The very small-angle neutron scattering (VSANS) instrument

J. G. Barker,[1] J. J. Moyer,[1] D. Adler,[1] L. Wroten,[1] C. Wrenn,[1] D. Pierce,[1] G. Jensen,[1,2] C. Gagnon,[1,3] S. R. Kline,[1] J. Chabot,[1] T. Thai,[1] A. Malone,[1] B. Dickerson,[1] N. Maliszewskyj,[1] C. Parikh,[1,2] N. Shmunis,[1,2] S. Pheiffer,[1] D. Ogg,[1] D. W. Ogg,[1] M. Rinehart,[1] S. Mullendore,[1] D. Johnson,[1] and C. J. Glinka[1,2]

The new 48-m VSANS (Very Small Angle Neutron Scattering) instrument is now a fully subscribed member of the CHRNS instrument suite. New design features of the instrument expand the capabilities of our two 30 m SANS instruments. The minimum momentum transfer q is reduced by a factor of up to five by utilizing a higher resolution (1 mm) detector. Meanwhile, new optics increase beam intensity by a factor of 100-fold to 4,000-fold over standard pinhole collimation when using these collimation conditions. Expanding from one to nine 2D detectors placed on three carriages instead of one greatly expands the range of scattering angles from a single measurement. Typical experiments now measure a larger range of scattering angles. The ratio of the maximum to minimum angles is now 130, a factor of seven larger than that obtained on 30 m SANS instruments.

VSANS also has the ability to adjust the wavelength resolution to better match the needs of the experiment. For typical SANS instruments a neutron velocity selector (NVS) is used to choose the range of wavelengths. For VSANS the full-width-half-maximum (fwhm) of the NVS spectrum is 12.5 % of the mean wavelength. As an option to provide higher resolution, two reflections from highly oriented pyrolytic graphite (HOPG) crystals reduce the fwhm to 1 %. Alternatively, the wavelength resolution can be relaxed to increase the beam intensity through the use of filters to limit the white beam produced by the cold source to a range of 4 Å to 8 Å. A polycrystalline Be filter removes wavelengths shorter than 4 Å, and an "X" shaped supermirror insert removes wavelengths longer than 8 Å. This filtered white beam has four times the intensity produced using the NVS of the same mean wavelength of 5.3 Å, as shown in Figure 1.

The front and middle carriages use four 2D detectors composed of arrays of 8 mm diameter linear position-sensitive tube detectors. Each tube can measure count rates similar to that allowed on the entire 2D detector on the 30 m SANS. Thus, VSANS count rates now can be 100 times higher. The positions of the detector arrays are adjustable such that a center square opening allows the smaller scattering angles to pass downstream to the next carriage. Typically, the front carriage is placed at a distance one fifth of the middle carriage, thus increasing the maximum scattering angle by a factor of five. The four panels generally cover 100 cm x 100 cm area, which is 50 % wider than the 65 cm x 65 cm detector used on the 30 m SANS instruments. This expanded angular range allows measurements of samples that are changing with time without having to measure with more than one set of detector distances.

By simultaneously increasing the sample-to-detector distance, sample aperture and beam stop sizes, the beam intensity can be increased by up to a factor of six over current practice on the 30 m SANS instruments. To take advantage of this possibility, a new version of our demountable liquid cells has been implemented with an increased sample size but with the same outer profile as the cells currently used in our existing temperature control blocks. Then, increasing the beam stop size from 50 mm to 100 mm, the sample aperture size from 13 mm to 22 mm diameter, and doubling the detector distance, the beam current for the same q_{min} can be tripled. The larger beam stop size also doubles the contribution of gravity to the q-resolution, but the insertion of prisms eliminates the effect of gravity.

The rear carriage uses a ^{6}LiF-ZnS(Ag) scintillation screen to convert the neutron to a burst of light that is then intensified and its position detected via three charge coupled detectors (CCD). The position of the neutron is detected to an accuracy of 0.9 mm fwhm using the rear detector. This rear carriage has three small beam stops: 12 mm diameter for converging beam collimation, and 6 mm and 12 mm wide by 300 mm tall rectangular beam stops used with narrow slit collimation.

The converging beam refractive lens optics used on VSANS achieves a minimum q of 2.6 x10^{-4} Å^{-1}, a factor of three smaller than can be realized using lenses on the 30 m SANS instruments, thus enabling measuring objects three times larger. The new optics has nearly equal pathlengths of 24 m before and after the sample using a 6.7 Å wavelength beam that is blocked by a 12 mm diameter beam stop. Using standard pinhole collimation, the source and sample aperture diameters would be 6 mm and 3 mm, respectively. By using refractive lenses to focus the beam, the sample aperture is enlarged to 10.4 mm, increasing the beam intensity by a factor of $(10.4/3.0)^2 = 12$. By inserting a series of intermediate masks, twelve separate beams are defined. Inserting refractive prisms after the lenses corrects the parabolic trajectory of the neutron beam,

1 NIST Center for Neutron Research, National Institute of Standards and Technology, Gaithersburg, MD 20899
2 University of Delaware, Newark, DE 19716
3 University of Maryland, College Park, MD 20742

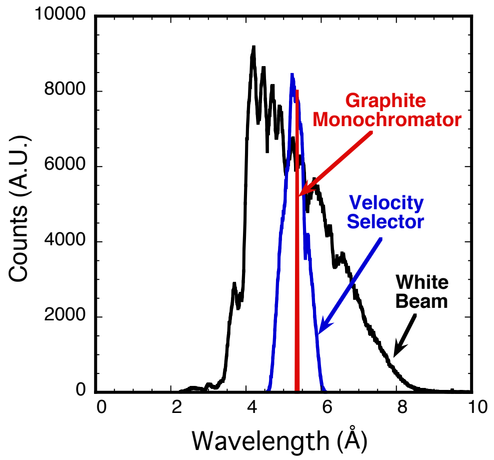

FIGURE 1: Time of flight (TOF) spectrum for the three wavelength resolution options. The blue curve is obtained using a NVS with rotation speed chosen to produce a mean wavelength of 5.3 Å. The black curve is the spectrum from the filtered white beam. The red curve is a calculated spectrum for the HOPG monochromator. The beam intensity is proportional to the area under the curve.

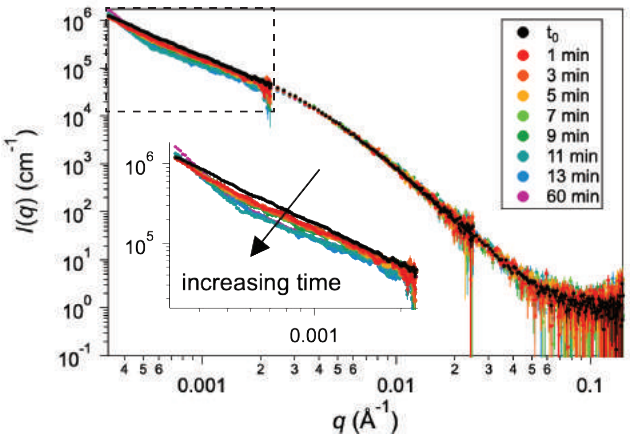

FIGURE 2: Transient structural evolution measured for a sheared sample of Vulcan XC-72 carbon black suspended in propylene carbonate.

allowing all 12 beams to converge to the same spot on the detector. To minimize attenuation of the beam from phonon scattering, the prisms are cooled to 155 K. The observed gain in beam intensity using 12 beams versus pinhole collimation is 120 after correcting for lens and prism attenuation. A new larger demountable liquid cell and temperature-controlled sample blocks are now being designed to take full advantage of this option.

Narrow slit collimation is obtained using two motorized XY slits where the width and height of a rectangular aperture is adjustable over the full width and height of the incoming neutron guide which has a width of 60 mm and a height of 150 mm. These two slits can be adjusted to produce a narrow beam at the rear detector with a width smaller than the smallest 6 mm wide beam stop. Using 6 Å wavelength, a source aperture that is 2.5 mm wide by 150 mm tall and sample aperture that is 1.25 mm wide by 75 mm tall, the measured beam intensity is 31,000 s^{-1}. An additional factor of four increase in beam intensity is achieved by using the filtered white beam wavelength option. The measured parasitic background outside the beam stop was 91 s^{-1}. Test scattering measurements have been made to q_{min} = 1.5x10^{-4} $Å^{-1}$. Commonly available quartz capillaries of 2 mm diameter can be used for liquid cell holders.

Polarized beam experiments are also possible at VSANS. In fact, several improvements have been made to the beam polarization compared to the NG7 30 m SANS instrument providing flipping ratios of up to 300. Mathematical simulations of beam depolarization caused by instrument magnetization profile led to redesigning of the permanent magnet and soft steel structures surrounding the double V shaped polarizing super mirror insert before the source aperture and the RF coil located upstream of the sample. The redesign minimized nonuniformity of magnetic field lines in the beam area which was causing beam depolarization in the simulations. The beam polarization at the sample position is now measured to be 99.3 % with a measured flipping efficiency of the RF flipper approaching 100 % within our measurement capability of +/- 0.0003. The large 2 m long sample area allows room for a ^{3}He spin filter downstream of the sample. Several experiments have been performed at VSANS, measuring all four polarization scattering cross-sections.

As an example of the capabilities of VSANS, the structural evolution of a sheared carbon black sample was measured using four converging beams and the high-resolution detector on VSANS [1]. The structure of carbon black suspensions play a pivotal role in the design and processing of electrode formulations used in electrochemical energy storage applications. Unfortunately, this structural evolution occurs at length scales too large for typical SANS experiments and time scales too short for USANS experiments. However, VSANS, with its lower attainable *q*-range and high neutron flux, provides a unique measurement of these structural changes under shear. VSANS data (Figure 2) shows the transient densification of carbon black agglomerates that occurs at low shear rates, as evidenced by a decrease in intensity at low *q*. This densification is related to a time-dependent decrease in both the viscosity and electrical conductivity of the suspension. Therefore, the combination of this time-dependent VSANS data and steady-state USANS data provides useful information for understanding the microstructural origin of the transient properties of carbon black suspensions.

Nearly all of the experiments in the last year utilized at least one of the new capabilities provided by the new instrument. Further developments in the next year will concentrate on constructing and modifying sample environment equipment to permit routine user experiments that take full advantage of the higher beam intensity collimation options available on VSANS.

Reference

[1] J. B. Hipp, J. J. Richards, N. J. Wagner, *in preparation*, (2019).