Frequency Modulation Feedback Control for Near-Field Acoustic Characterization of Mesoscopic Fluid Films

Research Paper

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**ABSTRACT**

The enigmatic properties of the mesoscopic fluid confined between two solid objects or adsorbed on a substrate have puzzled scientists in the field of condensed matter. One of the current methods to describe mesoscopic fluids is Shear Force Acoustic Near-Field Microscopy (SANM). SANM consists of a fabricated probe attached to a quartz tuning fork that vibrates at resonance. With the substrate and the probe as two solid boundaries, the dynamics of the liquid are recorded from the changes in the tuning fork’s amplitude and the ultrasonic signals generated by the probe’s mechanical motion. The shrinking amplitude of the tuning fork can be attributed to multiple variables and this is the central deficiency in SANM. The addition of frequency modulation to match the driving frequency to the local resonance frequency is used to improve the instrumentation of SANM. Frequency modulation consists of three phases: Lock-In Amplifier, Proportional Integral Differentiator Microcontroller (PID), and Voltage Controlled Oscillator (VCO). The Lock-In Amplifier is used to detect the phase shift of the output signal of the tuning fork. That phase shift runs to the PID and calculates an error against a set point of ninety degrees. The calculated error causes the VCO to drive the tuning fork at the local resonance frequency. As this cycle continuously repeats, resonance of the tuning fork is guaranteed. Current results prove the claim of successful implementation of a Lock-In Amplifier, PID, and VCO. Future research of research of frequency modulation is to characterize the mesoscopic fluid films.

**I. INTRODUCTION**

The physical properties of mesoscopic fluids in confinement between two solids greatly differ from fluids in their bulk form. The effective shear viscosity increases dramatically as the confinement gets tighter, unusual molecular ordering is displayed in the vicinity of the substrate (which contrasts the highly disordered bulk water), and viscoelastic time responses are prolonged. Despite their relevance not only in friction phenomena (huge economical applications) but also in biological processes (including folding/unfolding of proteins), the strikingly dynamic behavior of these mesoscopic fluids is not easy to understand. It is still a puzzle why these fluids start to acquire solid-like properties.

Current techniques used to characterize mesoscopic films under shear stress are limited in their experimental findings due to the complexity of the mesoscopic fluid. The dynamics of the mesoscopic fluid are very complex that using a tapered probe (a currently used technique), about 10-50 nanometer apex diameter, to analyze the responses of the fluid can be affected by a number of factors. Since currently available techniques are unable to provide a convincing unifying explanation, it is highly desirable to develop new techniques, ideally of high resolution, to be able to characterize these mesoscopic fluids.

The expected outcomes of this project are to successfully incorporate frequency modulation into the Shear-Force Acoustic Near-Field Microscopy apparatus to increase its metrology. With better instrumentation, the next steps of characterizing mesoscopic fluids under confinement can properly be analyzed and evaluated. If the SANM apparatus were driven at a single driving resonance frequency, a decrease in the probe amplitude could be attributed to the damping effect of the mesoscopic fluid, the thermal energy dissipated from the mesoscopic fluid, or to a shift in the resonance frequency of the quartz tuning fork due to resistance from the fluid. With frequency modulation, instead, the probe will always be driven at the current resonance frequency and this technique will eliminate any unwanted effects on the probe's amplitude from the thermal activity of the mesoscopic fluid and any shifts in the resonance frequency of the quartz tuning fork due to added resistance. Frequency modulation allows that any decrease in probe amplitude can be related entirely due to the damping effects of the mesoscopic fluid. In addition, frequency modulation will keep track of the frequency shifts and these frequency shifts will indicate the new elastic force component of the mesoscopic fluid. With these unique developments in place, the properties of the mesoscopic fluid can be effectively recorded and these properties can be examined under better instrumentation.

**II. SHEAR FORCE ACOUSTIC NEAR-FIELD MICROSCOPY**

The Shear Force Acoustic Near-Field Microscopy (SANM) apparatus is the central component in controlling the approach/retraction of the tip into the fluid. The SANM apparatus consists of a tapered probe attached to one of the tuning fork’s tines. The piezoelectric tuning fork is driven by an electric voltage. This quartz tuning fork vibrates at an amplitude on the scale of nanometers. This device of the probe and the quartz tuning fork is built onto the platform of the MCL piezoelectric. The MCL piezoelectric is the equipment that controls the approach and retraction of the tip to the substrate. The MCL is a piezoelectric that expands or contracts depending on the magnitude of the voltage and the sign of the voltage supplied. Through a LabVIEW program and a FPGA card, the MCL is automatically and manually controlled to approach the substrate at certain specified velocities. The MCL operates on a voltage range from -10 to 10 volts and expands or contracts to a maximum length of 60 microns.



Figure 1 represents the SANM approach/retraction method. The probe vibrates laterally at the resonant frequency and penetrates the mesoscopic layer. In the process, the ultrasonic signals are recorded by the acoustic sensor.

The functioning of the SANM is computer interfaced and utilizes LabVIEW Programming and a Field Programmable Gate Array (FPGA) card. FPGA cards are top of the line technology to control sophisticated electronic instrumentation and in general, a FPGA card allows a faster response compared to that of a computer. The unique characteristic of FPGA cards is that they can be configured to the user's necessities and these cards are very adaptable in the field. In the SANM experimental setting, the FPGA card is customized to control the approach/retraction of the tip. This is very important because the FPGA card allows maintaining the integrity of the SANM tapered probe approach. The probe-sample distance ranges typically from a zero to twenty nanometers and the implementation of FPGA control allows the tip from crashing into the solid surface by utilizing its quick data acquisition and fast controls.

**III. EXPERIMENTAL METHOD**

Frequency modulation is the crucial component of this project that enhances the instrumentation to characterize mesoscopic films. The theory behind frequency modulation is to measure the frequency and phase of an input signal and output a signal that matches the frequency and phase of the input signal. In SANM, frequency modulation is used to measure the input signal's frequency and phase and output a signal with a ninety degree phase change and frequency that correspond to those of the input signal.

Frequency modulation is used to control the quartz tuning fork input and output voltages and therefore, keep the quartz tuning fork vibrating at a particular frequency. This particular frequency is the resonant frequency of the quartz tuning fork where it results in the maximum amplitude of the vibration of the tuning fork. At the resonant frequency of the quartz tuning fork, the phase change of the output signal is exactly ninety degrees out of phase from the input signal. Frequency modulation will exploit this unique condition of the quartz tuning fork and always maintain a ninety degree phase shift between the input and output signals of the tuning fork. With a constant ninety degree phase shift between the two signals, the tuning fork is guaranteed to vibrate at the resonant frequency at all times and the resonance frequency results in the vibration of the maximum amplitude of the tuning fork.

With the application of the FPGA card, LabVIEW, and a properly designed PLL, frequency modulation with the SANM apparatus can be implemented. The Phase Locked Loop (PLL) control system is the electronic circuit behind frequency modulation. Frequency modulation is made up of three critical stages which constitute the PLL: Lock-In Amplifier, PID Microcontroller, and the VCO. The Lock-In Amplifier is inserted to monitor the input signal and output a signal that is related to the phase of the input signal. The Lock-In Amplifier itself has its own integrated PLL. The second stage of frequency modulation consists of a PID and a VCO that constitutes of the second PLL. The second PLL integrates a Proportional Integral Differentiator (PID) microcontroller and a Voltage Control Oscillator. The PID compares the phase of the input signal provided by the Lock-In Amplifier against a phase set point of ninety degrees. Then, the PID outputs an error signal through three different forms: a proportional circuit, integrator circuit, and a differentiator circuit. In the frequency modulation approach, the proportional and integrator circuit are used to calculate the phase error. This error signal is the input for the Voltage Controlled Oscillator that outputs a signal whose frequency and phase directly relates to the error signal. By maintaining a 90 degree phase shift between these two input and output tuning fork signals, the tuning fork will always operate at resonance.

Quartz Tuning Fork

Figure 2 represents the complete electronic circuit of frequency modulation or also known as the Phase Locked Loop (PLL)

The integration of SANM and frequency modulation provides a better scope on the enhanced instrumentation of characterizing mesoscopic films. By testing the concept of frequency modulation into the SANM apparatus, the responses analyzed by the quartz tuning fork and the acoustic sensor could provide us with valuable information on the dynamic properties of these fluids.

As the SANM apparatus controls the approach of the tip towards the substrate, frequency modulation will work in the background to update the shifts of the tuning fork frequency. When the tip plunges into the mesoscopic fluid, the acoustic sensor in the SANM apparatus will pick up ultrasonic vibrations and these increases in ultrasonic vibrations will correspond to multiple shifts in the resonant frequency of the quartz tuning fork. Frequency modulation will ensure that the tip vibrates at the maximum amplitude and the readings of the acoustic sensor can be credited to the damping effect of the mesoscopic fluid. Both of these unique implementations will be mounted on the approach/retraction stage and work in tandem.

**IV. EXPERIMENTAL RESULTS**

The experimental results obtained in this study were conducted to verify the instrumentation of frequency modulation and to prove the reliability and accuracy of frequency modulation. The quartz tuning forks used in this study are commercially manufactured and have a typical resonance frequency in the 32 kHz range. The fabricated tips were made through an electrochemical process that included the use of HCL. These tips are essentially atomic tips and to verify the correct features, they were examined under an electron microscope. If the tips contain all the necessary features, the tip would be attached to one of the sides of the tuning fork.

The first stage of frequency modulation is the Lock-In Amplifier. The Lock-In Amplifier is tested against a commercially verified Lock-In Amplifier called the SR-850. The SR-850 is a Lock-In Amplifier that is commercially sold on the market that also contains a digital signal generator. Below is a comparison of the frequency sweep results taken of the amplitude of the tuning fork.

Fig. 3 represents the Digital Lock-In Amplifier frequency sweep from 32.740 kHz - 32.790 kHz.

The graph of the Digital Lock-In Amplifier Frequency vs. Time displays the frequency sweep taken in this experiment. The frequency sweep taken was from 32.740 kHz - 32.790 kHz and the resonance frequency was detected at a frequency of 32.740 kHz at the scan length of 140 seconds. This key data point is crucial in understanding the set of graphs displayed in this section.

Fig. 4 and 5 are respectively amplitude vs. frequency and amplitude vs. time graphs taken by the SR-850 and the Digital Lock-In Amplifier

As shown in the two graphs above, the shape of both graphs look very similar and the congruence between these two graphs and the two instrumentation will be proved through quantitative evidence. In fig. 4, the graph spikes at around 32,756 Hertz and yields a maximum amplitude of 60 nanoamps. In fig. 5, the graph spikes around a time value of 140 seconds and yields a maximum amplitude of 58.5 nanoamps. On fig. 3, the time value of 140 seconds corresponds to a frequency of 32,756 Hertz and it can be observed that the SR-850 Lock-In Amplifier and the Digital Lock-In Amplifier agree on the identification of the maximum amplitude at the same frequency.

Fig. 6 and 7 are respectively phase vs. frequency and phase vs. time graphs taken by the SR-850 and the Digital Lock-In Amplifier

As shown in figures 6 and 7 above, the graphs correlate in very identical shapes in terms of their troughs and peaks. The key interest in both graphs is to observe the phase shift that is detected by both instruments. In the SR-850 phase vs. frequency graph, it is noted that the 90 degree phase shift is detected around 32,762 Hertz. In the other corresponding graph taken by the Digital Lock-In Amplifier, the 90 degree phase shift is detected around 32,756 Hertz. The interesting disparity between the two data points suggest an experimental error. The experimental error is assumed to be due to the high amount of electrical noise carried by the BNC cables that attach the tuning fork to the SR-850 Lock-In Amplifier. Another experimental error is the initial settings of the graphing function on the SR-850 Lock-In Amplifier. In future research, multiple tests using the SR-850 Lock-In Amplifier will be taken with lower electrical noise and proper settings to ensure maximum accuracy.

Fig. 8 and 9 respectively display the graphs of the PID Microcontroller and the Voltage Controlled Oscillator.

The last of set of graphs contain the readings of the PID Microcontroller and the Voltage Controlled Oscillator. The graphs are identical in nature and that feature displays the cooperation of these two stages. The flat point of the curve symbolizes the detection of a 90 degree phase shift. When a 90 degree phase shift is detected, the PID stops outputting an error and the slope, as a result, is zero as there is no change. In accordance, the VCO shifts in response and does not shift its base frequency which drives the tuning fork.

**V. DISCUSSIONS AND CONCLUSIONS**

In conclusion, frequency modulation with SANM is a much better instrumentation that can potentially characterize the mesoscopic fluid. Each individual part of the phase locked-loop has reliable functionality and the cooperation of these parts displays unity. By comparison to the SR-850 Lock-In Amplifier, the digital Lock-In Amplifier is a low cost and effective piece of instrumentation. The purpose of the experiment is to increase the metrology of SANM through frequency modulation. The two goals of implementing superior instrumentation in this discipline requires two distinguished aims: to enhance or create new techniques that are superior to current techniques and construct instrumentation in the most economical and effective way. The current technique used to characterize mesoscopic films is the SANM method. Frequency modulation enhances the instrumentation and capabilities of SANM in characterizing the mesoscopic layers. The SR-850 Lock-In Amplifier was used to properly scrutinize the results offered by the Digital Lock-In Amplifier. By verifying the reliability and reproducibility of the LabVIEW implemented frequency modulation, it can be proven that tens of thousands of dollars in equipment and material costs have been saved through designing frequency modulation as a software component. The SR-850 Lock-In costs about 8,000 dollars whereas this Digital Lock-In Amplifier costs about 2,000 dollars and has frequency modulation as an additional customizable component. A software written frequency modulation is as effective as physical equipment designed for the same purpose.

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