

Progress in High-resolution Neutron Imaging at the Paul Scherrer Institut - The Neutron Microscope Project

# **Progress in High-resolution Neutron Imaging at the Paul** Scherrer Institut – The Neutron Microscope Project

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Abstract. Here we report the recent advances in the Neutron Microscope project at the Paul Scherrer Institut. We demonstrate the recent improvement on the capability of neutron imaging that allows us to acquire neutron images with isotropic spatial resolution of about 5 micrometres.

# 1. Introduction

Neutron imaging of the highest possible spatial resolution is a major demand of the neutron imaging user community with several scientific disciplines applying this technology. However, while X-ray imaging can be performed routinely with  $\mu m$  and even sub- $\mu m$  spatial resolution even using table-top instrumentation, the spatial resolution of isotropic neutron imaging has long been limited to the deca-micrometre domain [1-4].

The sub-10 micrometre spatial resolution neutron imaging has been hitherto routinely available (in one direction only) in the case of anisotropic imaging by using a tilted detector setup (Boillat et al. [5]). There are also hybrid detectors that apparently allow for sub-10  $\mu$ m spatial resolution of neutron imaging; however, their performance is currently rather limited due to their temporal resolution [6]. Another interesting development that may provide a sub-10 µm spatial resolution is based on axisymmetric focussing mirrors (so-called Wolter optics) that promise possible "dramatic

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improvements in the signal rate and (spatial) resolution" [7]. However, the current resolution of coldneutron Wolter optics is reported to be on the scale of many deca-micrometres ([7-10]).

A recent addition to high-resolution neutron imaging systems is a microscope developed within the "Neutron Microscope Project" at PSI by Trtik et al. [11]. The first prototype of the microscope (Neutron Microscope 1.01) uses a high numerical aperture magnifying lens, which projects the image from a 4- $\mu$ m thick gadolinium oxysulfide (Gd<sub>2</sub>O<sub>2</sub>S:Tb) scintillator screen onto a high-resolution pixelated detector magnified by a factor of 4.3. Because of this magnification factor, the pixel size of the neutron images made with the Neutron Microscope 1.01 reached 1.5  $\mu$ m, which translates to a true spatial resolution of 7.6  $\mu$ m [11]. Therefore, the first prototype of the Neutron Microscope delivered nearly fourfold enhancement of the spatial resolution compared to the standard neutron imaging available at PSI [4]. It should be highlighted here that, despite its name, the Neutron Microscope we are currently developing is not *per se* a neutron microscope that uses neutrons for magnification, because we do not focus neutrons using our device. Our microscope can be described more precisely as a "very high resolution, very high-numerical aperture optical microscope with the light input based on the high-performance neutron-sensitive scintillators".

In this paper, we report on a very recent enhancement of high-resolution neutron imaging capabilities within the "Neutron Microscope project".

#### 2. Status and preliminary results

For the further enhancement of our "Neutron Microscope", we employ a tailor-made dedicated objective with an even higher (five-times) magnification factor and link it with recently developed isotopically-enriched 157-gadolinium (capture cross-section 254,000 barn) oxysulfide scintillator screens [12].

The dedicated objective is composed of thirteen individual lenses. As the objective is to be intrinsically exposed to scattered neutrons and thus possibly suffer from radiation damage, the first three lenses (i.e. the ones closest to the scintillator) are made of a radiation hard glass. A schematic of an axial cross-section is shown in Figure 1. The objective is designed to provide a spatial resolution better than 5  $\mu$ m, while keeping the light transmission above 70% for the light spectral range of terbium doped gadolinium oxysulfide.



**Fig. 1:** Schematic of cross-sectional view of the PSI Neutron Microscope showing (from right to left): the sample holder (pink), adaptor with the scintillator screen (green and yellow), the dedicated magnifying objective composed of 13 individual lenses (light grey), and a CCD detector (red and black).

The dedicated objective is designed for a field of view of 10 mm x 10 mm and the object distance is 60 mm. The scintillator screen is based on the recently developed isotopically-enriched 157-gadolinium oxysulfide ( $^{157}$ Gd<sub>2</sub>O<sub>2</sub>S:Tb; hereafter referred to as 157-gadox). The thickness of the scintillator is approximately 3.5 µm, while the mean particle size of the 157-gadox powder was approximately 2.0 µm.

The magnifying objective linked with the isotopically–enriched 157-gadox scintillator was preliminarily tested at the ICON beamline, PSI Villigen. The test arrangement was positioned slightly downstream from measuring position No. 2 (L = 9.0 m). A full polychromatic beam with a 20 mm neutron aperture was utilized. Therefore, L/D ratio equalled 450.

The new spatial resolution test-object (a gadolinium Siemens Star) was developed in-house with the size of individual spokes as small as 4  $\mu$ m. The test object was imaged using the Neutron Microscope and a sCMOS detector of a pixel size of 6.5  $\mu$ m (Hamamatsu ORCA Flash 4.0). Given the magnification power of the objective, the nominal pixel size of acquired images is 1.3  $\mu$ m. Figure 2 shows the neutron image of the mentioned gadolinium Siemens Star test object. The images presented in Figure 2 are based on 33 single frames each with an exposure time of 30 seconds, giving a total exposure time of 16.5 minutes.



Fig. 2: Neutron radiography of the Siemens Star test object made with the PSI Neutron Microscope. The full field of view is shown on the left, and the right is zoomed-in to the high-resolution area. The Fourier ring correlation (FRC)-based spatial resolution of the image is equal to  $5.4 \mu m$ .

The spatial resolution of the images was assessed using the Fourier ring correlation (FRC) technique [13, 14] and was calculated to be 5.4  $\mu$ m. However, as can be seen in Figure 2, some (albeit faint) modulation was revealed at the thinnest parts of the spokes (4  $\mu$ m). When compared to the spatial resolution available from the Neutron Microscope prototype 1.01, the spatial resolution improved by about 30%.



Fig. 3: Neutron radiograph of 'Wood's metal' alloy based on the Neutron Microscope test arrangement. The image clearly shows dark contrasting segments representing the cadmium containing microstructural features.

In addition to the test object, we also imaged a thin sample (about 0.3 mm) of the Wood's metal alloy. The mean distance between the sample and the scintillator was approximately 0.5 mm. The Wood's metal is a eutectic alloy composed of bismuth, lead, tin, and cadmium (about 10%) with a melting point of approximately 70°C. As bismuth, lead, and tin are rather neutron-transparent, the vast majority of the contrast in the neutron image is therefore based on cadmium (capture cross-section 2,450 barn). The neutron radiography clearly revealed needle/platelet-like structures of cadmium in the microstructure of Wood's metal alloy. Dynamic studies of relatively slow processes of solidification/segregation of such eutectic alloys is therefore one of the application fields foreseen for the Neutron Microscope instrument.

#### 3. Outlook & discussion

The neutron capture efficiency in thin gadolinium-based scintillators has been recently improved by the provision of the isotopically-enriched version of this phosphor [12]. Next, we will attempt to produce microstructured scintillators based on this phosphor. The challenge of production of such scintillator screens lies in the limited amount of the input materials (both for the production of the isotopically-enriched phosphor itself and the manufacturing of the scintillator screens using such isotopically-enriched phosphors).

In order to incorporate the Neutron Microscope instrument into the user-programme of Paul Scherrer Institut, a new sample positioning stage with superior accuracy must be designed and implemented. operate Neutron Microscope The plan is to the device as а self-contained instrument independent from the current operating infrastructure of the PSI neutron imaging beamlines. This plan has two major consequences:

- (i) it will be possible to transfer the Neutron Microscope between different beamlines with only a moderate effort,
- (ii) due to the limited field of view of both the Neutron Microscope and the standard high-resolution setup (MICRO-setup) with respect to the size of the beam (e.g. at the measuring position 2 at ICON beamline), it is foreseen that both of the devices can be operated at the same time, thus utilizing the available (and costly) neutron beamtime more effectively.

It is foreseen that high-aspect ratio neutron beam collimators based on microchannel plates will be developed for both conditioning the divergence of the primary neutron beam and to suppress the signal from scattered neutrons [15]. As these collimators will lead to a decrease in the useful neutron beam intensity, the more efficient scintillators, and the tailored magnifying optics (as described above) will prove very important for the further successful development of the Neutron Microscope facility. In addition, the temporal resolution of imaging using the Neutron Microscope can be even further improved by using neutron sources with higher flux than that available at SINQ, PSI (e.g. ANTARES and potentially ESS and ILL).

## 4. Conclusions

In this paper, we reported on the progress of high-resolution neutron imaging at Paul Scherrer Institute under the Neutron Microscope Project. We demonstrated that the tailor-made high-numerical aperture magnifying optics linked together with a thin isotopically-enriched <sup>157</sup>Gd<sub>2</sub>O<sub>2</sub>S:Tb scintillator screens can provide images with a spatial resolution of about 5  $\mu$ m, which translates to approximately 30% enhancement in spatial resolution compared to the first Neutron Microscope prototype 1.01 [11].

## 5. Acknowledgements

The project is supported by financial contributions from SNF R'Equip, CCMX and PSI-CROSS funding bodies. The magnifying objective was designed in collaboration with Braunecker Engineering GmbH (Dr Bernhard Braunecker, Dr Massimo Biber) and produced at POG AG, Gera, Germany. Mr Heinz Richner and Mrs Gabrielle Peschke (ETH Zurich, Switzerland) are kindly thanked for the provision of the sample of Wood's metal alloy. We gratefully acknowledge manifold support for mechanical engineering by J. Hovind, M. Blumer, J. Welte and M. Meyer (all PSI). Our thanks also go to Ch. Grünzweig, A. Kaestner and P. Boillat (all PSI) for their submission of test devices and other important assistance with setting up the Neutron Microscope. Also, we kindly thank Ch. David and T. Neiger (both PSI) and the staff of Dr Gerhard Gassler GmbH for assistance with the production of scintillator screens. Last, but not the least, we would like to thank an anonymous reviewer for the kind expression of several comments that improved the quality of this paper.

- [1] Kaestner, A.P, Hartmann, S., Kühne, G., Frei, G., Grünzweig, C., Josic, L., Schmid, F., Lehmann, E.H, 2011. The ICON beamline – A facility for cold neutron imaging at SINQ, Nuclear Instruments and Methods in Physics Research A. 659, 387–393
- [2] Williams, S.H., Hilger, A., Kardjilov, N., Manke, I., Strobl M., Douissard, P.A, Martin, T., Riesemeier, H., Banhart, J., 2012. Detection system for microimaging with neutrons. Journal of Instrumentation 7, P02014
- [3] Tremsin, A.S., Kardjilov, N., Dawson, M., Strobl, M., Manke, I., McPhate, J.B., Vallerga, J.V., Siegmund, O.H.W., Feller, W.B., 2011. Scatter rejection in quantitative thermal and cold neutron imaging. Nuclear Instruments and Methods in Physics Research Section A. 6
- [4] Lehmann, E.H., Frei, G., Kuehne, G., Boillat, P., 2007. The micro-setup for neutron imaging: A major step forward to improve the spatial resolution. Nuclear Instruments and Methods in Physics Research A 576 (2-3): 389-396
- [5] Boillat, P., Kramer, D., Seyfang, B., Frei, G., Lehmann, E., Scherer, G., Wokaun, A., Ichikawa, Y., Tasaki, Y., Shinohara, K. 2008. In situ observation of the water distribution across a PEFC using high resolution neutron radiography, Electrochemistry Communications, 10, 546–550, doi:10.1016/j.elecom.2008.01.018,
- [6] Vavrik, D., Jakubek, J., Krejci, F., Zemlicka, J., Kaestner, A.P., 2014. Modular pixelated detector system for neutron imaging with micrometric resolution. In 10<sup>th</sup> World Conference on Neutron Radiography, Grindelwald, Switzerland
- [7] Liu, D., Hussey, D., Gubarev, M. V., Ramsey, B. D., Jacobson, D., Arif, M., Moncton, D. E., and Khaykovich, B., 2013. Demonstration of achromatic cold-neutron microscope utilizing axisymmetric focusing mirrors, Applied Physics Letters, 102, doi:10.1063/1.4804178

- [8] Trtik, P., Lehmann, E.H., 2013. Comment on "demonstration of achromatic cold-neutron microscope utilizing axisymmetric focusing mirrors" [Appl. Phys. Lett. 102, 183508 (2013)], Applied Physics Letters, 103, 236101
- [9] Liu, D., Hussey, D., Gubarev, M. V., Ramsey, B. D., Jacobson, D., Arif, M., Moncton, D. E., Khaykovich, B., 2013. Response to "comment on 'Demonstration of achromatic cold-neutron microscope utilizing axisymmetric focusing mirrors" [Appl. Phys. Lett. 103, 236101 (2013)], Applied Physics Letters, 103, 236102
- [10] Hussey, D., 2015. A Neutron Microscope for Energy and Materials Research, presented at VI European Conference on Neutron Scattering, ECNS2015, Zaragoza, Spain. September 2015
- [11] Trtik, P., Hovind, J., Gruenzweig, Ch., Bollhalder, A., Thominet, V., David, Ch., Kaestner, A., Lehmann, E. H., 2015. Improving the spatial resolution of neutron imaging at Paul Scherrer Institut – The Neutron Microscope Project, Physics Procedia, 69, 169-176
- [12] Trtik, P., Lehmann, E. H., 2015. Isotopically-enriched gadolinium-157 oxysulfide scintillator screens for the high-resolution neutron imaging, Nuclear Instruments and Methods in Physics Research Section A, 788, 67–70, doi:10.1016/j.nima.2015.03.076
- [13] van Heel, M., Schatz, M., 2005. Fourier shell correlation threshold criteria. Journal of Structural Biology.151, 250–262
- [14] Vila-Comamala, J., Diaz, A., Guizar-Sicairos, M., Mantion, A., Kewish, C.M., Menzel, A., Bunk, O., David, C., 2011. Characterization of high-resolution diffractive X-ray optics by ptychographic coherent diffractive imaging. Optics Express. 19, 21333-21344
- [15] Tremsin, A.S., Kardjilov, N., Dawson, M., Strobl, M., Manke, I., McPhate, J.B., Vallerga, J.V., Siegmund, O.H.W., Feller, W.B., 2011. Scatter rejection in quantitative thermal and cold neutron imaging. Nuclear Instruments and Methods in Physics Research Section A. 651, 145-14