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

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Principal Investigator

Margaret S. Greenwood
Pacific Northwest National Laboratory
P.O. Box 999, MSIN K5-26
Richland, WA 99352
509-375-6801 (phone)
509-375-6736 (fax)
Margaret.greenwood@pnl.gov

Co-Investigators

Leonard J. Bond
Pacific Northwest National Laboratory
P.O. Box 999, MSIN K5-26
Richland, WA 99352
509-375-4486 (phone)
509-372-4583 (fax)
Leonard.bond@pnl.gov

Anatol Brodsky
University of Washington
Seattle, WA 98195
206-543-1676 (phone)
brodsky@cpac.washington.edu

Lloyd Burgess
University of Washington
Seattle, WA 98195
206-543-0579 (phone)
lloyd@cpac.washington.edu

Graduate Student

Mazen Lee Hamad

Research Objectives

The objectives of the project are to investigate the use of 1) ultrasonic diffraction grating spectroscopy (UDGS) for measuring the particle size of a slurry and 2) shear wave reflection techniques to measure the viscosity of a slurry. For the first topic, the basic principle is to extend the methods that have been successful in optics, called grating light reflection spectroscopy (GLRS), to ultrasonics. At the University of Washington, researchers are using GLRS to measure nanometer-sized particles and particles up to two microns in size in slurries. The goal of the research with ultrasonics is to measure particles in the range of a few microns to about 100 microns, which is the range of particle sizes for slurries in the radioactive waste tanks at U.S. Department of Energy (DOE) sites. The collaborators at the University of Washington will provide the theoretical algorithm that extracts particle size information from the experimental measurements of the critical frequency and amplitude. For the second topic, the research will use multiple reflections of ultrasonic shear waves at the interface between a solid and a liquid or slurry. Such reflections are known to provide information about the viscosity, but the goal here is to develop a method to make on-line measurements. This will include using the self-calibrating method, developed by Greenwood, for which a patent application has been submitted. In both phases of the research, collaboration with partners at the University of Washington will provide the theoretical basis for analysis of the experimental data.

Research Progress and Implications

This report summarizes work after one year of a three-year project. In our experiments with UGDS, the diffraction grating consists of equally spaced triangular grooves machined into the face of a stainless steel half-cylinder, as shown in Figure 1. The incident ultrasonic wave travels through the stainless steel and strikes the back of the diffraction grating at an angle θ. This produces diffracted ultrasonic waves traveling in the liquid or slurry in contact with the grating, as well as a reflected wave observed by the receive transducer. In the experiment the ultrasound sweeps through a range of frequencies and at each frequency, the amplitude of the receive signal recorded. As the frequency of the ultrasonic wave decreases, the angle of the m = 1 diffracted wave in the liquid increases. At the critical frequency, this wave reaches 90°. In Figure 2, the location of the m = 1 wave in the liquid is shown as the frequency of the incident wave changes. At 90° it becomes an evanescent wave, which is an exponentially decaying wave

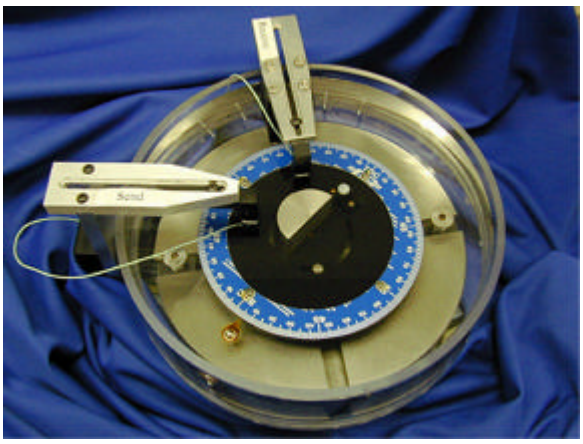

Figure 1. Experimental apparatus showing the immersion chamber, stainless steel grating, and send and receive transducers

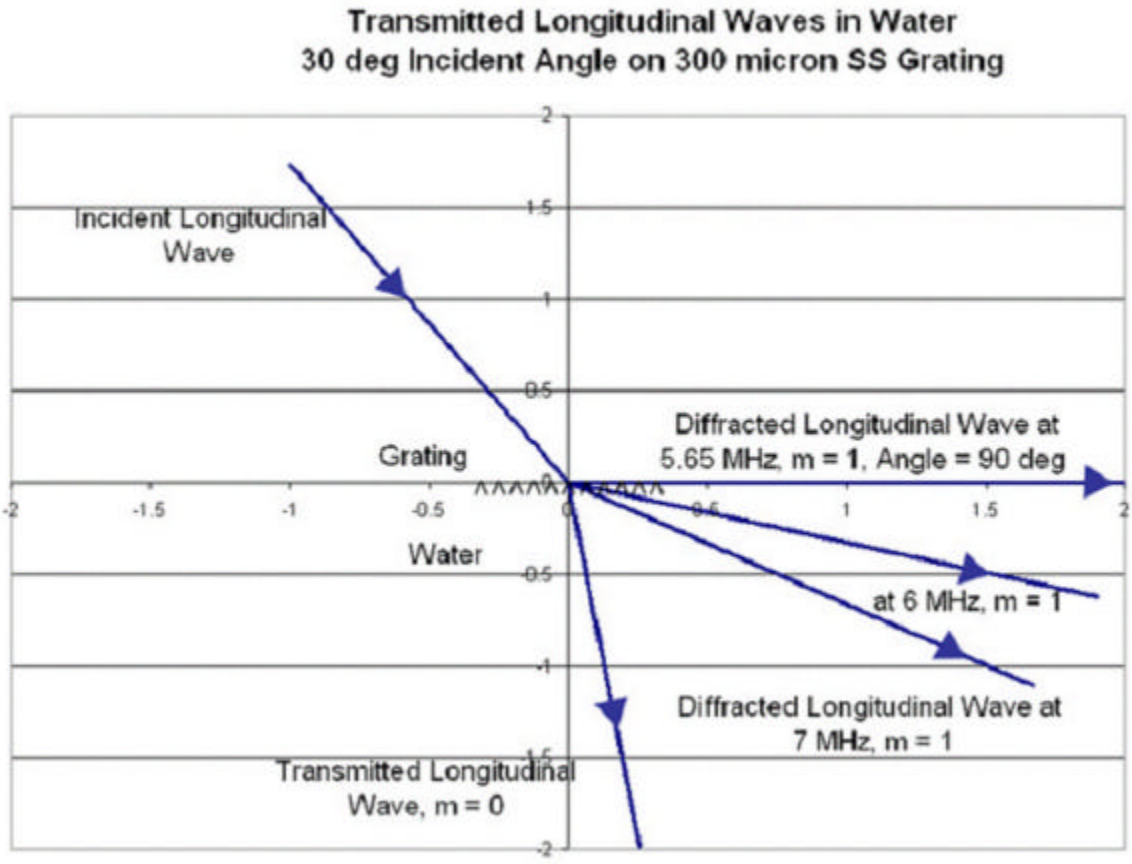

Figure 2. Movement of the m = 1 Wave in the Liquid to a Larger Angle as the Frequency Decreases

in the liquid or slurry. At a frequency below the critical frequency, the evanescent wave disappears and the energy is shared by all other waves. The objective is to observe the increase in signal at the critical frequency. For a slurry, the evanescent wave is attenuated due to Rayleigh scattering, and the amplitude of the receive signal at the critical frequency decreases. Because the attenuation is dependent upon particle size, this is the mechanism to determine particle size.

Because the critical frequency depends upon the grating spacing, the velocity of sound in the liquid, and the incident angle, the experiments were designed to vary all three parameters. The experiments were carried out using water and sugar water solutions, up to 30% by weight to vary the velocity of sound. The diffraction gratings consisted of 1) stainless steel (SS) with a 300-micron grating spacing, 2) SS with a 200-micron grating spacing, and 3) a Rexolite plastic grating with a 406-micron grating spacing.

For each SS grating, data were obtained at incident angles of 20°, 30°, 40°, and 50° for water, 10% sugar water (SW), 20% SW, and 30% SW. Figure 3 shows the data obtained for the

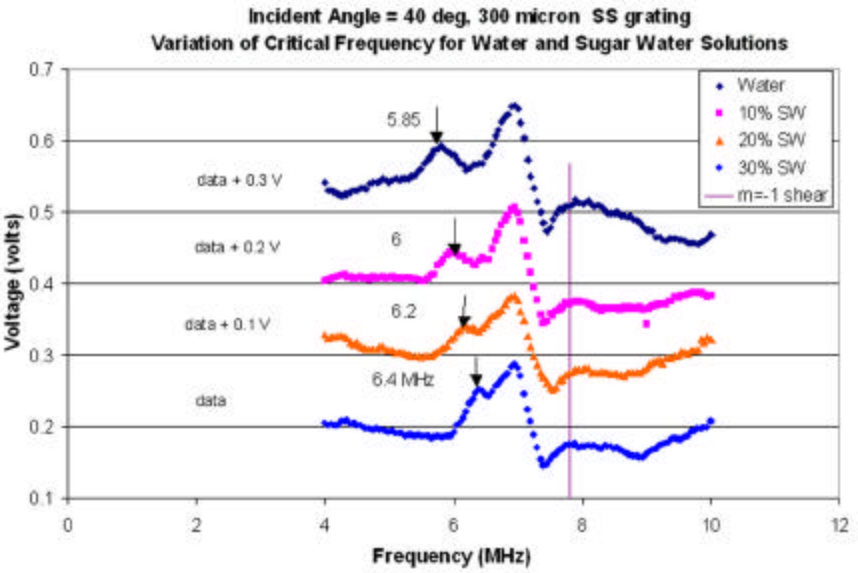

Figure 3. Data Obtained for 300-micron Stainless Steel Grating for an Incident Angle of 40°

300-micron SS grating at an incident angle of 40° and Figure 4, for the 200-micron SS grating at an incident angle of 30°. The experimental data are in *very good agreement* with the theoretical values of the critical frequency, shown by the arrows in Figures 3 and 4. The 32 sets of data for the SS gratings show similar agreement between the experimental and theoretical values of the critical frequency. This is a very important result, for the data show the ability to determine the critical frequency, which will be used in later experiments with slurries for the determination of particle size.

For the Rexolite grating, data were obtained for water at seven incident angles. At an incident angle of 25°, data were obtained for the following weight percentages of sugar water: 1.5%, 3%, 4.5%, 6%, 7.5%, 10%, 15%, 20%, 25%, and 30%. For water, the experimental data show good agreement with the predicted critical frequency. For the sugar water solutions, the critical frequencies cannot be distinguished from those for water. However, a peak in the graph of voltage versus frequency (Figure 5) shows that the height of the peak is very sensitive to the density of the SW solutions. Thus, an unexpected result is that the diffraction grating can act as a density sensor.

The immersion chamber shown in Figure 1 is mounted on a turntable. Within the last 3 months, the immersion chamber was motorized, and the data acquisition system was modified to

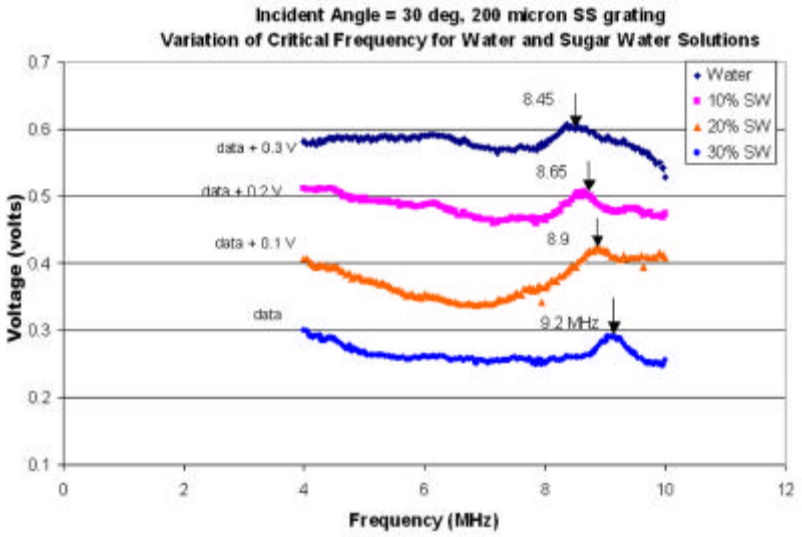

Figure 4. Data Obtained for 200-micron Stainless Steel Grating for an Incident Angle of 30°

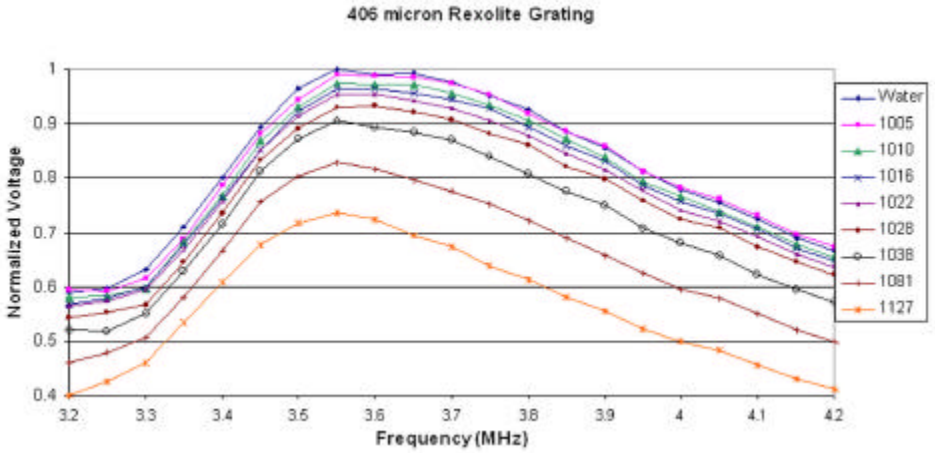

Figure 5. Data Obtained Using Rexolite Grating for Water and Eight Sugar Water Solutions. The legend gives the density of the sugar water solutions in kg/m^3

obtain the spectra of the diffracted waves in the liquid automatically. Thus, the send transducer is fixed at a given incident angle relative to the grating, while the receive transducer moves through a range of angles in order to obtain a spectrum of the diffracted rays in the liquid. This permits observation of the m = 1 diffracted wave in the liquid as it approaches 90° by choosing the appropriate frequency. The results show that the m = 1 peak increases in width as the angle increases. It is quite broad at 78°, and this is related to the width of the peak at the critical frequency, shown in Figures 3 and 4.

Ultrasonic diffraction grating spectroscopy has several advantages over laser technology now being used to monitor particle size in tank waste. First, ultrasound can penetrate farther into the slurry than visible light and will, therefore, give more information about the slurry composition. This is due to the fact that the evanescent wave penetrates into the liquid. Second, the ultrasonic sensor is noninvasive and is part of the pipeline wall. A sensor based upon UDGS would be very small—certainly an important advantage.

The following describes some of the theoretical results obtained by collaborators at the University of Washington. There are two important differences between optics and ultrasonics. The first is that light consists of transverse waves, while an ultrasonic wave can be either a longitudinal wave or a transverse (shear) wave. Second, when a glass optical grating is in contact with a liquid or slurry, the velocity of light in glass is *smaller* than the velocity in the liquid. However, for ultrasonics the velocity in the solid, such as in stainless steel, is *larger* than that in the liquid. These differences have some important consequences when considering the critical frequency at which the m = 1 diffracted wave in the liquid becomes 90° and the wave becomes evanescent. For example, for the ultrasonic data in Figures 3 and 4, small peaks occur at the critical frequency. The derivative of this curve gives positive slope, zero slope at the peak maximum, and then negative slope. A plot of the slope versus the frequency gives an S-shaped curve. In contrast, the optical case gives the reverse effect—a maximum for the derivative and an S-shaped curve for the amplitude versus frequency (or wavelength). This difference between optics and ultrasonics has been explained as a result of differences in the velocity of the two types of waves in solids and liquids, described above.

The shape of the curve at the critical frequency contains a great deal of information about the characteristics of the liquid or slurry. At the critical frequency, an evanescent wave is formed that decays exponentially *in the liquid*. That is, the measurement is a bulk measurement, not a measurement at a surface. At this point, the evanescent wave interacts with the liquid and with particles in the slurry. This interaction will depend upon velocity of sound, the dissipation parameters, and the coherence loss. The phrase *coherent* means that the propagation has the same direction as the original wave and also the same phase as the original wave. When the evanescent wave scatters from the particles in the slurry, loss of coherence results.

The critical frequencies shown in Figure 2 were calculated assuming that the velocity of sound was a real quantity. The velocity of sound can also have an imaginary part, usually considered small. The theoretical analysis shows that the value of the critical frequency depends upon both the real and imaginary parts of the velocity of sound. In future experimental studies, the effect of the imaginary part will be considered to see what role it plays.

In optics, the Rayleigh-Gans approximation was used to analyze the data and determine particle size. In anticipation of the experimental work soon to be carried out for slurries, an analogous formulation has been derived.

In future work, we will continue both experimental and theoretical studies of information provided by the data at the critical frequency. Information will be obtained from the data as a function of frequency and the derivative of this signal.

Planned Activities

Our planned activities include the following nine tasks:

1. Another stainless steel grating is being fabricated and is designed to enhance the signal at the critical frequency. Analysis of the data will be completed by July 15, 2002.

2. Parts for experiments to measure viscosity have been ordered, and experiments will be under way in July. Analysis of the data will be completed by September 1, 2002.

3. We will confer with collaborators about particle suspensions, i.e., particle size, for initial experiments with particle suspensions by September 1, 2002.

4. Apparatus for measurement with particle suspension will be designed and fabricated by October 2002.

5. We will carry out initial experiments with several particle sizes beginning in January 2003.

6. Data will be compared with theoretical calculations in May 2003.

7. We plan to obtain more experimental data over a large range of particle sizes and, if necessary, the theoretical algorithm for determination of particle size by August 2003.

8. In March 2004, we will refine measurement techniques for particle sizing so that it can be used for process control.

9. By September 2003, we will define the algorithm for particle sizing for use in process control.