Nano/Micro-Scale Imaging and Metrology by Electron and X-Ray 3-D Tomography of Innovative Materials - NIMETIM

Contract 2-CEEX 06 -11-23/2006
Project Director: Dr. Ion TISEANU

The main objective of the NIMETIM project was the development of methods and devices for X-ray tomography analysis with submicron resolution. In order to achieve this goal one have pursued the following paths:

O1. Development of sub-micron X-ray quantitative tomography techniques; design and fabrication of a nanoCT facility; development of standards for performances assessment of sub-micron spatial resolution tomography facility.

O2. Extension of the functionality of the TEM facility to 3D tomography with spatial resolution of tens of nanometres for polymeric composites and mesoporous materials.

O3. Application of the newly developed methods and instruments to the characterization of advanced materials and devices (superconductors, biomaterials, ceramics, nano/microelectronic devices, etc.).

Fig. 1 shows the newly developed X-ray tomograph with submicron resolution. Its overall parameters are listed in Table 1.

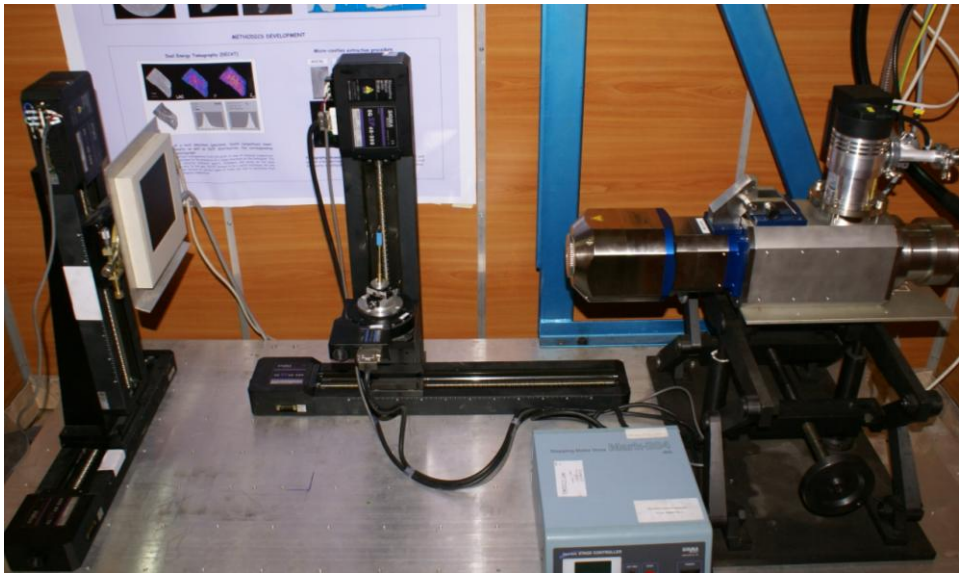

Figure 1 – X-ray tomography system with submicron resolution

The key component is a multifunctional X-ray source operated in micro- and nanofocus mode. The main parameters of the nanofocus tube are: maximum high voltage 225 kVp, maximum power 10/15 W depending on the irradiation head. Three type of sensors are employed for the X-ray detection: Image Intensifier (digital Output 10 bits) for fast inspections, amourphous silicon (a-Si) flat panels with more than one million pixels and pitch size of 48 or 100 µm (digital output of up to 14 bits) and a 1024 pixels linear array. The linear array detector is based on individual scintillator-photodiode with a very good digital output (16 bits) and it is devoted to tomography analysis of high density samples due to its large dynamic. Accurate sample positioning is done with a set of 5-6 computer controlled motorized stages. The acquisition and control software were realized within the NIMETIM project.

An effort has been done for the implementation of proprietary tomography reconstruction software. A modified Feldkamp algorithm has been parallelized for the processing of large image matrices (>1220x1216 pixels) towards the reconstruction of very large volumes (1024x1024x1024 voxels).

Thus we have demonstrated that one can reconstruct such a volume within 20 minutes on a 64 bit PC with 4 CPUs using 720 projections of 1600x1200 pixels as input data. The reconstruction software is designed to be hardware independent and it includes several features for visualization and virtual

navigation within the 3D reconstructed volume. In addition to both, parallel beam and cone beam scanning configurations a host of state of the art methods of tomography artefact compensations. have coded. One can note here the implementation for the first time of multi-material, multi-energy techniques in the frame of microtomography.

Table 1 – Overall parameters of the X-ray nonotomograph

	Overall parameters
X-ray source with micro- and nanofocus operation mode	Max. inalta tensiune: 225 kVp Putere max.: 10 W Detaliu minim detectabil: ~ 200 nm
Detectors	768 x 576 *Image Intensifier* 1248 x 1248 a-Si *flat panel* Liniar array 1024 pixels
Output Digital	Up to 16 bits
Micrometric motorized stages Axis X Axis Z Axis θ Fine adjustment of sample positioning	travel 800 mm, loading up to 50 kg travel 300 mm, loading up to 6 kg accuracy 0.02°, loading up to 30 kg accuracy 2µm, reproducibility 1µm, loading up to 2 kg
Magnification factor	< 2000
Space resolution	≥ 500 nm
Scanning time	< 15min. (720 views)
Reconstruction time 3D	< 20 min (1024x1024x1024 voxels)
Scanning methods	*Cone* beam CT Short scan (180° + 1/2 *fan angle*) Oblique View Cone Beam

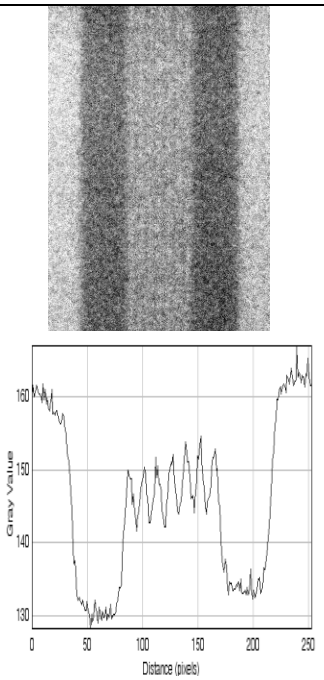

Figure 2 – Digital radiography of the JIMA mask (top); transversal profile on the 0.5 µm bar structure (bottom)

The demonstration of the submicron resolution of the newly developed tomograph has been done in two steps. Firstly, the space resolution of the source-detector assembly was measured with the Japan Inspection Instruments Manufacturers Association (JIMA) mask sample. Fig. 2 clearly demonstrates that the 0.5 µm JIMA bar structure is fully resolved in the digital radiography and the associated profile.

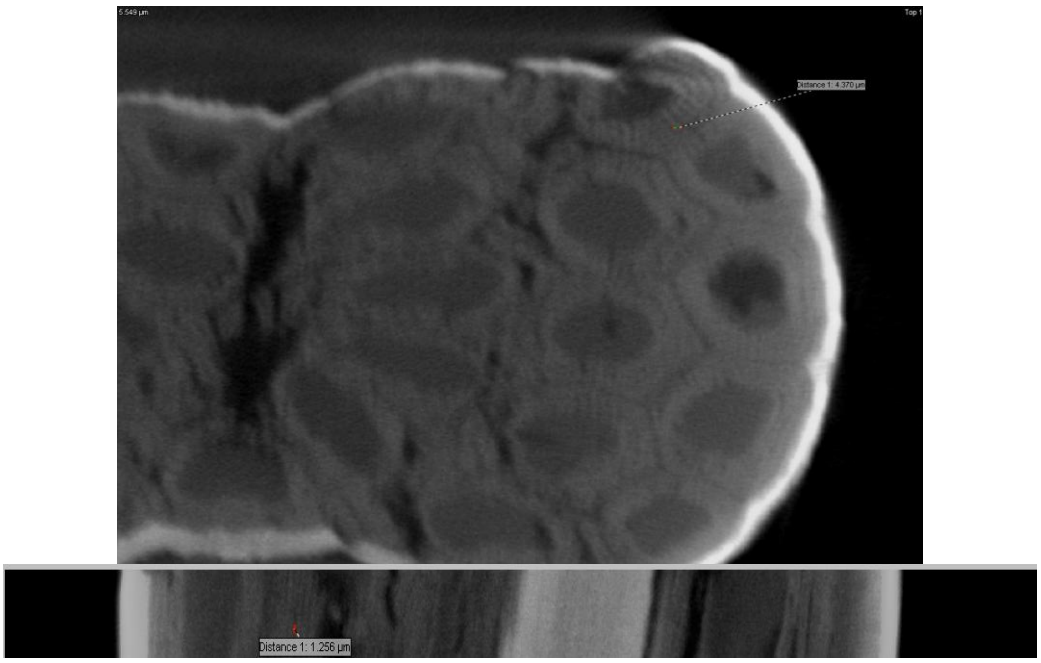

Figure 3: Tomographic cross sections on Nb3Sn ITER strand where submicron morphology is evidenced.

Finally, the validation of the system has been performed by tomography measurements on Nb3Sn superconductor strands. The role of this study is the modeling of the transport properties of the Nb3Sn wire in order to optimize the fabrication process in terms of the hysterezis losses, critical current and Cu/non-Cu ratio as stipulated by the technical requirements of the ITER fusion reactor.
Fig. 3 illustrates the extraordinary resolving power reached by the instrument as one can reveal for the first time on a conventional tomograph the detailed structure of the Nb filaments.

Electron tomography for materials science is developing, using TEM images (predominantly incoherent signals), as the basis for the tomography tilt series in order to reveal the 3D structure and composition of nanoscale objects. This required the upgrade of the image acquisition module using a high resolution CCD camera and of the goniometer for automatic control. Two different software packages and several reconstruction algorithms dedicated for the TEM-3D reconstruction were extensively tested. The experimental validation of the TEM-3D methodic has been carried out on a TEM Jeol 200CX microscope by acquiring few sets of tilted images with tilt angles between -65° to +65° in steps of 1-2°. Biopolymers and amorphous oxides materials were selected as typical samples.

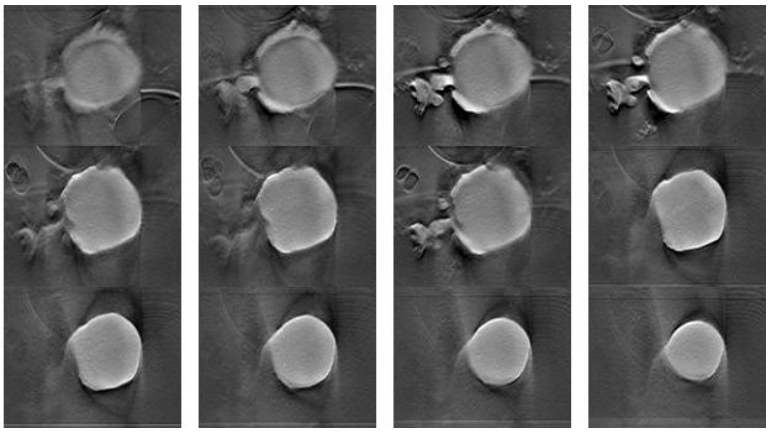

Figure 4 - TEM-3D filtered-backprojection tomography reconstruction of a polymer nano-capsule – the axial cross-sections are sampled at 10 voxels interval.

In Fig. 4 one introduces a set of tomography cross-sections of a polymer nano-capsule. The space resolution achieved of around 20 nm is well within the preset goals of the project. Also one have demonstrated that the alignment and reconstruction software on one hand and the acquisition and control software on the other hand are smoothly compatible.

In conclusion the objectives of the project were fully achieved by:
Design, construction and experimental validation of an X-ray transmission tomographic system with submicron resolution. It is based on the cone-beam scanning configuration and a modified Feldkamp reconstruction algorithm and it provides a host of state of the art methods of tomography artifacts compensations. One can note here the implementation for the first time of multi-material, multi-energy techniques in the frame of microtomography.
The extension, for the first time in our country, of the functionality of the TEM facility to fully 3D electron tomography with spatial resolution of few tens of nanometres, applied mainly for polymeric composites and mesoporous materials.
Finally, the newly developed methods and devices have been embedded in the non-destructive analysis of advanced candidate materials for the ITER fusion reactor.

The members of the NIMETIM consortium have published the following list of scientific papers.

Published papers with peer review (ISI Journals)

[1] Tiseanu, T. Craciunescu, T. Petrisor, A. della Corte. *3D X-ray micro-tomography for modeling of NB3SN multifilamentary superconducting wires*. Fusion Engineering and Design, 82, p. 1447–1453, 2007

[2] T. Kai, N. Okada, M. Baba, T. Takahashi, M. Misawa, I. Tiseanu, N. Ichikawa, *Structure of effective catalyst layers around bubbles in a fluidized catalyst bed*, Chemical Engineering Journal 130(2007)119-124.

[3] Augieri, G. Celentano, L. Ciontea, V. Galluzzi, U. Gambardella, J. Halbritter, T. Petrisor, A. Rufoloni, A. Vannozzi, *Angular properties of pure and Ca-substituted $YBa_2Cu_3O_{7-\delta}$ uperconducting thin films grown on $SrTiO_3$ and CeO_2 buffered Al_2O_3 substrates*, Physica C: Superconductivity and its Applications Volume 460-462 II, Issue SPEC. ISS., 1 September 2007, Pages 829-830

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[8] I. Tiseanu, T. Craciunescu, G. V. Aldica, M. Iovea. *X-ray micro-tomography as a tool for quantitative characterization of advanced materials manufacturing processes*. Advanced Materials Research, 47-50, pp. 698-701, 2008.

[9] Gh. V. Aldica, P. Nita, I. Tiseanu, T. Craciunescu, P. Badica. *High density MgB2 superconductor: structure and SEM investigations*. Journal of Optoelectronics and Advanced Materials, 4, pp. 929 - 932, 2008.

[10] A. Barau, V. Budarin, A. Caragheorgheopol, R. Luque, D.J. Macquarrie, A. Prelle, V. S. Teodorescu, M. Zaharescu, *A Simple and Efficient Route to Active and Dispersed Silica Supported Palladium Nanoparticles*, Catalysis Letters, 2008, 124:204–214, DOI 10.1007/s10562-008-9465-x

[11] . Tiseanu, T. Craciunescu, *Assessment of the Aplicability of X-Ray Tomography for Irradiated Samples*, in curs de publicare in Fusion Engineering and Design

[12] P. Badica, G. Aldica, T. Craciunescu, I. Tiseanu, Y. Ma, K. Togano, *Microstructure of MgB2 samples observed through X-ray Microtomography*, in curs de publicare in Superconducting Science and Technology

Papers published in International Conferences Proceedings

[1] Tiseanu, T. Craciunescu, T. Petrisor, A. della Corte. 3D X-ray micro-tomography for modeling of NB3SN multifilamentary superconducting wires. 24th Symposium on Fusion Technology, 11-15 Sept. 2006, Varsovia, Polonia

[2] Gh.V. Aldica, P. Nita, I. Tiseanu, T. Craciunescu, P. Badica, *High Density MgB2 Superconductor: Structure and Morphology through Microtomography and SEM Investigations*, 5th International

Conference, "New Research Trends In Material Science", ARM-5, Sibiu, Romania, 5 – 7September, 2007

[3] I. Tiseanu, T. Craciunescu, *X-ray Microtomography Experience at National Institute for Laser, Plasma and Radiation Physics, Bucharest, Romania,* Prezenata la Fachtagung Prozessnahe Röntgenanalytik Berlin, Germany, 15-16 November 2007

[4] M. Simon, I. Tiseanu, C. Sauerwein, S.-M. Yoo, I.-S. Cho, *Development of multi sensor and multi source computed tomography Systems,* DIR 2007 - International Symposium on Digital industrial Radiology and Computed Tomography, June 25-27, 2007, Lyon, France

[5] M. Simon, I. Tiseanu, C. Sauerwein: *Characterization of Automotive Parts by a Novel Multi-Scan Tomography System*, 4th International Conference Emerging Technologies in Non-Destructive Testing, April 2-4, 2007, Stuttgart, Germany.

[6] L Ciontea, G Celentano, A Augieri , T Ristoiu , R Suciu, M S Gabor, A Rufoloni, *A Vannozzi, V Galluzzi, T Petrisor, Chemically Processed $BaZrO_3$ Nanopowders as Artificial Pinning Centres*, 8th European Conference on Applied Superconductivity, EUCAS'07 Bruxelles 16-20 September 2007.

[7] L Ciontea, A Angrisani, G Celentano, T Petrisor jr., A Rufoloni, A Vannozzi, A Augieri, V Galuzzi, A Mancini, T Petrisor, *Metal Propionate Synthesis of Epitaxial $YBa_2Cu_3O_7$-x Films*, Prezentata la 8th European Conference on Applied Superconductivity, EUCAS'07 Bruxelles 16-20 September 2007

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[9] I. Tiseanu, T. Craciunescu, P. Badica, G.V. Aldica, M. Rindfleisch , *Characterization of Superconducting Wires by Cone-Beam Micro-Tomography*, acceptata pentru prezentare la *IEEE 2008 Workshop on X-Ray Micro Imaging of Materials, Devices, and Organisms*, 22-24 October 2008 Dresden, Germany

[10] M. Iovea, O.Duliu, G.Mateiasi, M. Neagu, M. Mangu, I. Tiseanu, T. Craciunescu, *Preliminary experiments for X-Ray dual-energy Micro-Tomography quantitative analysis*. Badica, G. Aldica, T. Craciunescu, I. Tiseanu, Y. Ma, K. Togano, *Microstructure of MgB2 samples observed through X-ray Microtomography*, acceptata pentru prezentare la *IEEE 2008 Workshop on X-Ray Micro Imaging of Materials, Devices, and Organisms*, 22-24 October 2008 Dresden, Germany

[11] M. Iovea, G.Mateiasi, M. Neagu, M. Mangu, O. Duliu, I. Tiseanu, T. Craciunescu, Materials microanalysis technique by dual-energy X-ray Tomography and Radioscopy, NDT 2008 15-18 September, Shrigley Hall Hotel, Nr Macclesfield, Cheshire, UK

[12] I. Tiseanu, T. Craciunescu, Assessment of the Aplicability of X-Ray Tomography for Irradiated Samples, 24th Symposium on Fusion Technology, 15-19 Sept. 2008, Rostock, Germany.