Neutron microtomography of MgB$_2$ superconducting multifilament wire

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Abstract

Neutron imaging of sub-10-micrometres spatial resolution has been recently achieved in 2D mode within the framework of the Neutron Microscope project at the Paul Scherrer Institut. Here we report on the development of the PSI Neutron Microscope instrument and the results of the first microtomographic imaging experiment of multifilament superconducting MgB$_2$ wire. The sample of MgB$_2$ superconducting 37 multifilaments embedded in copper-nickel matrix was investigated – in microtomographic mode – with the scientific interest regarding the distribution of boron within the individual superconducting filaments (about 40 µm in diameter). The resulting tomographic dataset revealed the distribution of boron within the entire 0.8 mm thick multifilamental wire with the isotropic voxel size of 2.6 micrometres.

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1. Introduction

1.1. Introduction - Neutron Microscope

The sub-10 micrometre spatial resolution neutron imaging has been recently achieved in 2D mode within the framework of the Neutron Microscope project at the Paul Scherrer Institut (PSI) [1,2]. This progress has been enabled by the development of the isotopically-enriched gadolinium-based scintillator screens [3]. In this paper, we introduce the instrument allowing acquisition of the three-dimensional neutron microtomographic images and show the first pilot results of the prospective user application.

Figure 1 presents a drawing the Neutron Microscope instrument. The high-numerical aperture magnifying objective equipped with a CCD camera is shown in two possible mounting positions (vertical and horizontal). The instrument is equipped with the high-precision positioning stages for the sample and multichannel plates. Also, the instrument is shown to be equipped with an optional X-ray source.

Due to the magnification of the objective, the focusing of the instrument is performed at the camera side of the instrument. The instrument is equipped with the high-precision six-axes sample positioning stage and a stage for the...
positioning of a multichannel plate collimator/scatter rejector. Also, thanks to the sensitivity of $^{157}$Gd$_2$O$_2$:Tb scintillator screen, the instrument can be optionally equipped with an X-ray source, thus providing an option for a future combined neutron/X-ray investigations. The entire arrangement shown in Figure 1 is designed as a self-contained instrument that is not tied to a particular neutron beamline and can be transferred between beamlines at PSI or even to the other neutron imaging facilities. However, the major part of development of the instrument has been performed at the ICON beamline at PSI [4], for which the instrument is designed in such a way that it can be used simultaneously with the existing Micro-setup instrument [4], thus making optimal use of the available neutrons at the ICON beamline.

1.2. Introduction - MgB$_2$ multifilament wires

Superconducting multifilamental MgB$_2$ wires are candidate conductors for the Superconducting Link Project at CERN [5].

![Fig. 2. Back-scattered electron image of the cross-section of MgB2 superconducting multifilamental wire. MgB2 – black, Nb – light grey, Ni-Cu alloy – dark grey](image)

Figure 2 shows metallographic cross section of an ex-situ wire with 37 superconducting MgB$_2$ multifilaments (Columbus Superconductors SpA). The wire was produced by the powder-in-tube (PIT) method. In this method, MgB$_2$ powder is inserted in Nb tubes, which also acts as a chemical diffusion barrier, and are subsequently cold drawn. Such tubes are then stacked into a second metallic tube (nickel-copper alloy) and cold drawn to a desired wire size. In the final step, MgB$_2$ powder is sintered by a heat treatment.

The mechanical properties [6] and hence distribution of MgB$_2$ powder (or the porosity) within the individual filaments are of interest for the manufacturers. Such distribution of the powder within the individual filaments have been hitherto investigated using FIB-nanotomography [7], that is spatial resolution-wise superior to the neutron imaging, but probes a limited sample volume.

Consequently, a sample of MgB$_2$ superconducting 37-multifilament wire was investigated in 3D using the Neutron Microscope at PSI.

2. Test arrangement, results and discussion

The Neutron Microscope has been placed at the ICON beamline (flux $\sim 10^7$ n cm$^{-2}$ s$^{-1}$) at a distance L=9.0 m downstream the beam defining aperture. The 20-mm aperture was used throughout the entire experiment, therefore the L/D ratio equalled 450. In order to limit the neutron dose on the instrument a $10 \times 10$ mm $\times$ mm beam limiting aperture was placed about 400 mm upstream the detector.
The wire of approximately 0.8 mm in diameter was attached to the sample holder using a UV-hardening glue in such a manner that the sample to detector distance was not larger than 2.5 mm.

An isotopically-enriched gadolinium oxysulfide ($^{157}$Gd$_2$O$_2$S:Tb) scintillator screen [4] of about 3.5 micrometres in thickness was used throughout the experiment. The Neutron Microscope was equipped with a sCMOS camera (ORCA Flash 4.0, Hamamatsu). The nominal image pixel size in this arrangement was equal to 1.3 micrometres and the field of view was about $2.7 \times 2.7$ mm × mm, however, the detector chip was binned by factor 2 in both directions leading to the resulting pixel size of the image to be equal to 2.6 micrometres.

181 projections spaced equiangularly over 180 degrees were acquired. In order to alleviate the negative influence of the gamma-spots in the projection images, the individual projections were taken with the exposure time of 30 s and 18 individual projections were taken at each angular position. Above that, 50 open beam images for the image normalization were acquired both before and after the tomographic experiment. Thus, the total acquisition time for the individual projection was equal to 9 minutes and hence the data acquisition of the entire tomogram lasted about 30 hours.

Figure 3a shows a horizontal slice from the reconstructed 3D microtomographic dataset showing –due to the neutron contrast of the elements occurring within the sample– the distribution of boron. The non-circular cross-sections of the 37 MgB$_2$ filaments (of approximately 40 micrometres in size) can be clearly observed. Above that, a rather transparent part of the Ni-Cu matrix can be also faintly distinguished from the outside air. Figure 3b presents the rendering of the boron rich areas based on the reconstructed microtomographic dataset.

The spatial resolution of the resulting 3D dataset was assessed by the Fourier shell correlation technique [8,9] and was found to be equal to 10.5 micrometres. Regarding the multifilamental MgB$_2$ wires, no clear inhomogeneity in boron distribution could be observed within the individual filaments themselves.
3. Conclusions and outlook

From the point of view of the detector development, we are pleased to report on the acquisition the first neutron microtomography at PSI of about 10 micrometre isotropic spatial resolution using Neutron microscope at ICON beamline. Further improvements in the technique are foreseen in the direction of the use of the microscope at (i) different beamlines within PSI (namely, POLDI beamline) or (ii) at other (more powerful) neutron sources, such as ANTARES beamline, MLZ, ILL, or in the more distant future, at ESS and PIK neutron sources.

From the point of view of the investigation of the MgB$_2$ superconducting wire, no clear areas of deficiencies in boron distribution were revealed in this pilot investigation. This indicates that all the inhomogeneities within the individual filaments are of smaller dimensions than the current spatial resolution of the technique, i.e. smaller than 10 micrometres. This seems to be true for the sample entire wire of 2.7 mm in length, which represents much more representative sample volume than that hitherto investigated by focused ion beam nanotomography. Further investigations of such superconducting wires using neutron microtomography are foreseen.

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