Conical Hollow Microhelices with Superior Swimming Capabilities for Targeted Cargo Delivery

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Inspired by flagellate microorganisms in nature, the microhelix is considered as an ideal model for transportation in fluid environment with low Reynolds number. However, how to promote the swimming and loading capabilities of microhelices with controllable geometries remains challenging. In this study, a novel kind of conical hollow microhelices is proposed and a method is developed to rapidly fabricate these microhelices with controllable parameters by femtosecond vortex beams generated from spatial light modulation along helical scanning. Conical hollow microhelices with designable heights (H = 45–75 µm), diameters (D = 6–18 µm), pitch numbers (P1 = 2–4), taper angles (θ = 0.1–0.6 rad), and pitch periods (ΔP = 10–30 µm) are efficiently fabricated. In addition, compared with straight microhelices, the forward swimming capability of conical microhelices increases by 50% and the lateral drift of the conical hollow microhelices is reduced by 70%. Finally, the capabilities of these conical hollow microhelices for nanocargo loading and release by the inner hollow core, as well as transportation of neural stem cells by the outer surface are demonstrated. This work provides new insights into faster and simultaneous transportation of multicrogdes for hybrid drug delivery, targeted therapy, and noninvasive surgery in vivo.

without pollution are the key requirements for fast and complete cleaning of toxic materials. Therefore, artificial micro-nanorobots with controllable parameters, superior swimming capabilities, and stronger loading capabilities are highly desirable.

Due to the low Reynolds number in microliquid environments and Brownian movement of micro-nanorobots, precise manipulation of micro-nanorobots is a challenge. Many efficient propelling strategies have been presented to overcome the Brownian motion and low Reynolds number, including chemical, optical, ultrasonic, and magnetic propulsion mechanisms. Among these strategies, magnetic propulsion has gained broad interest because of the fuel-free propulsion, wireless steering, high controllability, and nonpollution. Compared with fuel-driven propulsion, magnetic propulsion obviates detrimental and limited fluid environment. In addition, magnetically actuated micro-nanorobots are convenient to be applied to the human body because the magnetic field can penetrate the biotissues. Magnetic propelling also exhibits high precise manipulation in 3D space for better application in vivo. Therefore, magnetic fields have been widely used for steering artificial micro-nanorobots, especially in biomedical applications.

Inspired by bacteria in nature, the helix, as a classical structure in most magnetic micro-nanorobots that can change rotation in a plane to forward motion, has been widely investigated. For example, Zhang et al. fabricated composite metal microhelix by combining metal deposition, photolithography, reactive ion etching, lift-off process, and self-rolling of thin films with InGaAs/GaAs/Cr to form a microheliex due to gradient stress. In addition, Hoop et al. demonstrated that nanohelices were obtained by selectively etching the AAO template (by NaOH) and Cu segments (by HNO3) and covering magnetic layer Ni for bacterial contact killing. Moreover, Yan et al. proved that an alternative spirulina template-based hollow microhelices fabrication method was developed by absorbing Fe3O4 on the surface and annealing for targeted delivery. However, the uncontrollable aggregation of magnetite nanoparticles outside biotemplates leads to random geometry, coarse surface, and weak mechanical strength. How the geometry of hollow helices can be optimized for better magnetic propelling and steering are still unclear. Tottori et al. also took advantage...
of point-to-point direct laser writing (DLW) to fabricate microhelices with various parameters microhelices for microparticles transportation by femtosecond laser.[29] Nevertheless, helical microstructures were only limited to simple straight nanowires with a drawback of weak loading capacity and small relative contact area, which are significant factors for the application in microobject delivery or detoxification. Besides, helical micro-wires are fabricated with two-photon polymerization by single laser spot scanning, which is really time consuming, especially for the large batch production of these microstructures for following dilution in liquid environment and magnetic propelling. In addition, faster forward velocity of microhelix is desirable for faster drug delivery, which is currently limited by uncontrollable structures. Furthermore, lateral drift always appears with the forward movement, which causes big difficulty to precise guiding for biomedical application. Reduce of the drift movement is essential, which has not been solved in previous works. Taken together, it is highly desirable to design new microhelices with better swimming property and stronger transporting capability for biomedical applications.

In this study, femtosecond vortex beam is first produced by spatial light modulation (SLM) and then used for rapidly fabricating conical hollow microhelices based on two-photon polymerization by helical scanning. Compared with conventional point-to-point scanning strategy, the fabrication speed can be enhanced by 24 times. In order to optimize the swimming property and transporting capability of microhelices, 3D conical hollow microhelices with designable heights (45–75 µm), diameters (6–18 µm), pitch numbers (2–4), taper angles (0.1–0.6 rad), and pitch periods (10–30 µm) are flexibly fabricated. The swimming capabilities of different microhelices are analyzed in detail. We find that the maximum forward velocity of conical microhelix is 50% faster than that of straight microhelix while the lateral drift is reduced by nearly 70%. The superior swimming properties make 3D conical hollow microhelices easier to complete complicated trajectories and targeted cargo delivery with external magnetic manipulation, especially in an environment of high liquid viscosity and low magnetic frequency. Based on the optimized geometrical parameters and swimming performances, transportation of nanoparticles (SiO₂ and Ag) and neural stem cells (NSCs) is realized by these conical helices. Their applications in chemical microreaction (decomposition of H₂O₂ by silver nanoparticles (AgNPs)) and biomedical research (in vitro culturing of NSCs) are also demonstrated. This conical hollow microhelix holds great promise for faster multicargoes transportation and broad applications in hybrid drug delivery, targeted therapy, noninvasive surgery, and other biomedical studies.

The main concept for controllable fabrication of hollow microhelix in our experiment is schematically illustrated in Figure 1a. Ring-structure vortex beam is modulated by a computer-generated hologram (CGH) displayed on the Lcos-SLM. The CGH is composed of two parts shown in Figure 1b. Spiral phase plate (SPP) has an azimuthal phase distribution of \( \phi \). In

![Figure 1. Rapid controllable fabrication of hollow microhelix by femtosecond vortex beams based on SLM along helical scanning. a) Experimental setup. HWP: half wave plate; BE: beam expander; ID: iris diaphragm; M: mirror; Lcos-SLM: liquid-crystal-on-silicon spatial light modulator; L: lens; CCD: charge-coupled device. b) Illustration of the computer-generated hologram (CGH) composed by spiral phase plate with topological charge \( l = 20 \), and BG with period \( \Delta = 15 \) µm. c) Simulated light field \( (l = 20) \) at the focal region of high numerical aperture (NA = 1.35) oil-immersion objective lens. The right section corresponds to calculated and measured intensity distributions at planes of \( -6, -3, 0, 3, 6 \) µm from the central focal plane, respectively. d) 45° tilted SEM of microhelix arrays with high uniformity. The scale bar is 50 µm. e) Subsequent processing steps for functionalization, steering, and cargoes transportation: sputtering magnetic material, transporting in liquid environment, driving in Helmholtz coils system controlled by PC.](image-url)
microhelices with different geometrical properties including both left-handed and right-handed microhelices (Figure S6, Supporting information) are rapidly produced. Figure 2a–c shows microhelices with different height ($H = 45–75 \mu m$), diameters ($D = 6–18 \mu m$), and pitch numbers ($P_I = 2–4$), respectively. The scanning speed for all circular microhelices fabrication is 12 $\mu m \ s^{-1}$, and the used laser power is 60 mW. The microhelices are 60 $\mu m$ high and the fabrication time is 5 s, while it increases to 120 s with conventional single spot direct laser writing with a piezo stage (Figure S7, Supporting information). Furthermore, conical hollow microhelices with various parameters are designed and rapidly produced as well. As showed in Figure 2d, conical microhelices with taper angles ($T$) from 0.1 to 0.3 rad depending on bottom radius ($R_b$) and height ($H$) are realized (Figure S8, Supporting information). Different pitch periods ($\Delta P$) from 10 to 30 $\mu m$ are also realized by controlling the focus scanning trajectory, which keep the same taper of 0.3 rad (Figure 2e). The hollow core (Figure S9, Supporting information) can be clearly observed in each microhelix. The spiral features of microhelices are shown in Figure 2f by fluorescence image from microhelices lying on a glass substrate. Specially, a “USTC” pattern is designed and fabricated with taper angle of 0.3 rad, pitches period of 20 $\mu m$ by the optical vortex with $l = 20$ (Figure 2g,h), which demonstrates the flexibility of this fabrication method. When a uniform 20 Gs magnetic field with a constant direction is applied, the directions of the conical microhelices axes vary with the taper angles. Here, the angle between the magnetic axis of microhelices and the uniform magnetic field is defined as misalignment angle $\alpha$. Generally, the misalignment angle of straight microhelix has positive correlation with the helix angle (Figure S8, Supporting information). For conical microhelix, the average helix angle increases with the growth of taper angle, leading to constant improvement of misalignment angle (Figure 2i). The taper angles and misalignments approximately keep on with the Heaviside function in theory similar to straight microhelices.$^{[15]}$ The misalignment angle of conical microhelix with taper angle of 0.1 rad is less than 35°, where magnetic axis tends to be along the long axis. In particular, taper equal to 0.3 rad is a turning point, around where misalignment angle has a rapid growth. The misalignment angle is up to 82.5° when taper angle is equal to 0.6 rad in order to promote magnetic axis vertical to long axis for flagellate spiral motion. The misalignment angle always has influence on the swimming character of microhelices, which is further discussed in next part.

Efficient steering of microhelix is crucial to achieve its function. A Helmholtz coils system is constructed to complete universal steering. Three pairs of coils are driven by sinusoidal alternating current, as shown in Figure 3a. The moment of force exerted on microhelix is schematically shown in Figure 3b. Magnetic dipole moment ($\mathbf{M}$) interacts with external magnetic field ($\mathbf{B}$), producing magnetic moment ($\mathbf{T}$). It is worth to notice that magnetic moment is more efficient than magnetic gradient force to achieve microhelices steering.$^{[16]}$ The relation between geometrical properties and swimming capabilities is systematically studied. First, straight hollow microhelices with different diameters are steered in deionized (DI) water with surfactant (Triton X-100) in magnetic field with 10 Gs strength.
and 30 Hz frequency. Figure 3c shows the trajectory of straight microhelices within 3 s (Video S1, Supporting information). At the beginning, forward velocities always increase with growth of frequency. But a step-out frequency appears with the continuous growth of frequency, which leads to asynchronous steering of microhelices with the rotating magnetic field. The step-out frequency depends on three main factors: material category, helix geometry, and magnetic property, which is formulated as:

$$f_s = A \left(1 + \gamma^2 \right) \sin 2\alpha$$

where $A = \Delta \gamma VH^2/k_l$ is a characteristic parameter. The parameter $\Delta \gamma$ depends on saturation magnetization and $H$ could be changed by different materials category and the strength of magnetic field. $\gamma = \frac{k_{fi}}{k_{fi}} \tan \alpha$ is a “steerability” parameter affected by the microhelix geometry, where $\alpha$ is misalignment angle. Thus, the forward swimming velocity is formulated as:

$$v = \begin{cases} 2\pi R \cdot Ch \cdot f & \left(1 - \frac{2}{1+\gamma^2} \frac{f^2}{f_{i}^2} \left(1 + \frac{1}{1-f^2/f_{i}^2} \right) \right) \quad f < f_i \\ 2\pi R \cdot Ch \cdot f & \left(1 - \frac{2}{1+\gamma^2} \frac{f^2}{f_{i}^2} \left(1 + \frac{1}{1-f^2/f_{i}^2} \right) \left(1 - \sqrt{f^2 - f_i^2} \right) \right) \quad f \geq f_i \end{cases}$$

(3)

where $Ch$ is the chirality coefficient depending on microhelix geometry. According to the result of experiments and numerical calculations, the step-out frequencies are around 42, 28, and 24 Hz for microhelices with 3, 6, and 9 $\mu$m radius, respectively. The highest velocity achieved by experiments and calculations is around 120 $\mu$m s$^{-1}$, as shown in Figure 3d. In addition, conical microhelices with various taper angles (0.3–0.6 rad)
are also steered in Figure 3e (Video S2, Supporting information). The forward velocity of conical microhelix is also in direct proportion to the frequency similar to the straight microhelix (Figure 3f). The chirality coefficients $Ch$ are 0.1549, 0.1335, 0.1137, 0.0983 depending on the helix geometry, respectively. According to Equation (3), the dimensional slopes of the curves are $\approx 4.38$, 5.03, 5.36, 5.56 $\mu$m, respectively. As a result, the maximum forward velocity of the conical microhelices is faster than straight microhelices up to nearly $180 \mu$m s$^{-1}$ in 10 Gs magnetic field (Figure 3f). Furthermore, the forward velocity of conical microhelices with different field strength is investigated, and we find that the velocity of movement along the sharp end of
the cone is much faster than the opposite direction (Figure S10, Supporting information). Apart from different conics, the pitch period is another major factor that influences the forward swimming velocity. The conical microhelices with pitch period of 30, 20, 15, and 10 µm are steered in the same magnetic field, as shown in Figure 3g–j, respectively (Video S3, Supporting information). It is clearly found that microhelix with a longer pitch period has a higher forward velocity. The computed step-out frequencies are 35, 40, 26, 20 Hz for microhelices with 30, 20, 15, 10 µm helical pitch periods, and the best pitch period is around 20–30 µm so that the highest velocity can be up to 180 µm s⁻¹, as shown in Figure 3k. The slope values V/f are 5.08, 4.38, 3.46, and 2.63 µm for each microhelix, respectively. In short, the forward swimming characteristics of conical microhelices are improved by about 50% compared with straight microhelices.

Apart from forward swimming, the lateral drift swimming plays an important role in swimming characterization of microhelices. When the conical microhelix rotates at the same frequency, the rotation linear speed of the tail is higher than the head of conical microhelix due to the difference in diameter of each part. For a microhelix with a taper angle of 0.3 rad, the radius difference between tail and head is 9 µm. At a frequency of 5 Hz, the linear velocity of the tail is 282.7 µm s⁻¹ faster than the head. Thus, the head of microhelix is always reversed to rotation magnetic field to offset a part of the drifting velocity. Straight microhelix always keeps vertical state compared with the inclination conical microhelix in rotation magnetic field with 5 Hz frequency and 20 Gs strength (Figure 4a,b; Video S4, Supporting information). At the same forward speed, the lateral velocity of conical microhelix is less than half of straight microhelix. Since drift velocity always has positive correlation with forward swimming, ratio of lateral and vertical velocity is a significant parameter to evaluate the drift swimming character. At low frequency, the forward velocity is relatively low so that velocity ratio is rather high. Velocity ratio decreases with the improvement of frequency at first. When frequency is up to step-out frequency, velocity ratio increases again because of the reduce of forward velocity after step-out frequency. The velocity ratios of conical (T = 0.3 rad, Rₜ = 9 µm) and straight microhelices (R = 9 µm) are investigated with different frequency and magnetic strength (Figure 4c,d). Obviously, the initial values of velocity ratio are 0.85 and 1.43 for the conical and straight microhelices. At the frequency of 40 Hz, the velocity ratio decreases to 0.23 for conical microhelices. By contrast, the minimum velocity ratio is still 0.68 for straight microhelices at 25 Hz. So, the drift swimming is reduced by about 70% at suitable frequency. In addition, the velocity ratios of straight and conical microhelices both decrease with the growth of magnetic strength at 30 Hz rotation frequency, which keep stable after critical strength (Figure 4d). Finally, the relationship between velocity ratio and taper angle is studied (Figure 4e). With taper angles increasing from 0.1 to 0.3 rad, the velocity ratios decrease after initial growth at each step-out frequency. When the taper angle sequentially increases, the aspect ratio increases again due to the instability of the motion and the decrease of the forward velocity (Figure S12, Supporting information). The best taper angle for reducing the degree of drift swimming is nearly 0.3 rad, where the velocity ratio is only about 0.2 (Figure 4e). Above all, it is distinct that lateral drift motion is significantly mitigated by conical microhelices for a better guiding capability. At frequency of 10 Hz, the propelling of the conical microhelices in arbitrary directions is realized along with the change of rotation magnetic field (Supporting information). As shown in Figure 4f, microhelix is first steered to move ahead to the lower right corner. Then the direction is changed to the left side after 5 s. Finally, the microhelices are steered back to the top left corner along with a 10 Gs strength magnetic field. Moreover, the excellent orientation properties of conical microhelices make them easy to realize complex routes. Figure 4g–i shows different routes of “square,” “triangle,” and “NANO” patterns, respectively (Video S5, Supporting information). All of these show the precise steering ability, which is highly desirable for cargo transportation and drug delivery (Figure S13, Supporting information).

Based on the precise steering of conical hollow microhelices, micro/nanocargoes loading, transporting, and releasing are realized. It should be noticed that various cargoes can be transported simultaneously with both the hollow inner core and the outer surface. In the experiment, SiO₂ nanoparticles with 200 nm diameter are efficiently loaded in hollow microhelices (Figure 5a). It is clearly observed that many nanoparticles are loaded both on microhelix surface and inside the hollow core, which promote the capacity of loading by larger surface area compared with solid microhelices. Furtherly, the application of microhelices in chemical microreaction (decomposition of H₂O₂ by AgNPs) is demonstrated, as shown in Figure 5b. Conical hollow microhelices are filled with AgNPs at the beginning and dispersed in DI water. Then, along with the adding of H₂O₂ and Triton X-100 (100:1), bubbles are produced from hollow microhelices in 2 s (Video S6, Supporting information) because of the decomposition of H₂O₂ by AgNPs. Thus, the AgNPs are gradually consumed and released. Apart from the hollow core, the surface of the microhelices can also be loaded with microcargoes. Taking the advantage of this property, a great prospect is proposed that the neural stem cells can be controllably transported in vivo for cell delivery and damaged tissue repair in future, which is schematically shown in Figure 5c. In order to prove this concept, targeted transportation of single and double neural stem cells is realized with microhelices in vitro (Figure 5d,e; Video S7, Supporting information). Neural stem cells adhere to microhelices surface by coated poly-L-lysine (PLL) and rotate together with microhelices. In order to prevent the decrease of cell viability, conical hollow microhelices are coated with 20 nm biocompatible materials Ti (Figure S14, Supporting information). Generally, cells are always guided by themselves to move toward rigid substrate with lamellipodia and lamella expanding in vitro because cells on stiff substrates generated significantly stronger traction than those on soft substrates,[39] and finally released from microhelices. After 3 days of cultivation, the NSCs are released from microhelices surface and then proliferate onto glass substrate in vitro with 10% serum, which keep up with great viability as shown in Figure 5f. By adjusting the fetal bovine serum ratios to 1%, NSCs can also effectively differentiate into neural cells after 2 days of cultivation, for there are evident nerve dendrites on the cell as performed in Figure 5g. This method provides a valid solution for the simultaneous transportation of nanocargoes and microcargoes.
In this work, we present a novel method to rapidly fabricate hollow microhelices with controllable heights, diameters, pitch numbers, taper angles, and pitch periods based on femtosecond holographic processing method. The fabrication time can be decreased by 24 times compared with conventional point-to-point scanning. The relationships between swimming properties and geometrical parameters of microhelices are systematically analyzed in the experiment. It is found that conical microhelices have higher forward velocity compared with straight microhelices. In addition, conical microhelices exhibit much lower lateral drift ratio, which makes it easier to complete complicated trajectories with external magnetic manipulation. On the basis of the superior swimming characteristics, the conical hollow microhelices are used to load and release nanoparticles by its hollow core. Targeted delivery of cells with these conical microhelices is realized by transporting neural stem cells to predesigned trajectories.
destination area for subsequent proliferation in vitro. The novel fabrication method and the fabricated conical hollow microhelices hold great promise for a very broad application prospects in hybrid drugs delivery, targeted therapy, and noninvasive surgery and other biomedical studies.

Experimental Section

Fabrication of the Hollow Microhelices: The typical holographic femtosecond laser writing system source is a mode-locked Ti: sapphire laser oscillator (Chameleon Vision-S, Coherent Corp, central wavelength: 800 nm, repetition rate: 80 MHz, pulse width: 75 fs), and the CGH was displayed on a reflection type liquid crystal SLM (Pluto NIR-2, from Holoeye Photonics AG, Germany), with 1920 × 1080 pixels, the pixel pitch of 8 µm, and 256 gray levels. The vortex beam was generated by loading the prepared CGH on SLM. A commercially available zirconium–silicon hybrid sol–gel material doped with 4,4'-bis (diethylamino)-benzophenone photoinitiator at 1% by weight (SZ2080, IESL - FORTH) was used for photopolymerization in the experiment. The sample was prebaked to evaporate the solvent on a 100 °C thermal platform for 1 h. The movement of sample was completed by a nanopositioning stage (E545, from Physik Instrumente GmbH & Co. KG, Germany) with nanometer resolution and a 200 µm × 200 µm × 200 µm moving range to precisely locate microstructures. The vortex beam was focused by a 60 × oil-immersion objective (Olympus) with high NA (1.35) for high processing quality. The sample was developed in 1-propanol for 1 h to wash away all of the unpolymerized parts after laser photopolymerization. With the spontaneous evaporation of the developer, the hollow microhelices were

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Figure 5. Nanoparticles (SiO2 and AgNPs) and neural stem cells (NSCs) loading, transporting, and releasing. a) SEM images of hollow microhelice loaded with and without nano SiO2 particles (the upper and lower two figures depict the microhelices standing and lying on the substrate, respectively). b) Loading and consumption of AgNPs with hollow microhelices. c) The schematic illustration of NSCs transportation for cell delivery and tissue repair by conical hollow microhelices with superior swimming capabilities. d) Single NSC transportation and e) double NSCs transportation in cell culture medium. f) The NSCs are released from microhelix onto a pure glass substrate and proliferate after 3 days of cultivation. g) NSCs are released from microhelix onto a pure glass substrate and differentiate after 2 days of cultivation. Scale bar are 1 µm in (a), 50 µm in (b), (g), and 100 µm in (d),(e).
obtained on the glass substrate. Then, the sample was evaporated with 100 nm Ni/Ti bilayer at a rate of 2 Å s⁻¹ by magnetron sputtering (Kurt J. Lesker, LAB 18) with RF source of 600 W, vacuum degree of 10⁻⁶ mbar after plasma cleaning for 1 min.

**Characterization of Swimming Performance:** A triaxial Helmholtz coil system is composed of three parts: data acquisition card controlled by computer, power amplifier, and the Helmholtz coil used to generate rotating magnetic fields in arbitrary direction. The microhelices were swept down from the substrate by microprobe. Then, the microhelices were blown repeatedly using a microliter pipette so that they could be suspended in the droplet and transferred out to a square microtrap fabricated by PDMS and glass substrate for following micromanipulation and observation. A charge-coupled device (CCD) camera (MV-UB500M with 1280 × 960 pixels, 22 FPS) with an optical lens and a light-emitting diode (LED) light source was connected to the computer for videos and pictures capture.

**Cargo Loading and Release:** SiO₂ (1 mg) nanoparticles were combined with DI water (10 mL) to generate the suspension after 10 min sonication. The sample was immersed in the suspension cultured for 1 h in order to load nanoparticles. The scanning electron microscopy (SEM) images were collected with a secondary electron SEM (ZEISS EVO18) operated at an accelerating voltage of 10 kV after depositing ~100 nm gold. The AgNPs (30–50 nm diameter) were purchased from aladin-e. com (S110970). AgNPs (5 mg) were added in 10 mL DI water to generate suspension. Commercial poly(vinylpyrrolidone) (PVP) (1 mL) was dropped in the suspension in order to avoid the particles agglomeration. The sample was repositioned in H₂O₂ (10 mL) with Triton X-100 (0.1 mL) to generate bubbles after cultured for 1 h in suspension.

**Cell Transportation and Culture:** Neural stem cells were kindly provided by Stem Cell Bank, Chinese Academy of Sciences. The neural stem cells were maintained in minimum essential medium (Invitrogen, 11 090 081) supplemented with 10% fetal bovine serum (FBS, Gibco), glutamax (Invitrogen, 35 050), and nonessential amino acids, 100 × (Invitrogen, 11 140) at 37 °C in a humidified atmosphere of 5% CO₂ in biological operation cabinet. Before the cell seeding, the microhelices were immersed in 10 μg mL⁻¹ PLL (10 μg mL⁻¹) for 24 h coating. PLL is a common coating material to enhance NSCs attachment with microstructures, which is positively charged to absorb cell with negatively charged surface. After 30 min ultraviolet sterilization, NCs were seeded at a concentration of 10⁵ cells mL⁻¹ into a Petri dish containing microhelices. After 1 h cultivation, the Petri dish with microhelices was put in a Gelanholtz coil system for 3-day suspension. Subsequently, the Petri dish was placed in an incubator at 37 °C and measuring of samples. This work was partly carried out at the USTC Center for Micro and Nanoscale Research and Fabrication.

**Conflict of Interest**
The authors declare no conflict of interest.

**Keywords**
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**Supporting Information**
Supporting Information is available from the Wiley Online Library or from the author.

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