

## **EMSP Annual Report, June, 2004**

### **Investigating Ultrasonic Diffraction Grating Spectroscopy and Reflection Techniques for Characterizing Slurry Properties**

Project Number: 81889

Lead Principal Investigator:

Margaret S. Greenwood  
Pacific Northwest National Laboratory  
Richland, WA 99352  
509-375-6801

[margaret.greenwood@pnl.gov](mailto:margaret.greenwood@pnl.gov)

Co-Investigator:

Leonard J. Bond  
Pacific Northwest National Laboratory  
Richland, WA 99352  
509-375-4486

[leonard.bond@pnl.gov](mailto:leonard.bond@pnl.gov)

Co-Investigator:

Lloyd Burgess  
University of Washington  
Seattle, WA 98195  
206-543-0579

[lloyd@cpac.washington.edu](mailto:lloyd@cpac.washington.edu)

Co-Investigator:

Anatol Brodsky  
University of Washington  
Seattle, WA 98195  
206-543-1676

[brodsky@cpac.washington.edu](mailto:brodsky@cpac.washington.edu)

Graduate Student:

Mazen Lee Hamad  
University of Washington  
Seattle, WA 98195

[mhamad@u.washington.edu](mailto:mhamad@u.washington.edu)

### **Research Objectives**

The particle size of a slurry and the viscosity of a liquid or slurry are both difficult to measure on-line and in real time. The objectives of this research are to develop the following methods for such measurements: 1) ultrasonic diffraction grating spectroscopy (UDGS) to measure the particle size of a slurry, 2) UDGS to measure the velocity of sound in a slurry using reflection from a grating as opposed to ultrasound traveling through a possibly dense slurry, and 3) shear wave reflection techniques to measure the viscosity of a slurry.

## Research Progress and Implications

This report summarizes research in the third year of a three-year project. Our experiments with UDGS show the effect of particle size and this is a *major breakthrough* in our research this year. This is the **first observation** of such an effect using ultrasonics. In addition, two methods have been developed to measure the velocity of sound. Measurements with a fused quartz wedge show that the viscosity of 20% sugar water is 2.02 mPa-s, compared to a handbook value of 1.94 mPa-s at 20° C. For 60% sugar water, the viscosity is 39.4 mPa-s, compared to a handbook value of 42.7 mPa-s at 20° C.

A schematic diagram of the 240-micron stainless steel (SS) grating and the data acquisition system is shown in Fig. 1. Ultrasound from the send transducer travels through the SS and strikes the grating-slurry interface, where it is reflected to a receive transducer. The peak in this signal at the so-called critical frequency is used to determine the velocity of sound. The peak height for a slurry is reduced due to scattering from the particles, which is dependent upon particle size. Fig. 2 shows the data obtained for 10% sugar water. The critical frequency of 7.30 MHz is in agreement with the known velocity of sound. Experiments were also carried out with salt water solutions. The results show that, for the same velocity of sound, the peak height above background is the same for sugar and salt water solution. As the velocity of sound increases, the peak height above background increases. Thus, the velocity of sound can be obtained from 1) a critical frequency measurement and 2) the peak height above background.

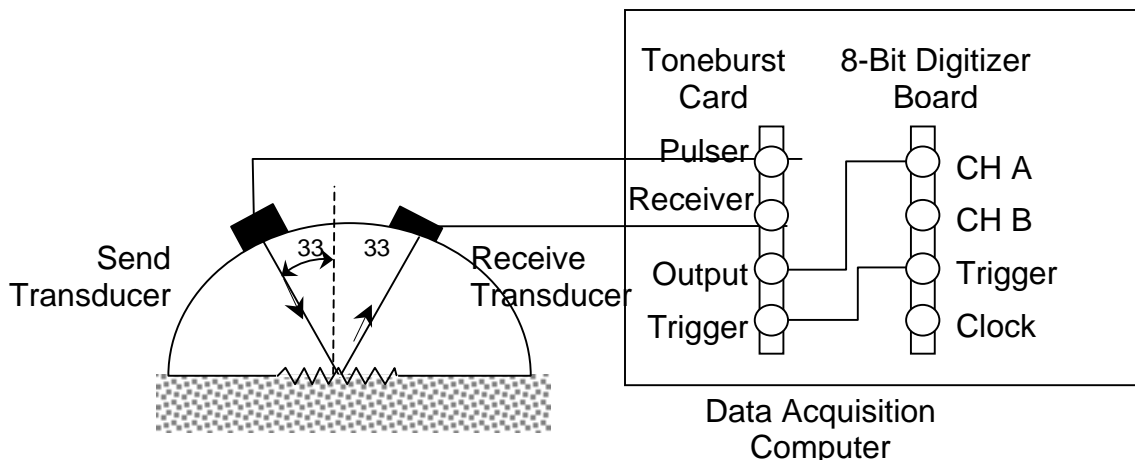


Fig. 1

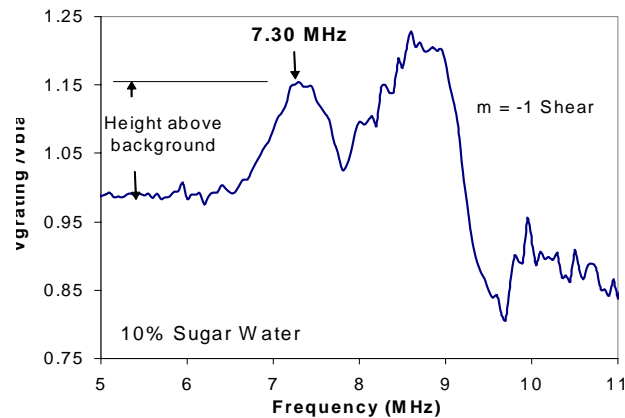


Fig. 2

The initial measurements with slurries were carried out using polystyrene spheres of various sizes in water. To contain the slurry, a cup (radius = 1.9 cm) was placed in front of the stainless steel grating unit, as shown in Fig. 3. A small magnetic stirrer was placed beneath the cup to provide a uniform suspension. As shown in the photograph, the send and receive transducer are fastened to flats on the SS unit, similar to Fig. 1. The ultrasonic beam, traveling in stainless steel, strikes the grating and produces a transmitted  $m = 1$  beam, as well as the reflected beam. The angle of this beam in the liquid increases with decreasing frequency and the critical frequency  $F_{CR}$  occurs when the angle is  $90^\circ$ . At frequencies below  $F_{CR}$ , this  $m = 1$  wave does not exist and its energy is shared with other types of waves. Thus, a peak is observed in the signal of the receive transducer at the critical frequency. However, at the critical frequency, the  $m = 1$  wave becomes evanescent and penetrates into the slurry. Since scattering from the particles occurs, some energy is dissipated and the peak height of the signal in the receive transducer is expected to be reduced. Since the scattering is dependent upon particle size, this effect can be used for particle size measurements. These are the effects we wanted to demonstrate experimentally.

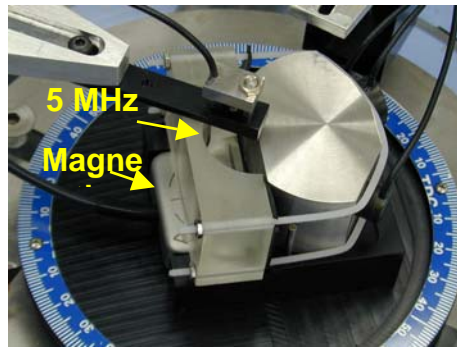


Fig. 3

The first set of data was obtained using polystyrene spheres in water with a size range from 180 to 250 microns at a concentration of 21% by weight. A scan-over-frequency for the receive transducer was obtained. During the data acquisition, the signals on the computer monitor immediately showed a decrease, compared to those with water. This, of course, was the expected result, but the immediate verification was very satisfying. The peak height for 10% concentration was larger than that for 21% and that for the 26% concentration was slightly smaller, as would be expected.

Experimental measurements were obtained for polystyrene sphere diameters of 16, 42, 74, 98, 136, and 275 microns at 10% by weight. The results are shown in Fig. 4. In order to compare the various slurries, the area under the peak, between the solid line and the dashed line, is integrated and listed on each graph. The peak integration for water is 1.38 and for 17% SW is 1.459, for which ultrasound is not absorbed. To calculate the ultrasound absorption by the spheres, data from slurries are compared with data from liquids and the results are shown in Fig. 5.

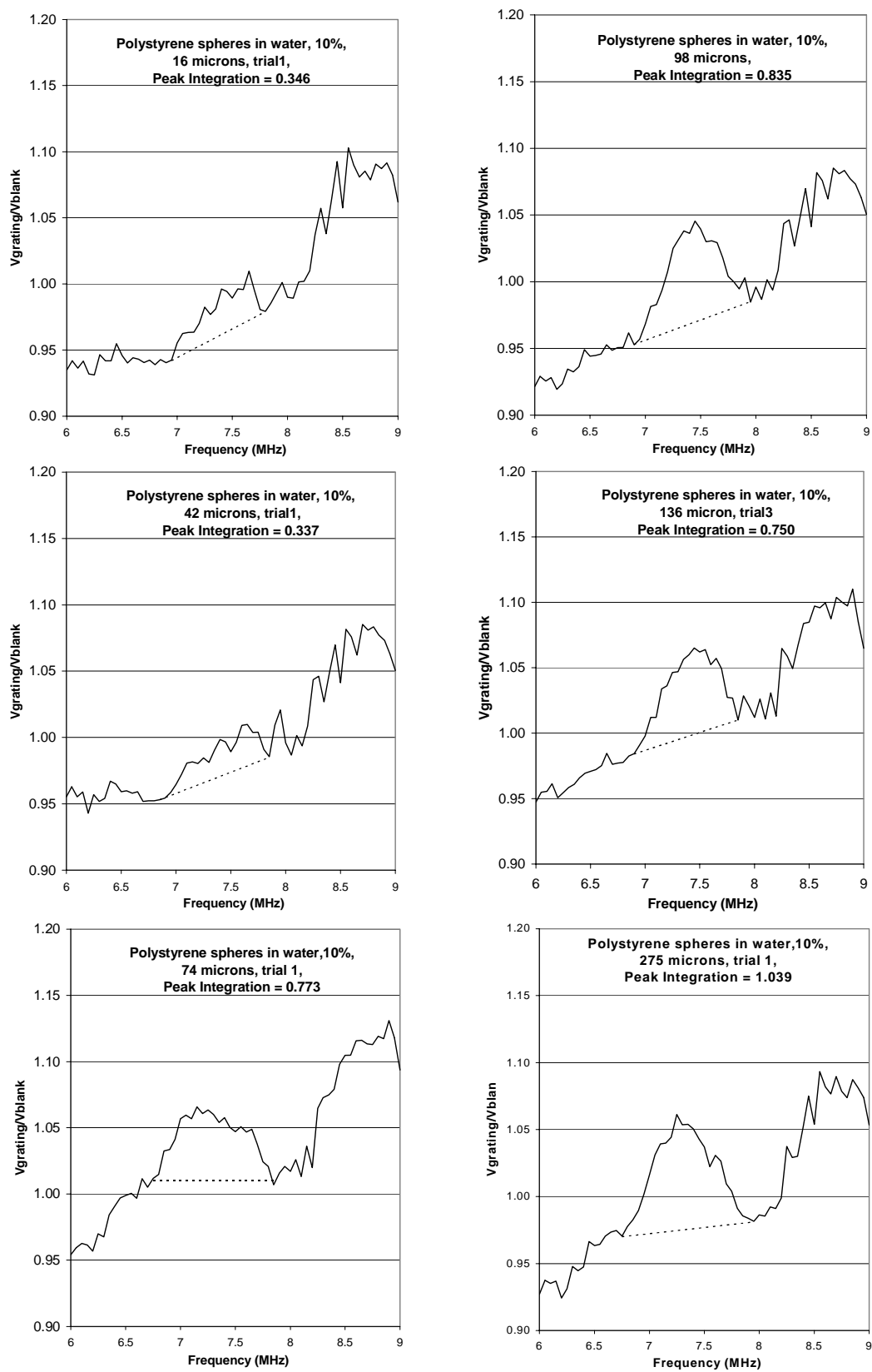


Fig. 4

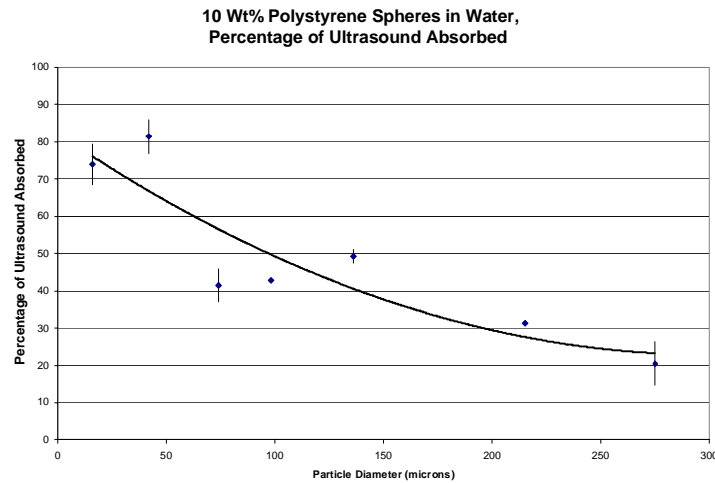


Fig. 5

During the last year, the main objective of the theoretical work at the University of Washington has been to develop a method to calculate the ultrasonic dissipation parameters from the experimental data with slurries. The theory of ultrasonic wave back scattering by a slurry at the boundary of a diffraction grating interface has been developed. Much information can be obtained near the critical frequency where the traveling waves are transformed into evanescent ones. Analysis of ultrasonic scattering in this region allows us to get rich information about both the surface and bulk properties of the sample. There are two mechanisms that occur when the evanescent wave interacts with particles in the slurry. One mechanism is coherence loss in the scattering process. The second one takes into account the wave absorption, which is dependent upon the kinematic viscosity. For small particles, wave absorption prevails, where  $R(v/\omega)^{0.5} < 1$ .  $R$  is the particle radius,  $v$  is the kinematic viscosity and  $\omega$  is the angular frequency. For large particles, where  $R(v/\omega)^{0.5} > 1$ , the dissipation mechanism is dominated by incoherent scattering effects. The dissipation function  $\varepsilon(R)$  can be found by analysis of the signal near the critical frequency. In particular, the peak height of the derivative of the signal at the critical frequency is inversely proportional to  $[\varepsilon(R)]^{0.5}$ . Comparison with the experimental data is in progress.

Measurements of the viscosity of sugar water solutions have been obtained using the fused quartz wedge, illustrated in Fig. 6. The 7.5 MHz transducer produces shear waves in the quartz, where the vibrations in the quartz are perpendicular to the plane of the paper (SH waves). Since liquids do not easily support shear waves, the reflection coefficient at the quartz-liquid interface is only slightly less than 1.0. As the viscosity of the liquid increases, the amount of ultrasound traveling into the liquid increases and therefore, the amount reflected decreases. The objective is to measure the reflection coefficient, which leads to a determination of the viscosity. To achieve the required sensitivity, the ultrasound must make many round trips, each losing more energy into the liquid. Fig. 7 shows the results obtained using a total of 16 echoes.

### Planned Activities

For the research in UDGS, an aluminum grating has been fabricated and 2.54 cm-long transducers have been mounted on this unit. The grating was anodized to prevent oxidation. The larger size transducer will increase the peak height of the signal, as well as reduce its width. The use of a lighter weight grating material means that a larger fraction of ultrasound will be transmitted into the liquid and hence a stronger signal in the receive transducer will be observed. This grating was fabricated in July and has just been received. A specialized transducer (0.32 cm diameter, 5 MHz, shown in Fig. 3) was fabricated to fit inside the cup, so that signals in the slurry can be observed and positioned at the desired angle by rotation of the chamber. The use of this transducer permits measurement of the velocity of sound and the attenuation of ultrasound through the slurry. Signals

from this specialized transducer can be compared with those for the receive transducer. The goal is increased sensitivity for particle size measurements.

Experiments are underway to use a smaller fused silica wedge for performing the viscosity measurements. Two wedges have been fabricated, with one having a frequency of 10 MHz and the other, a frequency of 14 MHz.

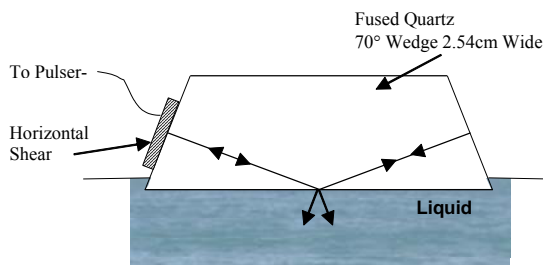


Fig. 6

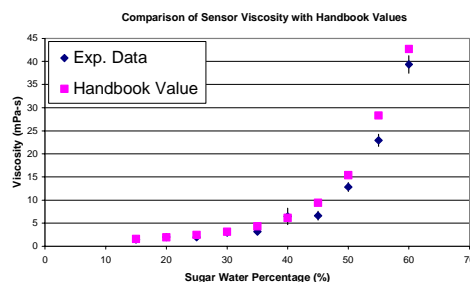


Fig. 7

### Information Access

1. M. S. Greenwood, A. Brodsky, L. Burgess, L. J. Bond, and M. Hamad, Ultrasonic diffraction grating spectroscopy and characterization of fluids and slurries, *Ultrasonics* 42, 531-536 (2004).
2. M. S. Greenwood, A. Brodsky, L. W. Burgess, and L. J. Bond, Using ultrasonic diffraction grating spectroscopy to characterize fluids and slurries, *Review of Progress in Quantitative Nondestructive Evaluation* 22B, 1637-1643 (2002).
3. M. S. Greenwood, A. Brodsky, L. Burgess, and L. J. Bond, Investigating Ultrasonic Diffraction Grating Spectroscopy and Reflection Techniques for Characterizing Slurry Properties, Invited presentation at the EMSP Symposium at the American Chemical Society Meeting in New York, September 10, 2003. The proceedings of this symposium will be published in a book.
4. M. S. Greenwood, J. D. Adamson, and L. J. Bond, Measurement of viscosity using multiple reflections of ultrasound within a quartz wedge, submitted for publication to the *Journal of the Acoustical Society of America*.
5. M. S. Greenwood, J. D. Adamson, and L. J. Bond, Measurement of the Viscosity-Density Product Using a Quartz Wedge, presented at the Review of Progress in Quantitative Nondestructive Evaluation, Golden, CO, July 25-30, 2004
6. M. S. Greenwood, L. J. Bond, A. Brodsky, L. Burgess, and M. Hamad, Observing Effects of Particle Size for a Slurry Using Ultrasonic Diffraction Grating Spectroscopy, presented at the Review of Progress in Quantitative Nondestructive Evaluation, Golden, CO, July 25-30, 2004

