Noncontact All-In-Situ Reversible Reconfiguration of Femtosecond Laser-Induced Shape Memory Magnetic Microcones for Multifunctional Liquid Droplet Manipulation and Information Encryption

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Shape memory polymers can change their shapes in a controlled way under external stimuli and thus promote the development of smart devices, soft robotics, and microfluidics. Here, a kind of iron particles (IPs) doped shape memory microcone is developed for noncontact all-in-situ reversible tuning between the tilted and upright state under near-infrared (NIR) irradiation and magnetic field (MF) actuation. The magnetic microcones are simply fabricated by a laser-ablated replica-molding strategy so that their height can be precisely controlled by adjusting the laser machining parameters. In addition, it is found that the droplets can be transported unidirectionally on the tilted microcones, which is related to the variation of the adhesion force induced by the length of the triple contact line (TCL). Finally, other multifunctional applications have also been realized, such as, selective droplet release, information encryption, rewritable display, and reusable temperature switch. This work may provide a facile strategy for developing multiresponsive smart devices based on shape memory polymers.

1. Introduction

Recently, stimuli-responsive shape memory polymers (SMPs) have attracted considerable interest due to their tunable mechanical properties under the external trigger,[1,2] such as light,[3–5] heat,[6–8] electricity,[9,10] and so on. When the SMPs are heated above the glass transition temperature ($T_g$), it would become soft, which could be changed into arbitrary shapes under external pressure. Then it would be solidified into the temporary state after cooling. When being reheated above $T_g$, the SMPs would rapidly recover their initial state.[11–14] Based on this intriguing property of SMPs, various functional structures, and smart devices have been exploited in diverse fields, including adhesive film,[15] actuators,[16] sensors,[17,18] biomedicine,[19,20] and rewritable chip.[21] For example, Bai et al.[12] fabricated a rewritable superhydrophobic SMP platform to achieve directional liquid transportation. Zhang et al.[22] designed a smart surface with tunable wetting states based on SMP and PNIPAAm under the temperature stimulus. Cheng et al.[23] reported a kind of SMP pillar array that can reversibly transit the wetting states through dynamically controlling the microstructure shape. It is noticed that the heating strategies used in the above works are mostly based on a heating plate or electrical temperature control chamber, which need the SMP sample to directly contact with the heating equipment. This leads to some disadvantages during the tuning process, such as inconvenient operation and dangerous manipulation,[24] which would severely limit the potential application of these devices in a complex environment.

To improve the heating condition, researchers proposed a simple strategy, such as photothermal heating, to trigger the shape memory effect of SMP through doping some particle materials. For instance, Zheng et al.[3] prepared SMP micropillar arrays by doping gold nanorods to achieve light-induced shape recovery. Mishra et al.[25] reported a kind of SMP with gold nanospheres and nanorods to achieve sequential actuation through wavelength-selective photothermal heating. Yi et al.[26] reported the photothermal shape recovery behaviors by doping nanocarbon hybrid-based composites which can be actuated by a near-infrared laser. However, those doped specific particle materials are either expensive or complex to prepare. In addition, a major drawback for those works is that the SMP structures must be exerted by external contact pressure if they are deformed again. Therefore, a simple, noncontact, all-in-situ tuning strategy of the smart SMP with the multistimuli response is urgently demanded.

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To realize noncontact all-in-situ tuning, magnetic nanoparticles with dual photothermal and magnetic responses were doped in SMPs. For instance, Ze et al.\cite{27} reported an SMP surface composed of Fe$_3$O$_4$ and NdFeB to achieve reversible actuation and shape locking of robotic grippers, which could be used for capturing a lead ball through a complex magnetic system. Liu et al.\cite{28} fabricated a kind of centimeter-level soft robotic grabber actuated by photothermal and magnetic reconfiguration of SMP composites for grabbing the circular fruits. However, the above mentioned smart devices with reversible tuning ability could not manipulate the microscale objects due to the large size of their structures (centimeter scale). In addition, the type of manipulation object is also confined to the solid object (ball and fruits). Recently, Liu et al.\cite{29} reported that the micropillar can be easily obtained by the self-assembly method, but the structure is hard to keep consistent. To the best of our knowledge, the noncontact, all-in-situ reversible configuration of magnetic particles doped SMP microscale structures to achieve the manipulation of liquid droplets with microscale size has rarely been reported (Figure S1, Supporting Information).

Herein, a kind of IPs-doped SMP microcone array with dual photothermal and magnetic responses for multifunctional microdroplet manipulation was reported. The microcone array was fabricated by a laser-ablated replica-molding method and could realize noncontact all-in-situ reversible tuning between the tilted and upright state. Under the NIR irradiation, the IPs can rapidly transform light energy into heat energy, and then the microcones can be bent by the magnetic field. The microcone will remain tilted state after removing the NIR and magnet in sequence. Finally, after turning on the NIR again, the microcone will recover the original upright state. It is worth noting that the whole tuning process does not need to directly in contact with the sample, which achieves the remote control of the SMP microcones. In addition, it is found that the tilted microcones have an obvious anisotropic adhesion force to the droplet along and against the tilted direction, which could be used for the multifunctional manipulation of microdroplet such as droplet unidirectional transport, selective release, and so on. Finally, the microcone array was also designed for information encryption, rewritable display, and temperature switch.

2. Results and Discussion

2.1. Design and Fabrication of the SMP Microcone Arrays

Figure 1a schematically illustrates the crucial fabrication process of the SMP microcone array. First, the regular microhole array was obtained on silica gel soft mold by a one-step femtosecond laser micro-drilling method, which exhibited the advantages of high efficiency, low thermal effect, and good controllability.\cite{30-35} Subsequently, the mixture of IPs/epoxy resin (EP) was deposited on a silica gel mold for complete curing. Finally, an IPs-doped SMP microcone array with many rough surfaces was obtained after being peeled off (Figures S2 and S3, Supporting Information). Based on the shape memory effect of EP and the photothermal effect of magnetic IPs, the as-prepared microcones could be noncontact all-in-situ reversibly tuned between the upright and tilted state under the NIR irradiation and MF actuation (Figure 1b; Movie S1, Supporting Information). The detailed tuning procedures were described below. At first, the SMP microcones were in the original upright state (permanent state). When the NIR light was turned on, the doped IPs could convert light energy into heat energy and the sample temperature increased to the $T_g$ of EP. Then the upright microcones would be bent when an additional MF was applied simultaneously. After removing the NIR and MF in sequence, the microcones could remain in a bent state (temporary state). Finally, the bent microcones would return to their upright state in an overwhelmingly short time when the NIR light was turned on again. The external MF used in this experiment was generated by two jointed square NdFeB permanent magnets under the sample. The specific measurement method of magnetic flux density and the variation trend of magnetic flux density are shown in Figures S4 and S5 in the Supporting Information, respectively. By simulating the MF of two jointed magnets (Figure 1c, detailed simulation process can be found in the Supporting Information), it can be found that the MF in the junction is stronger ($\approx$ 340 mT) and the bending angle of the microcone can be precisely. Figure 1d–f shows the different states of the microcone array during the tuning process, and it should be noted that the microcone could not bend under MF without NIR (Movie S2, Supporting Information). Additionally, the bending state of microcone with different heights and IPs contents were systematically investigated by controlling the magnetic flux density (Figure 1g.h). The results indicate that the tilted state of microcones could be precisely controlled under different magnetic flux densities.

2.2. Characterization of SMP Microcones

Compared with photolithography which needs various masks and multistep processes,\cite{3,22,23} femtosecond laser drilling is a mask-free, size-designable, and one-step ablation method. So, different heights of the microcone could be obtained by adjusting the laser machining parameters. It can be seen from Figure S6 in the Supporting Information that the height of microcone increases with the pulse number increasing under a certain laser power. Based on the convenient one-step femtosecond laser micro-drilling method, the microcones with different spaces could also be precisely and freely controlled (Figure S7, Supporting Information). Subsequently, the relationship between the bending angle of microcone with different heights and magnetic flux density was systematically explored (Figure 2a). Especially note the microcone with fixed space (300 µm) and concentration of IPs ($\approx$ 50 wt%). Then the different bending angles ($\beta$, defined in Figure 2a) were obtained by varying the magnetic flux density. While under the same magnetic flux density, the higher the microcone is, the greater the bending angle is. The microcones with a height of about 560 µm were used in the following experiment. Then we quantitatively studied the relationship among the IPs concentration, magnetic flux density, and bending angle under the NIR irradiation (Figure 2b). When $C = 50$ wt%, the bending angle increases from 0° to 54° with the increase of magnetic flux density. In addition, a higher IPs concentration generally results in a stronger bending angle when the magnetic flux density was constant. It is worth noting that when the IPs concentration is zero, no matter how the magnetic flux density changes, the microcone would not bend. In order to investigate the relationship between IPs concentration and photothermal performance, the SMP
substrates (15 × 15 × 2 mm³) with different IPs were chosen as experimental subjects, and the temperature-time curves were carried out when the NIR light was illuminated at 5 cm above the sample (Figure 2c). It can be seen that the surface temperature remained at ≈22°C without any thermal response when the SMP substrate with no IPs (C = 0 wt%). In contrast, the temperature would quickly increase above Tg (≈40°C, Figure S8, Supporting Information) under the NIR about 10 s of the SMP substrates with a high IPs concentration (10, 30, and 50 wt%, Figure S9, Supporting Information) and then it dramatically decreased to the room temperature when the light was turned off. There will be thermal diffusion in the SMP substrate (Figure S10, Supporting Information), so it will take a little longer for the temperature to rise above the Tg. It is worth mentioning that the tilted microcones (C = 50 wt%) could recover their original upright state in a very short time (Figure 2d; Movie S3, Supporting Information) and the recovery time of bending microcone with different angles was also explored in Figure 2e. In addition, the recovery time of the tilted microcones with C = 10 and 30 wt% was also very short, similar to that of the microcone with C = 50 wt%. The NIR
is directly irradiated to the microcone surface with less thermal diffusion. Therefore, the microcones with a maximum bending angle (≈54°) could recover their original state within 2 s. Finally, the fatigue test of shape memory polymers was performed and the SMP microcones were still shown a great shape memory ability after 100 cycles (Figure 2f), which shows great potential for practical applications. Especially note that the microcones with IPS of 10 and 30 wt% still have good fatigue resistance.

2.3. Mechanism of Droplet Unidirectional Transport

The tilted SMP microcone surface has superhydrophobic property (≈150°, Figure S11a, Supporting Information) and anisotropy of sliding angle (≈25° and ≈37°, Figure S11b,c, Supporting Information). Inspired by this anisotropic sliding ability, droplet unidirectional transport could be achieved on the tilted SMP microcone surface (Figure 3a). When the droplet moved to the right side with the lower sample movement, the upper single microcone did not have enough adhesion force ($F_{\text{driven}}$) to drag the droplet against the tilted direction (Figure 3a ii,iii). In contrast, when the droplet moved to the left side, the $F_{\text{driven}}$ was big enough to make the droplet move along the tilted direction (Figure 3a iv–vi). The mechanism of droplet unidirectional transportation is mainly related to the difference of adhesion force along and against the tilted direction, which is analyzed by both experimental and theoretical calculation.

Adhesion test set-up and test procedure are described in Supporting Information Figure S12 in the Supporting Information. Figure 3b shows the experiment result of the adhesion
force test between the water droplet and tilted microcones along and against the tilted direction. It can be calculated from Figure 3b that the maximum adhesion force along and against the tilted direction is about 5.5 and 9.13 \( \mu \text{N} \), respectively. Then we constructed the corresponding 3D models to theoretically illustrate the variation of contact state between the microcones and the droplet during the sliding process. When the droplet was dragged along the tilted direction (Figure 3c), it was subjected to a right-hand driven force \( F_{\text{driven}} \) and also a left-hand adhesion resistance \( F_{a1} \). On the contrary, when the droplet was dragged against the tilted direction (Figure 3d), it was subjected to a left-hand driven force \( F_{\text{driven}} \) and also a right-hand adhesion resistance \( F_{a2} \). According to the previous study, \[ 36 \] the magnitude of \( F_{a1} \) and \( F_{a2} \) mainly depends on the length of TCL, which could be described by the following equation

\[ F = \gamma \times L_{\text{TCL}} \]  

where \( \gamma \) is the surface tension of liquid and \( L_{\text{TCL}} \) is the length of the TCL. It is worth noting that a shorter TCL with five circular shapes was formed (diameter: \( d_1, d_2, d_3, d_4, d_5 \)) when the droplet was dragged along the tilted direction (Figure 3c). In the opposite direction (Figure 3d), a long TCL with four circular shapes (diameter: \( D_1, D_2, D_3, D_4 \)) and one linear shape (2L) was approximately formed. Herein, the \( F_{a1} \) and \( F_{a2} \) was calculated about \( \gamma \pi (d_1 + d_2 + d_3 + d_4 + d_5) \) and \( \gamma \pi (D_1 + D_2 + D_3 + D_4) + 2L_{\text{L}} \), respectively. For the sake of simplicity in discussions, we assumed that \( D \) is equal to \( d \), so the ratio of \( F_{a2} \) and \( F_{a1} \) could be simplified as \( 0.8 \pi (2L/5 \pi d) \), where \( L = h/3 \), and detailed calculation process can be found in Supporting Information. According to the SEM of the microcone, the height \( (h) \) and diameter \( (d) \) of the microcone were about 562 and 45 \( \mu \text{m} \), respectively, so we could obtain that \( F_{a2} \approx 1.33 F_{a1} \). It is worth noting that the difference of adhesion force along and against the tilted direction through theoretical calculation is consistent with the experimental results within the error range, which is mainly related to the variation of the contact length of the triple contact line.

2.4. Applications of SMP Microcone for Multifunctional Droplet Manipulation

By utilizing the tilted SMP microconed surface, we designed a complete set of the experimental platform that contained droplet unidirectional transport, droplet capturing/releasing, and droplets reaction, to demonstrate its potential application in the multifunctional manipulation of the microscale objects (Figure 4a). We chose sodium hydroxide (NaOH) and bromothymol blue (BTB) as the reaction liquid for convenient observation. First, the NaOH droplet was placed on the tilted
microconed surface ($T = 0$ s), while the BTB droplet was placed on another smooth substrate. Then we used another tilted microcone surface (upper sample) to drag the NaOH droplet from the left side to the right side ($T = 6–7$ s). Subsequently, the NaOH droplet was lifted by the upper sample under the effect of adhesion force ($T = 13$ s) and was transported to the position above the BTB droplet. At the same time, the upper tilted microcone became upright after turning on the NIR ($T = 14$ s), and the adhesion force decreased. Finally, the NaOH droplet was released from the upper sample under the effect of droplet gravity and reacted with the BTB droplet ($T = 15$ s; Movie S4, Supporting Information). The mechanism of the droplet lifting and releasing process is mainly related to the variation of the adhesion force induced by the length of TCL (Figure 4b,c). It is worth noting that compared with the capture function in recent works,[27,28] the size of the manipulation object in our work is much smaller ($\approx \mu$L; Figure S1, Supporting Information) and the object type is also different (their is solid and our is liquid), which may have potential applications in the field of biomedicine.[37] For example, we could precisely guide
the medicated droplet to the specified microwell as we need (Figure S13, Supporting Information).

Besides the above-mentioned applications, selective droplet release by using tilted microcones was demonstrated, which is difficult for common PDMS micropillars,[38,39] and the schematic diagram is shown in Figure 4d. First, the tilted IPs-doped microcones, that were realized under the existence of NIR and MF, capture two droplets and then lift the two droplets vertically from the substrate. Next, the microcones adhering to two droplets are moved to another receiver substrate. Subsequently, an intended microcone is selectively heated via a NIR light. The tilted microcone will quickly recover its original upright state after heated above its $T_g$, thus achieving the selective releasing of droplet 1 and 2, respectively. When the microcone becomes bent again, the next round of droplet selective transfer can be performed. The sequential screenshots of the selective release process are shown in Figure 4e (Movie S5, Supporting Information). In order to in situ capture the changing process of the microcone, we added a filter for NIR in front of the CCD. This method is expected to achieve selective release manipulation of large area droplet arrays and has potential practical applications in the field of biodetection, microreaction, and so on.

2.5. Other Applications: Rewritable Display, Information Encryption, and Temperature Switch

Based on the shape memory effect of SMP, we demonstrated a rewritable magnetic microcone array with selective bending as shown in Figure 5. The magnetic microcone array is made

Figure 5. Demonstration of the rewritable magnetic microcone array with selective bending. a) Schematic diagrams showing the mechanism of rewritable microcone array. b) Top view of microcone array in different states (upright, bending, selective bending). c) The optical images of the states (upright, bending, selective bending). d) Microcone array to achieve multiple pattern display, such as Arabic numerals “1”, “3”, and “4”.
of EP and IPs, which can be reversibly converted into three different states (upright, bending, selective bending) when stimulated by different external conditions, thus enabling erasable functionality. The conditions required for a reversible transition between the three states are depicted in detail in Figure 5a and Movie S6 (Supporting Information), and a reversible transition can be achieved between any two states. The upright microcone could be easily tilted by applying the MF and NIR, while the tilted microcone could restore to the upright state by applying the NIR. Figure 5b,c is a top-view schematic and of the optical photographs of three states (upright, bending, selective bending). Finally, we wrote the different Arabic numerals of “1,” “3,” and “4” on the microcone array after each time of erasing (Figure 5d).

Additionally, the information encryption can be achieved through the rational design of the microcones with iron particles (magnet-responsive EP + IPs) and carbon black (magnet-nonresponsive EP + CB), as shown in Figure 6a. First, we attach the mask plate with specific information onto the template. The IPs doped EP was then used to fill the specific microholes. In the next step, carbon black (CB) doped EP was poured over the template without a mask to fill the remaining microholes. The reason for choosing CB here is that its color is similar to that of IPs so that the color of the obtained microcone remains the same for the purpose of encryption. After sequentially curing and peeling off, the encrypted microcone arrays were obtained. In the presence of NIR and MF at the same time, the microcone containing IPs will bend, while the microcone containing CB will not change, so the encrypted information will be displayed. The decrypted information reverts to the encrypted state after irradiation by NIR. The whole decryption and encryption process of the information was recorded in Movie S7 in the Supporting Information. Figure 6b,c display the SEM images (top view) of information encryption and decryption of the letter “U,” respectively. In addition, other messages can also be encrypted and decrypted, such as the messages encrypted in Figure 6d,e are the letter “F” and the Arabic numeral “11,” respectively.

Finally, a kind of reusable temperature switch based on SMP microcones was also proposed as shown in Figure S14a in the Supporting Information. Special note, a gold film was deposited on the microcones to impart its conductivity. Initially, the microcones were tilted and could not touch the upper electrode, so the circuit was in the nonconduction state, and the indicator light was off. When the ambient temperature (hot plate) exceeded the $T_g$ of SMP, the tilted microcone would become upright and come into contact with the upper electrode to form a conducting circuit, so the indicator light would be on. Additionally, the microcones could recover the original tilted state by applying an MF, so the temperature switch based on the SMP is repeatable through periodic applying/removal MF. The detailed process was shown in Figure S14b and Movie S8 in the Supporting Information. When the ambient temperature was about 34°C ($<T_g$), the microcones were in a tilted state and the indicator light was off. The microcones would gradually become upright when the temperature reached the $T_g$ ($\approx 40\, ^{\circ}\mathrm{C}$) of SMP, and the light was on. When an external MF was applied at this time, the microcones would become tilted again and the indicator light

![Figure 6. Application of information encryption and decryption. a) The process of message encryption and decryption. Some microcones are made of epoxy resin mixed with iron particles (EP + IPs), which can be reversibly tuned by MF and NIR. Other microcones are made of epoxy resin mixed with carbon black (EP + CB), which is not responsive to MF. b,c) The top-view SEM images of information encryption and decryption on the bendable microcones “U” pattern. Reversible conversion between encryption and decryption can be achieved through external stimuli (NIR and MF) on microcones with EP + IPs. d,e) SEM images (top view) of different microcone arrays with EP + IPs after information decryption. The encrypted messages are the letter “F” and the Arabic numeral “11.”](image-url)
would turn off. After removing the MF (T < T_f), the microcones remained tilted and the temperature switch was restored. It is worth noting that the indicator light in the experiment can be replaced by an alarm bell to achieve a better alarm effect.

3. Conclusion

In summary, we have demonstrated a kind of IPs-doped SMP microcone array with noncontact all-in-situ reversible reconfiguration based on dual photothermal and magnetic responses for multifunctional droplet manipulation and temperature switch. The SMP microcones are simply realized by combining femtosecond laser ablation and duplicate template method so that the height and the tilted angle of microcones could be precisely tuned by adjusting the laser pulse number and the magnitude of MF. It is found that the droplet on the tilted microconed surface shows anisotropic sliding ability along and against the tilted direction, which is induced by the variation of TCL length between the microcone and the droplet. Furthermore, we have achieved the multifunctional manipulation of the microdroplet on an integrated test platform by utilizing the tilted SMP microcone surface, including droplet unidirectional transport, droplet capture, selective release, and droplet reaction. Finally, information encryption, rewritable display, and temperature switch were also demonstrated. We believe the IPs-doped SMP microconed surface with noncontact all-in-situ dual photothermal and magnetic responses may have great potential applications in the future design of stimuli-responsive devices.

4. Experimental Section

Materials: The iron particles (>99.99% purity) with a mean size of 5 μm (Figure S15, Supporting Information) were purchased from Guangzhou Metal Material Tech. Co., Ltd. The NIR with a wavelength of 808 nm was chosen for this experiment. The epoxy resin precursor and curing agent were obtained from Ji’an Yifu E-commerce Co., Ltd. The NdFeB magnets (40 mm × 40 mm × 2 mm) used in the experiment were purchased from Shanghai Zehe Mechanical & Electrical Co., Ltd. The release agent was provided by Wuhan Jieguan Biotechnology Co., Ltd. China. Temperature change during the NIR irradiation process was measured with a digital Gauss meter (HM-100, Huaming instrument Co., Ltd. China). Magnetic flux density was measured by the FLIR ONE thermal camera. The magnetic field was tuned by adjusting the laser pulse number and the magnitude of MF. It is found that the droplet on the tilted microconed surface shows anisotropic sliding ability along and against the tilted direction, which is induced by the variation of TCL length between the microcone and the droplet. Furthermore, we have achieved the multifunctional manipulation of the microdroplet on an integrated test platform by utilizing the tilted SMP microcone surface, including droplet unidirectional transport, droplet capture, selective release, and droplet reaction. Finally, information encryption, rewritable display, and temperature switch were also demonstrated. We believe the IPs-doped SMP microconed surface with noncontact all-in-situ dual photothermal and magnetic responses may have great potential applications in the future design of stimuli-responsive devices.

Conflict of Interest

The authors declare no conflict of interest.

Supporting Information

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

Acknowledgements

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Data Availability Statement

The data that supports the findings of this study are available in the supplementary material of this article.

Keywords

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