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Viscosity of liquids from the transfer function of microcantilevers

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ABSTRACT

A method is presented to obtain the viscosity and density of a fluid from the mechanical frequency response of a microcantilever beam immersed in fluids. The technique is a practical solution to perform the measurement when only very small quantities of the fluid are available. A novel algorithm is described to measure the viscosity and density of an ambient fluid by comparing experimental results with the theory. The theoretical results are analytical with a closed form solution. The algorithm presented is easier to implement than the standard method currently used by most atomic force microscope practitioners. In addition, unlike the standard method, the new algorithm is applicable to high viscosity fluids that do not produce resonant peaks in the microcantilever power spectra. Experiments were carried out using standard cantilevers in a fluid cell filled with air, water, methanol and commercial oils. The result of comparing theory with experiment validates the algorithm and thus we propose the algorithm as a way to measure density and viscosity of uncharacterized fluids.

1. Introduction

Studies of the response of microcantilevers to the surrounding viscous fluids began early in the development of atomic force microscopy. Perhaps the first published suggestion to use the instrument as a fluid viscometer was made by Oden et al. [1]. Those authors used a damped harmonic oscillator approach admitting that such a simple theory would not provide an accurate model over a wide range of viscosities and cantilever responses. In a series of papers, Sader addressed this issue with what has become accepted as the standard method for taking cantilever frequency spectra taking into account fluid viscosity and density [2–5]. This suite of papers is built on a solution to the Euler–Bernoulli equation for thin microcantilevers placed in a perpendicular uniform fluid flow [6]. While in principle a simple result, this approach involves a fairly complicated solution to implement. Sader recognizes this in his seminal paper writing, “The most simplistic model makes the heuristic analogy with the dynamical motion of a sphere through a viscous fluid. Despite its physically dubious foundations, this model has been used widely in many fields and applications.”

However, as early as 1967, it was shown that a string of spheres placed in a flow stream reduces to the solution for a cylinder [7]. Recently, this fact was recognized as being applicable to microcantilevers and the equations were solved using boundary conditions for a magnetically driven cantilever [8]. What we present below is a solution to these equations and boundary conditions, and compare to corresponding experiments from a thermally driven cantilever. This makes the relatively simple and easily implemented result available to the majority of microcantilever users who do not have instruments outfitted for magnetic drive.

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This viscous damping effect will exist in general when measuring forces on soft matter including biological molecules and tissues. Biological materials usually depend on ambient water for structural integrity. Thus useful information of biological matter can be obtained only when immersed in water. However, standard sensor calibration based on frequency response is performed in air. This calibration typically uses measured cantilever power spectra, specifically the central frequency and corresponding quality factor of the first mode resonance peak. Yet, those values are strongly dependent on the ambient fluid. For example, moving from air to water induces changes as large as a factor of ten in frequency and quality factor of typical microcantilevers. While theoretical frameworks have been developed that address the mechanical response of microcantilevers in fluids, it is well known that they lack accuracy due to an assumed rigidity of the cantilever. The result of these shortcomings is that, using the standard method, accurate measurements of cantilever behavior in fluids with densities and/or viscosities very different from air cannot be inferred from the method using calibration in air. We have developed a new method that solves this problem. We show that it works not only to take into consideration the water around the cantilever but also to measure the viscosity and density of other fluids, even high viscosity oils.

The motion of cantilevers submerged in viscous fluids is of interest for a variety of applications. Several theoretical and experimental studies have been published on the effects of viscous fluid damping on the mechanical behavior of microcantilevers. This is important for the purposes of calibration [2–5,9–11] as well as understanding fundamental thermomechanical noise in these oscillators [3,12,13].

We have an interest in analyzing the motion of cantilevers in fluids to characterize lubricants. Of particular interest is the case of ionic fluids which are candidates for space lubrication. Both in the development of these expensive fluids as well as during quality checks when they are in service, characterization using the smallest possible volume are desirable. Lubrication for space vehicle mechanisms is of special interest because standard terrestrial lubricants do not perform well in space due to the need to operate the systems at extreme temperatures and pressures. For example standard oils solidify at the low temperatures existing in space and evaporate under the low pressures as well. To address these problems, research groups have directed their attention to the possible use of mixtures of ionic liquids. With the proper recipe, they remain liquid to lower temperatures and exhibit inherently low vapor pressures. The ability to accurately measure viscosity of prototype samples of such ionic lubricants using small volumes would aid in their development.

Another application of fluid viscosity measurement that interests us involves clinical medical use of the technique. Currently, viscosity of blood is used in the analysis and treatment of several disorders [14] including blood clotting [14] and hemophilia [15]. Viscosity of other fluids like that found in joints is also of clinical interest [16]. The ability to make these measurements using microfluidic technology is well appreciated [17]. Most of these technologies require a measurement of flow in a capillary tube. A better understanding of how cantilevered beams move in fluids would allow for development of a complimentary technology for making clinical fluid viscosity measurements using very small samples or under in vivo conditions.

Silicon based cantilevers offer a relatively simple platform for measuring viscosity of fluid samples. This is desirable in cases where only a small sample is available or where a micro sensor is necessary. Damping of cantilever oscillations was demonstrated as a method for measuring viscosity in gases and low viscosity liquids [9,18,19] and for measuring viscosity in mixtures of water and glycerol [1,20]. These demonstrations used both resonant frequency shifts and changes in the quality factor, Q, to measure fluid viscosity. Our method works in those conditions and also when the resonant peaks disappear due to high viscosity damping. This feature makes the technique valuable even when low driving power (i.e. thermally driven microcantilevers) and large viscosity conflate to obliterate the resonance peaks.

Various physical models were developed to describe cantilever dynamics, and were applied to the measurement of viscosity of fluids. The analysis of highly viscous fluids, such as lubricant oils, presents technical difficulties. Typically, lubricant viscosity must be measured over a wide range due to changing operational temperatures and thickening caused by aging. Consequently, a robust calibration curve which spans a large viscosity range is necessary. It is desirable to have a relatively simple calibration process. For example, a calibration curve that requires only the use of air and a single liquid would simplify implementation. Accordingly, it is advantageous to have a single relationship between viscosity and an experimentally measurable cantilever property accurate over a viscosity range spanning air and very viscous liquids.

Measurement of viscosity by oscillating cantilevers is extensively discussed in the literature [1,3,21]. The general principle of operation of this kind of sensor rests on the fact that viscosity, an energy dissipative mechanism, affects the widths of the resonance amplitude vs frequency peaks. Thus, an overarching goal of these methods is the search for and validation of correlations between viscosity and some directly measurable quantity, typically the quality factor and/or location of the frequency at peak resonance amplitude. To contribute to this field, we developed a new approach that explicitly takes into consideration the many degrees of freedom of the beam sensor. Since the approach allows for differing relative velocities along the beam, we utilized a local interaction description between the sensor and the surrounding fluid. This work builds on our earlier attempt to measure viscosity by introducing first principles expressions for dissipative cantilever forces instead of empirical expressions [22,23]. This puts the method on a sounder foundation and provides a larger parameter range of applicability.

2. Dynamics of the cantilever beam

The viscosity sensor we consider is a tipless microcantilever, as shown in Fig. 1.

It is a micrometric prismatic beam connected to a relatively massive silicon chip on the left and free on the right. Often, the silicon chip is connected to a piezoelectrically actuated base allowing for driven oscillations of the