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Characterization of thick deposit found at the WEST inner divertor marker tile 32i after C4 campaign

Elżbieta Fortuna-Zaleśna, WUT

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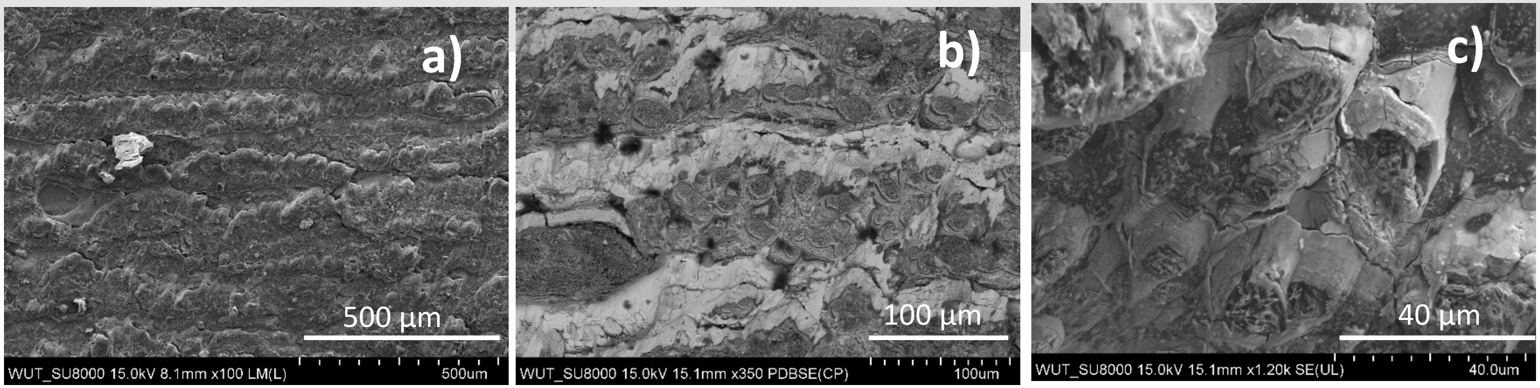
Outline

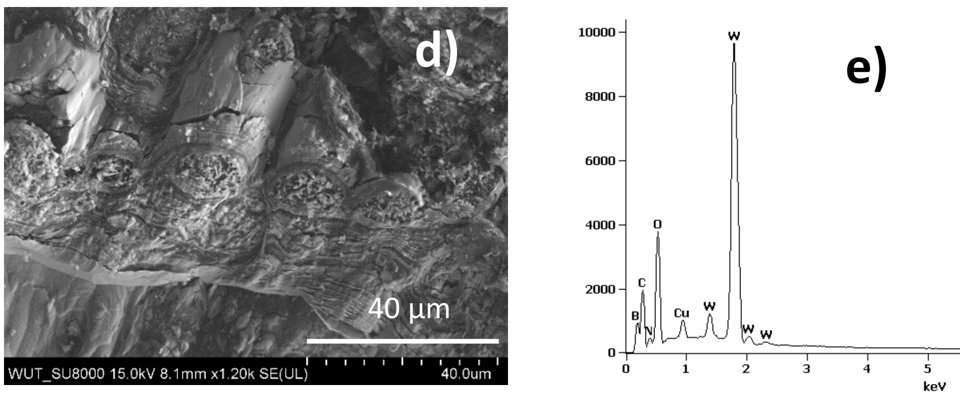
- ***Introduction***
- ***Deposit morphology***
- ***Deposit internal structure***
- ***Comparison of the structure of WEST deposit to those observed in other devices (AUG, JET)***

Introduction

- ✓ *The erosion and re-deposition patterns on entire erosion marker tiles from the inner and outer WEST divertor after C3 and C4 campaigns were determined at IPP using ion beam analysis.*
- ✓ *Further analysis were performed on small samples cut out from the above-mentioned tiles, using the first set of results as a guidance. They were distributed between several labs for additional surface analysis.*
- ✓ *The studies carried out at the Warsaw University of Technology comprised examinations of the surface morphology and chemical composition by SEM and measurements of the marker layer and/or re-deposited layer thickness at the FIB cross-sections. All together 9 samples were examined: 6 from C3 campaign and 3 from C4 campaign. Their research was aimed at:*
  - *Net erosion/deposition pattern on the divertor*
  - *Impurities in deposits*
  - *Evolution of microstructure*
  - *Comparison between C3/C4*
  - *How the WEST deposits compare with the other metallic devices (AUG)*
- ✓ *In todays talk the latest results concerning the characteristics of the thick deposit found at the WEST inner divertor marker tile 32i after C4 campaign will be presented (C4_32iJ).*

Deposit surface morphology



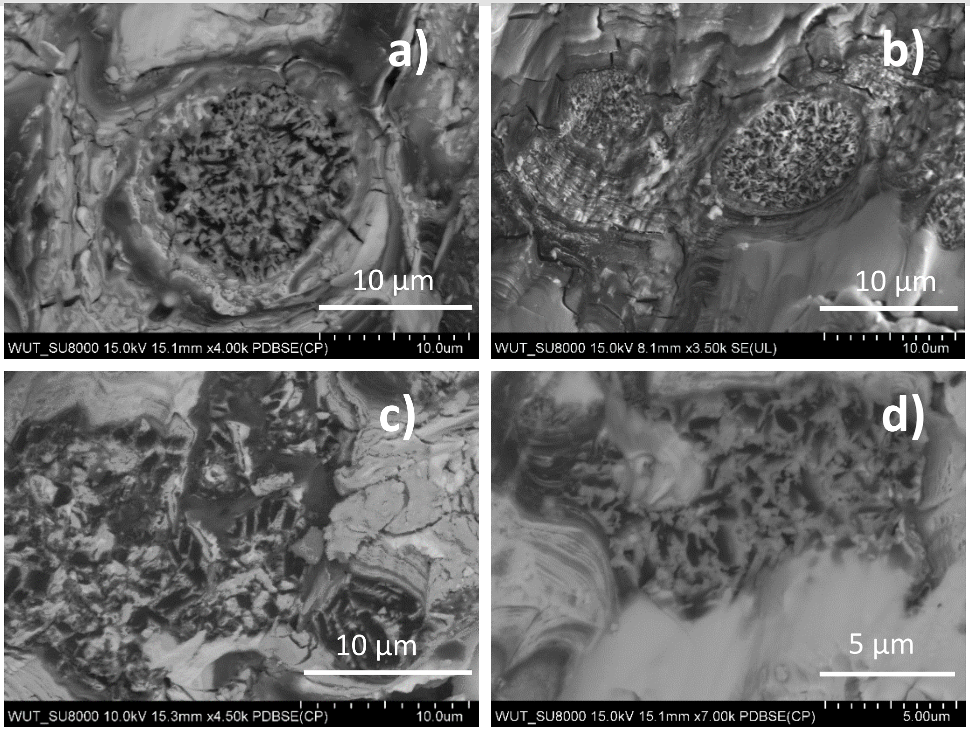


SEM images of the surface morphology (a-d) together with corresponding EDX spectrum from the area (e).

At low magnification, one can see lobes/sheets of the deposit, clearly separated from the neighboring ones (Fig. a). These areas are inhomogeneous both in terms of morphology and chemical composition. Within these flakes both spherical/elongated/tear-like elements (several tens micron in diameter) and stratified structures can be seen. Additionally, at the top of these structures, locally there are bands of deposit rich in tungsten. They are visible in the images registered in the BSE mode as white stripes. Deposit is cracked. In some placed fragments of deposits are missing.

EDX measurement from the area clearly show increased signal from B, O, C and Cu as well as small but distinct peak from N.

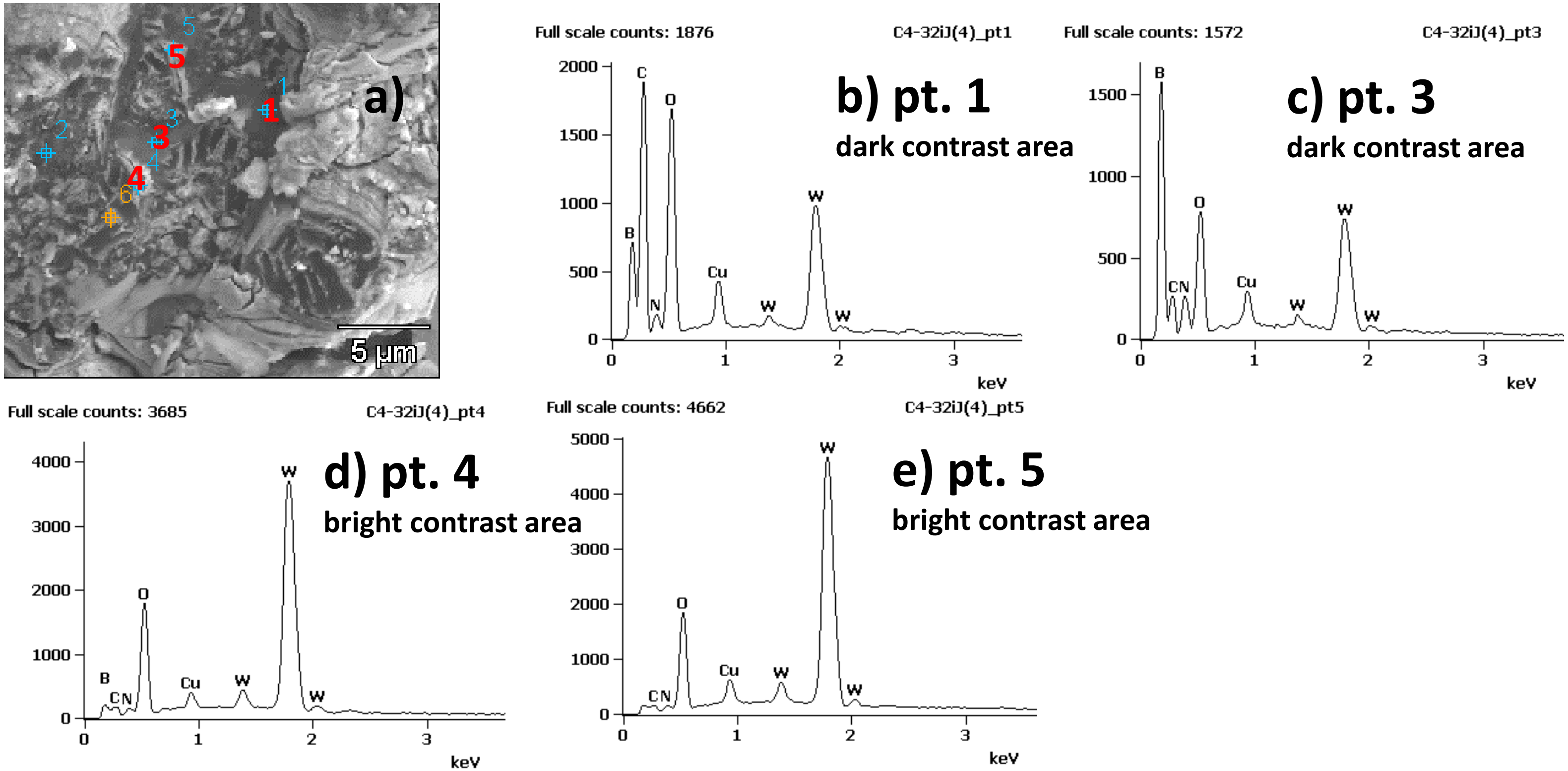
Deposit surface morphology



SEM images of the spherical elements morphology.

- ✓ *Inside the elongated elements very often the characteristic morphology, presented in Figs. a-d is observed. In their interior there are fine areas strongly differing in chemical composition (dark and bright contrast ones). The outer part of these outgrowths is ordinarily covered by a layered skin.*

Surface composition



SEM image of the elongated element interior (a) together with corresponding EDX spectra (b-e).

✓ *EDX point analyses show the dark contrast areas inside the elongated elements are rich in boron, whereas the bright ones in tungsten and oxygen.*

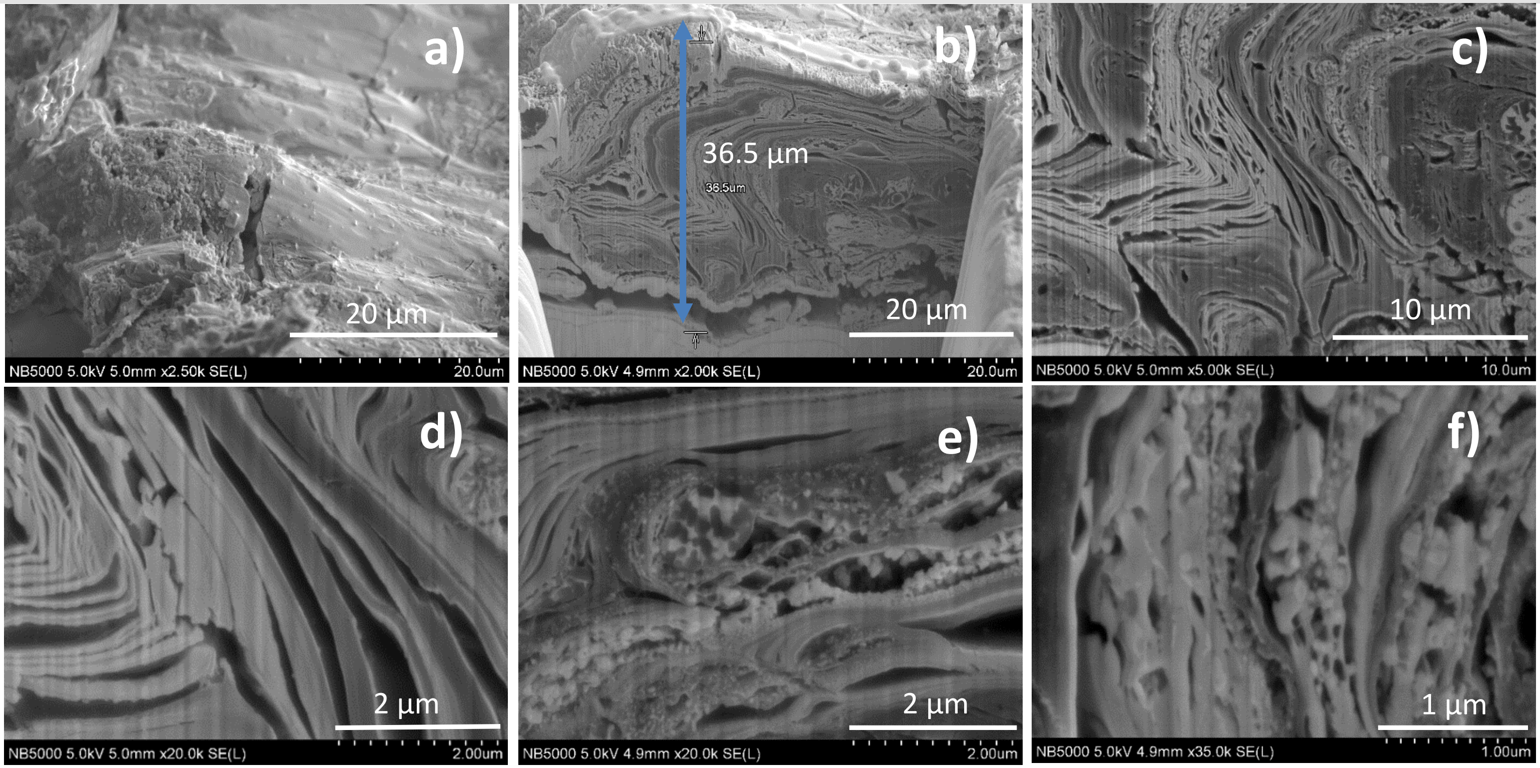
Summary

- ✓ *The structure of examined re-deposited layer is complex. At low magnification, one can see sheets/flakes of the deposit, clearly separated from the neighboring ones. These areas are inhomogeneous both in terms of morphology and chemical composition.*
- ✓ *Within these flakes both spherical/elongated elements (several tens micron in diameter) and stratified structures can be seen. Additionally, at the top of these structures, locally there are directional bands of deposit rich in tungsten. Deposit is cracked. In some placed fragments of deposits are missing.*
- ✓ *Inside the elongated elements very often the characteristic morphology is observed. In their interior there are fine areas strongly differing in chemical composition (dark and bright contrast ones). The outer part of these formations/outgrowths is usually covered by a layered skin.*
- ✓ *EDX measurement from the area clearly show increased signal from B, O, C and Cu as well as small but distinct peak from N.*

Deposit internal structure

- *Two FIB cross-sections - longitudinal and transverse ones were cut through the two randomly selected elongated elements in order to reveal their internal structure and measure the deposit thickness.*
- *As the examined objects are large, to cut them large and deep cross-sections had to be made (depth ~80 μm, length ~50 μm).*
- *Their structures were examined in detail.*

Deposit internal structure, longitudinal section



SEM images of the re-deposited layer and its cross-sections.

- ✓ *The cross-section was taken along an elongated, tear-shaped element, ~ 50 µm long, Fig. a.*
- ✓ *In the area of the particle, we can distinguish something like a nucleus/core and a deposit with a layered structure. The layers are not parallel to the surface but bend around the core. This structure indicates that layers have grown up on this element. Deposit thickness up to 37 µm.*
- ✓ *Large crack/delamination at the marker layer-deposit border.*
- ✓ *Clearly visible layered structure, although in some areas the boundaries between the layers are blurred / not visible. Most likely the material transforms into more equilibrium structures.*

Deposit composition, longitudinal section

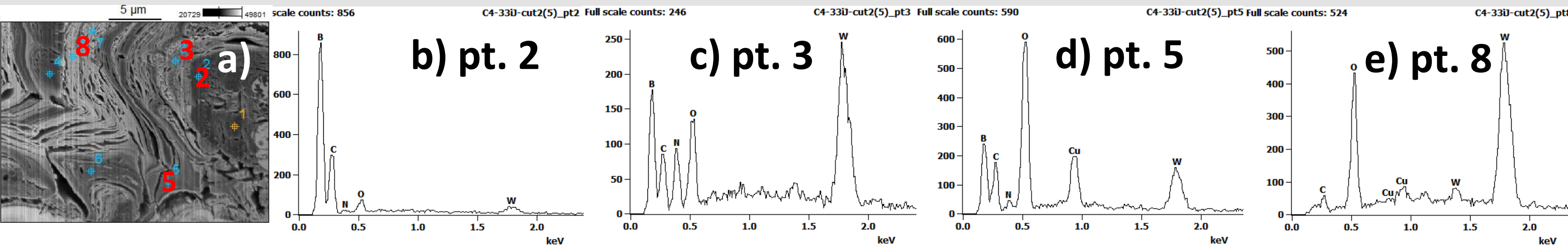


Fig. 1. SEM image of the deposit cross-section (a) together with corresponding EDX spectra (b-e).

✓ *EDX analyzes clearly show the variation in chemical composition between the layers. What can be noticed is the presence of oxygen in the layers rich in tungsten.*

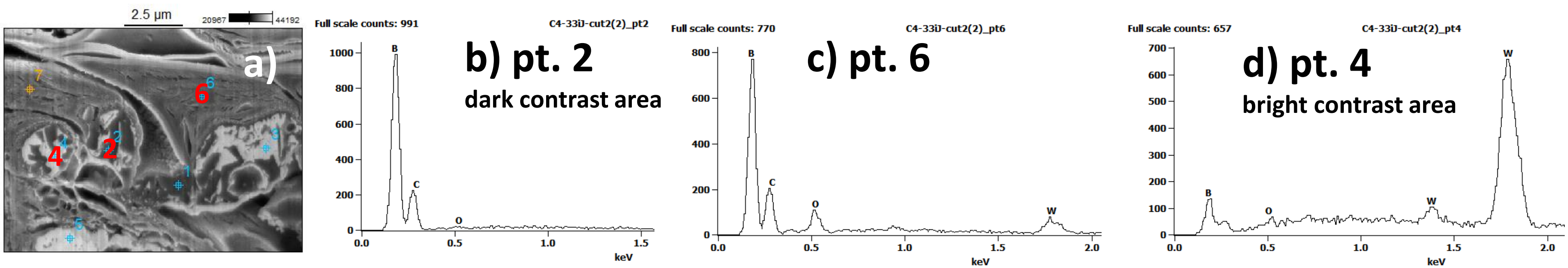
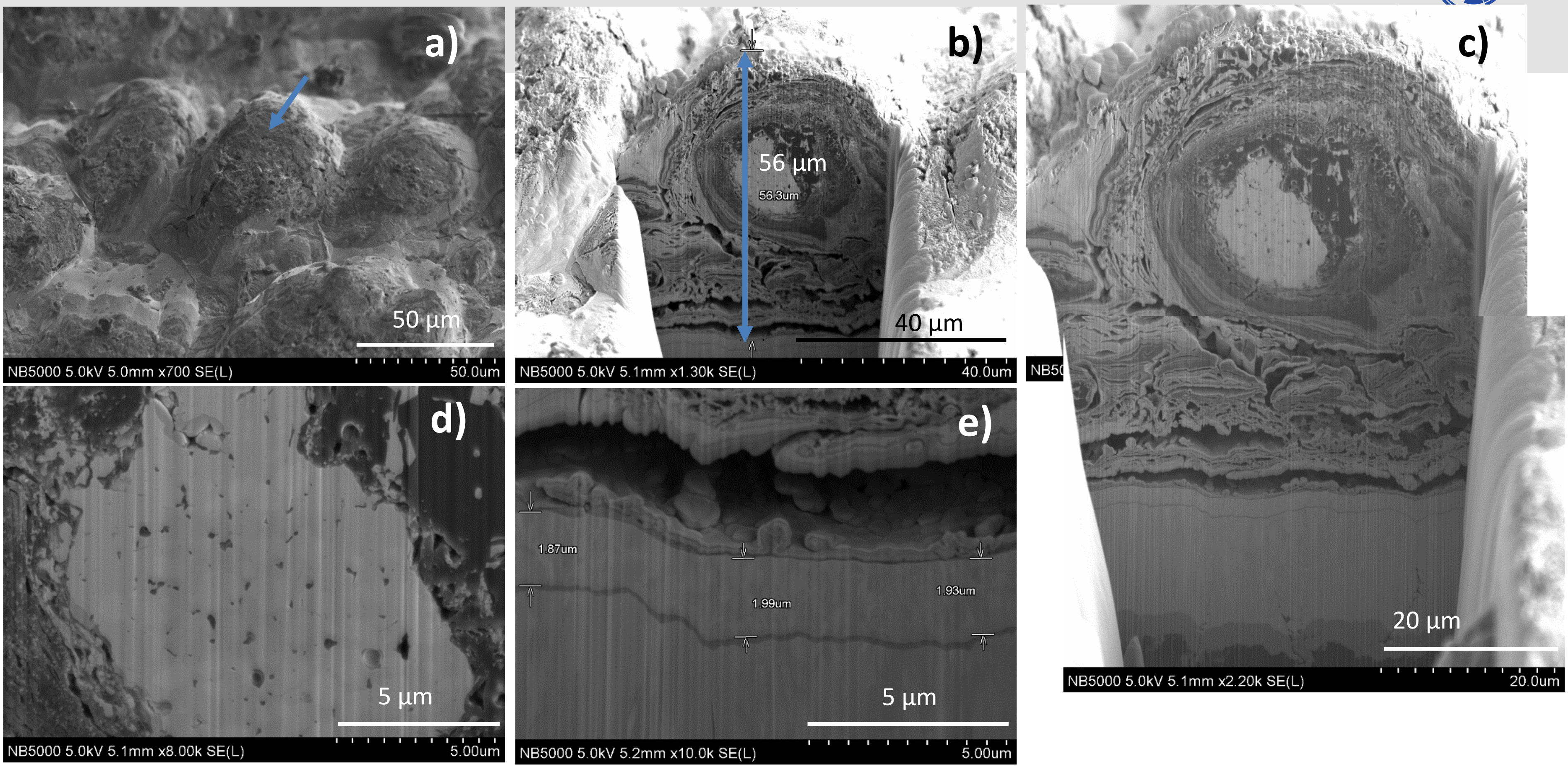


Fig. 2. SEM image of the deposit cross-section (a) together with corresponding EDX spectra (b-d).

✓ *Fig. 2a shows the areas with a two-phase structure. Dark-contrast crystals are rich in boron and light-contrast ones in tungsten.*

Deposit internal structure, transverse section



SEM images of the re-deposited layer and its cross-sections.

- ✓ *The cross-section was taken across an elongated, tear-shaped element, ~ 50 µm high, Fig. a.*
- ✓ *In the area of the particle, we can distinguish something like a nucleus/core, the details of which are shown in Fig. d, and a deposit with a layered structure. In the bottom part of the re-deposited layer the sub-layers are more less parallel to the surface, however the sublayers located higher are bent around the core. Such a structure indicates that these sub-layers have grown up on this element. Deposit thickness measured at the cross-section is up to 56 µm.*
- ✓ *The marker layer thickness is equal about 2 µm, Fig. e. Large crack/delamination at the marker layer-deposit border.*

Core structure and chemical composition

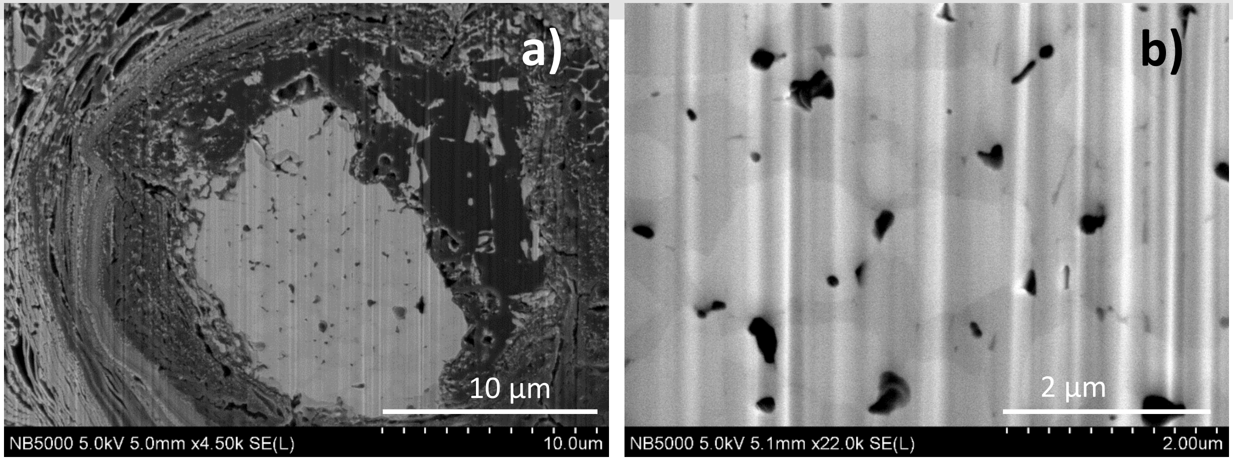


Fig. 1. SEM images showing details of the deposit internal structure.

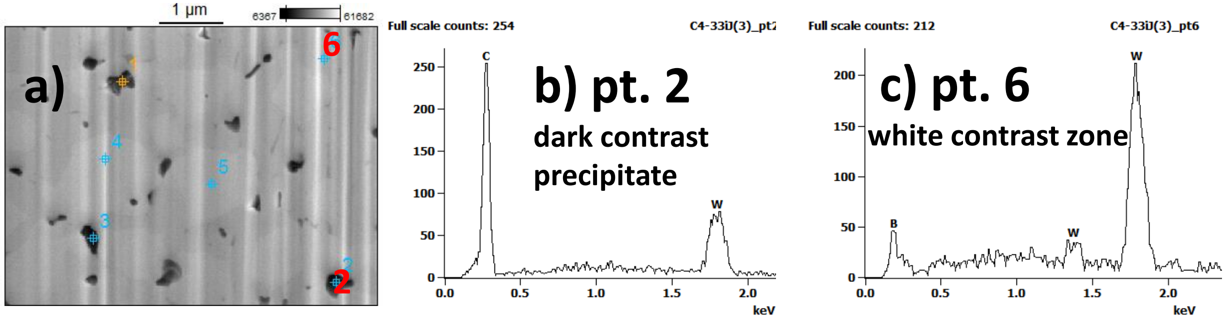


Fig. 2. SEM image of the core (a) together with corresponding EDX spectra (b-c).

- ✓ *In the central part of the cross-section there is a particle of the size of about 15 microns with a granular structure and a grain size of 1-2 microns. EDX analyzes show that it contains tungsten and possibly also boron (probably the intermetallic phase). At the grain boundaries, precipitation with a size of up to 400 nm rich in tungsten and carbon present (tungsten carbide?).*

Deposit - dark contrast crystals

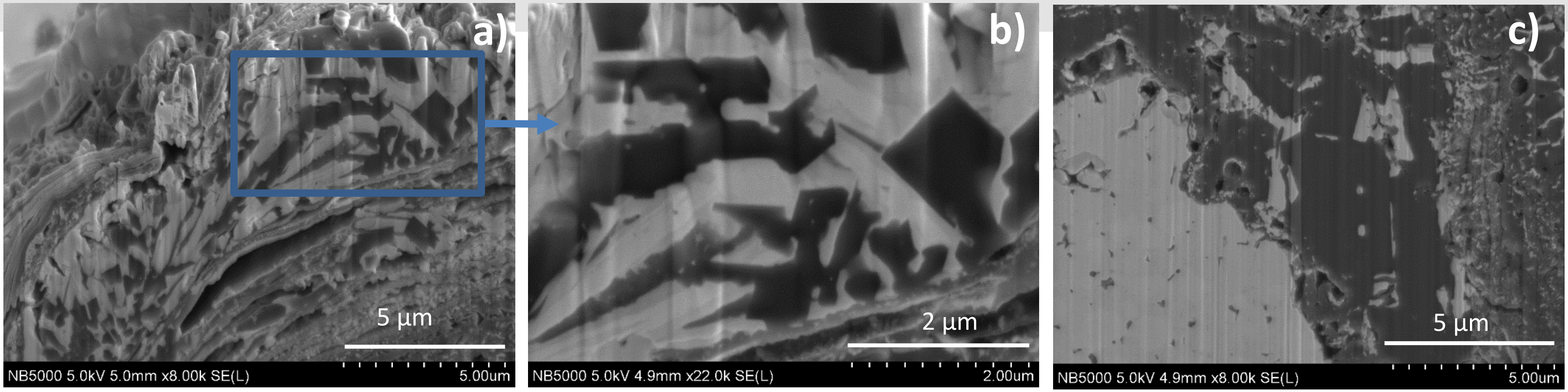


Fig. 1. SEM images showing regions with boron rich crystals (a-c).

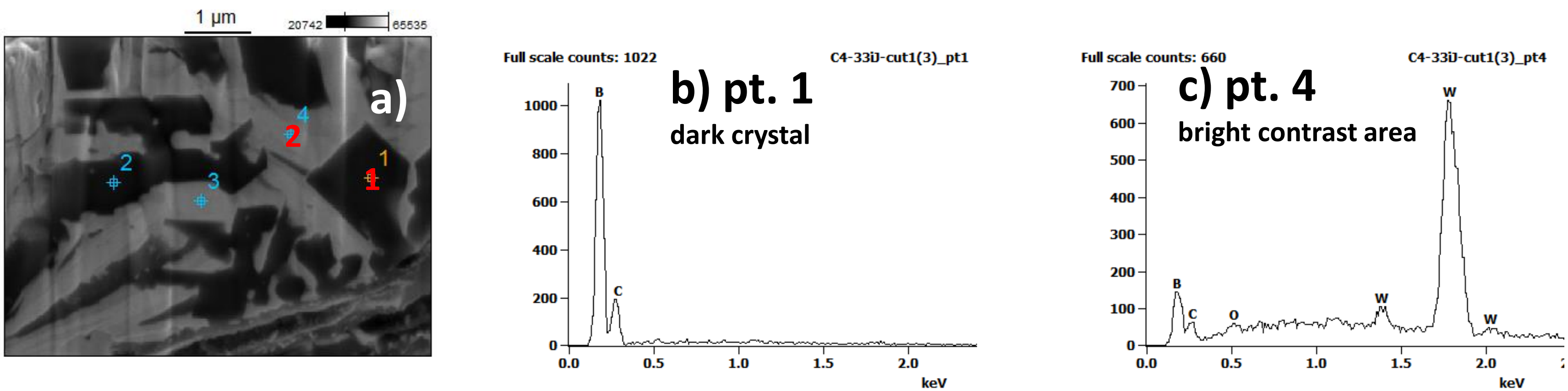
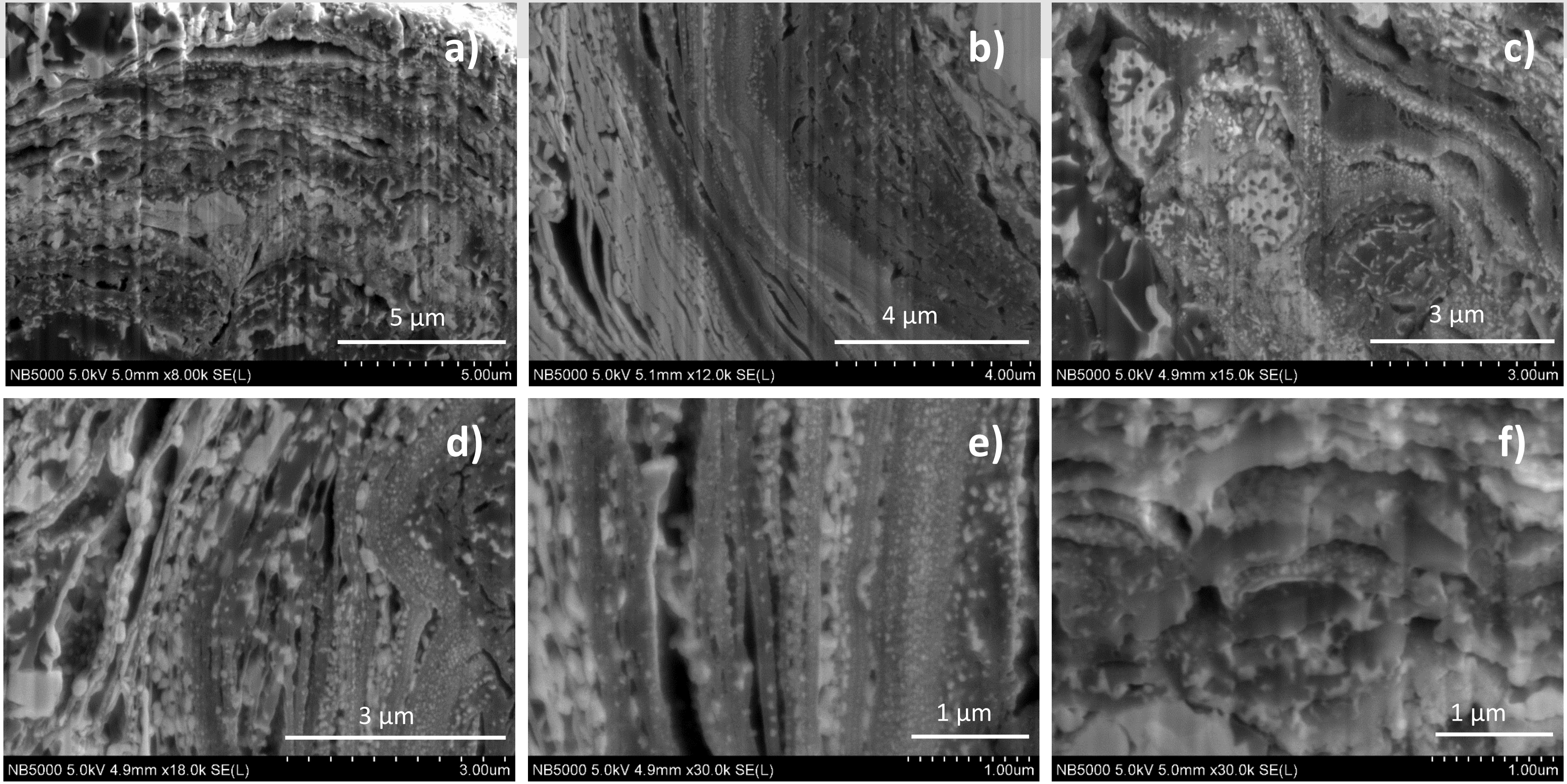


Fig. 2. SEM image of the boron-rich crystals (a) together with corresponding EDX spectra (b-c).

- ✓ *The areas containing boron-rich crystals are found both in the outer layer of the deposit and in its interior. EDX analyzes show that except boron they contain also carbon (most likely boron carbides).*

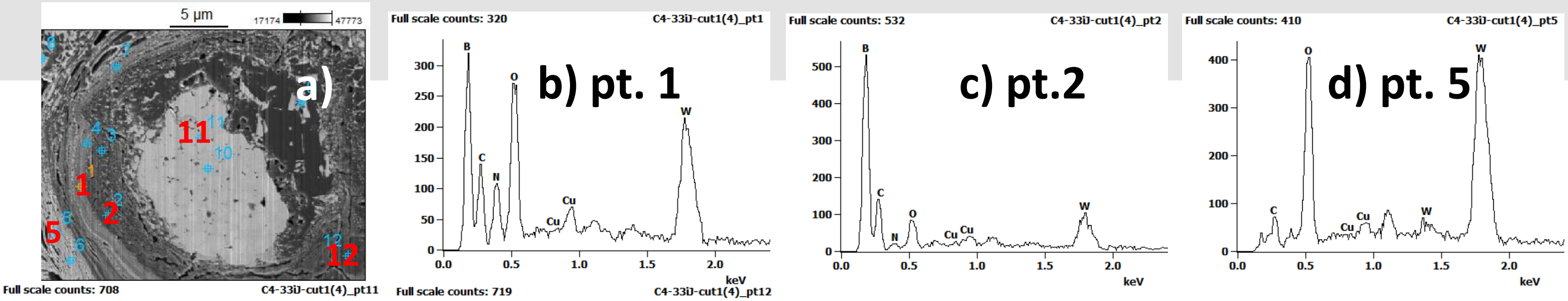
Deposit morphology - layers



SEM images showing the details of deposit internal structure.

- ✓ *Very inhomogeneous material. Strong variation in chemical composition observed in most sublayers.*
- ✓ *Most likely the material transforms into more equilibrium structures – Fig. c.*
- ✓ *There are zones with a similar chemical composition.*

Deposit chemical composition



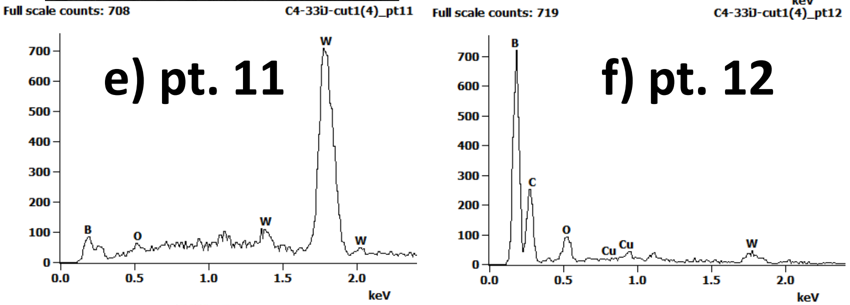


Fig. 1. SEM image of the deposit together with corresponding EDX spectra, illustrating the differences in the chemical composition between different areas/zones of the deposit.

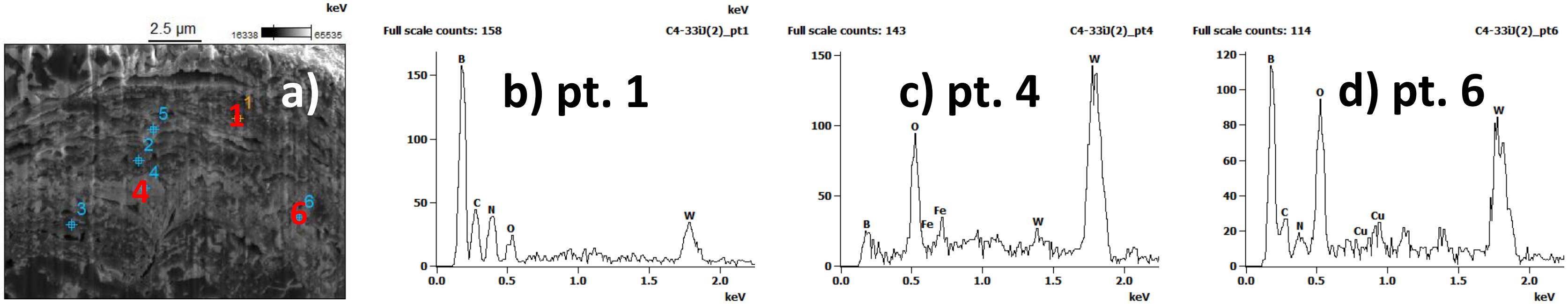
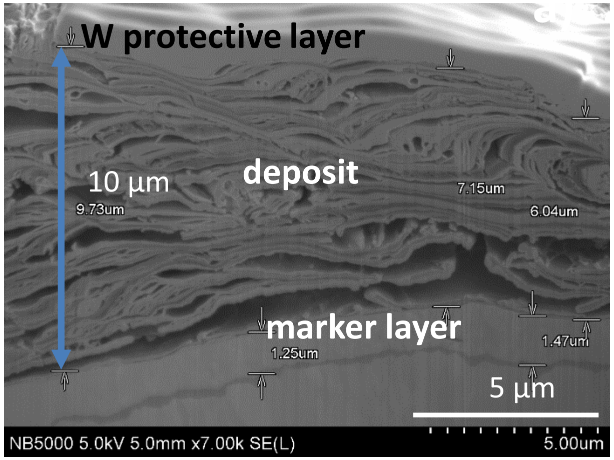


Fig. 2. SEM image of the deposit together with corresponding EDX spectra, illustrating the differences in the chemical composition between sublayers in the micro-scale.

- ✓ *Distinct differences in the chemical composition between individual zones and sublayers.*
- ✓ *In the case of tungsten-rich regions, two types of sublayers can be distinguished: (i) those that contain a significant amount of oxygen in addition to tungsten, and (ii) those that contain boron in addition to tungsten.*
- ✓ *Clear tendency towards segregation and formation of zones rich in boron and carbon.*

Summary/Conclusions

- *In the bottom part of the re-deposited layer there is a zone with the sub-layers more less parallel to the surface. This zone has similar morphology to what we have seen in the same location after C3 campaign, however, is much thicker (15-20 µm after C4 campaign vs. 10 µm after C3). After the C3 campaign, we also have not observed the elongated/spherical structures being a characteristic feature of the deposit formed after the C4 campaign.*



SEM image of the deposit and marker layer cross-section, sample C3_34iJ, s = 180.5 mm

- *The results of EDX point measurements made on the deposit cross-sections indicate a high probability of the presence of intermetallic phases, borides, carbides and oxides in the material.*

Summary

- ***In many ways, the structures observed in the deposit under study mirror those found in the AUG*** *(see morphology of dust particles with boron carbide crystals presented in Phys. Scr. T159 (2014) 014066 and deposit morphology in Nuclear Materials and Energy 9 (2016) or Fusion Eng. Des., 86 (2011), pp. 1753-1756].*

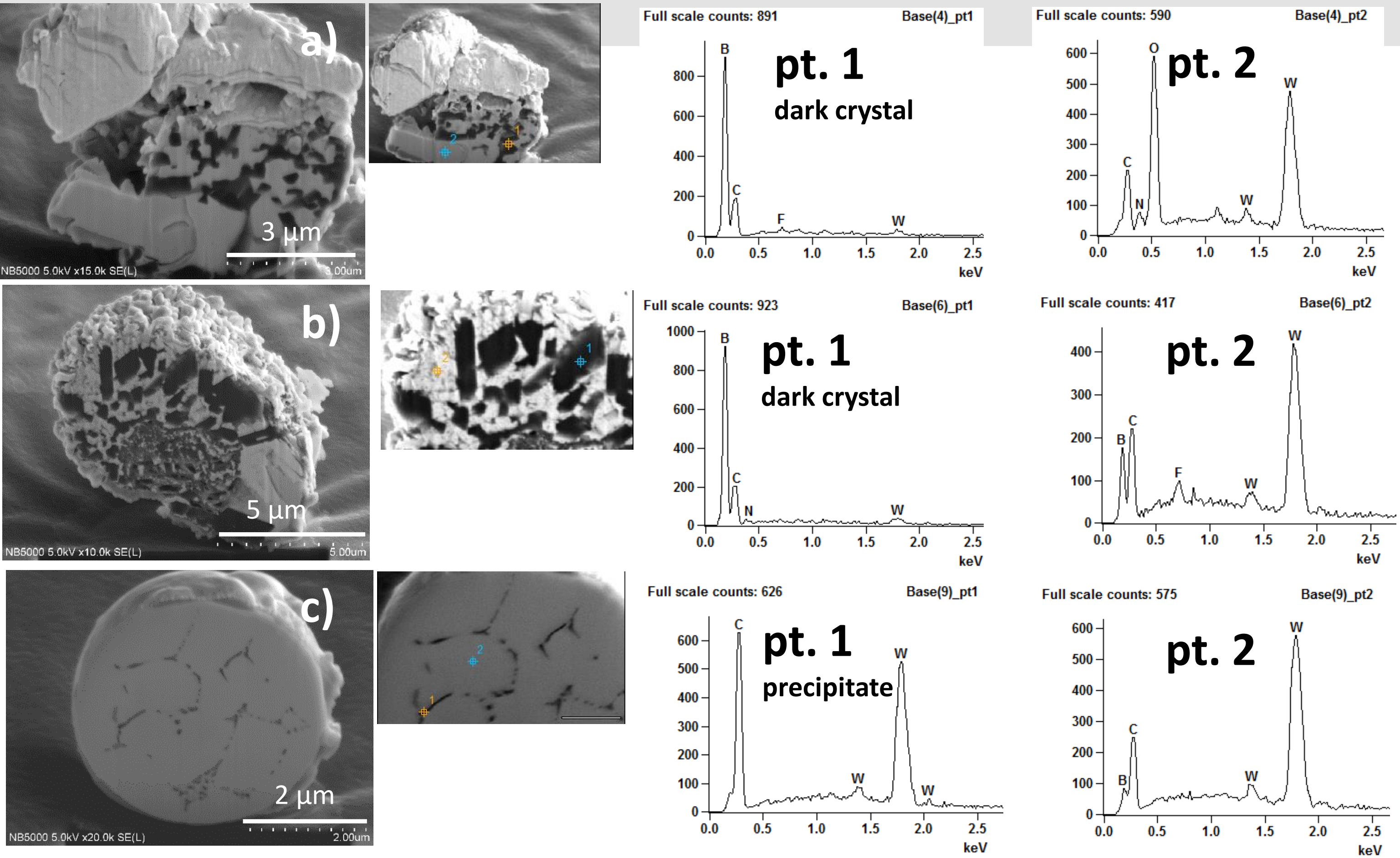
- *M. Balden [J. Nucl. Mater. 438 (2013) S220] studied the surface of polished tungsten specimens after exposure to the outer divertor plasma close to the strike point in the 2011 campaign. Porous, layered deposits formed around dust particles stacked at the surface, with an elongation at an angle of approximately 45 °to the B field lines.*

- *Changes in the deposit morphology under the influence of temperature (from a layered, amorphous to a crystal structure) were observed in the co-deposits grown in the JET reactor [see Fig. 5 in Nuclear Materials and Energy 12 (2017) 582-587 and 2021 Phys. Scr. 96 124038].*

- *The structure of two distinct types of carbon-based deposits, stratified and columnar/granular, is related to the temperature in the zone where the layers were formed. In the case of TEXTOR laminar structures occurred at surfaces where temperature usually did not exceed 600 K, while the granular were typical for high temperature range (even 2000 K) [Phys. Scr. T167 (2016) 014059]. It can be assumed that a similar dependence may also apply to co-deposits from all-tungsten devices (of course, the temperature does not have to be the only factor influencing the deposit morphology).*

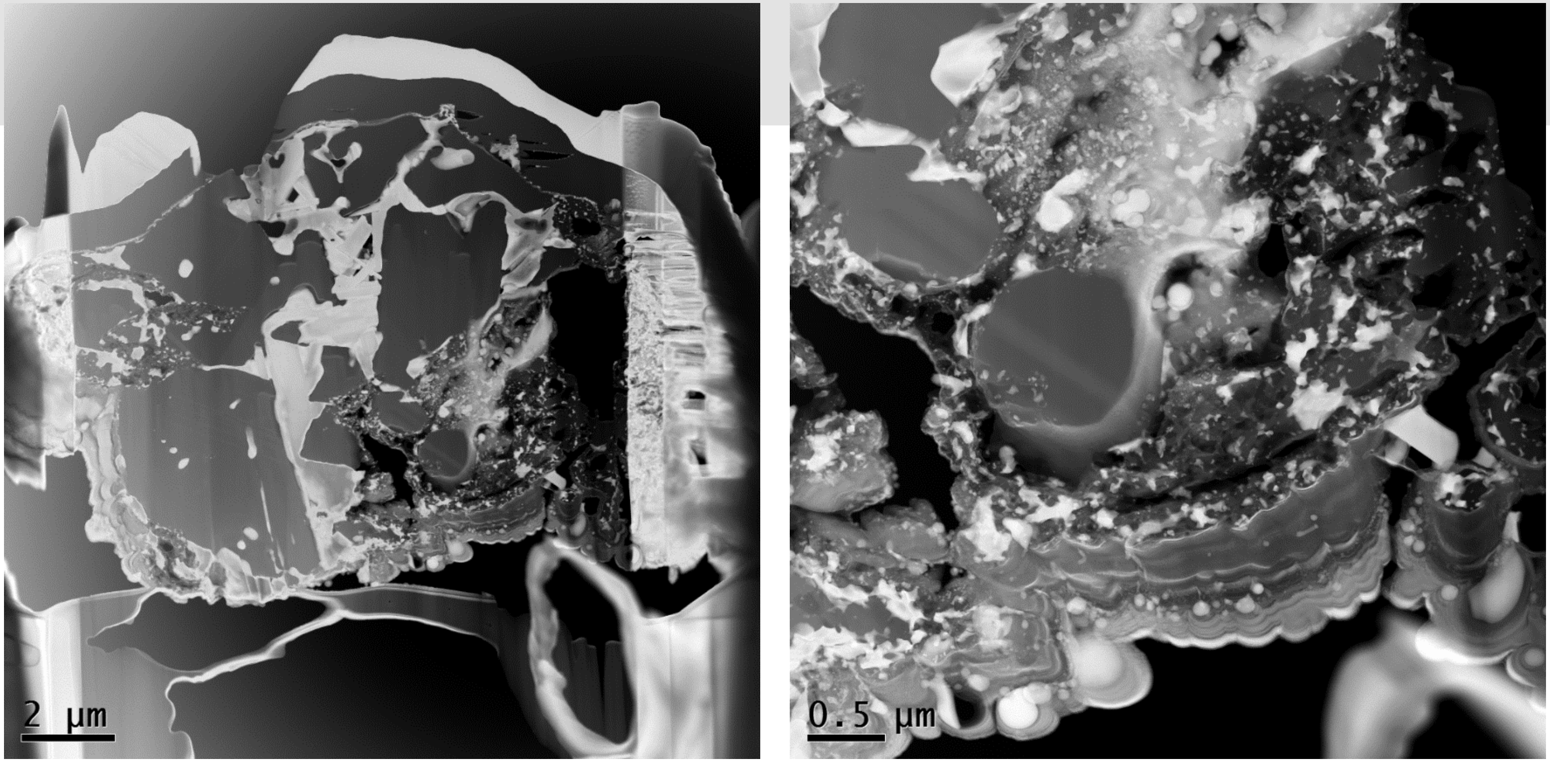
Morphology of re-deposited layers from AUG and JET

AUG dust internal structure



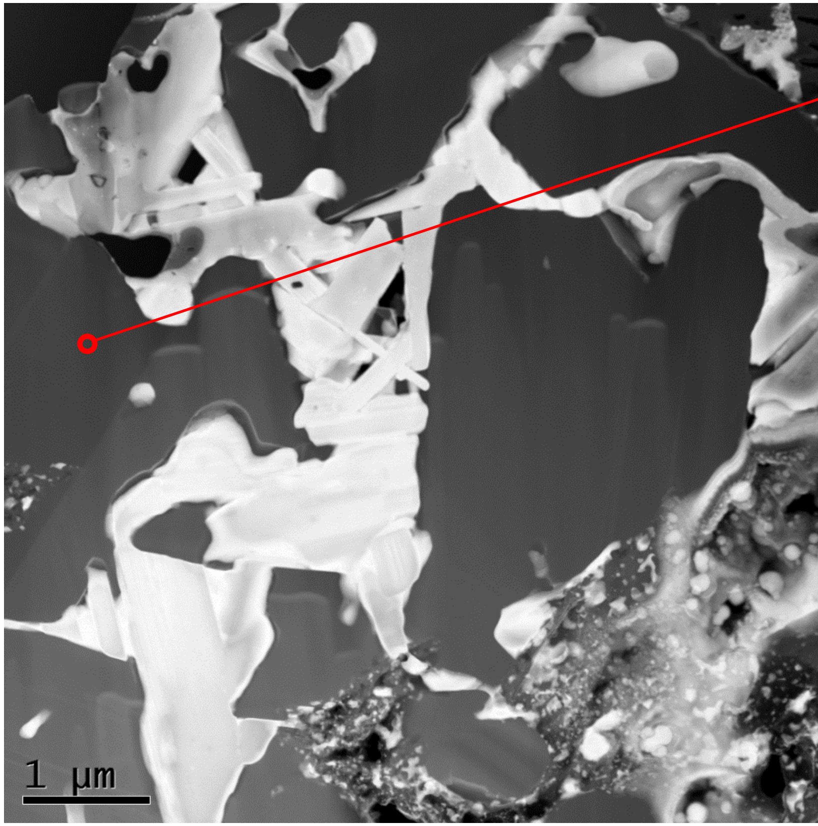
SEM images of dust particles cross-sections collected after the 2009 campaign using the filtered vacuum technique together with corresponding EDX spectra.

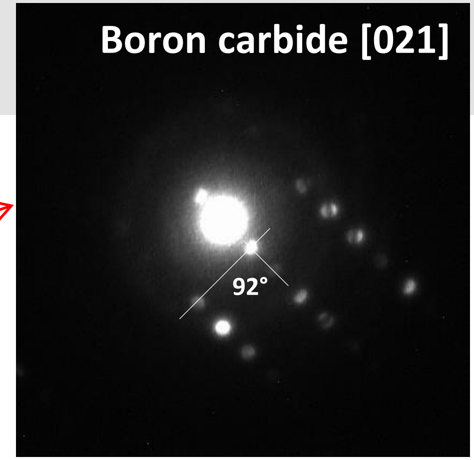
- ✓ *Boron–rich crystals found inside dust particles,*
- ✓ *Precipitation of carbides observed,*
- ✓ *The structure of particles with grains rich in boron is the same as the deposited layers at the inner divertor baffle region.*



TEM images of a thin foil cut out from the AUG dust particle.

- ✓ *Large dark contrast crystals rich in boron and bright contrast crystals rich in tungsten present.*
- ✓ *In the bottom part of the foil layered structure is visible.*
- ✓ *Transition area present between these two zones.*



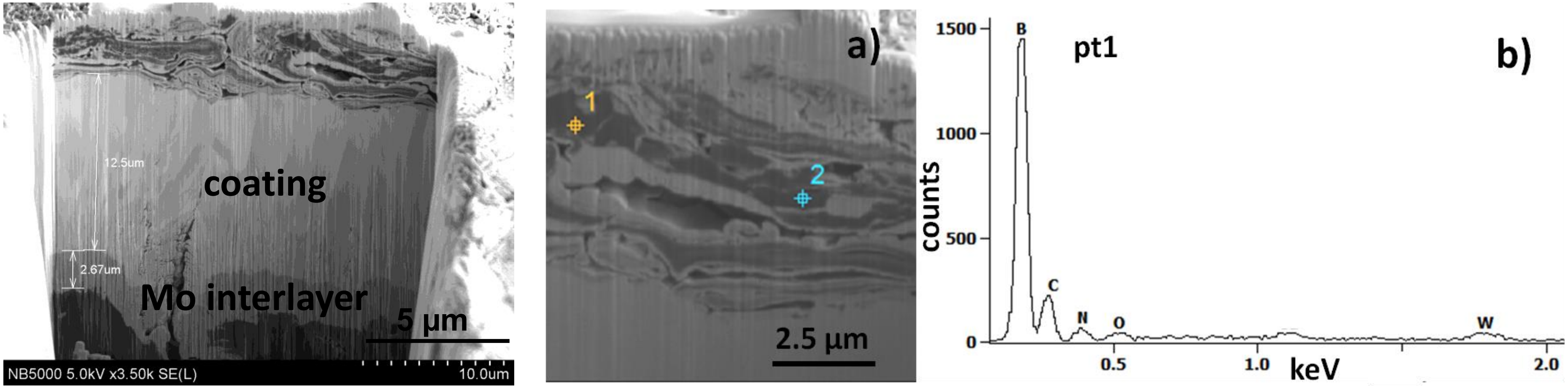


	Experimental data	Literature data
$d_{(21\text{-}2)}$	1.71 Å	1.76 Å
$d_{(1\text{-}12)}$	3.79 Å	3.81 Å
α	92°	92.32°

- ✓ *The dark phase was identified by diffraction patterns as boron carbide.*
- ✓ *Bright crystals have not revealed artifacts induced by ion milling which suggests that it is not pure tungsten but an intermetallic phase/phases.*

Work done by J. Grzonka. Results published in Phys. Scr. ***T159*** *(2014) 014066*

Morphology and chemical composition of AUG deposits

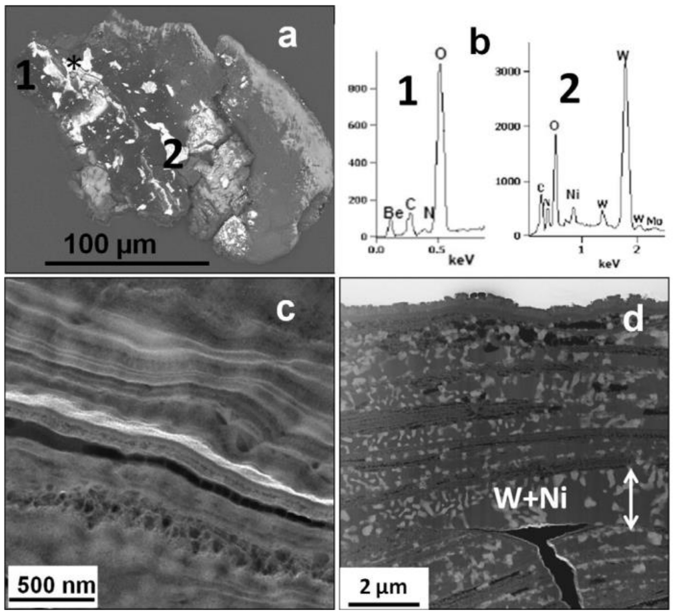


SEM images of the deposit present at the outer divertor strike point tile 1, private flux region (retrieved from the machine after 3 campaigns 2009-2013) cross-section together with corresponding EDX spectrum.

✓ *Crystals rich in boron and carbon found [Nuclear Materials and Energy 9 (2016) 128–131].*

Morphology and chemical composition of JET deposits

Changes in the deposit structure under the influence of temperature



Deposits on apron of Tile 1 after ILW-2 campaign in 2013–2014:

(a) a typical agglomerated particle,

(b) EDX spectra from two regions,

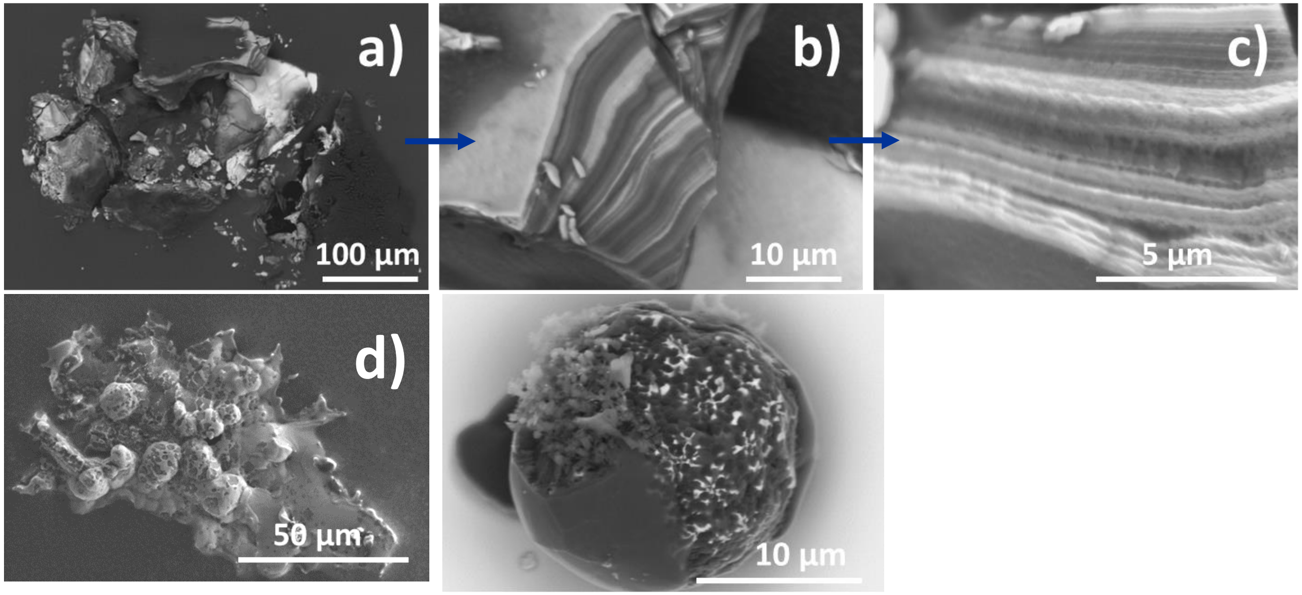
(c) stratified Be-rich deposit, lamella cut from the region indicated as 1;

(d) highly porous mixed deposit with light and heavy elements, lamella cut from the region indicated with asterisk.

Fig. 5 from E. Fortuna-Zalesna et al. Nuclear Materials and Energy 12 (2017) 582-587

Morphology and chemical composition of JET deposits

Changes in the deposit structure under the influence of temperature



Particles collected from Be limiter tile 2XR11 from the JET inner wall guard limiter.

E Fortuna-Zaleśna et al 2021 Phys. Scr. ***96*** *124038*

Thank you for your attention