Time-Dependent Effects in AFM Measurements of Disjoining Pressure of PFPE Films

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An important parameter for quantifying the spreading behavior of a perfluoropolyether (PFPE) film is its disjoining pressure. Previous work in this research group has described a method for calculating the disjoining pressure directly from AFM measurements. This work focused on looking at the disjoining pressure qualitatively with the goal of using this information to screen hard disk lubricants in a high-throughput manner. Here, we present new procedures for reducing time-dependent effects. Minimizing these effects is critical to obtaining accurate quantitative data from the AFM method. The accuracy of this data is verified by comparison with Hamaker theory.

The AFM method is based upon interpretation of force-distance curves obtained by probing silicon substrates dip-coated with the PFPE, Fomblin Z 03. Experiments are performed by bringing a spherical cantilever tip into contact with the film. Contact causes a meniscus of PFPE to form due to capillary forces. After contact is made, the AFM probe and the sample are separated until the meniscus between them is broken. Analyzing the stretched meniscus region of force curves with a model describing the Laplace pressure of a PFPE meniscus yields the effective radius of curvature of the meniscus. This radius of curvature is related to the disjoining pressure when the film on the surface is in equilibrium with the film in the meniscus. Equilibrium is defined as the state when the chemical potentials of the film in the meniscus and that on the surface are equal.

To reach equilibrium, it is necessary to consider time-dependent effects. Initial contact between the cantilever and the surface depletes the film in a region around the cantilever tip. To account for this depletion, a cantilever preconditioning step is used. This step involves leaving the probe in contact with the film for a prolonged period of time, then moving it to a new location for force curve measurements. This preconditioning wets the AFM probe with PFPE so less film is drawn from the surface at the location the force curve measurements are performed. Additionally, time-dependent fluid flow is a factor during an AFM experiment. To approach the equilibrium condition it is necessary to retract the sample from contact with the cantilever tip at speeds on the order of 0.1 nm/s. These slow retraction speeds allow PFPE adequate time to drain from the meniscus during the performance of a force curve.

Including these two modifications gave superior results. A plot of disjoining pressure versus film thickness for Fomblin® Z 03 demonstrated behavior in accordance with Hamaker theory; namely, thin films yielded extremely large disjoining pressures while thicker films had smaller disjoining pressures.