



Post-mortem analysis capabilities for W dust studies

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- *What is dust?*
- *Risks associated with the presence of dust in the reactor*
- *Investigations of dust in the JET and AUG reactors*

What is dust?



***Dust** – loose particles that are formed in fusion devices as a result of plasma-first wall interactions.*

*Safety-related issues concern **mobilizable in vessel dust** (size between 100 nm and 100 μm).*

Risks associated with the presence of dust in the reactor



The generation of dust particles in a reactor-class thermonuclear reactor is considered as a potential hazard, which creates severe issues regarding safety and the economy of operation.

- **Potential hydrogen production from the reaction with steam after an accidental water leak.**
- **Potential dust explosion following hydrogen production and an accidental air ingress.**
- **Tritium-contaminated and neutron-activated dust is also a radiological hazard.**
- **Dust can affect the plasma's stability if it gets into the area where fusion reactions take place.**
- **Decrease the optical transmittance of diagnostic windows when they become covered with a layer of deposits or dust particles.**

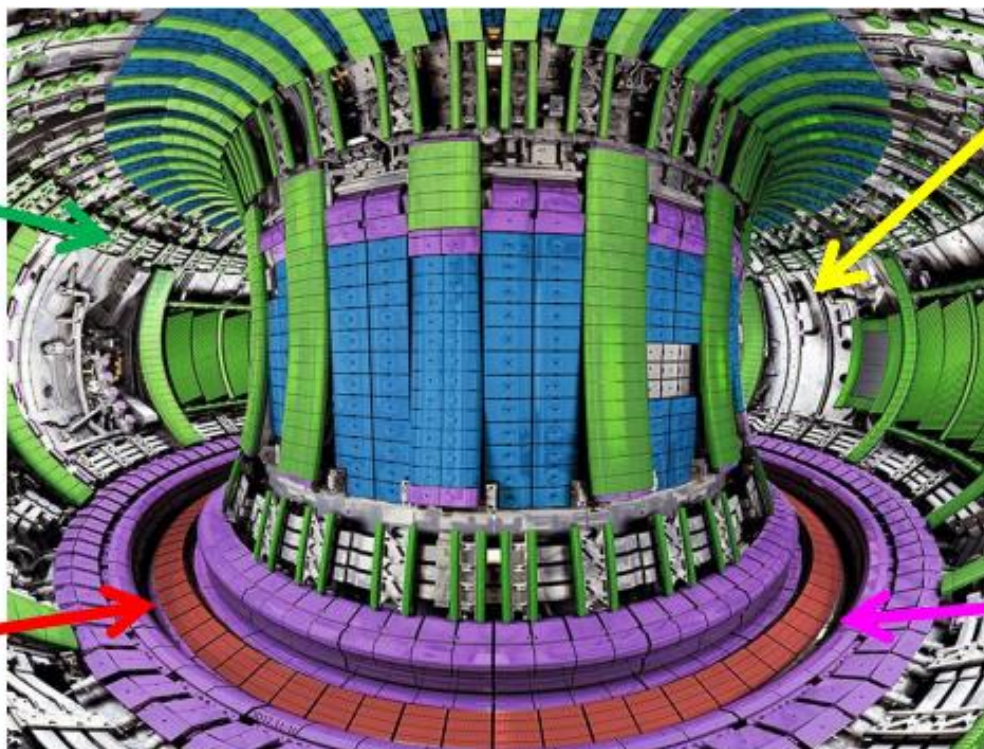
JET – ITER-like wall (ILW) vessel components



Bulk beryllium (Be)
Castellated structure



Bulk tungsten (W)
Lamellae structure



Incoel vacuum vessel

Tungsten (W) coating on
carbon-fibre composite
(CFC)



- Bulk Be PFCs
- Be- coated incoel PFCs
- Bulk W
- W- coated CFC PFCs

Due to courtesy of A. Widdowson



Operation of JET with the ITER-like wall (JET-ILW) composed of beryllium in the main chamber and tungsten in the divertor provides a unique opportunity for detailed studies of dust generation: quantity, morphology, location in the vessel, fuel inventory, etc.

Dust analysis programme on JET-ILW



Large international effort - EUROfusionWork Programme & EU-Japan Broader Approach (12 EU labs and the International Fusion Energy Centre at Rokkasho)

- Cycle of plasma operation (~20 hours) followed by intervention to remove tiles/probes for analysis,
- Thousands of measured and analysed points,
- Results provide data on: dust sources, location, quantity, morphology, composition and fuel retention, removal and collection methods, behaviour in plasma, predictions of dust generation and behaviour in reactor.

Types/Locations	Methods
Vacuum cleaning ILW - all	Optical microscopy
Sticky pads: Tiles 0, 1	SEM
Sticky pads: Tile 5	FIB
Sticky pads: Tile 8	TEM, STEM
Sticky pads: Tiles B, C	EDX, WDX, EPMA
Sticky pads: Be IWGL	μ XRF/XCT
Dust monitors: ILW-2 and 3	μ -NRA
Mirrors (FMT)	μ -PIXE
KY-6 Mirror ILW-1 and ILW-2	μ -RBS
Spatial blocks	HIERDA (dust monitors)
QMB crystals	ICP-OES
	TDS: H, D, T
	Full combustion: T
	Radiography: T
	Cameras (in discharges)

Detailed studies require a broad range of techniques and a network of specialized laboratories with ability and expertise in:

- (i) *analysing of a broad spectrum of species, from hydrogen isotopes to tungsten;*
- (ii) *handling tritium and beryllium-contaminated materials.*

Dust survey and collection methods



- *High-resolution photography of PFC at shut-downs (most comprehensive record of wall modification by plasma-wall interactions, identification of droplets/splashes);*
- *Remotely controlled vacuum cleaning of the divertor,*
- *Local sampling of loosely bound matter from PFCs (plasma-facing components, PFC) on microscopic tables with carbon sticky pads,*
- *Collection of mobilized dust on various erosion-deposition probes located in the divertor and the main chamber: dust monitors (Si plates), test mirrors, spatial blocks, and covers of quartz microbalances (QMB) devices.*

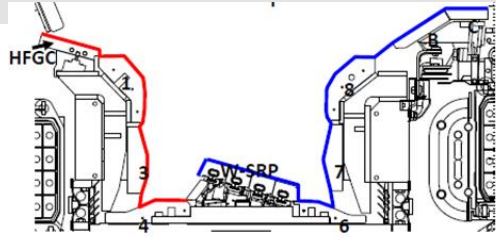
The aim was to have a complete overview of dust sources, location, and quantity, not just a number (even large) of analysis points and isolated findings.

Examples illustrate key findings resulting from W dust studies in JET-ILW.

Dust Collection: *Facts, Advantages, Drawbacks*



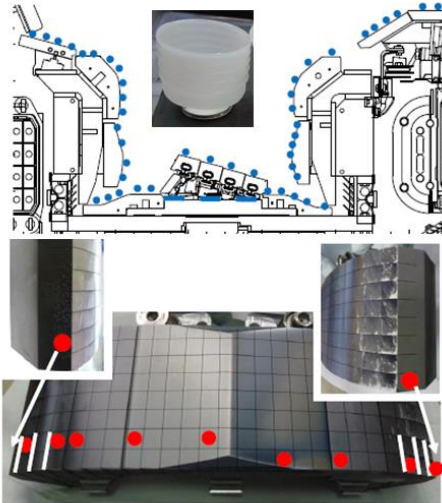
Vacuum cleaning: 360 °



Messages:

- Around 1 g after each campaign.
- Total amount of loosely bound matter in the divertor.
- Sampling for TDS (the only option)
- Composition: a mix of all particles.
- Possible disintegration of particles.
- Not possible to associate dust morphology with place.

Local sampling: sticky pads



Messages:

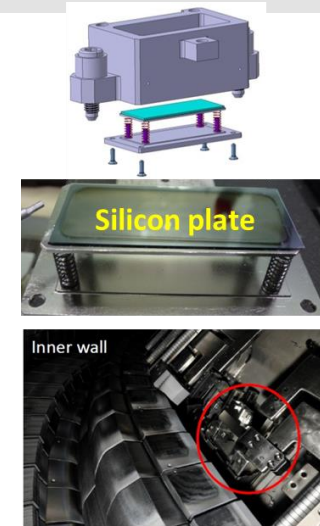
- Fairly small amount sampled.
- Good adherence of particles to sample.

Advantage: Detailed local analyses.

Drawbacks:

- The top layer of samples represents the bottom of deposits.
- Force is applied during the collection; more than just dust may be collected.

Dust monitors



Message:

Tiny amount collected.

Advantages:

- Collection of undisturbed mobilized particles.
- Precise analyses.

Drawbacks:

Bigger particles may be lost in the transportation of monitors from the torus to BeHF and then to analytical equipment.

Dust examination

fuel content



Fuel content measurements of individual particles

The distribution and content of D in the collected particles (Fazinic et al. 2020 Nucl. Fusion 60 126031 and Materials 2022, 15, 8353), and especially the relative fuel concentration in individual metal and carbon particles was determined to be:

- *D / C about 0.2;*
- *D / Be below 0.04;*
- *D / W at the detection limit*

Fuel retention is mainly related to the presence of carbon and is closely related to the type of particles.

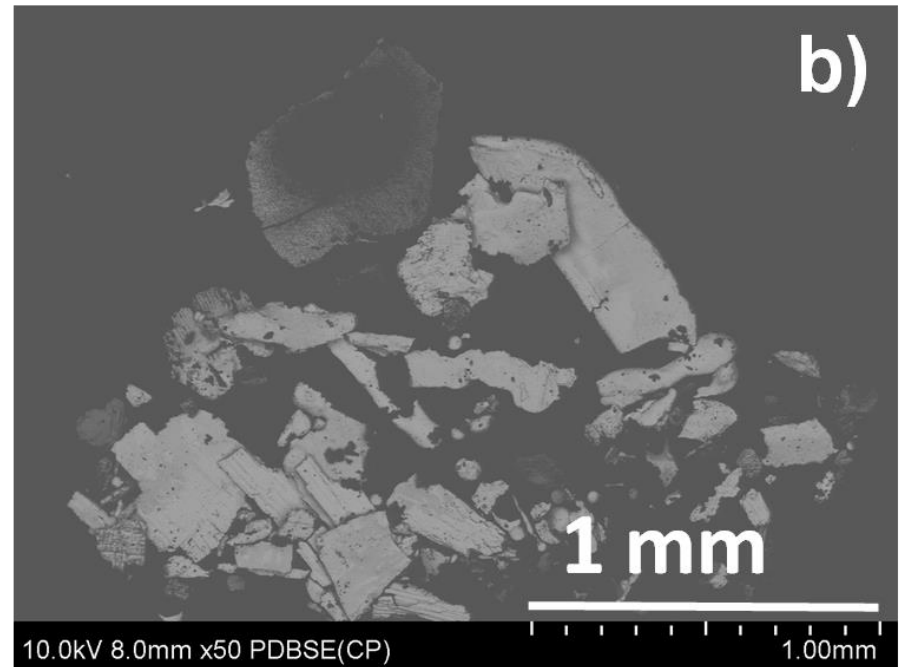
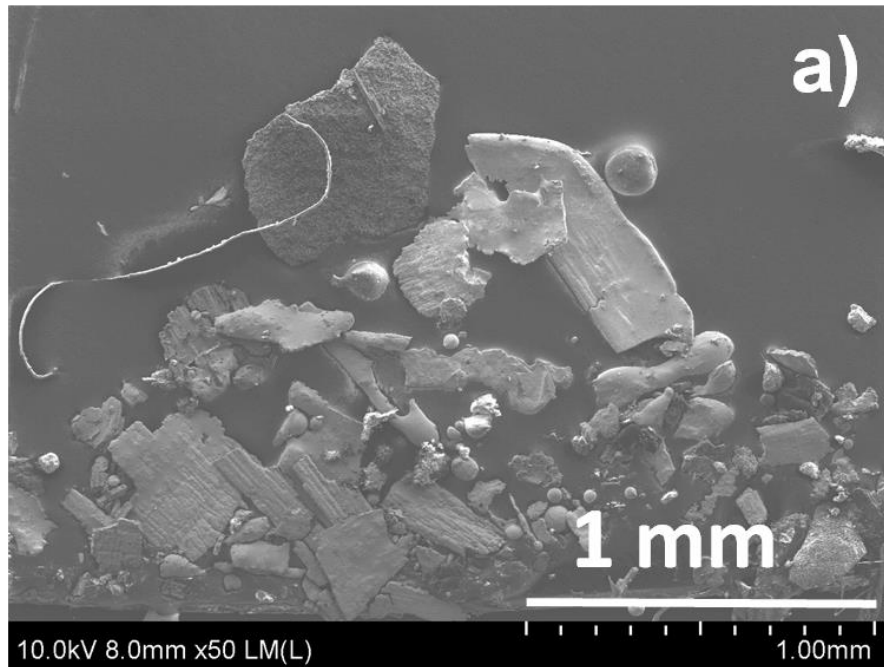
Similar results were obtained in T. Otsuka et al. works (tritium imaging plate technique), where radiographic studies have shown that the tritium content is 100 times higher in carbon-containing particles than in those whose composition was dominated by Be or W. (Nucl. Mater. Energy 17 2018 27 and Phys. Scr. 97 2022 024008).

Fuel content measurements of dust powder collected by vacuum cleaning

The retention of hydrogen isotopes was evaluated by thermal desorption spectrometry (TDS) measurements. 1.2×10^{21} D atoms/g dust ; <1 % of global fuel retention.

Morphology and chemical composition studies

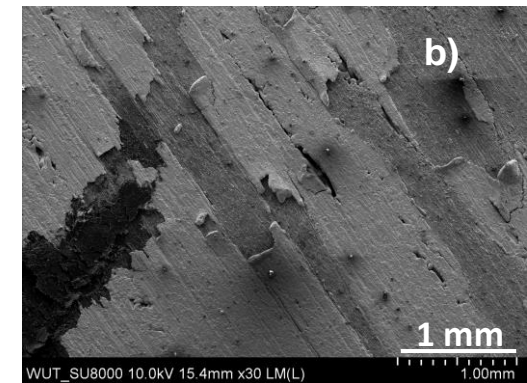
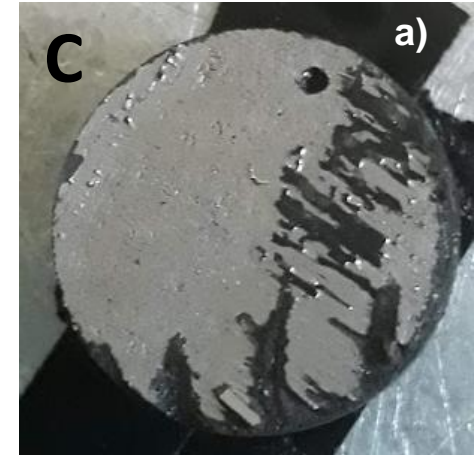
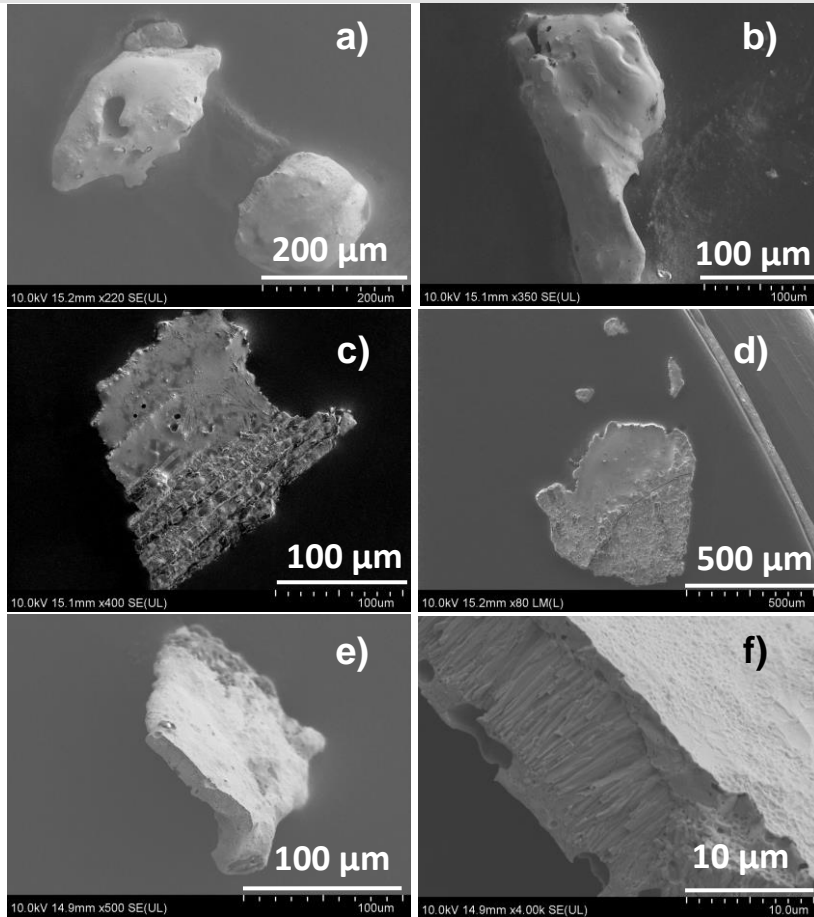
Dust collected from the outer divertor after ILW-3 by VC



- ✓ *The largest group of particles are tungsten ones. They are visible in the image recorded in BSE mode in white.*
- ✓ *Most are small fragments of tungsten coatings deposited on CFC tiles (size: 60 μm - 1.3 mm).*
- ✓ *Some of the flakes are more than likely from B and C tiles, where damage to the tungsten layer was found (most likely caused by the ICRF antenna).*

Morphology and chemical composition studies

Dust collected from outer divertor after ILW-3



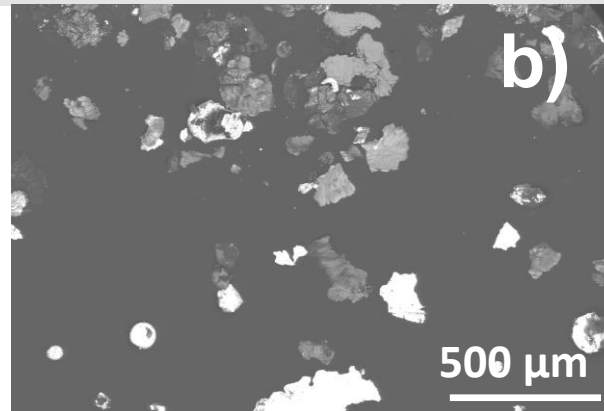
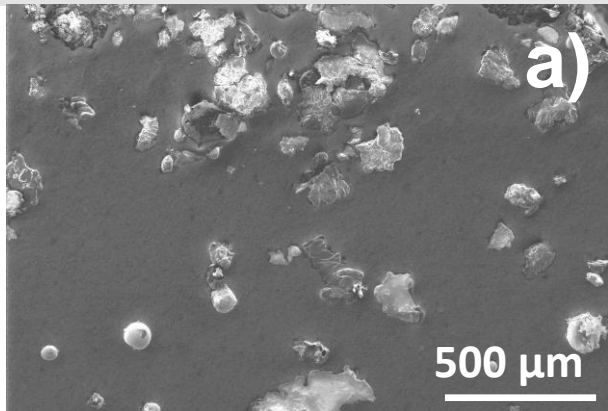
SEM images of W particles collected by VC from the outer divertor.

Images of:
a) the core sample cut out from the C tile ($\phi = 17 \text{ mm}$) and
b) its morphology.

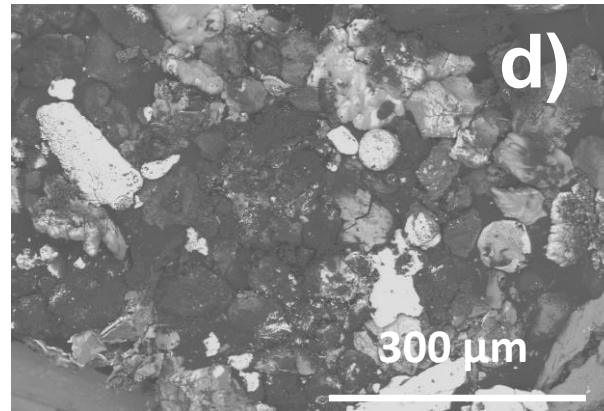
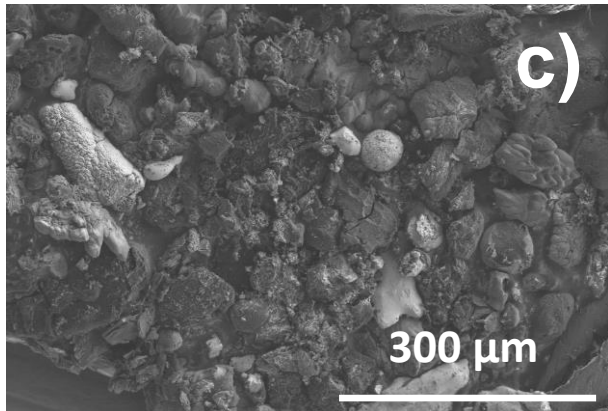
✓ *Tungsten dust occurs mainly as partially molten flakes originating from the W-coated tiles.*

Morphology and chemical composition studies

Dust collected from the inner divertor after ILW-3 by VC



SEM images of particles from the inner divertor.

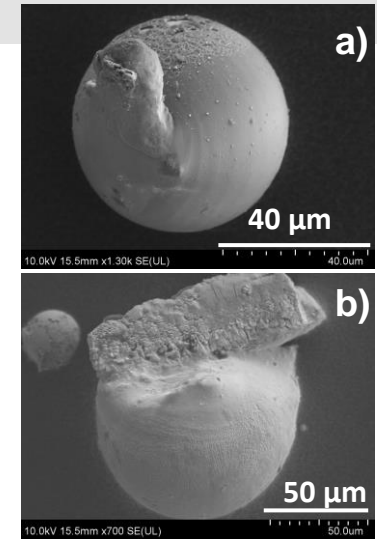
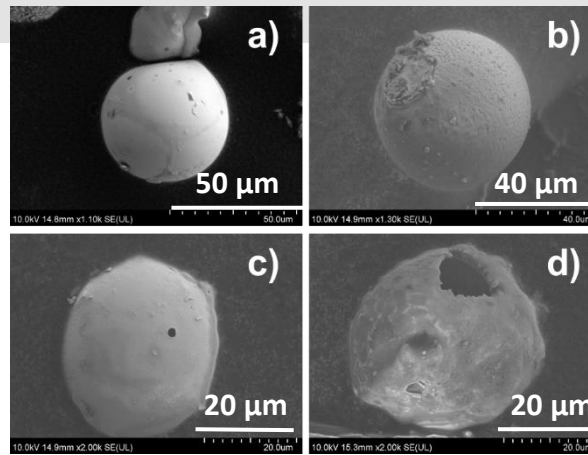
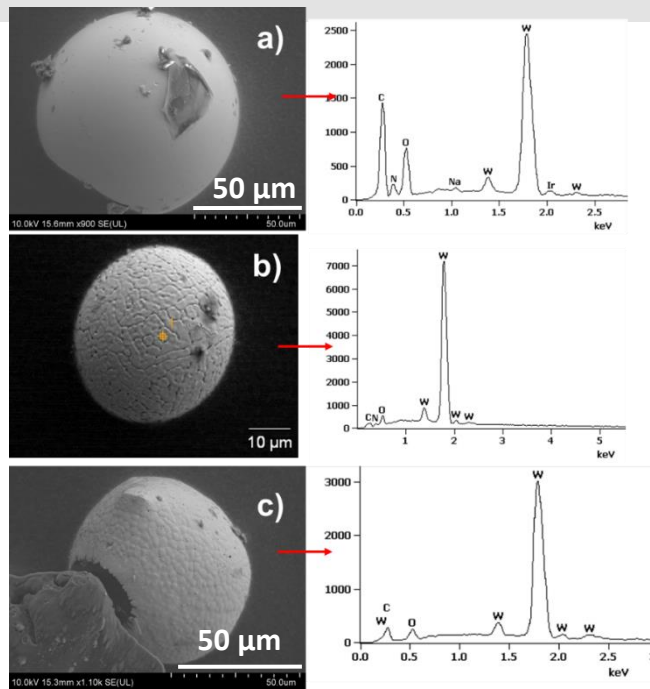


- ✓ A variety of dust particles ranging in size from tens to hundreds of micrometers.
- ✓ Particles of different morphologies (flakes, spherical particles, and irregular ones).
- ✓ There are both particles composed of heavy elements (W, Ni) and light particles, mainly beryllium deposits.

Morphology and chemical composition studies

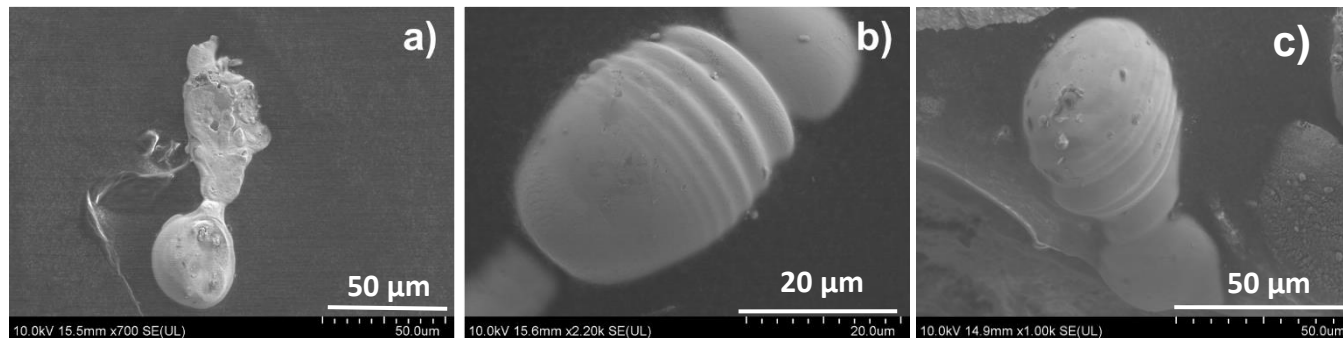


Dust collected from divertor after ILW-3



Particles with a protrusion that is a trace of the place where they were detached.

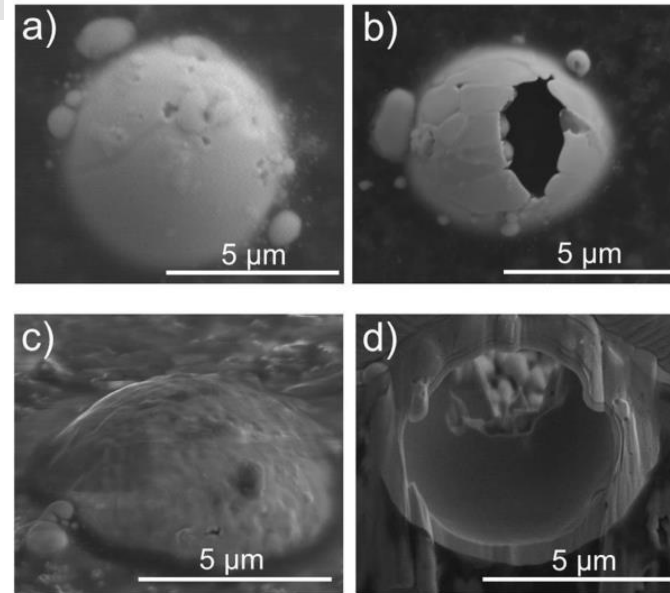
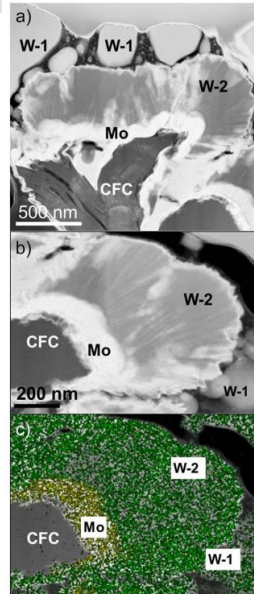
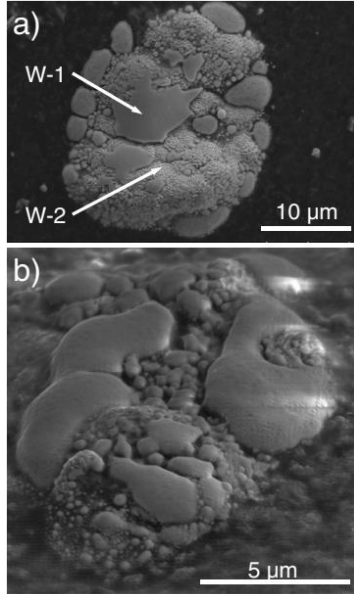
SEM images of W spherical particles found on Si dust monitors: bulk and with ball-like structures.



Morphology and chemical composition studies



Dust collected on sticky pads from Tile1 after ILW-1



Tungsten-particle originating from W coatings: W-Mo particle with marked areas of molten W-1 and original W-2 coating (a) and a mixed W-Mo-C particle (b).

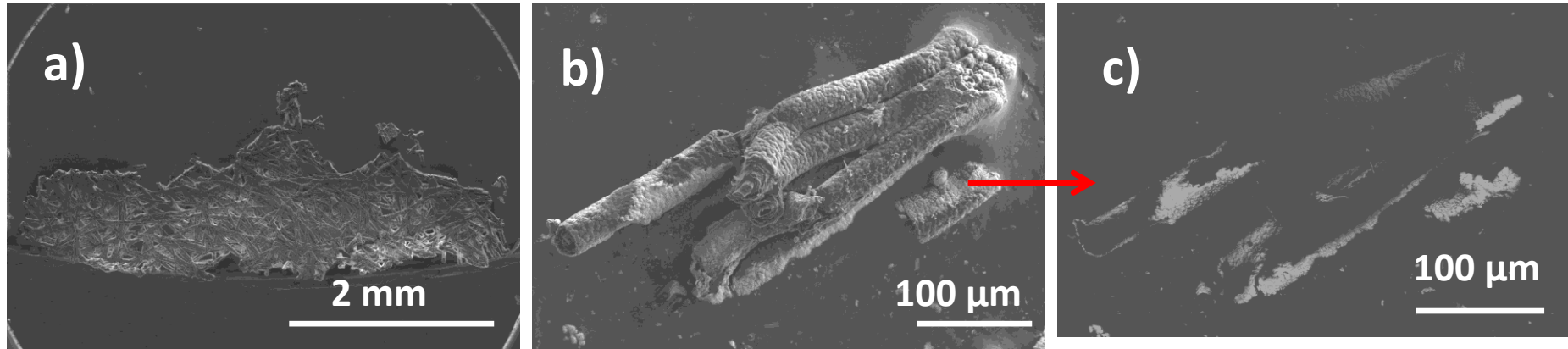
The internal structure and composition of the particle shown in Fig. 1a.

Tungsten particles : (b) broken shell revealing the empty interior (c); particle selected for cross-sectional FIB studies, (d) the FIB-made cross-section proving the ball-like particle.

A. Baron-Wiechec et al 2015 Nucl. Fusion 55 113033

Dust collected by sticky carbon pads

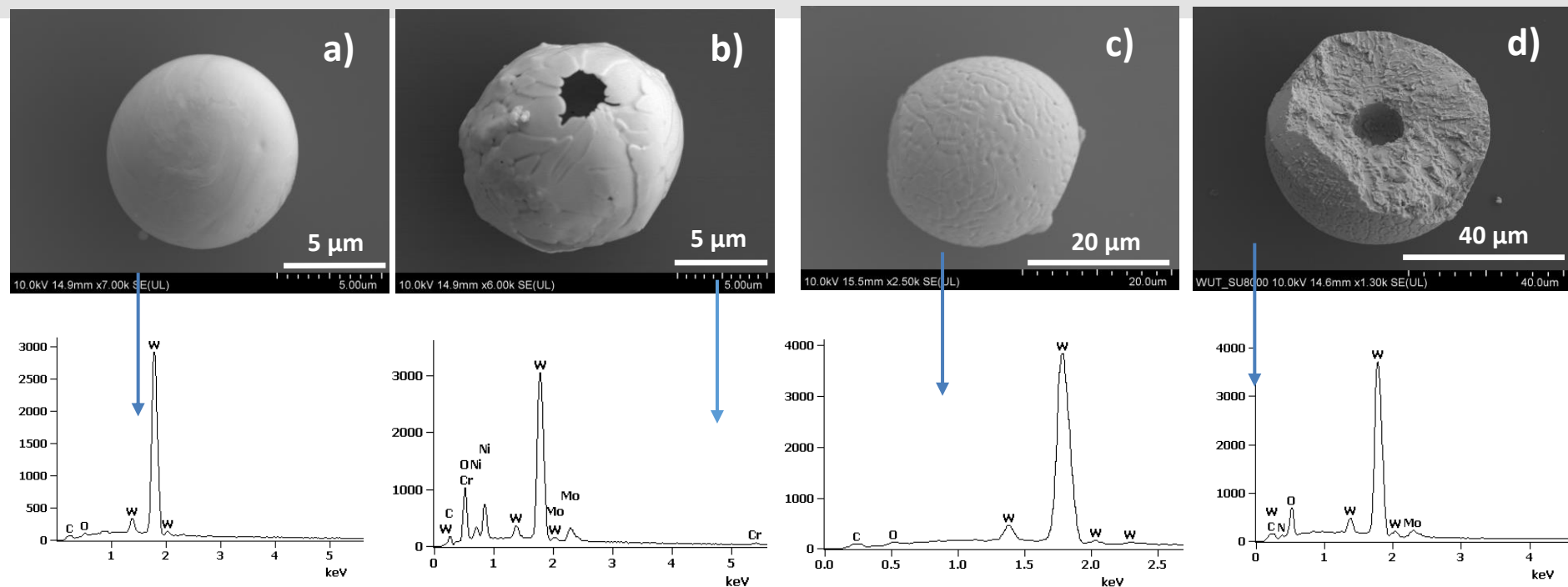
flakes W coating deposited on CFC divertor tiles



SEM image of a tungsten coating with a fragment of CFC composites, Tile 0.

In the images, we see the underside of the particle. Its upper part is embedded in the tape.

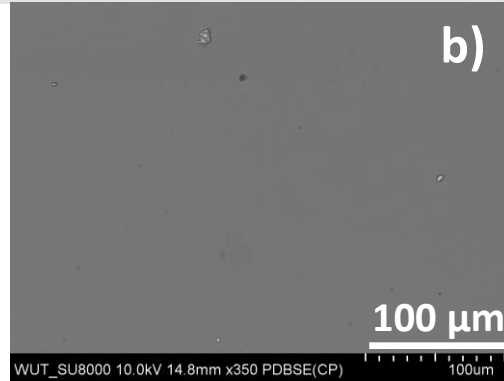
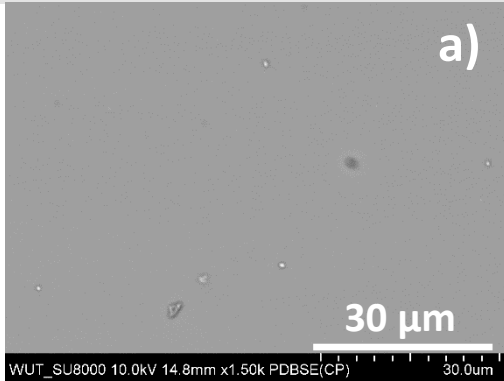
W dust on Si dust collectors



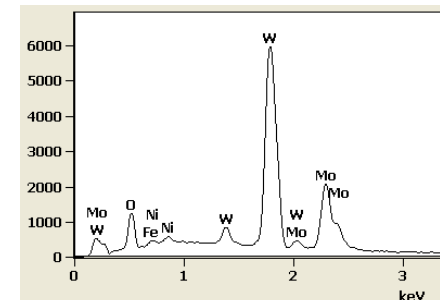
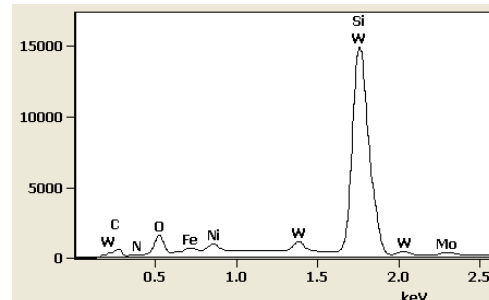
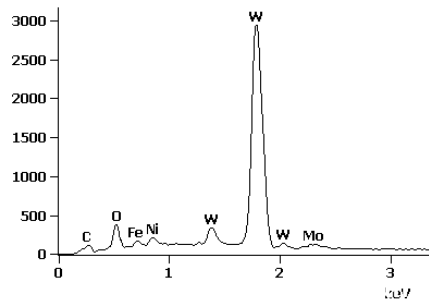
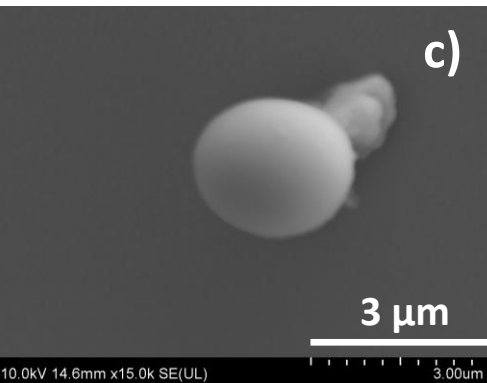
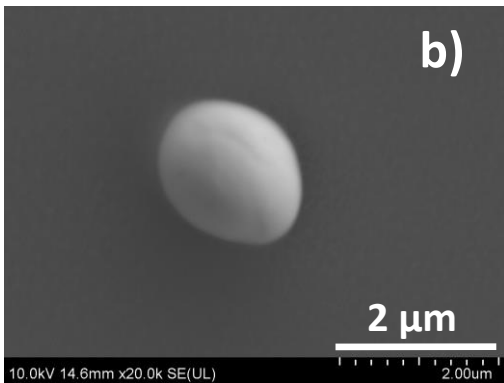
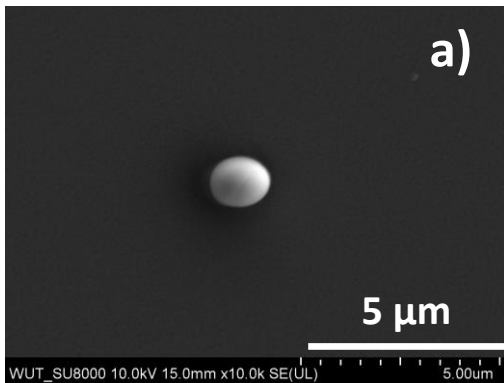
SEM images of W spherical particles together with corresponding EDS spectra: (a-b) OU6 outer and (c-d) IN6 inner divertor silicon dust collectors.

- Size: 3-55 μm.
- In the registered spectra of chemical composition, in addition to tungsten, there were also lines originating from molybdenum and carbon, which indicates the source of these particles - tungsten coatings.
- Particles differ in surface morphology. We observe particles with a relatively smooth surface, with a dendritic structure and „ball-like” shell structure, empty inside.

W dust on Si dust collectors



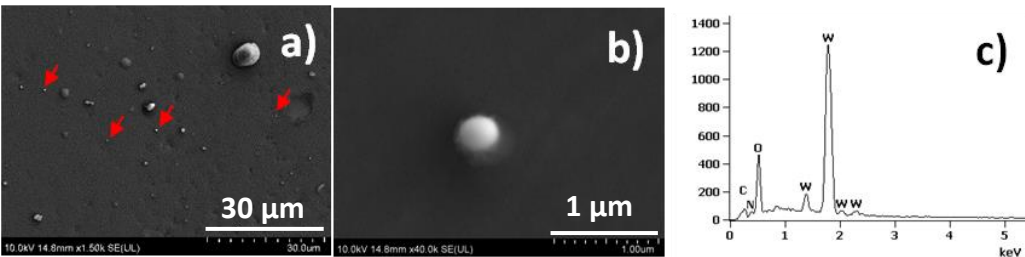
SEM image of tiny W and Ni particles (visible as white dots), BSE mode.



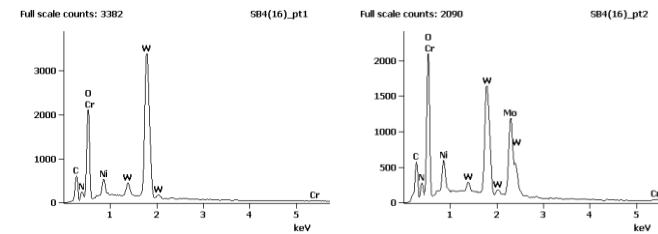
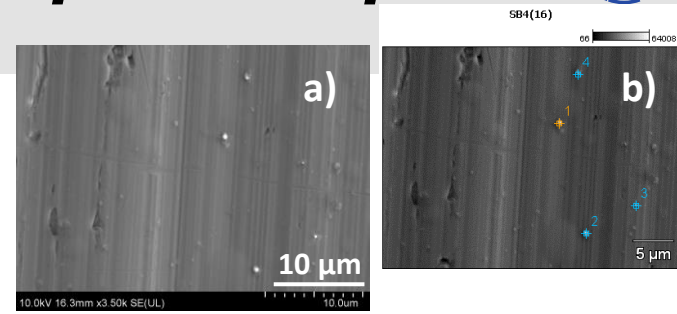
SEM images of tiny W spherical particles together with corresponding EDS spectra.

Size: submicron-3 μm.

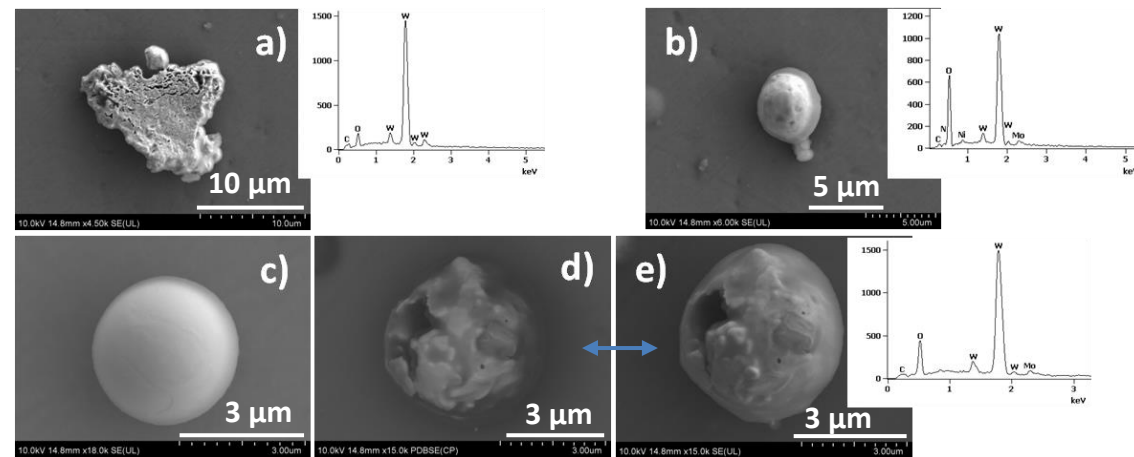
W particles at various erosion-deposition probes



SEM image of tiny W and Ni particles, mirror no 117.



SEM image of tiny W particles, SP4.



SEM image of W particles mirror no 117.

Analyses of dust in JET2

Network of laboratories



From global amount to finest details

CCFE:

- *Collection of particles*
- *Mass determination*
- *Tritium content (off gas)*



IFERC, Rokkasho, Japan

- *vacuumed dust from JET-C and ILW*
- *deposits on divertor tiles*
- *Tritium content (scintillography)*
- *Scanning microscopy*
- *Focused ion beam*
- *Transmission microscopy*
- *X-ray spectroscopy: EDX & WDX*
- *Thermal desorption*
- *X-ray diffraction (for divertor samples)*

IPPLM (locally sampled dust):

- *Focused ion beam*
- *Scanning and transmission microscopy*
- *X-ray spectroscopy: EDX and WDX*



RBI (locally sampled dust):

- *Micro-ion beam analysis:
NRA, PIXE, RBS*



IST, VR (dust monitors):

- *Ion beam analysis:
NRA, PIXE, RBS, HIERDA*

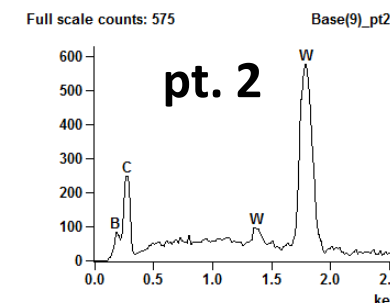
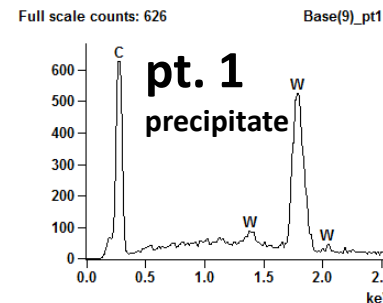
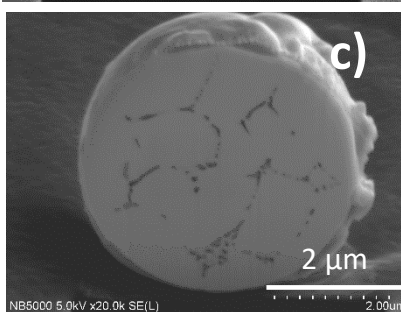
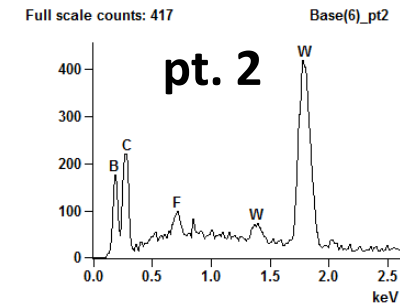
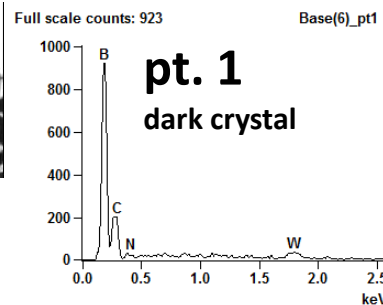
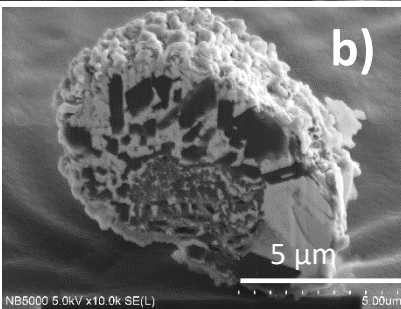
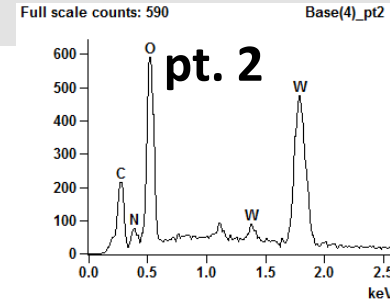
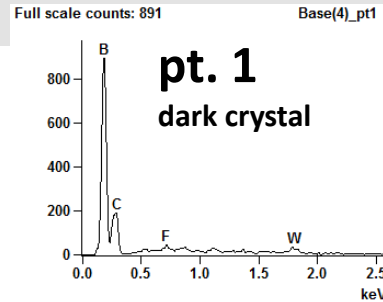
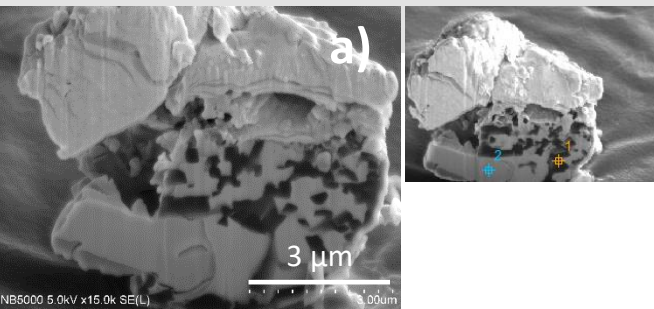
Dust analysis programme on AUG



During five consecutive operation campaigns (2007–2011), Si collectors were installed, which were supported by filtered vacuum sampling and collection with adhesive tapes in 2009.

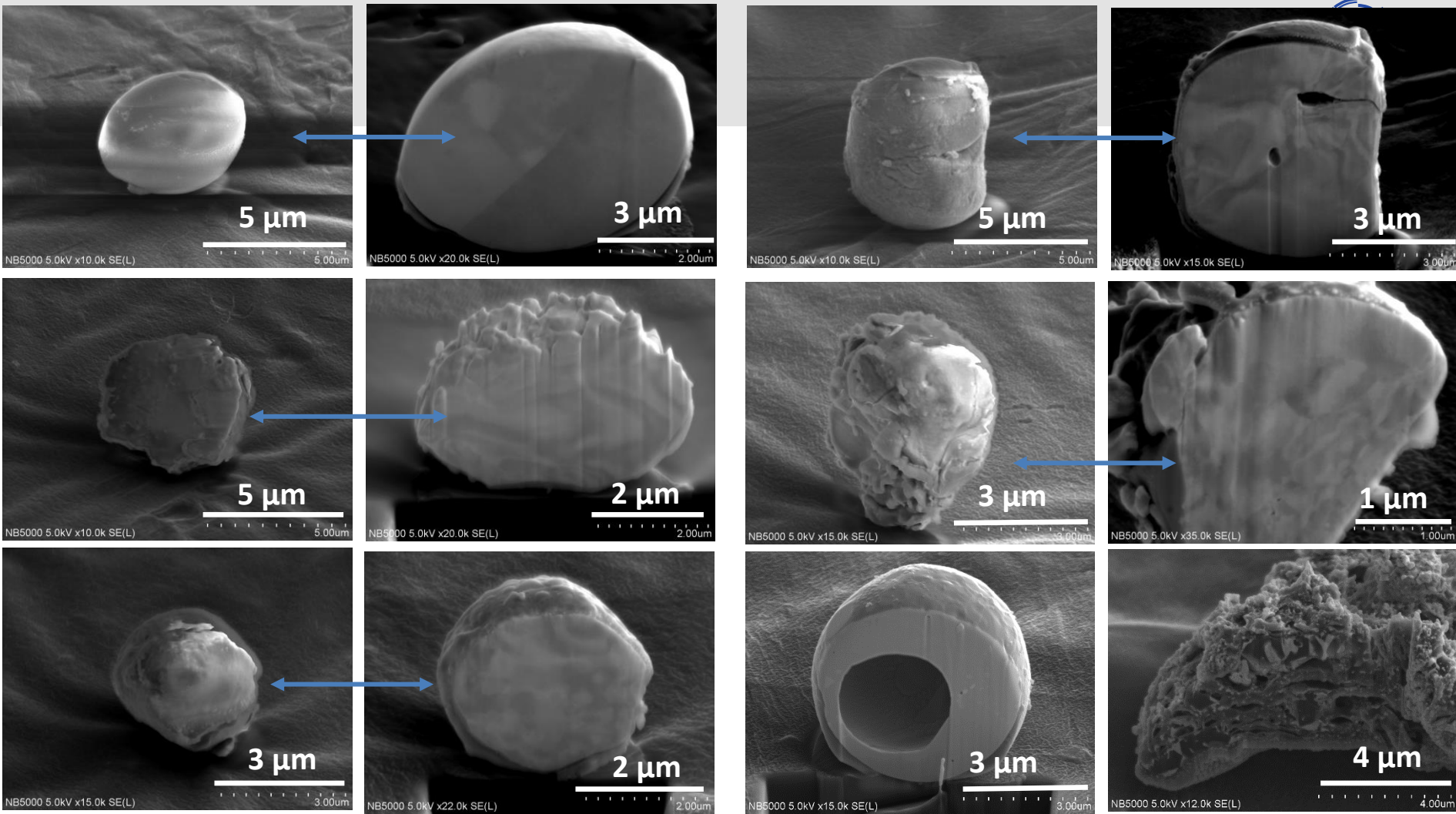
Summary given in: M. Balden et al. Nucl. Fusion 54 (2014) 073010

AUG dust internal structure



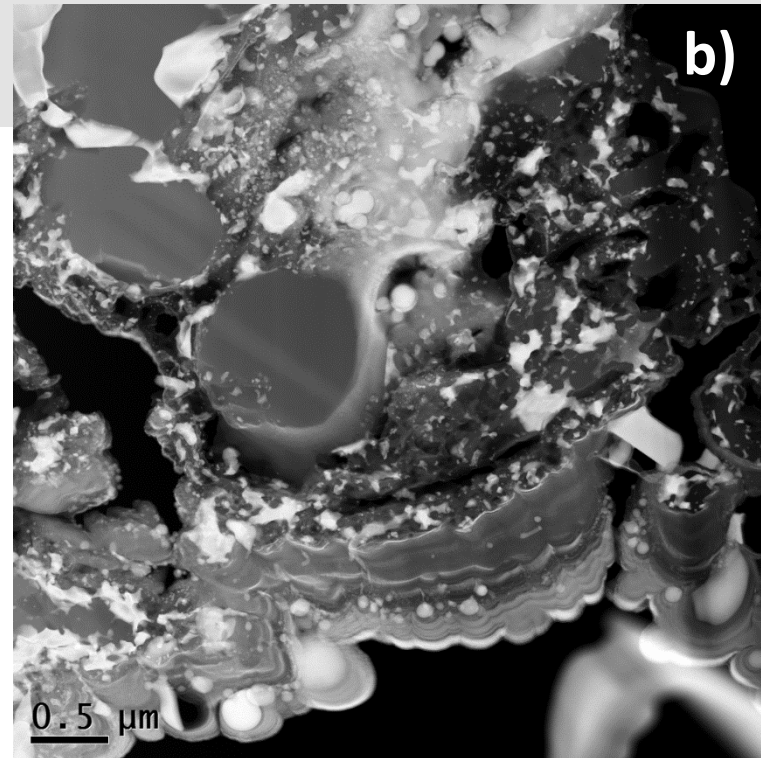
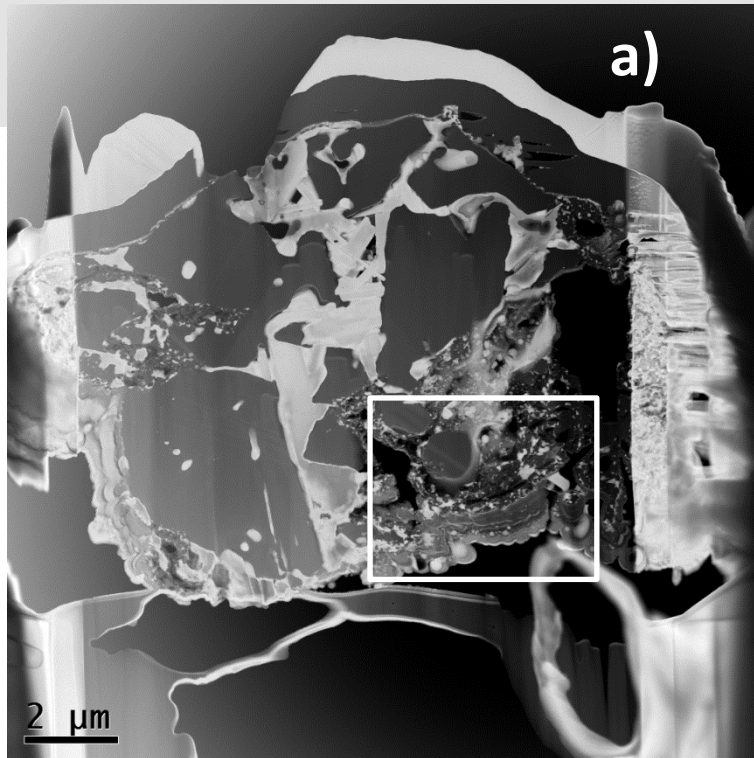
SEM images of dust particles cross-sections collected after the 2009 campaign using the filtered vacuum technique together with corresponding EDX spectra.

- ✓ 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.



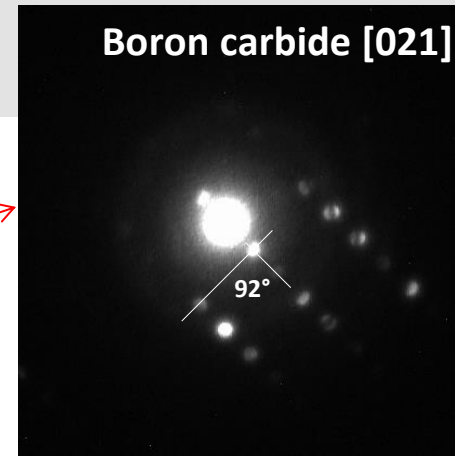
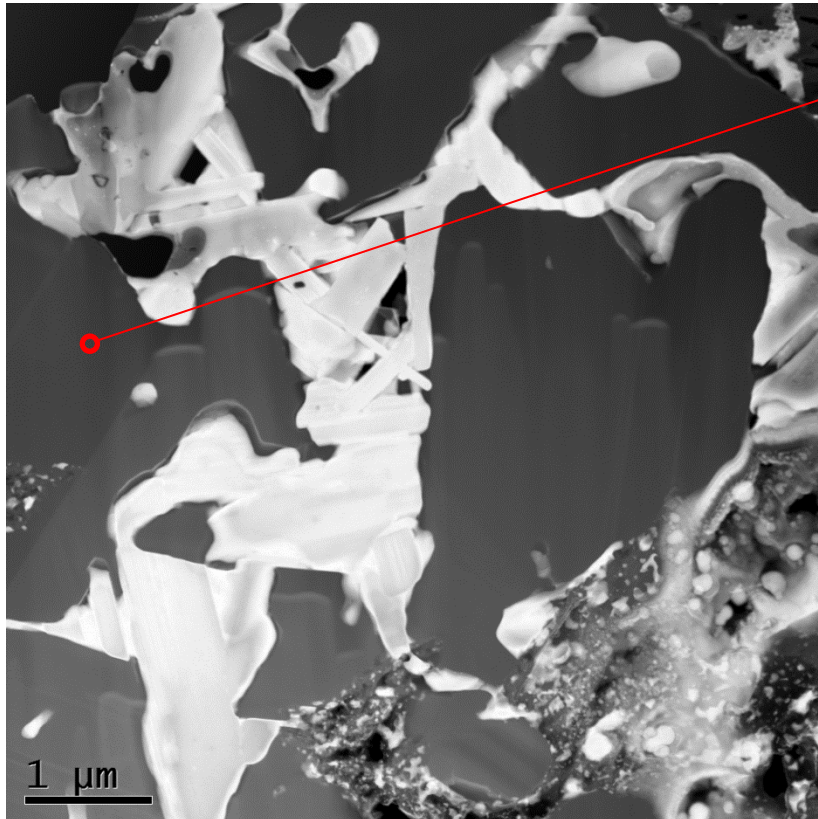
SEM images of AUG dust particles and their cross-sections.

Work done by M. Rasiński and J. Grzonka.



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.*

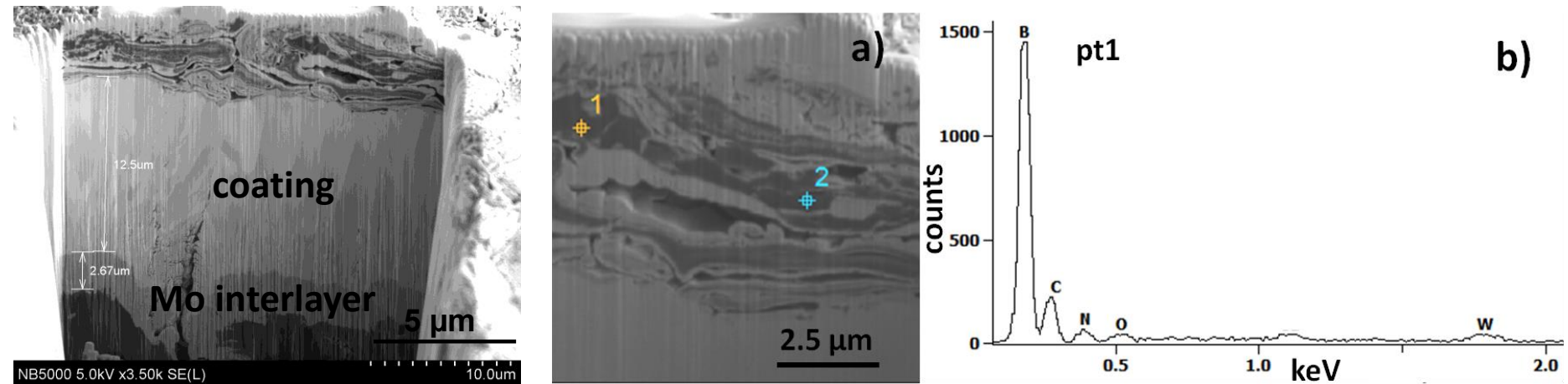


	Experimental data	Literature data
$d_{(21-2)}$	1.71 Å	1.76 Å
$d_{(1-12)}$	3.79 Å	3.81 Å
α	92°	92.32°

- ✓ *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

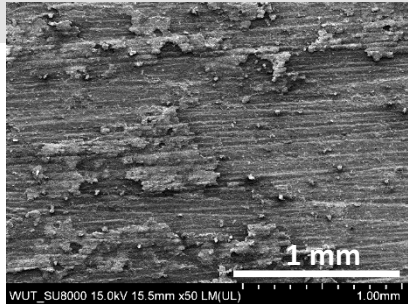
Morphology and chemical composition of AUG deposits



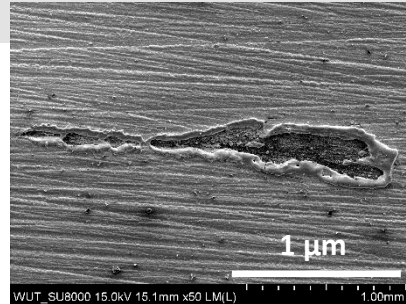
SEM images of the deposit present at the outer divertor strike point tile 1, private flux region (retrieved from the machine after 3 campaigns 2009-2013) cross-section together with corresponding EDX spectrum.

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

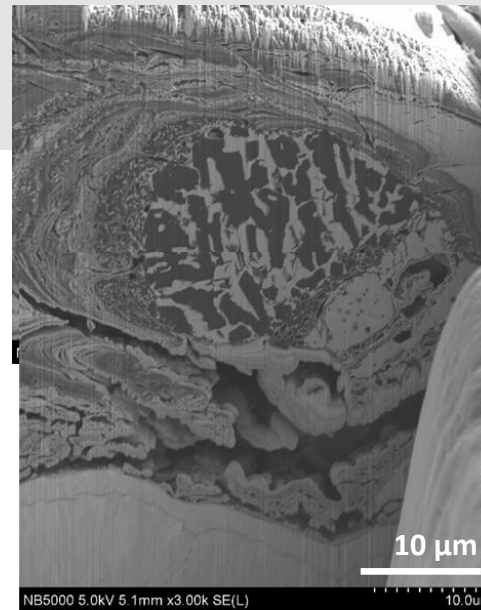
WEST marker tiles



SEM image of the C3 inner divertor sample.



SEM images of the arc track present at the surface, C3 campaign.



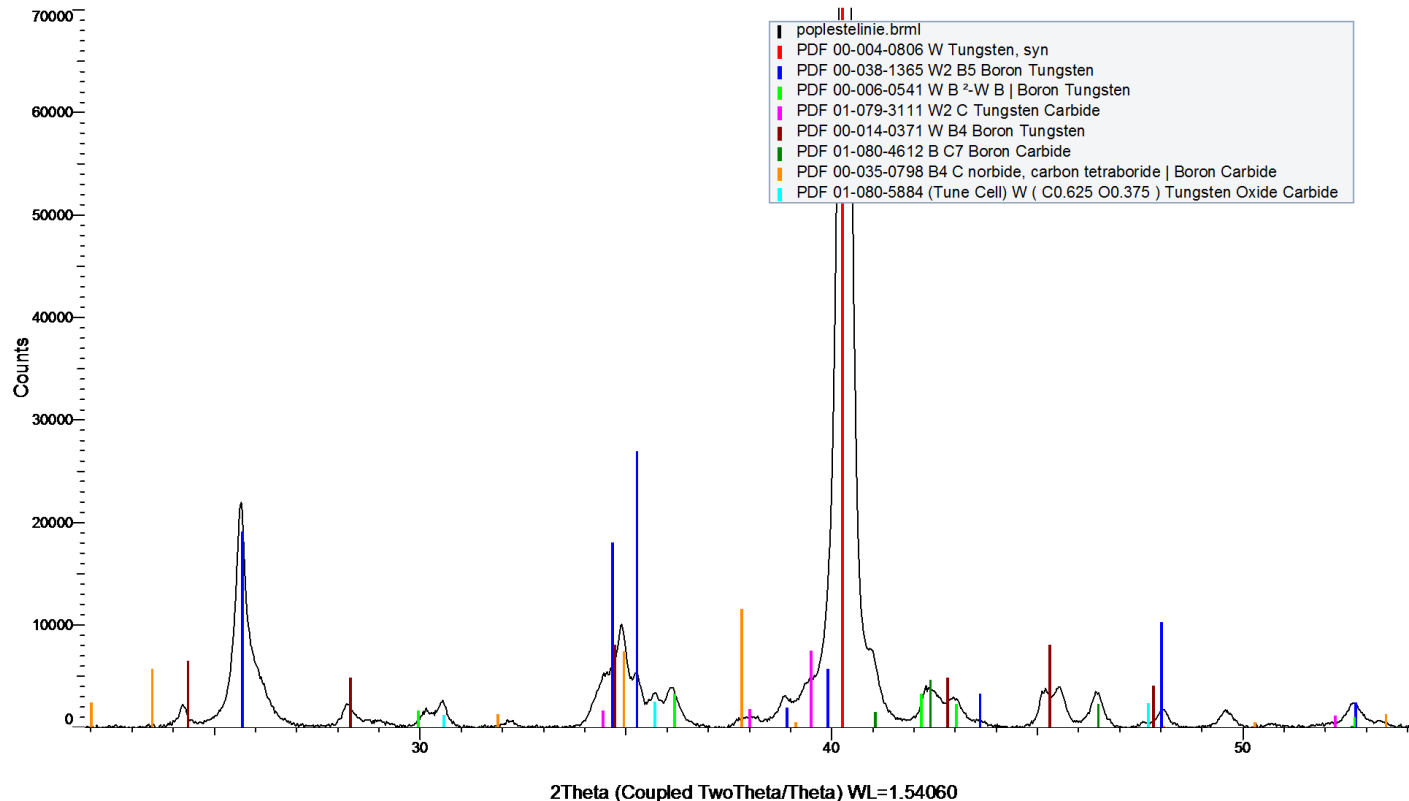
SEM images of the deposit and marker layer cross-sections, inner divertor, C5 campaign.

- ✓ *Co-deposits of similar morphology to those observed in AUG are found in WEST.*
- ✓ *In this machine, damage caused by arcing and co-deposited layers flaking and delamination is noticed as well.*

WEST samples



(Coupled TwoTheta/Theta)



XRD pattern, sample J, C4.

The main conclusion that can be drawn from the analysis of this spectrum is the presence of intermetallic phases from the W-B system, **tungsten borides**.

Concluding remark



The procedures for dust collection and sampling in JET and AUG, together with a broad range of analysis methods, constituted the most comprehensive approach to dust studies ever applied.