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Janus particle microshuttle: 1D directional self-propulsion modulated by AC electrical field

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A catalytic Janus particle is capable of gaining energy from the surrounding fuel solution to drive itself to move continuously, which has an important impact in different fields, especially the field of micro-systems. However, the randomness of self-propulsion at the microscale restricts its use in practice. Achieving a directed self-propelled movement would greatly promote the application of the Janus particle. We proved experimentally that an AC electric field was an effective way to suppress Brownian motion and control the direction of self-propelled movement. The self-propulsion and dielectrophoretic response of a 2μm Janus particle were observed and the related basic data were collected. Interdigital electrodes, 20 μm in width, were energized in pulsed style to modulate the self-propulsion, which resulted in a shuttle-style motion in which a single Janus particle moved to and fro inside the strip electrode. The change of direction depends on its unique position: the catalyst side is always pointed outward and the orientation angle relative to the electrode is about 60°. Numerical simulation also proved that this position is reasonable. The present study could be beneficial with regard to self-propulsion and AC electrokinetics of the Janus particle. © *2014 Author(s). All article content, except where otherwise noted, is licensed under a Creative Commons Attribution 3.0 Unported License.* [http://dx.doi.org/10.1063/1.4868373]

I. INTRODUCTION

The Janus particle, proposed by de Gennes in 1991, has a non-homogeneous structure on its two hemispheres, which enables some novel physical and chemical properties to be induced.[1] At present, the Janus particle has been fabricated by different methods including microfluidics, self-assembly, sputtering, and so on.[2,3] Among all applications, a very interesting example is self-propulsion (Fig. 1(a)), where one hemisphere of the Janus particle covered with a thin layer of catalyst (e.g. platinum (Pt)) serves to decompose the fuel reactants (e.g. $2H_2O_2 \rightarrow 2H_2O + O_2$) surrounding the particle.[4] Because the catalytic reaction generates asymmetry of the molecule number at the two sides of the Janus particle, the concentration gradient of molecules will force the Janus particle to move autonomously,[5] with the direction opposite to the catalyst side. Considering this compact structure and the fact that an additional battery is not required, self-propulsion of the Janus particle has attracted widespread interest in fields such as MEMS, self-assembly, and sensors, so far.[2,3]

For microsized Janus particles, the self-propulsion exhibits very complex behaviours and the related mechanism has been studied thoroughly. Commonly, it was believed that the movement of Janus microparticles is due to the competition between two different motions: random Brownian motion and self-propellant motion.[6] This results in inconvenience when one wants to control the direction of motion of Janus microparticles, such as in targeted drug delivery. Hence, artificial intervention has to be introduced for the self-propulsion of Janus particles. The key to generating an expected directional movement is to overcome the random rotation from Brownian motion.

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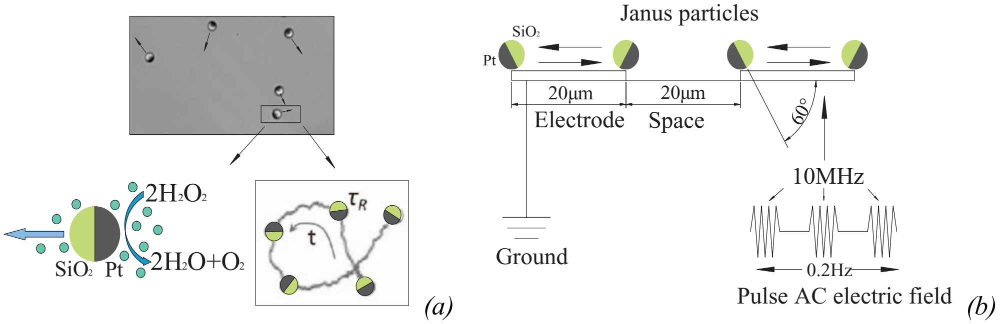

FIG. 1. (a) Schematic diagram of Janus particle's self-propulsion. (b) Microshuttle of Janus particle above the electrode under a pulsed AC electric field.

In 2012, Baraban *et al.*[7] published their results on how to realize directed self-propulsion of Janus particles in a magnetic manner. An additional magnetic cap was coated between the catalytic metal layer and SiO_2 particles, and an external magnetic field was used to direct the movement of the Janus particles. The strength of the magnetic field was very weak, so it served only to suppress the random rotation rather than driving the particles to move. Long range 1D directional motion was realized under a fixed magnetic field, and the flexible 2D planar motion of Janus particles was carried out by further adjusting the applied magnetic field in time. In principle, more complicated functions including transport, manipulation, and separation of Janus particles could be achieved too. The magnetic field method was also found in the case of using non-spherical Janus particles or other micro-devices.[8] In addition to the magnetic field, a light-driven method and a DC electrical field were also available to direct the self-propulsion of Janus particles.[9–11]

In this paper, we present a novel design utilizing an AC electrical field to control the orientation of the Janus particles and further to realize a controllable shuttle-style self-propulsion (Fig. 1(b)). In Section II A, the experimental procedure is introduced briefly. In Section II B, we show the stochastic characteristics using the effective diffusion coefficient D_{eff} and the probability distribution of displacement rotation angleγ. Next, in Section II C, we examine the dielectrophoresis (DEP) of the whole Janus particles under a high frequency AC electric field and determine the orientation angle of the Janus particle as a two-hemisphere structure. Based on the characteristics from Section II B and the manipulation manner described in Section II C, we put forward a strategy in Section II C in which by switching the AC electric field "on" and "off" with a certain frequency we can eliminate the negative effect of random motion and direct the Janus particles to move. Finally, the conclusions are drawn in Section III and information about the fabrication and materials is provided in the Appendix.

II. RESULTS AND THEORY

A. Experiment

The experiment was conducted on a MicroPIV platform (LNM, Institute of Mechanics, CAS). The trajectories of particles immersed into H_2O_2 solution were observed by video microscopy with an image field of view of 512 × 512 pixels. Then, the test solution was prepared by mixing the Janus particle aqueous solution and the hydrogen peroxide solution to give a certain H_2O_2 concentration. After the preparation of the solution, a 70 μL droplet was put on the glass slides. A CCD camera (Andor 897) was used to record videos. Image series consisting of 600–1000 frames were captured in one position located about 2 μm above the glass substrate. The particle positions (x, y) can be tracked by the software video spot tracker (V07.02). Then, a high frequency AC electric field generated by indium tin oxide (ITO) interdigital electrodes (IDEs) was applied. ITO material provides high transparency and good electrical conductivity, enabling visualization of the movement of particles by an inverted microscope. The electrode pad accesses the signal generator to load the pulsed signal. Similar observations were carried out to record the motion of Janus particles affected by the DEP force.

B. Stochastic characteristics of self-propulsion of Janus particle

A Pt-SiO_2 type Janus microsphere is a common experimental model used to carry out the study of self-propulsion phenomena and was adopted in most of the works in the literature.[4] Its non-uniform surface structure consists of one dielectric silica hemisphere and one Pt-coated hemisphere. When Pt-SiO_2 microspheres were dispensed into hydrogen peroxide solution, a catalytic reaction ($2H_2O_2 \rightarrow 2H_2O + O_2$) happened on the Pt surface, where the molecular number ratio of reactant and product is 2:3. Obviously, the overall number of molecules in solution is increased and the distribution becomes uneven at the same time. At the Pt side, there are more product molecules and the concentration is higher than at the opposite SiO_2 side. According to the viewpoint of thermal dynamics, Brownian motion is the result of random collision of small molecules with the particle surface. The more molecules collide, the higher the resultant random force is. Therefore, the Janus particle will be pushed away from the Pt-coated hemisphere.

Brownian motion originates from random collisions of surrounded liquid molecules. This process is equivalent to applying a random Brownian force on a particle. The expression of Brownian force is given by,[12]

$$F_{Brownian} = \xi\sqrt{\frac{12\pi k_B T \mu R_p}{\Delta t}} \tag{1}$$

Here, k_B is the Boltzmann constant, T is the thermodynamic temperature, μ is dynamic viscosity, R_p is the radius of the particle, Δt is the time interval, and ξ is a random number. Under the action of Brownian force, the particle will give rise to random translation motion. Furthermore, considering that Brownian force does not always pass through the centre of the sphere, it also results in a Brownian force moment that makes the particle rotate randomly at the same time. The rotation angular velocity Ω depends on the torque Γ_θ and viscous friction factor f_r of fluid and is decided by

$$\Omega = \frac{d\theta}{dt} \approx \frac{\Delta\theta}{\Delta t} \sim \frac{\Gamma_\theta}{f_r} = \frac{F_{Brownian} \cdot R_P}{8\pi\mu R_p^3} \sim \sqrt{\frac{3k_B T}{16\pi\mu R_p^3}} \cdot \sqrt{\frac{1}{\Delta t}} \tag{2}$$

The relationship between the rotation angle $\Delta\theta$ and the time interval Δt is obtained by

$$\Delta\theta \sim \sqrt{\frac{3k_B T}{16\pi\mu R_p^3}} \cdot \Delta t^{1/2} \tag{3}$$

From Eq. (3), it can be clearly seen that the rotation angle of the particle is proportional to the time interval. For a movement with a very small rotation angle $\Delta\theta$, it can be deemed as a straight motion. In the present case, the characteristic $\Delta\theta$ is set to $\pm\frac{\pi}{2}$ and the resultant characteristic time τ_R is about 10 s. Below this τ_R, the particle is able to maintain its direction and move forward.

We carried out the observation of the Janus particles (2 μm in diameter) and collected basic data in different concentrations of H_2O_2 solutions as shown in Fig. 2. The effective diffusion coefficient D_{eff} and the displacement rotational angle γ as a function of the time interval of observation Δt are used to describe the self-propulsion quantitatively. It can be seen that D_{eff} increases with Δt at the relatively short times, and then approaches the constant D_{eff} when Δt exceeds a certain threshold τ_R. The time threshold indicated that Janus particles change their motion from self-propulsion to Brownian-like motion. This transition can also be regarded as a change from straight to random motion. For different H_2O_2 solutions, the new constant D_{eff} is increased and the threshold τ_R is decreased as the concentration of H_2O_2 increases. The typical behaviours of self-propulsion have been studied in detail recently by Howse *et al.*[13] and also using our measurements.[14]

In Fig. 2(b), we show the probability of the γ angle at different time intervals in 5% H_2O_2 concentration. Obviously, in a short time period, small-angle rotations have large probability; for example, the particles' rotation is mainly concentrated between –50° and 50° when the time interval is 1 s. This means that the particles possess good directionality. The directionality decreases when the

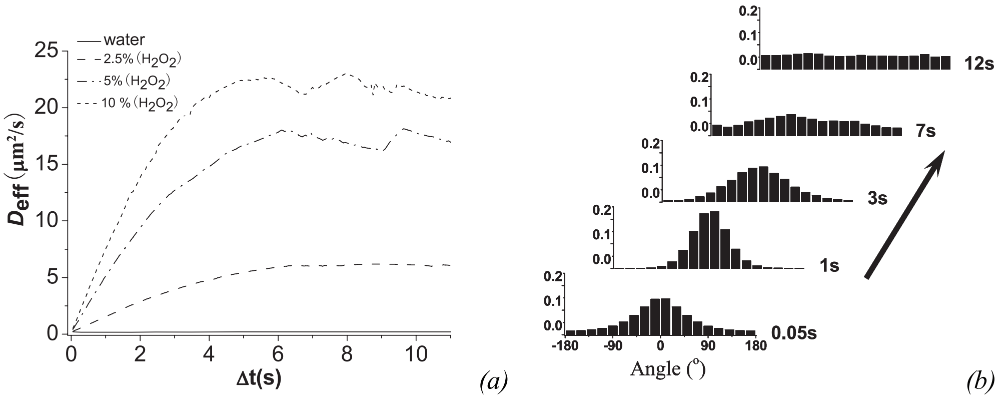

FIG. 2. (a) The effective diffusion coefficient as a function of the time interval for Janus particles in different H_2O_2 concentrations. (b) The probability distribution of displacement rotational angle at different time intervals in 5% concentration of H_2O_2.

probability of small-angle rotations is slightly lower as the time interval increases. The randomness of the particles rotation appears until the time interval is 12 s. At this moment, the angle of rotation has the same probability. So, the directionality of particles motion is available for our experiment for a short time interval. For example, for the short time $\Delta t = 2.5s$, where the particles have good directional motion, the effective diffusion coefficient is measured to be approximately 12 $\mu m^2/s$ in 5% H_2O_2, and the mean squared displacement $\langle L^2 \rangle$ is about 120 μm based on the well-known equation

$$\langle L^2 \rangle = 4D\Delta t \tag{4}$$

This means that the Janus particles have a large probability of undergoing almost straight propulsion with a typical length of 11 μm. This typical length is the basis for our design of microshuttle electrodes.

C. Controllability of Janus particles

1. Dielectrophoretic response of Pt-SiO_2 microsphere

According to bipolar theory, particles suspended in fluid media will be polarized under an external electric field and polarization charges of equal size and opposite sign are generated. Even for a neutral particle, the non-homogenous electrical field may induce the particle to move because the force at different poles cannot be counteracted, which is called DEP. For a homogenous sphere, the expression of the DEP force is given by,[15]

$$F_{DEP} = 2\pi\varepsilon_0\varepsilon_f r^3 \mathrm{Re}[\mathrm{CM}^*]\nabla E_{rms}^2 \tag{5}$$

where r is the radius of the particle, ε_0 is the permittivity of a vacuum, ε_f is the permittivity of the fluid medium, ∇E_{rms}^2 is the gradient of the RMS electric field, and CM^* is the Clausius-Mossotti factor. Re[CM^*] is the real part of CM^*,

$$CM^* = \left(\frac{\varepsilon_p^* - \varepsilon_f^*}{\varepsilon_p^* + 2\varepsilon_f^*}\right), \varepsilon_p^* = \varepsilon_p - j\frac{\sigma_p}{\omega}, \varepsilon_f^* = \varepsilon_f - j\frac{\sigma_f}{\omega} \tag{6}$$

where ε_p is the permittivity of the particle, σ_p is the conductivity of the particle, σ_f is the conductivity of the fluid medium, ω is the angular frequency of the AC electrical field, and $j = \sqrt{-1}$. Depending on the sign of CM^*, the sign of the DEP force could be positive or negative, in turn, to generate an attractive or repulsive force.

Currently, extensive works on the DEP response of micro- or nano-particles, cells, and nanotubes have been done,[16–18] however, few of them were about the manipulation of Janus particles. The DEP properties of Janus particles are quite different from those of ordinary dielectric particles (PS or

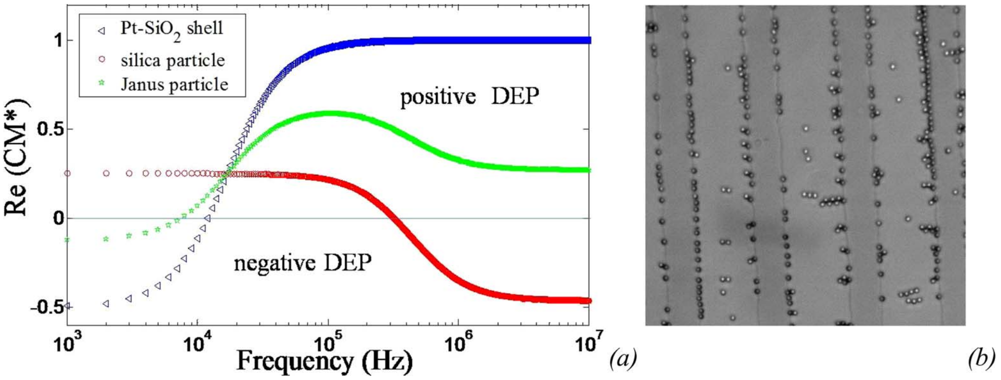

FIG. 3. (a) DEP response curves for SiO_2 particle, Pt-SiO_2 –shell, and Pt-SiO_2 Janus particle. (b) Snapshot showing pDEP response of Janus particles located at two edges of the electrode with the electrical parameters $V = 2v_{pp}$ and $f = 10$ MHz. Here, white particles are the control particles of uncoated SiO_2 particles, whose DEP response is nDEP.

SiO_2 particles) because there is a big difference in dielectric constant between metal and dielectric materials. Here, as reported by the literature,[19,20] a simple linear superposition model was used to consider the two-hemisphere structure. The integrate CM^* is calculated as the sum of the Pt-SiO_2-shell and the bare SiO_2 sphere,

$$CM^*_{Janus} = (CM^*_{Pt} + CM^*_{SiO_2})/2 \tag{7}$$

The response curve of CM^* versus applied AC frequency is given in Fig. 3(a). From this figure, it can be seen that CM^* of ordinary inorganic particles gradually decreases with increases in frequency and that the DEP force changes from pDEP to nDEP. The crossover frequency is about a few hundred kilohertz. The response of pure metal particles has the opposite trend. Different from two kinds of homogenous particles, the DEP properties of Pt-SiO_2-type Janus particles combine the properties of SiO_2 dielectric spherical particles and a Pt-SiO_2-shell.[19] We observed the DEP response of Pt-SiO_2 Janus particles with a frequency range from 1 KHz to 10 MHz. The crossover frequency from nDEP to pDEP is about 10∼20 KHz, which agrees with the experimental observation. A typical result in Fig. 3(b) shows that Janus particles are pDEP when the frequency is 10 MHz and the electrical potential is $2V_{pp}$. This is in agreement with the model above. It can be seen that Janus particles are attracted to two edges of the electrodes and keep a steady state. Especially, the side of the Pt-coated hemisphere faces away from the electrodes and the silica side points to the electrodes inside. Controlling the Janus particle's orientation is a key factor in our design and its mechanism will be explained by a numerical model below.

2. *Orientation angle of Pt-SiO_2 microsphere*

We calculated the electric field distribution and the energy of the system for different particle positions in order to understand why individual Janus particles are oriented in a unique direction as we observed in the experiment. The total electric energy E_e of the system can be obtained by integrating the local energy density, E_{es}, over the subdomain volume of the particle.

$$E_{es} = \frac{1}{2}DE = \frac{1}{2}\varepsilon_0\varepsilon_r E^2 \tag{8}$$

where D is the electric flux density, ε_0 is the dielectric permittivity of a vacuum (8.854×10^{-12}), and E is the electric field intensity. For our system, which is not in a vacuum, the electric flux density D is given by $D = \varepsilon_0\varepsilon_r E$, where ε_r is the relative permittivity of the particle.

We performed a 2D simulation with Comsol Multiphysics 4.1. As stated above, the Janus particle is attracted to the high field intensity area in the present electric field gradient so that the minimum potential energy should be reached. The voltage is kept constant and only the orientation angle of the Janus particle is changed. We then calculated the total stored electric energy (since it was a 2D simulation and we integrated over the area rather than the volume). The total electric

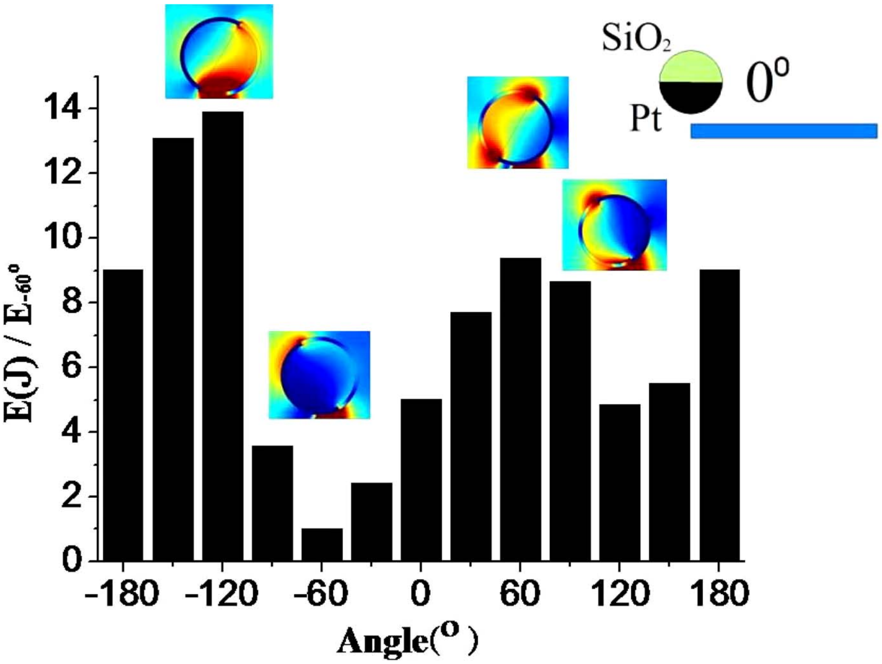

FIG. 4. The relative size of the electrical potential energy at different orientation angles; clockwise is negative.

energy of the system was calculated from –180° to 180° by rotating the particle about its Pt/SiO_2 interface axis in 30° increments. The potential energy is maximal at –120° and minimal at –60°. The latter corresponds well to the experimentally observed orientation of the particles (Fig. 4). It can also be straightforwardly judged from the fact that blue colour (cold colour represents low electrical potential) occupies almost the whole volume inside the particle.

D. Directed self-propulsion by AC electrical field

1. Time scale of modulation signal

The typical time scale τ_{DEP} of the self-propellant particle on IDEs is given by L/U_f, where L is the characteristic length dimension and U_f is the velocity of self-propulsion. For typical values of L (which is of the order of 10 μm as given by the half width of the electrode) and U_f (which is of the order of ~10 μm/s as given purely by the self-propellant velocity) in our experimental, the resulting time scale is about 1 s. It should be noted that the rotation time scale τ_R from Eq. (3) is used to depict the transition from self-propulsion to Brownian-like motion. The value of this characteristic time is about 10 s for our experiment (2-μm Janus particle and 5% H_2O_2 solution). Below the rotation time scale of $t < \tau_R$, the particle will move in a straight line in a direction from the Pt side to the SiO_2 side, and above the rotation time scale $t > \tau_R$ the particle will move randomly and can no longer be steered in only one direction. When the DEP force is modulated at the time scale ranging from $\tau_{DEP} < \tau < \tau_R$, we have the possibility to control the motion in time before the Janus particle begins to change its direction. The strength of the DEP force will be kept at a very low level so that it is just sufficient to change the orientation angle; in this paper, the voltage of $2V_{PP}$ is chosen.

2. Prototype verification of directed self-propulsion

To demonstrate the ability to direct the movement of the Janus particle in 1D mode, we performed prototype experiments where we introduced 2-μm Janus particles suspended in a 5% H_2O_2 solution and then applied pulsed DEP. The electrode parameters are $W_{IDEs} = W_{SPACE} = 20\,\mu$m, W_{IDEs} is the electrode width, and W_{SPACE} is the space between two adjacent electrodes. In the present design,

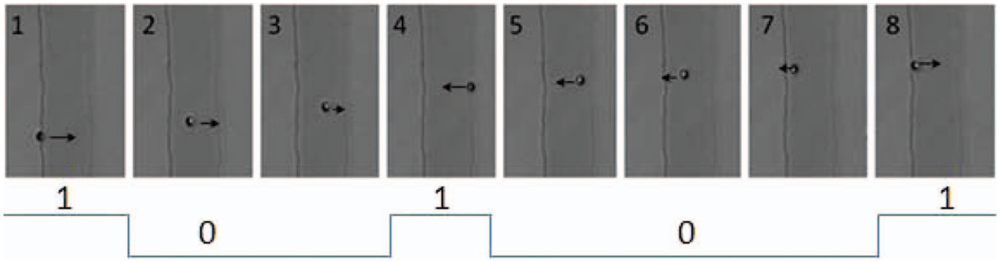

FIG. 5. Reciprocation of the Janus particle under the pulsed DEP with $V = 2\mathrm{v_{pp}}, f = 10$ MHz, $T = 5$ s.

we pulse the DEP force by turning it on and off with a 50% duty-cycle square wave with a frequency of 0.2 Hz. By tuning the time period, we can direct the movement of the Janus particles.

The results in Fig. 5 depict a time-series of images under pulsed operating conditions. In the first "on" period, it is shown that one Janus particle is attracted to the left edge of the electrode and the Pt-side of Janus particle faces the left (Fig. 5(1)). During this period, the DEP force at the edge of the IDEs is maximal and strong enough to overcome the Brownian motion, and the Janus particle is trapped steadily. Once the electrical field is powered off, no DEP force is applied to the Janus particle and self-propellant force is dominant. At the beginning of the first "off" period, the Janus particle moves from the left edge to the right edge inside the electrode. The path of the particle is nearly a straight line (Fig. 5(2)). At the end of the first "off" period, the Janus particle has moved to the vicinity of the right edge (Fig. 5(3)). In the second "on" period, the particle is immediately attracted to the right edge of the electrode, and at the same time the Janus particle instantly rotates by 180° and keeps its Pt-side opposite to the electrode again (Fig. 5(4)). In the following periods (Fig. 5(5,6,7)), the Janus particle recovers its self-propelled motion and comes back to the left edge of the electrode from the right edge. In the last image (Fig. 5(8)), the Janus particle returns to the initial state and the whole shuttle-style cycle is completed. The above procedure can be repeated many times only when pulsed DEP is applied and a fuel solution is supplied. The result has successfully verified the prototype design. Based on the present work, more complicated operations with directional self-propellant motion could be carried out in the future.

III. CONCLUSION

We have introduced a DEP-based modulation method that makes use of time-pulsing to generate 1D self-propulsion for Janus particles. Although it was found that the randomness of self-propulsion is significant due to the appearance of Brownian-like motion, at short time scales, directional movement is still approached. With the help of an AC electric field generated by IDEs, one can control the orientation of Janus particles when they experience positive DEP so that they are attracted to the edge of the IDEs. The time scale τ for a pulsing AC electrical field is decided by the nature of self-propulsion and the geometric dimension of the electrodes. By modulating the time precisely, Janus particles have enough time to arrive at the opposite side of the electrode and maintain approximately straight movement during this period. By repeatedly turning the electric field "on" and "off" many times, the shuttle-style motion of Janus particles is generated. We also developed a numerical model to explain the unique orientation of Janus particles and a simple analytical model to calculate the DEP response under an AC electric field, and the model predictions were consistent with the experimental observations.

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APPENDIX

1. Fabrication of Janus particles

The bare SiO_2 particles were brought from the standardization center of China University of Petroleum, and their nominal diameter was $\phi = 2061 \pm 18\, nm$. To prepare the Janus particles, a powder of SiO_2 particles was mixed with deionized water and a few tens of microliters of mixture were dispensed on the silicon wafer. Next, the silicon wafer was settled on a spin coater, and a rotation speed of 800 rpm was applied to form a thin layer of the mixture solution. This was followed by natural evaporation of the water, and the remaining particles gathered compactly to form an SiO_2 particle monolayer arrangement on the silicon wafer. Then, using an e-beam evaporation technique, a 7-nm thick Pt film was deposited onto the top hemisphere of the SiO_2 microspheres. However, the evaporation process of Pt cannot reach the bottom hemisphere and it is left unchanged. Thus, Pt-SiO_2-type Janus particles were fabricated. Under the microscope, the Pt side is black and the SiO_2 side is grey.

2. Fabrication of IDEs and electronics

The strip electrodes of the device are fabricated from ITO-coated glass slides.[21] The ITO glass slides were first cleaned and blow-dried and then dehydrated. A positive-tone photoresist AZ2001 was spun-coated at 2500 rpm and the slide was baked at 100 °C prior to standard photolithography. The slide was then exposed to ultraviolet light through a film mask. The pre-designed IDE pattern was transferred to the ITO glass surface. Subsequently, the slide was immersed into a developer solution for 20 s. The slide was then etched in a solution of ferric chloride (35 g), hydrochloric acid (250 mL), and deionized water (250 mL). The etching time was about 2 minutes, after which the slide was immersed in a 5% sodium carbonate solution to remove the surplus etching solution.

3. Fluidics, optics, and electronics

Before experimental observation, Janus particles were scraped from the silicon substrate with a razor blade. The powder of Janus particles was resuspended in 30% H_2O_2 and pure water (18 MΩ) in the different ratios required by the experiment. The volumetric concentration of the Janus particle suspension was about 5×10^{-3} mol/mL.

The particle trajectories in water and in H_2O_2 solutions with different concentrations were observed by video microscopy with an image field of view of 512 × 512 pixels. Image series consisting of 600∼1000 frames were captured in one position located about 2 μm above the glass substrate.

Two types of signals, continuous and pulsed, were applied to the electrodes to generate the DEP force. A sine-wave signal at 10 MHz with an amplitude up to 2 Vpp was generated by a universal signal generator. The electrical connection was realized by affixing a copper wire to a reserved ITO pad with silver conductive glue.

More details about the operation parameters can be found in our previous works.[14,21]

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