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High resolution Bragg edge transmission spectroscopy at pulsed neutron sources: Proof of principle experiments with a neutron counting MCP detector

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ABSTRACT

The high spatial and temporal resolution of a neutron counting detector using microchannel plates (MCPs) combined with Medipix2/Timepix readout can substantially improve the spatial resolution of neutron transmission spectroscopy, as shown in our proof-of-principle experiments. Provided that the neutron fluence and data acquisition time are sufficient, transmission spectra can be acquired in each $55\times55\ \mu m^2$ pixel of the detector, allowing high spatial resolution mapping of Bragg edge positions. Our first experiment demonstrates that energy resolution as high as $\Delta E/E<1\%$ or $\Delta E<4$ mÅ can be achieved. Variation of the residual strain in a well-characterized VAMAS round robin shrink-fitted Al ring-and-plug sample was measured with ~200 microstrain resolution through an accurate mapping of the first (1 1 1) Bragg edge. The measured stress profile agrees well with the expected values for that particular sample. More developments on the detector processing electronics are required in order to reduce the data acquisition times by enabling simultaneous measurements of spectra in a wide energy range covering multiple Bragg edges.

1. Introduction

The observation of Bragg edges in neutron transmission spectra due to coherent scattering in crystalline materials enables non-destructive studies of phase, texture, and residual stress [1–4]. A pulsed neutron source combined with neutron counting detectors can utilize time-of-flight techniques to measure either neutron diffraction or transmission energy spectrum. Previous experiments conducted at the ENGIN-X neutron beamline [5] at the ISIS pulsed spallation source demonstrated the capabilities of this technique with diffraction detectors (resolving $\sim1\ mm^3$ in the sample) as well as with a 10×10 Li glass pixel transmission detector (spatial resolution $2\times2\ mm^2$) [1–5]. The strength of the diffraction technique is its ability to obtain information within a small ($\sim1\times1\times1\ mm^3$) gauge volume. The transmission mode enables much higher spatial resolution, as will be shown in this paper (in principle down to a 55 μm level), but only allows a two-dimensional projection of measured physical parameters. However, the high spatial resolution of transmission spectroscopy is a very attractive feature for some engineering applications, especially if two-dimensional projection is sufficient. In this paper, we demonstrate the capabilities of time of flight neutron transmission spectroscopy utilizing a high resolution neutron counting detector.

2. Experimental setup

A proof of principle experiment was conducted on the pulsed-source neutron beamline ENGIN-X at the ISIS facility in the UK [5], Fig. 1. A well-characterized VAMAS round robin shrink fitted Al ring-and-plug sample was installed in front of the neutron counting position sensitive detector, Fig. 2, combining neutron sensitive MCPs, developed by Nova Scientific (Sturbridge, MA) and the Timepix readout [6] developed within the Medipix collaboration. The high spatial (~55 μm, [7,8]) and temporal resolution (~1 μs, [9]) of our detection system in principle enables the measurement of neutron transmission spectra within each $55\times55\ \mu m^2$ area. However, a limited neutron flux of the beam ($\sim10^6\ n/cm^2/s$ integrated over the full spectrum) as well as slow serial readout mode (~30 ms readout time) of the electronics required long acquisition times, in order to acquire sufficient statistics within such a small area.

The purpose of the experiment conducted in April 2008 was to verify the capabilities and to study the achievable resolution of

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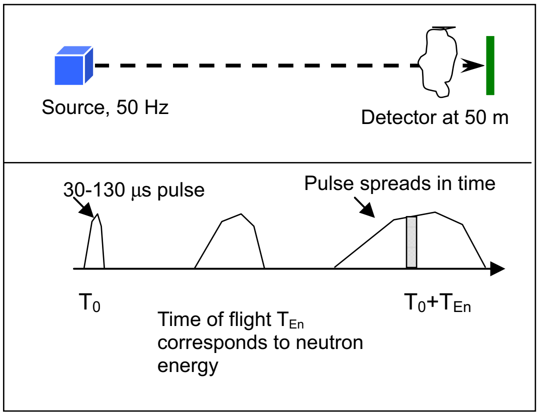

Fig. 1. Schematic diagram of time-of-flight transmission spectroscopy on a pulsed source. The moderator used in a particular beamline defines the width of the initial pulse, which, in turn, determines the width of measured Bragg edge. Detecting time-of-flight and *XY* position of each transmitted neutron enables spatially resolved transmission spectroscopy of the samples placed in the beam.

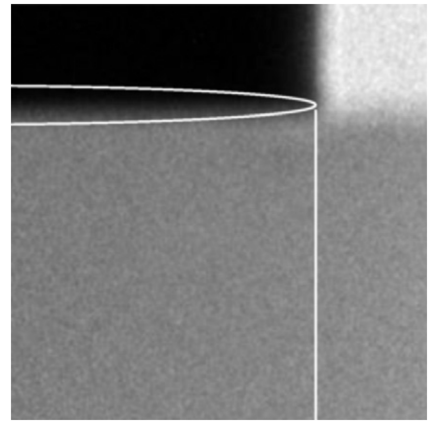

Fig. 3. The full spectrum neutron transmission radiographic image of an Al sample with an iron bar placed on its top. The white cylinder indicates the position of the shrink fitted Al plug. The image consists of 256 × 256 pixels, each 55 µm square. Integration time 100 s.

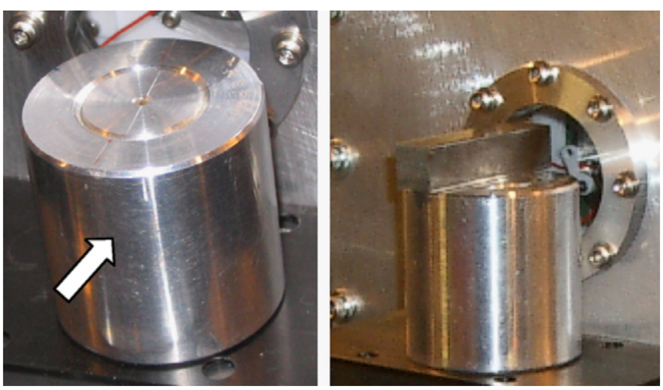

Fig. 2. VAMAS round robin shrink fitted Al ring-and-plug sample. The sample is 50 mm in diameter with a 25 mm press-fit Al cylinder. It is placed in front of a neutron counting detector with neutrons incident on the cylinder side. An iron bar is set on top of the cylinder for an accurate identification of the edge position in the transmission image. The arrow indicates the direction of an incident neutron beam.

new measurement system. A Medipix readout mode with a scanning 10 µs shutter was used in this experiment, which is not as efficient as Timepix in terms of duty cycle. All neutrons detected within a given 10 µs timing window were accumulated over a few thousand neutron pulses, thus providing the sample transmission image at a given energy. Our colleagues from the Czech Technical University in Prague [10] and our later experiments at the Spallation Neutron Source at Oak Ridge National Laboratory [11] made use of the Timepix timing resolution, substantially reducing the data acquisition time by simultaneous detection of multiple energies. However, much faster parallel readout electronics is needed in order to measure multiple Bragg edges simultaneously. The active area of our current version of the detector is limited to 14 × 14 mm^2 by the Timepix readout, while an active area of MCPs is > 25 mm in diameter. We plan to fabricate MCPs with 40 mm diameter active area, and then to tile or mosaic multiple Timepix readouts to increase the sensor area. We are also working on fully parallel readout electronics, which should allow simultaneous detection of multiple Bragg edges and allow even higher resolution of crystal lattice parameters through a Rietveld fit [1].

3. Data interpretation and results

3.1. Transmission radiography and reconstruction of Bragg edge spectra

A full spectrum transmission radiography image of the VAMAS sample is shown in Fig. 3. There is obviously no structure seen in the image as it has no features except for the gradual increase of transmission due to the variation of the sample thickness from its round shape. A neutron counting detector at a pulsed beam allows the introduction of a third dimension to that image, as neutron energy can be calculated from the time-of-flight for each detected neutron. Thus in principle our measurement system should allow transmission spectra to be built for a 55 × 55 µm^2 area, provided the neutron statistics are adequate. However, due to the inefficiency of our current data acquisition mode for each 55 µm pixel, we combined several pixels together while building the spectral data. The results shown below were obtained by building spectral data within a 165 µm wide area (3 pixel), extending from the top of the sample to the bottom of the imaged area (~8.8 mm). These integration regions are adequate as no significant variation of spectra is expected along the height of the cylinder, parallel to the cylinder axis. It should be noted that the spectra are built with 55 µm shifts, one third of an averaging width. A typical reconstructed (1 1 1) Bragg edge spectrum is shown in Fig. 4. The width of the measured Bragg edge is defined by the duration of an initial neutron pulse (~130 µs at a neutron wavelength of ~4.7 A for the cold methane moderator at an ENGIN-X). The next step is to reconstruct shifts of Bragg edge positions due to the residual strain in the sample.

3.2. Reconstruction of the strain profile

The typical residual strain values of the shrink-fit sample used in our measurements are known to be within the range of a few hundred microstrain. That corresponds to the change of the lattice parameter by a factor of ~10^{-4}–10^{-3} i.e. by 0.1–1 mA. Therefore the positions of Bragg edges need to be measured with the same 0.1–1 mA accuracy. Fig. 4 shows that the width of a measured Bragg edge is much larger due to the width of the source pulse.