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Historical perspective

Acoustic levitation of liquid drops: Dynamics, manipulation and phase transitions

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ABSTRACT

The technique of acoustic levitation normally produces a standing wave and the potential well of the sound field can be used to trap small objects. Since no solid surface is involved it has been widely applied for the study of fluid physics, nucleation, bio/chemical processes, and various forms of soft matter. In this article, we survey the works on drop dynamics in acoustic levitation, focus on how the dynamic behavior is related to the rheological properties and discuss the possibility to develop a novel rheometer based on this technique. We review the methods and applications of acoustic levitation for the manipulation of both liquid and solid samples and emphasize the important progress made in the study of phase transitions and bio-chemical analysis. We also highlight the possible open areas for future research.

Contents

1. Introduction

Foreign objects, including liquid drops, gas bubbles, solid particles etc., in a fluid can experience a steady time-averaged force when irradiated by a sound field, which is referred to as acoustic radiation force [1]. The intriguing phenomenon of dust particles forming a ring-like pattern in a glass tube, under the effect of a standing sound wave was reported by Kundt et al. in 1866 [2]. Afterwards, to understand the phenomenon, the acoustic radiation force on small particles, say, $r \leq \lambda$ (r is the radius of particle and λ is the sound wavelength), has been extensively studied since 1930s. In 1934, King proposed the

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acoustic radiation force for spherical incompressible particles in acoustic fields [3]. In 1962, Gor'kov developed a more general theory based on the acoustic force potential U_{ac} suitable to compressible particles, which can be written as [4].

$$U_{ac} = \frac{4\pi}{3} r^3 \left[f_1 \frac{1}{2\rho_M c_M^2} <p^2> - f_2 \frac{3}{4} \rho_M <v^2> \right] \tag{1}$$

$$f_1 = 1 - \frac{k_P}{k_M} \tag{2}$$

$$f_2 = \frac{2\left(\rho_p - \rho_M\right)}{\left(2\rho_p + \rho_M\right)} \tag{3}$$

where p and v are the sound pressure and particle velocity, c_M is sound velocity in the medium, k and ρ are compressibility and density, the subscripts M and P represent "medium" and "particle", respectively. Eqs. (1)–(3) built the relation between the acoustic radiation force, the sound field and the properties of the system, which also suggest that the acoustic force is sensitive to the compressibility difference between the particle and medium. Due to the elimination of the difference between k_p and k_m, the acoustic levitation in liquid medium is more challenging, though manipulation of micro-sized particles using MHz sound wave in acoustofluidic systems is possible [5]. It is well known that, acoustofluidics is already strongly impacting the *soft matter* field, ranging from particle aggregation studies [6] to 3D manipulation [7]. Here, we mainly study the acoustic levitation in air. We do not put emphasis on MHz acoustic resonator at the moment because it can only obtain micro-scale particle manipulation [8], but focus on the levitation which works at 20 k ~ 100 k Hz using power ultrasound [9].

1.1. Single axis levitator

For the acoustic levitation in air, the potential U_{ac} can be simplified as

$$U_{ac} = 2\pi r^3 \left[\frac{<p^2>}{3\rho_0 c_0} - \frac{\rho <v^2>}{2} \right] \tag{4}$$

where, ρ_0 is the density of air and c_0 is the sound velocity in air. The simplest and most popularly used acoustic levitator is usually called the 'single-axis levitator' which consists of a transducer and a reflector [10], as illustrated in Fig. 1(a). The samples, either solid or liquid, can be levitated at the pressure nodes of the sound field, each of which is separated by a distance of $\lambda/2$ [11]. It should be noted that it may exhibit different levitation performance at different pressure nodes. For the levitation of a liquid droplet, the goal is not simply to balance gravity. A force balance at the droplet surface must be obtained. The acoustic radiation pressure on the droplet is not uniform, usually positive (compression) at the polar area while negative (suction) at the equator, as illustrated in Fig. 1(b). The droplet will, in turn, adjust its surface curvature to adapt the radiation pressure. Meanwhile, the Bernoulli effect arising from the acoustic streaming may alter the force balance on the levitated sample and bring additional instability. To enhance the levitation ability and stability, the reflector [12] or both reflector and emitter [13] were often made concave. In general, there is a size limit of ~$\lambda/2$ for the sample that can be stably levitated which is determined by the maximum size of the potential well of single acoustic levitator. The typical sample size for levitation is around $\lambda/4$ ~ $\lambda/3$, which has been reported in previous experiments [14].

1.2. General applications of acoustic levitation

Since contamination from the container walls can be avoided and considering experimental simplicity, acoustic levitation has a wide range of applications in drop dynamics [12], biology [16,17], analytical chemistry [18–20], solidification [21] and pharmacy [22]. The main advantages of acoustic levitation over other levitation techniques, such as optical tweezers [23] both electrostatic [24] and electromagnetic, is that transparency and electro-magnetic properties of the levitated materials [25] are not required, which make it suitable for a broader range of materials. It should be pointed out that acoustic levitation is excellently amenable to soft matter, including complex drops, bubbles, foams, emulsion etc. Due to their softness, they can easily adapt their shape to the sound field, leading to good levitation stability. On the other hand, the mechanical or rheological properties of the levitating sample are reflected from its response to the sound field [26,27]. This is desirable for the study of viscoelasticity and relaxation of soft matter.

The unique environment provided by acoustic levitation, i.e., container-free condition and droplet internal flow, may result in novel or unusual physical/chemical effects. For instance colliding and mixing of acoustically levitated droplets gives a homogeneous system in 3–4 s [28], as illustrated in Fig. 2. The mixing was accelerated by at least an order of magnitude over that of three-dimensional diffusion, Moreover, by combining with other remote spectrum techniques [29], acoustic levitation provides an ideal platform to study the physical/chemical processes in the soft matter systems in contact-free condition, which is extremely beneficial to elucidate the effect of solid walls on these processes.

As compared with use of optical tweezers which can only manipulate small particles from tens of nanometers to tens of micrometers and is adept to the measurement of thermal-scale forces and weak interaction [23], acoustic levitation can produce stronger force trapping in larger spatial scale, therefore is competent to manipulate millimeter-sized drops. The working frequency of this technique is normally around tens to hundreds kHz, far below the frequency of Brillouin

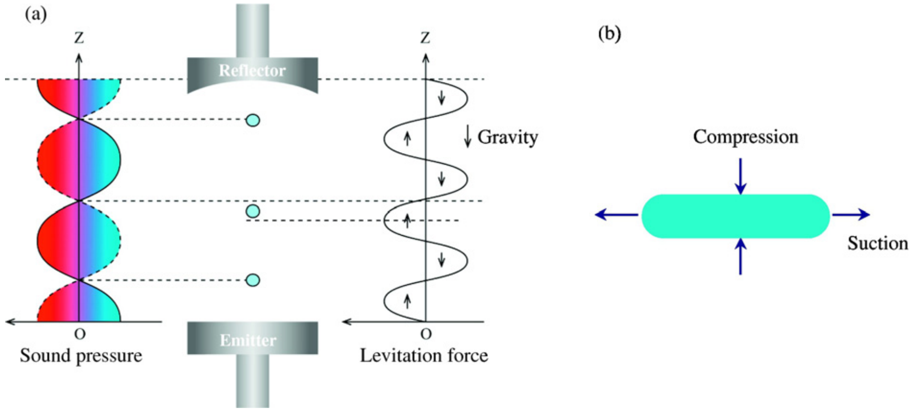

Fig. 1. (a) Schematic view showing the principle of acoustic levitation of a single-axis levitator. The samples can be levitated at the pressure nodes. (b) Illustration of acoustic radiation pressure distribution on a levitated liquid droplet. The arrows indicate the direction of force caused by acoustic radiation.

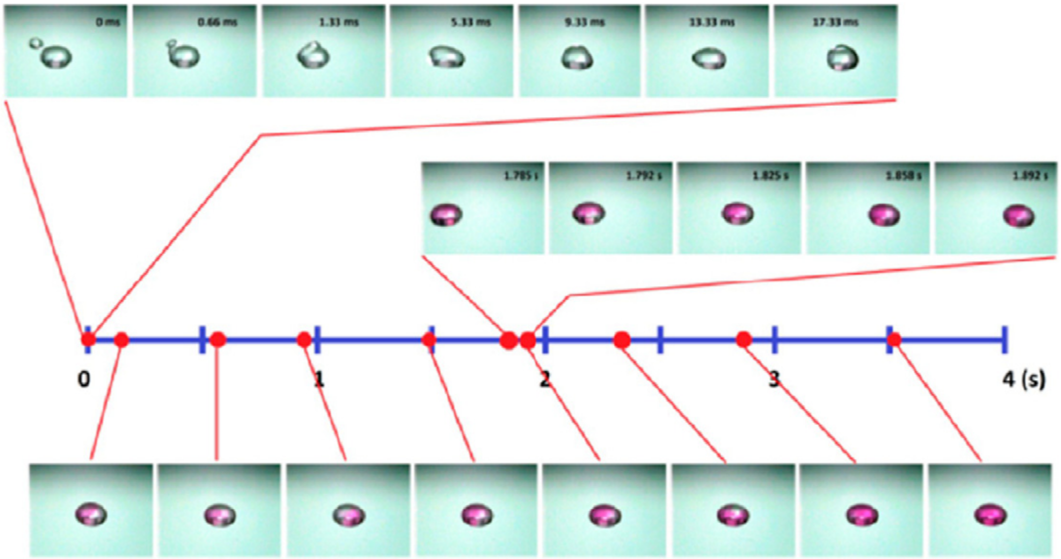

Fig. 2. Colliding and mixing of acoustically levitated droplets which show a much faster mixing. Volumes of smaller and larger droplets are 370 nL and 3 μL respectively. The spherical diameter of the mixed droplet is ~0.93 mm.
Reprinted with permission from ref. [28].

scattering technique which characterizes the acoustic properties of condensed matter in GHz range by extracting information from Brillouin shift and line width [30,31].

2. Drop dynamics

2.1. Oscillation of acoustically levitated drops

Drop dynamics upon impacting or vibrating a drop on solid surfaces has been extensively investigated [32–37]. However, in such situation the drop-substrate interactions cannot be neglected due to the effect of contact line friction [33–35]. Fig. 3(a) shows the interfacial wave patterns of a sessile drop caused by forced vibration with increasing substrate acceleration [37]. To research the free oscillation dynamics of drops, experiments under microgravity condition, i.e. in the space station, provide an efficient approach [38]. Because of the high cost of space experiments, it is necessary to design alternative experiments at ground level. Acoustic levitation, owing to the freedom from solid walls and the possibility of active stimulus, becomes one of the most convenient techniques to study drop dynamics. During levitation, the gravity of the levitated drop is balanced by the acoustic radiation force exerted on the drop surface, which consequently results in an internal pressure on the drop in addition to liquid surface tension. The competition between the internal pressure and Bernoulli pressure caused by sound inevitably leads to radial oscillation [39]. With further modulation of the transducer signals, the original radial oscillation can be amplified or even developed to complex sectorial oscillation of a high mode up to seven [15], as shown in Fig. 3(b). Recently it was reported that the eighth mode of oscillation can also be accomplished [40]. The sectorial oscillation in acoustic levitation, which is obviously different from the interfacial wave patterns obtained from the case of sessile drops, relates to the drop surface tension and the coupling with drop internal flow [41], however, the details of the mechanism still remains unclear.

It has been reported that, in the single-axis levitator, solid objects exhibit spontaneous oscillation in both the radial and axial directions [42]. The oscillation frequency was well explained by a simple model based on a harmonic oscillator. On the other hand, acoustically levitated liquid

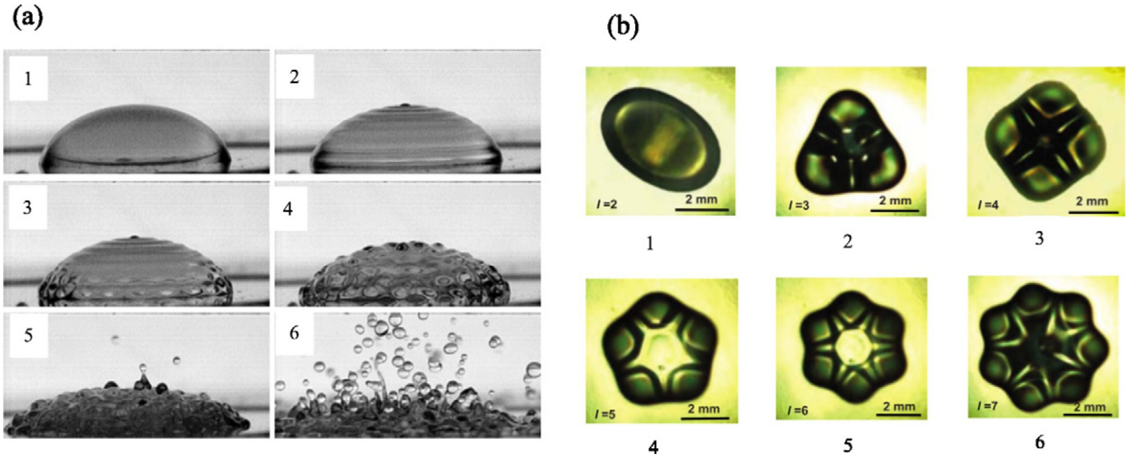

Fig. 3. (a) Dynamics of sessile drops (100 μL) under forced vibration with increasing substrate acceleration. (1) Unforced, (2) axisymmetric waves, (3) coupling of axisymmetric and azimuthal waves, (4) pre-ejection state, (5) ejection, and (6) atomization. Reprinted with permission from ref. [37]. (b) Sectorial oscillations of water drop stimulated by an active modulation of the power input of a single-axis levitator. (1)–(6) correspond to the modes 2 to 7 of the oscillation which was regulated by the modulation frequency. Reprinted with permission from ref. [15].

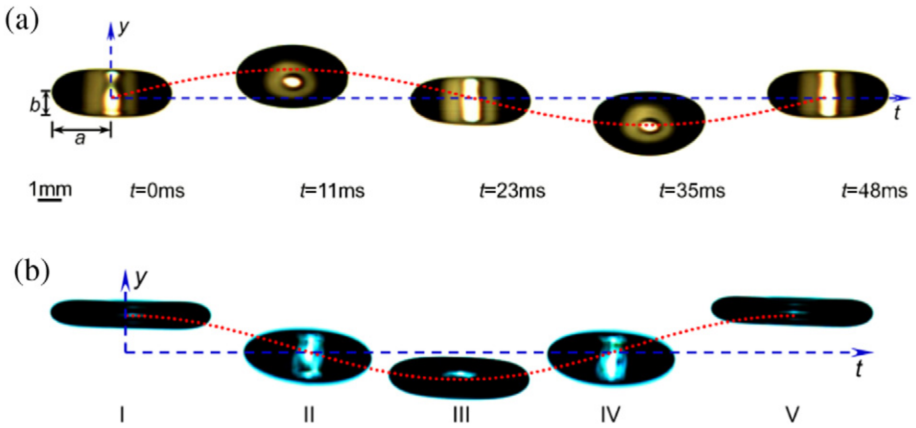

Fig. 4. Vertical vibration of acoustically levitated water drops. (a) Without shape oscillation, and (b) with shape oscillation. Reprinted with permission from ref. [43].

drops can undergo a vertical vibration if it is deviated from its equilibrium levitation position artificially, which also shows a harmonic mode [43], as illustrated in Fig. 4(a). In this case, the sum of acoustic radiation force and gravity acts as the restoring force. Under stronger sound intensity, the vertical vibration of the liquid drop often couples with its shape oscillation (Fig. 4(b)). The vertical oscillation can probably be damped via the acoustic streaming in the sound field [44].

2.2. Instability behaviors

In addition to drop oscillation dynamics, acoustic levitation also serves as an effective tool for the investigation of fluid instability because the levitated drops can exhibit many types of instability behaviors, including atomization [45], breakup [46] and buckling instability [39]. The drop atomization can be induced by the growing capillary waves arising from the radial or translational oscillation, as shown in Fig. 5(a). The strong acoustic streaming often causes Kelvin-Helmholtz instability at the edge of drops [47]. The buckling instability of acoustically levitated drops is characterized by the buckling of the liquid membrane and abrupt area expansion, which was first reported by Lee et al. [39]. This phenomenon could be triggered by increasing sound intensity or by external heating via laser as reported recently [48]. This may possibly be attributable to the enhanced suction effect at the membrane edge, which was reflected by the stretched shape of an oil film affixed with a water droplet [44], as illustrated in Fig. 5(b).

2.3. Surface rheology

The surface tension of the levitated drop can be obtained from its static shape determined by the balance of surface tension and acoustic radiation force [49]. However, this requires accurate extraction of the drop profile and complex analysis based on Laplace equation [50]. Alternatively, surface tension can also be derived based on drop oscillation dynamics through analyzing the natural frequencies [26] or fitting a modified Rayleigh model to the sectorial oscillation [51]. The obtained data were in good agreement with those reported in refs. [52–54], as shown in Fig. 6(a). The dynamics of acoustically levitated drops also contains the information of its rheological properties [55–57]. The viscosity can be obtained from the decay of sectorial oscillation based on Lamb's theory [51]. Recently, the interfacial dilational modulus of a levitated liquid marble has been investigated by taking the surface area dependence of surface tension into consideration [27], as illustrated in Fig. 6(b). Compared with the traditional Wilhelmy plate method, the acoustic levitation method is capable of investigating the compression moduli for the particle layer after the buckling transition [27].

3. Drop manipulation

Although superhydrophobic surfaces [58] and microfluidic devices can facilitate drop manipulation for bio-chemical reaction or analysis,

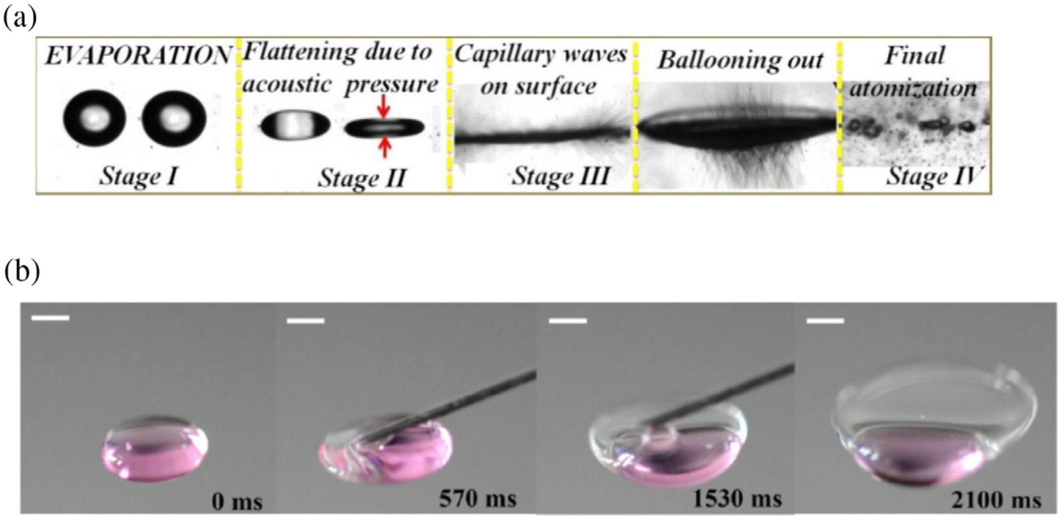

Fig. 5. (a) Different instability behaviors of acoustically levitated drops from flattening to capillary wave and atomization., Reprinted with permission from ref. [48]. (b) Formation of an abnormal shaped drop characterized by a stretched oil film affixed with a water droplet. Reprinted with permission from ref. [44].

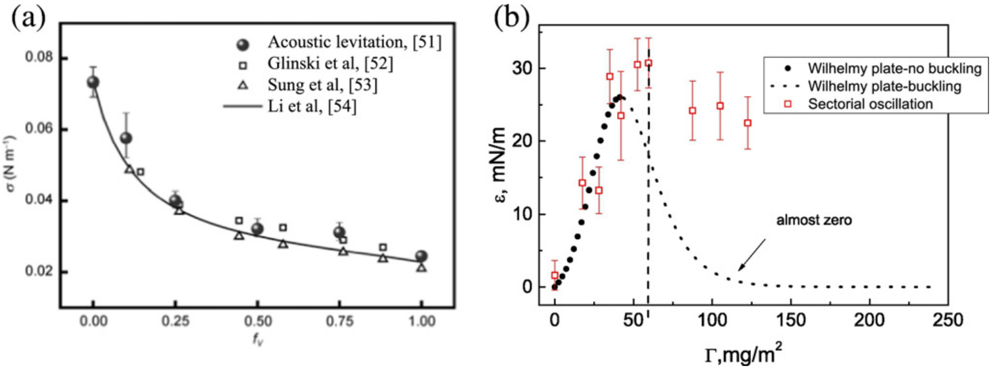

Fig. 6. Measurement of surface tension and interfacial compression modulus via acoustic levitation method. (a) Surface tension of ethanol aqueous solution versus volume fraction of ethanol f_v. Reprinted with permission from ref. [51]. (b) Interfacial compression modulus versus surface concentration Γ. Reprinted with permission from ref. [27].

these methods show an intrinsic disadvantage that liquid may form a residue on the surfaces of solid substrates or channels [17]. Compared with the lab-on-a-chip techniques where the drops are manipulated in tiny channels, acoustic levitation has prevented the contamination and confinement of solid walls, therefore, providing a cleaner environment and smart manipulation of drops containing chemical or bio-

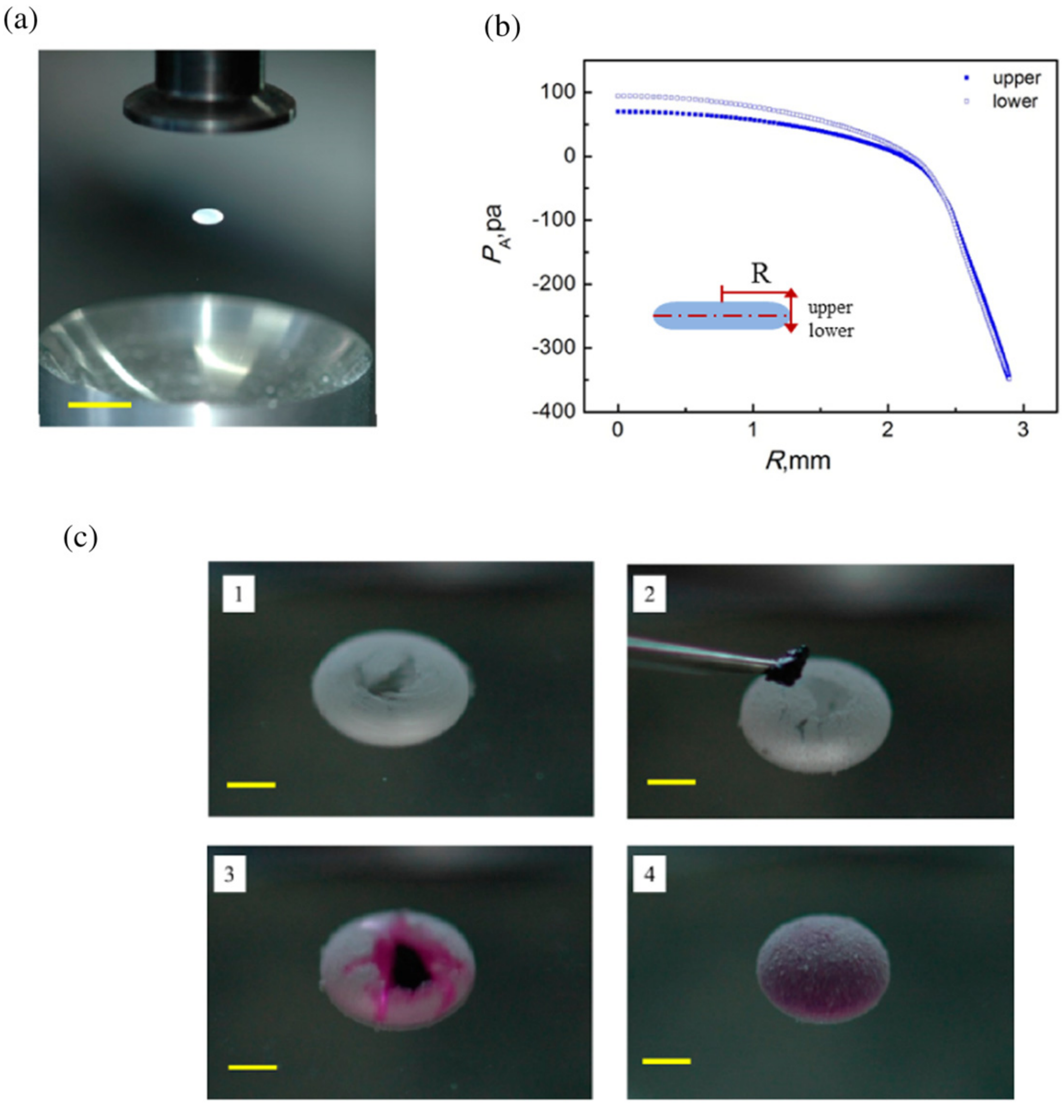

Fig. 7. Opening/closing switch of liquid marble via acoustic levitation. (a) Acoustically levitated liquid marble coated with Teflon particles (5 μm). (b) Acoustic radiation pressure distribution on drop surface showing that the drop was under compression at polar regions and suffered suction at the equator. (c) The particle layer coating on the liquid marble is opened and chemicals can then be introduced into the inner liquid. Afterwards, the hole on the liquid marble heals and recovers to its initial state.
Reprinted with permission from ref. [61].

materials. Furthermore, since optical scattering and refraction of the container are avoided, drop levitation has the advantages of in-situ observation and analysis by Raman spectroscopy or X-ray diffraction [29,59].

Since the levitated samples are trapped at the potential wells of the sound field, adjusting the position and shape of the potential well can generally result in sample manipulation. A facile approach has been developed by Andrade et al. [60] where the particle manipulation can be achieved by a non-resonant single-axis acoustic levitator simply through adjusting the position of the reflector while keep the transducer fixed. Through increasing or decreasing the sound intensity of the levitator, the surface of a particle coated drop (liquid marble) levitated in the sound field can be opened or closed in a controlled manner [61], as illustrated in Fig. 7. Poulikakos et al. [62] have proposed a new 'acoustophoretic' concept based on the spatiotemporal modulation of the levitation acoustic field, which allows continuous planar transport and processing of multiple objectives, and even orbital and spinning of samples in air [63]. These promising progresses in acoustic levitation enable three dimensional manipulations of millimeter-scaled objectives, which is highly desired in droplet coalescence, materials preparation, chemical and biological reactions.

Marzo et al. [64] have developed a technique using single-sided acoustic transducer arrays to simultaneously accomplish 3D acoustic trapping, translation and rotation of millimeter sized particles, as illustrated in Fig. 8. By optimally adjusting the phase delays of the transducers, different shapes of acoustic structures: tweezers, twisters or bottles, have been obtained which enhance the particle trapping and manipulation. Particularly, the one side geometry potentially enables in vivo manipulation.

Although in this work we focus on the acoustic levitation in air, it should be noted that acoustic tweezers, which normally can only be applied in a liquid medium, because MHz sound attenuates seriously in gas medium, also has potential to be applied in soft matter. Though not applicable to the demand of airborne chemistry [20], it is however, suitable for manipulation of micro sized objectives in liquid media. This enables us to perform in-situ study of cells in their culture liquid, for instance deforming a red blood cell [65] or even accomplishing 3D manipulation of single cells [66].

4. Phase transitions in acoustically levitated drops

4.1. Drop evaporation

It has been reported that solid surfaces can provide heterogeneous nucleation sites for phase transition depending on its wettability to the newly formed phase [67]. Since mechanical contact is completely avoided, i.e., all the heterogeneous nucleation effects are removed, the acoustically levitated drop becomes an ideal system for the study of undercooling of liquids and the phase transition/reaction therein.

Drop evaporation can be easily incorporated with acoustic levitation. Because droplet drying can lead to a wide range of materials, which is accompanied by sol-gel transitions, particle aggregation or crystallization, acoustic levitation is well suited to study these processes. The levitation facilitates the monitoring of evaporation kinetics of single drops [68,69], which has been applied to both single phase droplet [70], and binary or multiphase droplet [71,72]. The drying patterns of colloidal droplets depend on the particle-particle interactions, and are significantly affected by the substrates as well [73]. With the increase of substrate contact angle, the final residue pattern can be coffee ring-like stains (Fig. 9(a)) on hydrophilic substrate [74] and bowl-shaped residue (Fig. 9(b)) on a hydrophobic substrate ($\theta \sim 90°$) [73]. Under acoustic levitation, a ring-shaped residue has been obtained (Fig. 9(c)) [75], which exhibits a geometrical similarity with the initial dog-bone shape of the acoustically levitated droplet [39]. These results highlight the important role played by the substrate in drying pattern formation, and acoustic levitation provides the possibility to study drop evaporation at zero-contact condition and for different initial shapes. But it should be noted that the acoustic streaming, often inevitable during ultrasound levitation [76], may play a role in both the evaporation rate and the temperature gradient on the levitated droplet surface [71]. Under appropriate condition, the streaming can lead to the solidification of a levitated cyclohexane drop due to enhanced evaporation [77].

Crystallizations in the acoustically levitated drops often show notable differences compared to that in a container. For instance the amorphous liquid-like precursor has been observed in the crystallization of $CaCO_3$ salt due to the homogeneous precipitation resulting from the absence of solid walls [78]. The NaCl crystal obtained from acoustic levitation can be three times larger and show a much higher growth rate

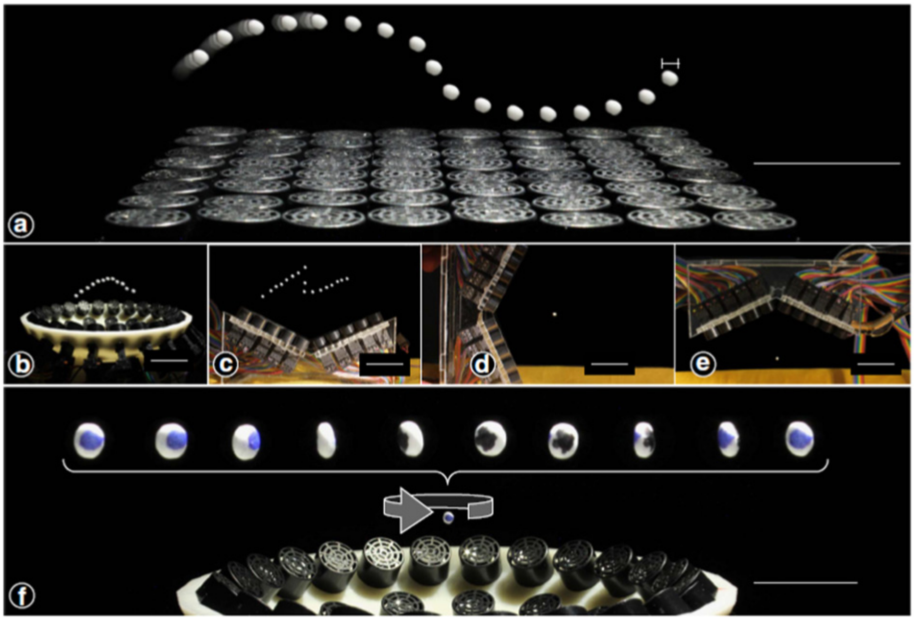

Fig. 8. Particle manipulation using single-sided arrays of acoustic transducers (40 k Hz). (a–c) The particles can be transported in 3 dimensions using different arrangements of the array without moving it. (c–e) The traps are strong enough to counteract gravity from any direction. (f) Asymmetric objects can be driven to rotate at up to 128 rpm.
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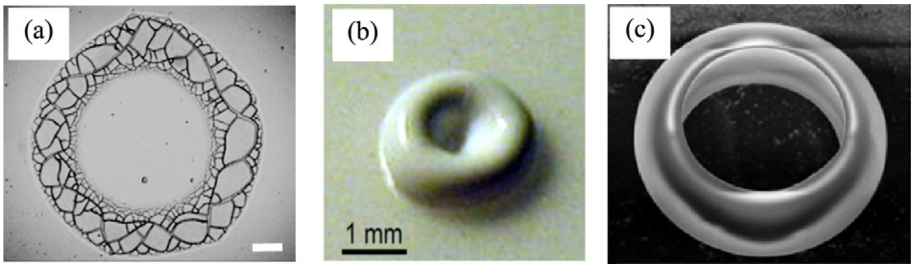

Fig. 9. Different drying patterns obtained from varied conditions. (a) Coffee ring-like stain on hydrophilic substrate, Reprinted with permission from ref. [68]. (b) bowl-shaped relic on hydrophobic surface, Reprinted with permission from ref. [67]. (c) Ring-shaped residue obtained from acoustic levitation. Reprinted with permission from ref. [75].

compared to that obtained in a vessel [79], as illustrated in Fig. 10. The authors also found similar results in crystallization of NH_4Cl and hen egg white lysozyme, and concluded that acoustic levitation shows positive effect for crystallization of both inorganic salts and proteins [79].

4.2. Combination with other techniques

Because of both the miniaturization and easy adaptation with other remote detection systems, such as fluorescence imaging, Raman spectroscopy, and X-ray diffraction, acoustic levitation can be widely used in bio-chemical reactions and analysis [16]. The container-free effect results in supersaturation of solutions, therefore leading to the formation of glass state pharmaceuticals [22] and high quality protein crystals [80] as well as unique self-assembly nanostructures of lipid or surfactant [81]. The structures in these processes can be in-situ observed via X-ray diffraction.

Recently, it has been reported that acoustic levitation can provide an alternative approach for the study of surfactant monolayer at the air-water interface [82], which takes the advantages of the traditional Langmuir trough by the reduction in materials consumption, and in preparation and observation time. Complex droplets, such as immiscible two phase droplets, can be stably levitated in the sound field, which provides a possibility to investigate the diffusion and phase separation in the drops [44,83]. This method can also be applied to bio-specific affinity partitioning [84]. Another important application of acoustic levitation is the processing and treatment of bio-matter, such as single cell experiments [85], Zebrafish embryos [86] and even small living animals [87].

5. Concluding remarks

Acoustic levitation in principle generates a standing sound wave which brings acoustic radiation force to the sample levitated in it. Compared with other levitation techniques, acoustic levitation does not require the electro/magnetic properties of the sample. Most importantly, this technique is gentle, by contrast to optical levitation; therefore it does not ruin the sample, and is safe even for biological specimens. The levitation stability is satisfactory with appropriate reflector geometry or using flexible/soft reflectors [88], which can be well maintained through adjusting the sound intensity according to the shape/volume change of the levitated droplet. Noteworthy, the vibration caused by sample deviation from equilibrium position can be damped via acoustic streaming [44]. The levitation may become unstable if the droplet shape change significantly and can not adapt itself to sound field due to the formation of solid crust for the drying of colloidal droplet. In this case, however, using arranged transducers in an optimized-designed pattern with accurate programmable phase modulation possibly can further enhance the levitation stability, which may also shed light on the potential to study the mechanical properties of soft matter via the levitation technique.

The special bio/chemical effects of acoustic levitation mainly arise from avoiding of solid wall contact; therefore high undercooling or supersaturation of liquids is expected. In addition, the sound field may generate unique acoustic radiation pressure distribution on the sample surface. The acoustic streaming may also induce a flow boundary outside the levitated sample [76] or influence the internal flow inside it [89]. These could also have effect on the physical/chemical processes. Moreover, it only consume tiny amount of materials in the levitation experiments which enable the levitated droplets to serve as very economic and efficient micro-reactors. Owing to the sufficient space of the levitator, determined by the sound wavelength, it is convenient to incorporate other remote detection instruments. This greatly facilitates the in-situ studies of the processes involved.

For the soft matter community, acoustic levitation is a high-performance technique which is not only perfectly adequate for sample handling but also shows promising applications in the study of surface

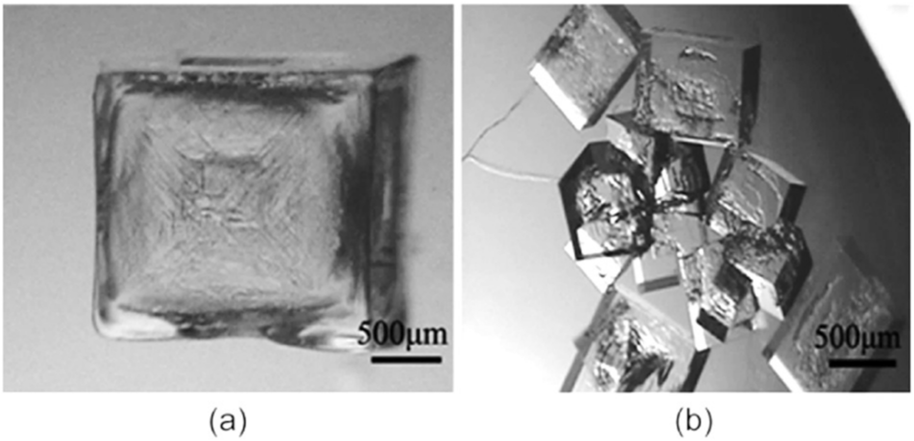

Fig. 10. NaCl crystal growing from different conditions: (a) acoustic levitation, and (b) in a vessel. Reprinted with permission from ref. [79].

tension, rheological properties (both bulk and interfacial) of the soft samples based on the coupling between drop dynamics and sound field. An input stimulus can be easily generated by tuning the power intensity or adjusting the emitter-reflector distance driven by a servo motor. The response of the levitated drop can be in-situ recorded via camera video. The viscosity of the system then can be extracted from the phase difference between stimulus and response. Alternatively, oscillation decay dynamics of drops after a sudden deformation contains the information of viscosity which can be easily monitored by acoustic levitation as well.

For the measurement of surface tension, acoustic levitation also provides a choice of menu, namely based on static equilibrium drop shape [50], oscillation dynamics [26] and unique instability behavior. Because the interfacial rheology is related to the area/concentration variation of surface tension, it is possible to obtain the rheological properties by combining the surface tension measurement and drop surface area analysis as the oscillating drop/bubble technique does [90,91], but completely eliminates the contact between measurement probe and sample. This suggests that both the sound field and its coupling with drop shape should be well understood, through numerical simulation and image analysis. From the experimental point of view, precise control of the sound field is required by either designing effective transducer patterns associate with accurate phase control or combining with servo motors or other external field stimuli. For some cases, a robotic system is necessary to enhance the sample positioning accuracy. Importantly, the theory which can interpret the relation between the drop dynamics, sound field and the rheology should be built. In this context, both experimental and theoretical works are expected to devote into this field.

Acoustic levitation provides a container-free condition, which is helpful to identify the effect of solid walls. This is of essential importance for the study of soft matters where the liquid-solid interfaces play an important role. The contact-free condition may greatly affect a wide range of physical-chemical processes, such as sol-gel transition, self-assembly, materials synthetics, and cell-cell interactions. However, the physics behind the process is not fully understood yet. Researches on these topics under acoustic levitation would obtain deeper insight into the nucleation, aggregation and dynamics in the systems, as well as the soft matter-ultrasound interactions.

Acknowledgements

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