nature materials

LETTERS

PUBLISHED ONLINE: 7 AUGUST 2011 | DOI: 10.1038/NMAT3090

Programming magnetic anisotropy in polymeric microactuators

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Polymeric microcomponents are widely used in microelectromechanical systems (MEMS) and lab-on-a-chip devices, but they suffer from the lack of complex motion, effective addressability and precise shape control[1,2]. To address these needs, we fabricated polymeric nanocomposite microactuators driven by programmable heterogeneous magnetic anisotropy. Spatially modulated photopatterning[3] was applied in a shape-independent manner to microactuator components by successive confinement of self-assembled magnetic nanoparticles in a fixed polymer matrix. By freely programming the rotational axis of each component, we demonstrate that the polymeric microactuators can undergo predesigned, complex two- and three-dimensional motion.

In the fabrication of miniaturized mechanical components for MEMS and lab-on-a-chip systems, polymeric materials have attracted a great deal of attention, as they are lightweight, flexible and easily processed. Various engineered polymeric materials, such as electroactive polymers, carbon nanotube composites and liquid crystal polymers, are currently under investigation for implementation as active actuating components in microscale devices[4–9]. However, most of these actuation schemes are based on simple translation or deformation of active materials, lacking the capacity for the sophisticated multi-directional movement controls required for integrated actuating elements with complicated motion. Furthermore, most active polymeric actuators must be physically wired to additional inorganic electrodes or control devices, which makes the fabrication process complicated and also often limits freedom in actuation range and surrounding media.

Magnetically actuated microdevices, on the other hand, can be controlled without any additional apparatus having physical contact with the actuator. As long as the actuation environment is magnetically transparent, they can be operated in many different media, including air, vacuum, conducting and non-conducting liquids[10,11]. The flexibility of magnetic actuation in conjunction with the advantages of polymeric materials makes magnetically driven metal–polymer composites attractive for many applications, such as magnetic beads in bioassays and magnetic gating valves in microfluidic control systems[12–20]. However, the conventional magnetic microcomponents reported so far have only offered simple motions such as monotonous translation and rotation, owing to intrinsic limitations in their actuation principle. There are two types of magnetic forces conventionally used: the magnetic translational force and the magnetic torque. The magnetic translational force has all parts of a microcomponent moving in only one direction, heading towards the positive magnetic field gradient[14,15,21]. The utilization of this force presents inherent difficulties in the design of multi-directionally moving microcomponents because it becomes harder to control the spatial variation of the magnetic field at the microscale. In regards to alternate motions using magnetic torque, movement is limited to simple rotation and bending[16,22–26]. Patterning multiple magnetic moments with different directions in a microstructure is very difficult to achieve using a conventional micromachining process. More fundamentally, if each functional part of a microcomponent has a differently oriented magnetic moment, they will attract each other without the external magnetic stimulation, resulting in unwanted aggregation of the microcomponents even before the actuation. Therefore, to achieve complex actuations with large deflection and precise shape control, we should investigate other energy transduction mechanisms and develop a new magnetic material system to apply such mechanisms.

A magnetically anisotropic material attempts to align its magnetic moment with one of the easy axes, which are the energetically favourable directions of the magnetization. By using a superparamagnetic material, which aligns its magnetic moments with the external magnetic field direction without spontaneous magnetization and magnetic hysteresis, multi-directional movement in a microactuator is possible. Each part of a superparamagnetic microactuator gains a different magnetic anisotropy, and thus can rotate towards its own magnetic easy axis in response to the application of an external magnetic field. This property, however, has not yet been used because the magnetic anisotropic torque is relatively small compared with other magnetic forces. Furthermore, the direction of an intrinsic magnetic easy axis is firmly determined by the shape and the magnetocrystalline structure of the magnetic material, and thus cannot be controlled in natural magnetic materials.

Here we present a new magnetic nanocomposite material system and *in situ* fabrication process that is not shape limited and allows the programming of heterogeneous magnetic anisotropy at the microscale. The key idea is to combine the self-assembling behaviour of superparamagnetic nanoparticles, which have stronger magnetization than that of general paramagnetic materials, with a spatially modulated photopatterning process. By repetitively tuning the nanoparticle assembly and fixing the assembled state using photopolymerization, we fabricate microactuators for which all parts move in different directions under a homogeneous magnetic field. To show the feasibility of our concept, we demonstrate polymeric nanocomposite actuators capable of two-dimensional and three-dimensional complex actuations that have rarely been achieved using conventional microactuators. Our approach greatly simplifies the manufacturing process and also offers effective rules for designing novel and complex microcomponents using a nanocomposite material with engineered magnetic anisotropy.

Figure 1 shows a schematic representation of the fabrication and actuation of the magnetic actuator. The polymeric nanocomposite material used here is based on a combination of a

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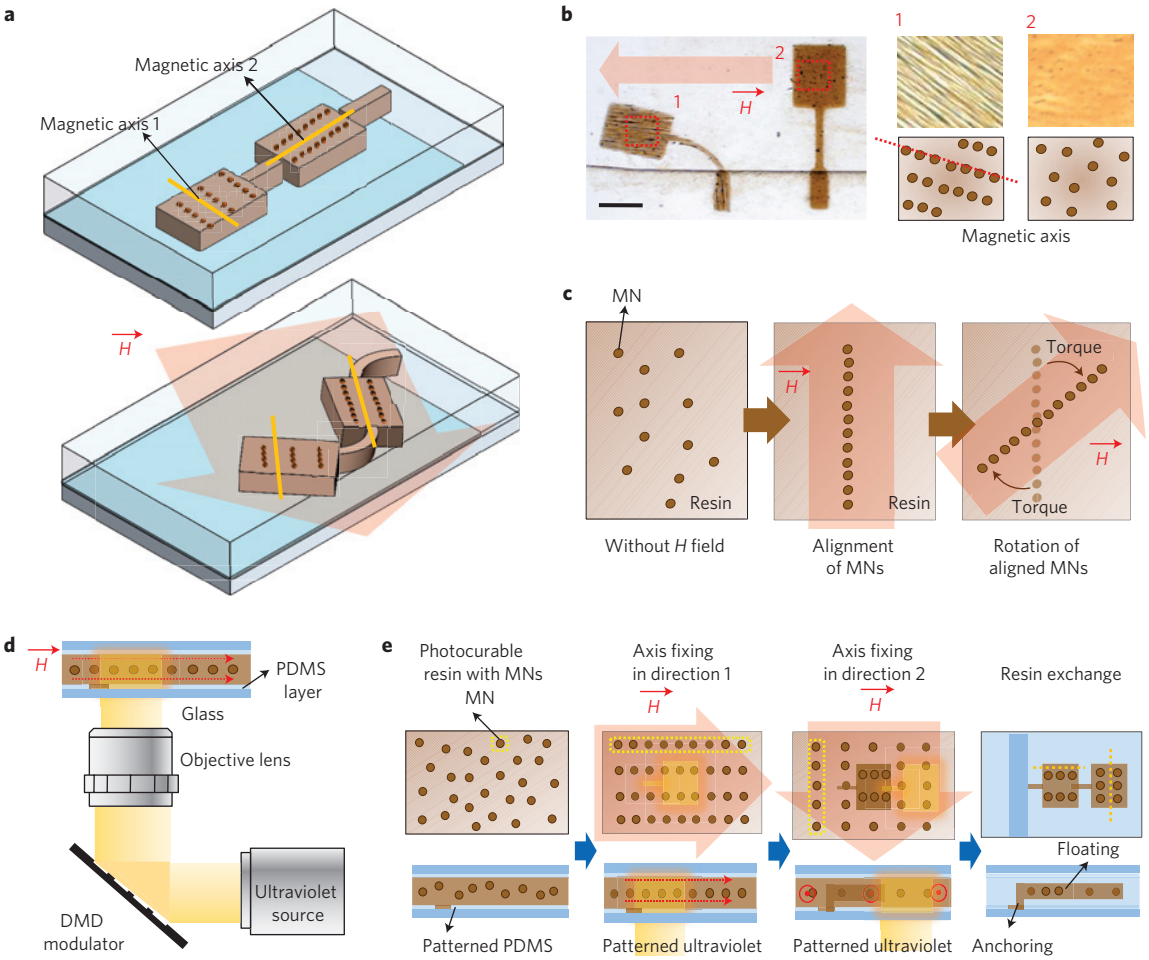

Figure 1 | Schematic of the fabrication of polymeric magnetic microactuators. a, The movement of a magnetic actuator. The magnetic actuator possesses two different magnetic axes, therefore it actuates in a zig-zag conformation when the magnetic field is applied. **b**, Actuation of a simple magnetic cantilever. Under a homogeneous magnetic field the magnetic cantilever, which contains self-assembled magnetic nanoparticles (MNs), only bends towards the field line. (Scale bar 50 μm). **c**, Magnetic nanoparticle self-assembly. Randomly dispersed magnetic nanoparticles are self-assembled along the uniform magnetic field line to minimize magnetic dipole interaction. When the magnetic field direction is changed, the assembled nanoparticles rotate along the field line. **d**, Maskless lithography set-up. The set-up is composed of an ultraviolet light source, a digital micromirror device (DMD) modulator and objective lenses. The light is patterned through a DMD modulator and focused on the microfluidic channel, polymerizing the resin. This process enables fabrication of microstructures with various shapes as well as the ability to fix the magnetic nanoparticles in the structure. **e**, Magnetic axis fixing process. A mixture of photocurable resin and magnetic nanoparticles is injected into the microfluidic channel. A magnetic field is applied in direction 1, and the microstructure embedding with aligned magnetic nanoparticles in direction 1 is fabricated using maskless lithography. A magnetic field is applied in direction 2, and another microstructure embedded with aligned magnetic nanoparticles in direction 2 is attached to the previous structure using the same process. Finally, the resin is exchanged and the fabrication process is completed. The completed actuator is anchored at one end and free-floating at the other end.

photocurable polymer and superparamagnetic nanoparticles. The superparamagnetic nanoparticles consist of several single-domain magnetite nanocrystals, their core capped with a negatively charged material in a silica shell[27]. Without an external magnetic field, the nanoparticles are randomly dispersed in a photocurable liquid resin. When the external magnetic field is applied, the superparamagnetic nanoparticles self-assemble, forming chain-like nanostructures along the magnetic field lines to minimize the magnetic dipole interaction energy of the system (Fig. 1c). Remarkably, when the magnetic field direction is changed, all chains rapidly rotate or realign along the changed magnetic field direction (Fig. 1c). Once polymerized, the chains induce cooperative torque[28], which is the driving force of microstructure actuation.

Figure 1d is the experimental set-up composed of an optofluidic maskless lithography system[29], which enables microstructure fabrication of various shaped microstructures, a microfluidic channel, which determines the geometric dimension of the microstructure, and a permanent magnet, which generates an external magnetic field. With this system, a sequential process is used to manufacture miniaturized polymeric actuators. First, a glass substrate is coated with polydimethylsiloxane (PDMS), which is responsible for the inhibition layer[30] used for generating the free-floating part of the component. The coated PDMS is partially removed to create the anchoring region where the structures are permanently attached to the intrinsic glass substrate. Then, the microfluidic channel is attached on the PDMS coated glass substrate and filled with a mixture of poly (ethylene glycol) diacrylate (PEGDA), photoinitiator and superparamagnetic nanoparticles. A homogeneous magnetic field is applied across the microfluidic channel, which causes the superparamagnetic nanoparticles in the resin to form chains along the direction of the applied magnetic field. The resin is photopolymerized in a tenth of a second using an optofluidic maskless lithography system, thus freezing the superparamagnetic nanoparticle alignments in the polymerized region.

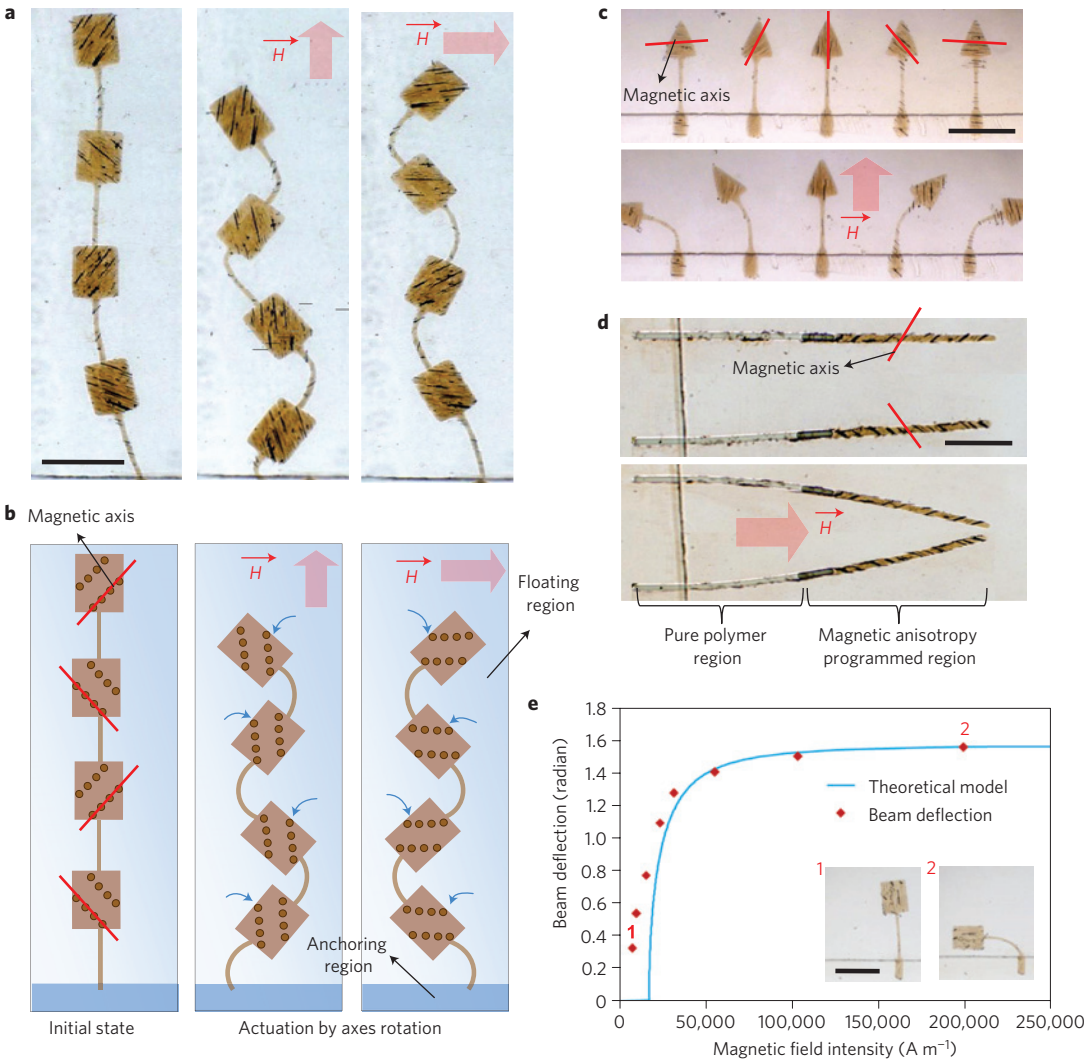

Figure 2 | Various types of actuation using programmable magnetic anisotropy. a, A microactuator containing four different easy axes recoils like a snake when a magnetic field is applied. (Scale bar 100 μm.) **b**, Schematic of the magnetic actuator shown in Fig. 2a. The magnetic nanoparticle chains play the role of the magnetic easy axis in the structure. Each part of the structure bends in different directions, owing to the alternating easy axes. **c**, Arrow-like magnetic actuator. Five identical arrow-like actuators bend in five different directions under the uniform magnetic field line. (Scale bar 50 μm.) **d**, Magnetic tweezer. The magnetic tweezer contains both a pure polymer region and a magnetic anisotropy programmed region. It tweezes under a homogeneous magnetic field. (Scale bar 50 μm.) **e**, Theoretical model and experimental result of beam deflection. Theoretical results are in agreement with the experimental result. As the magnetic field intensity increases, the beam bends at a greater angle with the same magnetic field direction. This result implies that we can regulate the actuator movement using the magnetic field direction as well as the magnetic field intensity. (Scale bar 200 μm.)

After photopolymerization, the magnetic field direction is changed, leading to the rearrangement of superparamagnetic nanoparticle chains in the remaining region along the altered magnetic field lines. Then the photopolymerization process is carried out again, creating a newly polymerized part that has a different chain direction to the previous part. This repetitive tuning and fixing is done until the microctuator manufacturing is completed. Before use, the remaining resin is exchanged for the chosen resin to provide an appropriate liquid environment for microstructure actuation (Fig. 1e). In comparison to thermal curing, the photopolymerization process is instantaneous and can fix the self-assembled nanoparticles fast enough to maintain the assembled state, allowing for high resolution and multi-exposure patterning, with the total fabrication time determined by the time necessary to change the magnetic field.

The significant advantage of our material system over conventional material systems for microsystems is that the magnetic properties of the polymeric microstructure, such as the direction of the magnetic easy axis or the amount of magnetization, are programmable. As mentioned above, the fixed alignment of magnetic nanoparticles in multiple polymerized regions creates magnetic anisotropy in the actuator. This enables a homogeneous magnetic field to independently actuate each part with the relative initial direction of chains in each component determining the final configuration of the actuator. Therefore, this principle enables the production of a variety of active polymeric microdevices by freely programming the rotational axis of each component.

Figure 2 exhibits several types of complex actuations of polymeric micro actuators which are not easily achievable using conventional materials and fabrication methods. The actuator in Fig. 2a is composed of four parts, which have different magnetic easy axes. Each part rotates with a different direction and angle, responding to the homogeneous external magnetic field, and as a result, it moves like a crawling snake as the magnetic field line changes direction (Supplementary Movie S1). The five identical actuators in Fig. 2c bend at different angles, owing to the magnetic easy axes being programmed in different directions in each

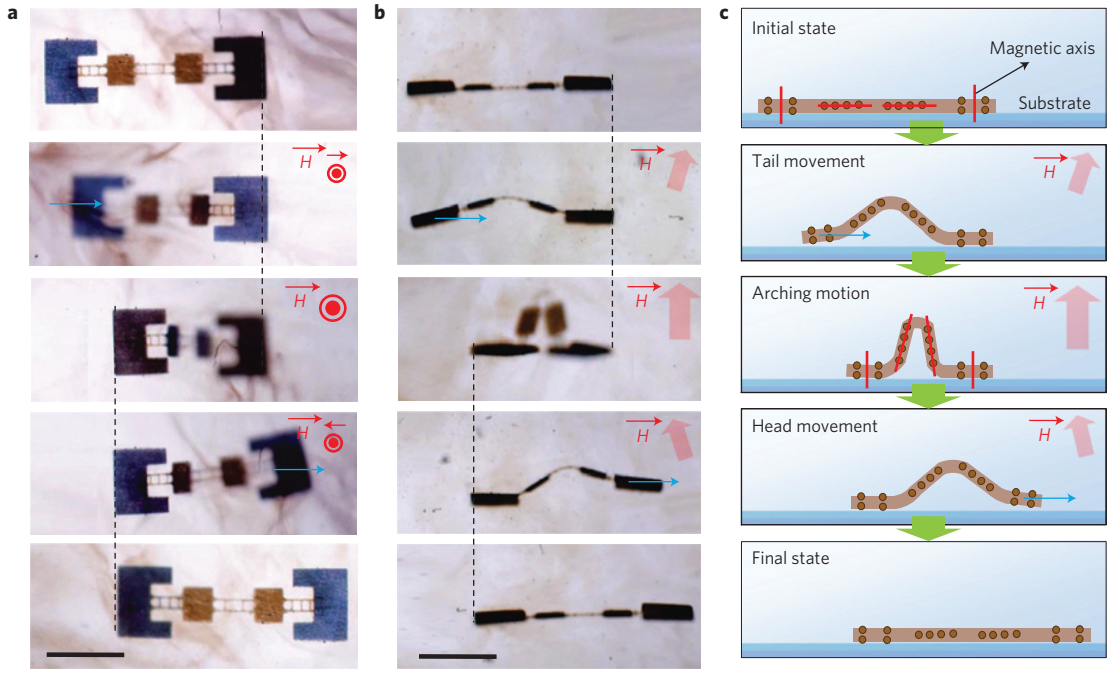

Figure 3 | Polymeric micro-looper. a,**b**, Sequential movement of a micro-looper (Scale bars 100 μm.) ((**a**): Top view, (**b**): Side view). The micro-looper creeps on the substrate as the external magnetic field is applied. First, a weak and slightly tilted magnetic field is applied. Thus, the micro-looper starts to bend and the tail moves towards the head. As the magnetic field becomes stronger and clearly vertical, the micro-looper completely arches. When the magnetic field is slightly tilted in the opposite direction, the head proceeds forward and the tail sticks to the substrate. After the magnetic field is removed, one sequential crawling process is finished. The micro-looper can crawl on the substrate as this process is repeated. **c**, Schematics of micro-looper movement. The micro-looper is composed of four bodies having different magnetic axis, such that it has various configurations according to the applied external magnetic field direction. By choosing an appropriate magnetic field direction and intensity for the desired configuration we can achieve an arching movement of the micro-looper.

actuator (Supplementary Movie S2). The magnetic microtweezer, shown in Fig. 2d, contains both a pure polymer region and a magnetic-anisotropy programmed region. These actuation examples show that, using our strategy, the design capability of polymeric magnetic actuators can be dramatically expanded (Supplementary Section S6). We can implant many magnetic axes in a single polymeric microstructure as well as combine the magnetic actuator into other types of functional polymer. In addition, the actuation is easily regulated simply by varying the direction of the external magnetic field lines instead of the magnetic field gradient, which is hardly controllable at the microscale.

The torque induced by the magnetic anisotropy can be analysed in terms of a simple analytical model (Fig. 2e). The prototype polymeric microactuator consists of a narrow cantilever beam made of photopolymerized PEGDA, which is anchored to the substrate at one end, and a free-floating polymer plate in which superparamagnetic nanoparticles are embedded. The superparamagnetic nanoparticles are arranged into one-dimensional chain-like nanostructures, and the nanostructures are uniformly distributed with constant spacings[31]. Under a uniform magnetic field, the microstructure experiences a magnetic torque on the plate due to the cooperative response of the assembled nanostructures, and a mechanical restoring force due to the thin cantilever beam deflection. These forces become equal at the equilibrium state.

The angular mechanical deflection of the microactuator at equilibrium is found to be $\varphi = K \sin 2(\varphi_{\text{initial}} - \varphi), (K = K_{\text{m}}/K_{\varphi})$, where φ_{initial} is the initial angle between the magnetic field line and the cantilever beam before stress, K_{m} is the mechanical stiffness of the thin beam and K_{φ} is the magnetic factor of the magnetic nanoparticle cluster. As the rotation of the magnetic microstructure is constrained by a mechanical spring, the magnetic torque causes the microstructure to rotate until the mechanical restoring torque of the beam spring compensates for the magnetic anisotropic torque and an equilibrium angular deflection α is attained. The magnetic anisotropic torque is induced by the magnetic dipole interaction of the aligned nanoparticles and the mechanical joint is regarded as a homogenous polymeric beam. The resulting equilibrium factor K varies according to the number of magnetic nanoparticle chains in a microstructure, the magnetic field intensity, the material property and the actuator geometry. Figure 2e shows the equilibrium deflection angle with respect to the external magnetic field intensity and this model corresponds to the experimental result. As the magnetic field intensity increases, the magnetic axis of the microstructure becomes parallel to the magnetic field direction. More detailed information is included in Supplementary Section S3.

As a practical application, we applied our material system to demonstrate the polymeric microrobot shown in Fig. 3. The polymeric microrobot is composed of two heads, two bodies and three joints. The inner two bodies have parallel magnetic easy axes and the outer two heads contain vertical magnetic easy axes with respect to the substrate. The three joints have a ladder structure to prevent unnecessary twisting (Supplementary Section S7). This microrobot engages in an arching motion through the application of an external magnetic field in a vertical direction to the substrate (Fig. 3). To convert the arching motion into the net forward movement on the substrate, we use the frictional force between a head and the substrate. First, we apply the magnetic field at a slight tilt from the perfect vertical direction to the substrate. Thus, the two bodies bend in different directions while the floating head slowly moves to the other head, at which point it sticks to the substrate. As the magnetic field lines become vertical to the substrate, the microrobot arches like a looper caterpillar. Once the magnetic field becomes slightly tilted again, the head that has been stuck to

the substrate following the initial state floats and moves forward, relaxing the arching of the bodies (Fig. 3c). Therefore, by carefully regulating the magnetic field direction, we can achieve forward crawling of the polymeric microrobot on the substrate.

In this paper, by combining the magnetic self-assembly of superparamagnetic nanoparticles and rapid photopolymerization, we have shown that one can freely programme heterogeneous magnetic anisotropy in a polymeric microstructure and make each part of the microstructure actuate in pre-programmed directions using magnetic stimuli. Rather than the fabrication of static microstructures, this programmable material allows the assembly of precise, complicated microstructures from a single flexible polymeric base. One can not only design many complex static microstructures by programming a single material system, but also create dynamic structures that react to, and affect, the environment. For example, this technology will provide a novel approach to fabricate functionalized microcomponents for advanced microfluidic systems that require fluidic control on the microscale. Furthermore, these microcomponents should enable the construction of a reconfigurable microrobot that changes its shape based on the function it needs to perform, simplifying the fabrication and operation methods that are required. Therefore, this material would make possible a host of applications that need smart microstructures.

Methods

Materials. The nanocomposite material is composed of superparamagnetic colloidal nanocrystal clusters (CNCs) and a photocurable resin monomer solution (Supplementary Section S1). For the polymeric microstructure generation, we used poly (ethylene glycol) diacrylate (PEGDA, Sigma-Aldrich, average $M_n \sim 258$) mixed with 10 wt% of photoinitiator (2,2-dimethoxy-2-phenylacetophenone, Sigma-Aldrich) as a photocurable resin. In the magnetic nanocomposite microstructure demonstration, superparamagnetic CNCs dispersed in the photocurable resin were used for the magnetic anisotropy programming region. The liquid for the actuation environment is also a solution of 10:1 poly(ethylene glycol) diacrylate (PEG-DA, Sigma-Aldrich, Mn = 258) : 10 wt% of photoinitiator (2,2-dimethoxy-2-phenylacetophenone, Sigma-Aldrich).

Substrate surface coating. Polydimethylsiloxane (PDMS) elastomer (Silgard 184, Dow Corning) was poured onto a glass substrate, and spin coated for 30 s at 3,000 r.p.m. The coated substrate is then thermally cured for 10 min on a 150 °C hot plate. The hardened PDMS surface was patterned chemically or mechanically to generate the actuator fixing region (Supplementary Section S4).

Magnetic field generation and maskless lithography. Polymeric microactuators were generated by photopolymerization using a spatial light modulator (SLM, Supplementary Section S2). An optical microscope (IX71, Olympus), ultraviolet light source (200 W, mercury–xenon lamp, Hamamatsu) and digital mirror device (DMD, Texas Instrument) were aligned to expose an ultraviolet pattern, as shown in Supplementary Fig. S3. A ×10 microscope objective lens having a numerical aperture (NA) of 0.28 projects the computer-controlled image pattern on the MEMS SLM to the final object plane with a demagnification factor of approximately 8.9. As the pitch of the micromirror array is 13.68 μm in the SLM plane, the pixel size in the object plane is approximately $1.54 \times 1.54\,\mu m^2$. The optical projection system and imaging optics share the same objective lens and their light paths are separated by a beam splitter. Using this system, we can reliably synthesize polymeric microstructures with a single pixel resolution ($\sim 1.54 \times 1.54\,\mu m^2$) at 0.1 s exposure time. A magnet was placed above the microfluidic device and calibrated using a gaussmeter (455 DSP Gaussmeter, Lakeshore).

Superparamagnetic CNCs synthesis. A $FeCl_3$/diethylene glycol (DEG) stock solution is prepared by dissolving 20 mmol $FeCl_3$ in 50 ml of DEG and heating it to 120 °C for 1 h under a nitrogen atmosphere. A NaOH/DEG stock solution is also prepared by dissolving 125 mmol of NaOH in 50 ml of DEG and heating it to 120 °C for 1 h under a nitrogen atmosphere. Superparamagnetic Fe_3O_4 core nanoparticles with tunable size are synthesized using a high-temperature hydrolysis reaction with polyacrylic acid (PAA) as a surfactant. In a typical synthesis, a mixture of 288 mg of PAA, 1 ml of $FeCl_3$ stock solution and 15 ml of DEG is heated to 220 °C in a nitrogen atmosphere for 90 min with vigorous stirring to form a transparent, light yellow solution. Then 1.8 ml of NaOH/DEG stock solution is injected into the above solution, which slowly turns black after about 2 min. The resulting mixture is further heated for 1 h to yield approximately 120 nm Fe_3O_4 colloids. The synthesized Fe_3O_4 colloids are first washed with a mixture of deionized (DI) water and ethyl alcohol, then with pure water several times, and finally dispersed in 3 ml of DI water. The silica coating of Fe_3O_4nanoparticles is performed using a modified Stöber method. Typically, 3 ml of a Fe_3O_4 colloid solution is mixed with ethyl alcohol (20 ml), NH_4OH (28%, 1 ml). Tetraethyl orthosilicate (TEOS, Aldrich) (0.1 ml) is injected into the solution. After 20 min, TEOS (0.1 ml) is injected one more time. After the reaction has finished, the Fe_3O_4/SiO_2 particles are collected by magnetic separation, followed by rinsing with ethanol three times, and finally dispersed in 3 ml of EtOH.

Received 15 February 2011; accepted 5 July 2011; published online 7 August 2011

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Acknowledgements

This work was partly supported by the Korea Science and Engineering Foundation (KOSEF) grant funded by the Korea government (MEST) (2011-0016491), supported by the Korea Science and Engineering Foundation (KOSEF) grant funded by the Korea government (MEST) (2011-0000545), and supported by Global Frontier Project grant (NRF-M1AXA002-2010-0029797) of the National Research Foundation funded by the Ministry of Education, Science and Technology of Korea.

Author contributions

Jiyun Kim, S.E. Chung and S.K. designed the experiment. Jiyun Kim and S.E. Chung performed the experiments and analysis. S-E. Choi synthesized the magnetic material and gave key advice for the experimental design. H.L. and Junhoi Kim gave key advice for the experimental design. Junhoi Kim also investigated the magnetic properties of the nanoparticles.

Additional information

The authors declare no competing financial interests. Supplementary information accompanies this paper on www.nature.com/naturematerials. Reprints and permissions information is available online at http://www.nature.com/reprints. Correspondence and requests for materials should be addressed to S.K.