

Characteristics of the Micro-Focus X-ray Tomography Facility (MIXRAD) at Necsa in South Africa

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Abstract

The South African National Centre for Radiography and Tomography (SANCRAT), located at Necsa, strives to be leaders in utilizing ionizing radiation as imaging probes in research and development as part of the South African National System of Innovation. The centre already encompasses a Neutron Radiography and Tomography facility (SANRAD) as well as a High Energy X-ray facility (HEXRAD). Recently the centre introduced a state-of-the art micro-focus X-ray machine to the already diverse arsenal of imaging equipment in the Micro-focus X-ray Radiography and Tomography facility (MIXRAD). It is anticipated that the equipment will be extensively used by post graduate students and researchers since this dedicated equipment is the first of this kind and type being made available, nationally, to conduct research for free if the research leads to the publication of peer reviewed papers and/or an upgrade to a higher educational degree. The characteristics and performance capabilities of the MIXRAD facility are presented through several case studies that were being conducted with the instrument.

Keywords: Micro-focus X-ray tomography, characteristics, capabilities, limitations, South Africa

1. Introduction

Micro-focus X-ray Computed Tomography technology becomes a very attractive radiation based research technique since high quality micron-level information of the interior as well as composition of samples can be obtained non-invasively and non-destructively. Micro-focus investigations are therefore extensively focused in research fields such as the geosciences with typical investigations on porosity [1], mineral distribution and quantification (e.g. 3D Volume fractions) [2], petrographical analyses [3], fracture (cleat) analysis [4], ect. The South African Nuclear Energy Corporation (Necsa) accommodated post graduate students in X-ray investigations in research projects previous with the aid of a 100kV X-ray source. There is however a big limitation to this particular system since the focal spot is relatively large (3 mm) and as a result, the spatial resolution obtained, even with small samples, is in the order of 0.08 mm.

Through the National Research Fund (NRF) initiative to upgrade national equipment capabilities to higher levels of sophistication, a state of the art micro-focus X-ray machine was installed at Necsa in May 2011. Consequently the MIXRAD facility, earmarked to be utilized by the national (and international) research communities was instigated. This machine enables



students and researchers from various research disciplines to perform investigations for free when results obtained is published in open literature after a peer review process or the study is a post graduate study towards a higher educational degree. For researchers to have access to this kind of technology in the past was very difficult since all similar machines in South Africa are privately owned and therefore beam time is not readily available and at a cost. Micro-focus X-ray investigations also forms the basis of most tomographic synchrotron-based investigations and consequently the MIXRAD facility enables researchers and students from South Africa to submit good quality proposals to synchrotron light sources to obtain beam time.

In this paper the characteristics and performance of the MIXRAD facility are being described and then highlighted by several case studies which were performed on the MIXRAD machine and compared to results obtained at a similar European facility. Its performance in the fields of biosciences, food science and mineralogy are addressed.

2. MIXRAD Facility

Nikon XTH 225 ST micro-focus X-ray tomography systems are fully assembled in Europe and meets very stringent quality and safety standards. The system basically consists of 4 separate functional units; a lead-lined cabinet, an external control module, an external chiller and PC's with software for acquisition, reconstruction into a 3D virtual image and for visualisation and analysis (See figure 1). The lead-lined cabinet houses the X-ray tube, sample manipulator and flat panel detector. The lead-lined cabinet meets international DIN radiation standards and is completely shielded off for the total hourly dose rate to measure less than 1 micro Sievert on cabinet surface.

The system voltage setting ranges between 30 and 225 kV whilst the beam current ranges from 0 to 1 mA (see table 1). With a maximum power rating of 30W it ensures that a wide variety of samples can be investigated, even when the density of the sample is relatively high.



Figure 1 Micro-focus X-ray machine at the MIXRAD facility

The specific X-ray tube that is installed in this system is easy to maintain and cost effective since off the shelf filaments are used. The spot size is 0.001 – 0.003 mm and therefore the geometrical unsharpness of the object associated with cone beam enlargement is minimized. A multi-metal reflection target enables the researcher to utilize a specific X-ray energy spectrum depending on the specific sample characteristics that is being investigated. The multi-metal target comprises out of 4 targets on one rod and consists of molybdenum, copper, silver and tungsten (which are mostly used in X-ray sources). Unfortunately the target suffers physical damage over time in the form of a small hole that is burned into the rod that affects the quality of the radiograph. This damage is instigated in a process called electron drilling or pitting due to the small focal spot and high energy electron beam impinging on the same area. Luckily this pit is very small and the target can be indexed for the electron beam to be always at an optimum energy.

Table.1 System characteristics of MIXRAD system

| System Characteristic | | Value |
|------------------------------|---|----------------------------------|
| X-RAY SOURCE | Voltage (kV) | 30-225 |
| | Current (mA) | 0-1 |
| | Spot size (μm) | ~1-3 |
| | Beam Angle (degrees) | 25 |
| | Multi target | Mo; Cu; W; Ag |
| | Filter kit with a range of thickness of foils | Cu; Al; Sn; Ag |
| TRANSLATION TABLE | Axis movement (directions) | 5 |
| | Sample Movement in Horizontal Plane (mm) | 200 |
| | Sample Movement in Vertical Plane (mm) | 300 |
| | Sample Movement in Beam Direction (mm) | 610 |
| | Rotation accuracy | 1/1000 th of a degree |
| | Tilt | 45° |
| | Movement | Ultra slow; Slow; Medium; Fast |
| DETECTOR | Type | Perkin Elmer |
| | Dynamic Range | 16 bit |
| | Physical Size (mm) | 400 x 400 |
| | Pixel Size (Micron) | 200 x 200 |

X-rays have to be generated in a vacuum and therefore the system is supplied with a turbo-molecular pump as well as a two-stage rotary vane pump. For normal operation to commence, vacuum is achieved within approximately 10 minutes. The thin tungsten tube filament, the most fragile component in the process to generate X-rays, has a life time of

approximately 35 – 100 hours depending on the operational conditions and parameters – maximum life time of the filament is being achieved at the MIXRAD facility with operation at 10W and less. The filament's functional life is, however, a trade-of between the acquisition time and current (mA) density. Replacement of a damaged filament is easy and cost effective and can be completed within 1 – 2 hours by eliminating the vacuum and opening the X-ray tube by quick-release latches. However, the correct setup of the system after replacement of a filament is tricky and time consuming.

The Perkin Elmer detector is a flat panel detector with a 16 bit dynamic range. This detector is state of the art and uses a scintillator material in conjunction with a direct output digital panel. The physical active size of the detector is 400 mm x 400 mm with a pixel size of 200micron x 200micron. For tomography applications the detector accommodates a sample of approximately the same size at lowest system magnification [5].

A 5-axis sample manipulator is installed in the lead-lined cabinet and is completely controlled by the acquisition PC (installed in the external control module). A maximum sample weight of 50 kg is permitted on the sample manipulator which extends the capabilities of the system to investigate larger samples. Joysticks as well as a CNC user interface on the image acquisition software enables the instrument scientist to obtain the optimum sample position before experimentation commences. The sample manipulator can move 200 mm in the horizontal plane, 300 mm in the vertical plane and 610 mm in the beam direction which allows for a high geometrical enlargement (zoom), depending on the size of the sample. The rotation movement is extremely accurate up to 1/1000th of a degree and consequently up to 4000 or more projections in 360° are possible where the amount of projections influences the resolution of the final scan.

The system is delivered with pre-installed image acquisition and sample manipulation software and has a fully automatic mode for tomography once the sample has been placed on the sample manipulator. Through Inspect-X, dedicated software developed by Nikon, projections for the CT scan are being acquired, the vacuum is managed, the sample manipulator is controlled, it communicates with the X-ray controller and determines the safety status of the system. Inspect-X is installed on the 32 bit image acquisition PC which is housed in the external control module together with the power supply and power distributor to the system. Projections are transferred via CAT-6 connection cable from the image acquisition PC to a 64 bit reconstruction PC at a rate of 1 GB per second. It is thus possible for images to be sent immediately to the reconstruction PC and therefore minimizes data transfer time.

2.1. Tomographic Process at the MIXRAD facility

The tomographic process of the MIXRAD facility is similar to that of the SANRAD facility [6] with the biggest difference in the amount of automation in the tomographic acquisition procedure at the MIXRAD facility. The sample is fixed on the sample manipulator in the micro-focus X-ray cabinet to ensure no movement of the sample occurs during scanning – it is then horizontally optimized, normally for maximum enlargement of the sample in order to obtain optimal spatial resolution. This adjustment is also to ensure that the sample is horizontally

included in each 2D radiograph at all angles of rotation and for correct normalization during the tomography reconstruction process. (figure 2).

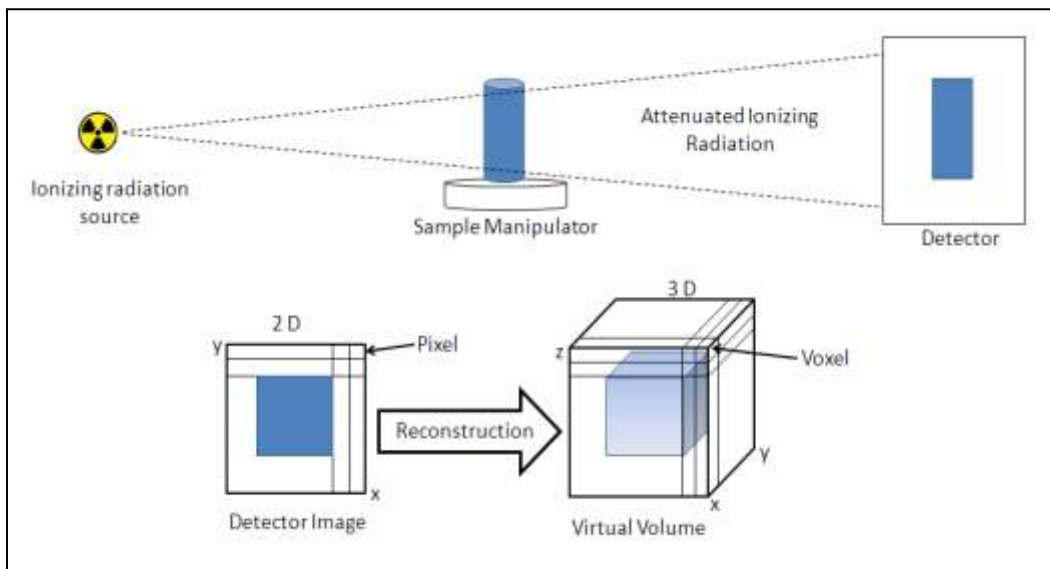


Figure 2 Tomographic Process at the MIXRAD facility

The energy and current density settings are individually adjustable while the system is in operation. This allows for careful adjustment to achieve at least 15% penetration through the sample but never exceeds 65535 grey values of dynamic range in areas without the sample. Filters are available to achieve a comfortable high dynamic range within the radiographs. Once the sample is correctly positioned, a shading correction algorithm is obtained through acquiring flat field radiographs under the exact same conditions as the sample images but with the sample removed from the field of view. Additionally, radiographs with the X-rays switched off are acquired in order to remove possible non-functional pixels and to determine the gray value of air during the normalization process. One flat field image (an average of up to 128 radiographs) and one “dark” image (an average of up to 128 radiographs) are usually obtained to develop the shading correction. The Inspect-X software incorporate these images in real-time during the scanning process so that the projections obtained from a CT scan are already normalized. The acquisition software also enables the researcher to correct for beam hardening artifacts and reconstruct in real time but this is not desired since it is a time consuming process. Micro-focus projection data sets are up to 30GB in size and it is a time consuming process to reconstruct the virtual image - therefore 64Bit PC's with solid state hard drives are recommended. .

The projections images are in an appropriate tiff-format for the reconstruction process to commence on the reconstruction PC the moment the scan is complete. CT-Pro reconstruction software, fully developed in-house by Nikon, is used to transform the 2D projections into a 3D volume (refer to figure 2). CT-Pro allows for rapid (< 2min) reconstruction with limited input from the operator – reconstruction parameters are optimized automatically so that the quality of a scan do not depend on a qualitative opinion of the operator. The final product from a reconstruction in CT-Pro is a RAW 3D volume file which can be directly imported in VGStudioMax visualization software [7] that allows and enables the researcher to distinguish between different materials based upon segmentation.

2.2. Comparison between the MIXRAD facility and a similar facility in Germany

The capabilities of the MIXRAD facility are evaluated against a leading German facility located in Berlin (Helmholtz Zentrum-Berlin), which is also a government funded research facility. Numerous different types of samples were tested ranging from anatomical to geological samples. The custom made Helmholtz facility's micro-focus X-ray machine has a major advantage of a higher resolution detector with the detector pixel size of 0.05 mm whilst that of the MIXRAD facility is 0.2 mm. However, a major disadvantage of the Helmholtz facility is the sample size that can be accommodated since its detector is a quarter of the size of the detector at the MIXRAD facility. Figure 3 depicts the results (slices of a tomogram) of scans made on small coal particles (5mm diameter max) at the Helmholtz facility and MIXRAD facility respectively. It is evident that the capabilities of the MIXRAD facility are comparable to that of the facility at the Helmholtz Zentrum-Berlin in terms of resolution.

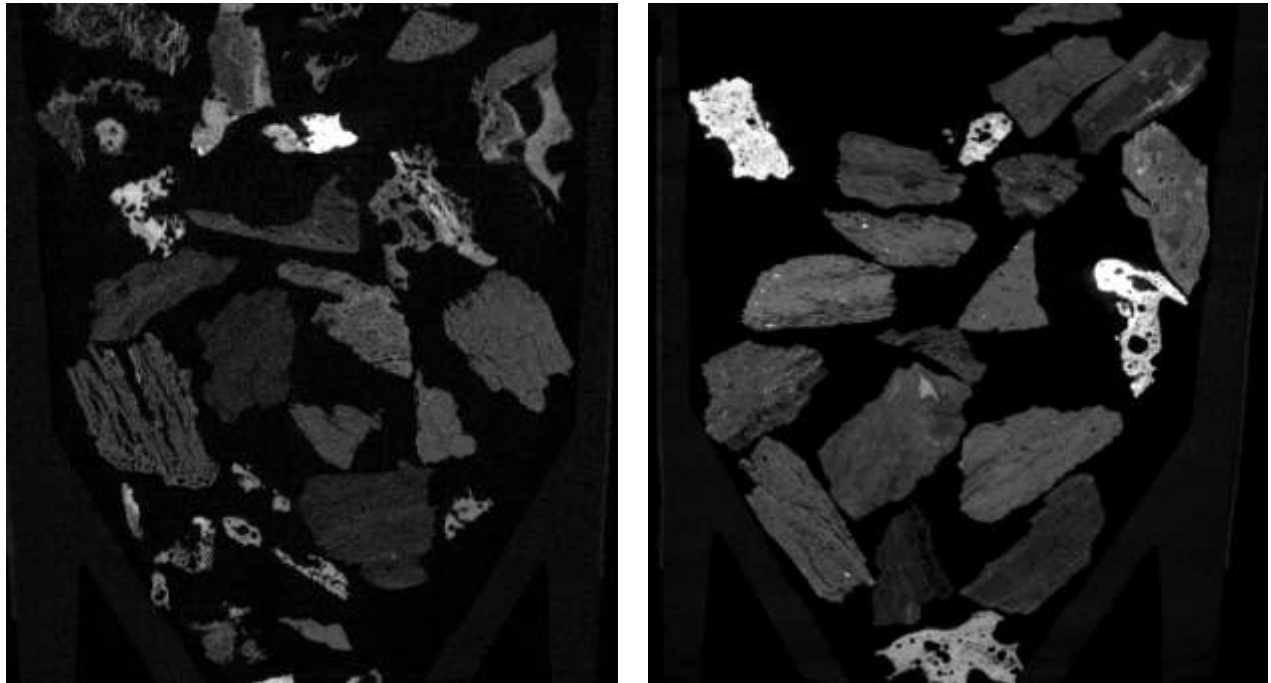


Figure 3 Coal sample scanned at Helmholtz (left) and MIXRAD (right)

The voxel size of the tomograms from Helmholtz and Necsa is respectively 0.0062 mm^3 and 0.0087 mm^3 - the magnification at both facilities is high and approximately the same. This indicates that the resolution of a micro-focus tomography scan is more dependent on the geometric enlargement capabilities of the source at very high magnifications than on the detector pixel size. The opposite is obviously true for very low magnifications.

2.3. Micro-focus X-ray tomographic investigations at MIXRAD

The capabilities of the MIXRAD system is being demonstrated in the next paragraphs by means of several case studies with examples that ranges from geology to biosciences.

2.3.1. Coal Sciences

Micro-focus X-ray tomography is not a new research technique in coal sciences and past research topics primarily consisted of evaluating a coal bed for carbon dioxide sequestration and methane extraction [8]. This evaluation consisted of quantifying the cleat/fracture network as well as non-destructive characterization of coal [9]. Very few researchers investigated a reactive process that occurs in the coal and consequently it was deemed appropriate to investigate coal pyrolysis and gasification at the MIXRAD facility. The spatial resolution ranged from 0.017 to 0.018 mm for all the scans and therefore it was possible to observe cracks and mineral inclusions larger than 0.018 mm.

It was possible to investigate the physical changes that occurred during pyrolysis and gasification by utilizing the non-destructive nature of micro-focus X-ray tomography. The changes in the same sample were observed by utilizing advanced volume rendering software like VGStudio, which enables the user to merge samples between successive reaction steps. Figure 4 indicates the development of fractures and pores within a coal sample at normal conditions as well as two different phases of pyrolysis. A non-destructive characterization prior to any reaction revealed information regarding the composition of the sample and the permeability of the organic matrix. This is especially important in making assumptions regarding the reactivity of the coal sample and where the most significant physical changes would occur.



Figure 4 Coal pyrolysis at different stages (left – baseline, middle – 50%, right – 100%)

Gasification is an industrially important reaction for the South African economy since numerous industrially important chemicals and fuels are produced through it. Pyrolysis is a physical process that is a precursor to the gasification reaction whilst the latter is a conversion process in which coal is transformed into syngas. There are numerous proposed reaction mechanisms that describe how gasification occurs and through micro-focus X-ray tomography it was possible to observe behaviour that is indicative of the shrinking core model. Figure 5 indicates the clear unreacted core of organic material in the coal sample that underwent 30% gasification.

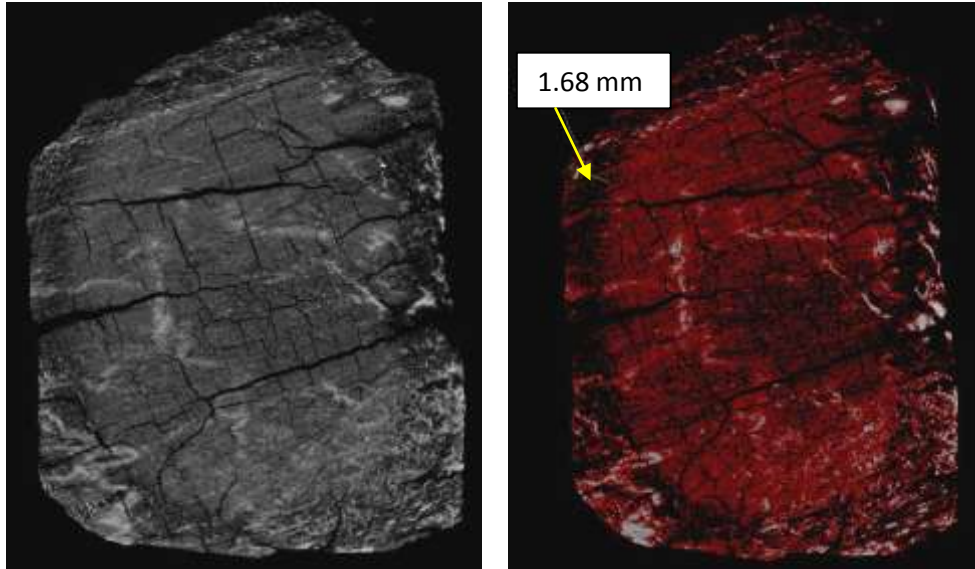


Figure 5 Coal sample at 30% gasification

2.3.2. Anatomy

Conventional medical X-ray CT imaging is expensive and the spatial resolution achieved is $\sim 0.500\text{mm}$ and significantly inferior than what is being achieved at industrial micro-focus X-ray systems with an intrinsic $0.001 - 0.006\text{ mm}$ spatial resolution capability. The MIXRAD facility enables anatomy students to obtain high quality research results for free. The capabilities of the MIXRAD system are being demonstrated through two anatomical research projects that are being considered a first in this field of applications. The spatial resolution was 0.069 mm in both cases since the samples were approximately the same size.

The Pterygopalatine fossa is a cavity on the side of the skull that is of importance in clinical applications. The maxillary nerve is present in this cavity and it is therefore vital to have knowledge of the Pterygopalatine fossa when administering anaesthesia in dental procedures. It is difficult for anatomy students to visualize this cavity since its shape is complex and the opening is covered by other bony landmarks. The entire cavity was extracted and visualized non-destructively for students to obtain a clear picture of the area and its location. Figure 6 (left) demonstrate the opening to the cavity in the skull whilst figure 6 (right) indicates the nerve openings inside the cavity.

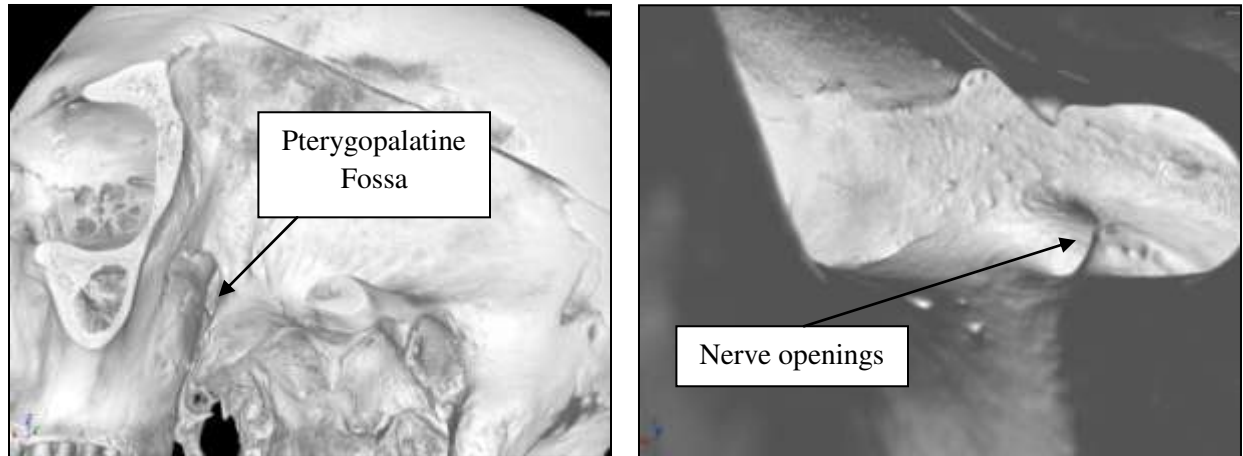


Figure 6 Pterygopalatine Fossa opening

A cochlea implant, which is an electrode implanted in the cochlea, restore hearing in numerous patients suffering from hearing loss. It is therefore necessary to have a complete understanding of the complex shape of each patient's cochlea and semi circular canals in order to model the implant. Micro-focus X-ray tomography enables researchers to fully extract the inner ear cavities and bones for modeling cochlea implants. Figure 7 (left) indicates the intricate shape of the cochlea and semi-circular canals whilst figure 7 (right) indicates the internal structure of one of the semi-circular canals. Micro-focus X-ray tomography is an invaluable research technique when the region of interest is small and this project proves the extreme capabilities of this technique since the whole cochlea is about 5 mm in diameter.

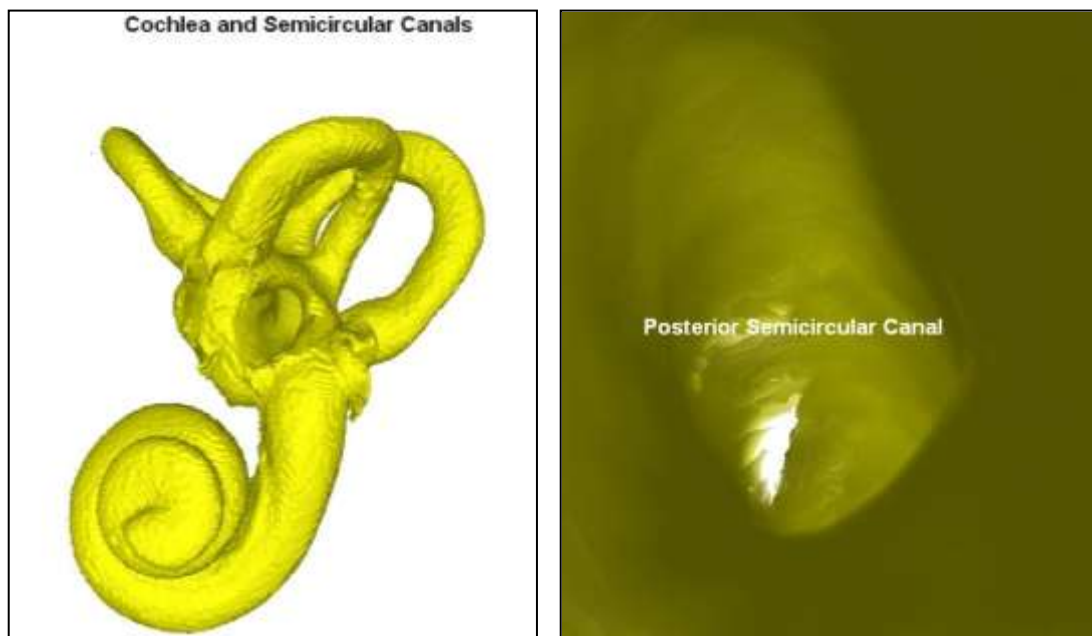


Figure 7 Cochlea and semi-circular canals

2.3.3. Agricultural Sciences

Maize forms an integral part of the diet of many South Africans and therefore scientific knowledge in this field of agricultural science is crucial. One problem that occurs with maize storage is the development of fungal infections when the maize is stored in silos. This presence of fungus obviously renders the entire stock in the storage silo inappropriate for human consumption and consequently an impact on the economy is inevitable. The modus operandi of the fungus is to infiltrate the kernel and start consuming the corn kernel at the germ and then moving inward into the kernel. The biggest problem in visual detection is that the damage by the fungus is only detectable at an advanced stage of infection. Figure 8 (left) indicates a corn kernel on the day of infection whilst figure 8 (right) indicates the same kernel after 18 days of infection. The damage is still not present on the outside of the kernel and it is possible to detect the damage earlier by using micro-focus X-ray radiography with a spatial resolution of 0.008 mm.

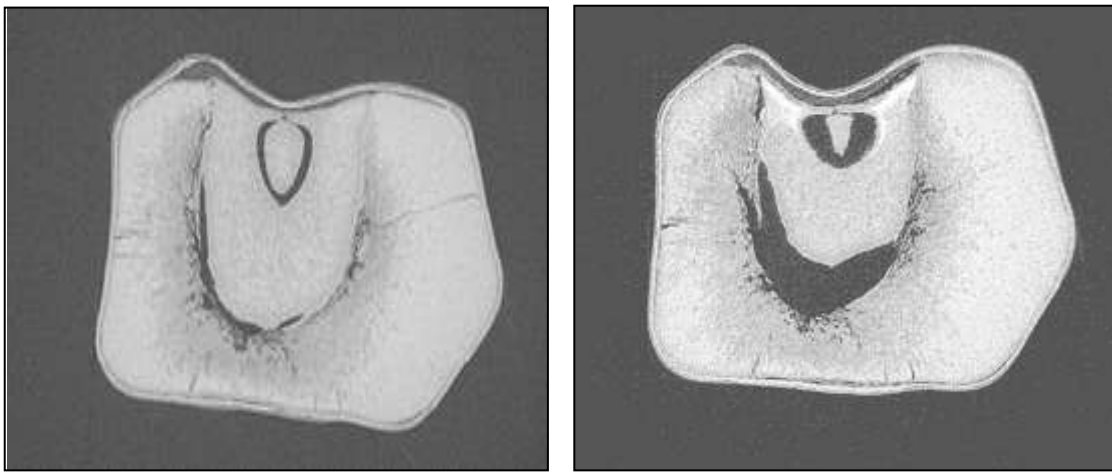


Figure: 8: X-ray tomogram slice of a Corn kernel (left - clean, right - infected)

South African economy also relies on exporting high quality oranges to foreign markets. A potential problem that can damage this market is insect infestation since foreign markets are very sensitive to infestations. The same problem (as with the kernels) of detecting an infestation early on is prevalent with the oranges. The larvae burrow through the skin of the orange by making a very small hole which is incredibly difficult to detect visually, especially at a large industrial scale packing plant. Micro-focus X-ray tomography makes it possible to visualize this infestation when there is still no sign of it on the skin of the orange since the spatial resolution of the scan is 0.045 mm. Figure 9 (left) indicates an orange prior to infestation whilst figure 9 (right) indicates an orange at an advanced state of infection. The red part is representative of the cavity that results due to activities of the larvae. This investigation served as a feasibility study to test different technologies to detect insect infestation in citrus fruit.

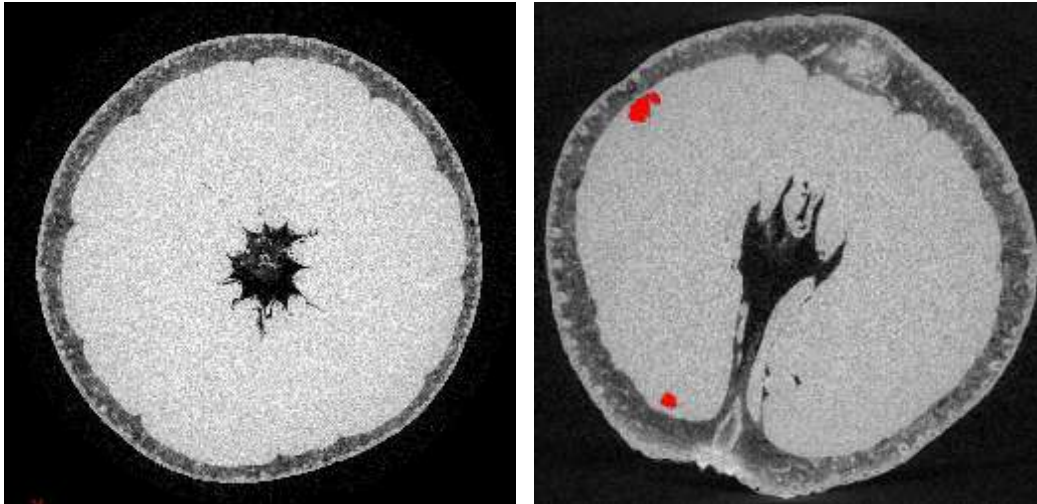


Figure 8 X-ray tomogram slice of an Orange (left - clean, right – infected red areas)

3. Conclusion

Micro-focus X-ray is a cutting edge technique that enables the user to study very intricate and fine samples in a non-destructive way. The MIXRAD facility, located at Necsa provides access for South African students and researchers to this technology and consequently enables them to gain experience in a relatively new but powerful non-destructive and non-invasive research- and analytical technique. The characteristics of the MIXRAD facility compared to those at German research facilities is similar and in some cases results in higher quality data.

Acknowledgements

The author would like to thank the staff at the Radiography and Tomography section (Radiation Sciences) of Necsa for assistance and support. The author would also like to acknowledge the support of the staff at the Helmholtz Zentrum-Berlin for their expertise in the field of micro-focus X-ray Tomography. The MIXRAD system was sponsored by DST-NRF.

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