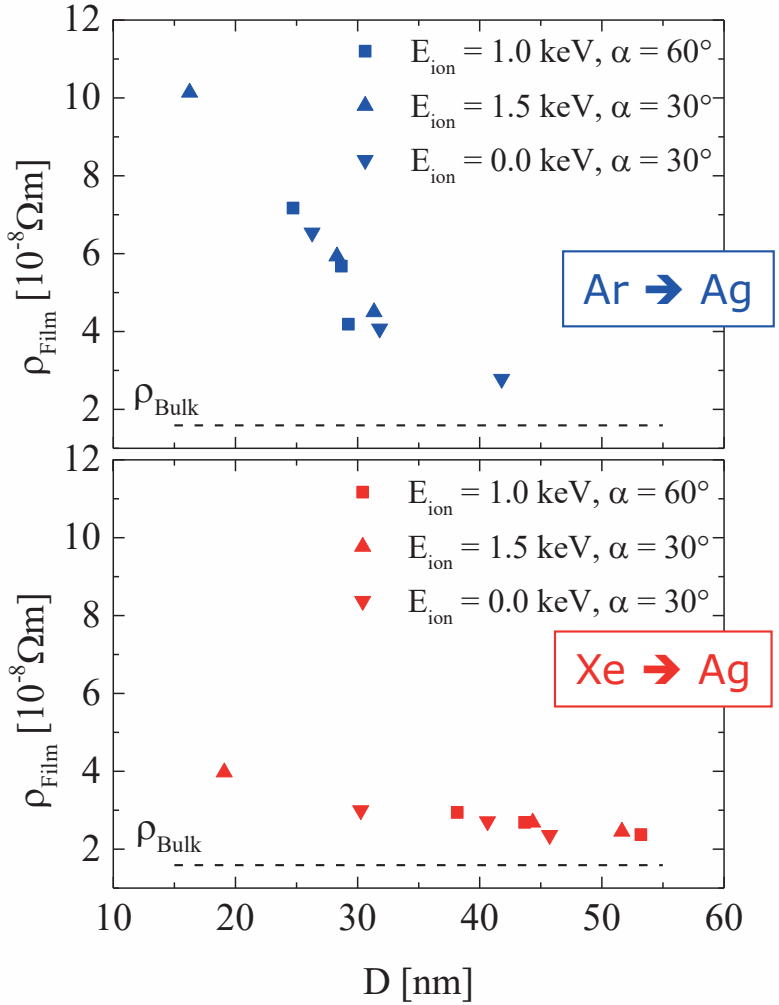


Electrical resistivity

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

C. Bundesmann, et al., Thin Solid Films 551 (2014) 46.

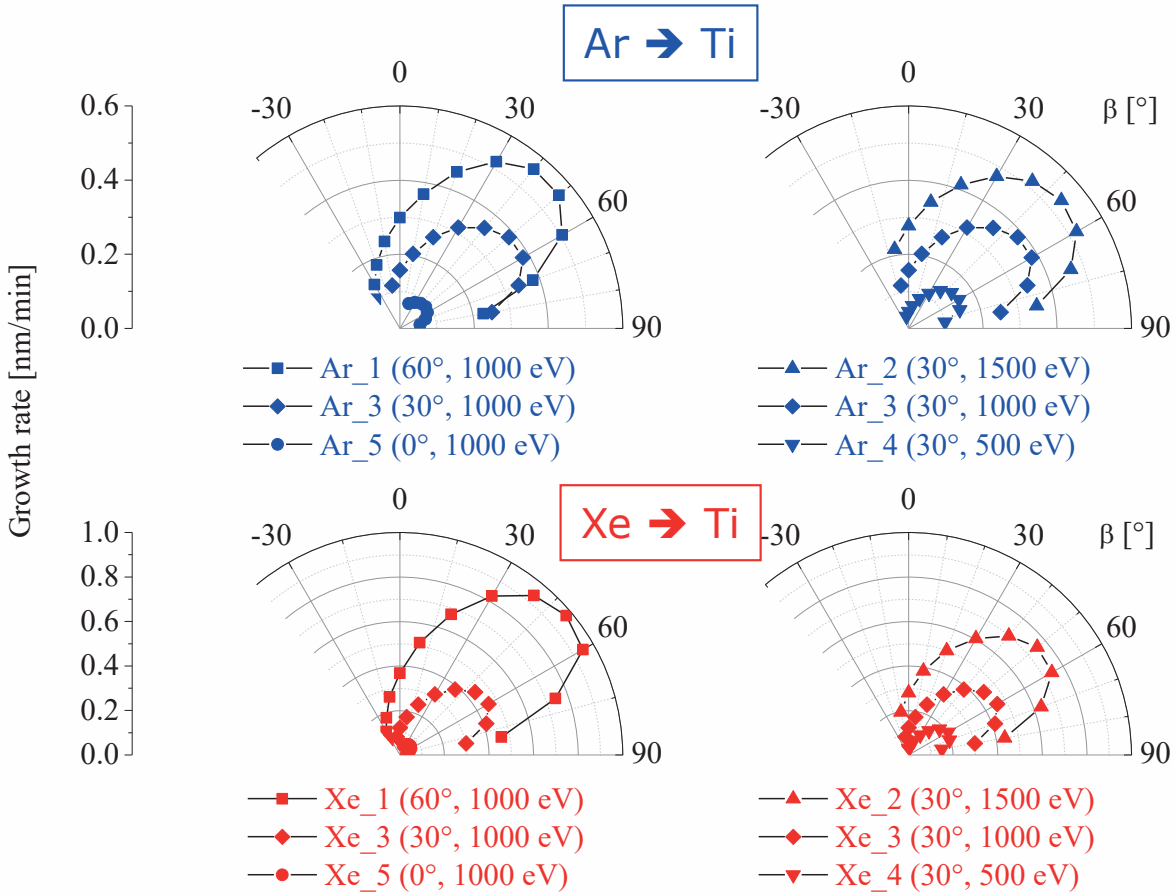
Ag films: Electrical resistivity vs. crystallinity (XRD)



- 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)

C. Bundesmann, et al., Thin Solid Films 551 (2014) 46.

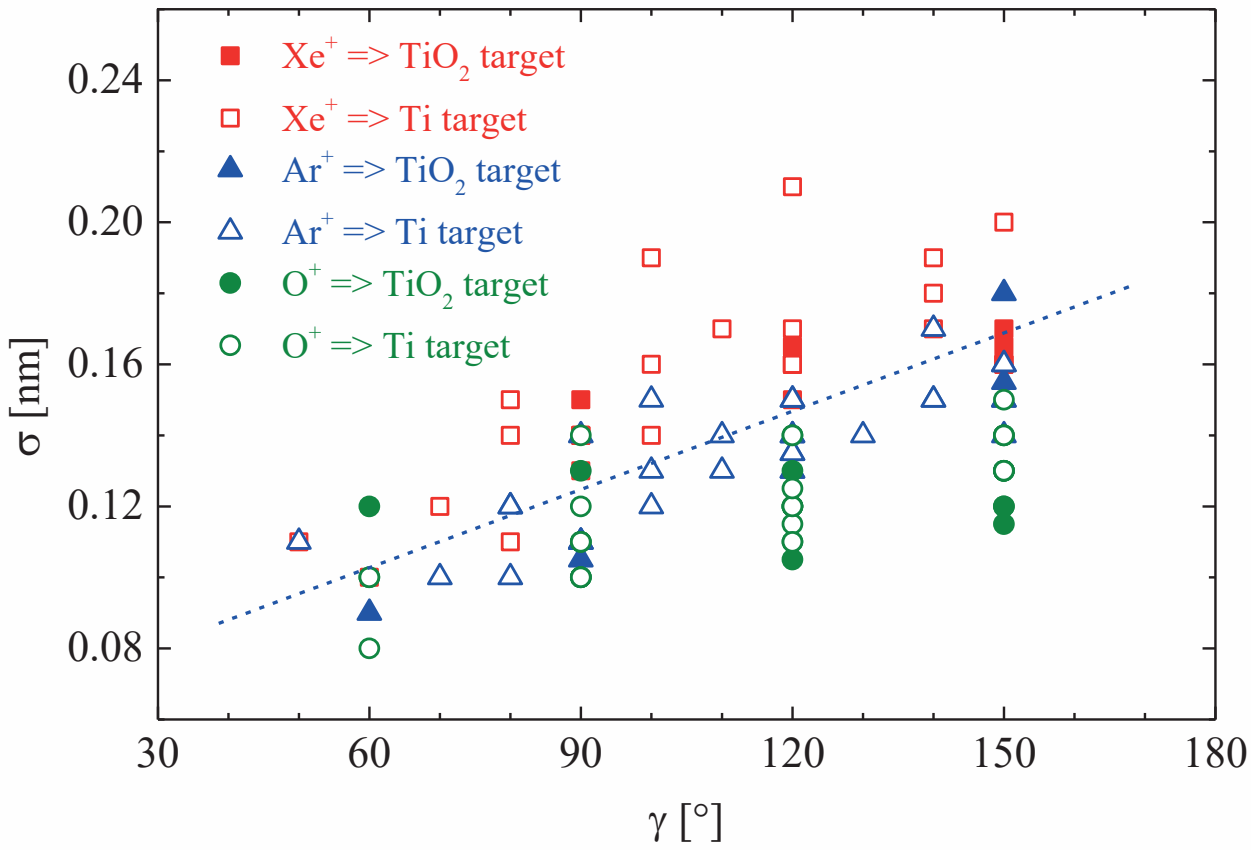
TiO_2 films: Growth rate



- 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

C. Bundesmann, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 395 (2017) 17.

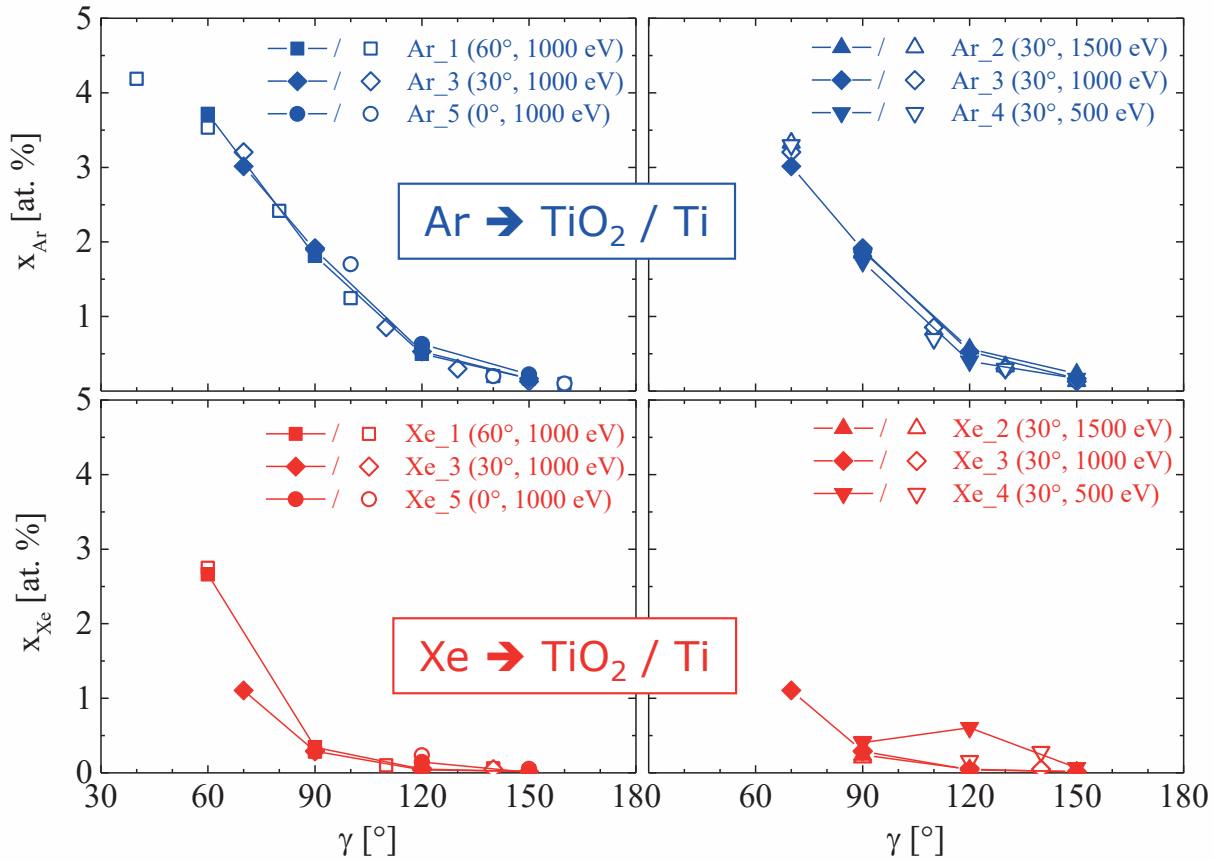
TiO_2 films: Surface roughness (AFM)



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

C. Bundesmann, et al., Appl. Surf. Sci. 421 (2017) 331.
C. Bundesmann, et al., Eur. Phys. J. B 90 (2017) 187.
L. Pietzonka, et al., in preparation.

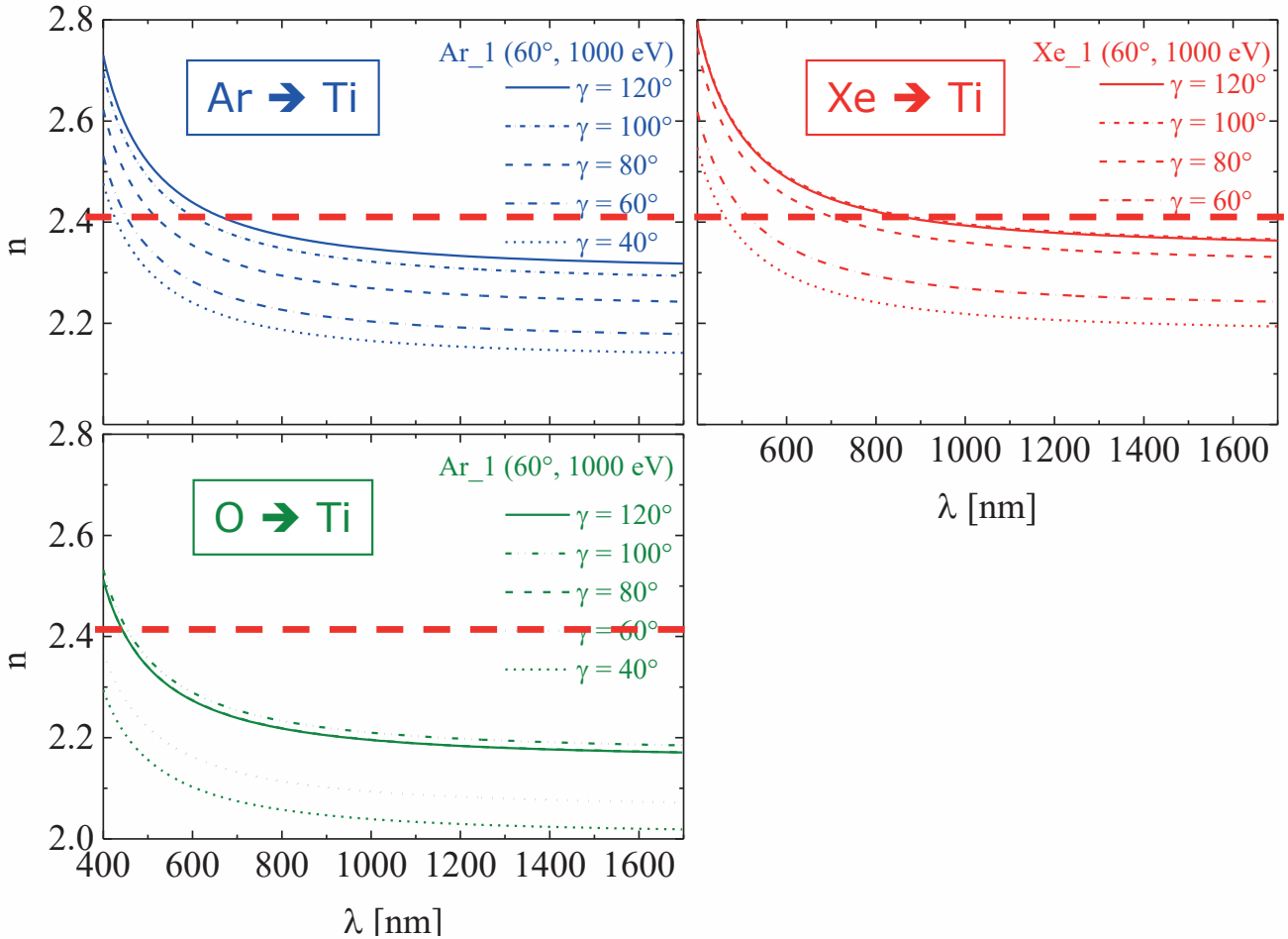
TiO_2 films: Composition (RBS)



- 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)

C. Bundesmann, et al., Appl. Surf. Sci. 421 (2017) 331.
C. Bundesmann, et al., Eur. Phys. J. B 90 (2017) 187.

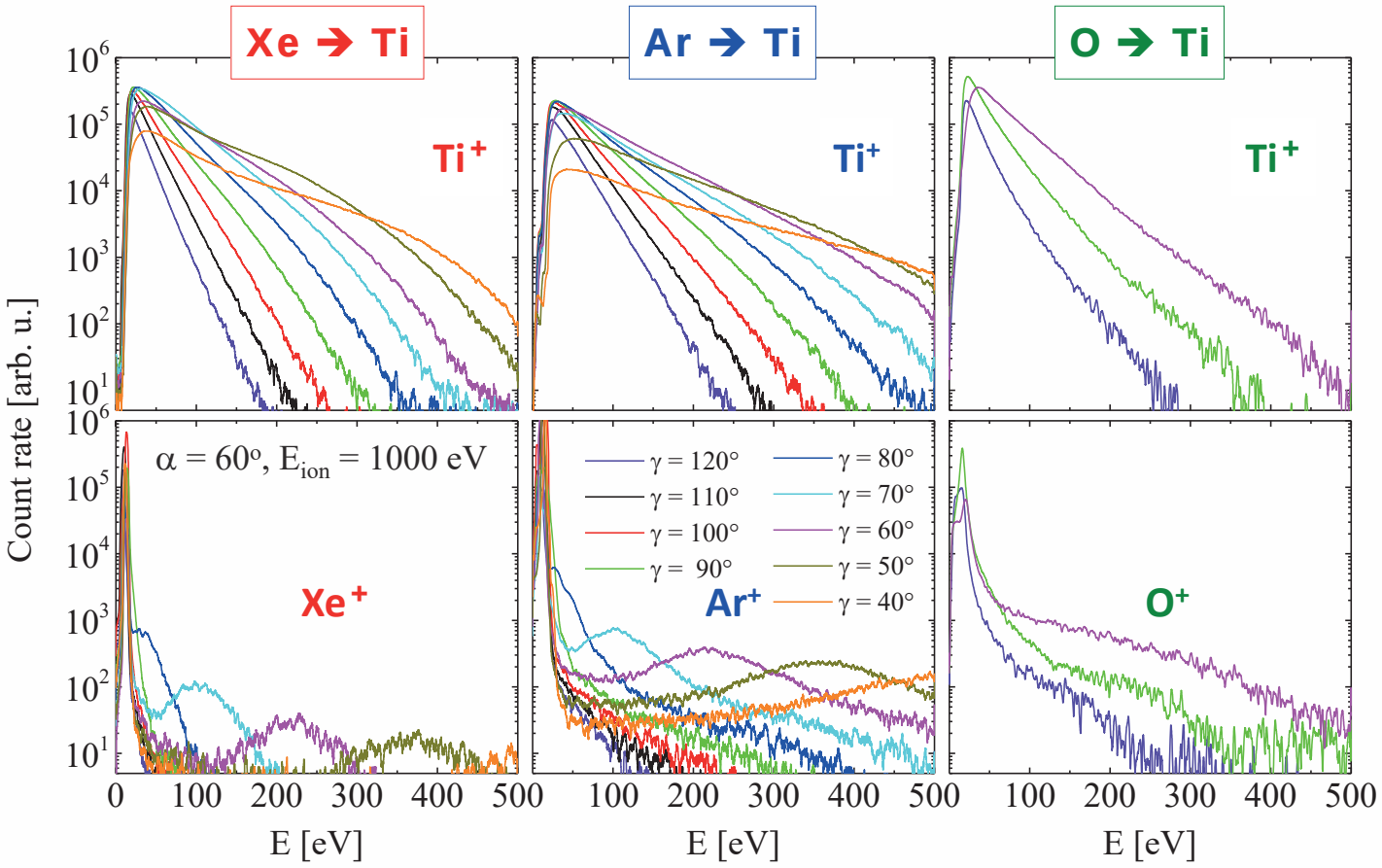
TiO_2 films: Optical properties (SE)



- 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: $n(O) \leq n(Ar) \leq n(Xe)$

Energy distribution of secondary particles

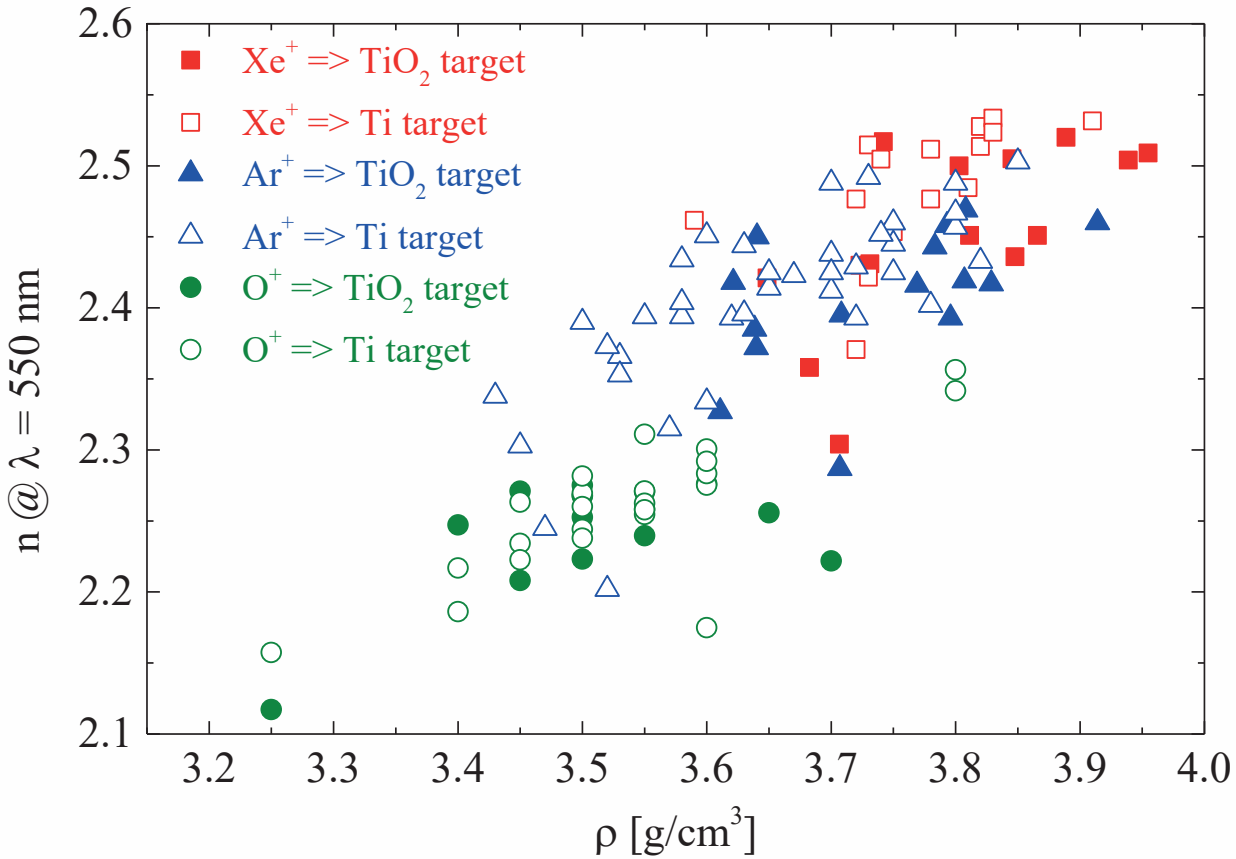


- Additional high-energy contributions due to direct sputtering or scattering events
- Maximum particle energy can gain several 100 eV
- Energy (and signal intensity) depends on ion energy, scattering angle, ion species

$$\gamma = 180° - \alpha - \beta$$

T. Lautenschläger and C. Bundesmann , J. Vac. Sci. Technol. A 35 (2017) 041001.
T. Lautenschläger, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 385 (2016) 30.

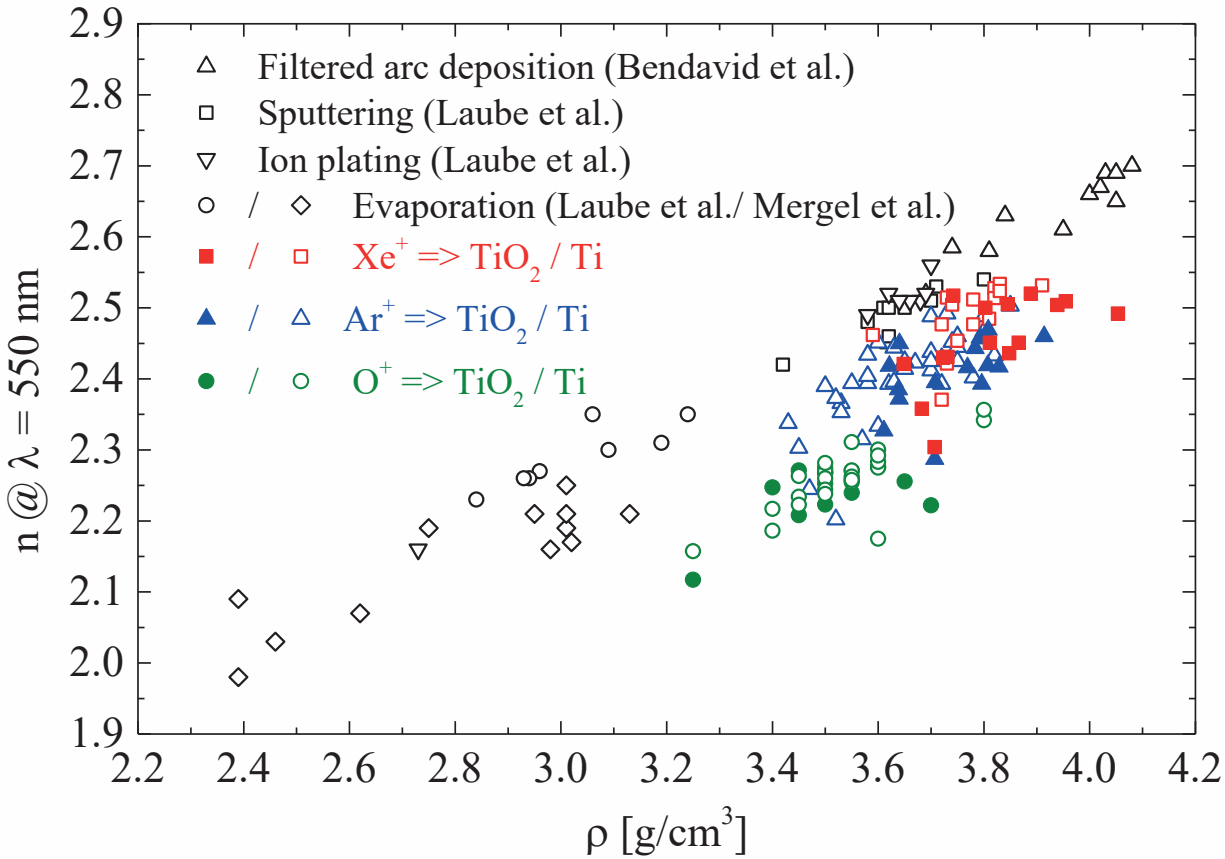
TiO_2 films: Optical properties vs. mass density



- Index of refraction *n* and mass density *ρ* show strong correlation

C. Bundesmann, et al., Appl. Surf. Sci. 421 (2017) 331.
C. Bundesmann, et al., Eur. Phys. J. B 90 (2017) 187.
L. Pietzonka, et al., in preparation.

TiO_2 films: Optical properties vs. mass density



- Index of refraction *n* and mass density *ρ* show strong correlation
- Systematics fits to data from other PVD techniques (related to different particle energy)

A. Bendavid, P.J. Martin, H. Takikawa, Thin Solid Films 360 (2000) 241.
M. Laube, et al., Nucl. Instrum. Methods Phys. Res., Sect. B, 113 (1996) 288.
D. Mergel, et al., Thin Solid Films 371 (2000) 218.

Outline

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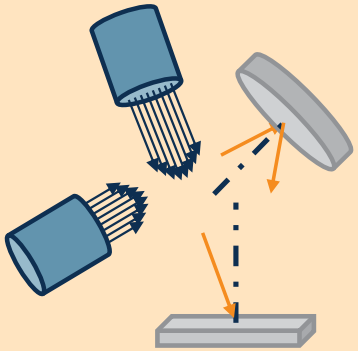
DIBSD

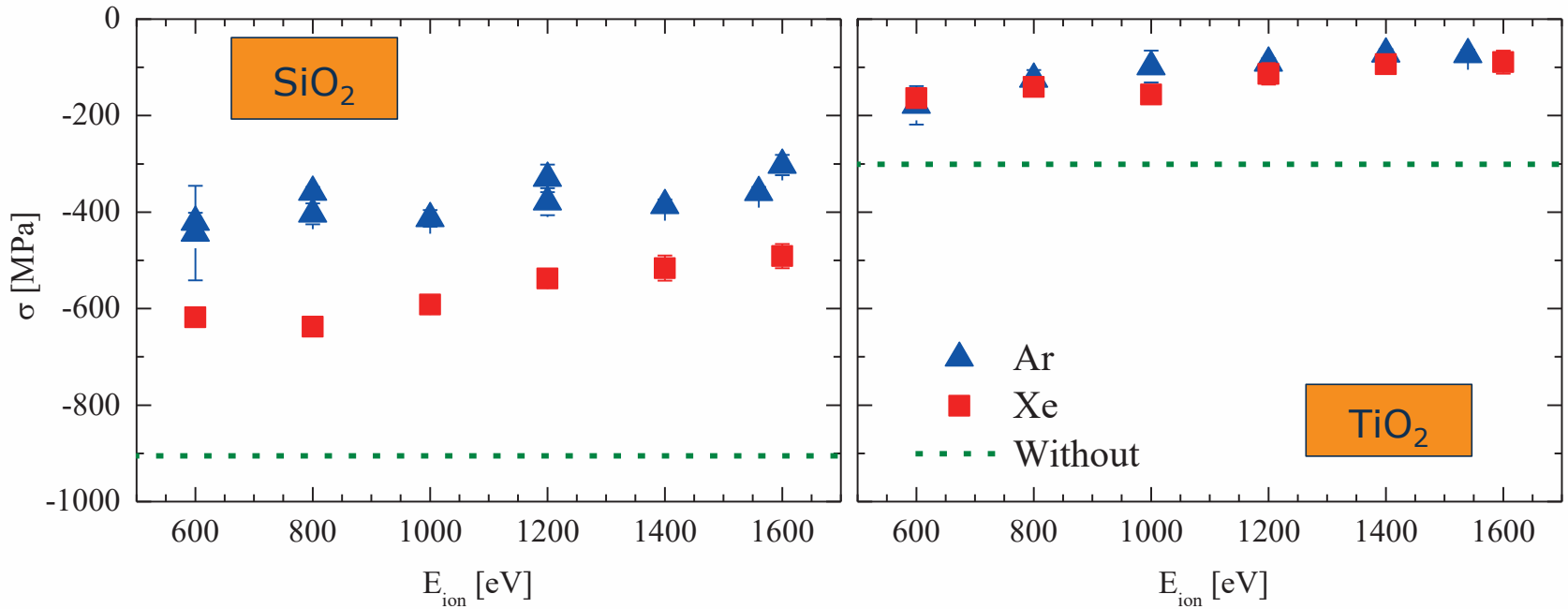
Thin Film Properties



DIBSD: Stress engineering of SiO_2 and TiO_2

- Reducing intrinsic stress by assisting ion bombardment

Film stress vs. energy of assisting ions



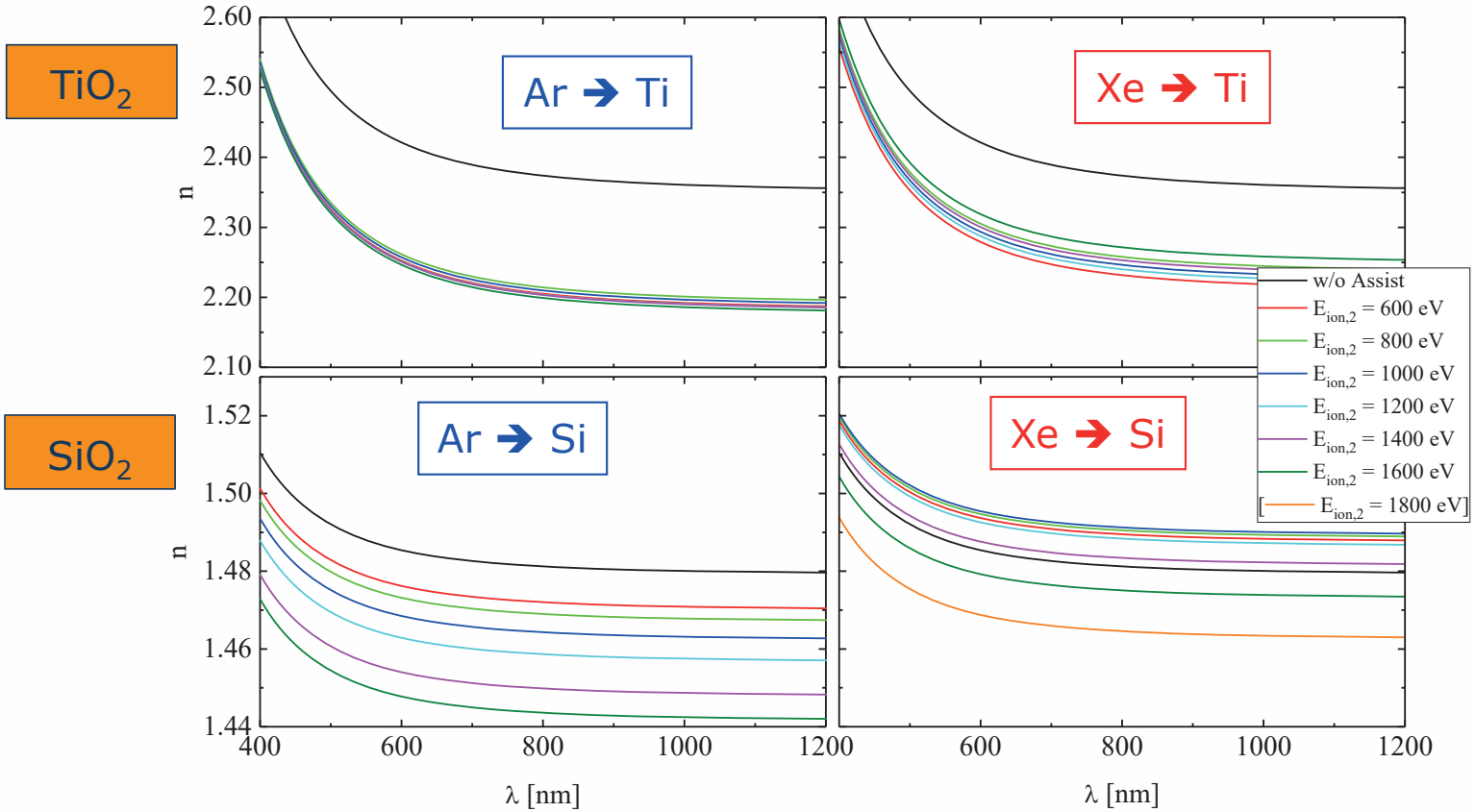
σ < 0

compressive stress

C. Bundesmann, et al., Thin Solid Films 516 (2008) 8604.

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

C. Bundesmann, et al., Thin Solid Films 516 (2008) 8604.

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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