

TT-TRITIUM BREEDING AND MATERIALS
TTMI – IFMIF**TASK: TW4–TTMI–003**
IFMIF, Test Facilities

Deliverable: HFTM: Implementation of suitable NDT inspection methods for the structural integrity assessment of instrumented capsules and rigs by micro-tomography. Experimental validation of real time micro-radiography of miniaturized samples under mechanical stress.

I. Tiseanu, T. Craciunescu, N.B. Mandache, F. Gherendi

National Institute for Laser, Plasma and Radiation Physics, Bucharest

1. Introduction

Presently, the X-ray micro-tomography is the only reliable solution for official inspections of the structural integrity of IFMIF complete assemblies before, during and after irradiation campaigns [1]. Hence the successful demonstration of micro-tomography on the assembled irradiation capsule is crucial.

During 2003 extensive NDT inspection of fusion materials miniaturized samples using transmission micro-tomography system were carried out. The goal was to establish the reference design for a micro-tomography system for IFMIF environment condition. 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, thermocouples and heaters. As typical examples we presented the reconstructions obtained for probes realistically simulating two test capsules, which were constructed and measured in our laboratory. Based on these results one concluded that we are in a position to accurately assess the structural integrity of the IFMIF High Flux Test Module (HFTM) irradiation capsules.

In 2004, in addition to conventional cone-beam tomography, new inspection configurations were developed in order to assess the integrity of the assembled irradiation capsules and rigs. Limited view tomography and laminography are the only possible NDT techniques to provide as precise as possible 3-D model of the instrumented capsule.

During the reported period a comparative NDT analysis on two micro-tomography facilities, our low energy, high magnification installation and a high-end industrial tomography facility with higher X ray energy and intensity was carried out. Thus, one could better assess the limitations of our facility when it comes to bulky samples made of high density materials. In order to prove the observation of flaws in the testing samples we assembled a dummy capsule containing various miniaturized specimens with intentionally incorporated defects. One can conclude that the current measurement represents the most realistic trial to qualify the tomography method as a reliable solution for official inspections of the structural integrity of IFMIF complete assemblies. Further tests will be carried out on the real irradiation capsule and rig that would be made available by our IFMIF partners.

Another topic initiated during the reporting period was tomographic measurements to accurately record the shapes evolution of compressed pebble bed samples. The goal of the experiments, relevant to tokamak blanket technology, is to determine the following quantities: radial and vertical void fraction profiles, contact numbers and contact areas of the pebbles and their position on the spheres.

The inspection configuration was also adapted in order to acquire real time micro-radiographies for monitoring the history of mechanical tests of the miniaturized specimens. Real time micro-radiography is a powerful tool for monitoring the history of mechanical/thermal tests of the miniaturized specimens. In the current study we performed real time X-ray imaging of compression tests on ceramic samples and fracture tests on metallic foils.

2. X-ray tomography of dummy irradiation capsules

The reference design for micro-tomography system for IFMIF environment condition was established for miniaturized fusion materials samples. However, it is of great interest to perform NDT inspection of large and complex samples as irradiation capsules and rigs of high density materials. Inherently, the low budget, low energy facility built in our laboratory would exhibit limitations when confronted with such demanding requirements. In order to identify these limitations we carried out a comparative NDT analysis was on two micro-tomography facilities, our low energy (NILPRP), high magnification installation [2-3] and a high-end industrial tomography facility with high X ray energy and intensity [4]. The study was performed in cooperation with Hans Waelischmiller (HWM) Institute from Germany. Table 1 presents the overall performances of the facilities used in the comparison.

Table 1: Overall performances of the microtomography facilities used in the comparative study

Item	NILPRP facility	HWM facility
Microfocus X-ray source	Max. high voltage: 160 kVp Max. power: 20 W Min. object-focus distance: 0.4 mm X-Ray cone: 170° Focus spot \cong 3 μ m	Max. high voltage: 225 kVp Max. power: 300 W Min. object-focus distance: 5.0mm X-Ray cone: 30° Focus spot 5-200 μ m
Detector elements	768 x 576 image intensifier (XII) 1248 x 1248 a-Si flat panel	1048 x 1048 a-Si flat panel
Digital Output	10 bits XII, 12 bits a-Si	16 bits a-Si
Effective Area of Detector	169x169 mm ² XII, 120x120 mm ² a-Si	400x400 mm ² a-Si
Micrometric manipulator	Four motorized axes	Six high precision axes
Magnification Factor	<2000	<400
Source-Detector Distance	1000 mm typical	1500-2500 mm
Nominal Spatial Resolution	\cong 10 μ m	\cong 10 μ m
Scanning Time	< 10min. (720 angles)	< 20min. (720 angles)
3D Reconstruction Time	< 2min. (256x256, 256lines) <5min. (512x512, 256lines) <20min. (1024x1024, 512 lines)	Never longer than scanning time
Probe dimensions	Diameter <40 mm Height <500 mm Weight < 4 kg	Diameter <400 mm Height <1500 mm Weight < 100 kg
Scanning Method	Cone beam CT Region of interest (ROI) CT	Cone beam CT Region of Interest (ROI) CT

From the comparison of the reconstructed details it resulted that the higher X-ray energy available at the HWM facility was essential for achieving sufficient space resolution in order to assembly an accurate 3-D model of the dummy irradiation capsule. This permits the precise determination (within half a voxel) of the position of the thermocouple wires and pipes. The conclusion of this comparison provided the elements for upgrading the reference design of the IFMIF micro-tomography facility.

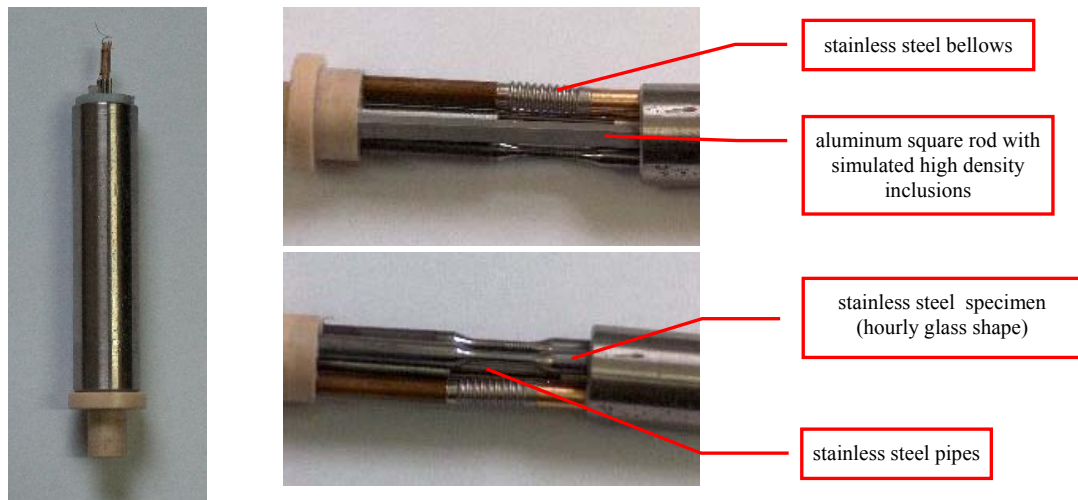


Figure 1 – Photographs of the dummy capsule

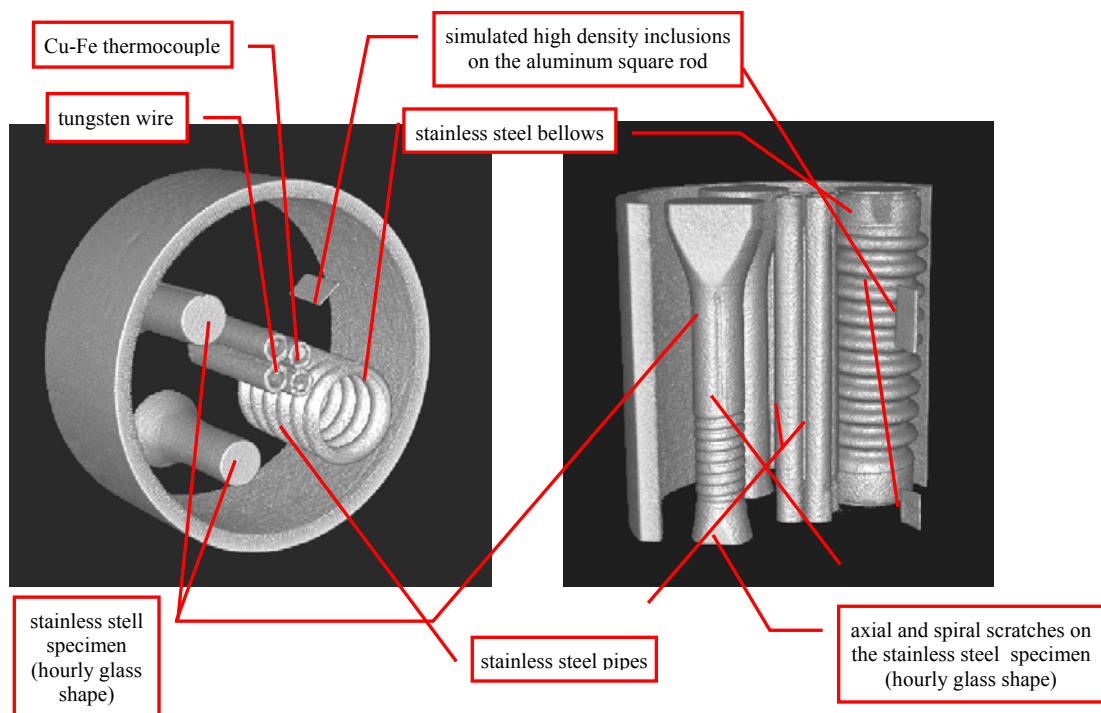


Figure 2 – 3-D reconstruction of the dummy capsule revealing scratches on one of the two stainless steel specimen (hourly glass shape)

The successful demonstration of micro-tomography on the assembled irradiation capsule is considered crucial. In order to prove the observation of flaws in the testing samples we assembled a dummy capsule containing almost all possible miniaturized specimens with intentionally incorporated defects (photography of the dummy capsule is presented in Figure 1). Four miniaturized elements are placed inside a steel capsule of height 70 mm, exterior diameter 16 mm and wall thickness of 0.8 mm. These are: two stainless steel specimens with an hourly glass shape (diameter 2 mm) one of them without induced defects and the other with two types of defects (axial and spiral scratches), miniature stainless steel bellows (exterior diameter 4.4 mm) and aluminum square rod (3 mm) with simulated high density inclusions. The center of the capsule is occupied by four stainless steel pipes (1.3 mm exterior diameter) one free, two containing a tungsten wire (100 microns) and another one with a Cu-Fe (100 microns) thermocouple. Tomography measurements were carried out at the

Micro-tomography Laboratory of the National Institute for Laser, Plasma and Radiation Physics (NILPRP), Bucharest. Optimum measurement parameters were around: $U=130$ kV, $I=85$ μ A, focal spot $3\div 5$ microns, spectrum filters 0.1 mm Cu, 0.9 mm Cd, geometrical magnification $6 \div 8$.

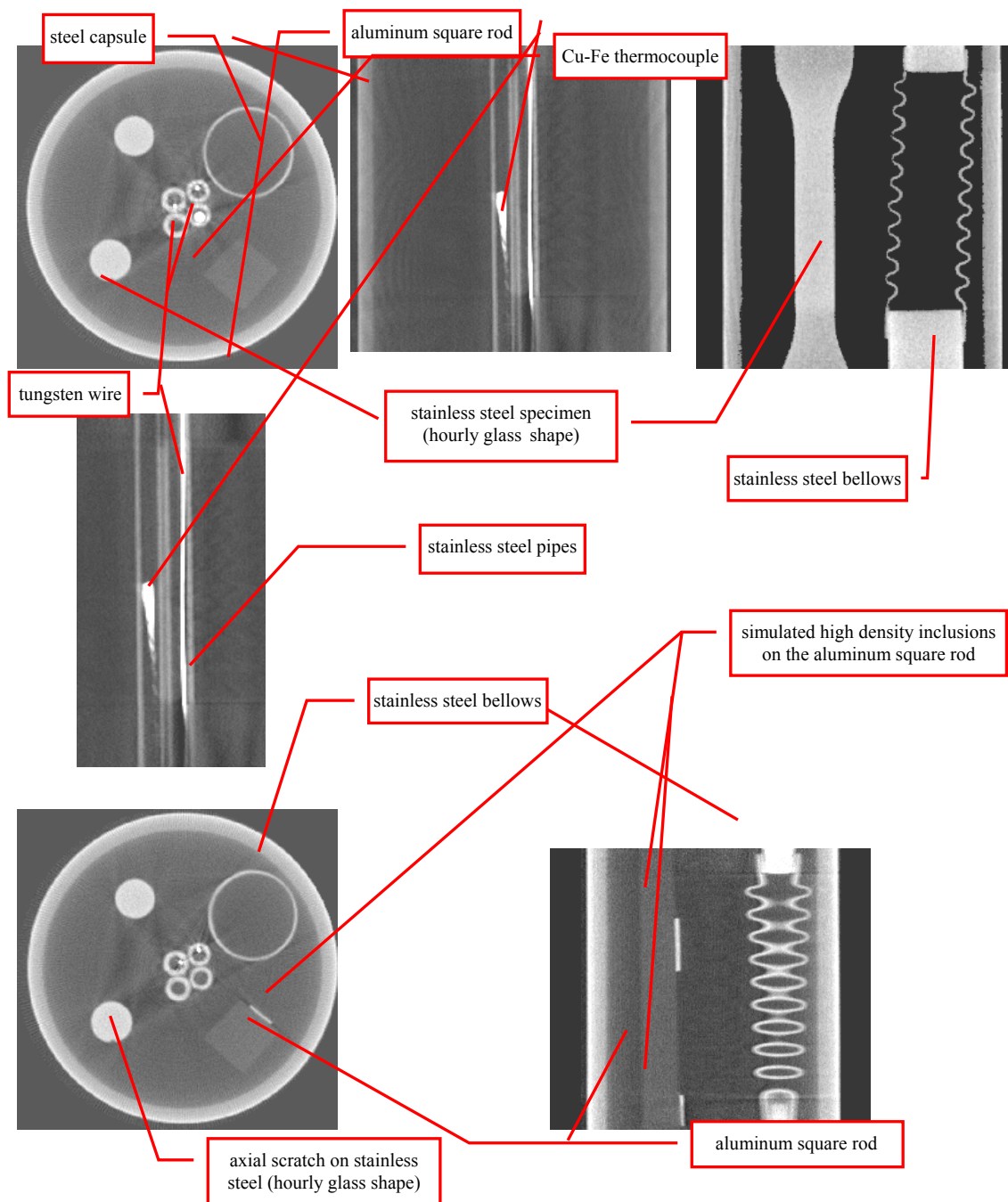


Figure 3 - Cross-sections through the 3-D reconstruction of the dummy capsule

This dummy sample incorporates several new elements in comparison with reported in the previous reporting period. Main differences consist in: low density material specimen (aluminum with simulated high density inclusion), stainless steel bellows, high density hourly-glass specimen with induced defects and more complex heaters/thermocouple structure. In addition the aluminum capsule clad was replaced by a relatively thick steel one. We believe that this capsule mock-up represent a better simulant of the real one which is still not available in the final form.

Reconstruction results are shown in Figures 2 and 3. Figure 2 presents the 3D reconstruction of the dummy capsule revealing the spiral and longitudinal scratches on one of the two hourly-glass specimen, the heaters/ thermocouple structure and the bellows surface. The reconstructed dimensions of the scratch defects are in good agreement with those produced. Various cross-sections through the reconstructed dummy capsule are illustrated in Figure 3. Visually one can recognize all elements enclosed in the capsule. More quantitatively, these cross-sections allow us to accurately measure the dimensions of the components and their relative positions. The absolute error of geometrical measurements is on the order of the voxel size: 32.8 microns. This is considered sufficient precision for the assessment of the structural integrity of the irradiation capsule and for the geometry description of the thermal-hydraulic modeling.

We can conclude that the current measurement represents the most realistic trial to qualify the tomography method as a reliable solution for official inspections of the structural integrity of IFMIF complete assemblies.

3. Tomography on pressurized pebble-bed samples

Another topic initiated during the reporting period is tomographic measurements to accurately record the shapes evolution of compressed pebble bed samples [5]. This is part of our efforts to acquire real time micro-radiographies/tomographies for monitoring the history of mechanical/ thermal tests of the miniaturized specimens.

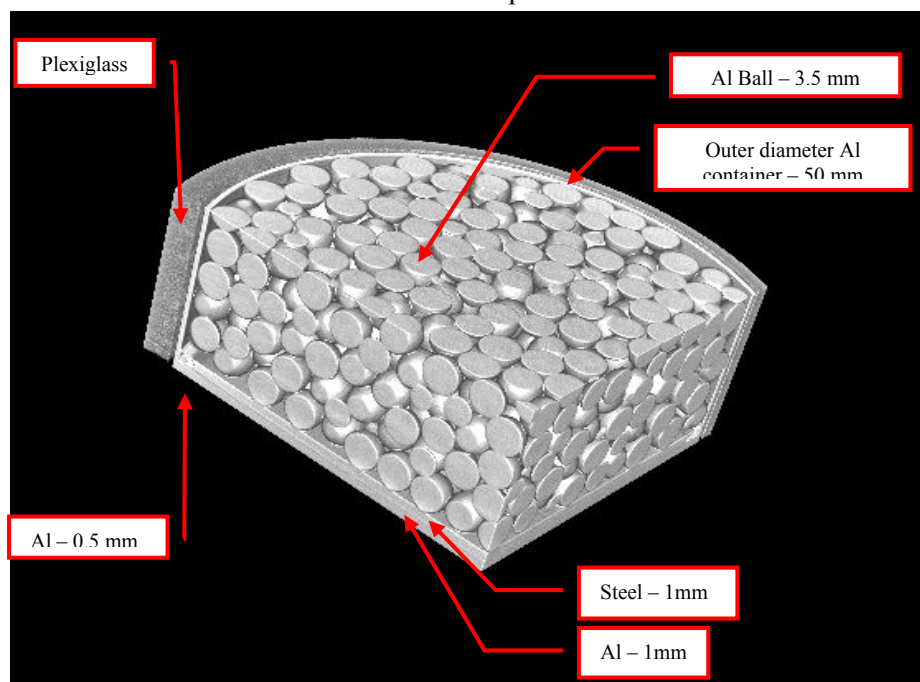


Figure 4. 3-D tomography reconstruction of compressed pebble bed sample.

The sample containing a compressed pebble bed consisting of 3.5mm aluminum pebbles was provided by a group of the Forschungszentrum Karlsruhe, Germany. This sample had already been investigated to a certain extent in the ESRF Grenoble [6]. The sample consists of a cylindrical Plexiglas container that contains a cylindrical aluminum container

(outer diameter 50mm, inner diameter 49mm; wall thickness at the bottom 1mm) with the aluminum spheres. At the container bottom there is an additional steel plate with a thickness of 1mm. The pebble bed sample was compressed at 8 MPa. The measurement conditions: HV 200 kV, current 170 μ A, voxel size 55.8 μ m, magnification 7.17.

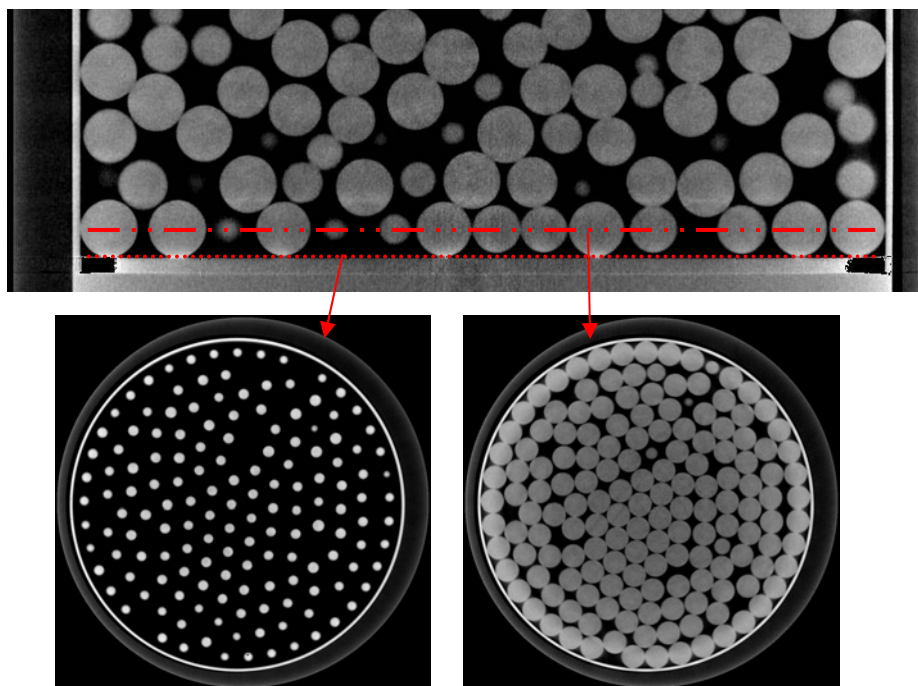


Figure 5 : Pebble bed sample: frontal cross-section (top); axial cross-sections through contact area between steel plate and aluminum balls (bottom-left) and through middle of bottom layer of aluminum balls (bottom-right).

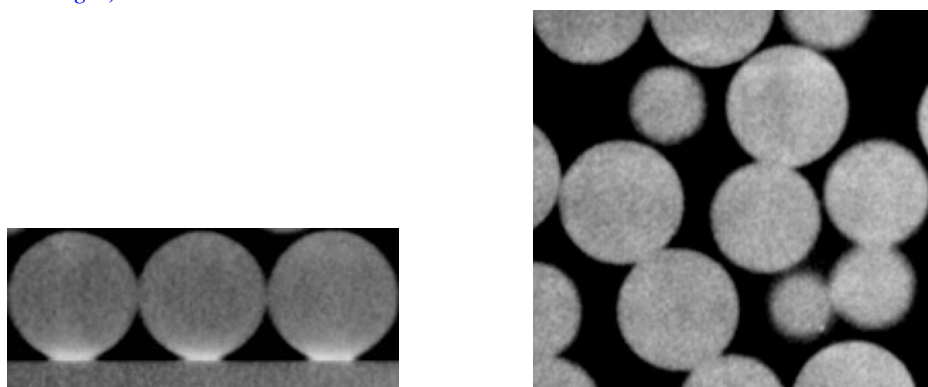


Figure 6: Pebble bed sample - frontal cross-sections: the bottom zone including those pebbles contacting the steel plate (left), zone at about the half height of the bed; the pebbles in this zone are characteristic for "bulk" pebbles (right).

Of special interest are two zones in the pebble bed:

- the bottom zone including those pebbles contacting the bottom,
- a zone at about the half height of the bed; the pebbles in this zone are characteristic for "bulk" pebbles.

The goal of the experiments is to determine the following quantities:

- radial and vertical void fraction profiles; the latter is of interest in the bottom zone,
- contact numbers and contact areas of the pebbles and their position on the spheres.

These quantities are different for pebbles in the bulk and those close to walls. The determination of these quantities is a very challenging task both in respect to the measurements (a space resolution in the range of 25 microns for a relatively large volume should be achieved) and the mathematical algorithms.

Here we report some preliminary results. Fig 4 shows a fragment of a typical 3-D reconstruction of the compressed pebble bed sample. Following we present frontal and axial cross section through some relevant regions (Figures 5 and 6).

4. Real time micro-radiography

Real time micro-radiography is a powerful tool for monitoring the history of mechanical/thermal tests of the miniaturized specimens. As our micro-radiography system is guaranteed for feature recognition down to a few microns this method could record very accurately shapes evolution as well as the development of micro-cracks, voids and even the fracture propagation process. A number of laboratories have used real time radiography to characterize the structure evolution of the various metallic foams, intergranular and exfoliation corrosion in high-strength Al alloys, dynamics of solidification pattern of metallic alloys etc.

To our knowledge only synchrotron sources like at ESRF, Grenoble, France have been used for in situ real time imaging of non-medical samples.

The measurements were carried out at the Micro-tomography Laboratory of the National Institute for Laser, Plasma and Radiation Physics (NILPRP), Bucharest. The micro-radiography facility used consists of a microfocus X-ray source (Phoenix 160-xt) and an image intensifier (SIRECON 17-2 HDR-M). The main feature of the microfocus source is the focal spot of only 3 microns. The image intensifier detector with an effective area of 128x170 mm² (576 x 768 pixels) can record X-ray images at up to 30 frames/second.

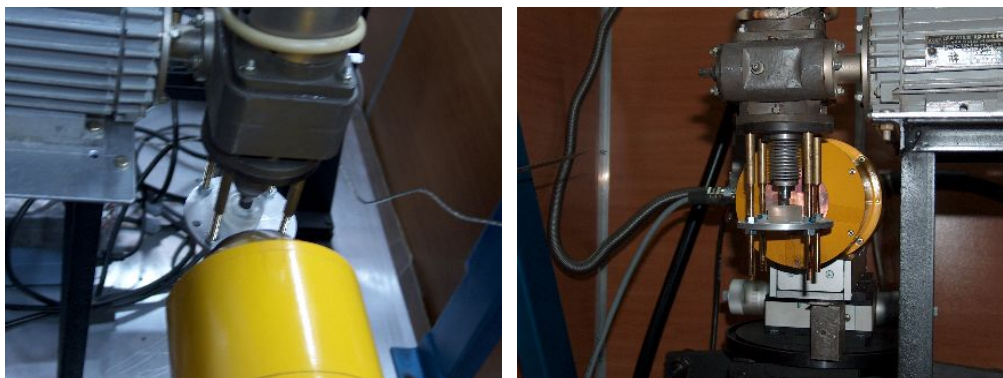


Figure 7 – Photographic views of the mechanical test device

In the current study we performed real time X-ray imaging of compression tests on ceramic samples and fracture tests on metallic foils. The mechanical tests device was

designed and constructed in our laboratory (Figure 7). It can deliver a constant pressure/ force over a displacement of about 5 mm in an interval of 10 seconds. The application of pressure/ force was synchronized with the X-ray images capture as well as with the capture of video/ audio signals. The X-ray images were acquired by a home- made software based on a special function (IMAQ-SEQUENCE) from the LabView-IMAQ package.

4.1 Compression test of ceramic samples

The purpose of compression tests is to investigate brittle engineering materials by detection of cracking in ceramic and coated materials. The sample (usually of tablet shape) is placed between two jaws that crush the tablet. This event is detected by an acoustic sensor. Usually, compression testing machines operate at constant speed. Our compression device was operated at constant speed of 0.5 mm/sec. Audio/video signals were recorded with a web camera with incorporated microphone. The audio data plot is correlated with the X-ray frames.

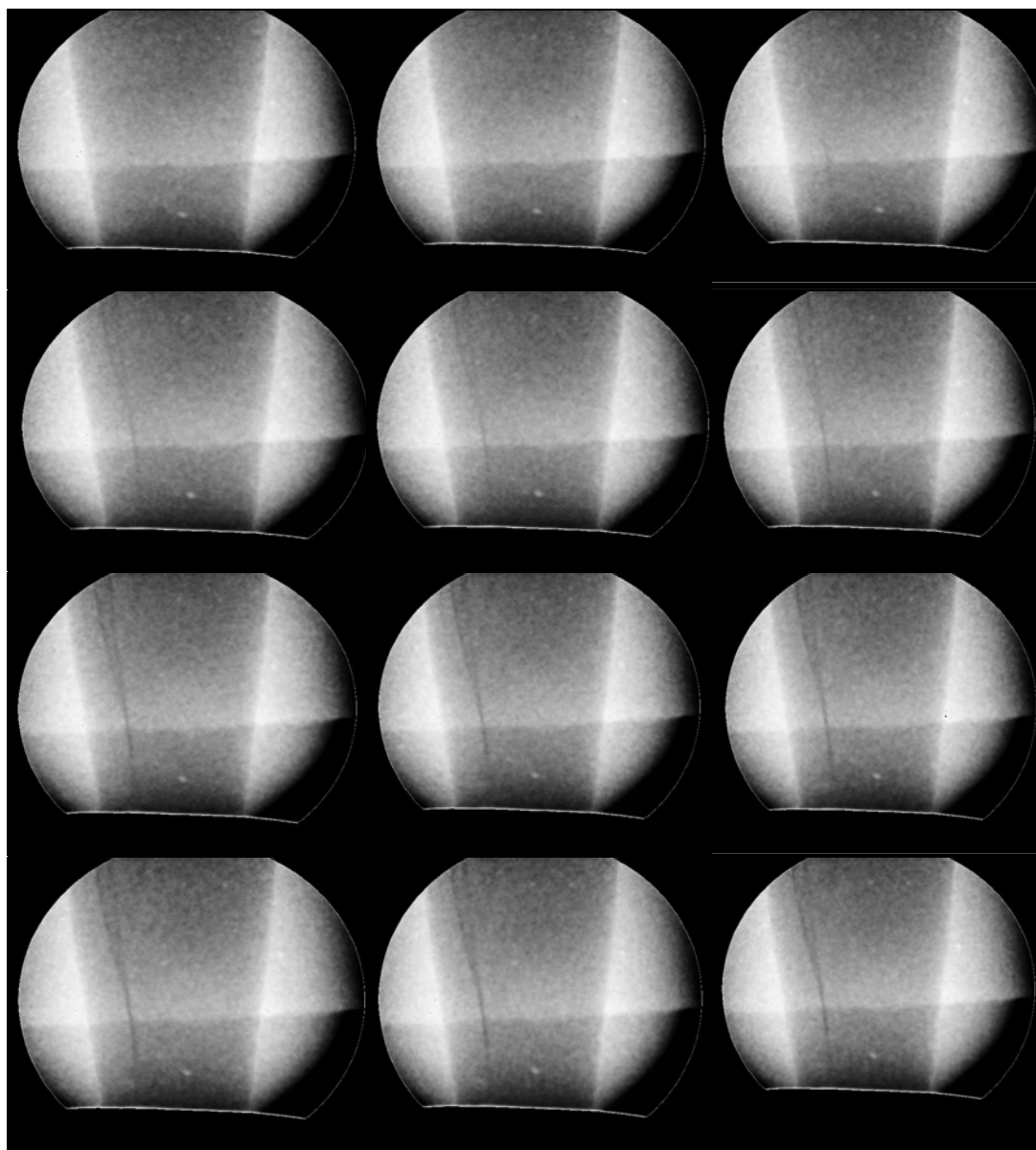


Figure 8 – Crack formation and propagation in an alpha-alumina cylinder with “dome shaped” end

The ceramic samples are alpha- alumina cylinders with a “dome shaped” end. Characteristics dimensions are: exterior diameter 7.2 mm, interior diameter 3 mm, cylinder height 6.2 mm, dome height 2.6 mm.

The ceramic samples follow a brittle fracture failure mechanism i.e. failure without significant amount of macroscopic plastic deformation prior to fracture. This mechanism was clearly identified in our compression tests. First a crack develops all along the sample height. The development time is around the sampling rate of our real time radiography system (66 msec). In a second step further increasing the stress will cause the crack to propagate in a catastrophic manner. The time constant of this step is about 800 msec.

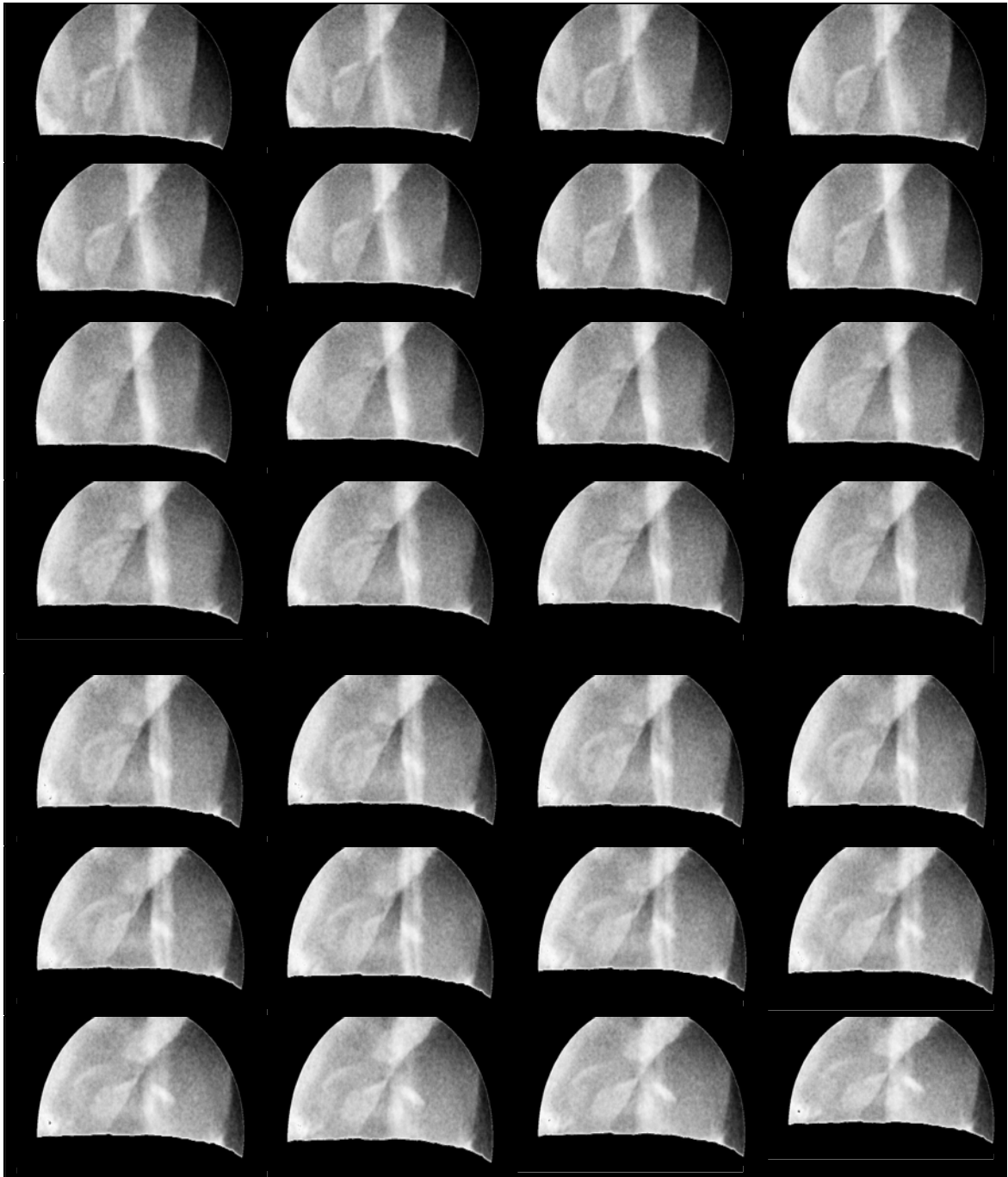


Figure 9 – Crack propagation in a catastrophic manner in an alpha-alumina cylinder with “dome shaped” end

Figures 8 and 9 display frame sequences corresponding to the two steps. The images were collected at $U=70$ kV, $I=60$ μ A, geometric magnification 21 and voxel size 10.5 microns. Figure 8 follows the initiation of the crack and its full development from top to bottom of the compression device. In Figure 9 one can see the catastrophic collapse of the alumina dome. The audio data plot represented in Figure 10 allows the time correlation of crack occurrence and development. From Figure 8 one can estimate the main crack width of 30-40 microns.

These measurements were designed to demonstrate the ability of our facility for monitoring the history of mechanical/thermal tests of the miniaturized specimens. One can remark that it has delivered a rather deep insight into the relatively complex failure mechanism of crack formation and propagation in ceramic samples.

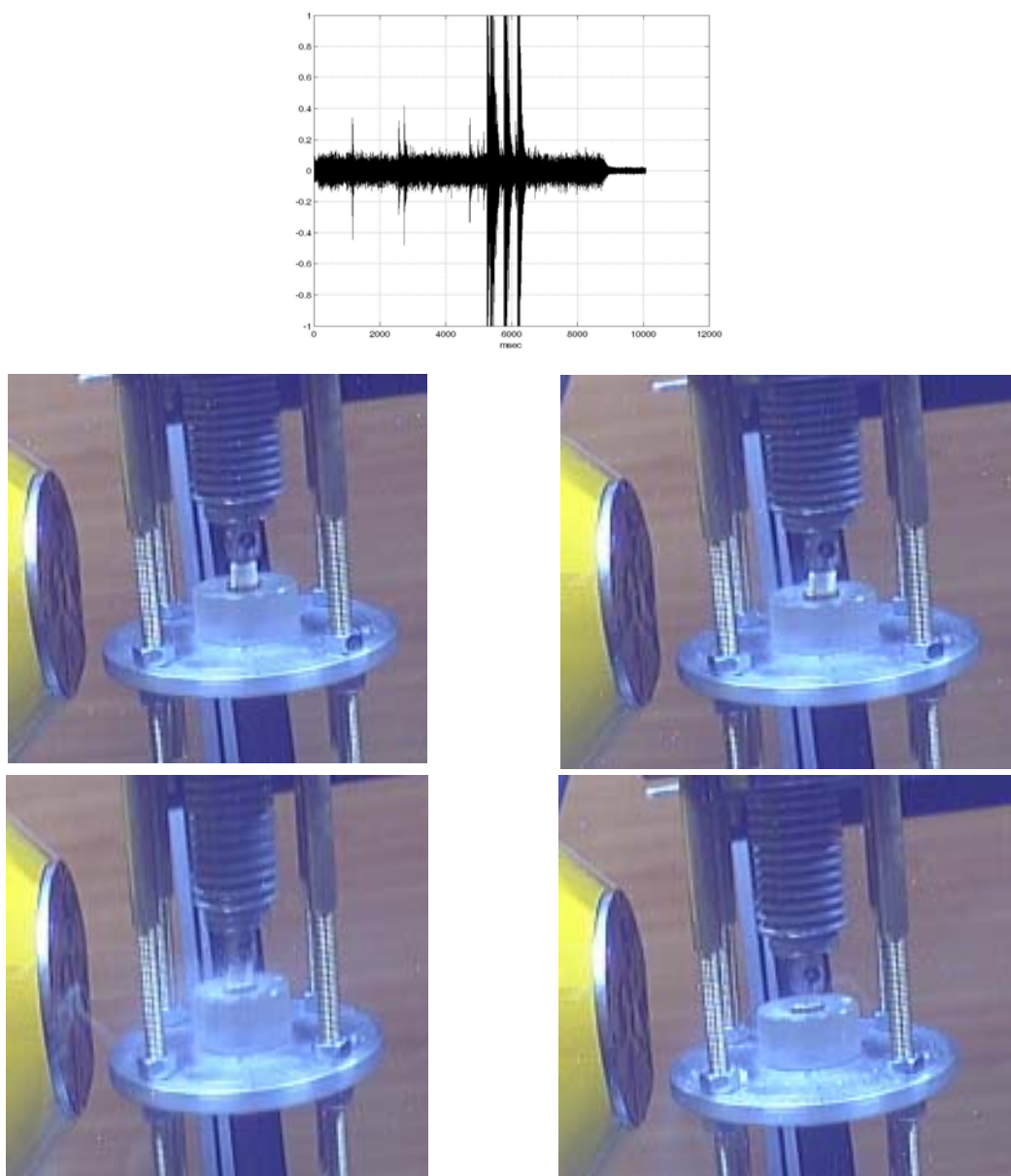


Figure 10 – Audio data plot (top) and some relevant video frames recorded during the compression

4.2 Tensile testing of thin metallic foil

Tensile testing of thin copper foil was visualized at constant strain rate. It is known that a pronounced effect of the thickness of thin copper foils is found in tensile testing under constant strain rate: the thinner a foil the smaller is the fracture strain. Foils that are thinner than 20 microns show macroscopically hardly any plastic deformation whereas the 250 microns thick foils still show the typical behavior of bulk copper.

In our experiments foils of 100 microns thick were used that should lead to a combination of the two material failure mechanisms: ductile fracture - failure that involves a significant amount of plastic deformation prior to fracture and fatigue fracture - failure associated with slow crack growth.

As tensile samples we used copper foils of rectangular shape 25x10 mm. To locate the fracture in the field of view of the radiographic system and transversal to the foil small dimension we initiated the fracture by short cuts of the foil on one or both sides of about 0.5 mm length and 100 microns average width. Figures 11 and 12 present the frame sequences in two representative measurements. For the sample in Figure 11 the initial cuts were applied on both sides of the copper foil at slightly different positions. One can remark a very interesting fracture mechanism. Initially the fractures propagate from both sides along the initiated directions, and then suddenly they change the trajectory to exactly match each other. A sampling rate of about 15 frames/sec is sufficient to follow the fracture mechanism. During propagation the tip of the fracture remains very narrow, approx. 20 microns. The X-ray absorption contrast for metallic foils provides the ability to study the fracture propagation in multiple layers samples. For example, Figure 12 shows the fracture dynamics in two parallel foils with initial cuts on both sides. The two foils follow the same fracture pattern mechanisms described above but separated in time.

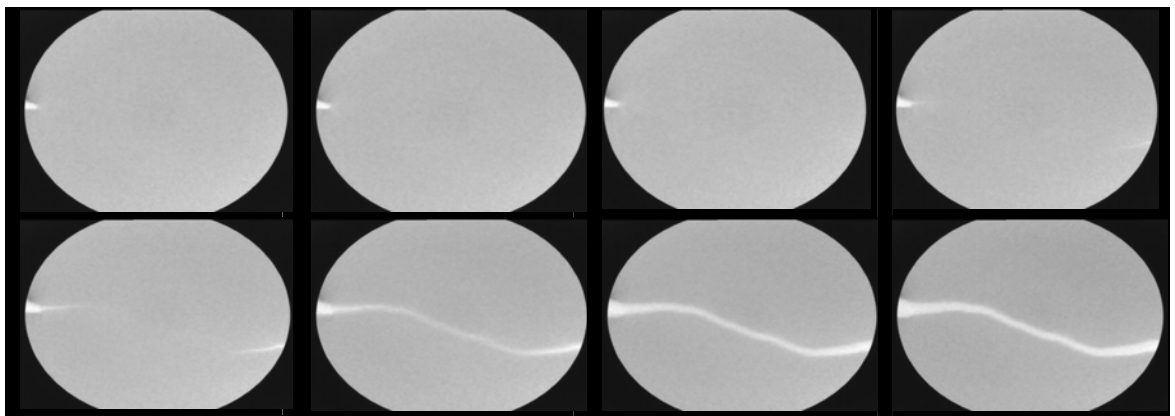


Figure 11 – Fracture mechanism of a thin metallic foil

Conclusions

During the reported period we focused our research on X-ray tomography of realistic irradiation capsules in order to assess their structural integrity. In addition a comparative NDT analysis was carried out on two microtomography facilities, our low energy, high magnification installation and a high-end industrial tomography facility with higher X-ray

energy and intensity. Thus, one could assess the limitations of our facility when it comes to bulky samples made of high density materials. It is actually the case with the real HFTM capsule that will be inspected in 2005. This analysis was supplemented by limited view tomography (laminography) measurements performed on same two facilities. We believe that this inspection technique is more adequate for flat samples as irradiation capsule. In order to prove the observation of flaws in the testing samples we assembled an additional dummy capsule containing various miniaturized specimens with intentionally incorporated defects. The absolute error of geometrical measurements is under the voxel size (approx. 30 microns). That is considered sufficiently accurate for the assessment of the structural integrity of the irradiation capsule and for the geometry description of the thermal-hydraulic modeling. One can conclude that the current measurement represents the most realistic trial to qualify the tomography method as a reliable solution for official inspections of the structural integrity of IFMIF complete assemblies. Further tests will be carried out on the real irradiation capsule and rig that would be made available by Forschungszentrum Karlsruhe, our IFMIF partner.

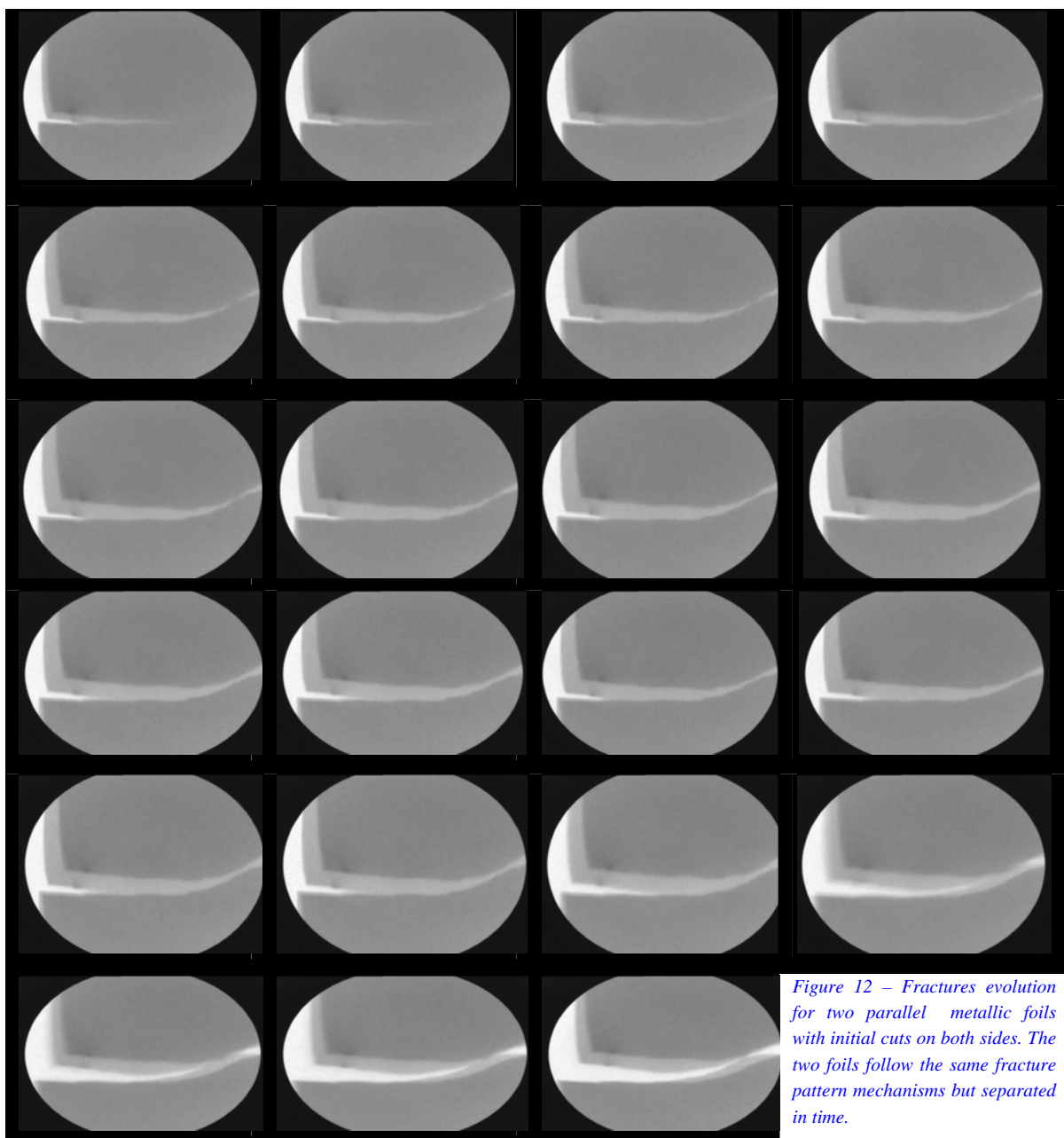


Figure 12 – Fractures evolution for two parallel metallic foils with initial cuts on both sides. The two foils follow the same fracture pattern mechanisms but separated in time.

The pre-existent and newly developed deformations around 20 microns in aluminum spheres of 3.5 mm diameter in a cylindrical aluminum container have been recorded. The goal of the experiments is to determine the following quantities: radial and vertical void fraction profiles, contact numbers and contact areas of the pebbles and their position on the spheres. To our knowledge this type of study was carried out only recently and only at very bright X-ray sources provided by synchrotron facilities. Here we demonstrated that a well tuned table-top cone-beam tomograph could also be successfully applied to same type of inspection tasks.

Our inspection configuration has been also adapted for the acquisition of real-time micro-radiographies of miniaturized specimens under mechanical stress. Real time X-ray imaging of compression tests on ceramic samples and fracture tests on metallic foils were carried out. The mechanical tests device was designed and constructed in our laboratory. The sampling rate (15 frames/sec) and the space resolution (20-40 microns) proved to be adequate for monitoring of fracture formation and propagation processes. Different fracture mechanisms were clearly identified in our compression tests. The X-ray absorption contrast for metallic foils also provides the ability to study fracture propagation in multi-layered samples. That type of experiment has become relevant for in-situ fracture test in the IFMIF test cell environment.

References:

- [1]. IFMIF Comprehensive Report Design 2003
- [2]. **Tiseanu I. and team**, EFDA Newsletter, Vol. 2003/6 December, 2003
- [3]. **Tiseanu I., Craciunescu T. and Mandache N. B.**, “*Non-destructive analysis of miniaturized samples and irradiation capsules by X-ray micro-tomography*”, presented at the 23rd Symposium on Fusion Technology, September 2004, Venice, Italy
- [4]. **Simon M., Sauerwein C., Tiseanu I., Burdairon S.**, “*Flexible 3D-Computertomographie mit RayScan 200, ZfP in Anwendung, Entwicklung und Forschung*”, Berlin, 21-23 Mai 2001, Berichtsband 75-CD
- [5]. **Reimann J., et al.**, “*Thermomechanics of solid breeder and Be pebble bed materials*”, Proc. ISFNT-6, San Diego, April 2002.
- [6]. **Joerg R., Pieritz R. A., di Michiel M., Ferrerom C.**, “*Inner Structures Of Compressed Pebble Beds Determined By X-Ray Tomography*”, presented at the 23rd Symposium on Fusion Technology, September 2004, Venice, Italy.