## High Resolution X-ray Tomography in Nuclear Energy Research

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## OUTLINE

Contribution to EURATOM FUSION

 Assessment of the Structural Integrity of a Prototypical Instrumented IFMIF High Flux Test Module Rig by Fully 3D X-ray Microtomography
 3D X-ray Micro-Tomography for Modeling of Nb3Sn Multifilamentary Superconducting Wires

- Neutron emission tomography for limited data set at JET tokamak
- Contribution to EURATOM FISSION

- Nuclear waste management: X-ray micro-tomography for crystalline host rock analysis

- X-ray Tomography for Nuclear Applications and Research
- Ultra fast X-Ray Computed Tomography for Transient Phenomena
- Gamma Emission Tomography
- OUTLOOK

Fan-beam geometry makes use of a linear x-ray detector and a divergent fan beam of x-rays. X-rays that are scattered in the same plane as the detector can be detected, but x-rays that are scattered out of plane miss the linear detector array and are not detected. Scattered radiation accounts for approximately 5% of the signal in typical fan beam scanners.

Cone-beam geometry, also known as volume or 3D scanning, is a recently developed technique that can greatly speed up collection of CT data from an object. For relatively large and dense samples cone beam geometries can lead to four detected scatter events for every detected primary photon. This geometry is currently in use in our CT.





# Reconstruction of time-resolved energy spectrum of short-pulsed neutron sources

The neutron flux shape is recorded by a number of detectors placed at various distances from the source.



The shape of the phantom simulates both the neutron emission caused by the interaction of an azimuthal or radial deuteron component with the dense plasma target (the two peaks at the beginning of the emission) and by the interaction of an axial deuteron beam with a dense plasma target (the peak in the latter phase of emission).



#### Phantom (test distribution)

• The rectangle shape of the three spikes is used to evaluate the <u>shape distortion</u> inherently caused by the limited view angle domain.

• Equal height of spikes monitor reconstruction with <u>erroneous</u> <u>heights</u>

• The <u>resolution of the shape</u> <u>reconstruction</u> is monitored by the size of the spikes and also by the relative shifts and gaps between spikes.

• A background of 10% of the spike's height is superimposed to appreciate the quality of signal to noise ratio reconstruction

Numerically simulated tomographic projections







#### Maximum entropy - ME

Algebraical Reconstruction Technique - ART





### The **SPEED2** operation parameters:

### Discharge 8778

- capacitor bank voltage: U=180 kV, W=57 kJ
- deuterium pressures: p=4.25 hPa
- total neutron yield per discharge Yn =2.45 10^10 neutrons;

The time-of-flight diagnostics system: 4 scintillator-photomultiplier detector assemblies placed at distances:

- ≻ 1.85 m
- ≥ 9.65 m
- ≻ 20.15 m
- ≥ 29.75 m



## Experimental data (tomographic projections)

#### **Discharge 8778**

Data analysis and interpretation

Superposition of a relatively broad spectrum (700-800 keV full width) with a dominant peak at about 2.5 MeV, arising at about 25-30 ns from the neutron emission start. Two-component deuteron distribution function (with an average deuteron energy of 100 keV): -a quasi-axial component (producing the 2.5 MeV peak) -a radial-azimuthal component (producing the broad spectrum) Relaxation of the two-component ion distribution by collisions with plasma and background gas  $\Rightarrow$ neutron emission in the later part of the pulse



Tomographic reconstruction. The experimental conditions and the numerical procedure provided an energy resolution of 25 keV and a time resolution of 2.5 ns.

## **X-RAY MICROTOMOGRAPHY FACILITY**

#### Goal:

to provide an universal tool for the NDT analysis of fusion material samples (structural and breeders) before and after various tests (including irradiation).

#### **Applications:**

•3D modelling of miniaturized samples;
•density variations, micro-cracks formation by mechanical/thermal cycling;
•percolation and pore network connectivity in porous materials;
•structural integrity of various components;

•high magnification microradiography for in-situ inspection of samples under mechanical and/or thermal stress.

#### **Overall performances**

- Magnification < 2000
- Spatial resolution  $\cong 5 \mu m$
- Density resolution > 0.5%
- -3D Reconstruction Time  $\cong$  2 min.

the state is the state for

(512x512x512 voxels)

-Probe dimensions: Diameter < 40 mm Height < 500 mm A computer tomograph is configured to take many views of an object in order to build a three-dimensional (3-D) model of its internal structure. 2-D slices through the object volume can be viewed as images, or the 3-D volume may be rendered, sliced, thresholded and measured directly.



The microtomography facility was designed and constructed with European Community support (EFDA Newsletters: Vol. 2003/2, April 5, 2003 ,Vol. 2003/6 Dec. 2003)

#### **INFLPR UPGRADED X-RAY subMICROTOMOGRAPHY FACILITY**



#### X-ray source

- Max. 225 kV
- Focus spot <0.8μ</li>
- Detector
  - a-Si flat panel
  - 1210x1216 pixels
  - 0.1x0.1 mm<sup>2</sup>
- High precision six-axis manipulator

## HIGH END X-RAY MICROTOMOGRAPHY FACILITY



#### X-ray source

- Max. 225 kV
- Focus spot 5-200μ
- Detector
  - a-Si flat panel
  - 2048x2048 pixels
  - 0.2x0.2 mm<sup>2</sup>
- High precision up to seven-axis manipulator

## **Advanced Computed Tomography System for the Inspection of Large Aluminium Car Bodies**



3D CT of an aluminum laser weld









Rivet joint extracted from a car body

## Space resolution benchmark (old facility)

#### Gold foil spiral (foil thickness – 5 µm)



#### Metallic catalyst sample (thinnest wire - 3 µm)



Optical microscopy

Current facility: Nanofocus tube (<0.4 µ ...,

X-RAY MICROTOMOGRAPHY of the IFMIF HFTM IRRADIATION CAPSULE

#### **IFMIF - International Fusion Materials Irradiation Facility** 3-D tomography of miniaturized samples

#### Radiography

### Density plot

Sagital view

nunnun,

SUS traction probe 2 mm diameter

Calibration sample: Cu tube 2.2 mm, Ag central wire 0.5 mm and coiled kanthal wires 0.18 and 0.08 mm

Push-pull fatigue specimen

Steel bellows of 6.5 mm



## **IFMIF - International Fusion Materials Irradiation Facility**

Assessment of the structural integrity of a prototypical instrumented IFMIF high flux test module (HFTM) rig by fully 3D X-ray microtomography















Tomography reconstruction of the HFTM irradiation capsule: longitudinal cross-section (left - microfocus tube, 225 kV, right - 450 kV tube, and middle – a comparison with its CAD model.



Tomographic reconstruction for optimum combination of the irradiation parameters and enhanced magnification for detail visualization. (microfocus tube, U= 220 kV, voxel size = 60m)

## **IFMIF - International Fusion Materials Irradiation Facility**

Assessment of the structural integrity of a prototypical instrumented IFMIF high flux test module (HFTM) rig by fully 3D X-ray microtomography (con't)



Tungsten wire Heater clad Groove

Tomographic cross-section that illustrates the gaps (lack of thermal contact) between the heater coils and the groove channels. Heater diameter approx. 1 mm, tungsten wire 100 microns diameter. Microfocus tube, U= 220 kV, I = 300A, voxel size = 60m

## **Steel sample with brazed heaters inside – IFMIF HFTM**

B:/Teil2-2\_20060327114451/Teil2-2\_20060327114451.v





30 mm



## Emission tomography facility for IFMIF irradiated capsules: technical concept

Established by numerical simulations and validated by experimental tests

Reconstruction of transversal (left) and longitudinal (bottom) cross-sections of a numerically simulated test capsule



Experimental test phantom (10 x 10 cm Plexiglas with rods surrounded by a volume, non-uniformly filled with <sup>131</sup>I liquid solution) The whole gamma spectroscopy chain and the collimating configuration were accurately described by full 3-D Monte Carlo simulations.

Emission tomography measurement tests were carried out on an adapted gamma-scanning system in the Hot-Cell facility at the Institute for Nuclear Research, Pitesti, Romania.

The samples were selected to simulate the IFMIF irradiation capsule.

Both the simulated and measured tomographic projections were processed within a proprietary reconstruction program based on the criterion of maximum entropy.

The design parameters for space resolution and isotope selectivity are well within reach.

X-RAY MICROTOMOGRAPHY for ITER

## ITER- Compressed pebble bed

3-D tomography reconstruction insert: frontal cross-sections of the bottom zone showing pebbles in contact with the steel plate





Frontal cross-section (top) axial crosssections through contact area between steel plate and aluminum pebbles (bottom-left) and through middle of bottom layer of aluminum pebbles (bottom-right)

## CFC-JET/ITER - Tomography

#### Graphite



Sample sectioned to about 4 x4x4 mm<sup>3</sup>: 3D tomography reconstruction and axial cross section Space resolution 18 microns/ image pixel, minimum detectable feature 15-20 microns

## **ITER- Welded Steel Pipe with Cu cables**

![](_page_22_Picture_1.jpeg)

![](_page_22_Picture_2.jpeg)

Microtomography measurements with 17 microns space resolution

## Welding steel pipes: offset position of the pipes

![](_page_23_Picture_1.jpeg)

## **3D X-ray micro-tomography**

3D X-ray micro-tomography ( $\mu$ CT) for to the characterization of structural integrity of Nb3Sn superconducting wires of differing topology

 Detection of the manufacturing defects and anomalies inside the complex structure
 Filamentary Twist Pitch factor evaluation

The evolution of the density distribution of ceramic samples manufactured by Field Assisted Sintering Techniques (FAST)

sinterized Ni powder and high density MgB2 superconductors

#### X-ray microtomography on ITER Type Nb3Sn superconductor wires

![](_page_25_Picture_1.jpeg)

![](_page_25_Picture_2.jpeg)

![](_page_25_Picture_3.jpeg)

Oxford Instruments (OST), for ITER Type I 0.81 mm (NbTi)3Sn strand, 19 subelements), Single Ta barrier, Cu:non-Cu ratio 1

#### OST strand of 0.7 mm Nb3Sn filaments

![](_page_25_Picture_6.jpeg)

![](_page_25_Picture_7.jpeg)

![](_page_25_Picture_8.jpeg)

![](_page_25_Picture_9.jpeg)

Voxel resolution (from left to right): 3 µm, 1.7 µm & 0.8 µm

### Innovative method for non-invasive twist-pitch measurement

![](_page_26_Picture_1.jpeg)

![](_page_26_Picture_2.jpeg)

Filaments evolution inside the crosssections, revealed after filtering and segmentation.

![](_page_26_Figure_4.jpeg)

![](_page_26_Figure_5.jpeg)

Illustration of the method for twist-pitch calculation

![](_page_26_Picture_7.jpeg)

Extraction of the central filaments (left) and of the Ta sleeve (right).

![](_page_26_Figure_9.jpeg)

The evolution of twisted structure revealed by screwing together successive tomographic reconstructions TP=15.2  $\pm$  4% mm

Traditional vs. non-invasive methods for measuring the filamentary twist pitch (TP) parameter

![](_page_27_Picture_1.jpeg)

![](_page_27_Picture_2.jpeg)

The traditional method for measuring the filamentary twist pitch (TP) parameter consists of a 0.5 mm lamination followed by etching the copper in a nitric acid solution, thus evidencing the twisted structure despite of the Ta barrier.

> Results TP=14.7 ± 1 mm (traditional) TP=15.2 ±4% mm (non-invasive) Manufacturer specifications 15 mm

![](_page_27_Figure_5.jpeg)

Voids formation in MgB2 superconducting wires

![](_page_28_Picture_1.jpeg)

![](_page_28_Picture_2.jpeg)

![](_page_28_Picture_3.jpeg)

![](_page_28_Picture_4.jpeg)

![](_page_28_Picture_5.jpeg)

## Structure and morphology of high density MgB2 superconductor

•High density superconducting MgB2 (undoped and doped with SiC and B4C) is prepared using FAST (Field Assisted Sintering Technique) from very porous and mechanically weak mixture of Mg and B.

•Although, at present, it is not possible to significantly advance the understanding of the milling-properties relationship,  $\mu$ CT can reveal clear differences between the samples.

•For our particular bulk MgB2 superconducting samples,  $\mu$ CT allowed observation of certain 3D density patterns and their evolution with the milling time of the precursor mixtures.

•We shall emphasize that one important limitation of the  $\mu$ CT is that it cannot give any information on crystal quality and composition.

## MgB2 samples and processing conditions

The second se			
Sampl e	Mixing/millin g	Heat treatmen t temp. (°C)	Heat treatmen t time (h)
А	Manual in Ar	700	lh
В	Mechanical milling in Ar, 0.5h	700	1h
C, C1	Mechanical milling in Ar, 1h	700	lh
D, D1	Mechanical milling in Ar, 3h	700	lh
Е	Mechanical milling in $H_2$ , 0.5h	700	lh
F	Mechanical milling in $H_2$ , 1h	700	lh
G	Mechanical milling in H <sub>2</sub> , 3h	700	lh

![](_page_30_Picture_2.jpeg)

Representative  $\mu$ CT images of the heat treated bulk samples. Images were taken for a voltage of 40KV and a current of 100 $\mu$ A, sampling rate 3.87 $\mu$ m. Scale bar is 100 $\mu$ m.

#### **Summary**

X-ray micro-tomography may represent an appropriate solution for the microstructural investigation of relevant material issues as identified during manufacturing processes in a number of cases when the traditional techniques are not sensitive enough or are not the most appropriate to reveal the key characteristics of the investigated advanced materials. Neutron emission tomography for limited data set at JET tokamak

#### KN3 tomography

![](_page_33_Picture_1.jpeg)

Highly undetermined problem:

- 19 equations
- 35 X 20 unknown

(90 mm pixel size)

#### Maximum likelihood solution

The emission is assumed to be a Poisson process, and **P**<sub>km</sub> is a sample from a Poisson distribution whose expected value is:

$$\sum_{i} w_{ik} f_i$$

Then, the probability of obtaining the measurement **p** if the image is **f** is the so-called likelihood function:

$$L(P/F) = \prod_{k} \left[ \frac{1}{p_{k}!} \left( \sum_{i} w_{ik} f_{i} \right)^{p_{k}} \times \exp\left( -\sum_{i} w_{ik} f_{i} \right) \right]$$

#### Smoothing

Smoothing in magnetic surfaces may supply the lack of information.

![](_page_33_Figure_13.jpeg)

![](_page_33_Figure_14.jpeg)

median filtering using a circulating window along the magnetic contour lines

Additional smoothing can be obtained by resampling the experimental projection. Projection resampling implies the introducing of virtual lines of sight which ensures an improved coverage of the reconstruction domain

### Testing the method - phantoms

![](_page_34_Figure_1.jpeg)

Unfolding and Tomographic Techniques for Fusion Applications - TFD Tech. Meeting 1-2 July 2008

#### Information about the temporal evolution of the neutron emissivity

![](_page_35_Figure_1.jpeg)

Temporal evolution of the D-T 14 MeV neutron emissivity in a trace tritium experiment showing the relaxation of the high density plasma, which is heated by D neutral beams after the T-puff - shot 61141 (from 60.87 s to 61.32 s).

#### Nuclear waste management: Combined 3D tomography and X-ray fluorescence for host rock

Main goal of research was to implement methods for correlation of the host rock structure and composition

- structure determined by X-ray tomography
- composition determined by X-Ray fluorescence
- structure & composition determined by Dual Energy Tomography
- ➢Fabrication of a microbeam fluorescence facility for surface composition mapping (20 µ resolution)

Implementation of innovative combined transmission-fluorescence tomography method

#### X-ray tomography measurements on Grimsel sample

- 2D CT for full sample diameter FOV
- 3D µCT for small sample

### 2D CT cross section:

high penetration, low space resolution

Scanning parameters: Linear array 1024 pixels x 0.4 mm pitch Scanning method translate-rotate U= 300 kV, I=2.0 mA 1.5 mm Brass pre-filter Reconstruction matrix: 500x500 Voxel size: 0.25x0.25x0.8 mm<sup>3</sup>

![](_page_37_Picture_6.jpeg)

![](_page_37_Picture_7.jpeg)

**3D μCT and axial cross section** *Scanning parameters:* Flat panel array 1220x1216 pixels x 0.1 mm pitch scanning method rotate-rotate U= 100 kV, I=0.08 mA Cd & Cu pre-filter Voxel size: 46x46x46 μ<sup>3</sup>

#### Fully 3D macro CT : high penetration, low space resolution Scanning parameters: Source Bosello 450 kV , focus size 0.3 mm U = 370 kV, I=1.5 mA, 720 projections full scan, Prefilter Brass 2 mm, Detector PE 1640 AN1 ES CT – 1024x1024 pix2, 16 bits, 0.4x0.4 mm2 Source – Object distance : 519.75 mm Source – Detector distance : 1505.3 mm Magnificattion 2.9 Voxel size 0.138x0.138x0.138 mm3

![](_page_38_Picture_1.jpeg)

![](_page_38_Picture_2.jpeg)

![](_page_38_Picture_3.jpeg)

![](_page_38_Picture_4.jpeg)

![](_page_38_Picture_5.jpeg)

#### **METHODICS DEVELOPMENT** Micro-cavities extraction procedure

![](_page_39_Picture_1.jpeg)

![](_page_39_Picture_2.jpeg)

Tomography reconstruction of a crystalline rock axial, frontal and sagital cross-section. The region of interest is marked in green and the cavity is extracted by a sequence of image processing algorithms that finally provides the 3D cavity boundaries. High-Resolution Cone Beam Tomography for Two-Phase Flow Diagnostics

## Cone-Beam CT for two-phase flow

![](_page_41_Picture_1.jpeg)

## Upward bubbly flow 7 nozzle injection β=3.25%, jl=1.8 m/s, Height = 40D

![](_page_42_Figure_1.jpeg)

Ultra fast X-Ray Computed Tomography for Transient Phenomena

![](_page_44_Figure_0.jpeg)

## Construction of Fast X-ray CT

![](_page_45_Figure_1.jpeg)

## Instantaneous Bubble Interface Visualized by Fast X-ray CT

![](_page_46_Picture_1.jpeg)

**Bubbly Flow** 

**Slug Flow** 

![](_page_46_Figure_4.jpeg)

#### Void Fraction and Integrated Bubble Perimeter in Flow Channel Cross Section

![](_page_47_Figure_1.jpeg)

## Experimental Parameters in Fluidized Bed Test

Fluidized Bed Composition (from T Kai with Univ of Kagosh	ima)	1.43
SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub>	(13% : 87% by weight)	1
Particle Diameter:	36 mm (average) 46	2
Solid-alone density :	2600 kg/m <sup>3</sup>	145
Porous density:	980 kg/m <sup>3</sup>	200
Loosly-packed densit	ty: 560 kg/m <sup>3</sup>	
Fluidized Bed Test Section	LAP 11 1 1 1 1 1 1 1 1	100
Orientation:	vertical	22
Static height:	860mm X-ray	644
Tube Outer Diameter:	: 50mm 8	360
Tube Inner Diameter:	46mm	
CT-Measuring Height	:: 600mm	1
jg=0.005 - 0.07m/s	Lo 9 24 5 9 24 5 7 24 5	1
X-ray CT Scanner	The second a second a second	E.
X-ray power	100kV, 5mA (Nominal)	145
Vacuum:	1.2x10 <sup>-6</sup> Torr	de.
ROI elevation	L/D=13.0	ti A
Measuring Period	2 sec. (500 slices)	123

## "Bubble" Interface in Alumina Powder

![](_page_49_Figure_1.jpeg)

jg=0.04m/s.

jg=0.07m/s.

Gamma Emission Tomography for Nuclear Fuel Engineering

## Principle of Emission Tomography Maximum Entropy implementation

![](_page_51_Figure_1.jpeg)

 $\Psi = H - \sum_{k=1}^{N} \sum_{m=1}^{M} L(k,m) \cdot \left[ p_{km} - \int_{r(m,k)}^{r(m,k+1)} ds \cdot \int_{-\infty}^{\infty} f(s \cdot \cos(\theta_m) - u \cdot \sin(\theta_m), s \cdot \sin(\theta_m) + u \cdot \cos(\theta_m)) du \right]$ 

## Emission Tomography Equipment

![](_page_52_Picture_1.jpeg)

![](_page_52_Picture_2.jpeg)

High purity Germanium detector: 18 % Efficiency
Adjustable slit collimator. Height: 5 / 50 mm Width: 0.10/0.25/0.50
Scan device with motion control (ACB). Step motors. Axial/Radial/Rotational.
Multichannel analyzer (ORTEC). Gamma spectra acquisition.
Personal computer. Motion & Acquisition Control. Spectrum analysis.
Tomography.

## Cs-137 Profile "

![](_page_53_Figure_1.jpeg)

![](_page_53_Figure_2.jpeg)

CANDU FUEL - Cs-137 migration
large depression activity in the middle of the rod
accumulation of Cs-137 in the last mm of the rod

lack of pellets interfaces Cs-137 Axial Profile

how we we walk have more thank the water when the should be a second the second second the second se

![](_page_54_Figure_0.jpeg)

Current projects FEMaS (FP7) CFC-JET/ITER Tomo-Analytic CT Metrology CT Mobile (FP7) CERN CT PCB manufacturing

#### http://tomography.inflpr.ro

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#### PCB manufacturing X-ray tomography

![](_page_59_Picture_1.jpeg)

![](_page_59_Picture_2.jpeg)

![](_page_59_Picture_3.jpeg)

![](_page_59_Picture_4.jpeg)

Flip chip bonded solder ball (Pb-Sn), with 10 micron fracture by thermal stress

FacilitySpring-8, Oblique View Parallel BeamX-ray energy29keVDetectorCCD, 2000x1312 pixelsPixel size0.47 micron meter equivalent, optical magnificationx20 after scintillator (actual magnification x23.6)360 deg., 1.0 deg steps (0.05 deg. step data available)Image format16bit TIFF, no compressionTomography angle70deg. (60deg. data available)

Experimental setup

![](_page_60_Figure_3.jpeg)

2000x1312 pixels input images Reconstruction 1200x1200x201 0.47 µm voxel size

![](_page_61_Picture_1.jpeg)

![](_page_61_Picture_2.jpeg)

One can see the biased distribution of Pb phase in Sn balls

## Computer-aided tomography (µCAT)

 $\mu$ CAT systems are configured to take many views of the object in order to build a 3-D model of its internal structure. 2-D slices through this volume can be viewed as images, or the 3-D volume may be rendered, sliced, and measured directly.

For the NDT inspection of miniaturized samples the microtomography analysis is guaranteed for feature recognition down to a few tens of microns. 3-D tomographic reconstructions are obtained by a proprietary highly optimized computer code based on a modified Feldkamp algorithm.

## The microbeam fluorescence (µXRF)

component is a configurable film thickness and composition measuring tool. It include mechanical or optical X-ray beam collimation options, Amptek PIN diode X-ray detector and analysis software, motorized micrometric x-y-z stage for accurate sample positioning. A visual image of the sample is synchronized with the  $\mu$ XRF scanning in order to pinpoint measurement location, as well as for historical documentation.

![](_page_62_Figure_5.jpeg)

#### Overall performances

#### Microtomography

Spatial Resolution ≅ 20µm Density Resolution > 1 % Probe Dimensions: Diameter < 40 mm, Height < 200 mm Reconstruction time ≅ 5 min

#### Microfluorescence

Spatial Resolution ≅ 20µm Thickness Resolution ≅ 2 % of total layer Probe Dimensions: 100×100 mm<sup>2</sup>

# 2D composition mapping by micro-beam fluorescence

![](_page_63_Figure_1.jpeg)

The XRF analysis with fundamental parameters converts elemental peak intensities to elemental concentrations and/or film thicknesses - this enable composition mapping

![](_page_63_Picture_3.jpeg)

![](_page_63_Picture_4.jpeg)

## CFC-EK386

![](_page_64_Figure_1.jpeg)

## Radiography and Tomography results for JET CFC samples

## Graphite

![](_page_64_Figure_4.jpeg)

## CFC-NB31

![](_page_64_Picture_6.jpeg)

![](_page_64_Picture_7.jpeg)

![](_page_64_Picture_8.jpeg)

![](_page_64_Picture_9.jpeg)

![](_page_64_Picture_10.jpeg)

## Reconstruction of a Sepia cuttlebone

![](_page_65_Picture_1.jpeg)

Structural elements – septal thickness, pillar spacing and chamber height in the range of 5  $\div$ 200 µm