미세유체 소자를 이용한 균일한 다공성 미세구형입자의 제조

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Generation of Uniform Porous Microspheres in Microfluidic Devices

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Introduction

An emerging field that benefits from crystalline microporous materials is the field of photonic crystals, that is, three-dimensional (3D) dielectric composites with lattice spacings of the order of wavelengths of light (about 500 nm) (1). These crystals can be used to create photonic band gaps (frequency ranges that will not propagate light because of multiple Bragg reflections) (2) that induce useful optical properties, such as inhibition of spontaneous emission or photon localization (1). The generic examples that are sure to find use in many experiments and devices: a perfect dielectric mirror, which reflects light; a resonant cavity, which traps light; and a waveguide, which transports light (2). And the typical example is display devices. Several colors can be appear because the angle of incidence of the illuminating white beam varies with respect to the crystal planes (1). In this paper, the property that water droplets generated when the water phase introduced into the continuous oil phase is applied to generation of uniform microspheres. After calcination, Microspheres which have 300nm air spheres and 6㎛ in diameter are generated. By this method a lot of uniform microspheres could be generated for short time. And microspheres which have different pore size can be generated only by replacing PS latexes of difference size in water phase.

Experimental

An initial design is sketched in a CAD design program, and the pattern of chromium on a glass-the patterned mask for photolithography-is generated using a mask writer (Figure 1) (3,4). Linewidth of a channel is 40μm.

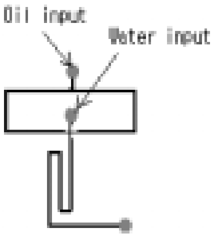

Fig 1. Microfluidic channel design by using a CAD design program

The wafer coated in photoresist is exposed to UV radiation for 100 seconds using a standard UV exposure unit (Karl Suss MA6 contact mask aligner). The wafer is then immersed in developer solution consisting of developer and deionized water until the pattern is clearly seen. The wafer has been rinsed with distilled water to discard any trash on it. After development, the wafer are hot baked for 30 minutes to round the flow channels in the mold then the bas-relief pattern of the photoresist serves as the master for generating molds in PDMS. A negative replica of the photoresist master-the top part of a microfluidic chip-is generated by casting an uncured prepolymer of PDMS against it. Microscope cover glass is prepared as the bottom part of a microfluidic chip. The top and the bottom are sealed and a microfluidic chip is fabricated (Figure 2).

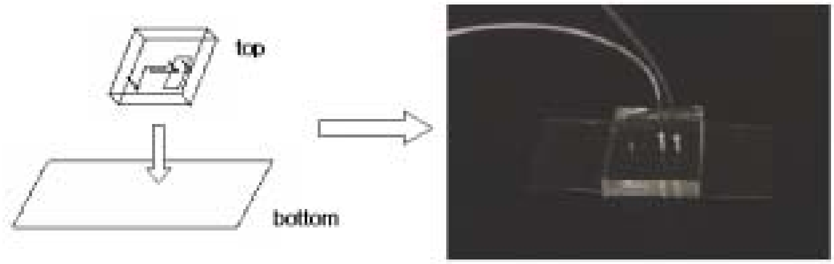

Fig 2. Fabication scheme for microfluidic channels and the shape of a microfluidic chip.

Polystyrene (PS) latex microspheres with carboxylate (COOH) were synthesized by emulsifier-free emulsion polymerization method and Ludox HS-40 colloidal silica was purchased from Aldrich Chemical Co.. The diameter of PS beads was about 300nm. Tween 20 is used as a stabilizer in water phase. KF 96-500, used as oil phase, was purchased from ShinEtsu silicon oil Co.. because other oils are not stable in contact with PDMS. The fluids are introduced into the PDMS microfluidic devices through pressurized reservoirs containing water solution of PS latexes and silica particels and oil. The reservoirs are connected Bronkhorst Hi-TEC mass flowmeters and pressure controlled by MFCs (mass flow controllers) purchased from Pungjurn instruments inc.. Pressure was applied to the reservoirs with compressed nitrogen gas, and the device output channel was allowed to vent to the atmosphere.

Research and discussion

Fig. 3 contains photomicrograph of the discontinuous water phase introduced into the continuous oil phase. When oil flow rate becomes higher, the space between droplets becomes narrower and generation rate of droplets becomes higher but droplets conglomerate so often (5). Once conglomeration is started, the larger droplet and a lot of following droplets conglomerate continuously and becomes larger and larger because larger droplets move slower than smaller one. This huge droplet flows at the end of the channel or is crystallized and immobilized in microchannel. If microchannel is clogged by this huge crystal, this channel becomes useless. But on the other hand, when oil flow rate becomes slower, the space between droplets become wider and conglomeration seldom occurs. But the number of crystals acquired are smaller than in a former case. If crystals are taken out from the chip, water droplets are generated continuously and lifetime of a chip will be longer. And at least the piling position of crystals would be controlled.

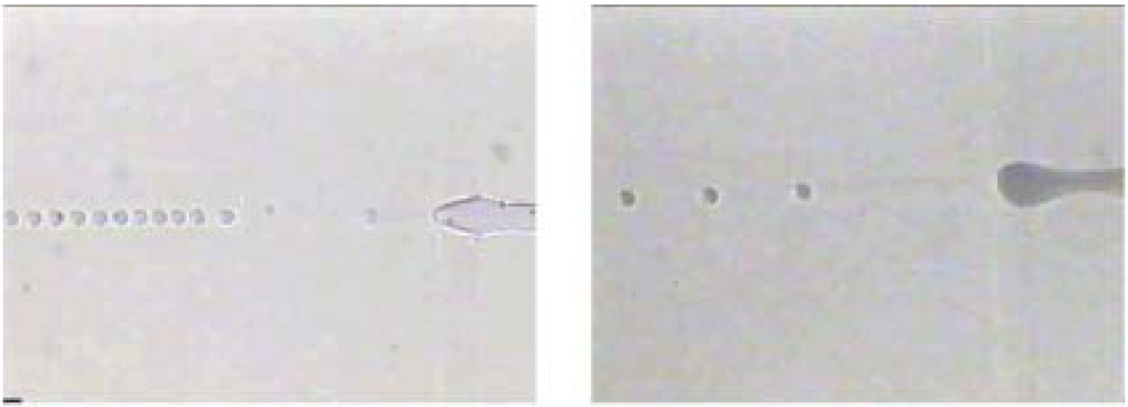

Fig 3. Photomicrograph of the discontinuous water phase introduced into the continuous oil phase. (A) For high oil flow rate. (B) For low oil flow rate.

Generated crystals are piled up in microchannel or stay at the end of the channel. Usually a larger droplet is immobilized in microchannel and act as a trap for following crystals. The pile of crystals is collected in Hexane. The shape of generated crystals can be confirmed by SEM images. But it is impossible to earn the SEM image of just gathered crystals because they are dripping wet with silicon oil. Hexane is used to remove oil but crystals as well as oil are swept away from a glass or PDMS plate. So a 2D patterned plate is used to fix the crystals on a plate. Hexane is dropped continuously over crystals fixed on a 2D patterned plate. By using this method oil was almost removed. Figure 5 contains SEM images showing two crystals fixed on a trough. Figure 6 shows surface of a supraball. Their supraball structure is confirmed and it is expected to gain porous microspheres by calcination method from them.

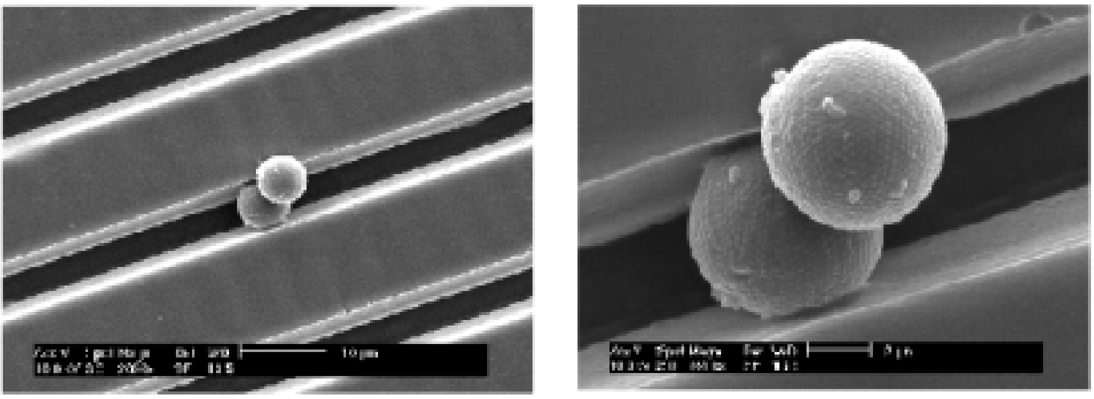

Fig 5. SEM images showing two crystals fixed on a trough.

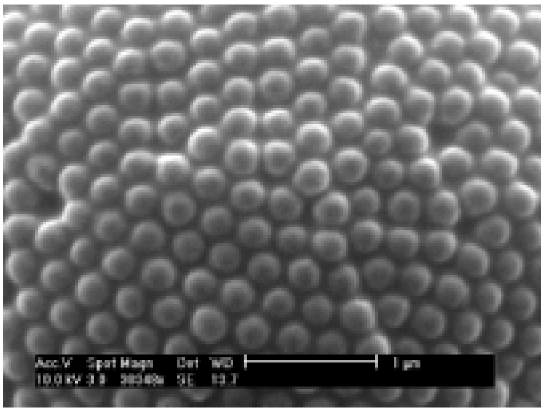

Fig 6. SEM image of surface of a supraball.

Summary and conclusion

Microchannels are fabricated by soft lithography(3) and oil phase and water phase are introduced in the channel. Then at the junction of the two flow water droplets are generated and water are diffused in oil continuously. So we put PS latexes and silica particles in water phase and generated uniform water droplets. Immobilization of PS latexes and silica particles in water droplets are observed by a confocal microscope. Their detailed shape of supraball is portrayed by SEM images. It is expected to gain porous microspheres from supraballs by calcination. Pore size of supraballs can be controlled as PS latexes which have different size are used. If crystals are taken out from the chip, water droplets are generated continuously and lifetime of a chip will be longer.

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