Progress in industrial applications using modern neutron imaging techniques

Christian Grünzweig*, David Mannes, Anders Kaestner, Florian Schmid, Peter Vontobel, Jan Hovind, Stefan Hartmann, Steven Peetermans, and Eberhard Lehmann

*Corresponding author. Tel: +41 56 310 4662
E-mail address: christian.gruenzweig@psi.ch.

Paul Scherrer Institut, CH-5232 Villigen, Switzerland

Abstract
Neutron imaging as a technique for non-destructive testing has its application range where the more common X-ray methods will come to their limits: transmission through thick layers of heavy metals, detection of small amounts of hydrogenous materials and some other light elements like Boron or Lithium. There are at least three approaches for the further methodical improvements in respect to industrial applications: better spatial resolution, adequate time resolution and the optimization of the contrast between components. Dedicated examples for the methodical improvements are shown.

1. Introduction
The reason why neutron imaging (NI) is much less common as non-destructive testing method in research and for industrial applications can be found in the limited number of sufficiently strong neutron sources and suitable installation dedicated for this challenging technique. Presently, only a dozen facilities world-wide dispose of an adequate performance for studies on competitive high level. Nevertheless, NI has proved to be a powerful, competitive and promising methods for material research, for many industrial applications and as a tool for different branches in fundamental research. A major reason for the
progress is given by the development and application of dedicated digital neutron imaging devices. The exposure time has been reduced from hours to seconds and nowadays even down to micro second scale, which will allow for dynamic neutron radiography investigations.

As free particles, neutrons can be used for imaging like X-rays if a directed beam is transmitted through an object and the resulting neutron distribution is measured with a two-dimensional detector. The “shadow image” is produced by the interaction between the sample material and the neutron field. Although neutron imaging data look at a first glance similar to X-ray images it must be underlined that the interaction mechanism of the sample material with neutrons differs fundamentally from photon interaction. X-ray interaction with matter occurs with the electrons in the atomic shells of the inspected materials. Therefore, the interaction probability is directly proportional to the number of electrons, namely the atomic number. Light elements, like hydrogen, have little chance to absorb X-rays, whereas heavy elements absorb a lot. This has specific consequences for transmission and contrast: hydrogen is nearly invisible, but metallic layers absorb much of the X-ray beam.

Neutrons interact principally only with the atomic nuclei of the tested materials. Most of the relevant materials scatter neutrons at the nuclei, but there are some prominent materials with huge absorption capability like Gadolinium (Gd), Cadmium (Cd), Boron (B) and Lithium (Li). Thermal and cold neutrons have the ability of higher penetration through heavy elements (e.g. metals) whereas a high contrast is given for light materials such as hydrogen, lithium or boron. Also a isotope specific contrast can be achieved.

Hence, it depends on the elemental composition of the studied object or material whether X-ray or neutron imaging will be more the appropriate testing method.

1.1. Neutron imaging beamlines at PSI

The configuration and characteristics of every NI beamlines are special concerning spectrum and geometry. Each beamline was designed and optimized for the local conditions parameters of the neutron source. Relevant parameters for an NI-facility are for example the neutron spectrum, the distance between source and detector, the beam aperture, the beam size, the neutron flux and space in the experimental area. The PSI operates three neutron imaging beamlines; NEUTRA [1] and ICON [2] and BOA. NEUTRA was the first imaging beamline at the spallation neutron source SINQ and has been operational since 1998. ICON was taken into operation in 2005, while BOA is in operation since 2012. The three facilities differ in their specific properties. Depending on the studied question or object/material it has to be decided which of the available facilities is the most appropriate. To avoid flux losses from the source to the experimental position the beamlines are equipped with vacuum flight tubes. The experimental positions dispose of linear positioning units and turntables. All instruments are equipped with suitable digital detection systems, i.e. scintillator-CCD-camera systems, imaging plates. Due to a variety of camera and lens systems the beamlines represent versatile user instruments, which can be adjusted to the necessities of specific investigations.

NEUTRA disposes furthermore of an option for X-ray imaging, called X-TRA, based on a 320 kV X-ray tube. The X-ray tube can be moved into the radiation position allowing for investigations using similar imaging optics as for the neutron beam. Thus, the X-ray data can be compared or combined pixel-wise with the neutron data. All beamlines have similar infrastructure in terms of sample positioning and detector systems.

The main difference between the beamlines consists in the energy spectra available at each beam line; NEUTRA disposes of thermal neutron spectrum whereas ICON and BOA possess cold neutron spectra. The beamline BOA features furthermore the possibility of using polarized neutrons. The attenuation coefficients for materials are generally larger for cold neutrons. A large attenuation coefficient has the effect that smaller amounts of an element can be detected with cold neutrons, i.e. higher contrast can be achieved. Large attenuation coefficients do on the other hand limit the sample size as the beam intensity
is attenuated more rapidly.

2. Object dimensions and spatial resolution

At the spallation neutron source SINQ at PSI, for the ICON, NEUTRA and BOA beamlines mainly three different imaging setups for radiographic and tomographic investigations are available:

(i) *Maxi-setup* with a maximum field of view of 300 x 300 mm² and a resolution of 150 μm/pixel.
(ii) *Midi-setup* with a field of view ranging from 30 x 30 mm² up to 150 x 150 mm² with a resolution ranging form 30 μm/pixel up to 75 μm/pixel.
(iii) *Micro-setup* with a field of view of 27 x 27 mm² and a pixel size of 13.5 μm/pixel.

These detector systems differ in the field of view covered by the respective system as well as the resulting pixel size as measure for the spatial resolution. Hence, every setup covers a certain range with respect to the object dimensions observable and the spatial resolution obtainable with the respective system and guaranties a high flexibility. The following shows typical examples of samples from industrial applications area.

2.1. Results obtained with the maxi setup

The maxi-setup is generally used for investigations of large objects. The presented example is a canned diesel particulate filter (DPF) from a van (cf. Fig. 1(a)). The silicon-carbide monolith has a diameter of 144 mm (5.66”) and a height of 130 mm and is enclosed within a steel cylinder. The measurement of the entire canned DPF contains spatially resolved information on the local level of the deposits within the monolith. The neutron tomography (NT), with a resolution of 150 μm/px, is shown in Fig. 1(c). Fig. 1(b) shows a three-dimensional volumetric view of the DPF. One can clearly distinguish the monolith with its individual segments and the filter channels. For the NT measurement 625 projections over an angular range of 360° were taken with an exposure time of 20 s per projection. The examination of an outer segment using scanning electron microscopy (Fig. 1(c)) reveals the thickness of the soot layer (250 μm) on the filter wall.
2.2. Results obtained with the midi setup

A great number of samples, which have to be non-destructively characterized have dimension in the range of several centimeters. Such small to medium-sized objects can best be examined using the midi setup; this setup allows adjusting the field of view to the object size and thus the highest spatial resolution achievable a respective object. A typical example is shown in the following.

This example consists of a test sample, which was used to demonstrate the possibilities and limitations of investigations of glued metal samples; it shows a multilayered glued aluminium cube (edge length 100 mm), with varying layer thicknesses (1mm – 10mm). A photograph of the aluminium cube is shown in Fig. 2(a). The cube is composed of 14 glued metal layers with different thicknesses. The results of NT investigations in form of the volume rendered data are shown in Fig. 2(b). The individual slices of the NT data of the adhesive joints are shown in Fig. 2c, where 6 out or the 13 joints are shown. For the tomography 625 projections were recorded over an angular range of 360 ° with an exposure time of 25 s per projection. The resolution is ca. 150 μm/px with a given field of view of 150 x 150 mm².
2.3. Results obtained with the micro setup

The example for the high-resolution tomography with the micro-setup (resolution 13.5 μm/px) consists again of a part of the diesel particulate filter sample as shown in Fig. 1. For the high resolution measurements the sample had to be cut to fit the size to the field of view of the detector (27 mm x 27 mm). The sample is shown in Fig. 3 (a). 625 projections over an angular range of 360 ° with an exposure time of 90 s per projection were recorded. The three-dimensional representation of the tomography data is shown in Fig. 3 (b). With the help of the tomography data, the different materials could be segmented according to the histogram criteria, Fig. 3 (c). An animation of the high-resolution tomography can be downloaded from [4].
3. Time resolution

Dynamic neutron radiography as a non-destructive testing method made big steps forward in the last years. Depending on the neutron flux, the object and the detector a time resolution down to 50 μs per single frame is possible. In the case of repetitive processes the object can be synchronized with the detector and better statistics in the image can be obtained by adding radiographies of the same phase. In that respect, a combustion engine is an ideal sample for the explained technique, because the motor block of metal is relatively easy to penetrate. Several attempts have been made to perform dynamic neutron radiography experiments [6, 7], where it is reported that a small fired model aircraft engine was imaged, as well as four-stroke car combustion engines that was however externally driven by an electro motor.

3.1. Dynamic neutron radiography of a fired two-stroke engine

The example for the dynamic measurements consisted of a conventional two-stroke engine from a chain saw. As the chain saw engine was running fired the exhaust gases had to be removed from the measurement area. This was realized by an exhaust extraction system, which was additionally installed at the beam line for such measurements.

In collaboration between PSI and the Andreas STIHL AG & Co. KG company it was possible to image the moving parts in the engine of a two-stroke (TS 410A, 66.7 ccm, 4.4 PS) chain saw engine running fired in idle speed (3700 rpm) and at 8000 rpm. The goal of the investigation is to visualize the fuel and lubricant distribution inside the cylinder in real-time conditions under operation of the fired
motor.
To ensure a more clear view into the motor, the cooling rips were removed as shown in the static images in Fig. 4.

Figure 4 (a) Experimental setup for dynamic neutron radiography measurements with a two-stroke engine TS400 cut off saw (small image) from STIHL; static neutron image of the original engine (b) and the machined engine with removed cooling fins (c).
Figure 5 Dynamic neutron radiography measurements of the prepared two-stroke engine at different crank angle positions between the bottom dead centre (BDC) and the top dead centre (TDC).

For dynamic measurements the detector system and the running engine have to be synchronized,
because the engine ignites unevenly while running idle. Technically, the image acquisition was synchronized by a trigger signal taken from the crank angle position using chevron mark indicator. As the running process is cyclic, several radiographic images acquired at the same cycle position were cumulated into a single frame for the movie. The exposure time of a single image was 50 μs providing an angular resolution of the crankshaft position at idle speed (3700 rpm) of 1.1 degree and at maximum speed (8000 rpm) of 2.3 degree. Each frame in the movie is composed of 1000 single images. By giving a delay to the camera trigger signal a movie was recorded. Fig. 5 shows 6 out of 72 different crankshaft positions. The movie of the full motorcycle contains 72 frames. The full movie can be downloaded from [8]. For the measurements the spatial resolution of the detector system was about 100 μm.

4. Contrast enhancement

4.1. Influence of the spectrum

To show the influence of the neutron spectrum on the linear attenuation coefficient and thus the image contrast the hydrogen distribution in zircaloy cladding tubes of nuclear fuel rods was investigated. The main interest in this study was to quantify the hydrogen content in such zircaloy tubes, which necessitates a high sensitivity for the element of interest. Six zircaloy tubes (Ø 11mm) with different hydrogen to zircaloy ratios ranging from 0 to 1.92 at. % were tested. This samples set was investigated at the NEUTRA beamline with a thermal spectrum (λ mean= 1.8 Å), at the ICON beam line (λ mean= 3.1 Å) and the BOA beam line (λ max= 3.7 Å), both with a cold spectrum. Fig. 6 shows the results obtained at each beam line where the linear attenuation coefficient for each zircaloy tube with different hydrogen ratios is shown. Clearly seen is the higher sensitivity for a colder neutron spectrum.

The contrast increase due to the influence of the spectrum can also be used for investigations such as fuel cell research, inspections of the moisture content in wood or concrete.
Figure 6 The linear attenuation coefficients over the H/Zr-ratio at the NI-beamlines at the Paul Scherrer Institut featuring each a different energy spectrum. The $\Sigma_{net}$ values are normalized to the sample with the zero H/Zr ratio.

4.2. Contrast agents

For individual applications often the individual composition of materials does not provide enough contrast, as most often the layer thicknesses are too thin, or the material itself does not provide enough contrast. A typical example is the fired two stroke engine, already presented in the dNR-paragraph, where the fuel inside the combustion chamber can not be seen, as the amount of fuel is below the detection limit. To overcome this problem a contrast agent was developed, which should allow to enhance both, the contrast of the fuel within the neutron images and thereby improve the detection accuracy. The tracer is based on a gadolinium oxide nanoparticles which are physically dispersed in the fuel. Gadolinium is an element showing one of the highest attenuation coefficients for neutrons ($\Sigma=1500 \text{ cm}^{-1}$). The two-stroke fuel containing the contrast agent has an attenuation coefficient of $\Sigma=70 \text{ cm}^{-1}$, allowing to detect a fuel layer thickness of 10 microns. The experimental neutron imaging results for different layer thicknesses of this contrast-enhanced fuel in comparison with normal fuel is shown in Fig. 7.
The future task is to conduct the dynamic neutron experiment with the running two-stroke engine under the use of the contrast-enhanced fuel. The concept of fuel containing contrast agents was furthermore successfully realized for the contrast enhancement of Diesel fuel utilising Gadolinium molecular compounds. This fuel was used for loading diesel particulate filters to increase the detection efficiency of soot and ash [MTZ]. Many other applications where contrast-enhancing materials can be used are imaginable for example to raise the contrast in lubricants, solvents or even aqueous solutions.

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