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Probing Micro-Newton Forces on Solid/Liquid/Gas Interfaces Using Transmission Phase Shift

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Supporting Information

ABSTRACT: Many of the nature and life systems are driven by capillary interactions on solid/liquid/gas interfaces. Here, we present a profilometry technique called transmission phase shift for visualizing the liquid/gas interfaces in three dimensions with high resolution. Using this approach, we probe the change in tiny forces with particle radius at a solid/liquid/gas interface. We provide the first direct evidence that in the issues of floating versus sinking at small-scale, Archimedes' principle should be generalized to include the crucial role of surface tension and reveal the dominant regimes of floating particles based on the Bond number. Remarkably, the measured forces are in the range of micro-Newtons, suggesting that this terse methodology may guide the future design of a liquid microbalance and will be a universal tool for investigating capillarity and interface issues.

INTRODUCTION

Liquid/gas interfaces are found throughout nature and play important roles in almost all scientific fields from chemistry,^{1,2} physics³ to biology⁴ and engineering.⁵ Floating versus sinking of solids on a liquid/gas interface has not only been driven by countless technological applications such as flotation,^{6,7} self-assembly,^{8–10} encapsulation,¹¹ liquid sloshing,^{12,13} or for the air–liquid interface cell culture¹⁴ but also is a matter of dead or alive for water-walking insects that live on the water surface.^{15,16} These raise the natural question, When can an object float? The more specific question is, Why small objects that are denser than the liquid can still float? In this vein, Archimedes, who over 23 centuries ago, first discovered the law of buoyancy, also known as Archimedes' principle, which may be stated as buoyancy = weight of the displaced fluid.

In spite of its terseness and elegance, however, Archimedes' principle does not consider the surface tension acting on the floating object. The generalization of the theory has always been actively investigated^{17–21} since Galileo's first glance at the tiny deformation of the liquid/gas interface,²² that is, what we would now call the meniscus. In this context, Keller formulated that the weight of the liquid displaced by the meniscus comes from the effect of surface tension, which provides part of the upward force, also known as the capillary force, on a partly submerged body.¹⁷ However, there is still no experiment to date that provides the direct evidence, as probing tiny forces at a flowable liquid/gas interface has always been a big challenge. In the recent study on measuring the capillary force on a liquid/gas interface, Lee and Kim carried out an elegant work on the role of superhydrophobicity in the adhesion of a floating

horizontal cylinder.²³ The method they used is the classical tensiometry technique, whose resolution is up to 0.1 μN . Using the same method, Pitois and Chateau measured the force–path curves on the removal of small spheres initially floating on a liquid/gas interface and analyzed the effect of contact angle hysteresis on the force of the spheres.²⁴ This technique is very simple, yet highly accurate; however, it is only applicable for the detaching or pressing process of a floating object. Moreover, it cannot visualize the liquid/gas interface, which the three-dimensional imaging of the interface would greatly enhance the understanding of the dynamics water-walking problems.²⁵

To resolve the problem, the measurement of surface tension or surface stress may provide a way: they are based on monitoring the deformation of interfaces, which is determined by the competition of forces, and indicates characteristics of the underlying forces.^{26–30} Using this principle, Lee and Kim evaluated the interfacial tension force depends on the two-dimensional meniscus profile and studied the sinking of a small sphere through an interface.³¹ Danov et al. visualized the two-dimensional shape of the capillary meniscus around a charged particle at a liquid/liquid interface, which can also be used to calculate the normal force acting on the particle.³² However, such measurements are limited to one-dimensional situations. Furthermore, visualizing a liquid/gas interface in three-dimensions is more hardly achievable owing to its specular

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and rheological properties. In our previous works,^{33,34} we discussed the reconstruction of the topography of a liquid/gas interface using a grid or dot pattern. The resolution of these techniques, however, was not high enough to measure the very tiny deformation of the interface. In addition, the relation of liquid deformation with surface forces has not yet been explored.

In this paper, we develop a profilometry technique called transmission phase shift (TPS) inspired by transmission electron microscopy³⁵ and present the first direct measurement of micro-Newton forces at a solid/liquid/gas interface based on the reconstructed topography. We demonstrate that the balance of floating versus sinking should be described by a generalization of Archimedes' principle, which includes the crucial effect of surface tension. This technique has important implications for any processes involving a liquid/gas interface, for example, investigating the hydrodynamics of walking on water,²⁵ dynamics of wetting and spreading,³⁶ issues in capillary wave,³⁷ and so forth.

METHOD DEVELOPMENT

The experimental configuration for TPS profilometry is shown in Figure 1a. A camera, a glass tank filled with water, and a

Figure 1. Illustration showing the visualization of a solid/liquid/gas interface. (a) Experimental configuration. The digital projector projects a sequence of digital fringe patterns and their transmission patterns are captured by the camera. (b,c) Partially submerged particle on the liquid/gas interface. (d) Captured images, which the fringes are distorted with the deformation of the interface.

digital projector (e.g., tablet PC) are set up in a vertical line. Four digital phase-shifted fringe patterns are projected, and their corresponding virtual images, that is, the transmission fringe patterns, are recorded by the camera. The basic principle of TPS profilometry is, when the interface deforms out-of-plane, the transmission fringe patterns are distorted in-plane because of the reflection of transmission light, as shown in Figure 1b–d. Hence, the out-of-plane measurements of the interface can be converted into the in-plane measurements of the fringe distortion of the transmission fringe patterns.

According to Snell's law and the triangular relationship (see the Supporting Information for the derivation process), the topography of the liquid/gas interface correlating with the phase difference of the fringe patterns can be written as

$$h(x, y) = \frac{u^2 + \sqrt{(n^2 - 1)u^2 + n^2}}{u[1 - \sqrt{(n^2 - 1)u^2 + n^2}]} \cdot \frac{p}{2\pi} \Delta\Phi_x(x, y) + H \quad (1)$$

where $u = \frac{\partial h}{\partial x}$ is the partial derivative of $h(x, y)$, n is the refractive index of liquid, p is the period of the fringe pattern, $\Delta\Phi_x(x, y)$ is the phase difference of the fringe patterns and is extracted using phase-shifting, H is the initial depth of liquid, and $h|_{x=0} = 0$ is the initial condition, which represents the water level of the interface.

For a very small deformation of the liquid/gas interface with small angles of incidence and emergence, eq 1 can be simplified to $\frac{\partial h(x, y)}{\partial x} = \frac{n-1}{n-1} \frac{p}{H} \Delta\Phi_x$ (see the Supporting Information for the derivation process). Therefore, the measurements of minimum variation depend on the magnification of the camera, the depth of the liquid, the period of the fringe pattern, and the phase resolution. To obtain a high-phase resolution, we utilize the phase-shifting technique³⁸ because of its robustness and accuracy in phase analysis with a theoretical phase resolution $\Delta\Phi_{\min} = 180^\circ/256$ ($\cong 0.01$ rad).³⁹ Considering the known and most commonly used parameters of, refractive index of water $n = 1.333$, water depth $H = 60$ mm, and period of fringe pattern $p = 1$ mm, a common megapixel camera with a pixel size of $x_{\text{pix}} = 10$ μm , a theoretical vertical resolution of up to 1 nm is achievable. This methodology is ultrasensitive to interfacial variation.

RESULTS AND DISCUSSION

Using this approach, we performed a series of experiments for visualizing the out-of-plane deformation of a liquid/gas interface and measured tiny forces on the interface. The experimental setup mimicked the geometry of Figure 1a. A transparent glass tank ($200 \times 200 \times 100$ mm³) was placed onto a flat iPad tablet and filled pure water with 60 mm depth. The thickness of the bottom glass δ approaches zero as the depth of water H was large, that is, $\delta/H \rightarrow 0$. A fast camera (IL5-H, Fastec Imaging) assembled with a 10 $\times$ lens was set up above the tank and focused on the tablet.

Phase Distribution. Four phase-shifted fringe patterns were first prepared in the tablet and sequentially projected in real time (25 fps) (see Supporting Information Figure S2). They were captured by the camera, as shown in Figure 2a, which can be expressed as $i_k = a(x, y) + b(x, y) \cos[\phi(x, y) + \frac{k\pi}{2}]$, where $k = 0, 1, 2, 3$ is the phase-shifted index, $a(x, y)$ is the intensity of background, $b(x, y)$ is the modulation intensity, and $\phi(x, y)$ is the phase distribution of the fringe patterns. These patterns served as the reference.

Then, a particle made of polytetrafluoroethylene (PTFE) was gently placed on the air–water interface and viewed from the above by the camera. The four identical phase-shifted fringe patterns were projected by the tablet and their corresponding transmission images were captured by the camera, as shown in Figure 2b, which can be expressed as $I_k = a(x, y) + b(x, y) \cos[\Phi(x, y) + \frac{k\pi}{2}]$, where $\Phi(x, y)$ is the modulated phase distribution of the deformed fluid interface. These four phase-shifted patterns served as the deformed patterns. By varying the particle radius, different groups of deformed patterns could be observed. Table 1 lists the main experimental parameters.

Figure 2. Captured fringe patterns of (a) undistorted liquid/gas interface. The inset represents the intensity of the fringe in transverse direction. The fringe period was calibrated as 0.62 mm. (b) Distorted interface deformed by a floating particle. The circle island located at the center of the images is the PTFE particle with a radius $R = 1.58$ mm. (c) Calculated phase map of $\phi(x,y)$, $\Phi(x,y)$, and $\Delta\Phi_x(x,y)$. The positive and negative values represent the downward and upward menisci. NaN represents not a number.

Table 1. Main Experimental Parameters

R (mm)	ρ_s (kg/m ³) ^a	p (mm)	H (mm)
1, 1.5, 1.58, 2, 2.3	2.3×10^3	0.622	60.00 ± 0.50
n	ρ_L (kg/m ³) ^b	γ_{LV} (N/m) ^c	θ_A (deg) ^d
1.333	1.0×10^3	7.20×10^{-2}	89.7

^aThe density of PTFE particles. ^bThe density of pure water. ^cThe surface tension of the air–water interface. ^dThe contact angle of water wets on PTFE.

As shown in Figure 2b, the circle island located at the center of the images was the spherical PTFE particle. The upper-left corner of the images was defined as the coordinate origin, and the vertical and parallel fringes were identified as the x and y axis, respectively. It is shown that the fringes at the right and left sides next to the solid/liquid/gas contact line are greatly distorted. The larger the distortion, the greater the deviation of the fluid interface from the horizontal plane. It shows that the fringes are attracted by the contact line but, nevertheless, the fringes right above and underneath the contact lines are less distorted. This is due to the fact that the fringe pattern is insensitive to the orientation of the fringes. The phase difference of the four transmission fringe patterns thus can be extracted by phase-shifting, phase-unwrapping, and phase subtraction operations, as shown in Figure 2c. From the perspective of coordinate origin, the right distortion of the fringes indicates a downward meniscus, whereas the left indicates an upward one, revealing that the phase variation has opposite values, as is evident in Figure 2c.

Interface Reconstruction. Once the phase difference $\Delta\Phi_x(x,y)$ had been obtained, reconstruction of the fluid interface was carried out. We adopted a numerical method to solve eq 1 (see the Supporting Information for details). Figure 3a shows the reconstructed air–water interface with respect to that shown in Figure 2. The curve shape of section $abcd$ crossing the center of the contact line was plotted with respect to particle radii $R = 1, 1.5, 1.58$, and 2 mm, as shown in Figure 3b.

Figure 3. (a) Reconstructed topography of a liquid/gas interface with respect to particle radius $R = 1.58$ mm. (b) Section of the fluid interface crossing the center of the particle with respect to particle radius $R = 1, 1.5, 1.58$, and 2 mm.

Force Probing. For a floating object, the classical Archimedes' principle formulates that the buoyancy provided by the hydrostatic pressure is $F_b = \rho_L g V$, where g is the acceleration due to gravity and V is the displaced liquid volume within the solid/liquid/gas contact line and the partly submerged body which is calculated by the reconstructed liquid/gas interface. We found that the calculated buoyancy did not match with the weight of the floating particle, whose

mass is measured by a 1/10 000 g electronic balance. There must be some effects that have been missed by the classical Archimedes' principle.

This effect, which we now know comes from the surface tension of the liquid/gas interface, gives an additional capillary force to the floating object. Using the proposed TPS profilometry technique, the weight of liquid displaced by the meniscus can be measured by $F_c = \rho_L g \iint_S h(x, y) dx dy$, where S is the area of meniscus outside the solid/liquid/gas contact line projected to the horizontal. As shown by the dots in Figure 4a,

Figure 4. Force evaluation of spherical PTFE particles floating at an air/water interface. (a) Comparison of the analytical and experimental results of gravity, buoyancy, and normal capillary forces with a relative error within 5%. (b) Dimensionless supporting forces vs the Bond number.

we measured the buoyancy F_b and the weight of liquid displaced by the meniscus F_c with four particle radii R and found that the balance of forces is achieved from the resultant force of F_b and F_c to counteract the gravitation of the object.

This gave the first direct evidence of experiments that the effect of surface tension, that is, the normal capillary force (projection of the capillary force onto the vertical axis), is equal to the weight of liquid F_c displaced by the meniscus. The normal capillary force is hard to be measured from surface tension, while can be alternatively measured using TPS profilometry from the viewpoint of displaced volume of the meniscus. This force must be included in the generalization of Archimedes' principle. The forces probed in these experiments were in the magnitude of micro-Newtons, which are within the range of capillary actions. Furthermore, it gives hints to the design of a liquid microbalance, which can weigh tiny objects through the displaced meniscus.

As the capillary force is equal to the weight of the liquid displaced by the meniscus, the surface tension can be measured using TPS profilometry by

$$\gamma_{LV} = \frac{\rho_L g}{2\pi r \sin \varphi_r} \iint_S h(x, y) dx dy \quad (2)$$

where r is the contact radius and φ_r is the angle between the horizontal line and the tangent line at the solid/liquid/gas contact point [see Figure 1c]. The approximate φ_r can be obtained by numerical derivation and then arc-tangent operations at the end of the meniscus profile, which is reconstructed using TPS (see Figure S4). In addition, r is obtained by $r = R \sin(\theta_A - \varphi_r)$. In this paper, the surface tension of the air/water interface was measured to be 7.20×10^{-2} N/m at 25 °C.

For a particle with radius R floating at a liquid/gas interface, the buoyancy is

$$F_b = \rho_L g \left[\frac{4}{3} R^3 - \frac{1}{3} R^3 (2 + \cos \alpha)(1 - \cos \alpha)^2 + d \sin^2 \alpha \right] \pi R^2$$

where $[\dots]\pi R^2$ is the displaced volume within the solid/liquid/gas contact line and the submerged body and d is the pressing depth of the contact line.⁴⁰ The normal capillary force is $F_c = \gamma_{LV} 2\pi r \sin \varphi_r$, where $r = R \sin \alpha$ and $\alpha = \theta_A - \varphi_r$ (see Figure 1c). The gravitation is $G = \rho_s g \frac{4}{3} \pi R^3$. Considering the force balance of the sphere, that is, $Mg = F_b + F_c$, we obtained the mapping relations between φ_r and R (see Supporting Information Figure S4). Thereafter, the supporting forces F_b and F_c are the functions of radius of sphere R . Therefore, the analytical buoyancy, normal capillary force, and gravitation of the floating particles versus radius are plotted in Figure 4a, revealing that the analytical solutions were in good agreement with the experimental results. The relative error is within 5%, better than the tiny force measurement obtained using light shadow.⁴¹

Dominant Regimes of Floating Particles. Both the normal capillary force and buoyancy increase with the increase of radius, of which the former quickly increases and then slows. F_c is larger than F_b at small radii until a critical radius of $R \approx 2.11$ mm. After that, it sharply drops to zero with a slight increase of radius ($R > 2.24$ mm, approaching to the capillary length of water $l_c = 2.71$ mm).

For floating versus sinking, it is generally believed that an object with a density greater than that of the liquid will float not only due to surface tension but also due to the characteristic length of the object $L \ll l_c$ ($l_c = \sqrt{\gamma_{LV}/\rho_L g}$ is the capillary length), that is, the Bond number $Bo = (L/l_c)^2 \ll 1$.⁴² Figure 4b shows the dimensionless supporting forces, $\mathcal{F}_b = F_b/Mg$ and $\mathcal{F}_c = F_c/Mg$ (M is the mass of the objects) versus the Bond number. As the Bond number increases, the dimensionless normal capillary force $\mathcal{F}_c$ decreases, whereas the dimensionless buoyancy $\mathcal{F}_b$ increases. The former plays a major role for small Bond numbers of $0 < Bo \leq 0.6$ and the latter dominates for $0.6 < Bo \leq 0.67$. The variation of the dimensionless forces is nonlinear, indicating that there is a complex interplay between them. For a Bond number of $Bo > 0.67$, the air/water interface is unable to support the particle and hence it sinks. This shows that a spherical particle can only float at a liquid/gas interface with a small Bond number due to surface tension. This may explain why the legs of semiaquatic insects living at air–water interfaces have not evolved into a spherical shape.

CONCLUSIONS

We report an optical profilometry technique applying TPS for the first time to accurately probe micro-Newton forces at a liquid/gas interface. This technique, which is mainly based on the phase variation of transmission fringes, can visualize the liquid/gas interface in three dimensions. Using this approach, the mechanics of floating particles with different radii have been discussed. The competition of the forces, that is, the normal capillary force, buoyancy, and gravitation, are decoupled, indicating that the small particles can only float at a fluid interface if the Bond number is small. In particular, we gave the first experimental demonstration of a generalization of Archimedes' principle, which includes the effect of surface tension. We envision that this new methodology can be applied to exploring a wide range of interfacial phenomena and capillary interactions, such as the hydrodynamics of water-walking, dynamics of wetting and spreading, issues in capillary waves, elastocapillary problems, and so forth.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: [10.1021/acs.langmuir.8b03922](https://doi.org/10.1021/acs.langmuir.8b03922).

Equation derivation process and detailed measuring process (PDF)

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Author Contributions

Z.L. designed the research; Z.L. and Y.P.Z. supervised the research; X.H., H.D. performed the experiments; X.H. and Z.L. analyzed the data and wrote the paper, and X.H. and H.D. contributed equally.

Notes

The authors declare no competing financial interest.

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