

Cellular Architecture – Overview of X-ray and Neutron Radioscopy and Tomography

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Structure and microstructure analysis are very important to understand macroscopic properties like the mechanical, thermal or acoustic attributes of cellular materials. Metallographic methods can give us fundamental information about the ready sample, e.g. cell size distribution, critical cell wall thickness, cracks, etc. But in most cases the formation of the structure is the key process to be able, not only to understand the formation, but to influence it and the final structure in the desired matter. To be able to follow in-situ and non destructive the formation dynamic of cellular metallic structures, X-ray and neutron radioscopy and tomography have been used. In this contribution a description of the different beam sources, beam geometries, and comparison of these two methods show the advantages and limitations of different analysis configuration possibilities. Some results achieved with different methods are presented and examples of their contribution to the foaming development and foam properties improvement are given.

1 Introduction

For the improvement of metal foam technology the study of the foam structure, that correlates directly with the foam properties, and a closer process control are needed. To improve foam quality more knowledge about the physics of metal foaming is required. Real-time in-situ diagnostics are necessary in order to follow the dynamic of the process to understand, e.g., the kinetics of blowing agent decomposition and of pore growth, the effect of drainage or the details of foam degradation. For these investigations non-destructive methods such as X-ray radioscopy [1] and tomography [2] were proposed and successfully applied. X-ray radioscopy has been shown to be a powerful tool for real-time diagnostics, not only for the metallic foam process [3], but also for observing liquid metals [4,5], metal solidification [6], or flaw detection in Al castings [7]. Complementary to X-ray analysis neutron radioscopy and tomography are also more and more considered and applied [8] because of the specific advantages of this method like great penetration depth, specific atomic contrast, hydrogen sensitivity, etc. We will try to compare and emphasize the advantages of these complementary analysis methods, specially for the analysis of cellular material.

2 Principles

The way of interaction of the radiation with the matter determines the attenuation properties of the materials. The attenuation of the incident beam by the corresponding material can be described in the conventional radiography by an exponential function of two parameters – the material thickness $d = \int_{path} dz$ and the linear attenuation coefficient $\mu(x,y,z)$ for the corresponding material as shown in Eq. (1)

$$I(x, y) = I_0(x, y) \exp \left[- \int_{path} \mu(x, y, z) dz \right] \quad (1)$$

where the $I_0(x,y)$ and $I(x,y)$ are the beam intensities before and after the transmission in a plane (x,y) transversal to the propagation path z.

The attenuation coefficient $\mu(x,y,z)$ is a product of various physical events which lead to a beam attenuation. In the radiography theory we mainly consider two attenuation processes: absorption and scattering in the investigated sample. For radiography purposes in non-destructive investigations mainly two kinds of radiations are used: X-rays and neutrons. As a charge-neutral particle the neutron mainly interacts with the core of the atom in contrast to the X-rays which interact with the electronic shell. This determines the increase of the attenuation coefficients for X-rays with an increase of the atomic number of the elements, respectively, the number of the electrons in the electronic shell. The probability for interaction of neutrons with the atom core depends on a parameter called coherent scattering length a_{coh} which does not depend on the atomic number of the element. Therefore, the attenuation properties of the elements for neutrons are independent on the atomic number as shown in [Fig. 1](#).

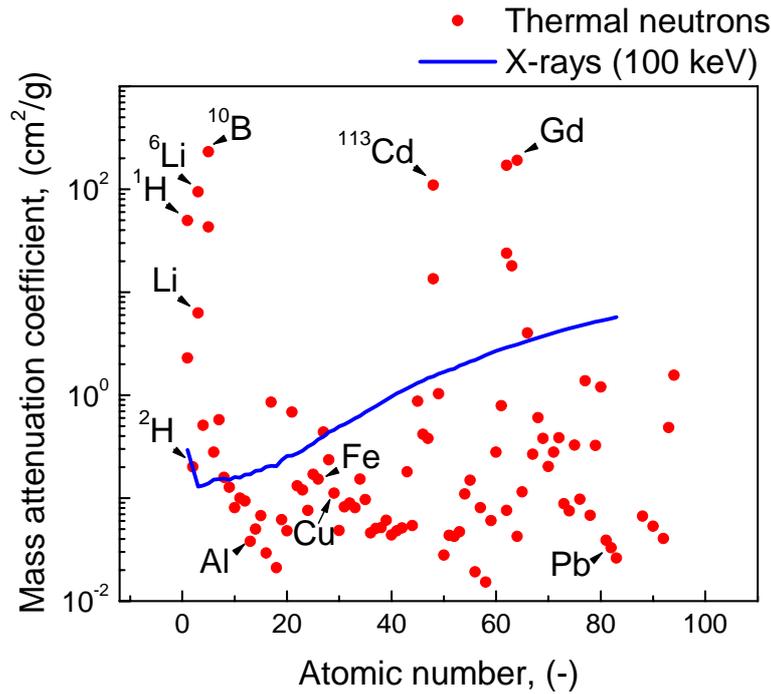


Fig. 1. Comparison of the mass attenuation coefficients for thermal neutrons and X-rays (100 keV) in dependence on the atomic number.

The attenuation properties of the materials depend on the energy of the applied radiation. For radiography purposes the energy of the neutrons is in the order of meV the so-called thermal or cold neutrons and the X-ray energies are in the order of a dozen to hundreds of keV. If we compare the mass attenuation coefficients for different elements in case of X-ray and thermal neutron transmission (see [Fig. 1](#)) the following main conclusions can be made:

- The neutrons are very sensitive to light elements like H, Li, B where the X-rays do not provide a good contrast.
- The distribution of attenuation coefficients for neutrons is independent on the atomic number which helps to achieve contrast even for neighbor elements, for X-rays it increases more or less exponential with the atomic number.
- The neutrons easily transmit metals like Pb, Fe and Cu which is difficult for the standard X-ray imaging facilities possessing energies of some hundred keV.
- The neutrons can distinguish between the isotopes of one element, for instance ^1H and ^2H , which is not the case for X-rays.
- X-rays allow a small penetration depth in the case of real-time radioscopy of Al metallic foams. This is an advantage because a reduced amount of cells can be studied this way.

In this context the two radiations appear to be complimentary in case of the radiography non-destructive investigations. To show the difference in the provided contrast from both radiations the following example is shown in [Fig. 2](#). Depending on the different absorption, different structures can better be realized with X-rays or with neutrons.

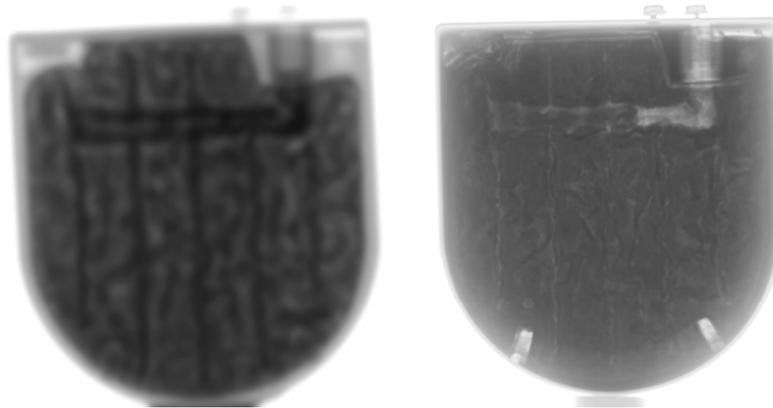


Fig. 2. Comparison between radiography images of LiI battery performed with neutrons (left) and X-rays (right).

3 Beam sources and imaging geometries

3.1 Sources

The sources for X-rays and neutrons determine the intensity of the radiation, the brilliance, if parallel or cone imaging geometry is used, if poly- or monochromatic radiation can be used, the possibility of real-time measurements, the detectors needed, etc., and of course also the beam disposability. The measurement time in a synchrotron or a neutrons reactor is limited, in comparison e.g. to a X-ray microfocus tube which can be used in a small lab.

3.1.1 X-ray sources

There are two main X-ray sources available for radioscopy and tomography, the X-ray tube with cone geometry and the X-ray synchrotron with parallel one. There are also other differences between them like the brilliance (see [Fig. 3](#)), the energy range, the irradiation section or the chromatism:

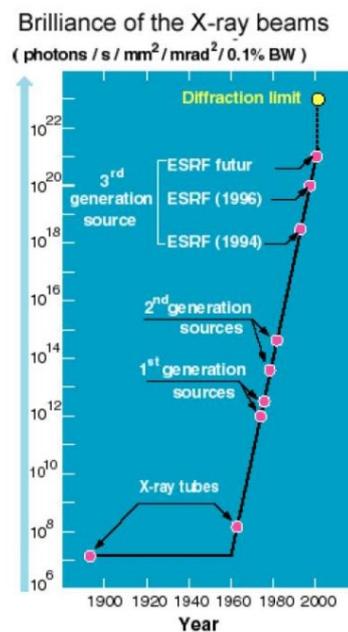


Fig. 3. Comparison of the brilliance of the X-ray beams for tubes and for different Synchrotron sources (source: ESRF [11]).

Synchrotron X-ray "light" is generated in electron accelerators and electron storage rings. The spectrum of the light emitted from these sources extends from the infrared to the vacuum ultra violet (VUV) and the X-ray region. BESSY [9], DESY [10], ESRF [11], SSRL [12], SRC [13], etc. are some of these facilities. The most important advantage of a synchrotron in comparison with a X-ray tube is their great brilliance (see [Fig. 3](#)) that is at least 10 orders of magnitude higher. But this is not the only difference, as we will see.

X-ray tubes for radiography and tomography are normally microfocus ones, with a higher acceleration voltage in comparison to tubes used for diffraction. The microfocus is necessary to reach enough resolution and the high voltage to have enough penetration depth. Their energies vary from 20 keV to several hundred keV. With increasing energy the penetration depth also increases, but that automatically means the reduction of spatial resolution (increase of the beam spot), because the focusing e-beam in the tube cannot exceed a certain energy density, in order not to melt the target material used. For this reason microfocus X-ray sources usually have a Tungsten target and the source current is limited. Due to the cone geometry relatively big samples can be irradiated and magnification is possible. At present the evolution of the microfocus sources is going on, e.g. transmission tubes are considered, with diamond targets and water cooling [14] to increase the brilliance and the penetration depth, but keeping a micrometer spot.

3.1.2 Neutron sources

To extract an intense neutron beam for radiography purposes a large scale facility like a research reactor or a spallation source is needed. In the research reactors, the so-called steady sources, the neutrons are produced due to the fission process in Uranium. The energy of the fission neutrons is in the order of a few MeV. These high energies are not appropriate for experimental purposes so the neutrons are moderated to energies of a dozen meV in a corresponding moderator, usually water or heavy water. Examples for research reactors in Europe are the powerful ILL reactor in Grenoble, France [15], two reactors in Germany – the new FRM 2 [16] and BER 2 [17], the reactor in Hungary KFKI [18] and so on. The advantage of a steady source in case of neutron imaging is the stability of the neutron flux with the time which is very important for the longer tomography experiments.

The spallation sources are accelerator-driven sources where the neutrons are produced at the excitation of the nuclei in a heavy-metal target (often Ta or W alloy) from which neutrons 'evaporate'. These fast neutrons are slowed in a moderator set around the target. The accelerator has a defined repetition rate giving a corresponding time structure of the neutron beam. This time structure is very appropriate for time-of-flight experiments and energy-selective radiography investigations, respectively. In Europe there are only two neutron spallation sources: SINQ at PSI in Switzerland [19] and ISIS [20] in England where the SINQ can rather be classified as a steady source due to its high repetition rate of 50 MHz.

From the view of the neutron radiography purposes both of the sources (reactors and spallation sources) provide enough high neutron flux of $\sim 10^6$ to 10^7 neutrons/cm²s at the sample position which is sufficient to perform a radiography experiment in the order of a dozen seconds. Exceptions are the high flux facilities at ILL and BER 2 ($\sim 10^9$ neutrons/cm²s) where a radiography image can be taken in one tenth of a second.

Another characteristic of the neutron sources is the mean energy of the neutron beam spectrum provided for experiments. As mentioned above the neutrons are moderated to thermal energies of a dozen meV, the so-called thermal neutrons. A further slow down is possible by the utilization of the so-called cold source where in a medium with a very low temperature, usually liquid deuterium at 30 K, the neutrons reduce their energy to a few meV, the so-called cold neutrons. Due to their low energy these neutrons have a higher probability for interacting with the matter and correspondingly the attenuation coefficients for the materials in this energy range are higher.

Other types of neutron sources for radiography purposes are the mobile sources based either on portable linear accelerators or a combination of neutron emitting isotopes. This type of source is currently with a poor performance providing a low neutron flux with no optimum image resolution and complicated shielding and operation systems. So this is the reason why they are not spread among the standard radiography methods.

3.2 Imaging geometries

Two beam imaging geometry configurations have been considered: The parallel and the cone beam geometry (see [Fig. 4](#)). In both cases the acquired projections have to be reconstructed for the tomography, in the same way as it is known from the medical tomography. This is made by an algorithm that applies a special inversed Fourier transformation, the so-called Radon transformation which is described in detail elsewhere [21].

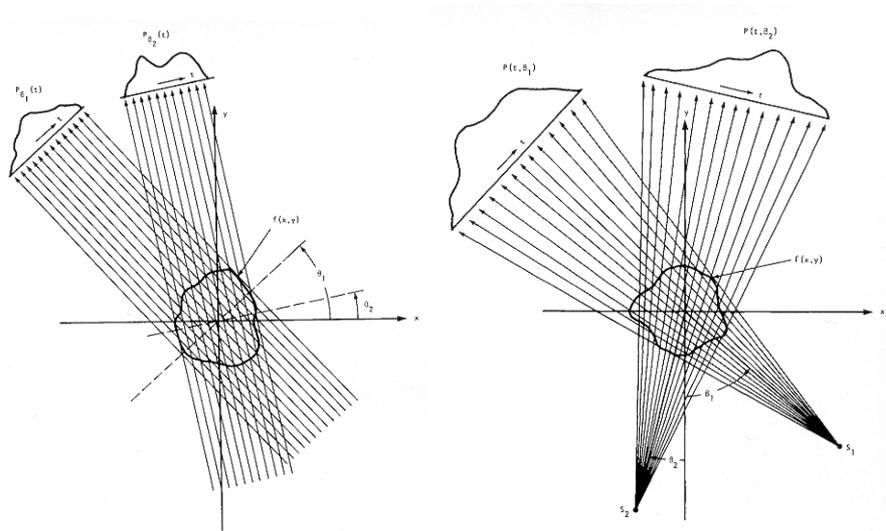


Fig. 4. Parallel (left) and cone (right) beam geometry for X-ray and neutron radioscopy and tomography [21].

The parallel beam geometry, typical for the neutron tomography, has the advantages that there is no distortion in the images like in the cone geometry (see [Fig. 4 \(right\)](#) and [Fig. 5](#)) and that the reconstruction algorithms of the pictures are more simple. Depending on the sample dimensions and imaging arrangement the distortion can be neglected (see [Fig. 5](#)). On the other hand the cone geometry allows magnification. This is important e.g. because the parallel synchrotron beams analysis area is normally limited to a small windows of a few square centimeters. Bigger samples cannot be studied or have to be scanned. Due to magnification in the cone geometry the resolution can also be increased if the detector pitch is small enough.

3.2.1 X-ray imaging arrangements

A single X-ray image can simply be recorded with a photo plate as we know from the medical radiographies. Series of analogue pictures for real-time radioscopy can be recorded with a video recorder and a video camera focused on a scintillation screen. As analogue single pictures are not appropriate for the tomography, digital recording is favored at present. X-ray detectors are based on a X-ray sensitive screen, an optical image amplifier, and a CCD camera. An alternative is the last generation of flat panel detectors where all detector components are integrated on a flat screen. They have a digital output with a resolution down to 50 μm pitch size and save place.

In the special case of parallel synchrotron X-ray radiation the typical imaging arrangement consists of a source, a beam line, a shutter, a filter, a monochromator, a slit, the sample and a detector. As we get a wide frequency spectrum of the light, a monochromator, e.g. a Si <111> crystal or multilayer, is used to filter this white radiation to the desired wavelength. The loss of intensity can be compensated through the high brilliance of a synchrotron. The beam size normally varies between some square millimeters to several square centimeters. Larger samples have to be scanned.

For a microfocus source the cone geometry has to be considered with a magnification geometry (see [Fig. 5](#)). One implication is the possibility to adjust the required magnification M . It is given by $M = b/a$ (a is the distance between source and sample and b between source and detector). Useful M 's can be as high as 10 for our configuration. To find the best imaging parameters with, e.g., the best spatial and time resolution and the desired magnification, other factors have to be considered such as the sample geometry, the source pitch (d_{source}), the detector pitch (d_{detector}) or the divergence distortion $d_{\text{div}} \approx t \cdot h / 4 \cdot a$ for $h \ll a$. At a given magnification a higher distance between source and detector will reduce d_{div} , but the intensity will also be reduced as $I \sim 1/b^2 \sim 1/M^2$. The resolution is determined not only by the detector pitch but also by the magnification and the source pitch.

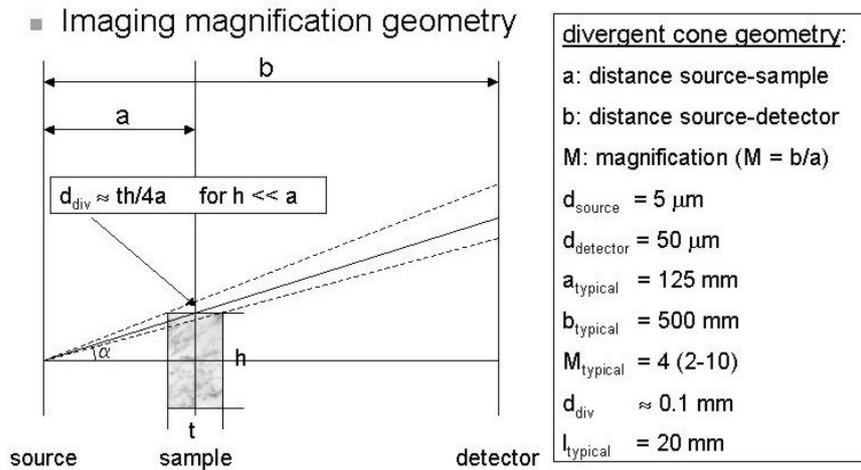


Fig. 5. Typical cone imaging geometry for metallic foams radioscopy [3].

As the absorption of X-rays increases with the atomic number, the dimensions of the samples are limited. Also the beam energy and the brilliance play a role. Typically they can vary from down to a millimeter to several centimeters. The sample dimensions that are allowed depend strongly on the absorption properties of the material.

3.2.2 Neutrons imaging arrangements

One neutron radiography facility consists of a collimation system which directs the neutrons to the sample, a sample environment which allows to scan the sample through the beam and to rotate it in case of a tomography investigation and a detector which converts the beam attenuation to a 2D image saved in an electronic format.

The neutrons, due to their electro-neutrality, could not be focused or deflected by electrical or magnetic fields like the X-rays or electron beams. So the only possibility to influence the neutron beam is to use a different combination of blends and slits made from high absorption materials like B_4C , Li or Cd. In the neutron radiography the collimator system usually consists of a large evacuated tube with blendes placed along the beam propagation increasing their aperture with the distance from the source, the so-called flight tube. This way a measure for the beam collimation can be defined as the ratio of the used collimator distance L to the diameter of the collimator entrance aperture D , the so-called L/D . The higher the L/D ratio the better beam collimation is achieved. Typical L/D values for standard neutron radiography facilities are between 200 and 500. The best L/D at the moment is achieved at the new neutron tomography facility ANTARES at FRM 2 in the order of 800 [22]. The beam collimator determines the limits in the spatial resolution of the recorded radiography images. At a defined L/D ratio each point of the sample will be projected on the detector plane as a spot with a diameter $d=l/(L/D)$, where l is the distance between the sample and the detector, Fig. 6. For tomography experiments we should rotate the sample around a defined axis which does not allow to use very close sample-to-detector distances. This makes the beam collimation parameter L/D very important for the quality of the radiography images. In this case at a fixed L/D ratio of 500 and given sample-to-detector distance of 5 cm the correspondent spatial resolution will be in the order of 100 μm .

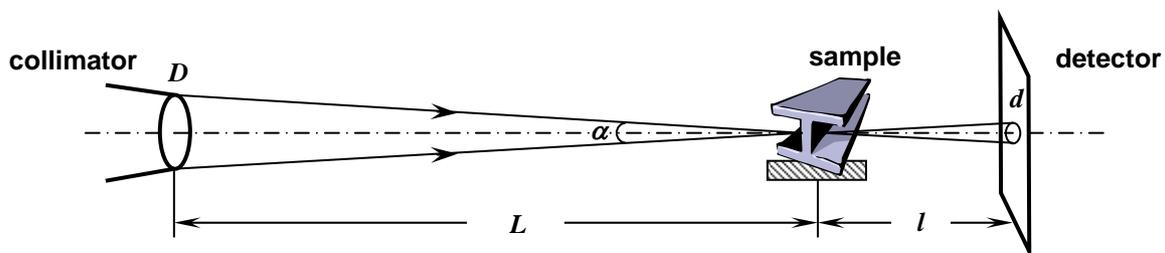


Fig. 6. Schematic representation of a neutron radiographic experimental geometry

Another parameter which determines the image resolution is the resolution of the detector system itself. The principle of the position-sensitive detectors is based on the detection of light photons obtained at the ionization

of a thin scintillation medium (called emitting screen or simply scintillator) from the used radiation. Since the neutrons could not ionize the medium we need some additional converter which captures the neutrons and emits some ionizing (secondary) radiation like X-rays or α -particles. Two types of converter materials are mainly used in the neutron radiography detectors: ${}^6\text{Li}$ (n,α) and Gd (n,γ). This secondary radiation is converted once again to light by a standard scintillating material such as ZnS where the mean free path of the secondary radiation in the scintillation material limits the spatial resolution of the detector in the order of $100\ \mu\text{m}$. A typical neutron radiography detection system is the combination of a scintillator screen which is observed by a CCD camera through a mirror and a lens system. Other often used detector systems are the imaging plates, flat panels and the classical X-ray film covered by a neutron to X-ray converter.

4 X-rays

4.1 Radioscopy

Real-time imaging

Recording a series of single radiographs of an expanding metal foam (see [Fig. 5](#)) allows us to follow the dynamic process in real-time. The X-ray intensity of a microfocus source is much smaller than that of a synchrotron source resulting in limitations with short exposure times imposed by the signal-to-noise ratio. However, with a measurement configuration like in [Fig. 5](#) the intensity is enough to get a satisfactory contrast for many applications, even for real-time picture acquisition at moderate image acquisition rates of around 1 Hz, even with the highest spatial resolution of $5\ \mu\text{m}$. However, time and spatial resolution are limiting each other. For very fast imaging the use of synchrotron rays is mandatory due to the higher brilliance.

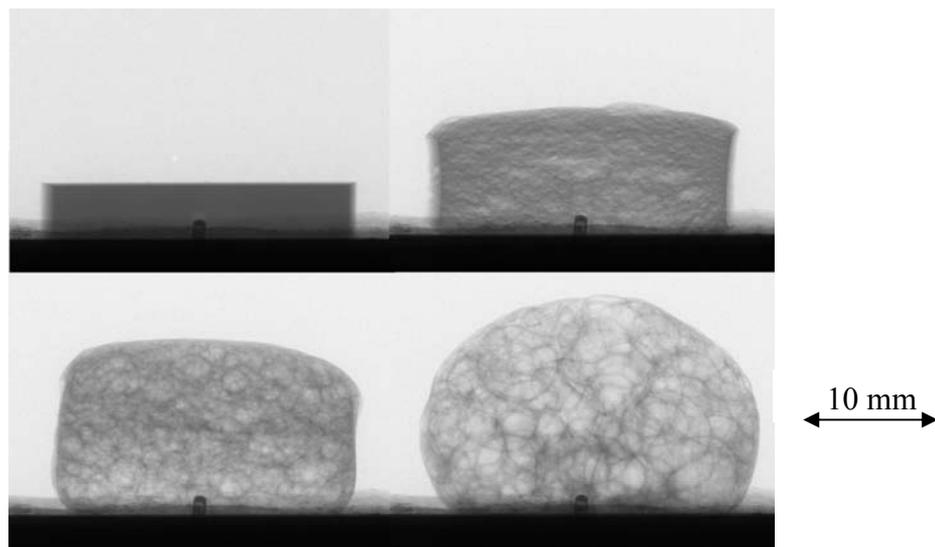


Fig. 7. Real-time X-ray radioscopy of an expanding Al-foam.

Phase contrast imaging

Contrast generation due to different absorption (attenuation) of the materials, thickness or element dependent, is the standard way of acquiring radioscopic pictures. But also in some special cases, where the absorption contrast is too poor, phase contrast can be used. In this case the phase of the X-rays changes due to the transition through media with different refractive indexes. Several radiographies, with different sample to detector distances, are taken, e.g. between 10 cm and 1 m. This method is described in detail e.g. by Cloetens et al. [23].

4.2 Tomography

Tomography is a powerful non-destructive method for the investigation of a large variety of different objects. It allows to visualize the inner volume of a sample without destroying or dismantling it. The tomography principle is based on the mathematical reconstruction of the 3-dimensional volume from 2-dimensional projection images collected while the sample is rotated around a defined axis. With the reconstructed data 3-dim. material and pore analysis are possible. Using filter and different algorithms very informative pictures can be obtained. Mostly one or several phases are shown or hidden to analyze their location and distribution. [Fig.](#)

shows a Zn-foam with small TiH_2 particles. A bimodal pore size distribution can be found. Depending on the software representation method (see Fig.) pores, matrix or particles can be visualized separately.

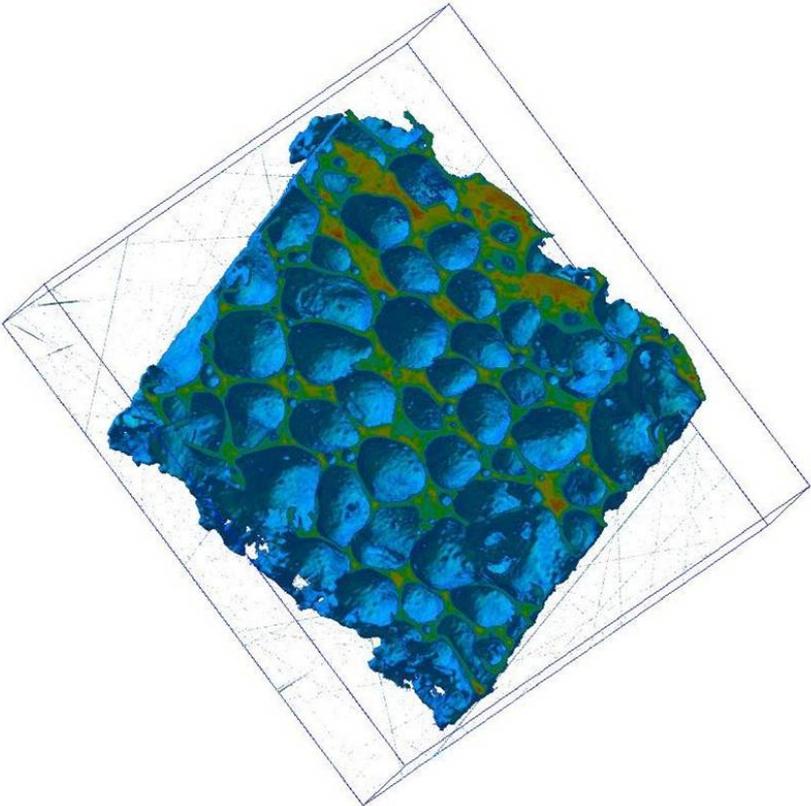


Fig. 8. Tomography of a Zn-foam with TiH_2 particles $< 28 \mu\text{m}$ [24].

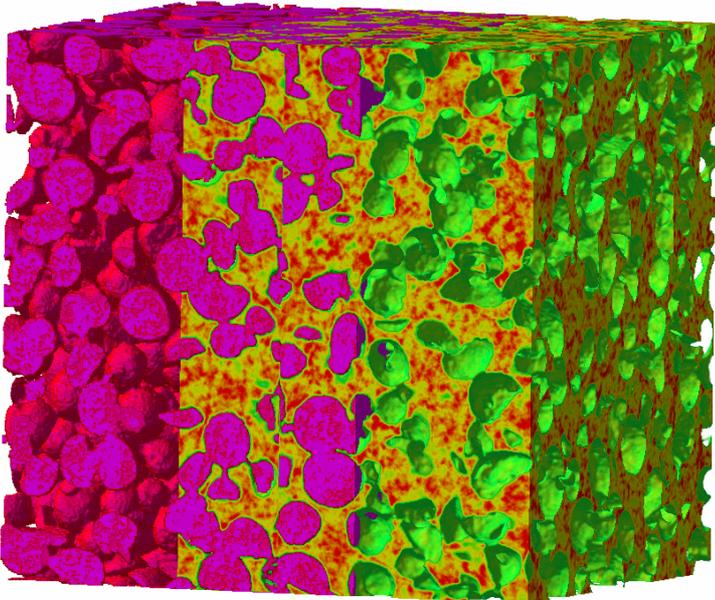


Fig. 9. Al-foam tomography reconstruction with different software representations. Pores, matrix or particles can be analyzed separately [24].

5 Neutrons

5.1 Radioscopy

Real-time imaging

The brilliance of neutron sources is much smaller than the synchrotron sources. The maximum possible thermal neutron flux of 3×10^9 neutrons/cm²s with a beam size of ca. 20 cm of diameter is available for radiography purposes at the radiography station at ILL called Neurograph [25]. High-flux radiography station using cold neutrons with a comparable flux is constructed recently at HMI, providing a rectangular beam size of 3×5 cm². Under such experimental conditions the possible shortest snapshot radiography times are in the order of some milliseconds. Some gain can be achieved if the so-called stroboscopic technique for repetitive processes is applied. An example for it is the presented below experiment performed at the cold neutron radiography station at HMI called CONRAD[26].

As a test sample for the real-time imaging experiment at HMI, a model air-craft combustion engine was investigated. The dimensions of the motor were (W×H×T) 70×70×30 mm³. For the first tests, the engine was driven by a coupled electromotor.

Data collection was based on the so-called stroboscopic method for investigations of repetition processes: a set of images was recorded exactly at the same position of the piston in the cylinder. After adding up all the images for the corresponding piston position, an average snapshot with better statistics was obtained. The whole combustion cycle was visualized by such “frozen” snapshots. Images at four piston positions are shown in [Fig. .](#) The next step in this work will be to investigate the fuel injection in a running motor driven by a combustion process.

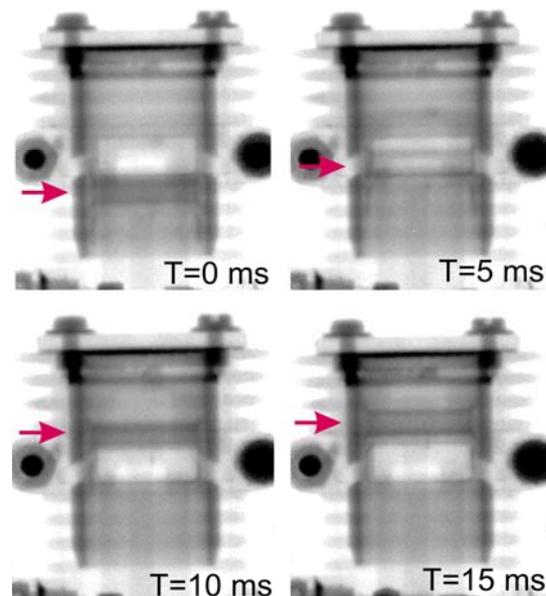


Fig. 10. Four neutron-radiography snapshots of a combustion engine at different piston positions. The arrows show the position of the piston. The experiment was performed at a rotation speed of 1110 rpm. The exposure time was 1 ms. For a defined piston position 200 images were recorded. The time delay between two piston positions was set to 1 ms.

Phase-contrast imaging

Analogous to the X-ray the neutron phase-contrast radiography is an edge-enhancement technique which allows to visualize objects which provide a very low contrast with the standard absorption radiography technique. The image formation in this case is based on the phase variations transformed to intensity variations in a beam with a high lateral coherence by its transmission of the object. Due to the fact that neutrons could not be focused in a spot with a micrometric size like X-rays we need a small diaphragm and a large distance between the diaphragm and the sample in order to achieve a good lateral coherence length in the neutron phase-contrast radiographic experiments. A comparison between phase-contrast and conventional radiography imaging of an aluminum foam sample are shown in [Fig. 6](#) [8].

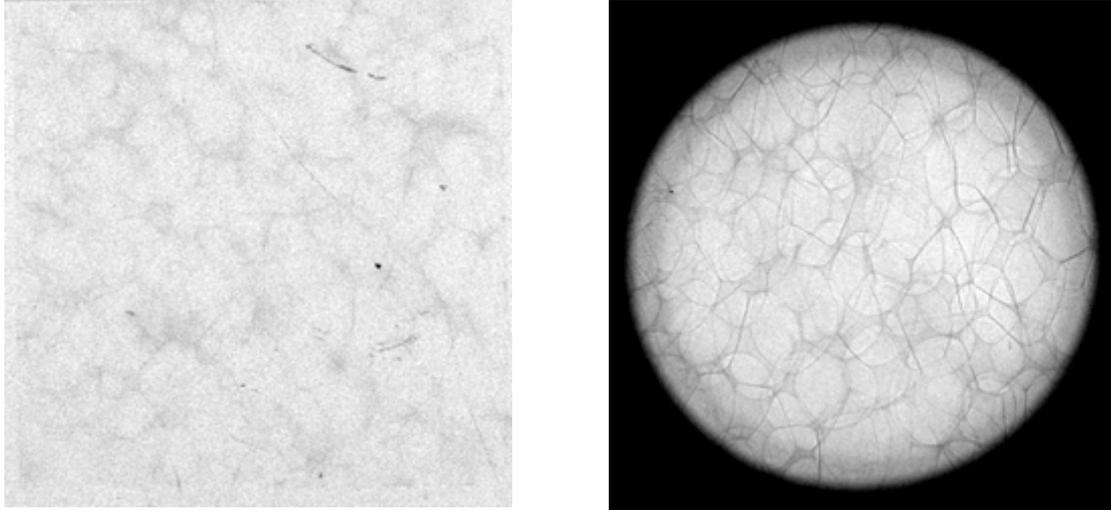


Fig. 6. Conventional thermal neutron radiography at 15 s exposure time (left) and phase-contrast neutron radiography (right) of a piece of aluminum foam. The phase contrast experiment was performed at: 120 cm sample to detector distance; 7 m source to object distance; pinhole: 0.5 mm and exposure time: 120 min.

This technique will be extended in the future providing a phase-contrast tomography option where the sensitivity of the neutrons to hydrogen can be used for an investigation of the gas diffusion in the foam after its production.

5.2 Tomography

The neutron tomography is analogue to the X-ray tomography. The limitation of the image resolution to 100 μm determines the number of projections used in the tomography experiments by the simple rule $M/N \sim \pi/2$, where M is the number of projections and N the number of used rays. Typical beam size in case of neutron radiography imaging is 10 x 10 cm^2 which determines 100 x 100 rays taking into account the resolution of 100 $\mu\text{m}/\text{ray}$ mentioned above. In this case for a neutron tomography experiment the number of projections is determined by $M = 100 \pi/2 = 157$. Using a CCD chip with 1024x1024 pixel with an integer format per pixel (2 Byte/pixel) and 200 projections per standard tomography experiment the data size for a tomography data volume could be estimated to be 500 MB which is no problem for existing computer systems nowadays. The measuring time at a standard neutron tomography facility is approximately 2-3 hours per experiment and the reconstruction procedure afterwards takes no less than 5-6 hours (ca. 20s per reconstructed slice) which means that for the maximum of 10 hours the tomography volume could be established. The next procedures which take much more time are the 3D-processing and segmentation of the data and the data archiving.

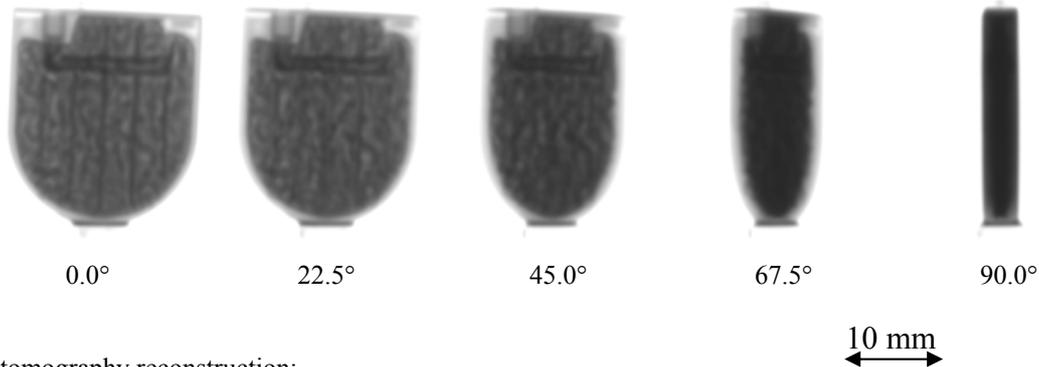
The neutron radiography is mainly used in cases where a high sensitivity to hydrogen is required or larger samples with high attenuation properties should be investigated. Some examples from the new neutron tomography facility at HMI are presented below.

Tomography investigation of a Lithium Iodide battery

Lithium (Li) has one of the highest attenuation coefficients for cold neutrons among the elements. So the task to investigate a Lithium Iodide (LiI) battery by means of neutron tomography was a challenge for the recently established tomography facility at HMI. Despite of the difficulty that for some of the 2-dimensional projections the neutrons had to pass through almost 3 cm of LiI, it was possible to successfully reconstruct the 3-dimensional image ([Fig. 7](#)).

The results of this investigation may help to increase the life-time of LiI batteries used in pacemakers.

2D-radiography projections at different rotation angles:



3D-tomography reconstruction:

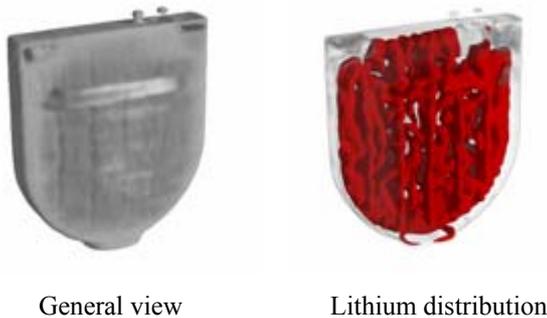


Fig. 7. Some of the 200 single neutron radiography projections (above) used for the 3D-tomography reconstruction (below).

Geological samples

In a further experiment, the density variation and the distribution of different minerals in a granite-sample were investigated ([Fig. 8](#)). This information helps to make conclusions about the formation of granite. In this case the presence of Kaolinit mineral (marked with orange) in the sample is an evidence that the granite material has underwent a hydrothermal alteration probably due to the flow of hot water through it. In this case the big Feldspar crystals frequently presented in granite have been transformed to Kaolinit by the following chemical reaction: $\text{KAlSi}_3\text{O}_8 + \text{H}_2\text{O} \rightarrow \text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. This kind of experiment may help to find a precise quantitative classification of different types of geological materials.

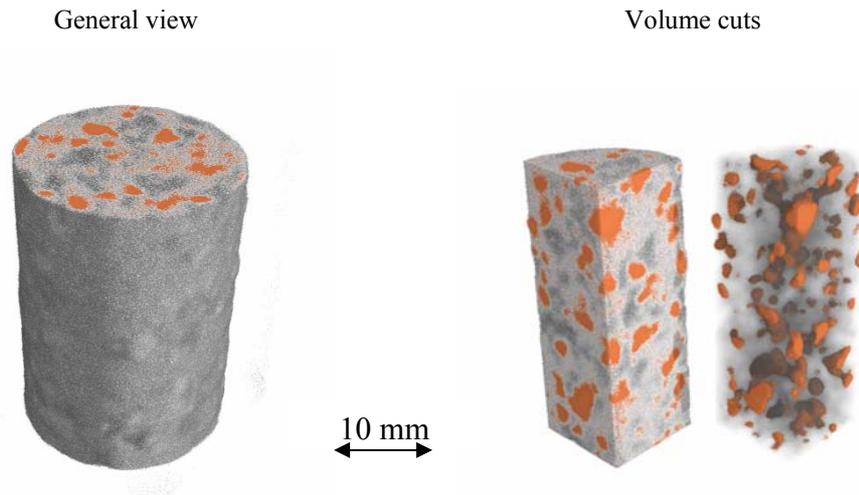


Fig. 8. The tomographical reconstruction of the granite cylinder shows the spatial distribution of the mineral Kaolinit ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$) marked with orange in the sample. The presence of four hydroxyl groups in the chemical composition leads to a strong contrast for Kaolinit crystals in the tomography reconstruction due to high scattering properties of hydrogen.

Conclusions

Non-destructive radiosopic and tomographic analysis methods are a powerful tool for material science development, especially for cellular materials. Several configurations are possible: neutrons or X-ray, large source or lab source, parallel or cone beam, attenuation or phase contrast, etc.

The neutron tomography appears as a complimentary technique to the X-ray imaging methods for cellular materials. It allows a higher sensitivity to hydrogenous materials and some light elements like Li and B which is very useful for investigations of glued joints of metallic parts, observation of diffusion processes of gasses and liquids in closed systems, water uptake and distribution in plants and building materials.

Some further developments in the X-ray and neutron detectors, sources and optics will help to improve the time resolution and the image resolution. Real-time radioscopy and tomography are very attractive techniques among the non-destructive material analysing methods.

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