Surface Analysis Techniques for the Study of Plasma Wall Interactions

Nicolas Bekris

Karlsruhe Institute of Technology

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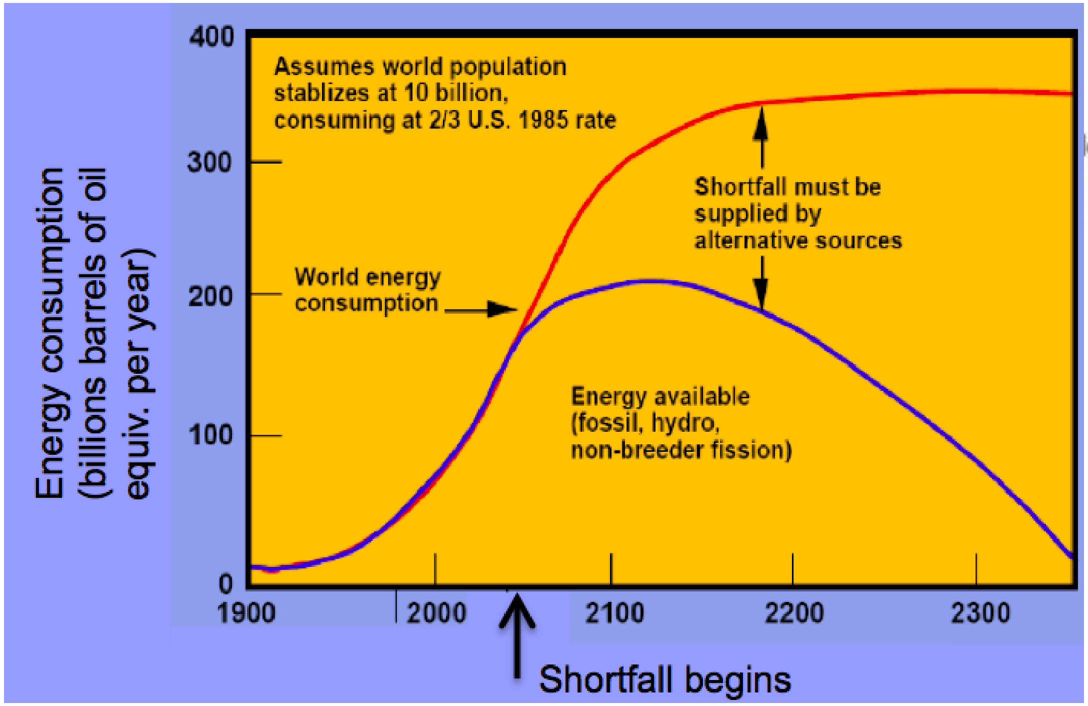
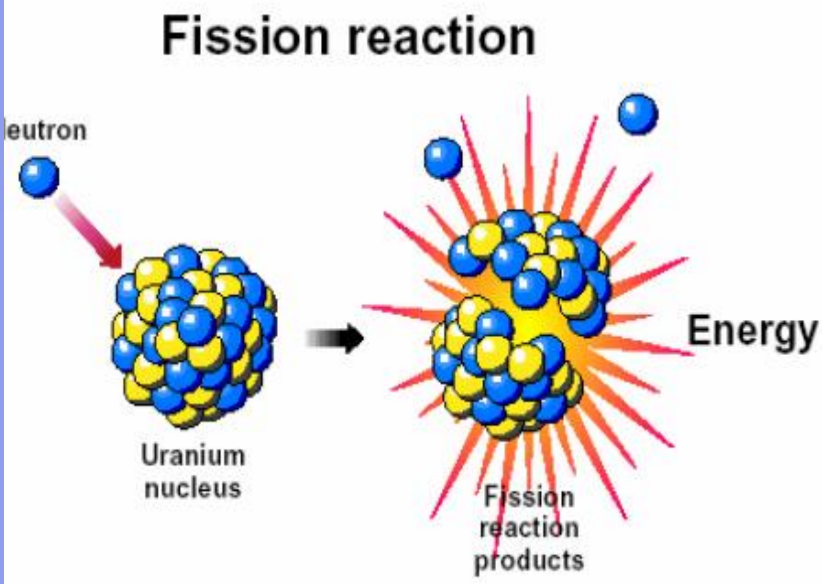
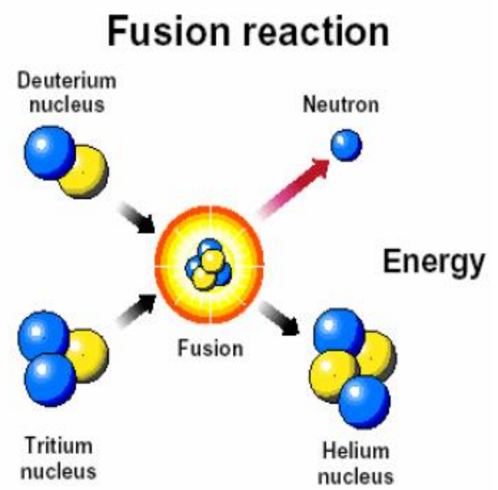
The views and opinions expressed herein do not necessarily reflect those of the European Commission.

Why Fusion?







Fusion reaction has the potential to provide energy in the future in a sustainable way exploiting reactions between hydrogen ions

Fusion Energy Research; Ions, Neutrons & Fusion Products

Examples of Fusion Reactions	Temperatures Needed
$^{2}H + ^{2}H \rightarrow ^{3}He + n + 3.2$ MeV	170 Million Degrees
$^{2}H + ^{2}H \rightarrow ^{3}H + ^{1}H + 4.0$ MeV	170 Million Degrees
$^{2}H + ^{3}H \rightarrow ^{4}He + n + 17.6$ MeV	80 Million Degrees
$^{2}H + ^{3}He \rightarrow ^{4}He + ^{1}H + 18.3$ MeV	400 Million Degrees

D-T is favoured: - Higher cross-section, Best yield at lowest temperature (ion energy)

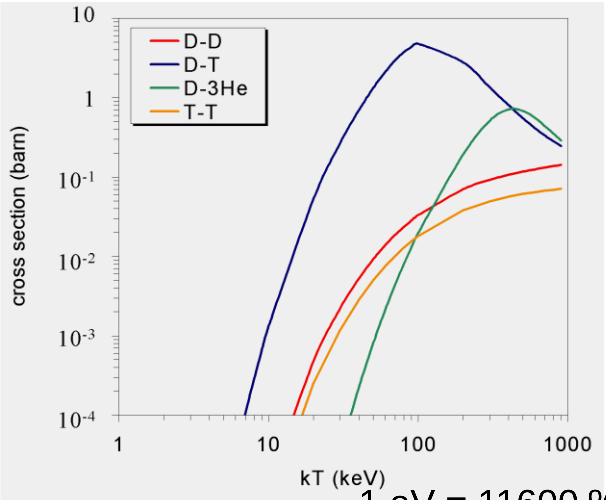
Need *containment* at high temperature and density

In the Sun – Gravitational containment
On Earth – Magnetic containment
– 'Tokamaks' like JET are now favoured

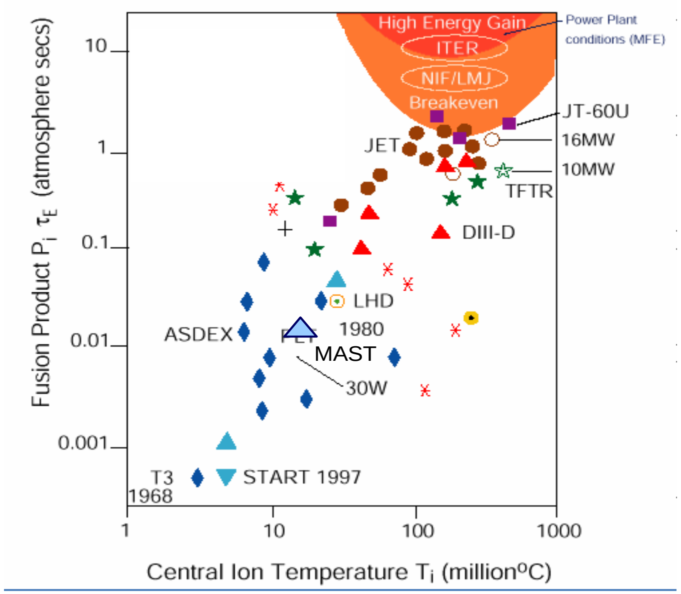
- **Maximise Fusion Product = ni.Ti.τe**

Arising points for Diagnostics

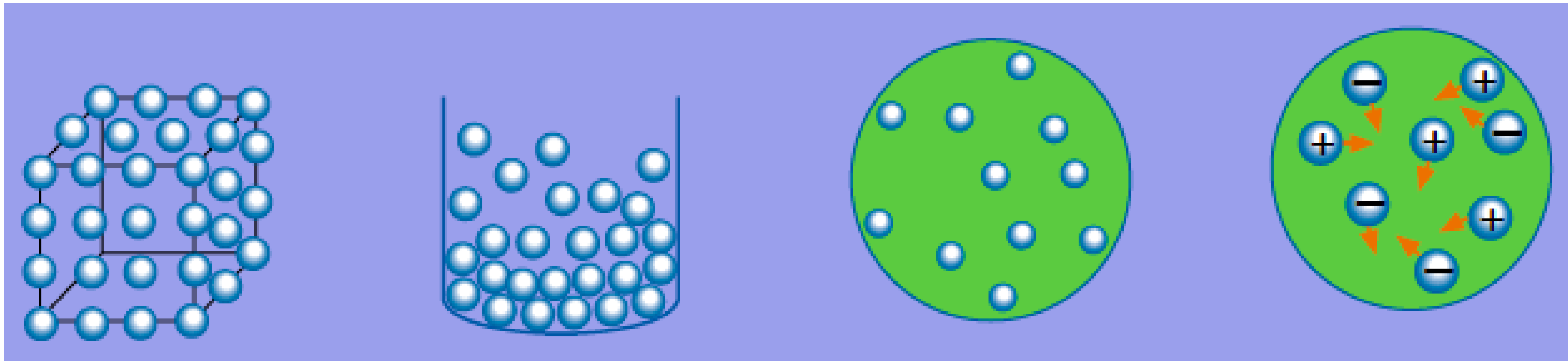
- Neutrons measure the fusion yield – directly
- Same number of p, ³T ions as n in a D,D plasma
- There are ³T ions + 14 MeV neutrons, even in D,D plasmas
  - From Triton 'burnup'
  - And tritium adsorbed in the walls



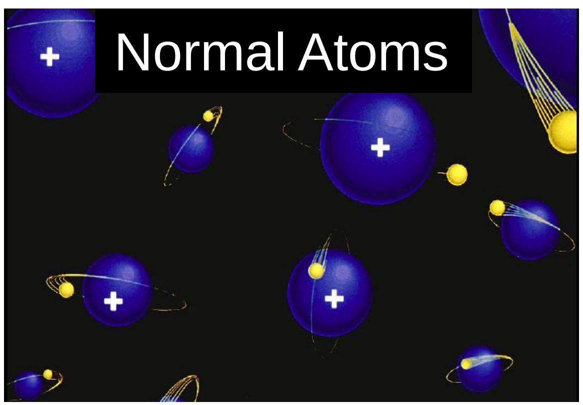
1 eV = 11600 ºC

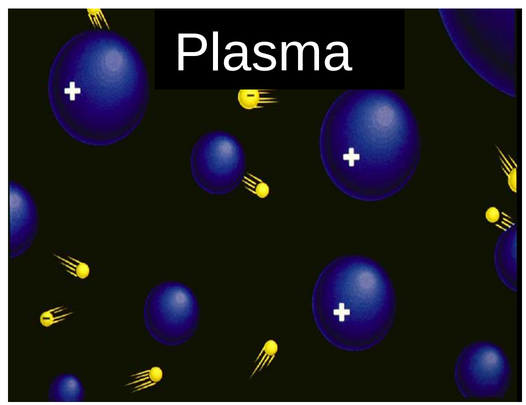


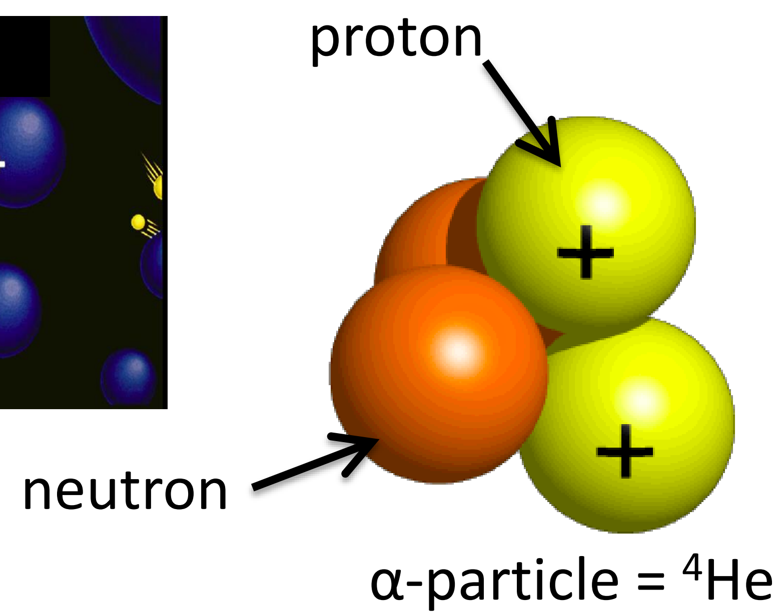
What is the Plasma?



Solid → Heat → **Liquid** → Heat → **Gas** → Heat → **Plasma**

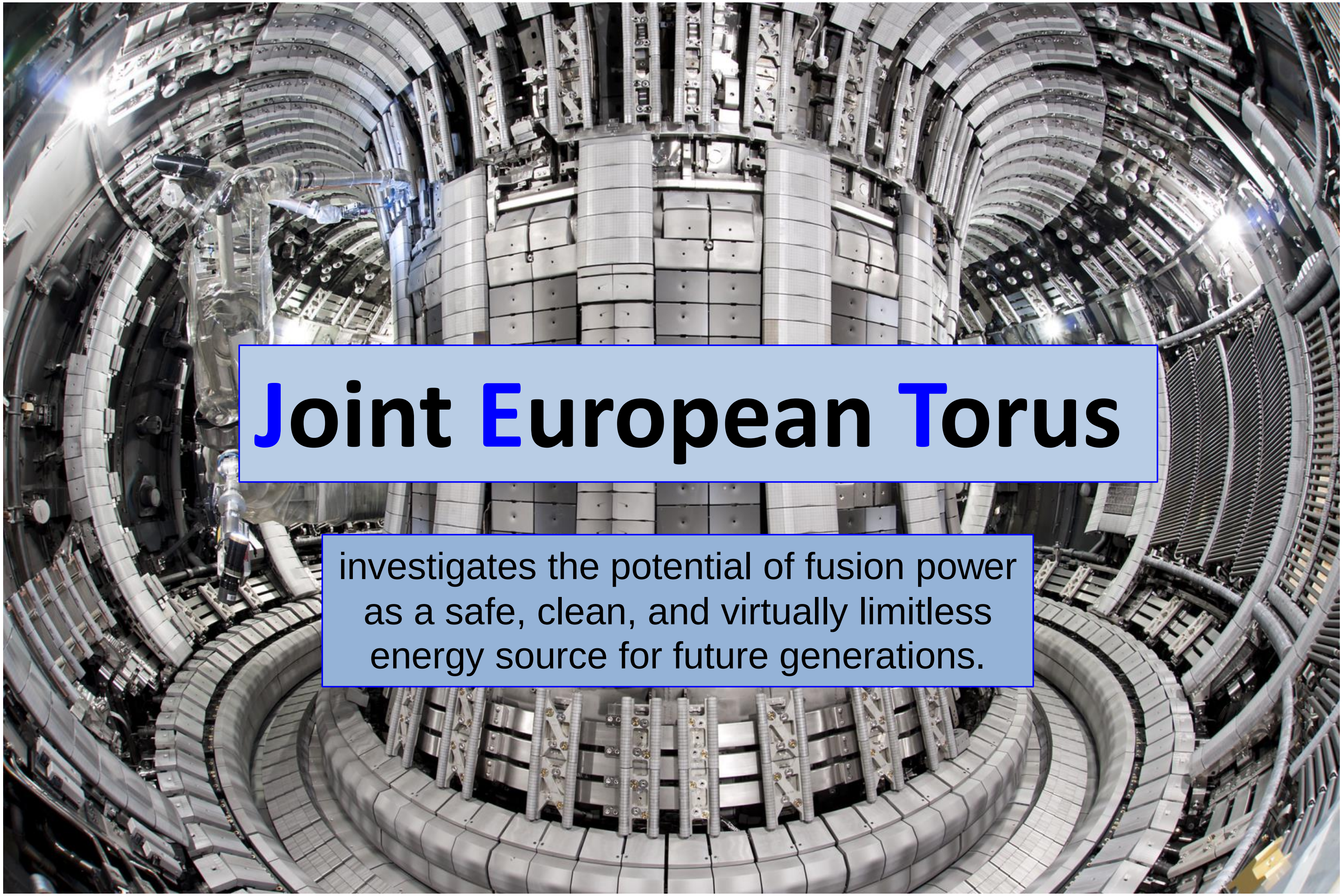






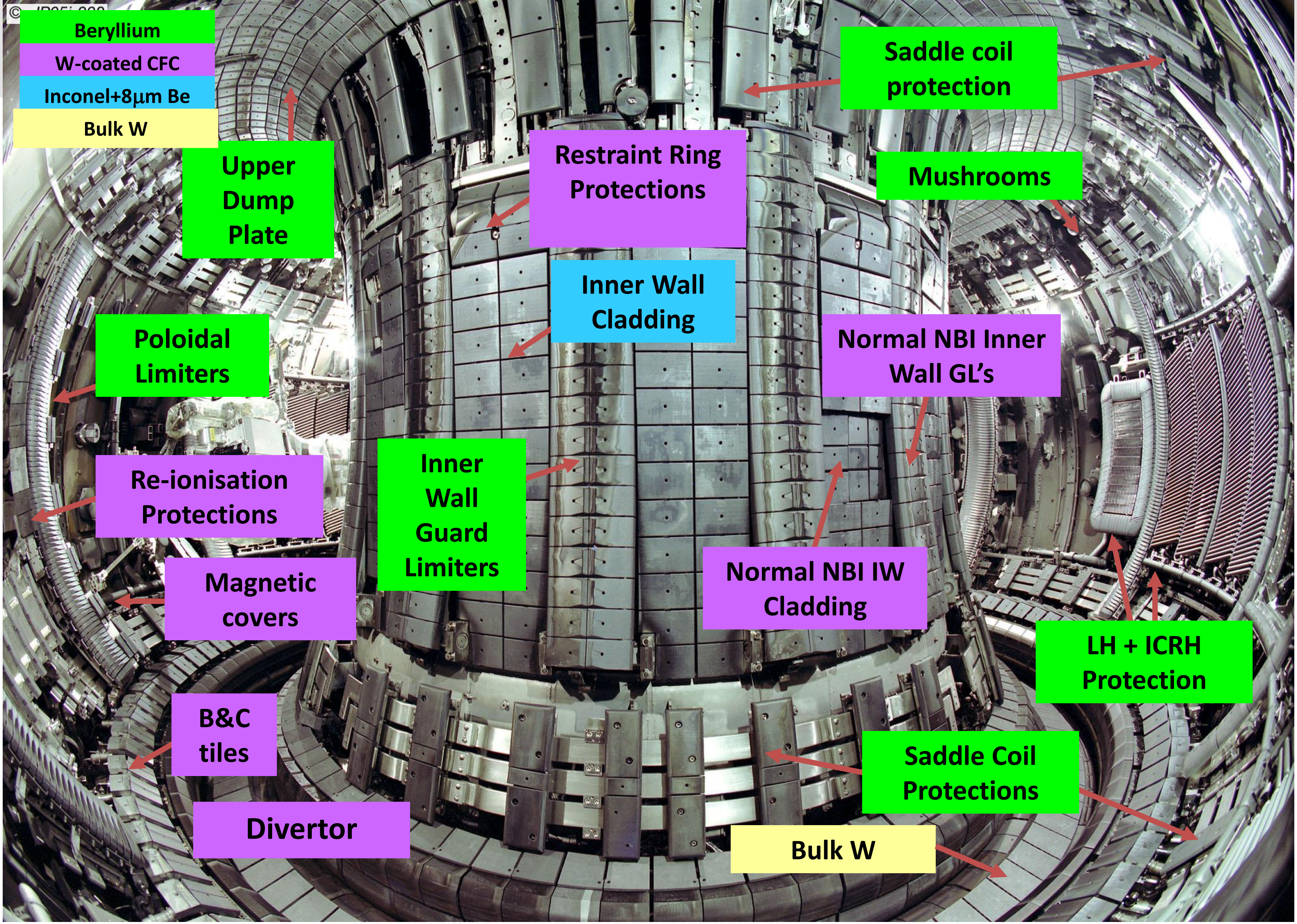
$^{2}H^{+}$, $^{3}H^{+}$, n, e^{-} and alphas

JET - The ITER-like Wall –May 2011

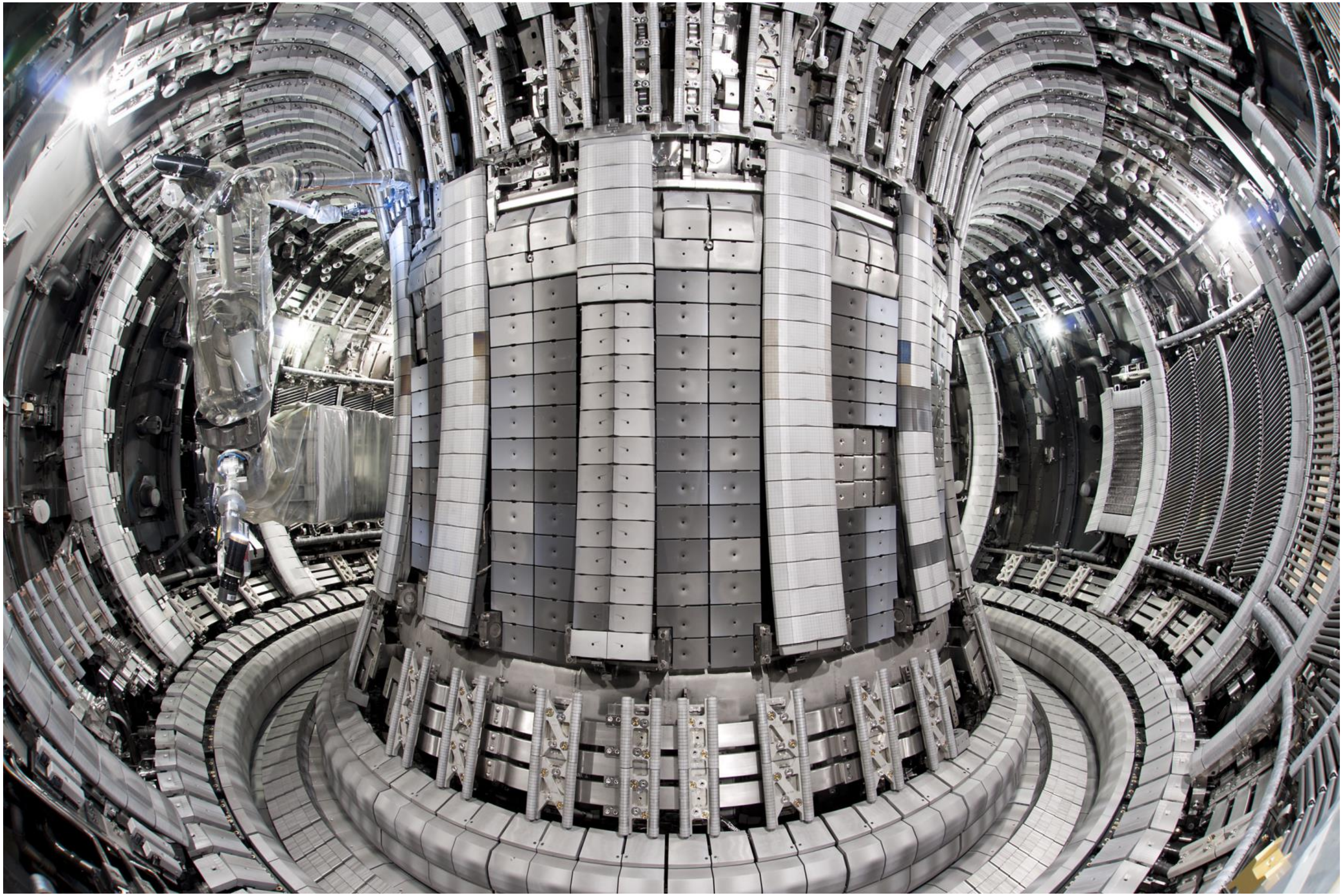


Joint European Torus

investigates the potential of fusion power as a safe, clean, and virtually limitless energy source for future generations.

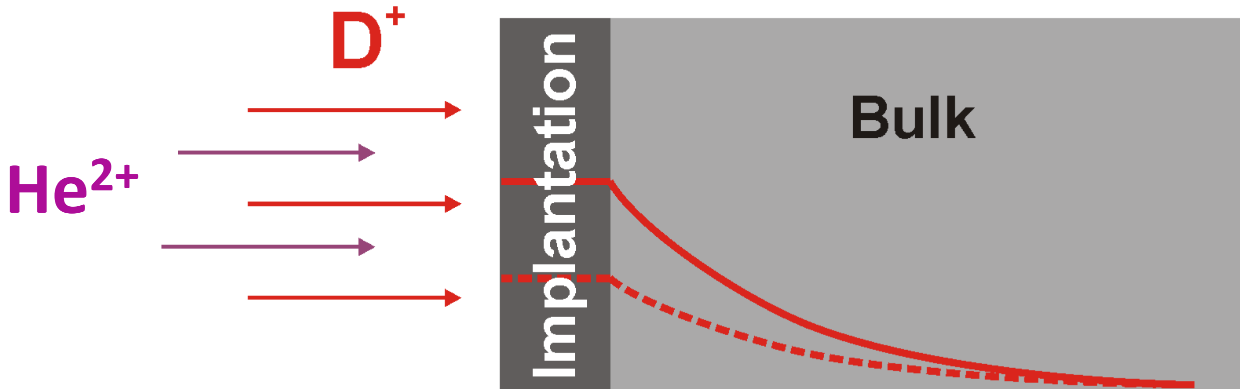

Beryllium
W-coated CFC
Inconel+8μm Be
Bulk W
Upper Dump Plate
Restraint Ring Protections
Saddle coil protection
Mushrooms
Inner Wall Cladding
Poloidal Limiters
Normal NBI Inner Wall GL's
Re-ionisation Protections
Inner Wall Guard Limiters
Magnetic covers
Normal NBI IW Cladding
LH + ICRH Protection
B&C tiles
Saddle Coil Protections
Divertor
Bulk W

JET - The ITER-like Wall –May 2011



Plasma Wall Interaction Consequences

1. Incident deuterium ions saturate the implantation layer (~ up few hundred nm)
2. The level of saturation is defined by the [D] the nature of the FWM, but when saturation is reached then we have a dynamic balance between adsorption and ion-induced desorption
3. From the implantation layer, D 'diffuses' further into the bulk material along the grain surfaces (inter-grain diffusion) or inside the grains (intra-grain diffusion) of the pores. Diffusion is temperature depending and also on some physical parameter (such as weave of the CFC fires)



PWI: Erosion Deposition & Fuel Loss

During Fusion reaction tritium leaves the plasma and after interaction with the first wall materials remains in the wall increasing the vessel inventory

(in ITER safety limit 700 g tritium inside the machine)

Questions:

1. ***What is the distribution of the eroded material inside the vacuum vessel and where are the deposition zones ?***
2. ***Where, how and why fuel is stored ?***
3. ***How does the material erosion and transport mechanism influence the fuel inventory ?***

Why Care about Fuel Loss ?

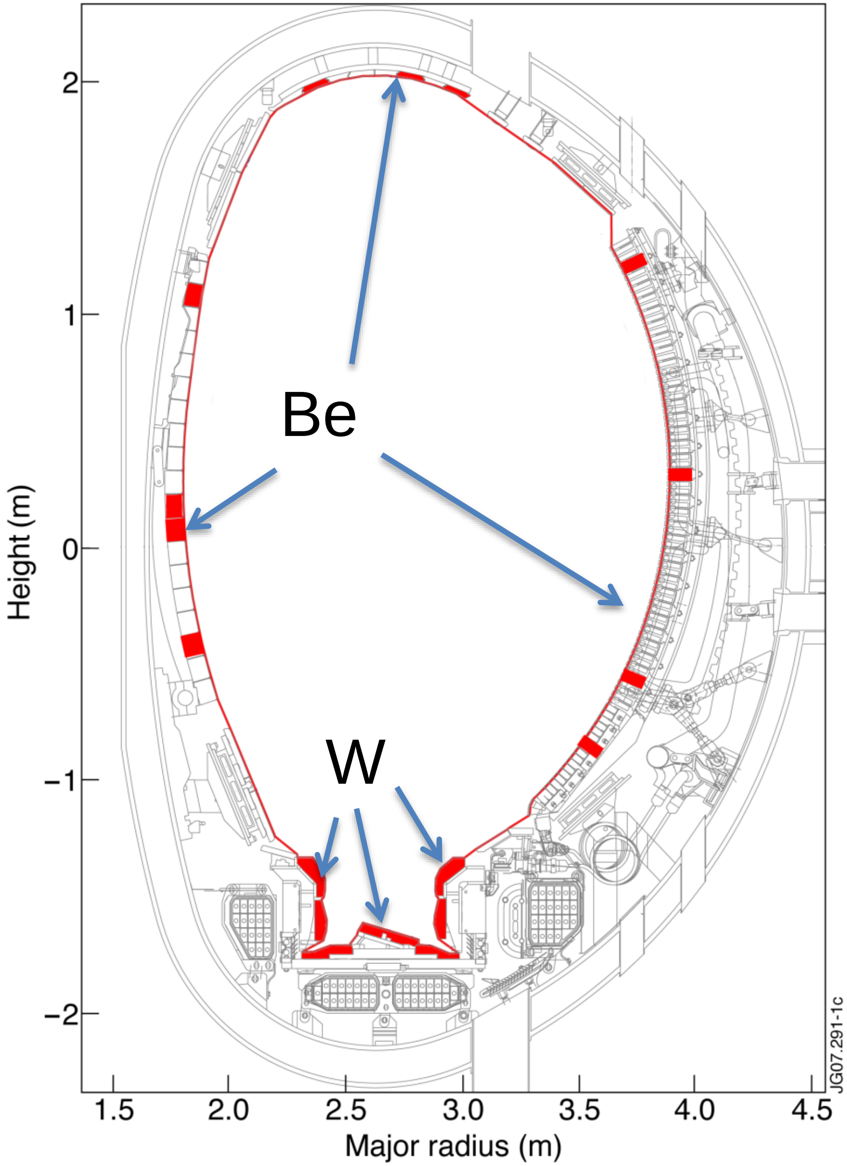
Cost & Reactor safety

One bottle of 6400L at STP D_2 cost ~£ 5000

Cost: Tritium is bought from Canada and cost ~ £ 65 000/**g** (Au £24/g) if you think that ITER will use about 10-15 **kg** (1-1.2 **kg**/y) tritium during its lifetime operation….

Safety: 1 g tritium ~10 000 curies (1Ci= 3.7 E10 decays/s)
Permissible release rate = 1 curie/day

Post-Mortem Surface Analysis



Post mortem analysis of tiles

- Poloidal cross section removed from vessel
- Some tiles have marker coatings

Non-destructive

Ion Beam Analysis;
(RBS, NRA, PIXE, PIGE)
& IP (auto-radiography)

Destructive

SIMS, AMS, FC-LSC

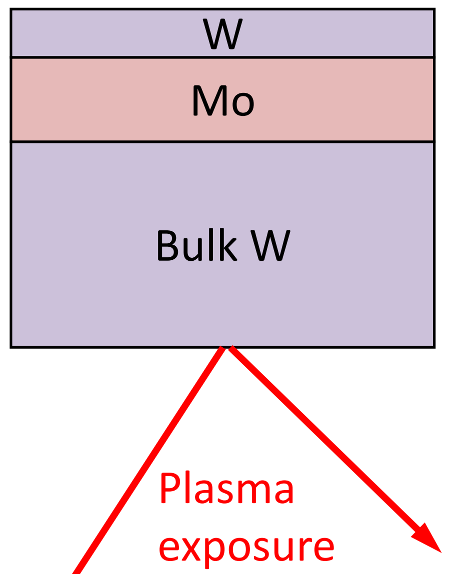
The Coating Principle

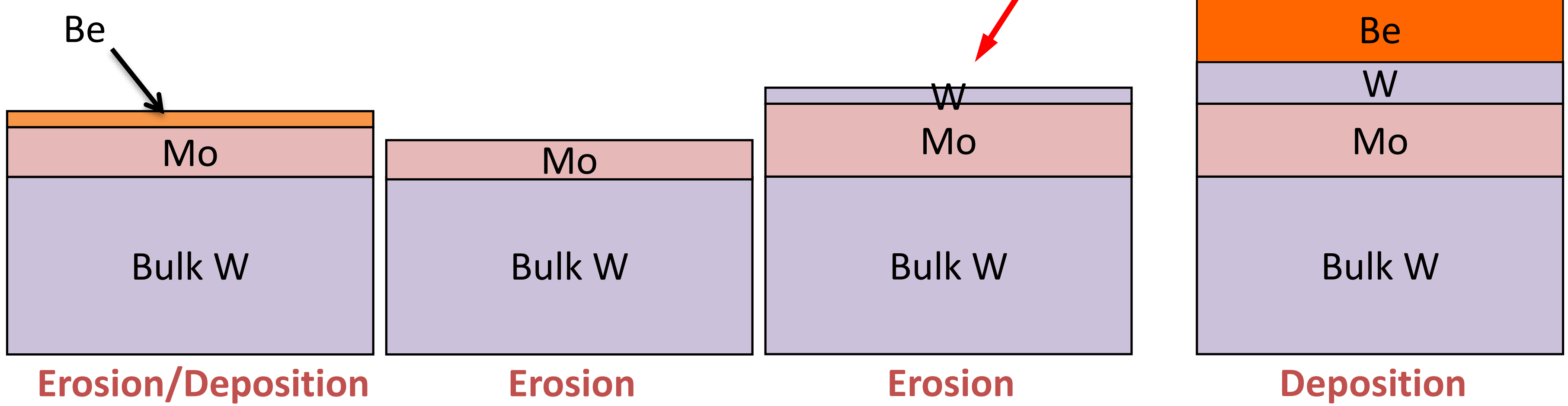
How determine the W erosion in JET divertor ?

- Marker stripe of various metals on the W divertor tiles

W-layer (14 µm PVD or 30 µm PVD) on CFC

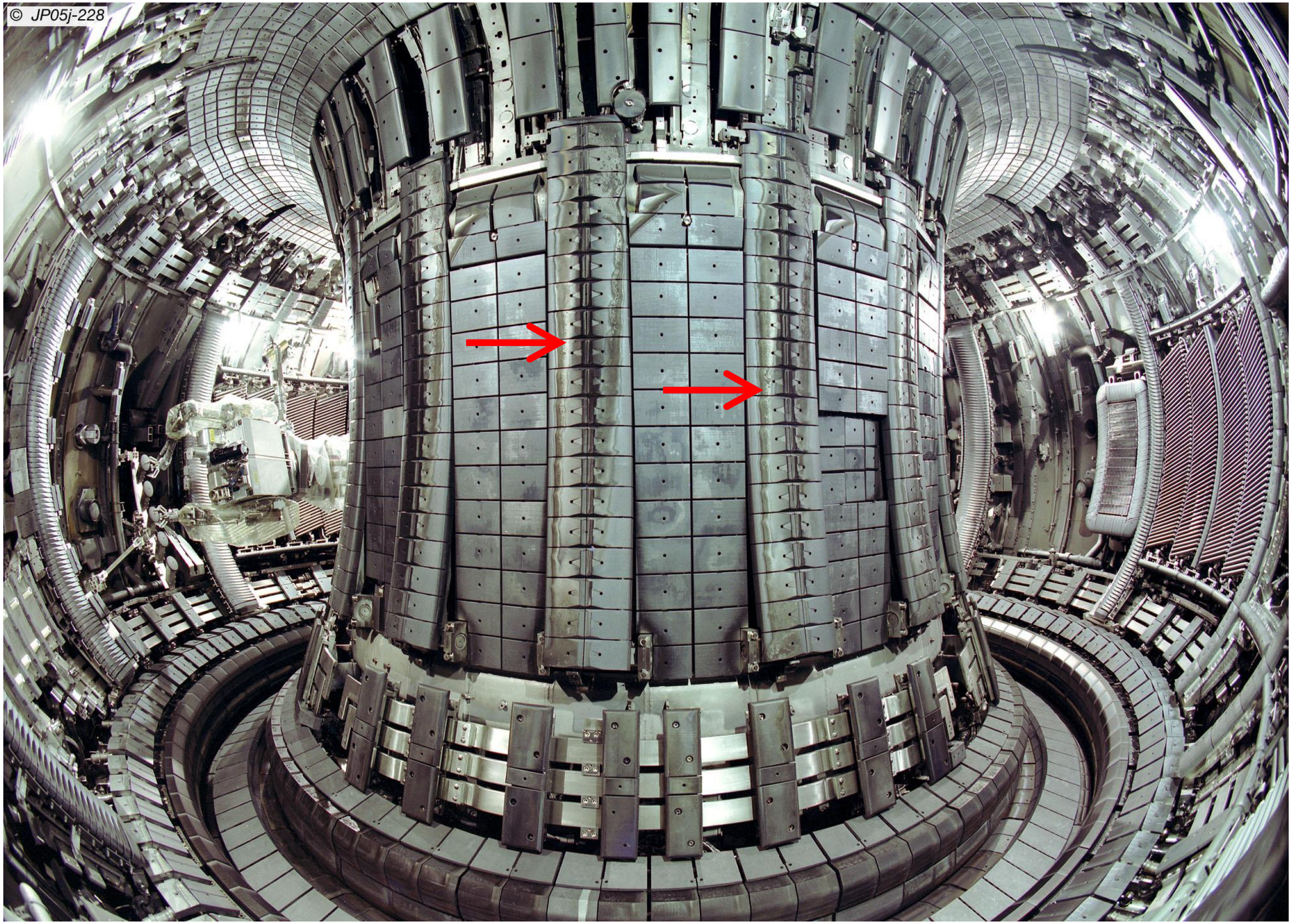
⇒ **Use marker layer of 5 µm W and 7 µm Mo**





M. Mayer, JW8-FT-3.41, Tungsten Erosion of JET Divertor. JET Report

JET 2009 – Carbon wall

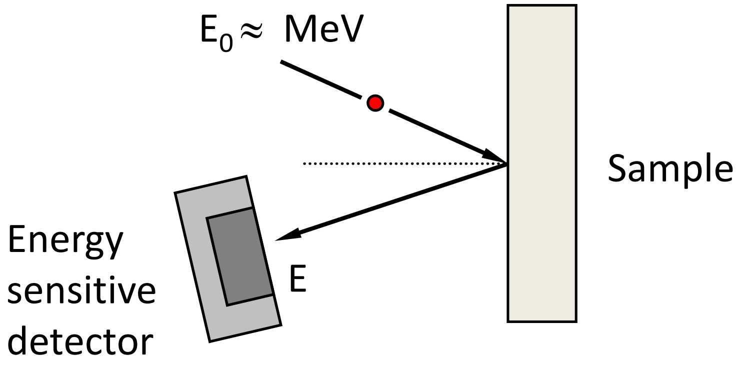


I. Non-destructive Surface Analysis

IBA – Ion Beam Analysis

Group of methods for the near-surface layer analysis of solids

- Elemental composition and depth profiling of individual elements
- Quantitative without reference samples
- No (or small) matrix effects
- Non-destructive
- Analysed depth: 0.5 µm (heavy ions) to 40 µm (using protons)
- High sensitivity ≈ ppm



1. Detection of charged particles
2. Detection of photons

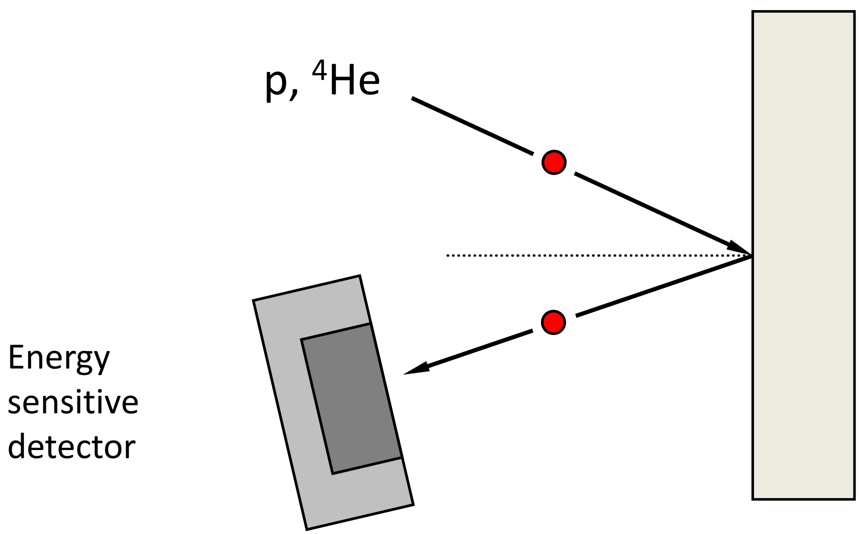
IBA: 1. Detection of charged particles

What is common for all IBA methods: Incident particles have MeV energies

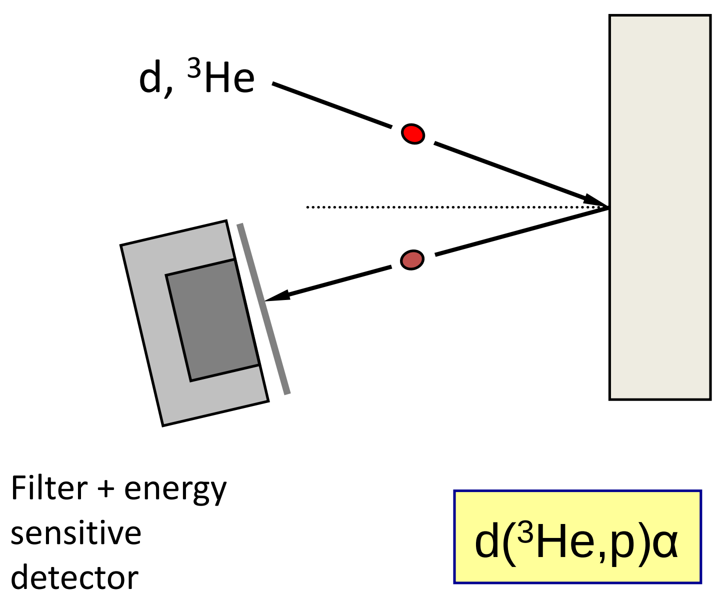
⇒ Methods can often be used in the same experimental setup

⇒ Methods can sometimes be used simultaneously

Rutherford Backscattering (RBS)

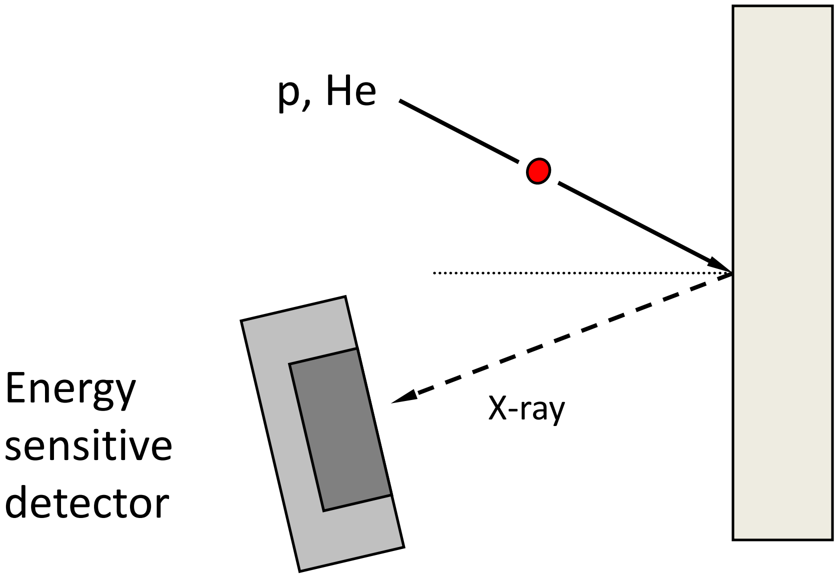


Nuclear Reaction Analysis (NRA)



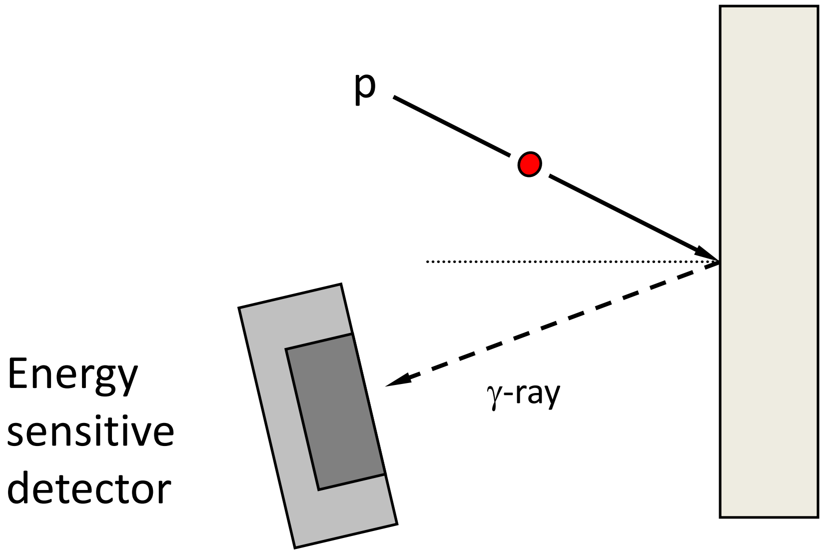
IBA methods: 2. Detection of photons

Particle induced X-ray emission (PIXE)



($10^{-10}>\lambda>10^{-12}$ m)

Particle induced γ-ray emission (PIGE)

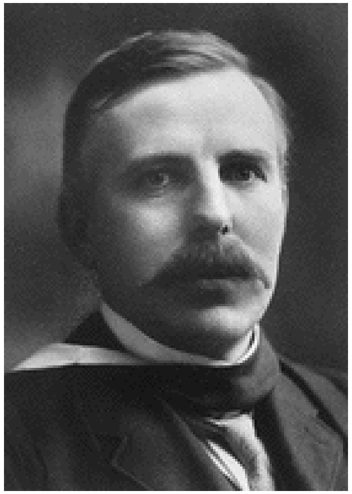


($\lambda<10^{-12}$ m)

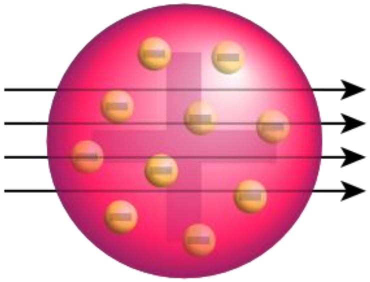
The Rutherford Backscattering Spectroscopy

Sir Ernest Rutherford (1871 - 1937)

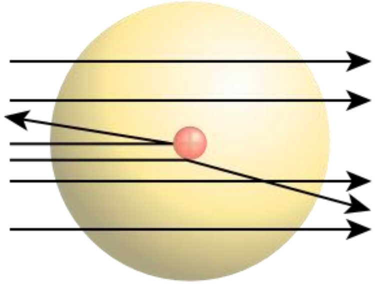
- 1909-1914: Rutherford's scattering experiments: ^{4}He on Au



Rutherford supervised a series of experiments carried out by Hans Geiger and Ernest Marsden between 1909 and 1914 studying the scattering of alpha particles <u>through</u> metal foils.
Rutherford suggested that Marsden attempt to measure backscattering from a gold foil sample. According to the then-dominant plum-pudding model of the atom (J.J. Thomson 1904), backscattering of the high-energy positive alpha particles should have been non-existent



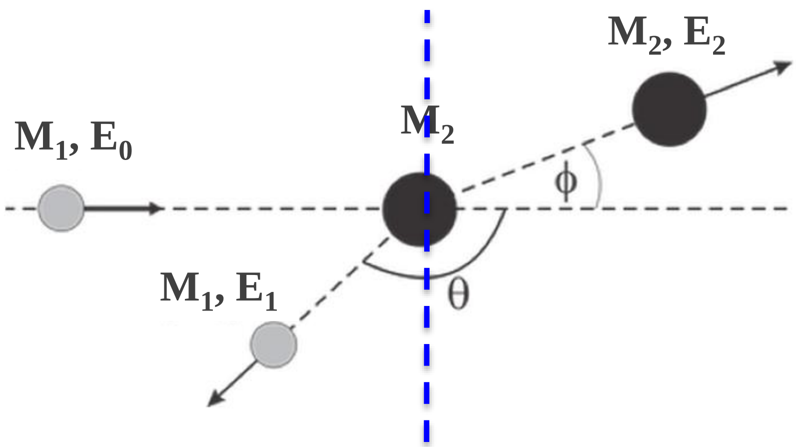
At most, only small deflections should have occurred as the alpha particles passed almost unhindered through the foil.
Instead, when Marsden positioned the detector on the same side of the foil as the alpha particle source……He immediately detected a noticeable backscattered signal. Obviously there were many collisions!!

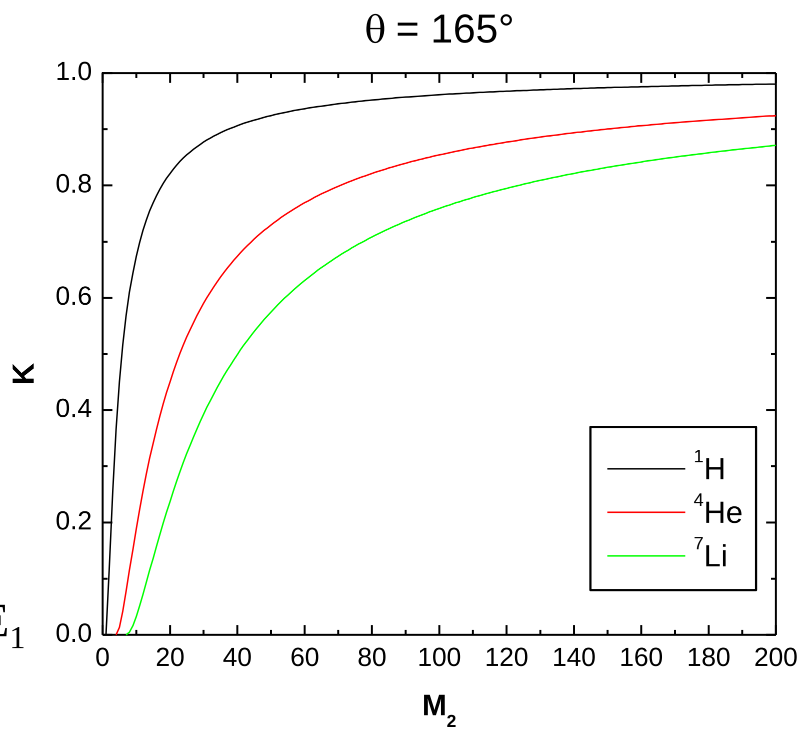


"It was quite the most incredible event that has ever happened to me in my life. It was almost as incredible as if you fired a 15-inch shell at a piece of tissue-paper and it came back and hit you." (E. Rutherford)

RBS: Elastic Scattering Kinematics

During the collision, energy is transferred from the incident particle to the target atom.
The change in energy of the scattered particle depends on both masses. During this elastic scattering the total energy is preserved : $E_0 = E_1 + E_2$





After the collision, the residual energy E_1 of the scattered particle at angle θ can be expressed as: $E_1 = k E_0$ where k is known as the *kinematical factor*

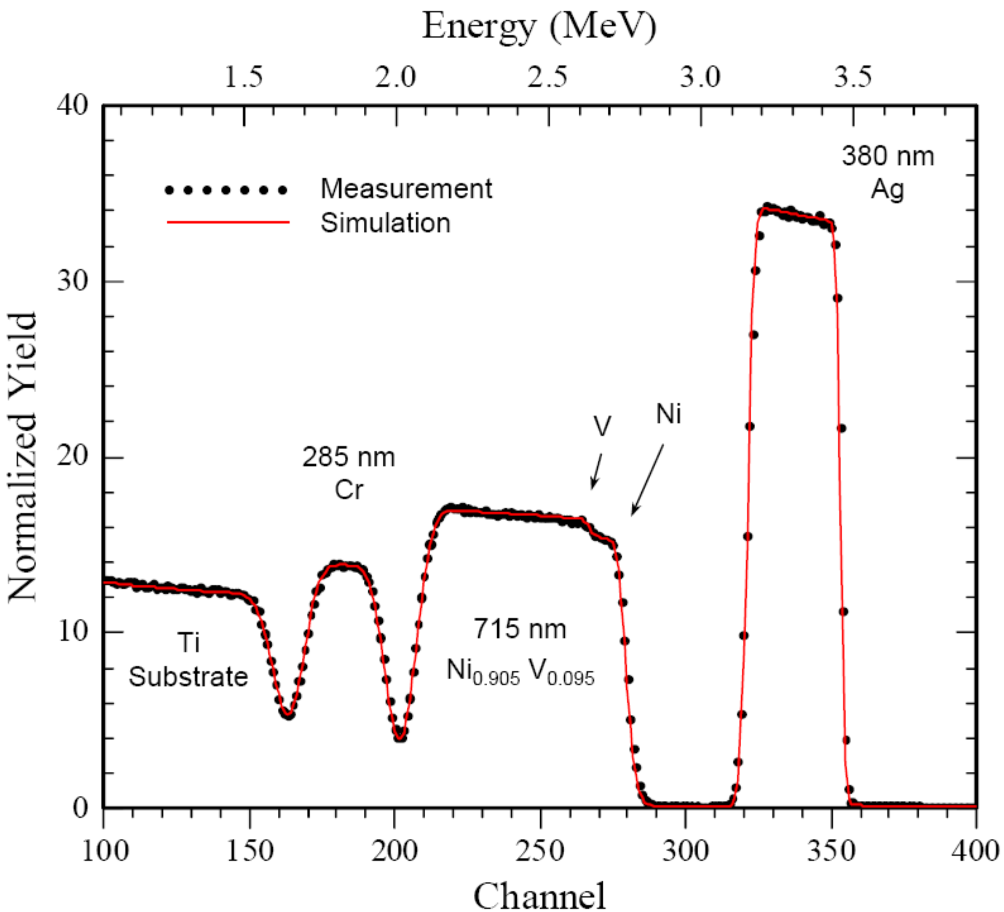
$$K = \left[\frac{\left(M_2^2 - M_1^2 \sin^2\theta\right)^{1/2} + M_1 \cos\theta}{M_1 + M_2} \right]^2$$

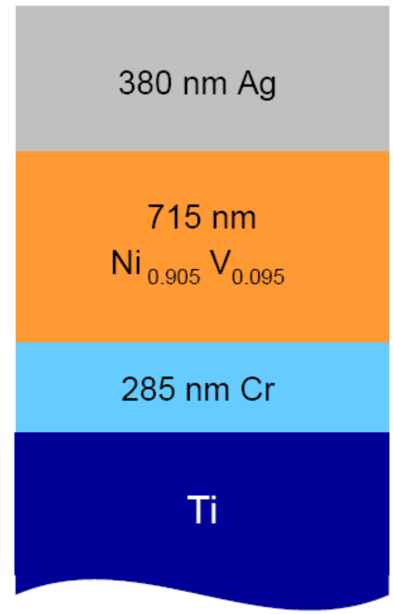
$$\Delta E_1 = E_0 \frac{dK}{dM_2} \Delta M_2$$

ΔE_1: Energy separation
ΔM_2: Mass difference

⇒ mass resolution decreases for heavier elements

RBS Typical spectrum



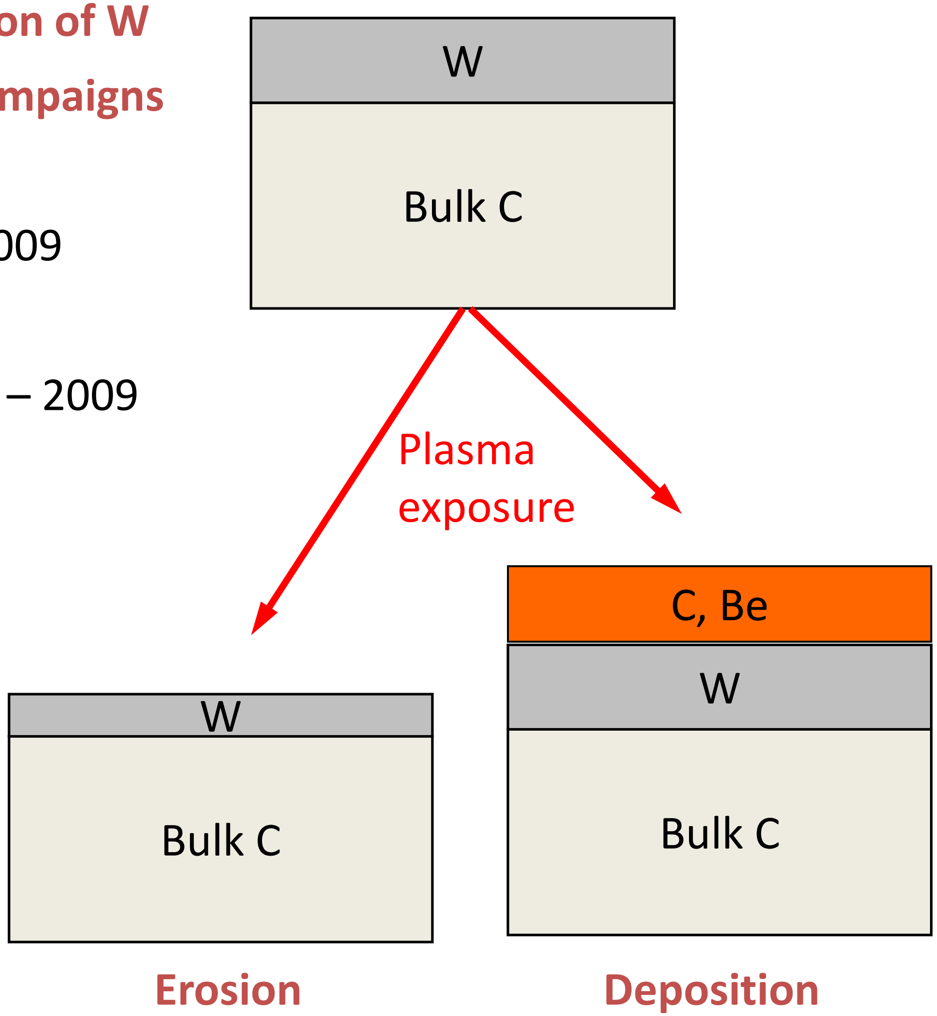


The Ag peak is well separated from the substrate signal, which is due to the higher mass (M_2) of Ag (108) compared to the rest of the transition elements (Ti, Cr, Ni, V m=47-64). In this elastic scattering process, the He particles (M_1) are scattered with a much higher recoil energy than the He from the substrate.

RBS at JET

RBS at JET is used mostly to determine erosion of W and C markers during the last operational campaigns of JET

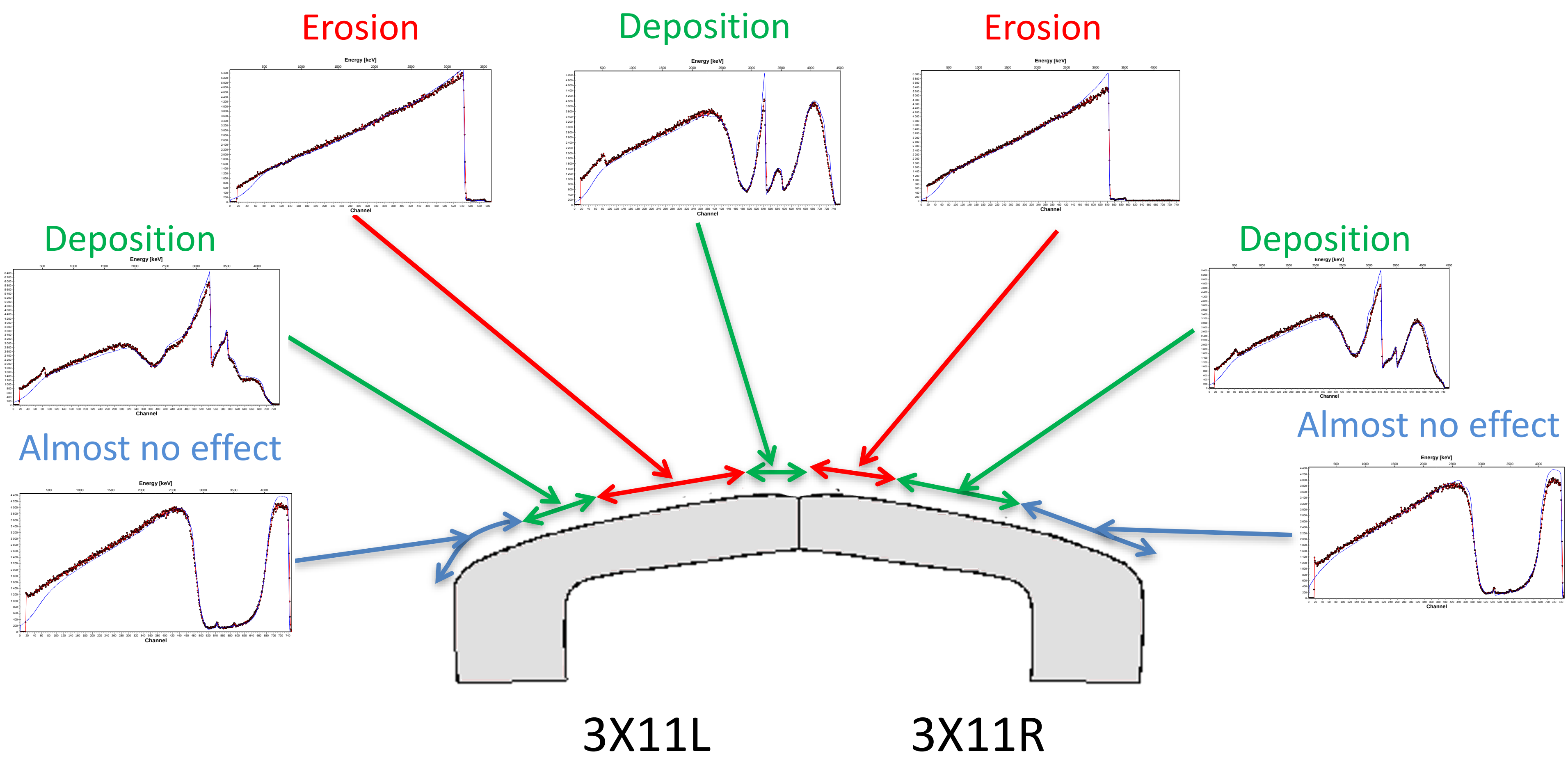
- Divertor tiles LBSRP, 7, 8 exposed 2007 – 2009
- Divertor tiles 6, 7, 8 exposed 2004 – 2009
- Inner wall guard limiter tiles exposed 2004 – 2009



To determine the erosion taking place at the inner wall

- 9 sachet samples (2004 – 2009)
- 2 flap units exposed 2004 - 2009

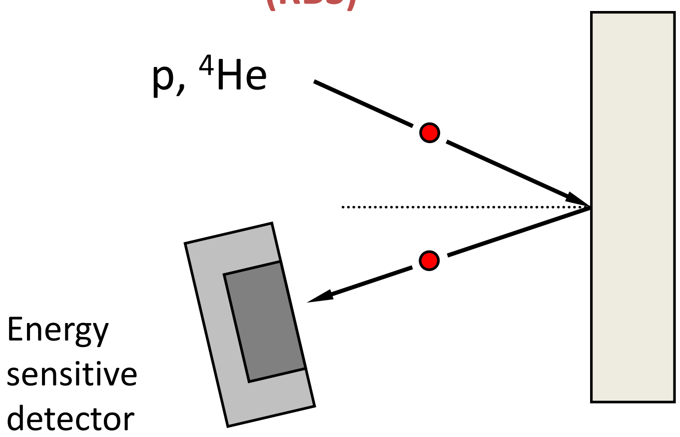
RBS: Erosion – Deposition map



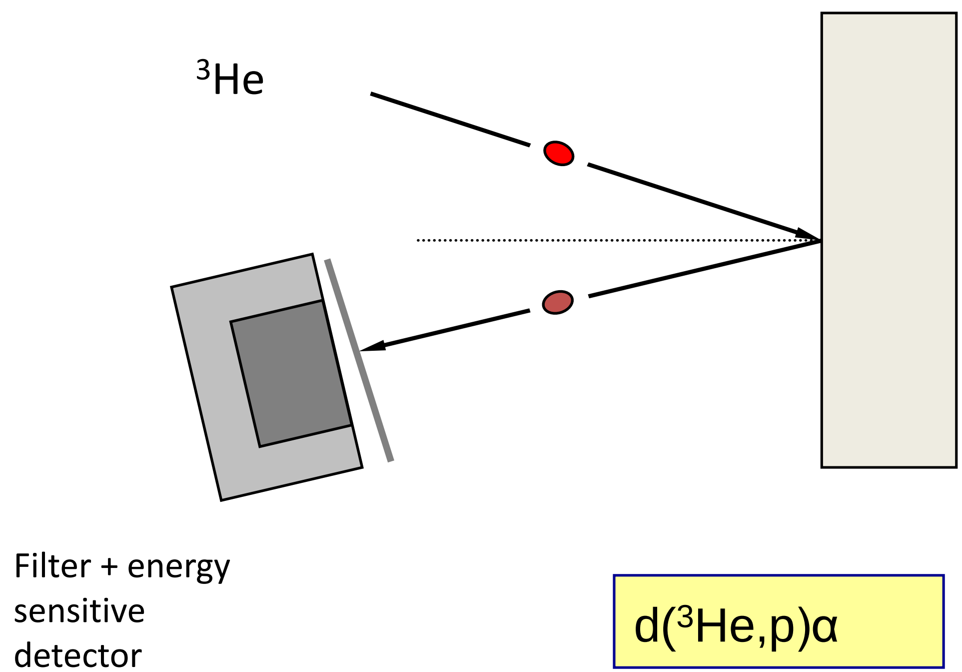
- Total amount of deposited C, extrapolated to the whole limiter: 109 g
- Erosion in erosion-dominated areas > 3 µm W

IBA: 1. Detection of charged particles

Rutherford Backscattering (RBS)



Nuclear Reaction Analysis (NRA)

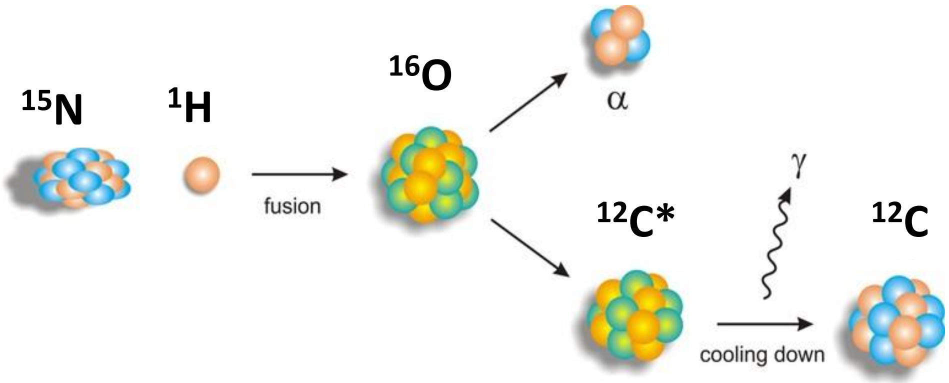


$d(^3He,p)\alpha$

Nuclear Reaction Analysis

The NRA uses accelerated particles, which initiate a nuclear reaction.
The emitted radiation is characteristic for this reaction and can then be detected while from the intensity of the radiation we can determine the concentration of the particular atomic species.
NRA is well suited for the detection of light elements since other methods fail in this field.

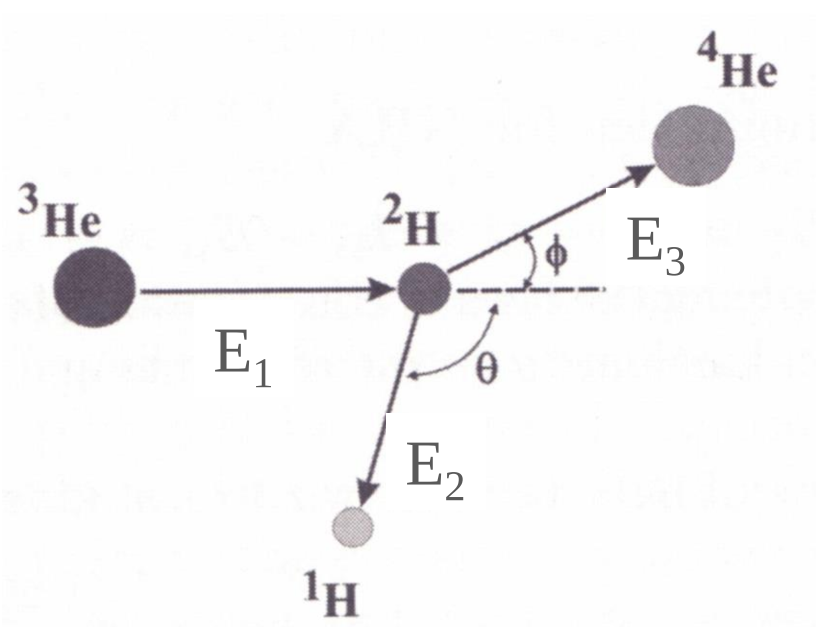
The products of the nuclear reaction are charged particles, gamma ray or both



The decay of the ^{16}O which is in an excited state generates an alpha particle and an excited ^{12}C isotope The latter emits a gamma quantum with a well-defined energy of E_γ = 4.43 MeV to reach its ground state which is the measured photon characteristic for this nuclear reaction.

NRA Kinematic

For Deuterium detection (for practical reasons) instead of ^{15}N, we are using ^{3}He.
The kinetic energy E_1 of ^{3}He, must be higher of the Coulomb barrier, then we have a nuclear reaction and production of a scattered p (^{1}H) and recoiling ^{4}He.
The excess kinetic energy of the final product is the Q-value of the reaction.



$$Q = \left(\sum M_r - \sum M_p\right)c^2 = \sum T_p - \sum T_r$$

The nuclear reactions can be *endothermic* (Q<0) or *exothermic* (Q>0). If Q>0 any E_1 generates a reaction, however, for the low values of E_1 the cross section is low.
For Q<0 there is a critical energy E_1 above which the reaction becomes possible

Note. For Q=0 we have Elastic scattering

The energy of the fast proton detected depends on the depth of the deuterium atom in the sample

NRA for Deuterium Measurement

For the Deuterium depth profile measurements a ^3He ion (+1 or +2) beam is used
We distinguish 3 cases Q>0, Q<0 or Q=0. However….

$$^3_2He +^2_1 D \longmapsto ^4_2 He +^1_1 p \qquad Q = 18,35\ MeV$$

$$^3_2He +^2_1 D \longmapsto ^1_1 p +^1_0 n +^3_2 He \qquad Q = -2,73\ MeV$$

$$^3_2He +^2_1 D \longmapsto ^2_1 D +^2_1 D +^1_1 p \qquad Q = -6,52\ MeV$$

$$^3_2He +^2_1 D \longmapsto ^3_1 T +^1_1 p +^1_1 p \qquad Q = -1,95\ MeV$$

Nuclear Reaction

All these reactions produce various products, which can be detected in the detection set-up. However, as Q<0 for most of them we can get rid of the parasitic reactions (2-4).

$$^3_2He +^2_1 D \longmapsto ^3_2 He +^2_1 D$$

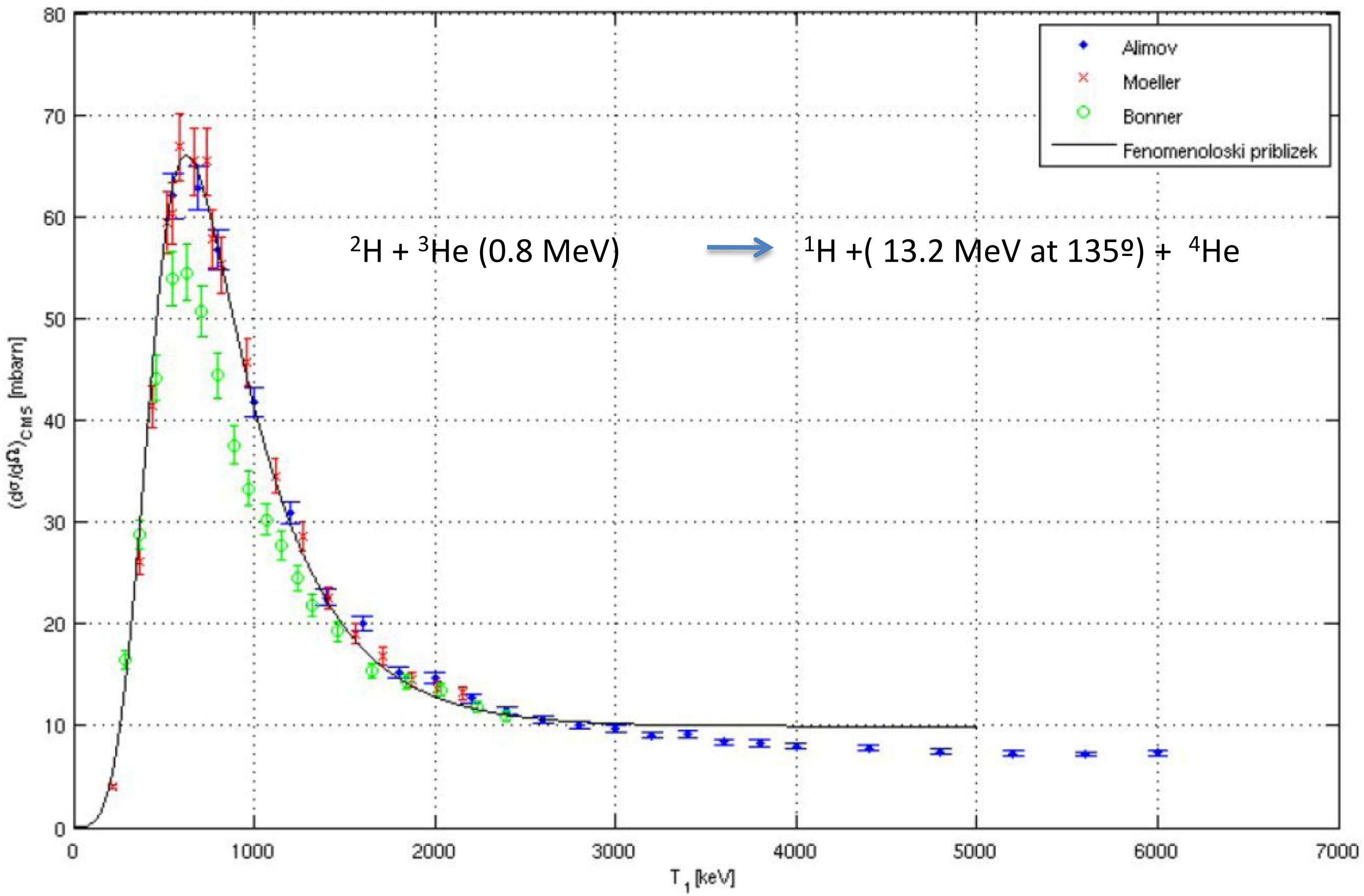
$$^3_2He +^{12}_6 C \longmapsto ^{12}_6 C +^3_2 He$$

$$^3_2He +^{13}_6 C \longmapsto ^{13}_6 C +^3_2 He$$

Elastic Scattering

The Elastic scattering reactions of ^3He with D are forming a low-energy background. However, we resolve the problem by placing a protective mylar foil in front of the detector. Fast protons are passing through, while the foil stops the scattered helium ions.

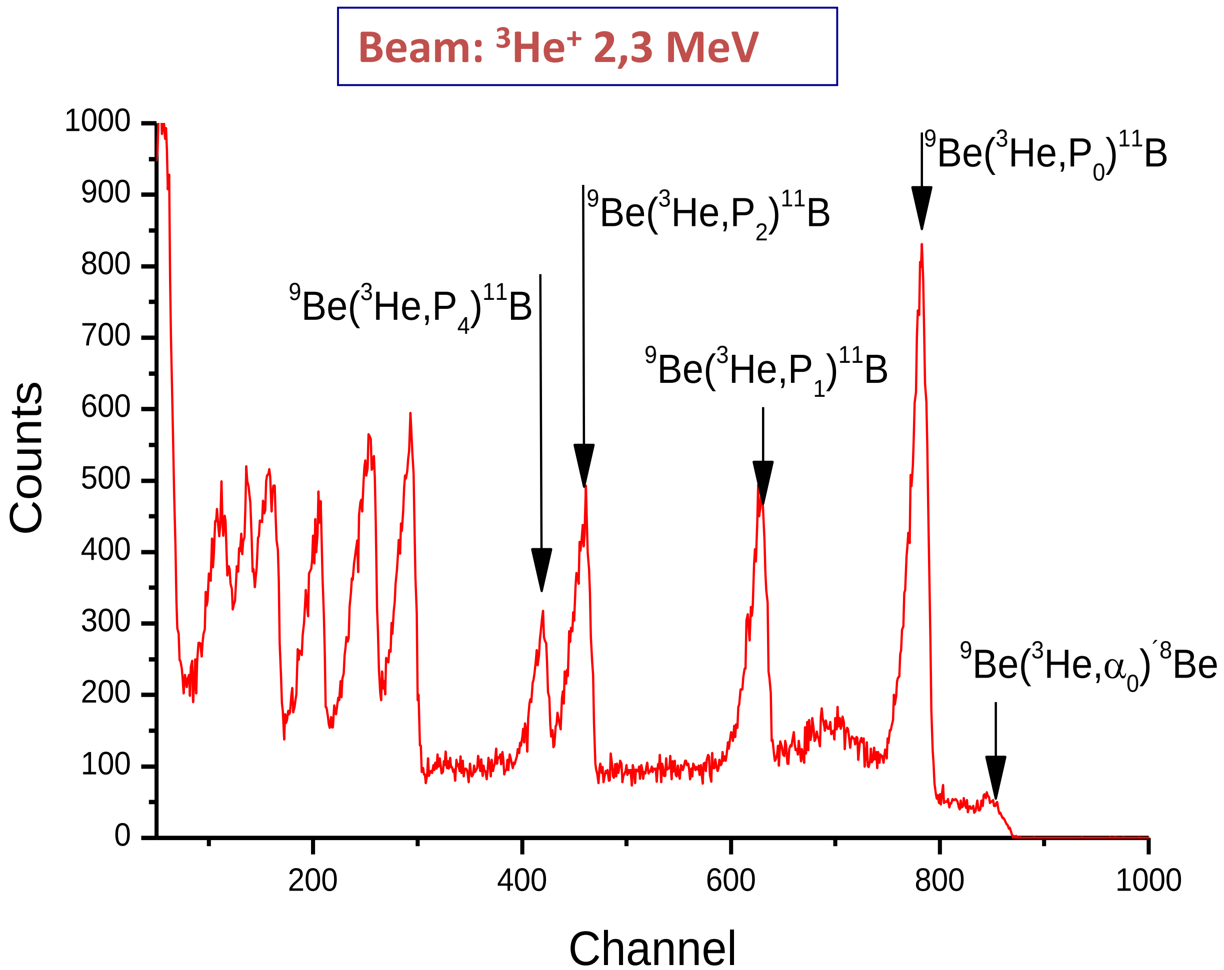
Cross-Section d(^{3}He,p)^{4}He



^{2}H + ^{3}He (0.8 MeV) → ^{1}H +(13.2 MeV at 135º) + ^{4}He

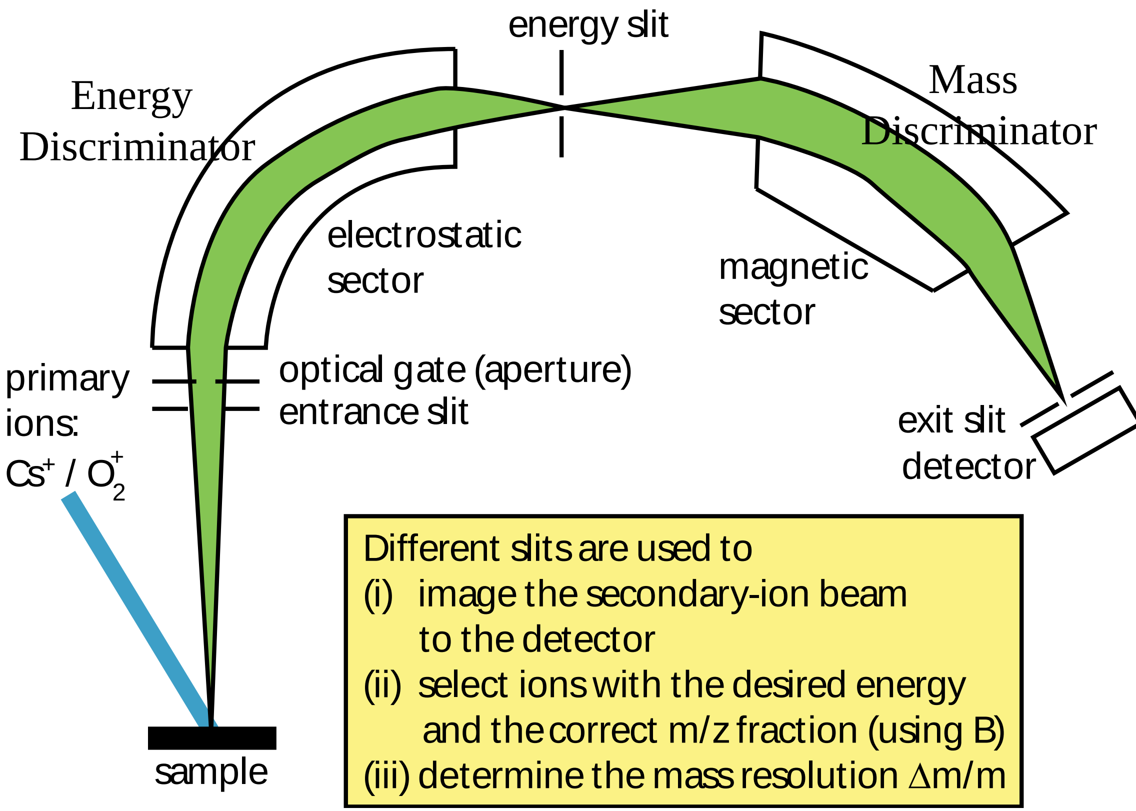
NRA spectrum of a Be sample

Several reaction channels are excited with emission of $^{1}H^{+}$(identified as Po..P4). The peaks allow the profile o Be in any solid sample down to the ppm level.



II. Destructive Surface Analysis: SIMS

SIMS uses an ion beam for sputtering the surface to investigate while we are analysing the masses of the ions of the sputtered particles.
Ion beams are (Cs^+, O_2^+ , Ar+, Ga+) of energy in the range 1 to 30keV (primary ions)
The sputtered ions (to analyse) are the secondary ions → Secondary Ion Mass Spectrometry (SIMS).

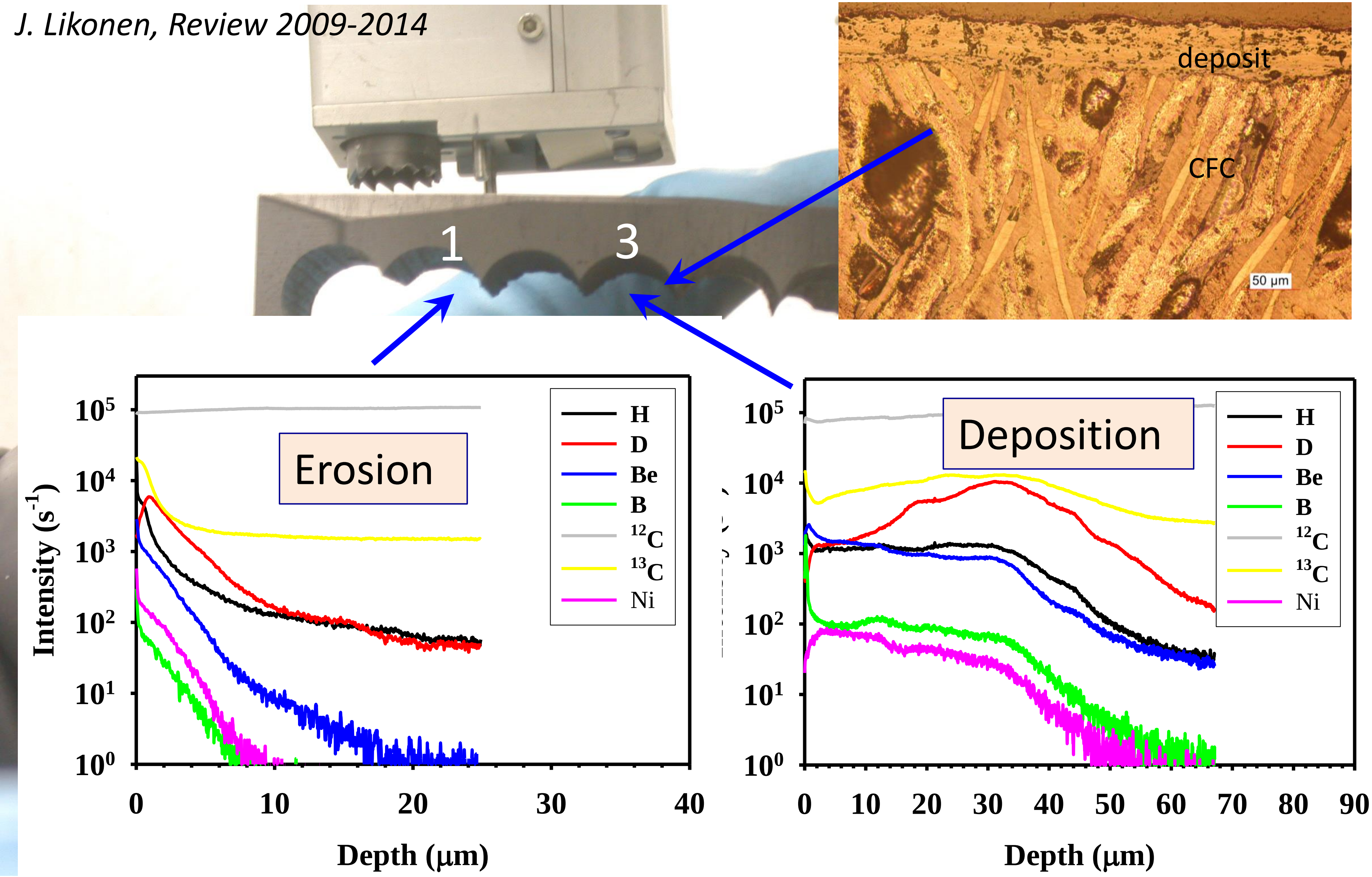


In this configuration (*forward geometry*), multiple ion beams can be measured simultaneously.
If the magnetic sector precedes the electrostatic sector (*reverse geometry*), then mass resolution is improved at the cost of losing the ability to measure multiple ion beams simultaneously.

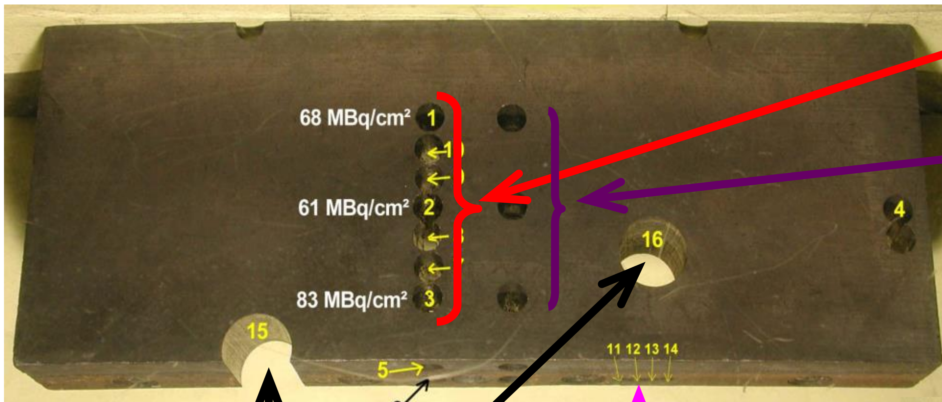
Oxygen bombardment increases the yield of positive ions and caesium bombardment increases the yield of negative ions.

SIMS Erosion Deposition Profiles

J. Likonen, Review 2009-2014



Tritium Analysis by Full Combustion



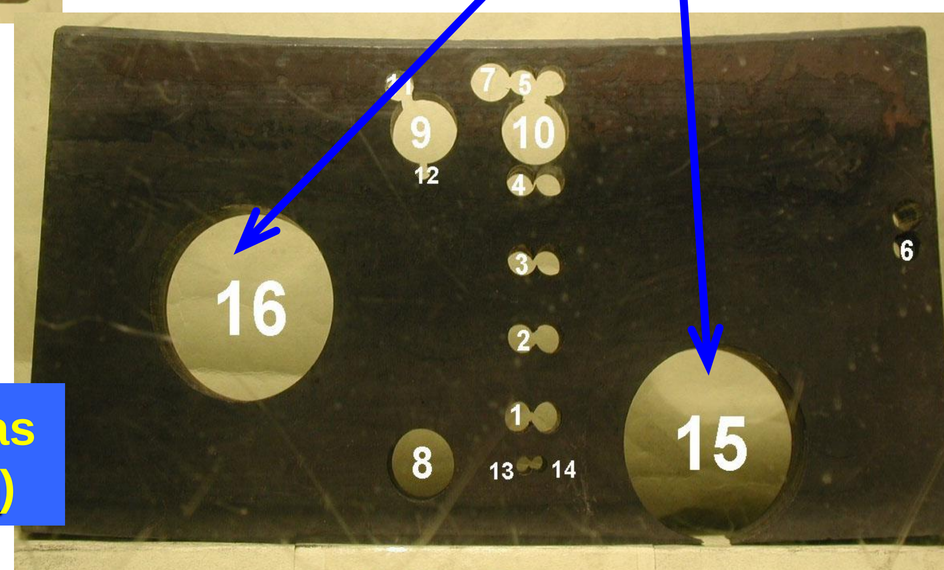
Combustion
(tritium concentration in **mm range**)

Ion Beam Analysis (D analysis)

Calorimetry and BIXS

Laser
(**detritiation**)

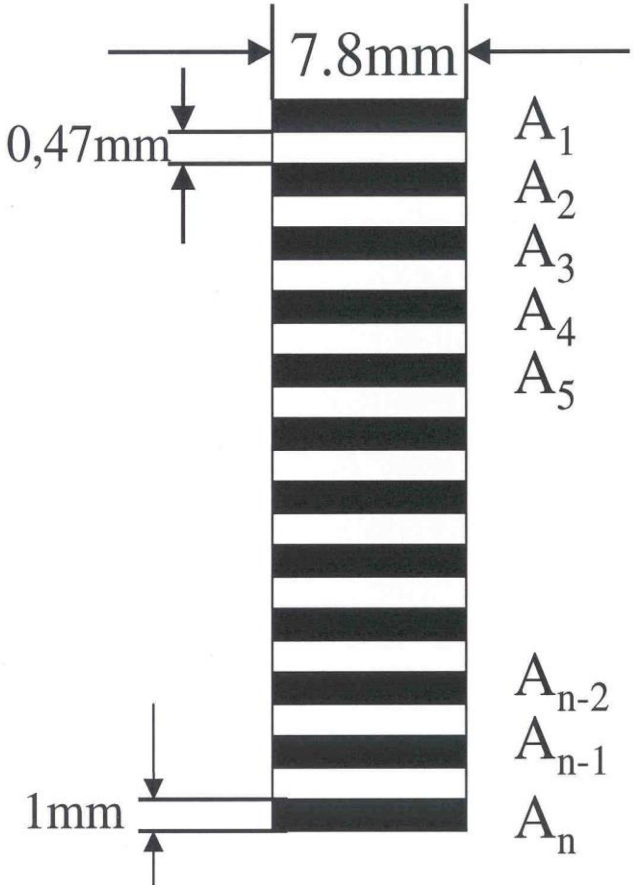
AMS
(tritium concentration in **µm range**)



For autoradiography the whole tile has been used (surface tritium distribution)

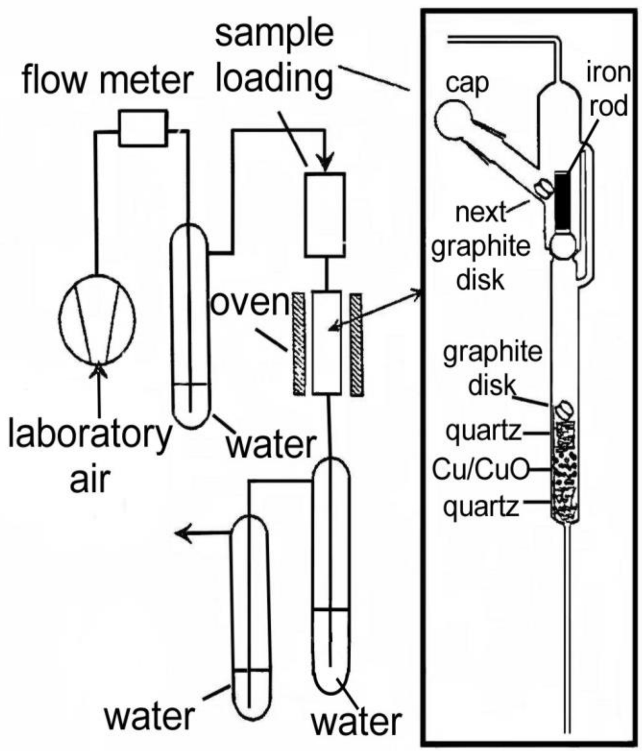
Sampling & Full Combustion Measurements

Graphite (left) and CFC (right) cylinders as well as some typical disks



Specimen designation A1 = Plasma exposed side

The Vance Apparatus



Tritiated Water is analysed by LSA

The reactions

$$2C_{(s)} + O_{2(g)} \rightarrow 2CO_{(g)}$$

$$2C_{(s)} + 2O_{2(g)} \rightarrow 2CO_{2(g)}$$

It was establish that the presence of water increases significantly the combustion rate.

$$2H_2O_{(g)} + C_{(s)} \rightarrow 2H_2 + CO_{2(g)}$$

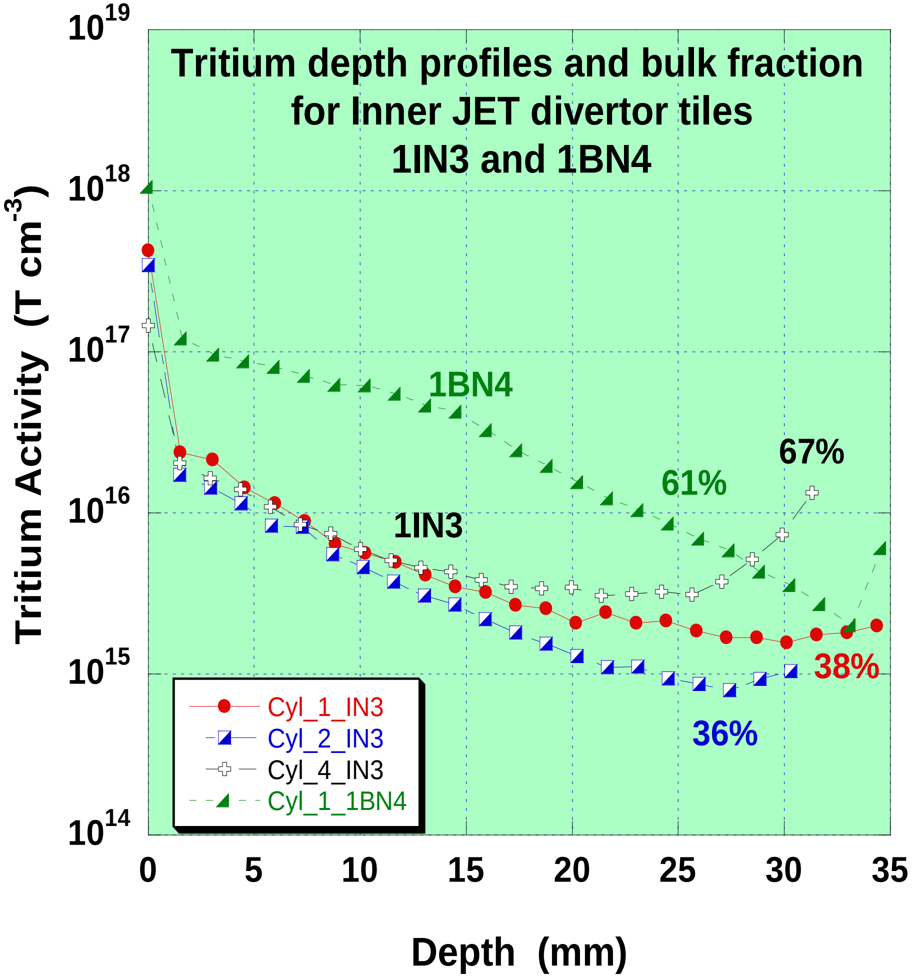
$$H_2O_{(g)} + C_{(s)} \rightarrow H_{2(g)} + CO_{(g)}$$

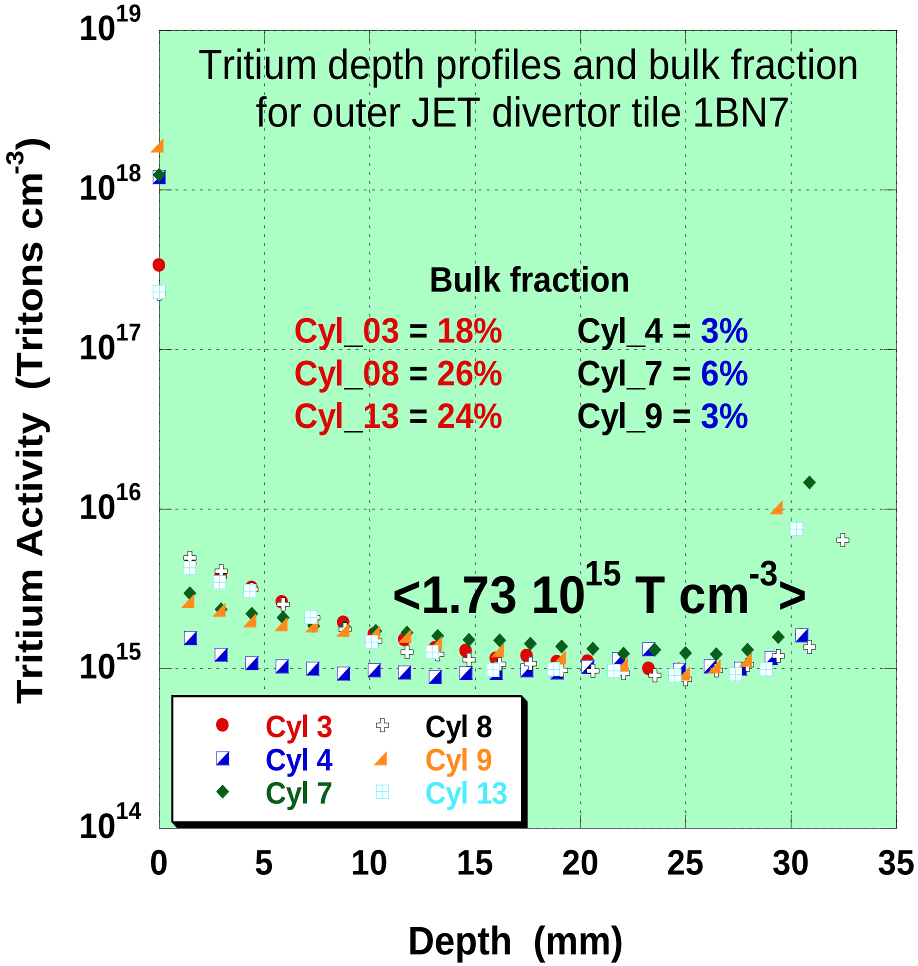
or more probably

$$2H_2O_{(g)} + C_{(s)} \rightarrow 2H_2 - CO_{2(tr)}$$

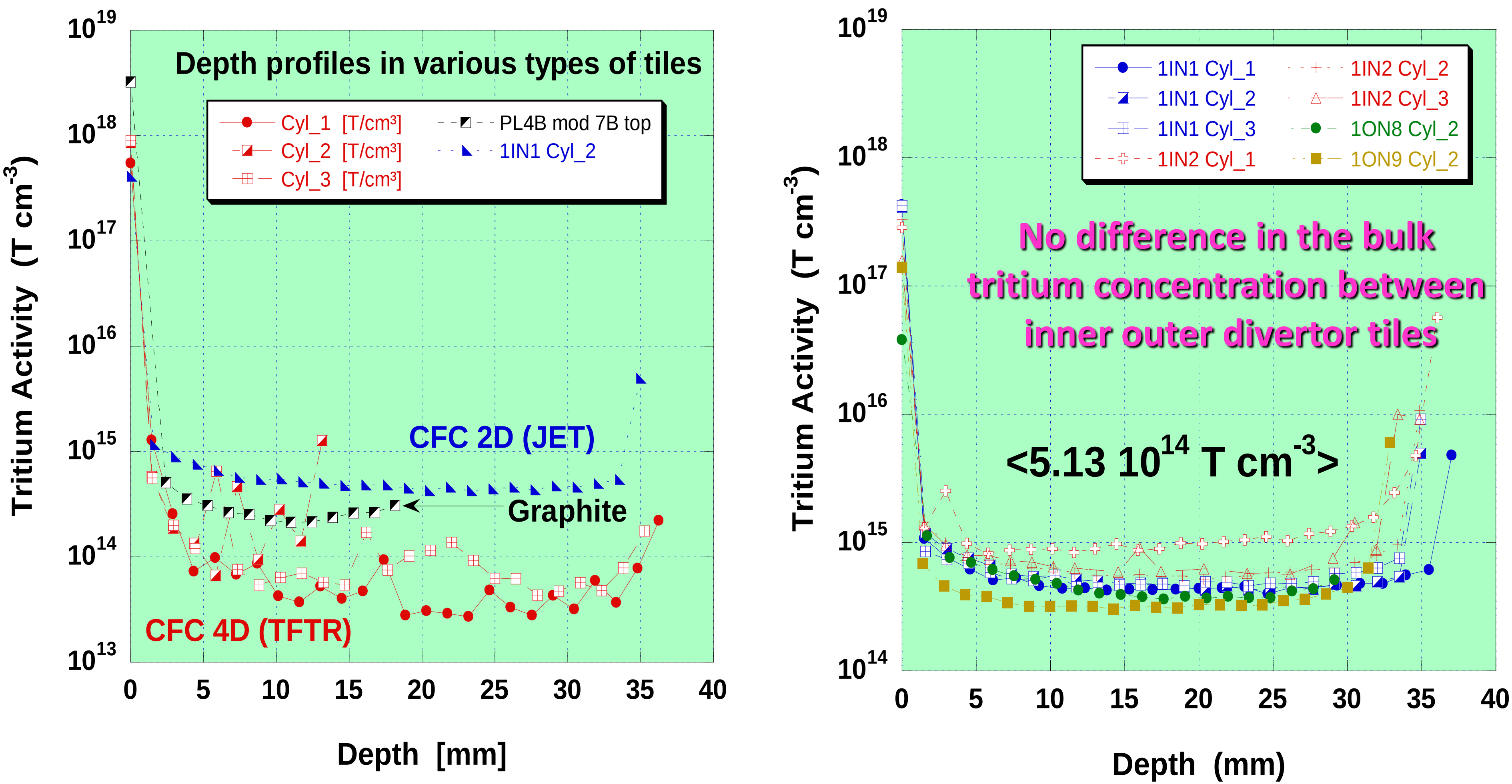
$$CO_2 - 2H_{2(tr)} + O_{2(g)} \rightarrow CO_{2(g)} + 2H_2O_{(g)}$$

Typical Tritium Profiles for JET Tiles



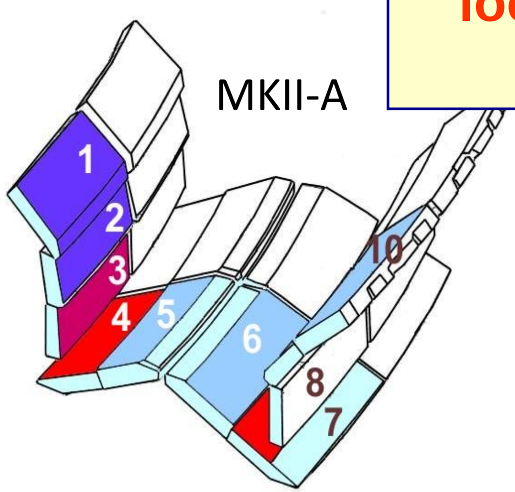


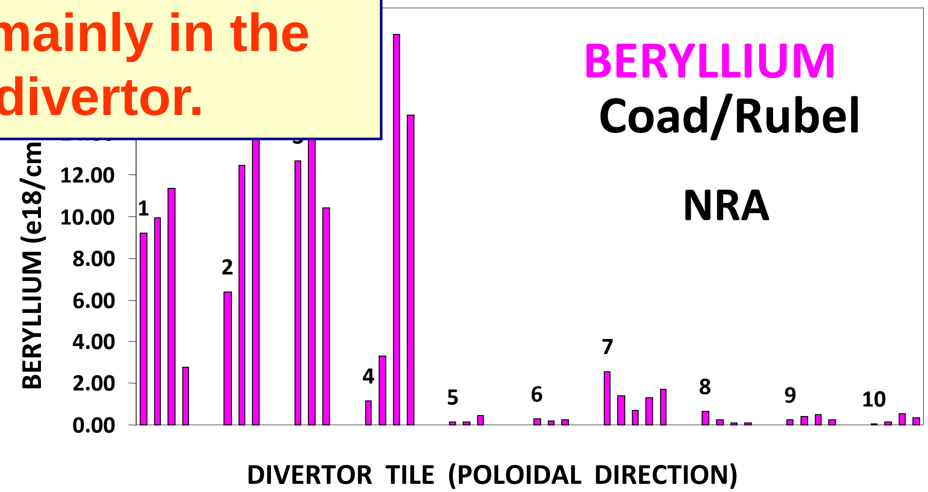
Tritium profiles in 2D & 4D CFC tiles

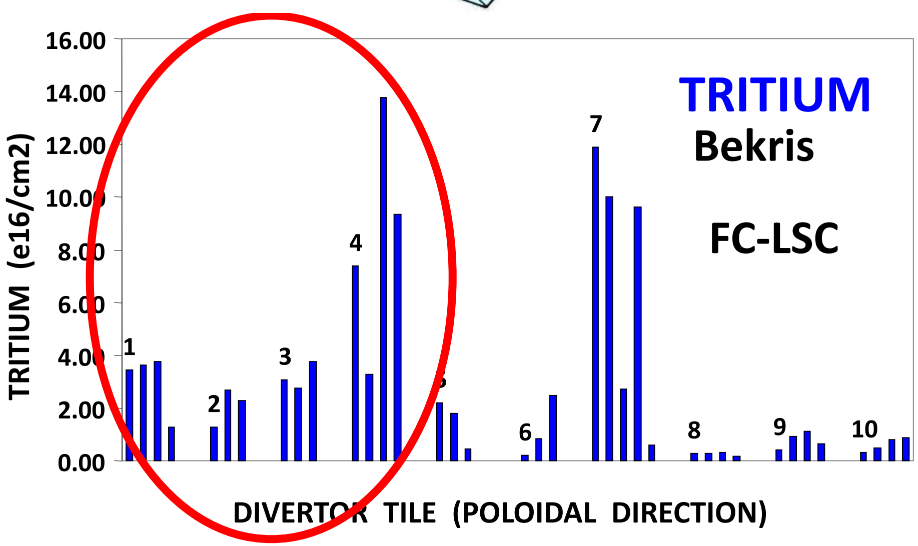


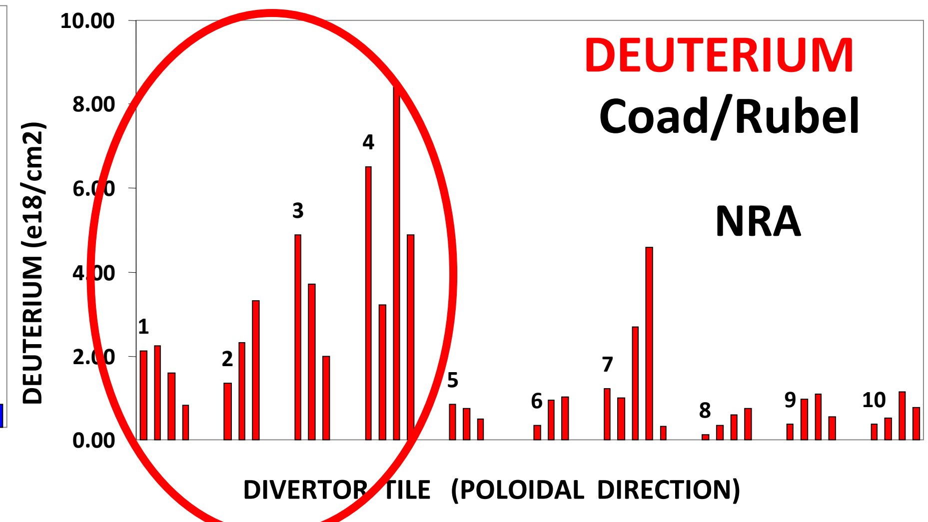
Comparison between the T, D and Be distributions

High fuel inventory (D + T) localised mainly in the inner divertor.









Imaging Plate (Non-Destructive)

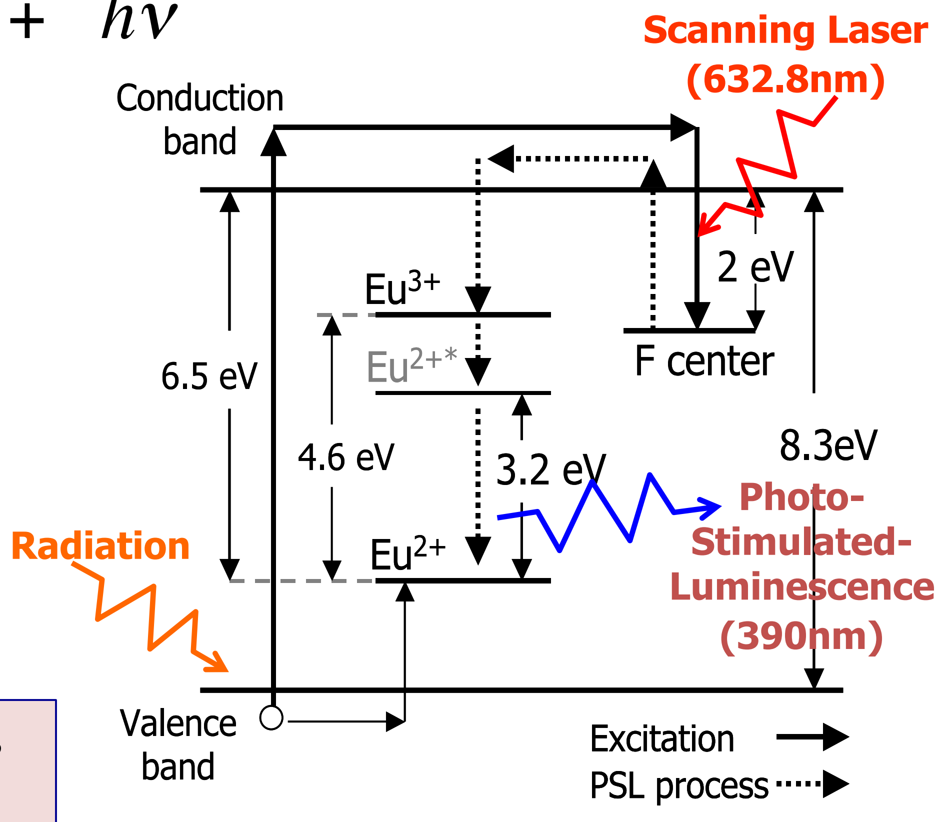
Image plates 2D cartography of [T]. It exploits the photo-stimulable properties of inorganic material. The Fuji image plates used here had BaFBr:Eu^{2+} composition. Europium acting as a luminescence centre

$$Eu^{3+} \xrightarrow{e} Eu^{2+} + h\nu$$

Radiation photons or electrons are absorbed by phosphor

Photoelectron ejected into the conduction band of the phosphor crystals and becomes trapped in a lattice defect created by the absence of halogen ions (F-center)

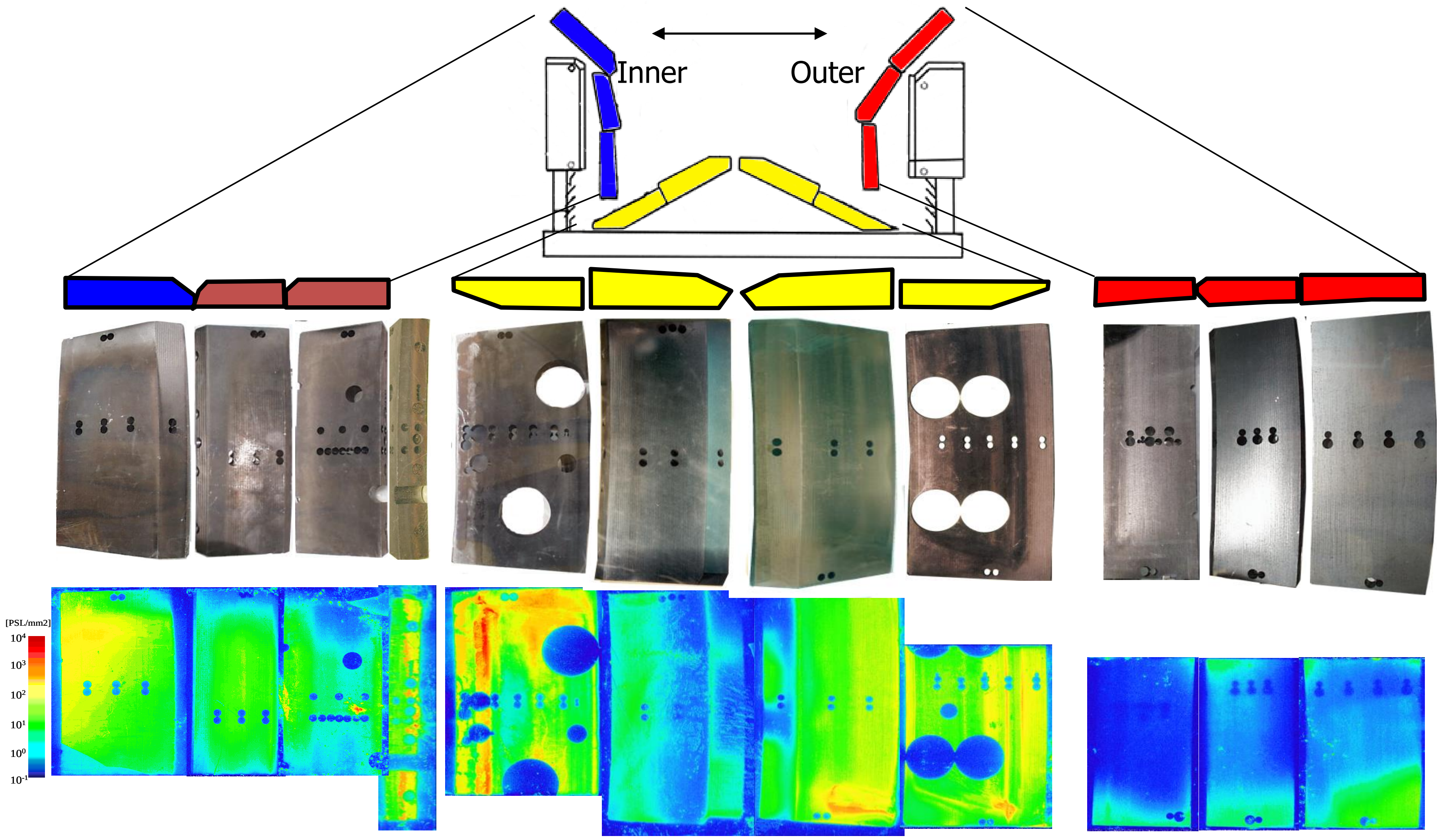
These defects were intentionally introduced during the manufacturing process.



The number of pairs (electron-vacancies) is proportional to the absorbed energy.

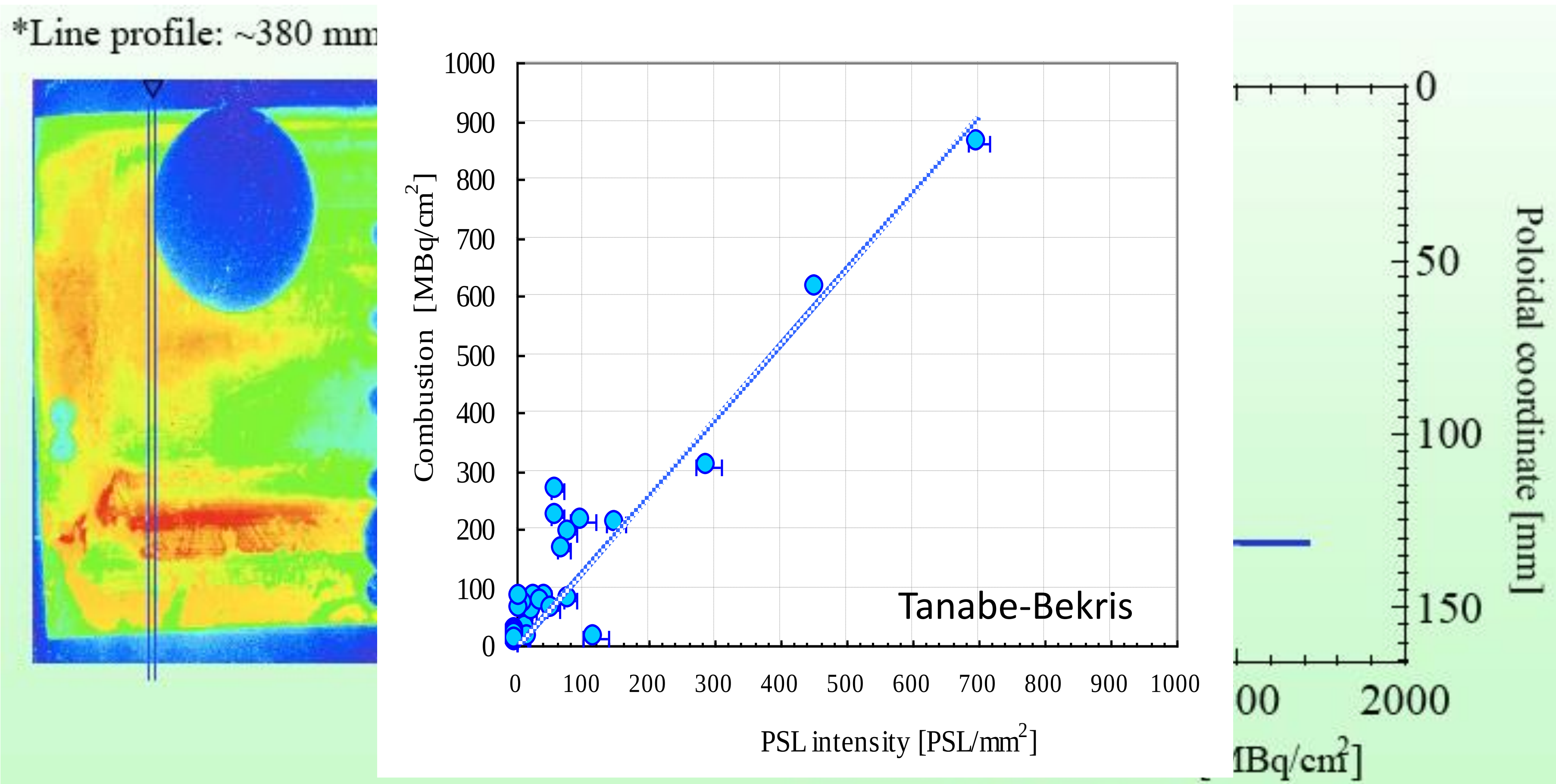
IP Analysis on MK-IIA divertor Tiles

T. Tanabe et al., J. Nucl. Mater. 345 (2005) 89-95

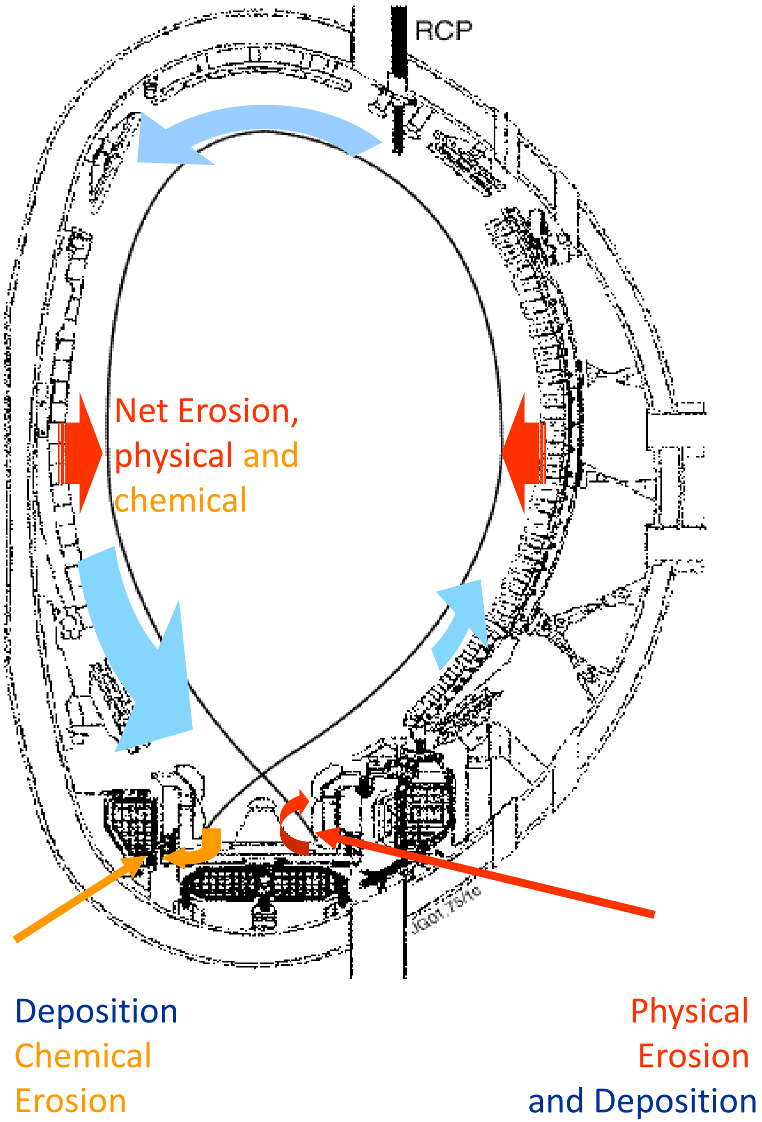


Converting PSL intensity to T activity

Surface Activity [MBq/cm²]=1.278±0.048 [PSL/cm²]



Conclusions



- Thanks to post-mortem analysis, we could establish that plasma fuel (tritium and deuterium) is co-deposited with the eroded carbon thus retaining fuel.

- The numerous surface analysis using a variety of destructive and non-destructive techniques helped us to establish a mechanism describing the material migration and fuel retention taking place inside the vacuum vessel

- Retention of fuel has safety and operational implications and need to be tackled as

  Deposits can lead to:
  Formation of dust and flakes
  Degradation of diagnostics

Acknowledgments

N. Bekris[1,2], M. Mayer[3], J. Likonen[4], K. Heinola[5,6], A. Widdowson[5] and JET Contributors*

EUROfusion Consortium, JET, Culham Science Centre, Abingdon, OX14 3DB, UK

[1]EUROfusion, ITER-Physics Department, Programme Management Unit
[2]Karlsruhe Institute of Technology, Tritium Laboratory (TLK), Karlsruhe, Germany
[3]Max-Planck-Institut für Plasmaphysik, Garching, Germany
[4]VTT Technical Research Centre of Finland, Finland
[5]Culham Centre for Fusion Energy, Culham Science Centre, Abingdon, UK
[6]University of Helsinki, Finland

** See the Appendix of F. Romanelli et al., Proceedings of the 25th IAEA Fusion Energy Conference 2014, Saint Petersburg, Russia*

Karlsruhe Institute of Technology

CULHAM CENTRE FOR FUSION ENERGY

University of Helsinki

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Fusion Technology Contributors