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Roughness-induced strong pinning for drops evaporating from polymeric surfaces

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ABSTRACT

The dynamic behavior of water drops evaporating from poly(methyl methacrylate) (PMMA) substrates with different roughness was examined. The relaxation of the wetting diameter, height, volume and the contact angle of a drop was monitored during the evaporation process. For the smoother substrates with $R_q \leq 19$ nm, four stages were identified during evaporation: the initial spreading stage, the constant contact radius (CCR) stage, the constant contact angle (CCA) stage, and the final stage. In contrast, for substrates with large roughness ($R_q \geq 209$ nm), the CCA stage was not observed, and the contact line was pinned at its original position during almost the entire lifetime of the drop. The energy barrier associated with the pinning and depinning of the contact line was estimated using the Shanahan model, and the energy associated with PMMA substrates of roughness $R_q = 4.9$ and 19 nm was found to be on the order of 10^{-8} J/m. The influences of the liquid withdrawal rate and the internal flow field were also studied, and the weak to strong pinning transition was found to be roughness dominant.

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1. Introduction

In their pioneering work, Picknett and Bexon [1] found that there are two distinguished modes for evaporation of single component fluids on solid substrates: a constant contact angle (CCA) mode and a constant contact area (CCR) mode. Other researchers later confirmed this observation [2,3]. Birdi and Vu reported that for smooth solids, the contact radius remained constant while the contact angle, θ , decreased for wetting liquids ($\theta < 90^\circ$). However, for non-wetting liquid/solid pairs, the contact angle remained constant during evaporation [4]. Similar results were shown by McHale et al. [2] and Erbil et al. [5].

A third mode, the “stick and slip” mode, was later discovered for various liquid/substrate combinations [6–11]. Bourges-Monnier and Shanahan [12] observed that four distinct stages occur during the course of evaporation on polymeric substrates. As described by Bourges-Monnier and Shanahan, in the initial stage (stage I), the surrounding atmosphere is saturated, and therefore, a small decrease in the drop height and contact angle can be seen, while the contact radius remains essentially constant. After stage I, the atmosphere is no longer saturated, and the contact angle decreases more quickly than in the first stage, while the contact radius

remains unchanged with the contact line “pinned”. This is followed by stage III, where the contact line is “depinned”. At this stage, the drop height and radius decrease simultaneously, while the contact angle stays relatively constant. However, slight pinning can still be observed in this stage. Multiple “stick-and-slip” behaviors are seen, which are characterized by abrupt changes in the wetting radius and the contact angle during evaporation. Finally, in stage IV, the contact angle, the contact radius and the height simultaneously decrease until the drop completely disappears.

Bormashenko et al. observed similar behaviors [3,8,13]. They performed experiments on high-energy surfaces and low-energy surfaces and found differing behaviors. For high-energy surfaces with chemical binding on the order of 1 eV, strong pinning was observed, and a mode similar to constant contact area evaporation mode was discovered. However, for low-energy surfaces with energy orders of $k_B T$, where k_B is the Boltzmann constant and T is the temperature, a multi-stage pinned-depinned behavior that was similar to what Bourges-Monnier and Shanahan reported was observed. The strong pinning and weak pinning behaviors were also observed in other recent studies. Pittoni et al. showed that multiple polymeric substrates, including acrylonitrile butadiene styrene (ABS), polycarbonate (PC), poly(methyl methacrylate) (PMMA) and polyvinyl chloride (PVC), exhibit similar behaviors [10,14].

The exact mechanism of contact line pinning is still under debate. In the literature, the mechanism has been attributed to contact angle hysteresis originating from surface heterogeneity

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[6,15–19], adsorption of the substrate to the fluid drop [20], and line tension [21–23]. In particular, Shanahan derived a simple model in 1995 that can be used to estimate the line energy barrier required for the contact line to depin using experimental data [6]. Various studies have used the equation [3,8], and the line energy barrier has been found to be on the order of $10^{-6} - 10^{-7}$ J/m, which is on the same order of magnitude as typical line tension values found in the literature [24–28]. Whether the line energy, *i.e.*, the energy barrier for the contact line to depin, can be directly correlated with the line tension is still under debate [13,29].

While the exact mechanism that leads to pinning is not fully understood, the “slipping” or “depinning” stage, where the contact angle assumes a constant value, has been identified with the receding contact angle (CA) [10,30]. Though minor discrepancies exist in the definition of advancing and receding contact angle [13,31–34], the advancing and receding CA has been identified as an important characterization of a given solid-liquid-vapor system. However, no universally acknowledged standard exists for measuring advancing and receding contact angles, and disparities between contact angles obtained from differing procedures were found in the literature. For example, Bormashenko et al. [35] and Erbil et al. [10] showed that different CA measurement methods yielded disparate results for water on various polymeric substrates. Additionally, Erbil et al. showed that the liquid withdrawal or introduction rates influenced the measured receding contact angles using the needle-syringe method [10]. As a result, Pittoni et al. [36] suggested that a standardized scheme for dynamic CA measurement should be devised to solve this problem. In this study, we adopted the definition used by Pittoni et al. [36] and identified the stage III contact angle as the receding contact angle.

Furthermore, surface roughness has also been reported to greatly affect the dynamic behavior of triple line. Previous studies found that for hydrophobic substrates, increasing surface roughness could lead to greater hydrophobicity and lower hysteresis [32,37–39]. However, recent studies showed that the effect of roughness on contact angle hysteresis is more complicated. Li et al. found that when roughness was increased, three types of wettability behaviors were observed for various polymeric substrates [40], while Chang et al. investigated the effect of roughness by studying the stability of air pockets and the imbibition of liquid into conical holes and found three types of wetting behaviors [41]. On the other hand, pinning was also found on structured surfaces and rough surfaces. Several researchers found that evaporation of drops on structured hydrophobic surfaces can trigger the wetting transition from Cassie-Baxter state to Wenzel state [42–44], thus locally pinning the triple line [8]. Locally pinned contact lines were typified by a stick-and-slip behavior, where the wetting radii exhibited regular jumps during evaporation [45]. While hydrophobic substrates generally did not exhibit strong pinning, rough hydrophilic substrates were shown to exhibit strong pinning. Shanahan and Bourges reported that for water on rough epoxy resins, the constant CA stage was not reached on a rough surface [7,12]. Similarly, Soolaman and Yu reported that for water on various substrates, *e.g.*, gold, Teflon, silicon wafer, and polycarbonate, pinning, depinning and hysteresis were dominated by surface roughness rather than chemical heterogeneity [46]. In recent studies, Pittoni et al. reported that even low-energy polymeric surfaces, such as PC, may exhibit a transition from normal four stage dynamics described previously into complete pinning, *i.e.*, without stage III and with the contact line pinned during the entire lifetime [14,36].

We were curious as to whether this roughness dominant phenomenon is universal for all polymeric substrates, and the aim of this work was to examine the role of substrate roughness on the pinning/depinning dynamics of triple line on polymeric substrates. In this study, the evaporation of water drops on PMMA substrates of various surface roughness was investigated using the

sessile drop method with video-enhanced image digitization technology and best-fitting algorithms. The dynamic behavior of the triple line of pure water on PMMA substrates was studied in terms of drop height, wetting diameter, drop volume, drop shape and contact angle.

Pittoni et al. showed that relative humidity during evaporation had a negligible effect on the dynamics of the contact line [36]. As a result, the relative humidity was conveniently chosen to be $H_r = 65\%$ for all evaporation experiments. To further verify the phenomenon, the needle-syringe method was employed, and various flow conditions and withdrawal rates were tested.

2. Materials and methods

2.1. Apparatus

In this study, an apparatus similar to the video-enhanced sessile drop tensiometer utilized by Lin et al. [30] was used. The apparatus was composed of an image forming and recording system, an ambient temperature control system, a humidity control system, and a video-image profiler digitizer.

The image forming and recording system was used to create and record silhouettes of sessile drops on solid substrates. The image forming system consisted of a light source (halogen lamp with constant light intensity; Oriel, QTH 63200), a lens system for producing a collimated beam, a quartz neutral density filter and a pinhole. The image recording system consisted of an objective lens (effective focal length 60 mm, f/no. 7.1), a video recorder, and a solid-state video camera [MS-4030 charge-coupled device (CCD), Sierra Scientific Co.]. The samples were installed on adjustable stages placed on a vibration isolated workstation.

The drop forming system consisted of a stainless-steel needle connected to a normally closed port of a three-way miniature solenoid valve (Lee Co.) via 1/16 in. i.d. Teflon tubing. The common port of the valve was connected to a gas-tight Hamilton syringe placed in a syringe pump. The valve was controlled by a D/A card with a personal computer.

The video-image system (Pico, Euresys) digitized images into 480×640 bitmaps with 8-bit gray level resolution. The rate of image acquisition was 30 images/s.

The ambient temperature and humidity control system consisted of an air chamber, a fan, a heater, a cooling pipe, a humidifier, a thermistor and a humidity probe. The chamber was made of a 1 cm thick poly(methyl methacrylate) (PMMA) walls. The temperature was controlled with a PID controller connected to the heater.

2.2. Materials

The water used in this study was purified by a Barnstead NANOpure water purification system with a specific conductance of less than $0.057 \mu\text{S}/\text{cm}$. The PMMA (CAS 9011-14-7) used in this work was purchased from Sun-Fung Co. Ltd., Taiwan. In the experiment, substrates underwent grinding for 15 minutes using sand paper (ISO 6344) with grit sizes of 5, 8, 14, 22, 35 μm .

Atomic force microscopy (AFM) analyses were conducted to investigate the topography of the substrates prior to and after the grinding process. A $100 \times 100 \mu\text{m}$ area was scanned in contact mode for each sample, and root mean squared roughness, R_q , was found to be 4.9 nm for the original PMMA substrate and 19, 209, 296, 391, and 1110 nm for the other ground substrates. The morphologies of the substrates are shown in Fig 1.

2.3. Methods

The experimental procedure described as follows was utilized in all experiments. The polymer substrate was rinsed with pure

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