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On-chip Magnetic 3D Soft Microactuators Made by Gray-scale Lithography

Yoko Yamanishi, Shinya Sakuma, Yuki Kihara, Fumihito Arai, *Member, IEEE*

***Abstract*—In this paper we describe a novel fabrication method of three dimensional polymeric magnetically driven microtools (MMT) for non-intrusive and no contamination experiments on a chip. In order to obtain precise and complicated three dimensional patterns of magnetically driven 3D microtools, a grayscale photolithography technique has been applied by making good use of thick nega-photoresist as sacrificed mold. By controlling an amount of ultraviolet light with a gradation of gray tone mask, we have fabricated smoothly curved (100 μm gap) object without steps which tend to be appeared in case of conventional layer by layer photolithography techniques. A wide range of on-chip application of microactuators can be proposed by using a softness of polymer-based 3D MMT. For example, microrotor was operated as miromixer and micropump, also microloader was actuated by the magnetic and fluidic force. The surface of the 3D MMT was fabricated with a group of spiked pattern to reduce the contact area between PDMS (Poly dimethyl siloxane) microchannel and PDMS-based 3D MMT.**

The produced 3D MMT can be applied to complicated on-chip manipulations of sensitive materials such as cells.

I. Introduction

THIS paper presents a fabrication and applications of 3D magnetically driven microtools (MMT) which can be operated as 3D microrotor and microloader. In the past study [1-4] we have reported novel magnetically driven polymeric microtool for non-intrusive and no contamination experiments on a chip. The composites were formed by suspending magnetite nanoparticles in polydimethylsiloxane (PDMS). In order to obtain precise and complicated pattern of magnetically driven mictotools, a photolithography technique has been applied by making good use of thick KMPR-1050 photoresist as sacrifice-mold [3-4]. The main features of them are 1. fabrication of any 2D shape, 2. soft and biocompatible, 3. no stiction (Teflon coating), 4. mass production with low cost. We have provided many examples of 2D microactuator such as stirrer, valve and so on. However it was desired to have three-dimensional microactuators which generate larger actuation force and which allow more complicated motion to manipulate cells in microchip.

For this paper, we have established novel fabrication method of 3D MMT by means of gray-scale lithography. Conventionally, fabrication of three-dimensional structure has done by, for example, photosensitive resin in a layer-by-layer process [5] or LIGA process with X ray [6], whilst gray-scale lithography technique is proposed to fabricate smooth curve simply and directly with low cost and mass productive [7]. The gray-scale lithography has been mainly introduced to produce the optical component such as lenses [8, 9]. Gray-scale lithography use a mask with a gradation of grey tone to express the curvature of the structure which control the amount of the parallel ultra violet light or the etching depth of the photoresist. Although the gray-scale photolithography has a limitation (about 400 μm [10]) of the depth of etching for a single exposure, this limitation of depth is enough to produce a microactuator to put in the microchip. Moreover the gray-scale lithography has an advantage to fabricate smooth curve line with high resolution compare to the layer-by-layer process. The fabrication method of MMT in the past work was difficult to apply to multi-step exposure due to the bubbles in the KMPR photoresist which lose accuracy of fabrications. Thus, this gray-scale photolithography provides many possibilities to fabricate 3D microtools with orbital shapes.

Gray-scale lithography required a gray tone mask with high-accuracy. It is well-known HEBS mask [11] as high resolution gray mask, however we have used a high resolution image setter film (3600 dpi) to produce the emulsion mask by reducing projection exposure method with 1/20 reduction lens system due to low cost and simple process of fabrications. This process used the glass substrate instead of Si substrate for back side exposure technique.

For the current study we have firstly applied gray-scale lithography technique on KMPR thick nega-resist and fabricated 3D MMT with high accuracy and operate them as microactuators in microchip.

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Y. Yamanishi is with Dept. of Bioengineering and Robotics, Tohoku University, 6-6-01, Aramaki-Aza-Aoba, Aoba-ku, Sendai, Miyagi-ken, 980-8579, Japan (phone: +81-22-795-6968; fax: +81-22-795-7035; e-mail: yoko@imech.mech.tohoku.ac.jp).

S. Sakuma is with with Dept. of Bioengineering and Robotics, Tohoku University, 6-6-01, Aramaki-Aza-Aoba, Aoba-ku, Sendai, Miyagi-ken, 980-8579, Japan (e-mail: sakuma@imech.mech.tohoku.ac.jp).

Y. Kihara is with with Dept. of Bioengineering and Robotics, Tohoku University, 6-6-01, Aramaki-Aza-Aoba, Aoba-ku, Sendai, Miyagi-ken, 980-8579, Japan (e-mail: kihara@imech.mech.tohoku.ac.jp).

F. Arai is with Dept. of Bioengineering and Robotics, Tohoku University, 6-6-01, Aramaki-Aza-Aoba, Aoba-ku, Sendai, Miyagi-ken, 980-8579, Japan (e-mail: arai@imech.mech.tohoku.ac.jp).

II. FABRICATION OF 3D MMT

A. Theory of Gray-scale Photolithography

Figure 1 shows the concept of Grayscale photolithography. A thick KMPR-1050 photoresist (Kayaku MicroChem Co., Ltd) was used for our grayscale lithography techniques. KMPR is most sensitive when the wave length of the UV light is about 365 [nm]. Generally the rate of absorption of the UV light of the photoresist changes by the wave length of the UV light source. When the radiated light contains the light of several wavelengths, the effect of light for each wave length could be combined to produce complicated phenomenon. For example, the relationship between the amount of exposure and cured depth can be complicated which may produce the exposed and unexposed area which results in partially-cured region in places. For the present study, the exposed light of mercury lamp was masked with a band pass filter which has a peak pass-wavelength of 365 nm. The intensity of the light at the wavelength of 365 nm was measured by a illumination photometer and which was 15.4 [mJ/cm^2]. Now we assume the KMPR thick nega-photoresist is one of the photosensitive resin, the equation of amount of radiation of UV light as a function of etched depth can be introduced.

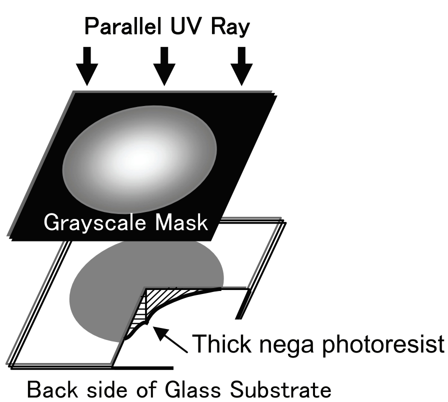

Fig. 1: Concept of Grayscale photolithography.

Assuming the E_0 [mJ/cm^2] is the radiation energy on a unit area of the surface of the photoresist, the amount of exposure at the resist depth of z [mm] can be expressed as (Lambert-beer's law[12]):

$$E(z) = E_0 \times \exp(-z/D) \quad (1)$$

where D [mm] is the penetration depth where the intensity of the UV light radiation on the surface of photoresist is 1/e, and which is the characteristic number of the KMPR photoresist. Suppose the critical number of the amount of the exposed light is E_c [mJ/cm^2], the photoresist is cured from the surface of the photoresist to the depth of G_d [mm] for a given amount of exposure light E. The G_d can be expressed as

$$E_c = E_0 \times \exp(-G_d/D) \quad (2)$$

$$G_d = D \times \ln(E_0/E_c) \quad (3)$$

where G_d is the depth of cured photoresist at a given amount of exposed light E_0 and D is the depth of the transmission of the light and E_c is the critical amount of exposure light.

B. Fabrication of 3D MMT by grayscale lithography

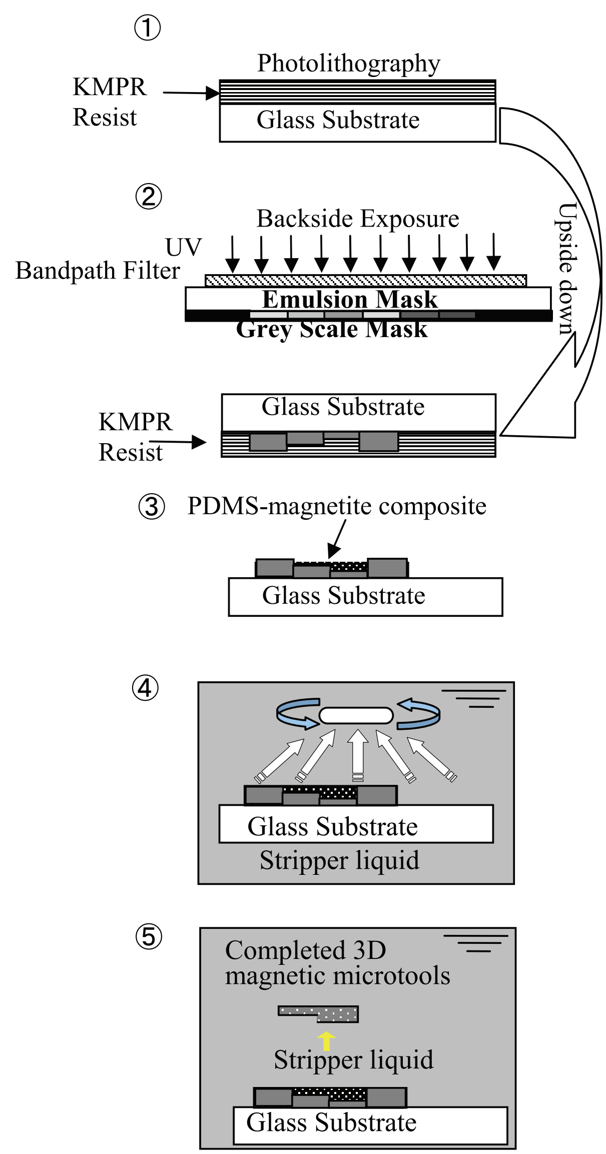

Fig. 2: Fabrication processes of 3D MMT.

Figure 2 shows a novel method to produce magnetically driven 3D microtools. The first (Fig.2①) uses KMPR photoresist. It is important to use transparent glass substrate instead of Silicon substrate. Gray-scale photolithography techniques require an emulsion glass mask with a gradation of grey tone which controls the amount of UV light. Because KMPR is the nega-resist, the photoresist cured by the UV light from the surface of resist to the bottom of resist (the surface of silicon wafer). Therefore the cured pattern by gray-scale mask may be washed away by the process of developing pattern. To fix the gray-scale pattern on the substrate, it is required to use transparent glass substrate and set it in the exposure machine upside down (*back side exposure*). By using the method, the gray pattern can be formed from the bottom of substrate to remain on the substrate. The KMPR photoresist mold for 3D-MMT was fabricated by patterning the photoresist layer on a *glass* substrate (photoresist height = 100 μm)(Fig.2②). The PDMS-magnetite composite (50w% of Fe_3O_4) is molded into a designed configurations of magnetically driven microtools and baked at 80 ℃ for 20 min to cure the composite (Fig.2③). The patterned substrate is then put in a stripper bath (Remover PG at 70℃) with a large commercial stir bar made of a permanent magnet that keeps the temperature of the stripper liquid steady (Fig.2③). KMPR resist is dissolved by the stripper liquid, leaving a gap between the KMPR resist and PDMS-magnetite composite. With dissolving the KMPR resist, the large stir bar can collects a number of patterned PDMS-magnetite composite automatically due to the magnetism of composite (Fig.2③&④). This is used to produce small, fine, complex 3D pattern of magnetically driven microtools.

III. CALIBRATION OF GRAY-SCALE PHOTOLITHPGRAPHY

To control the depth of the photoresist precisely, it is crucial to calibrate the gray photomask. Generally the gray patterns on the mask tend to be expressed by the density of the black dots on a mask. We have used high resolution of imagesetter film of 3600 dpi. However the size of each dots of the film is about 60 μm and this is larger than the size of the resolution of the exposure device (about 20 μm). Hence the gray-like mask was patterned as a contrast of density of "black dots" due to a large difference of the transmissivity of the area with and without the black dots. Therefore we have used a reduced projection (1/20) of gray-scale mask as shown in Figure 3, then the resolution of each dot of mask become smaller than the resolution of exposure device and the contrast of density of black dots are averaged and uniformly gray pattern can be obtained [7]. For the present study, the target pattern has been magnified 20 times and printed it on the imagesetter film. Then the image of the imagesetter film has been reduced at 1/20 on the emulsion mask by the reduced projection devices to obtain the gray-scale emulsion mask. (it is required to design an inversion of black and white due to the characteristics of emulsion mask). Figure 4 shows the patterned emulsion mask for the calibration of 256 gray levels. The KMPR photoresist was spin coated with a height of 120 μm on a glass of 3cm square and the back side exposure has been carried out with the calibration gray emulsion mask. The intensity of the mercury lamp with 365nm bandpath filter was 15.4 mW/cm^2 and the 1300 dose was provided to the KMPR photoresist. The depth of the pattern has been measured by a step profiler (KLA-Tencor). Figure 5 shows the condition of photoresist on the glass substrate after the exposure. When the amount of irradiance level of UV light is small, the most of the UV light is absorbed by the KMPR photoresist. Therefore a number of absorbed photons of UV light is sensitive to the amount of UV light.

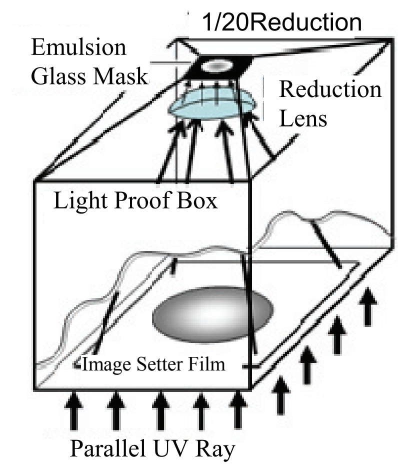

Fig. 3: Reduced projection of Gray-scale mask.

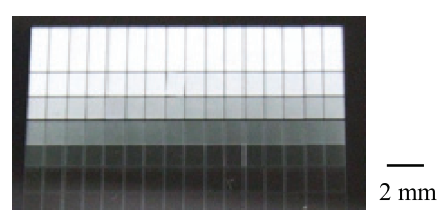

Fig. 4: Emulsion mask for Gray-scale calibration.

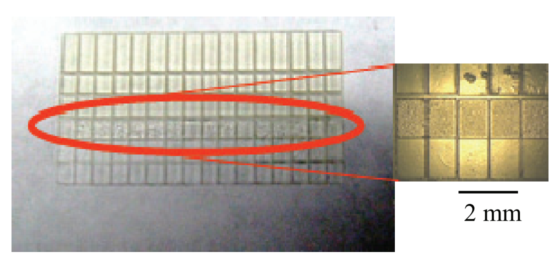

Fig. 5: Patterned KMPR photoresist for calibration

It was observed that a sudden change of the surface of exposure at the gray level of 70-75% as shown in Figure 5. When the amount of irradiance of UV light becomes large, the amount of cured photosensitive resist increased. Then the number of the photon of UV light increased without being absorbed by the photoresist and the UV light start to penetrate the photoresist and be saturated. This phenomenon can be expressed by the equation of (3) of the relationship between the arbitral amount of exposure E and cured depth G_d. Figure 6 shows the calibration curve of gray-scale lithography which is express by a relationship between a UV lamp power which derived from the transmissivity of light at each gray level and the cured depth of the KMPR photoresist. The calibration curve in Figure 6 can be fixed by the condition of exposure and consequently, the designed cured depth can be calculated. It is clear that the KMPR cured depth can be controlled up to about 120 μm (1300 dose with UV light bandpath filter).

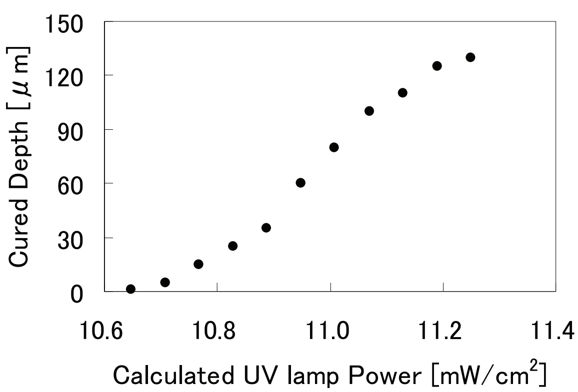

Fig. 6: Calibration curve of the grey-scale photolithography

IV. PRODUCTION AND OPERATION OF 3D MMT

A. Production of 3D MMT

The gray-scale calibration described in Chapter 3 allows to control a depth or height of 3D MMT. For example, a gear-shaped MMT has been developed with a large dimple (ϕ ≈ 500 μm) on the surface of MMT. Figures 7 and 8 show the fabricated KMPR mold for 3D MMT with and without UV bandpath filter respectively (exposure dose was 1300 dose for both conditions). It is observed that the KMPR pattern with UV bandpath filter (Figure 8) could obtain smooth curvature of 3D fabrication compare to that without UV bandpath filter (Figure 7). This is due to the exposure by multiple wavelengths which fluctuated the cured condition of photoresist as described in Chapter 3. Figure 9 shows the depth of the patterned KMPR measured by a step profiler (KLA-Tencor). The gap of the KMPR pattern was about 110 μm. Figure 10 shows the molded MMT from the KMPR pattern shown in Figure 8, and it is confirmed the smooth curvature was successfully transcribed from the KMPR pattern. By using the techniques, it is easy to apply to produce a gray scale mask with complicated 3D design. Figure 11 shows one of the examples of the complicated pattern of KMPR by means of gray-scale photolithography. The good point of the technique is only one exposure is enough to fabricate such a complicated shape. Figure 12 shows the 3D MMT fabricated which can be used as on-chip 3D rotor.

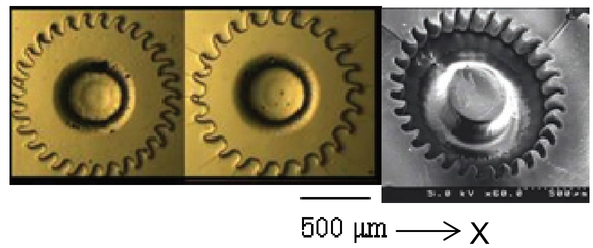

Fig. 7 Grayscale Patterned KMPR without UV bandpath filter

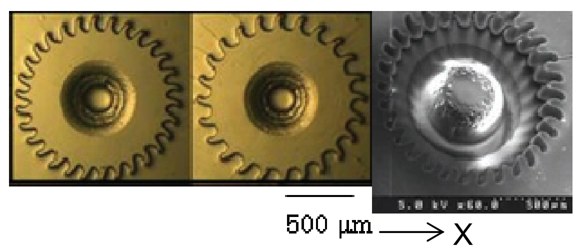

Fig. 8 Grayscale Patterned KMPR with UV bandpath filter

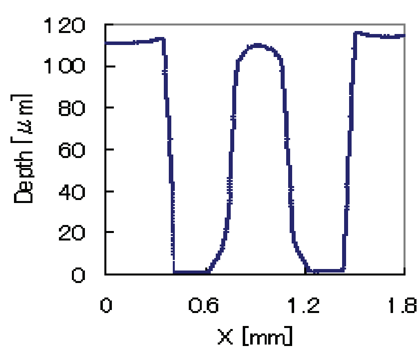

Fig. 9 Depth of Grayscale Patterned KMPR

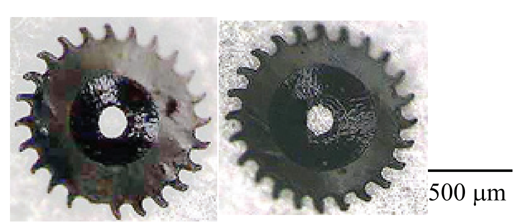

Fig. 10 Produced 3D MMT (Depth ≈ 100 μm)

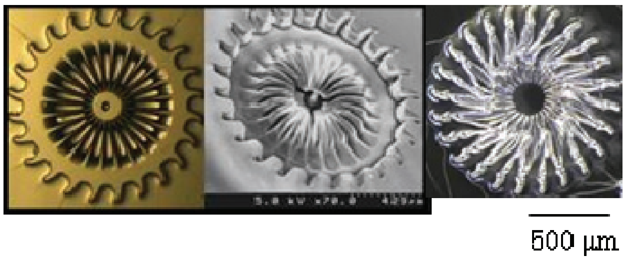

Fig. 11 Complicated 3D patterning of KMPR

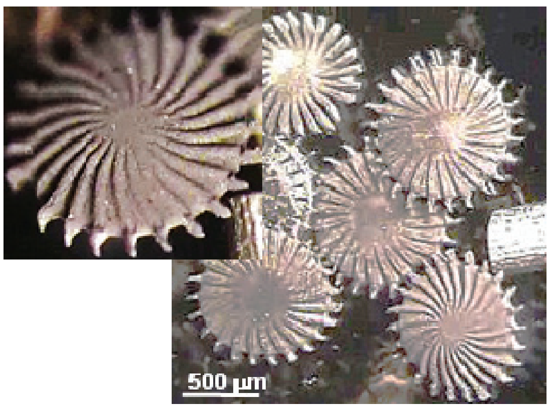

Fig. 12 Complicated 3D MMT

V. OPERATION OF 3D MMT

A. Actuation module of MMT

Figure 13 shows the actuation system which provides rotating motion to the MMT. Loading and rotor functions can be applied to 3D MMT with the actuation system. The motor is mounted below the microchip to initiate the rotating motion to manipulate flow or particle in microchannel.

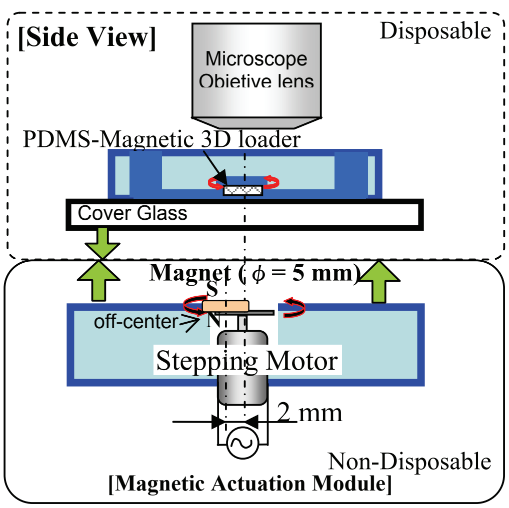

Fig. 13 Schematic of actuation module

B. Loading of multiple particles

Figure 14 present a concept of loading system of microparticles by a combination of magnetic and fluidic forces. It is important to use a fluidic force in addition to magnetic force because it is required to release from the MMT where the particle is in the exit area. Figure 15 shows a gear-shaped MMT to load multiple particles. The gear-shaped MMT is actuated of rotation by a magnetic force beneath the MMT (see Figure 13) and a fluidic force from the microchannel. This gear-shaped MMT has features to transport multiple particles one by one without any clogging. The fluid flow in the region of the exit helped to transport the particles to the downstream smoothly. The speed of MMT was controlled by stepping motor up to 70 rpm with off-center magnet.

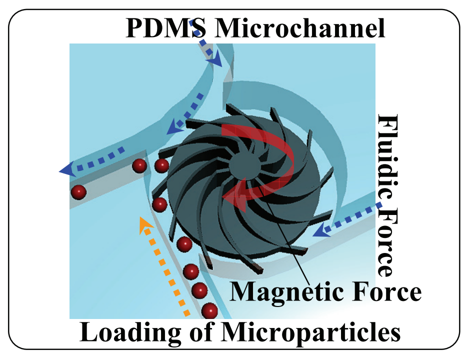

Fig. 14 Concept of Loading system by a combination of Fluidic and Magnetic forces

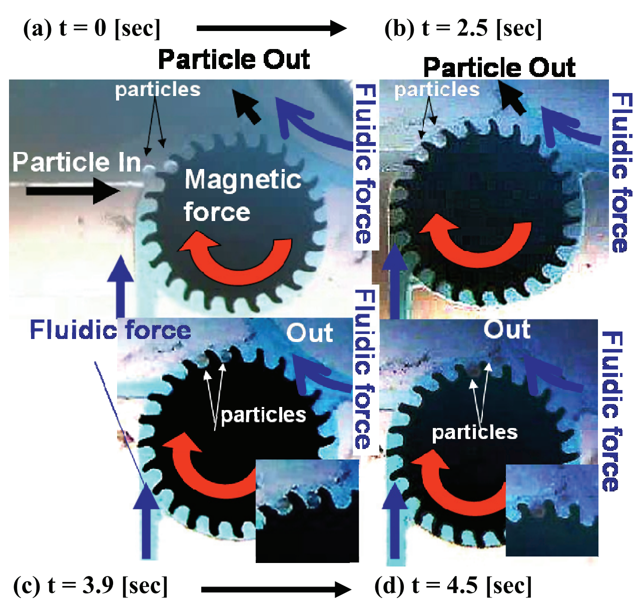

Fig. 15 Loading of multiple particles by gear-shaped MMT

The loading MMT in figure 15 was rotated at 3.3 rpm. The fluid flow through the microchannel is set-up using a controlled microsyringe pump (Kd-Scientific model 230) that generates a pressure flow with a flow rate set at 10 μLmin^{-1} and which push and swipe a particle in a gear of MMT to the downstream of microchannel.

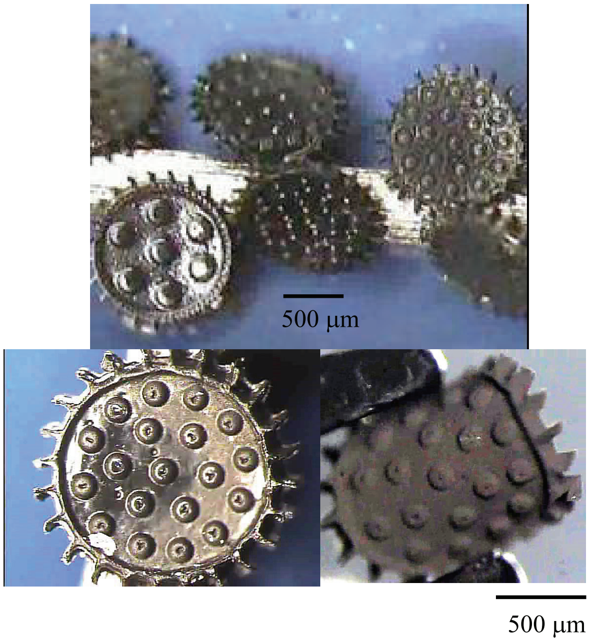

Fig. 16 A series of 3D MMT loader and its flexibility

It is important to reduce the friction force between the PDMS wall and PDMS-based MMT for the effective operation of loading. Figure 16 shows the spiked MMT loader fabricated by using the gray-scale photolithography techniques. The MMT has a features of its flexibility (Young's Modulus ≈ 5 MPa) due to polymer based materials as shown in Figure 16, and hence the MMT can be used with sensitive material such as cells.

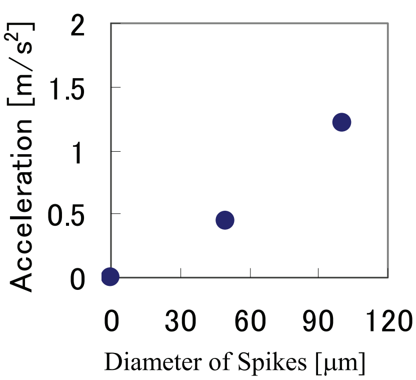

Fig. 17 The acceleration of spikes 3D-MMT in microchannel

Figure 17 shows the acceleration of MMT in the fluid channel. It was observed that the spikes which have larger diameters tend to have a higher acceleration of MMT. It was confirmed the spiked surface has a effect on the smooth actuation of MMT in the PDMS microchannels.

VI. Conclusion

We have successfully established to fabricate 3D-MMT with gray-scale lithography techniques. By using the technique 3D structure with 100 μm gap can be achieved. The novel gear-shaped MMT has been demonstrated as microloader by a combination of magnetic and fluidic force successfully. The spiked pattern has been fabricated on the surface of the 3D MMT to avoid any stiction between the PDMS-based MMT and PDMS microchannels which were confirmed to work effectively. The operation of 3D MMT has opened the possibilities for complicated manipulations in the microfluidic environment with sensitive materials such as cells.

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