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## TECHNICAL NOTE

# Photolithographic fabrication of arbitrarily shaped SU-8 microparticles without sacrificial release layers

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## Abstract

We report on an efficient high throughput method for the photolithographic fabrication of well-defined arbitrarily shaped SU-8 microparticles without a sacrificial release layer. The procedure eliminates the spincoating of a sacrificial layer otherwise needed for particle lift-off, thereby reducing processing time and costs. Statistical analysis of the size distribution revealed a standard deviation of less than 2.3% in size. The particles can be immediately released into aqueous solution. This allows for anisotropical functionalization of the particles with, for example, biological loads or elements of molecular recognition after the development of the SU-8 structures.

(Some figures in this article are in colour only in the electronic version)

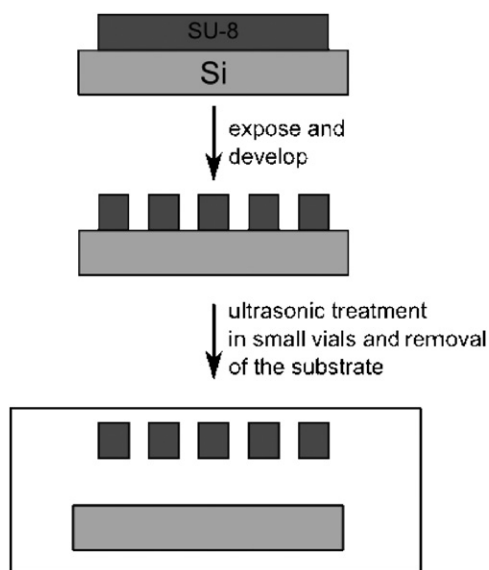
## 1. Introduction

Microparticles are important key elements in biotechnological, chemical and physical sciences. Possible applications range *inter alia* from carriers for vaccines [1], self-assembly of new materials [2], photonic materials [3], carriers in microfluidic channels [4] to model systems for acoustics in media [5]. However, most of the commercially available particles are spherical. Therefore their field of application with respect to shape-dependent phenomena is strongly limited. As a result, several approaches were made to synthesize nonspherical microparticles employing many different materials and fabrication strategies [6–13]. Two major strategies have been developed. On the one hand, particles were fabricated within the liquid phases of microfluidic systems where their shape was determined by the shape of the channels [8, 10, 11]. On the other hand, the particles were structured on a substrate and released either mechanically [9], with acids [7], solvents [13] or sacrificial layers [12].

The most common materials for non-spherical particles are e.g. poly(ethylene glycol) diacrylate [9–11], silicon

dioxide [7] and the well-characterized epoxy-based negative photoresist SU-8 [12, 13]. SU-8 is a standard material in MEMS technology due to its mechanical and chemical stability and its excellent processing properties [14]. In principle, it offers the possibility to create structures with high aspect ratios at submicrometer resolution (e.g. *via* e-beam lithography) and is therefore ideally suited for microparticle fabrication. Furthermore an ISO 10993 conform study by Kotzar *et al* shows that SU-8 seems to be fully biocompatible and even suitable for biomedical implantation [15].

However, SU-8 is usually not suited for lift-off processes complicating the release of the fabricated particles [14]. Therefore, sacrificial layers of Omnicoat [12] or polyimide were commonly used to release SU-8 patterns. A typical protocol contains the following steps: (1) spincoating and baking of the sacrificial layer, (2) spincoating and prebake of SU-8, (3) exposure to UV light and postbake, (4) development of the SU-8 structures, (5) dissolution of the sacrificial layer and particle release and (6) centrifugation and redispersion of the particles in a favored solution to remove the chemical



**Figure 1.** Schematic fabrication process for SU-8 microparticles. After a standard photolithography routine, the substrates are dropped in a vial and then placed in an ultrasonic bath, where the particles are released into a surfactant solution.

agent for dissolution of the sacrificial layer (repeated up to three times).

In this technical note, we propose a simplified procedure to fabricate SU-8 microparticles without sacrificial layers or removal agents. The technique reduces the number of processing steps from 6 to 4, is cost efficient and freely scalable to large throughputs.

## 2. Fabrication

The fabrication procedure is illustrated in figure 1. Silicon wafers (4 inch, Crystec, Germany) were cleaned in carboxylic acid for 20 min (1:3 mixture of hydrogen peroxide (p.a.) and 95% sulfuric acid (p.a.) both obtained from VWR, Germany) and rinsed with deionized water from a MiliQ Biocel System for several minutes. Centrifugation and heating up to 200 °C for 20 min on a contact hotplate removed residual water.

SU-8 (2) (Microchem Corp., USA) was applied to the wafers and spincoated at 500 rpm for 5 s and subsequently at 2000 rpm for 30 s (Spincoater ST147, Convac, Germany). After the prebake procedure (40, 60 and 90 °C each for 5 min, ramp rates: 3, 2, 2 °C min<sup>-1</sup>) a chromium-glass mask (DeltaMask, the Netherlands) was used to initialize cross linking of the resist with the i-line of a mercury pressure lamp with approximately 140 mJ cm<sup>-2</sup>. The samples were postbaked with the same parameters as for prebake and developed in MR-DEV 600 (Microresist, Germany) for 30 s, rinsed with acetone (p.a.) and isopropyl alcohol (p.a.) (both obtained from VWR, Germany) and dried with nitrogen (Linde, Germany) afterward.

Prior to the characterization of the particles on the substrates with a scanning electron microscope (JSM 880, Jeol, Japan), a 20 nm gold layer was sputtered on the samples in a MED 020 sputter system (BAL-TEC, Liechtenstein).

For particle release, the structured substrate is cut into 5 × 10 mm<sup>2</sup> pieces and dropped into a vial (6 ml) together with 5 ml DI water. The vials were placed in an ultrasonic bath (Transsonic Digital S, Elma, Germany) at 140% power for 10–30 s until the particles were fully released. Afterward the silicon substrate was removed.

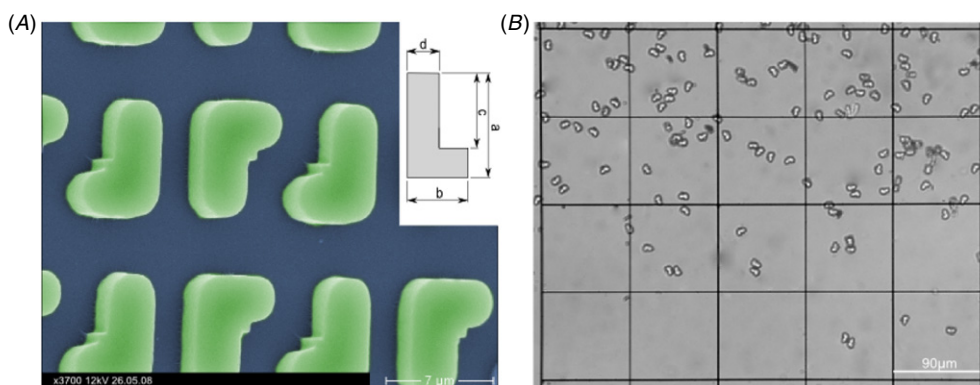
## 3. Results and discussion

Figure 1 illustrates the simplified lithography procedure for producing SU-8 microparticles with just four processing steps: (1) a SU-8 film is spincoated and prebaked. This step controls the height of the particles. (2) The photoresist is exposed to UV light and postbaked. The shape of the particles is controlled by the UV transparent regions on the mask. (3) The photoresist is developed. (4) Release of the particles by placing the silicon substrate in a vial filled with DI water in an ultrasonic bath.

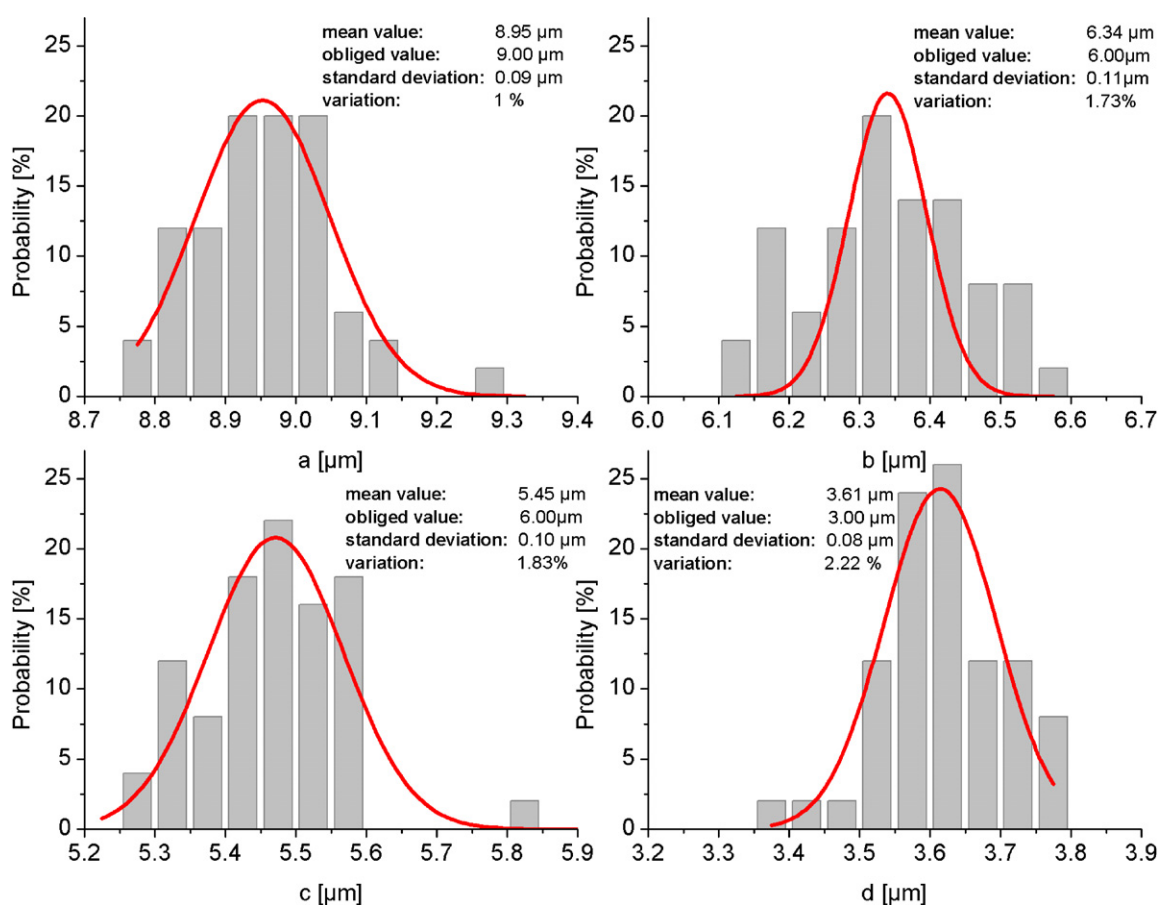
As an example, L-shaped particles are shown in figure 2 before and after release. Optical inspection revealed no destroyed or broken particles after sonification and release. Furthermore, no intra-particle adhesion was observed. An analysis of the size distribution by scanning electron microscopy is given in figure 3. The deviations are less than 2.3% and therefore competitive compared to recently published lithographically formed SU-8 particles; Badaire *et al* demonstrated 6% deviation using Omnicoat [12] and Moon *et al* 10–50% by using interference lithography and polyimide for lift-off [12]. Figure 4 shows more examples of microparticles, fabricated with our technique before (A)–(C) and after lift-off (D).

The fact that the SU-8 particles can be released easily without a sacrificial layer is supported by figure 5. The silicon wafer with unreleased particles was sputtered with a 20 nm gold layer and then placed in an ultrasonic bath. The treatment was interrupted before completed lift-off after several seconds. Different mechanisms of release can be identified: the particles are just released without hitting further particles (figure 5(B)), or a release cascade is started when a few particles start to slide along the substrate and remove further particles (figure 5(A)). The pathways can be seen because the sliding particles scratch off the gold layer.

SU-8 is usually considered as a hard to remove photoresist [16, 17]. Successful lift-off of SU-8 microstructures has so far only been demonstrated by using an additional sacrificial layer. After SU-8 development, the sacrificial layer is dissolved and the particles are released. Traditionally SU-8 was used to generate widespread patterns going along with large areas of contact between SU-8 and the substrate. For single microparticles, however, the area of contact is small easing the lift-off process. The adhesion might also be disturbed by different processes during the fabrication procedure. All fabrication steps lead to film internal mechanical stress: soft bake (50% contribution), exposure (30%), post bake (15%) and development (5%) [14]. The mechanisms rely on solvent evaporation, polymer shrinkage upon cross-linking and thermal expansion and retraction during baking and cooling ( $52 \pm 5$  ppm K<sup>-1</sup> for post bake at 95 °C) [14]. The maximum stress is observed during postbake with 16–19 MPa



**Figure 2.** Scheme of the L-shaped particles to define dimensions. (A) Scanning electron micrograph of the structured particles while they are still bound to the silicon substrate prior to removal. (B) The L-shaped particles in a free solution after they have been released from the substrate.



**Figure 3.** The measured four characteristic dimensions of the L-shaped particles in comparison to their corresponding obligated values and standard deviations/variations. The identifiers a, b, c and d correspond to those in figure 2(A). The sample size is  $n = 50$ .

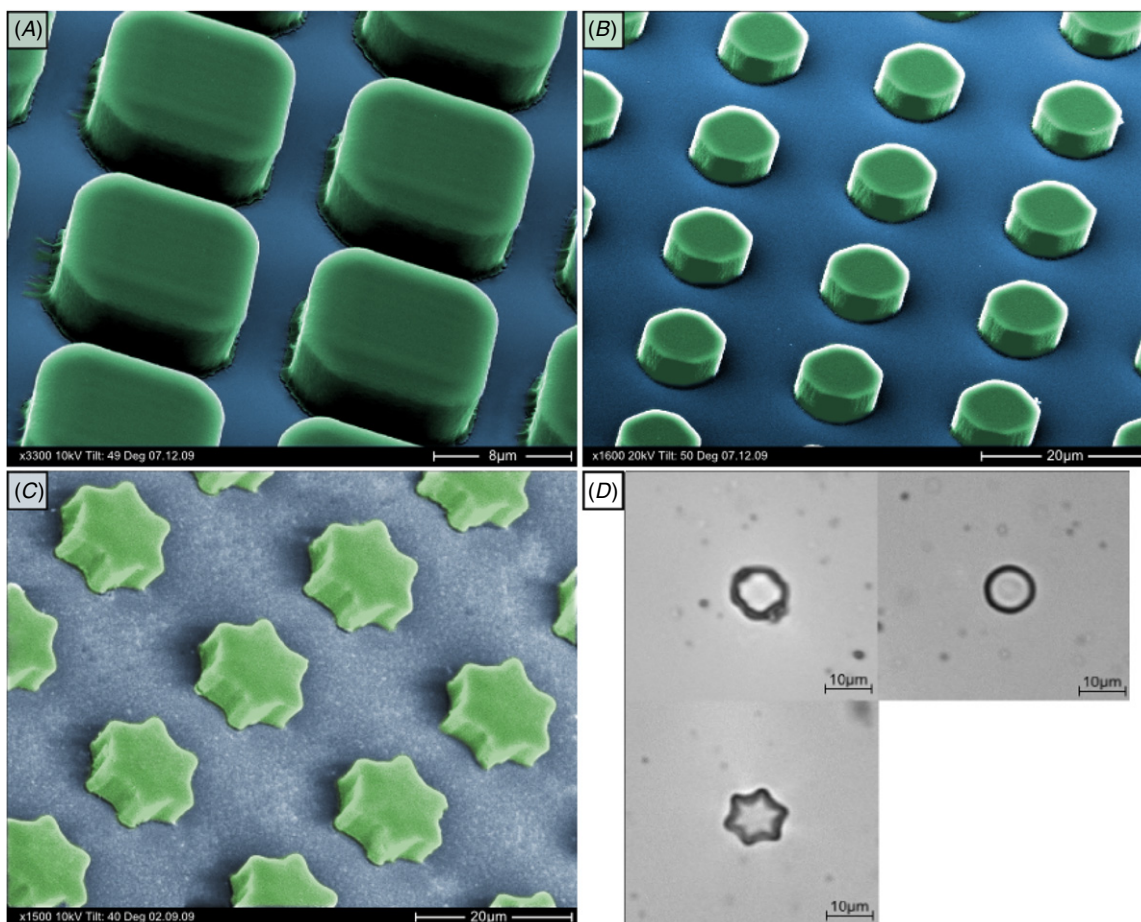
[14]. All this stress might weaken the adhesional forces and eases lift-off.

#### 4. Conclusions

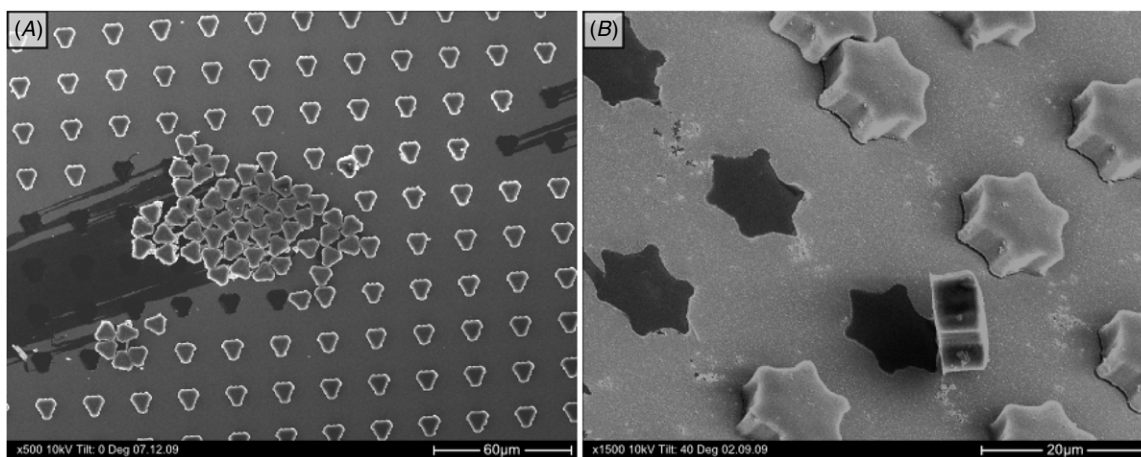
A simplified method for the production of non-spherical SU-8 microparticles of controlled shape without a sacrificial layer has been presented. The main advantages compared to established methods are: our procedure is faster because

the spincoating and the baking of the sacrificial layer can be omitted. Costs are reduced since neither the sacrificial layer nor the corresponding remover is necessary. Finally, the particles are immediately dispersed in the favored solution, making particle centrifugation, washing, purification and the exchange of the solvent obsolete. The procedure can be upscaled as well making it suitable for mass production.

SU-8 offers attractive properties for the production of anisotropic particles: the material is mechanically stable and biocompatible, an aspect highly relevant for biological



**Figure 4.** Scanning electron micrographs (colored) of further arbitrarily shaped microparticles before their lift-off: cuboids (A), cylinders (B), stars (C). (D) The particles from A, B and C after successful removal from the substrate in a free solution via optical brightfield microscopy.



**Figure 5.** Scanning electron micrographs of particles during an interrupted lift-off process in an ultrasonic bath.

applications. SU-8 particles could be steered using electric fields, because of their zeta potential of  $-36 \pm 5$  mV [12]. Combining such transport effects with hydrodynamics may lead to novel migration mechanisms of anisotropic SU-8 particles suitable for morphological separation in microfluidic environments, a project that is currently pursued in our lab.

Furthermore, the fabrication on a substrate allows for an anisotropic functionalization or coating of the particles. For example, Moon *et al* showed how to functionalize SU-8 particles on a substrate with metals, PEO chains and amine groups for further biotechnological experiments [13]. Herewith so-called janus particles with differently functionalized surfaces and arbitrary shape could be

fabricated. Furthermore mixing the SU-8 resist with magnetic nanoparticles or fluorophores before spincoating could further expand the range of application of the SU-8 microparticles.

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