Ultrasound Sample Environment for In Situ Small-Angle Scattering Experiments

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Background, Motivation and Objective

Ultrasonic sonicators are common tools in lab and industrial settings which produce cavitation vapor bubbles for cleaning, emulsification, cell lysis, etc. Aside from the high pressures and stresses from cavitation events, free radicals and high local temperatures can result from cavitation bubble collapse. The effects of sonication at the macroscopic scale are visible, but its effects at the molecular to nano-scale are not fully understood. We present a new sample environment designed specifically to study changes at the nano-scale due to acoustic forces using small-angle X-ray or neutron scattering measurement.

Statement of Contribution/Methods

The sample environment uses two focused ultrasound transducers coaxially aligned normal to the direction of beam scattering. A third PVDF transducer fixed beneath the sample was used as a cavitation detector. By correlating cavitation detection to the X-ray or neutron scattering data, measured structural changes can be correlated to acoustic pressure and cavitation. *In situ* ultra-small angle X-ray measurements were conducted at the Advanced Photon Source in Argonne National Lab. Small-angle neutron scattering measurements were obtained at the NIST Center for Neutron Research and the high flux isotope reactor in Oak Ridge National Lab. A wide variety of samples and measurements were made including cavitation of pure fluids, emulsification of oil droplets, formation of Pickering emulsions, and ultrasound-assisted P3HT nanofiber formation.

Results/Discussion

The ultrasound sample environment enables the study of nano-scale structural changes during sonication using small-angle X-ray or neutron scattering techniques. Changes down the molecular or nano lengths were easily measured and correlated to acoustic cavitation using the system. X-ray and neutron scattering measurements were insensitive to the cavitation event itself because of the long acquisition times (ranging 1 minute to 1 hour) relative to the short lifetime of the cavitation bubble (microsecond time scale). However, specialized time-resolved X-ray or neutron scattering instruments with down to femtosecond time resolution can be used in conjunction with the ultrasound sample environment to study nano-scale structural changes during the cavitation process.

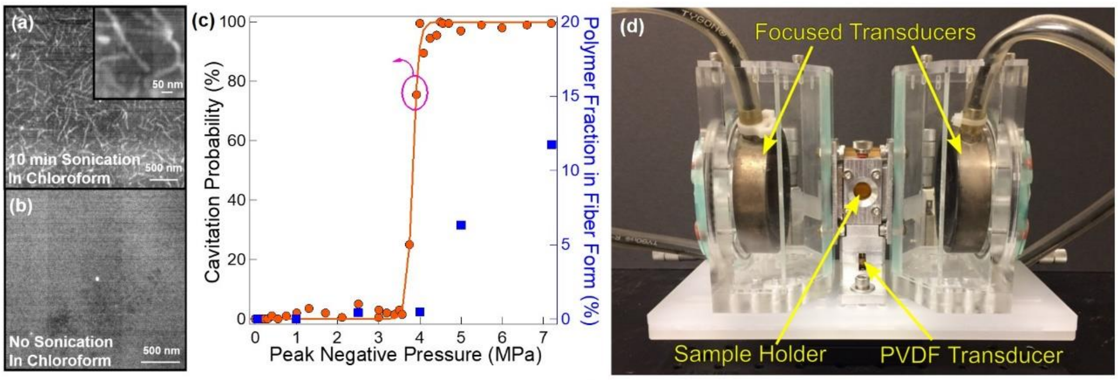

Figure 1: Scanning transmission electron microscopy images of P3HT samples (a) with and (b) without sonication. (c) Formation of P3HT nanofibers only occurred when acoustic cavitation was detected. (d) Small-angle X-ray and neutron scattering measurements were carried out in a custom build ultrasound sample environment enabling *in situ* measurements.