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Measuring hydrocarbon viscosity with oscillating microcantilevers

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ABSTRACT

This experimental–theoretical paper presents a reconstruction algorithm that enables the measurement of viscosity from the experimental resonance spectrum of a microcantilever immersed in the fluid under study. An analytical, closed-form solution of the equation of motion, including damping and internal friction, for the microcantilever frequency response is used which allows direct measurement of the fluid damping coefficient. This damping coefficient has a simpler relationship to viscosity than the commonly used quality factor, thus simplifying the process of calibrating and obtaining viscosity from experimental data. We validate the method by experimentally confirming that the relationship between viscosity and damping constant is quadratic. This is done with hydrocarbon liquids spanning a wide range of viscosities. Finally, we apply the method developed here to measure the viscosities of Hexadecane and Dodecane, obtaining very good agreement with accepted values.

1. Introduction

Measurement of the viscosity of liquids and gases by means of oscillating cantilevers has been extensively discussed in the literature. The general principle of operations of this kind of sensors rests on the fact that viscosity, an energy dissipation mechanism, affects the widths of the resonance amplitude-vs.-frequency peaks. Thus, an overarching goal of these methods is the search, proposition and validation of correlations between viscosity and some directly measurable quantity, typically the quality factor of a frequency resonance. To contribute to these developments, we present in this article an innovation based on the recognition that viscosity is linearly related with a parameter introduced in our model, the friction coefficient squared, defined in the body of this article. This relationship, based on theoretical considerations, is validated using extant published results. Then, using viscosity standards, we perform a calibration of our system. Finally, we use the sensor to predict the values of viscosity for Hexadecane and Dodecane and to compare them with their viscosities as obtained from other methods. Of particular interest is the fact that the cantilever we used to develop the sensors presented in this article, are the same ones used in atomic force microscopy (AFM). AFM started to be extensively used in the nineteen nineties and has evolved dramatically in many directions. However, one area that is still open for technological advances is that of AFM operation in a liquid environment, mostly driven by the interest of using the microscope in biological buffers. The problems of AFM in liquids can be roughly divided into tip-sample interaction issues, and cantilever related ones. The first category entails the understanding of the modification of nanoforces due the presence of the liquid environment. The second aspect is related with the dynamics of cantilevers in fluid. An incomplete understanding of the latter aspect can render a reconstruction algorithm invalid (for example an algorithm to convert tip-sample force into a voltage at the AFM photodiode).

This work contributes to generic principles of physical sensing and instrumentation [1] as follows. Driven by industrial needs, we require that our sensor be inexpensive, robust, accurate and compact. In order to reduce cost and increase accuracy we have relied on computer design, whereby the mechanical subsystem was designed from

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first principles and computer programs were written to extract parameters from the experimental curve. Indeed, to streamline the design of the sensor, we begin from a general differential equation that describes the motion of cantilever. This equation contains information about internal dissipation and dissipation due to the interaction between the cantilever and the surrounding fluid. The latter term is central to our design concept and we were able to demonstrate a linear relationship between the strength squared of this term and the surrounding viscosity, a fact that makes data processing particularly simple.

Silicon microcantilevers (MC) offer a relatively simple platform for measuring viscosity of very small samples of fluid. This is desirable in cases where only a small sample is available or where a micro sensor or array of sensors is necessary. Damping of MC oscillations was demonstrated as a method for measuring viscosity in gases and low viscosity liquids [2–4] and for measuring viscosity in mixtures of water and glycerol [5,6]. These papers used both resonant frequency shifts and changes in the quality factor, Q, to study fluid viscosity. Various physical models have been developed to describe the MC dynamics, and have been applied to the measurement of viscosity of fluids. One of them relies on the linear spring approximation, and incorporates damping, effective mass and geometric [7]. Another accounts for viscous damping by considering the MC as a rigid oscillating beam interacting with the surrounding fluid [8]. It was in turn used to obtain viscosity of low viscosity fluids where Q was relatively large [4]. On the other hand, the analysis of highly viscous hydrocarbons, such as lubricants, 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 is necessary which spans a large viscosity range. It is desirable to have a relatively simple calibration process. For example, a linear calibration curve that only requires the use of air and a single liquid would simplify implementation. Accordingly, it would be advantageous to have a single linear relationship between viscosity and an experimentally measurable MC property, accurate over a viscosity range spanning air and very viscous liquids.

In this paper we experimentally demonstrate a linear relationship between viscosity and the viscous damping coefficient squared. This relationship, being linear, is simpler than the standard one between Q and viscosity. We begin from the equation of motion for a MC that includes an external damping term and an internal friction dissipation term. A closed form solution to this equation was derived by us [9] and provides an explicit solution for the frequency response of the MC. The use of this solution offers several benefits over previous methods (using Q) for extracting fluid viscosity from the MC vibration spectra. First, the frequency spectrum is explicitly dependent on β, the damping coefficient. This allows β to be obtained directly from the resonance spectrum even for an elastic MC model including bending, damping, and internal friction terms. In contrast, for an elastic MC model, Q has a complicated (analytically unknown) dependence on the resonance spectrum. To overcome this problem Q is usually derived using the harmonic oscillator approximation [4]. Second, and more importantly, under typical conditions, β has a simpler relationship to viscosity than Q. Third, without complicating the data analysis process, the present model explicitly accounts for internal friction.

2. Theory

We use the continuum model for an elastic MC driven by an external sinusoidal force [9],

$$\frac{EI}{A}\frac{\partial^4 u(x,t)}{\partial x^4}+\rho\frac{\partial^2 u(x,t)}{\partial t^2}+\beta\frac{\partial u(x,t)}{\partial t}+\gamma\frac{\partial^4 u(x,t)}{\partial x^3\partial t}=0 \quad (1)$$

Here, $u(x,t)$ is the vertical deflection at position x along the cantilever at time t. E is the Young Modulus, I the cross sectional moment of inertia, A the cross sectional area, ρ the effective density, γ is the internal friction damping coefficient, and β is the damping coefficient due to fluid viscosity. The effective density accounts for the mass of the cantilever and the mass of the fluid that moves along with the cantilever.

The following assumptions are inherent to the model:

- The deflection of the cantilever tip is small in relation to cantilever length.
- The beam has constant density and elasticity.
- The fluid is incompressible and Newtonian.
- The Reynolds Number is less than 1.

By definition, Reynolds's number $R \equiv \frac{\rho_F VD}{\eta}$, where V is the velocity of the cylinder moving perpendicular to its axis, D its diameter, $\rho_F$the density of the fluid, and η the viscosity of the fluid. Although the understanding of the drag on a solid by an embedding fluid is incomplete, even for a cylinder, there is an extensive amount of theoretical and empirical work. For an extensive review of the literature, the reader can consult the excellent and current book by Zdravkovich [10]. For R small (as we will show is the case for the conditions of the MC), it has been shown that $\beta = C\eta^{1/2}$, where C is a constant proportionality factor for a given cantilever [11].

As a first step of the work, we will confirm experimentally that $\beta = C\eta^{1/2}$ is indeed satisfied for MC embedded in liquids. Two comments are in order about this equation. First, it establishes a linear relationship between the damping coefficient squared and viscosity. This immediately suggests a powerful technique to measure viscosities: for a given MC one chooses two fluids of independently known viscosities (for example air at room conditions, and a viscous hydrocarbon). For them, one measures their frequency power spectra and by fitting to theory (as described below) obtains the corresponding βs. This provides two calibration points in the (β,η) plane. Then, to measure the viscosity of an unknown fluid, one extracts the β from its own frequency power spectrum and, to obtain η, interpolates between the two calibration points. Second, strictly, $\beta = C\eta^{1/2}$ was derived for a circular cylinder, but here we present experimental results for a MC with a rectangular cross section. This dilemma is resolved in the Appendix, where we show that the two expressions do not differ by more than 5% for a typical MC.