OPTICAL INTERFEROMETRIC APPLICATIONS TO FLUID VISCOMETRY

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ABSTRACT

Optical interferometric monitoring during spin coating (optospinography) of fluid coatings has allowed close observation of their temporal evolution (typically at 700-3000 rpm, 1KHz microcomputer-assisted data acquisition). When the fluids are non-volatile, kinematic viscosity data can be determined using a simple analytical model. Validity of the method was tested with a standard (mineral oil OP20) with nominal kinematic viscosity $\nu = 37 \pm 1$ cS (temperature fluctuation of $\pm$ 0.5 ^{o}C), for which the optospinography method, combined with Abbe refractometry, provided $\nu = 36.1 \pm 0.1$ cS (angular speed uncertainty of $\pm$ 5 rpm). This is an indication that we now have available an absolute, contactless optical viscometry method for thin (thickness less than 10 μm) fluid films in small samples.

INTRODUCTION

As liquid films have been largely used in industrial processes for decades, their viscosity at formation stages still remains a relevant parameter for process evaluation, control and reproducibility.

For spin coating in open air, the optospinography technique, where an interferometric representation is obtained for the temporal variation of the film optical thickness during the process, has allowed us to distinguish four main stages of evolution: (I) pre-spinning oscillations in the sol, (II) ultrafast convective mass flow, (III) convection-evaporation, and (IV) limited evaporation. [1]

These two last stages are greatly simplified when spin coating is performed in the absence of significant evaporation, therefore allowing simple analytical modelling of the process. In a previous work, we proposed adaptation of the technique to the measurement of viscosity of liquids during spin coating, and considered its potential and validity for spinning sols [2].

In this study, we focus our attention in non-volatile oils. Monitored by optospinography, temporal variation of optical thickness is presented and discussed, as well as its relationship with kinematic viscosity. Validity of the method is tested with an oil standard.

EXPERIMENTAL

Analysis is made of a laser beam reflected from the liquid film on a silicon wafer or glass substrate, spinning at speeds in the range of 700-3000 rpm, in the experimental set up described in [1]. The ratio between the sample and the reference signal is computed after processing by an A/D converter, and readings are taken at every 1ms. Alignment of the system is critical, to ensure that the illuminated spot remains at the center of the substrate at all spinning times, which are typically up to 60 s. Conversion from optical to physical thickness is performed by index measurement using a commercial Abbe refractometer.

THEORETICAL

Assuming a horizontal, plane rotating disk infinite in extension, and a thin liquid film with negligible Coriolis forces and Newtonian behavior, the Emslie, Bonner and Pack (EBP) model [3] predicts a thickness time dependence in the form:

$$d = d_0[1+(\frac{d_0}{b})^2 t]^{-\frac{1}{2}} \quad ; \quad b^2 = \frac{3n}{4w^2} \qquad (1)$$

where $\mathbf{d_0}$ is the initial thickness, $\mathbf{w}$ the angular speed, and $\mathbf{n}$ the kinematic viscosity (coefficient of viscosity per unit density). For times $t >> \frac{b}{d_0}$,

$$d \approx bt^{-\frac{1}{2}} \quad . \qquad (2)$$

It should be also pointed out that no mass loss is considered in this model, which therefore cannot be applied to situations where there is significant evaporation to the surrounding atmosphere.

VALIDATION AND RESULTS

In order to validate the results obtained from optospinography , we applied our method to an oil standard with kinetic viscosity value ν_1^0 = 0.37 ± 0.01 Stokes (mineral oil OP20) at 22 °C. This value was obtained from interpolation, with a second degree polynomial fitting, starting from the supplier specifications at a set of temperatures; the uncertainties correspond to a temperature fluctuation of ± 0.5 °C in our experimental conditions. A typical optospinogram, where reflectance modulation was measured in real time, is shown in figure 1.

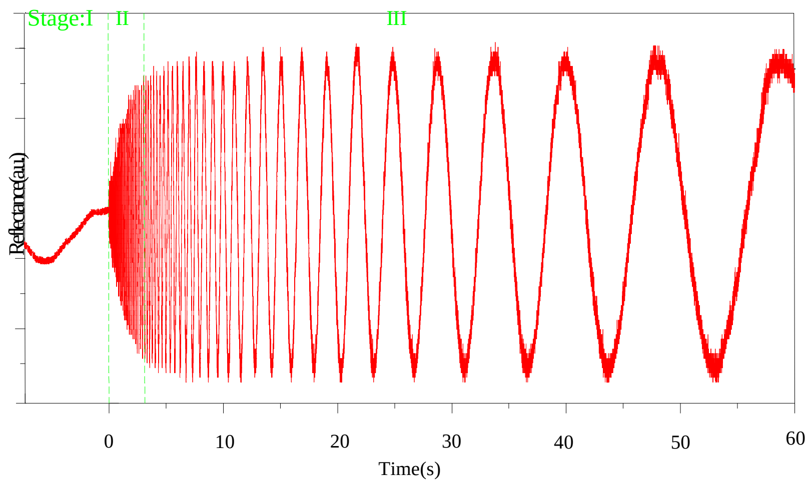

Fig. 1 - A typical optospinogram for mineral oil standard OP20, in open air, at 1000 rpm.

As inferred from the optospinogram data,[1] fig. 2 shows an interferometric representation of the optical thickness temporal evolution at three different processing speeds.

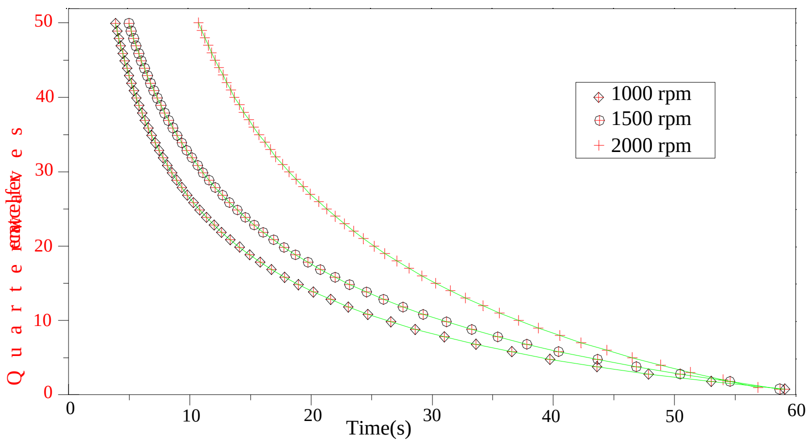

Fig. 2 - Optical thickness variation with time, in quarterwave units, for a spinning liquid coating (mineral oil OP20) at three different speeds of rotation. For 1000 rpm, data was extrated from figure 1. Counting of quarterwaves starts from the last reflectance extreme in each optospinogram.

From these curves, kinematic viscosity may be inferred, using relations (1) and (2).[4] The measured temporal evolution of the oil optical thickness at 1500 rpm is shown in fig. 3, where time is scaled considering the model prediction.

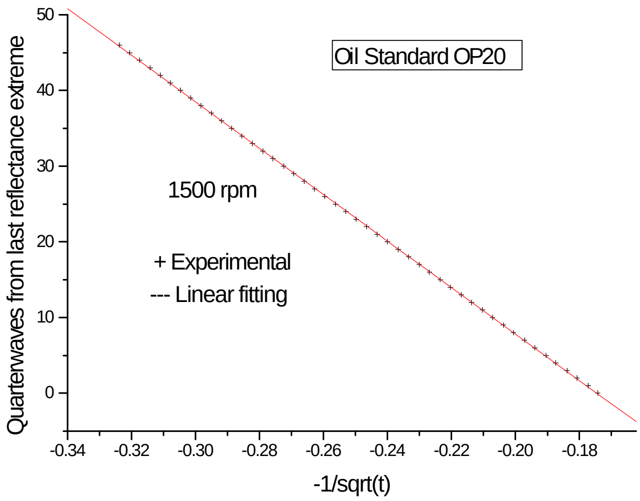

fig. 3 - Optical thickness variation of oil standard OP20 (in quarterwaves, at 632.8nm and 1500rpm) versus inverse square root of spinning time ($s^{-1/2}$), preceded by a minus sign for plotted variation with increasing time, in open air. Linear fitting corresponds to the EBP model prediction, for a Newtonian fluid with n_1 = 0.361 Stokes.

DISCUSSION AND CONCLUDING REMARKS

The fact that a straight line is obtained, corresponding to a constant ν, implies that the liquid is Newtonian and that the EBP model is valid in all measured range. Conversion from optical to physical thickness is made possible by a direct measurement with an Abbe refractometer, giving $n_1 = 1.46 \pm 0.01$. From this procedure, the obtained result for the oil standard is $\nu_1 = 0.361 \pm 0.001$ Stokes, where the uncertainties correspond to the observed angular speed fluctuation of $\pm$ 5 rpm.

The matching achieved between the known kinematic viscosity of the oil standard and the result obtained by optospinography, within the experimental uncertainties, indicates validity of the method.

We therefore have good theoretical and experimental indications that, despite the simplifying assumptions in this study, absolute kinematic viscosity of non-volatile fluids can be determined from contactless optical measurements during spin coating.

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