NON-DESTRUCTIVE ANALYSIS OF FUSION MATERIALS SAMPLES BY MICROTOMOGRAPHY

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1. Reference design for transmission micro-tomography system and underlying tests for confirmation

According to the current status, all milestones of the IFMIF subtask "Micro-tomography design, fabrication and test" were successfully accomplished during the year 2002 (EFDA Newsletter, Vol. 2003/2, April 5, 2003, and December). This allows us to provide the fusion materials community with a unique instrument for NDT inspection of individual miniaturized samples as well as for verification of irradiation capsules integrity. Due to its penetration ability and contrast mechanism, X-ray microtomography is the only known tool that has the ability of monitoring voids, micro-cracks and flaws in activated and completely assembled rigs, test modules and the Li-target back-wall.

Extensive NDT inspection of fusion materials miniaturized samples using the transmission micro-tomography system in order to establish the reference design for the micro-tomography system for IFMIF was performed. In addition to the inspection of miniaturized samples (presented elsewhere) we carried out micro-radiography/tomography studies of instrumented capsule mock-ups in order to determine the accurate positioning of the specimens and thermocouples and the NaK level.

As a typical example we present here the results obtained for probes realistically simulating two test capsules which were constructed and measured in our laboratory (Figure 1). Based on these results one can conclude that we are in a position to accurately assess the structural integrity of the IFMIF High Flux Test Module (HFTM) irradiation capsules.





Figure 1.a 3-D reconstruction (top) of the test capsule (bottom). SUS specimens and thermocouples are clearly identified.





Figure 1.b 3-D reconstruction (top) of the square test capsule (bottom). Kanthal wires 0.018 mm introduced inside of two out of three thermocouples are clearly identified.

In order to establish the reference design, special attention was also paid to the analysis of samples which feature structures of a few microns in magnitude. Very good reconstructions were obtained for these samples (Figure 2).

An effort was also made for the development of new methods to cope with the challenging problem of beam hardening in high density materials. Fully 3D Monte Carlo radiation transport simulations combined with validation experiments represent our main tools in approaching these problems.

Currently we are performing a comparative study which involves our facility and a high end tomography system from abroad. The goal of this study is the assessment of the dependence of the reconstruction quality on the X-ray energy (up to 225 keV), detector area (up to 400x400 mm²) and its digital output (up to 16 bits).

The micro-tomographic facility is available for cooperation between the EURATOM Associations within the framework of the EFDA Technology Workprogramme 2004 and 2005. These activities will be focused on implementation of suitable NDT inspection methods for the structural integrity assessment of instrumented capsules and rigs by micro-tomography and experimental validation of real time micro-radiography of miniaturized samples under mechanical stress.



Porosity characteristics of sand sample (minimum detectable feature 5 µm)



Reconstruction of a *Sepia* cuttlebone (structural elements – septal thickness, pillar spacing and chamber height – are features in the range of $5 \div 200 \,\mu\text{m}$)

Figure 2 3 D reconstructions revealing the ability of the tomographic system to detect features down to few microns.

2. Emission tomography conceptual design and preliminary tests

Gamma Emission Computed Tomography (GECT), is a non-destructive technique for generating quantitative cross-sectional images of industrial objects and in particular for the visualization of miniaturized samples and the internal structure of irradiation capsules by monitoring the gamma emission from the neutron activated isotopes.

A problem which arises in GECT for neutron activated samples analysis is the relatively large time necessary for the acquisition of experimental data: the sample is placed in a hot cell and its gamma radioactive content is measured via a long collimator which determines a small detection efficiency. For this reason only a few number of projections (views) of the two-dimensional isotope distribution, which is reconstructed by the GECT algorithms, are available in a reasonable time. These characteristics set this tomographic problem apart from the transmission tomography (where several hundreds projection angles can be easy achieved). Therefore specific tomographic methods (based on the use of a supplementary criterion to compensate the lack of information) must be adapted/developed and evaluated. In the present work a method based on the maximum entropy (ME) algorithm for GECT reconstruction was developed, evaluated and implemented. The maximum entropy method provides good results when the number of views is

small and has several advantages: removes detrimental effects caused by the missing projections, suppress streaking artefacts, gives the smoothest achievable reconstructed image by eliminating details which are not significantly above noise and so flattening artefacts that arise from systematic errors. A large number of computer experiments were performed in order to validate the ME algorithm. Good reconstructions of various phantoms (simulated objects), with shapes extensively used in this kind of tomography, were obtained for the situation of a strong attenuation and a small number of projections (Figure 3.a).



Figure 3.a – Reconstruction of various phantoms (simulated objects).



Tomographic reconstruction ¹³⁷Cs (662 keV) distribution in the crosssection of a CANDU-type fuel rod. 15 projection angles, 50 x 50 pixels



The reconstruction of an experimental test phantom - 15 projection angles and 256 x 256 pixels;



Axial gamma profile of a spent ⁶⁰Co (1173 keV and 1332 keV) sealed sources recovered from nuclear medicine

Figure 3.b - Reconstruction of real gamma-radioactive samples

Several experiments were performed on real gamma-radioactive samples in the hot-cell laboratory (HCL) of the Institute for Nuclear Research, Pitesti, where techniques of postirradiation examination and also a variety of facilities for handling and examination irradiated structural materials are available. An effort was made to adapt the existing gamma-scanning system to a suitable tomographic geometry. A mechanical system for rotating the collimator was designed and constructed in order to measure the tomographic projections by radial scanning of the sample (Figure 4).



Figure 4 – The adapted tomographic system: details on the slit width selector

In Figure 3.b we present the results obtained for: i) a CANDU type fuel pin (sintered uranium dioxide $5.7\%^{235}$ U, diameter=12.15 mm., cladding material: Zircaloy-4, 0.4 mm thickness - irradiated during a period of 9.5 months), ii) an experimental test phantom (10 x 10 cm Plexiglas with circles with dimensions varying from 0.8 cm to 4.0 cm surrounded by a volume, non-uniformly filled with ¹³¹I liquid solution). The ability of the system to analyse neutron activated samples is demonstrated by an experiment performed on a spent ⁶⁰Co source for medical radiotherapy (400 mCi). This sample was investigated by axial gamma scanning using an aperture of 0.1 mm for the slit of the collimator. The axial profile reveals the structure of the source, composed by 9 slices of 2.0 mm.

Neutron activation calculations of the fusion structural materials for the IFMIF neutron spectrum were used for the assessment of the design parameters (space resolution, acquisition time, isotope selectivity) of the emission tomography system. By fully 3-D Monte Carlo simulations (Tiger/SNL, MCNP/ORNL) we accurately described the whole gamma spectroscopy chain as well as the collimating configuration. For the desired space resolution (50÷200 μ m) the acquisition of gamma spectra for tomographic reconstruction needs ~30 minutes if the newly developed energy selective linear detector array based on a CdTe semiconductor associated with a multithread collimator is used.

3. Conclusions

The reference design for the transmission micro-tomography system was established for IFMIF environment conditions. Analyses of samples of fusion materials miniaturized samples were performed. An effort was also made for the development of new methods to cope with the challenging problem of beam hardening in high density materials.

Based on the results obtained for probes realistically simulating HFTM irradiation capsules one can conclude that we are in a position to accurately assess their structural integrity. Conceptual design of an emission tomographic system for miniaturized samples and irradiation capsules is established by numerical simulations and validated by experimental tests. The reconstructions presented in this report show that the design parameters for space resolution and isotope selectivity are well within reach. This encourages us to strongly propose the fabrication of an emission tomograph prototype in the EVEDA phase of the IFMIF project.

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