Transmission Bragg edge spectroscopy measurements at ORNL Spallation Neutron Source

A S Tremsin1, J B McPhate1, J V Vallerga1, O H W Siegmund1, W B Feller2, H Z Bilheux3, J J Molaison3, C A Tulk3, L Crow3, R G Cooper3, D Penumadu4

1Space Sciences Laboratory, University of California at Berkeley, Berkeley, CA 94720, USA
2Nova Scientific, Inc, Sturbridge, MA 01566, USA
3Spallation Neutron Source, Oak Ridge National Laboratory, Oak Ridge, TN 37831 USA
4Professor and JIAM Chair of Excellence, Department of Civil and Environmental Engineering, University of Tennessee, Knoxville, TN 37996, USA

E-mail: ast@ssl.berkeley.edu

Abstract. Results of neutron transmission Bragg edge spectroscopic experiments performed at the SNAP beamline of the Spallation Neutron Source are presented. A high resolution neutron counting detector with a neutron sensitive microchannel plate and Timepix ASIC readout is capable of energy resolved two dimensional mapping of neutron transmission with spatial accuracy of ~55 μm, limited by the readout pixel size, and energy resolution limited by the duration of the initial neutron pulse. A two dimensional map of the Fe 110 Bragg edge position was obtained for a bent steel screw sample. Although the neutron pulse duration corresponded to ~30 mÅ energy resolution for 15.3 m flight path, the accuracy of the Bragg edge position in our measurements was improved by analytical fitting to a few mÅ level. A two dimensional strain map was calculated from measured Bragg edge values with an accuracy of ~few hundreds μstrain for 300s of data acquisition time.

1. Introduction

Modern pulsed neutron sources that provide sub-100 μs bright neutron pulses uniquely enable non-destructive studies of texture, phase and residual strain within metal samples with high resolution [1-5]. Presently there are a number of instruments used for strain mapping from neutron diffraction with time of flight (TOF) measurement for each detected neutron [2-5]. The measured time of flight can be converted into neutron energy with the resolution determined by the width of the initial pulse, length of the flight path and the timing resolution of the neutron detection system. The conventional TOF neutron diffraction techniques measure the scattered neutrons. This method allows the measurement of the lattice parameter within the volume corresponding to the intersection of incident (constrained by slits) and diffracted beams (defined by detector collimators). Scanning this volume through the sample allows non-destructive measurement of the wavelengths of Bragg edges and as a result spatially correlated lattice parameter variations within the sample along the incident beam direction.

An alternative approach is to measure the spectrum of neutrons transmitted through the sample [1-3,5]. In this case, the lattice parameter is averaged over the neutron path length within the sample. However, in this mode the entire 2-dimensional map of averaged lattice parameter variation can be
reconstructed if transmission spectra are measured in each pixel of the detection system. Thus a high resolution position sensitive detector in the transmission mode allows accurate two-dimensional mapping of combined effects from all the scattering and reaction processes occurring in the sample. The spatial and temporal resolutions of the detection system are the crucial parameters defining the accuracy of neutron transmission spectroscopic measurements.

In this paper we present the results of proof of principle transmission Bragg edge spectroscopic experiments performed at the SNAP beamline of the Spallation Neutron Source of Oak Ridge National Laboratory. A two dimensional map of the Fe 110 Bragg edge positions was measured for a pre-stressed steel screw sample. The map of residual strain was then calculated from Bragg edge positions with accuracy of ~few hundred µstrain.

**Figure 1.** Photograph of bent steel screw used in the measurements. Dashed square shows 14x14 mm² area imaged by the detector.

**Figure 2.** Neutron transmission image of the screw. Full beam spectrum is used. 100 sec acquisition. Squares indicate areas where strain histograms of Figure 6 are measured.

### 2. Experimental setup

A high resolution neutron counting detector with a neutron sensitive microchannel plate and a Timepix ASIC readout [6] was used in our measurements. The active area of the detector was limited by the TimePix readout, which is 14x14 mm², with a 256x256 matrix of 55 µm square pixels, while the neutron sensitive microchannel plate stack has ~25 mm active area in diameter. The high detection efficiency (~40% for cold and ~20% for thermal neutrons [7]), spatial (55 µm [8,9]) and temporal (~µs [10,11]) resolution proven by our recent experiments enabled high resolution measurement of two dimensional neutron transmission spectrum in time of flight mode. The distance between the source of neutrons and the detector was only 15.3 m, compared to a 50 m flight path at ENGIN-X facility [3,11]. The neutron transmission spectra in our measurements in principle could have been acquired in each 55 µm pixel of our detector, providing there are enough statistics. The energy resolution of measured raw data was limited by the duration of the initial neutron pulse (~30 to 130 µs, depending upon the neutron wavelength), corresponding to ~7-33 mÅ. However, the position of Bragg edge could be determined with much better accuracy (down to ~1 mÅ or ΔE/E~0.025% at 4.08 Å) after the analytical fit to the measured edge data.

A pre-bent steel screw shown in Figure 1 was installed ~14 mm from the active area of our detector. The SNS neutron source operated at 600 µA proton beam current at 60 Hz frequency. The beamline has a decoupled poisoned supercritical hydrogen moderator. The full spectrum transmission image of the screw acquired over 100s is shown in Figure 2. The open beam area was used for flux...
normalization in our measurements. Our current serial detector readout electronics does not allow simultaneous measurements of neutrons across a wide range of energies, and only a narrow range around a particular Bragg edge was measurable in one acquisition run. The 110 Bragg edge data shown below was acquired in 300 seconds. Position (with \( \sim 55 \, \mu m \) accuracy) and timing (\( \sim 1 \, \mu s \)) of each detected neutron was recorded into raw data files, which then could be rebinned into spectral data with a given spatial and spectral resolution.

![Figure 3](image3.png)

**Figure 3.** Experimental 110 Bragg edge of steel screw measured in the area 2 of Figure 2. The width of the edge is defined by the width of the initial neutron pulse. Analytical fit improves the accuracy of \( \lambda_0 \) position to \( \sim \)few mÅ level. 300 sec data acquisition.

![Figure 4](image4.png)

**Figure 4.** Strain map image of the steel screw. The strain is calculated from 110 Bragg edge positions \( \lambda_0 \) mapped across the sample. Spectral data is calculated for a 20x20 pixel area around each pixel. The colour bar indicates strain values in \( \mu \)strain.

3. Results and discussion

Due to limited neutron statistics (only 37 counts per 55x55 \( \mu m^2 \) pixel within 170 mÅ range around 110 edge) our measurement did not provide spectral information in each pixel. Therefore we have rebinned data into spectra calculated within 20x20 pixel area with 10 \( \mu s \) timing (2.59 mÅ energy) resolution. Thus each pixel in our 256x256 2-dimensional map of Bragg edge spectrum contains data from a 20x20 pixel area. Figure 3 shows a typical transmission curve calculated for the box N2 of Figure 2. The width of the wedge is determined by the width of the initial beam pulse and by the flight path of 15.3 m. Despite relatively poor statistics and large edge width, the position of Bragg edge can be calculated quite accurately with the help of analytical function fitting [1,2]. In each pixel, the wavelength of Bragg edge centre \( \lambda_0 \), was calculated from the spectral data integrated over the 20x20 pixel area, and the resulting map of \( \lambda_0 \) values was converted into the residual strain map. Figure 4 shows the map of calculated residual strain in the sample. The strain in that image varies within \( \pm 4 \, \mu \)strain. The diagonal cut through the bent screw area in that image is shown in Figure 5. The noise on that curve indicates that the resolution of a few hundred \( \mu \)strain in each pixel of strain map was achieved in our measurement. The histogram of strain values in boxes shown by dotted lines in Figure 2 are shown in Figure 6, together with the histogram for the entire strain map image. It is clearly seen that compression in box 1 and stretching in box 2 are on the scale of \( \sim 1.5 \, \mu \)strain, while areas 3 and 4 do not have much of the strain, as expected in that sample.
4. Conclusion
The transmission Bragg edge spectroscopy measurements with MCP-Timepix detector installed at a pulsed neutron beamline indicate that 2-dimensional maps of strain can be measured with accuracy of ~few hundred μstrain and spatial resolution as good as the pixel size of the detector, providing enough statistics is acquired. Five minute acquisition experiments at SNAP beamline and SNS operating at 50% power are clearly not enough to resolve strain within 55 μm pixels. However, increased neutron flux, dedicated beamline optimized for this type of measurements and optimized detector electronics should enable very high resolution strain mapping in the near future.

Figure 5. Cross section through the image of Figure 4 showing the strain variation across the bent area of the screw.

Figure 6. Histograms of strain values from Figure 4 measured in the areas shown in by boxes in Figure 2 as well as that of the entire stain map image.

Acknowledgments
We would like to thank Dr. A. Stewer from ESS for his very helpful discussions and Antonio Des Santos of ORNL for the help with the measurements. This work was supported in part by the U.S. Department of Energy under Grant No. DE-FG02-07ER86322, DE-FG02-08ER86353 and National Science Foundation Grant No. DMR-0753599.

References
[1] Steuwer A, Santisteban J R, Withers P J, Edwards L, Fitzpatrick M E 2003 J. Appl. Crystallography 36, 1159
[2] Steuwer A, Withers P J, Santisteban J R Edwards L 2005 J. Appl. Phys. 97, 074903
[3] Santisteban J R, Daymond M R, James J A, Edwards L 2006 J. Appl. Cryst. 39, 812
[4] Wang X -L et al. 2006 Physica B 385–386,673
[5] Kiyanagi Y et al. 2009 Nucl. Instr. Meth. A 600, 167
[6] Llopart X, Ballabriga R, Campbell M, et al. 2008 Nucl. Instr. Meth. A 585, 106
[7] Tremsin A S, McPhate J B, Vallerga J V, Siegmund O H W, Hull J S, Feller W B, Lehmann E 2009 Nucl. Instr. Meth. A, in press, doi:10.1016/j.nima.2009.01.041
[8] Siegmund O H W, Vallerga J V, Tremsin A S, McPhate J B, Feller W B 2007 Nucl. Instr. Meth. A 576, 178
[9] Tremsin A S, Vallerga J V, McPhate J B, Siegmund O H W, Feller W B, Crow L, Cooper R G 2008 Nucl. Instr. Meth. A 592, 374
[10] Tremsin A S, Feller W B, Siegmund O H W 2008 IEEE Trans. Nucl. Sci. 55, 1664
[11] Tremsin A S, McPhate J B, Kockelmann W A, Vallerga J V, Siegmund O H W, Feller W B 2008 IEEE Nucl. Sci. Symposium N42-2, subm. IEEE Trans. Nucl. Sci