Chemical Phase Formation in Material Mixing

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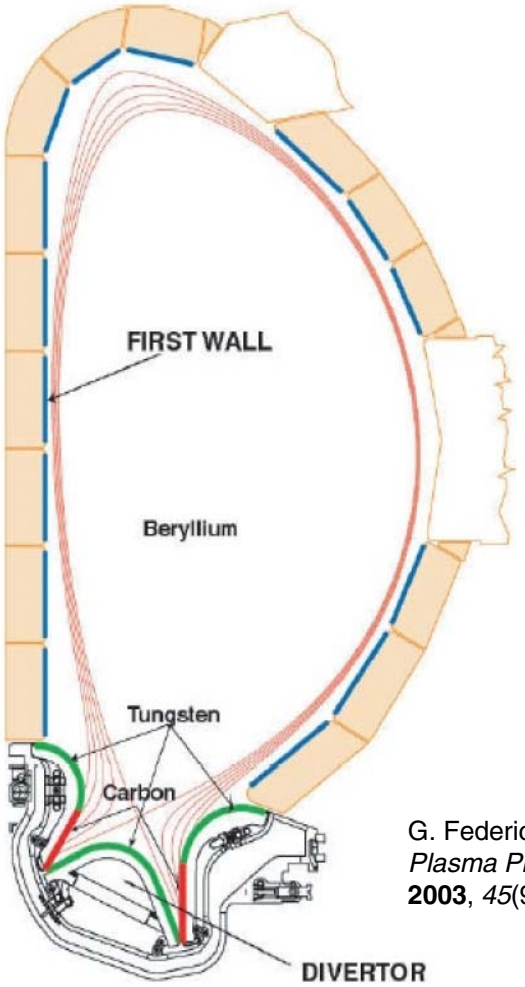
Interaction of energetic oxygen ions with Be_2W

Investigations with synchrotron photoelectron spectroscopy

Motivation I

- Beryllium, carbon and tungsten are proposed as first wall materials for ITER
- Oxygen from plasma impurities and from surface oxides after air exposure (e.g. maintenance)
- Formation of “mixed materials” due to:
  - Erosion
  - Transport
  - Redeposition



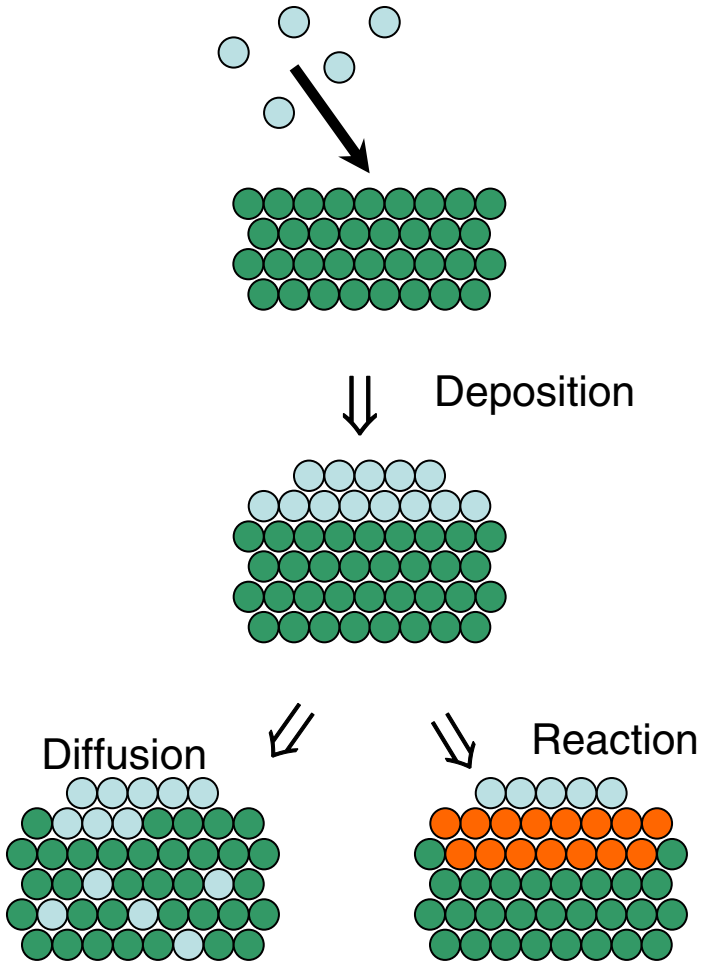
G. Federici et al.,
Plasma Phys. Control. Fusion.,
2003, *45*(9), 1523 - 1547

Motivation II

⇒Mixed Materials have new properties:

- Physical (e.g. melting point, thermal conductivity)
- Hydrogen retention
- Erosion

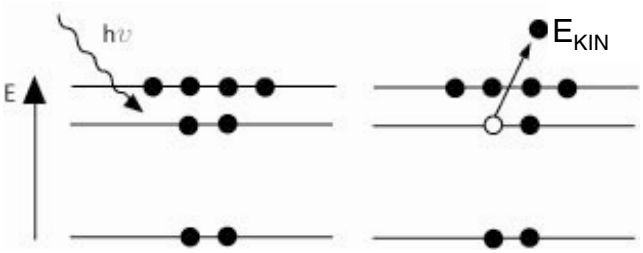
- Fundamental research of solid state reactions
  - Phase formations
  - Diffusion

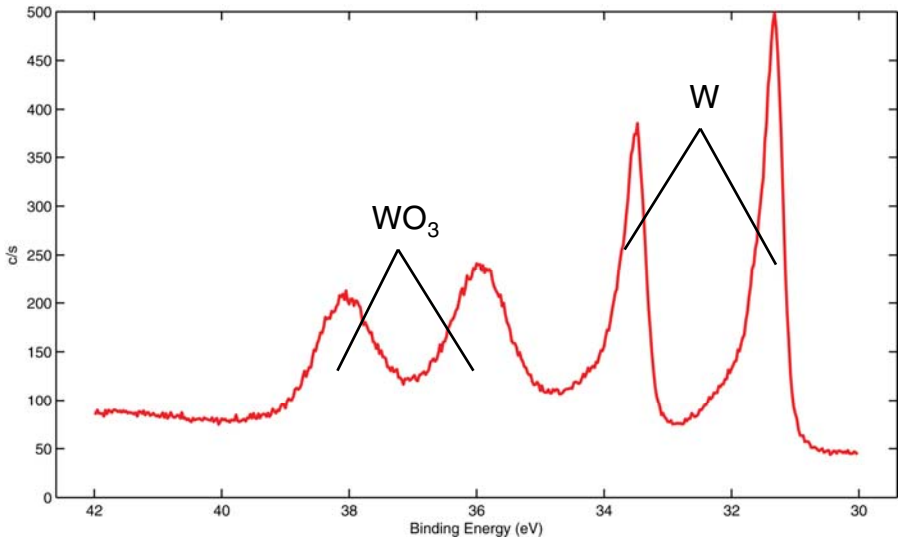


Methods: XPS

- Elemental analysis required due to different wall materials
- Thin layers require surface sensitivity
- Phase formation requires identification of chemical states

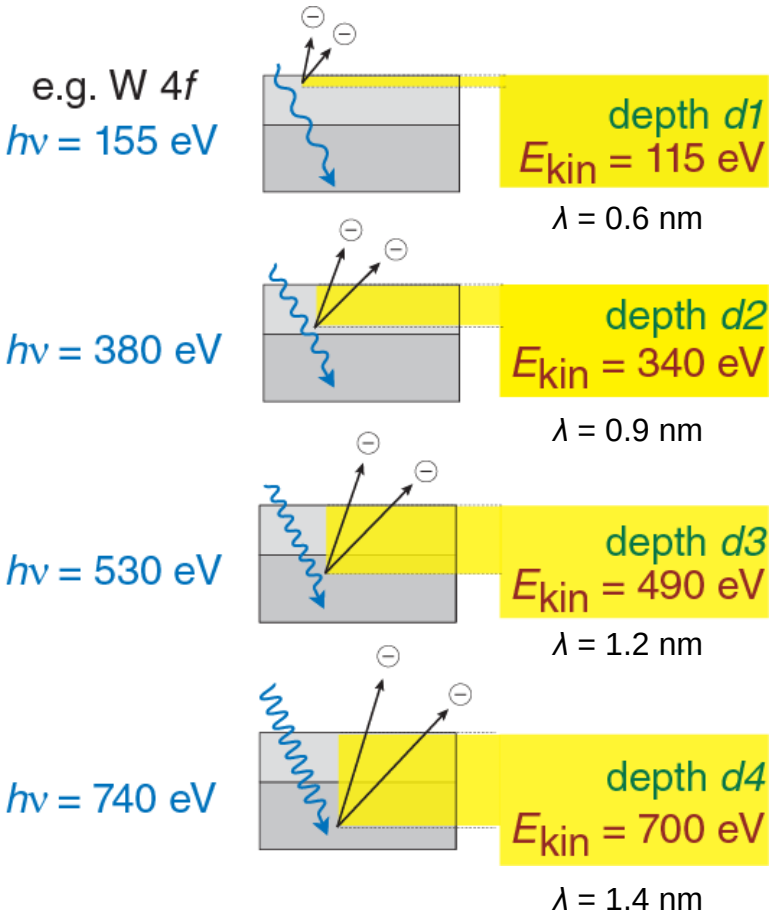
$$E_{kin} = h\upsilon - E_b - \Phi$$





⇒X-ray photoelectron spectroscopy (XPS)

Methods: Synchrotron-XPS



$$E_{kin} = h\upsilon - E_b - \phi$$

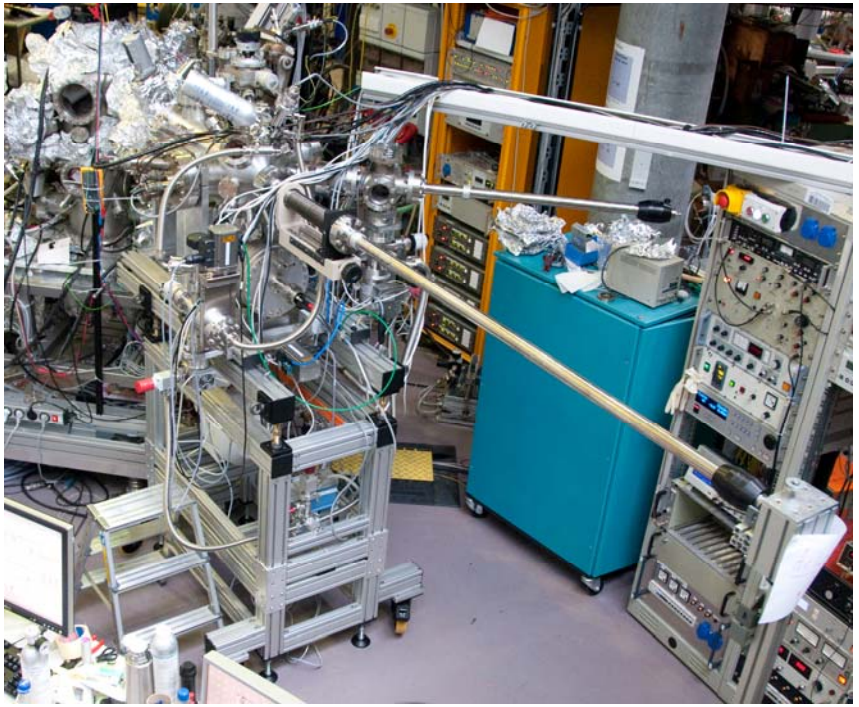
- Investigation of diffusive processes requires depth resolution
- Information depth depends on the kinetic energy of the photoelectron
- The kinetic energy depends on the energy of the photon

⇒ **Varying the photon energy changes the information depth**

⇒ **Only possible with synchrotron radiation**

Experimental Setup

- Sample preparation LAICA
  - 3 electron beam evaporators
  - Ion source (energies up to 5 kV)
  - Sample heater (up to 1370 K)
  - Auger system

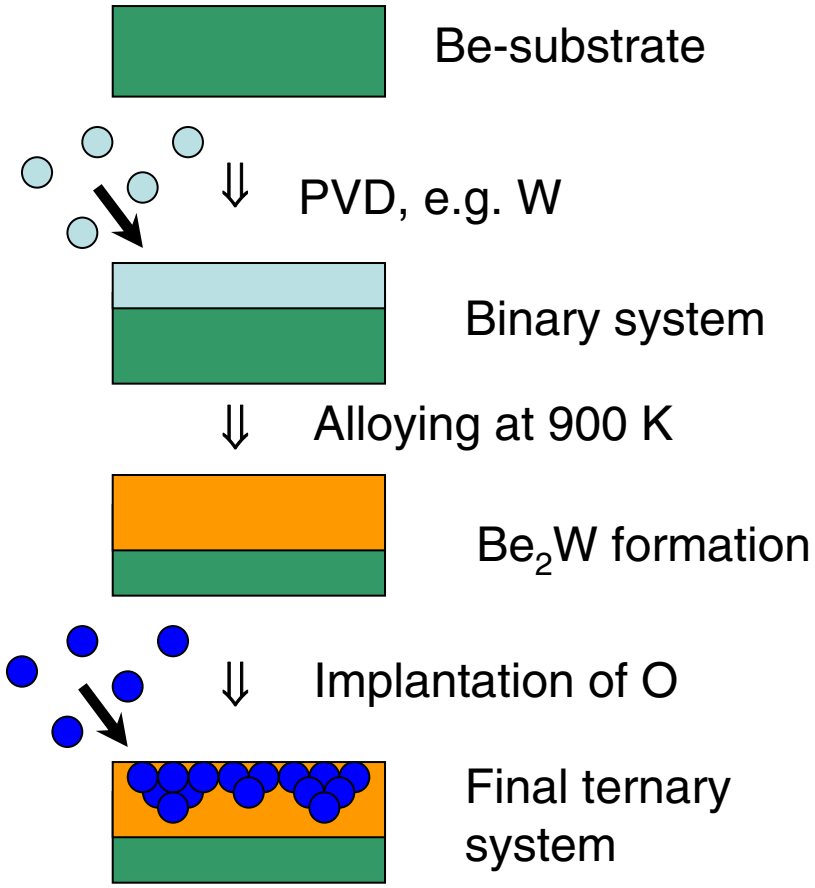


- Analysis chamber SurICat
  - Dedicated XPS system
  - Hemispherical analyser, energy resolution = 0.1 eV

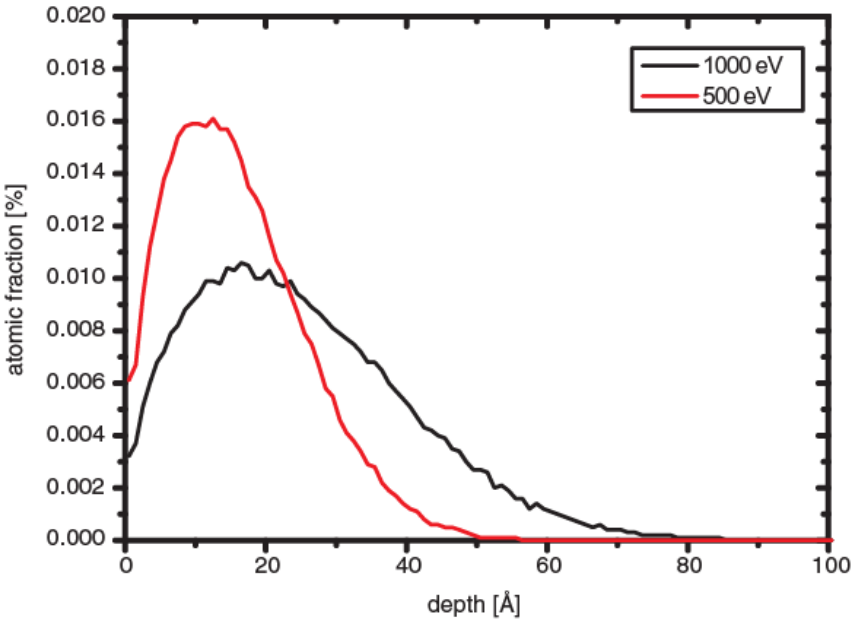
Sample preparation

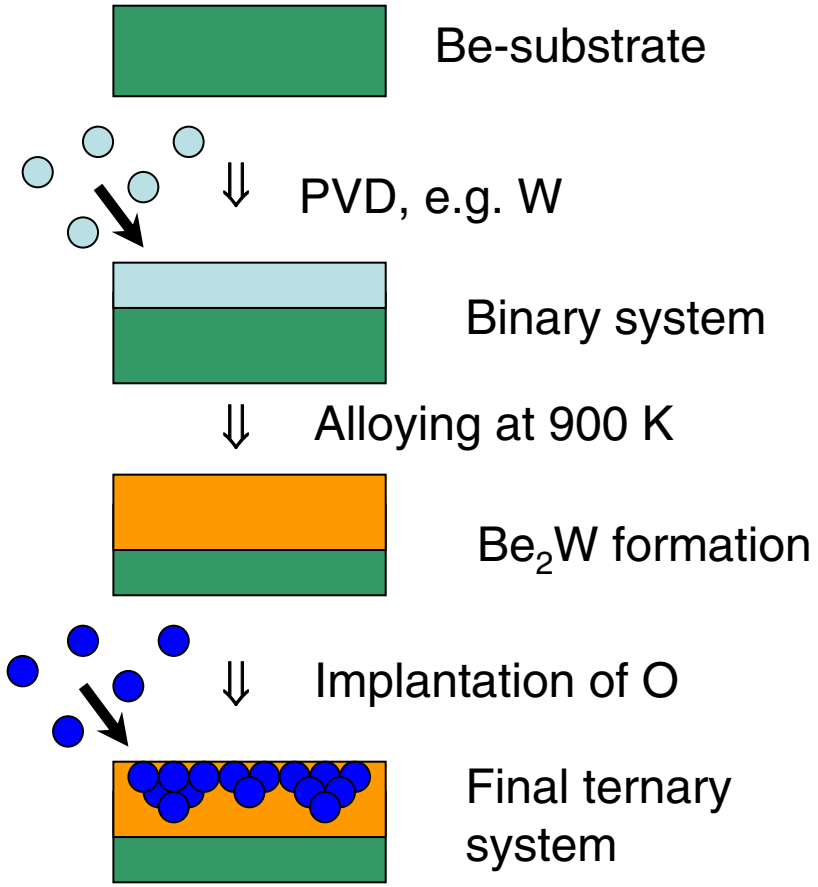
- In-situ sample preparation by physical vapour deposition (PVD) and ion implantation to simulate plasma processes:
  - Thin layers (nanometre range)
  - Interfaces
  - Ion implantation
- Heat treatment to temperatures up to 1370 K
- Binary systems of Be, W and O are investigated and well understood (several PhD Theses and articles)
- Classic XPS investigation of the Be on different tungsten oxides already performed

⇒ **Depth-resolved XPS on ternary systems of these elements**



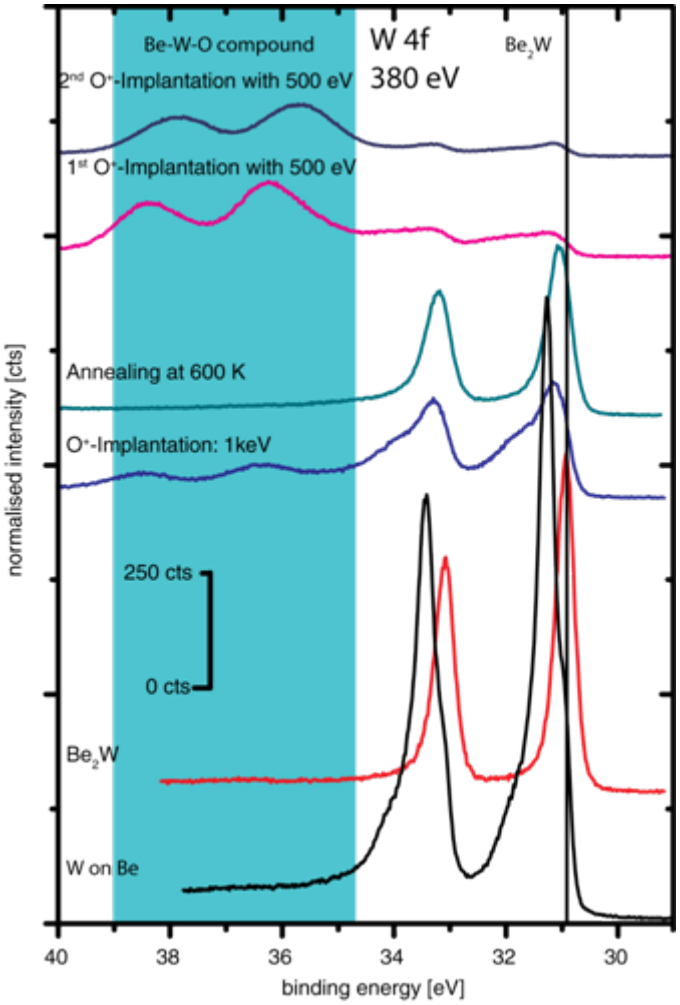
Sample preparation





Results

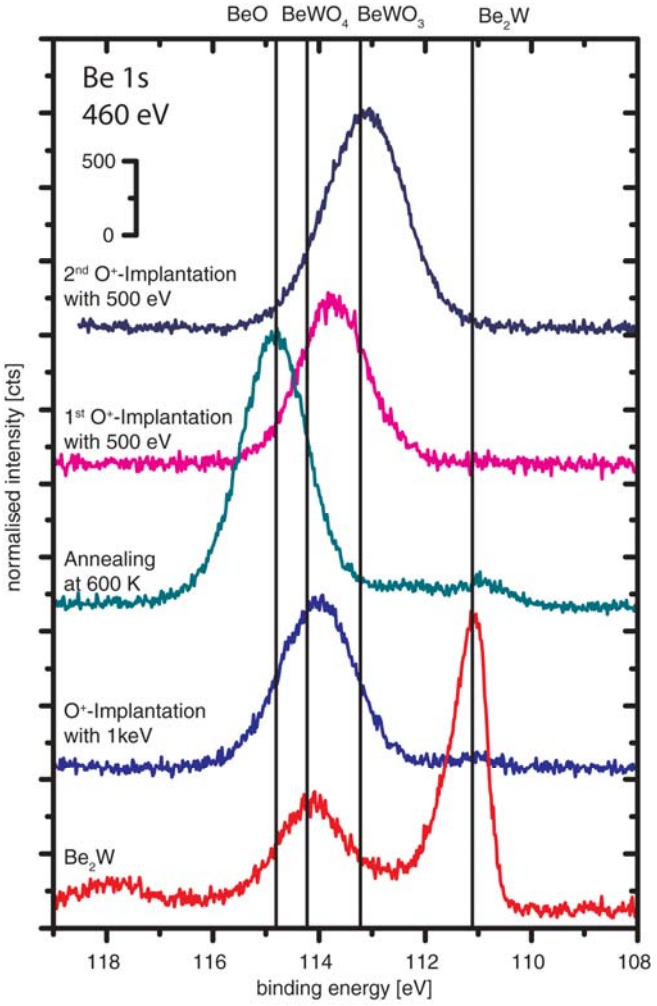
- Graph shows W 4*f* signals during the experiments in one depth ($h\upsilon$ = 380 eV, ID ~2.9 nm)
- After oxygen implantation: $BeWO_4$ visible
- After Annealing at 600 K: Decomposition of $BeWO_4$ although the tungstate is stable until 1000 K in Be-poor environment



Results

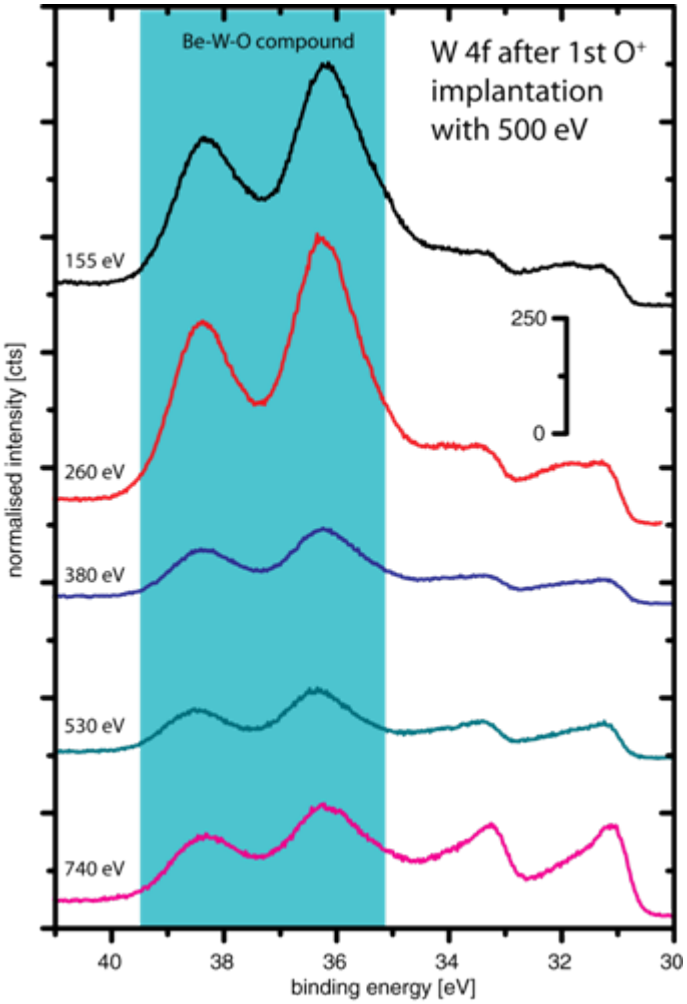
- After oxygen implantations: $BeWO_4$ and $BeWO_3$ also visible
- After annealing: shift to higher binding energies: BeO is visible

➢ Oxygen is bound in the sample as BeO



Results

- W 4*f* recorded at different information depth after O-implantation at 500 eV
- Ternary compounds mainly form in the surface near region



$h\upsilon$ [eV]	155	260	380	530	740
E_{kin}(W 4*f*)	115	220	340	490	700
λ [nm]	0.61	0.81	0.97	1.18	1.41
Est. ID [nm]	1.83	2.43	2.91	3.54	4.23

Summary

- Material investigation using synchrotron XPS:
  - Chemical information as gained by normal XPS combined with faster measurement times important for difficult systems (e.g. high oxophily of Be)
  - Method delivers additional depth resolution while being non-destructive

Summary

- Oxygen implanted beryllium tungsten alloy:
  - Oxygen implantation leads to reproducible formation of ternary Be-W-O compounds
  - Compound formation depth-dependant
  - Annealing at 600 K leads to destruction of ternary phases
  - Oxygen stays in the sample, bound as BeO after annealing

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