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Surface patterned polymer micro-cantilever arrays for sensing

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ABSTRACT

Microinjection molding was employed to fabricate low-cost polymer cantilever arrays for sensor applications. Cantilevers with micrometer dimensions and aspect ratios as large as 10 were successfully manufactured from polymers, including polypropylene and polyvinylidenfluoride. The cantilevers perform similar to the established silicon cantilevers, with *Q*-factors in the range of 10–20. Static deflection of gold coated polymer cantilevers was characterized with heat cycling and self-assembled monolayer formation of mercaptohexanols. A hybrid mold concept allows easy modification of the surface topography, enabling customized mechanical properties of individual cantilevers. Combined with functionalization and surface patterning, the cantilever arrays are qualified for biomedical applications.

1. Introduction

Micro-cantilevers (μCs), similar to those used in scanning force microscopes (SFM), have become increasingly popular as transducers in chemical and biological sensors [1–8]. They convert physical, chemical, and biological stimuli into measurable signals. Various detection methods have been introduced to measure the bending of the μCs in the range of few nanometers with extremely high accuracy. A compelling feature of μC sensors is that they operate in air, vacuum, or liquid environment [7]. Like many micro-machined devices, μCs are typically made from glass, silicon or other rigid materials. In the field of biomedicine, silicon-based μCs have to be cleaned or even sterilized for repetitive use. For single usage they are often too expensive. The fabrication is based upon single crystalline silicon wafers to be processed in cleanroom facilities. The high costs compromise many applications and calls for low-cost, disposable sensing elements. Polymer materials offer tailored physical and chemical properties to be combined with low-cost mass production. Therefore, compared to silicon-based μCs the polymeric μCs can exhibit better biocompatibility and much better adaptability of rapid prototyping along with mechanical properties, which make them particularly sensitive [7]. Despite these advantages polymeric μC arrays are not yet commercially available. Polymer μCs can be prepared in a variety of ways, whereas the type of polymer often determines the fabrication method [7,9]. So far, polymer μCs were realized using photolithography. It is limited to the suitable materials and the μCs fabrication is rather expensive [10]. Molding of microcomponents from thermoplastic

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polymers has become a routinely used industrial production process and is one of the most promising fabrication techniques for non-electronic micro devices [11]. Fabrication costs of molded parts are hardly affected by the complexity of the design. Once a mold insert is available, several thousand parts can be molded with modest effort. Furthermore, different polymers can be used to obtain parts of almost identical shape with a high degree of reproducibility. Micro-patterns on the mold can be replicated into the molded device, too, making it possible to integrate different dimensions and topographies into one single tool. The mechanical properties of polymer μCs can be tailored choosing appropriate dimensions and surface morphologies. The cost of the raw material in most cases is negligibly low, because only small quantities are required for micrometer-sized components. Therefore, parts fabricated by micromolding, even from high-end materials, are suitable for applications requiring low-cost and disposable components. Several thermoplastic molding processes such as hot embossing, injection molding (IM), injection compression molding and thermoforming give rise to micro-parts with high precision and repeatability [11,12]. Polymeric replication techniques based on nanoimprint and casting of curable polymers can be used to produce structures with sub-100 nm resolutions [13,14]. The hot embossing and the IM seem to be the most industrially viable processes for molded micro-parts [15]. Polystyrene (PS) cantilever beams of thicknesses between 2 and 40 μm with a stiffness ranging from 0.01 to 10 N m^{-1} have been produced using IM [10]. The acceptance of μC sensors in research and commercial, analytic applications crucially depends on the robustness, the ease of use, the reproducibility and finally the price. The question arises if disposable polymeric μC arrays can be fabricated on the basis of standard thermal IM using precisely machined metal molds. It is the aim of the present scientific activities to adapt IM, well established on the millimeter scale and above, to molds with 30 μm-thin cavities, 500 μm long and 100 μm wide to realize polymer μCs with a performance comparable to the presently used silicon-based arrays (hence termed micro-injection molding (μIM)). Sensitivity enhancement using customized surface structuring within the mold cavity is also being studied.

2. Materials and methods

2.1. Comparing established rigid μCs with polymeric ones

μCs respond to impacts ranging from surface stress via mass change to temperature. Their sensing involves the detection of μC deflections and of μC resonance frequencies. The laser beam projection technique provides the μC deflection induced by the forces acting on the cantilever [4]. Forces in the pN-range are detectable, since the setup can uncover sub-nanometer deflections of the apex of the μC sensors. These forces comprise expansions or contractions acting on one side of the cantilever surface [16]. One applies static and dynamic modes for more or less sophisticated sensing. In static mode, the surface stress generated from selectively adsorbed molecules on one side of the cantilever is measured. The free-end deflection Δ_z as the result of the surface stress $\sigma_{surface}$ is often quantified using the well-known Stoney formula [4,7], for example in the form of Sader [17]:

$$\Delta_z = \frac{3(1-\nu)L^2}{Et^2}(\Delta\sigma_{surface}) \quad (1)$$

where $\Delta\sigma_{surface}$ is the difference of surface stress between top and bottom sides of the cantilever, ν is the Poisson's ratio and E is the Young's modulus of the cantilever material and L and t are the length and thickness of the cantilever, respectively.

In dynamic mode, the *resonance frequency* of the cantilever f_{res} is monitored during mass adsorption on the cantilever [18]. The related shifts in resonance frequency Δf_{res} are given for homogeneously distributed adsorbents by

$$\Delta f_{res} \approx -f_{res}\frac{\Delta m}{2m_0} \quad (2)$$

where Δm is the absorbed mass and m_0 is the initial mass of the cantilever [18]. The frequency shifts per mass change on the typically applied cantilever of rectangular shape is

$$\frac{\Delta f_{res}}{\Delta m_0} = \frac{1}{4\pi n_l L^3 w}\sqrt{\frac{E}{\rho^3}} \quad (3)$$

with $\rho = m/Lwt$ as mass density and $n_l \sim 1$ as characteristic geometrical μC parameter [1].

The frequencies for the ith resonance mode, f_i, can be estimated using the μC geometry, L and t, and the materials density ρ, 2330 and 3180 kg m^{-3} for Si and Si_3N_4 as well as 1220, 900, and 1220 kg m^{-3} for the polymers polyvinylidenfluoride (PVDF), polypropylene (PP), and polyoxymethylene copolymers (POM-C), respectively:

$$f_i = \frac{t}{2\pi}\left(\frac{\alpha_i}{L}\right)^2\sqrt{\frac{E}{12\rho}} \quad \alpha_i: \quad \alpha_1 = 1.9; \quad \alpha_2 = 4.7; \quad \alpha_3 = 7.8; \quad \ldots \quad (4)$$

α_i is a constant obtained by numerically solving the beam frequency equation [7]. The Q-factor characterizes the resonator's bandwidth B relative to its centre frequency and the μC damping δ during ring-off:

$$Q = f\frac{\pi}{\delta} = \frac{f}{B} \quad (5)$$

The sensitivity of the sensor depends on the mechanical parameters Young's modulus E, ~130–188 GPa for Si, 310 GPa for Si_3N_4, as well as 6.7, 1.9 and 6.7 GPa for PVDF, PP, and POM, respectively, and Poisson ratio $\nu \sim 0.22$ for Si and 0.24 for Si_3N_4 as well as 0.3–0.5 for most polymers. In order to fabricate polymer μCs with sensitivity comparable with silicon ones (typical dimensions of $L \sim 500$ μm and 1–5 μm thickness), while keeping L constant, the μCs have to be one order of magnitude thicker to compensate the hundred times smaller E.

For the selective sensitivity to detect contaminants in gases or dedicated species in liquids, the cantilever surfaces have to be functionalized. For this purpose, one μC side is coated or patterned to enhance selective binding of the species of interest chemically or by featured surface morphology. Chung et al. [16], for example, used field ion beam milling to build nanostructures on the μC surfaces. The mechanical properties of the μCs depend on the coating and its thickness as well as the morphological features including pattern sizes. Field ion beam milling modifications soften cantilevers [19,20], whereas corrugations generated by means of stencils stiffen cantilevers and membranes [21]. μIM belongs to the attractive approaches to manufacture polymer μC with pre-defined surface microstructures.

2.2. Microinjection molding

A modular injection molding tool has been developed that consists of a high quality steel cylinder (Polmax Uddeholm) 30 mm in diameter as mold insert with two internal resistive heating cartridges (Watlow Firerod, 230 V, 180 W, 49 W/cm^2) fixed in the three-plate molding tool 'handy mold' with ejector pins (see Fig. 1, left side). This setup enables us to proceed with both isothermal and variothermal heating schemes with short heating times for temperatures as high as 320 °C in the vicinity of the mold cavities. The tool is installed in the clamping unit of an Arburg 320 Allrounder (Arburg, Lossburg, Germany) with a maximum clamping force of 600 kN.

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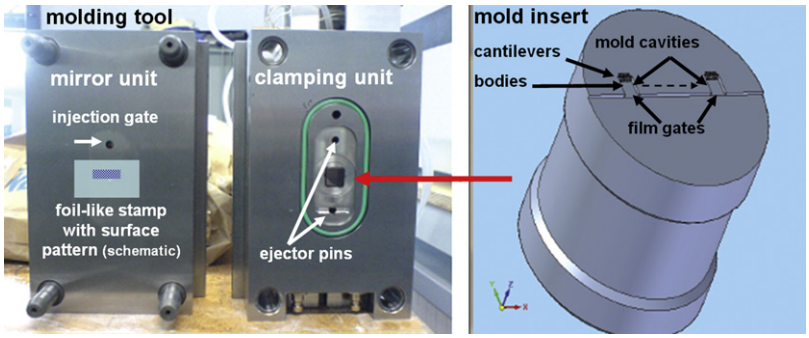

Fig. 1. Molding tool (handy mold) with two sides (left side). The mirror side contains the gate (top) and the location, where the patterned foil is placed. The clamping unit contains the mold insert (right side) with two mold cavities.

In contrast to the work of Andrew et al. [10] with one mold cavity composed of two halves placed in the opposite mold units of the IM machine to generate cantilevers with symmetric position, the present mold system comprises only one cavity located on the closing side [22,23]. The other side is free for mirror plates with designed micro- or nano-features.

The two parallel mold cavities (see Fig. 1, right side) were fabricated using laser ablation, and placed into the central part of the flat end of the cylinder. They are connected to the injection gate via a large plate-like cavity through 2.5 mm-wide gates for filling. The μC chip was designed with outlines of a micro-machined 500 μm-thick silicon μC with a $3.5 \times 2.5\ mm^2$ large body. It has eight 80–130 μm-wide μC beams with a 500 μm pitch on one side. The thickness chosen was usually in the range between 20 and 40 μm. To guarantee fast and complete filling also molds with 60 μm depths were applied (see Fig. 2, top micrograph). For the venting, at the end of each beam cavity thin, 5 mm-long, $10 \times 10\ \mu m^2$-wide channels were incorporated. The polished steel plate with one injection gate is the flat counterpart opposite to the closing unit. Therefore, the upper side of each μC beam has a polished finish (see Fig. 2, bottom micrograph) later used for laser beam reflection. Surface patterned beams require, thus, an additional mold insert with a micro- and nano-relief to be introduced at the mirror side. In place of another mold insert, we incorporate a thin, patterned polymer foil (see Fig. 1, left side). This foil-like mold prepared by hot embossing, typically 25–100 μm thick, forms the interface between the two units of the IM machine and is subjected to related pressure and heat. To ensure repeated alignment during injection and demolding, it is directly fixed onto the polished top by adhesive tape or clamps. The mold temperatures and pressures have to be low enough to enable a sufficient number of replications without degradation of the surface relief. The main advantage of the method lies in the simple integration of gratings with different sizes and orientations. It is particularly useful for test series. Even for mass production the method is promising, since polymer foils can be patterned in roll-to-roll processes [24,25].

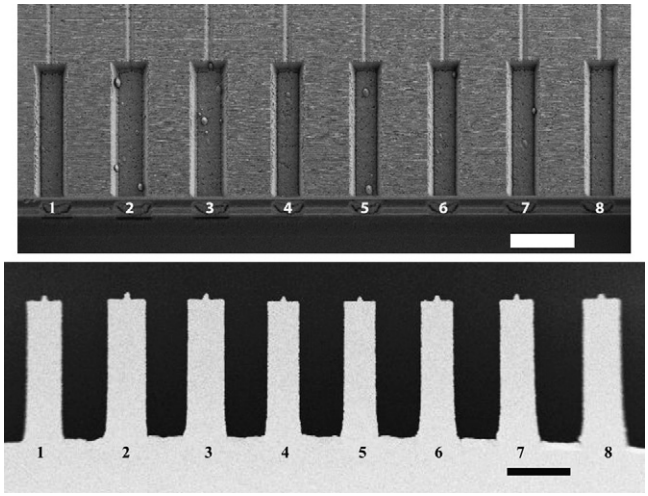

Fig. 2. The top SEM micrograph shows the array of eight laser ablated cantilever cavities in the steel mold insert. The cavity width varies from 80 to 130 μm. The scale bar corresponds to 200 μm. The SEM micrograph on the bottom is an image of an injection molded PP micro-cantilever array. Small tips at the cantilever end demonstrate the complete filling up to the venting channels.

2.3. Microinjection molded polymer materials

The polymers used are different grades of poly(etheretherketone) (PEEK: Solvay Advanced Polymer AvaSpire AV-650 BG15, Solvay Advanced Polymer KetaSpire KT-880NT, Victrex 150G), poly(propylene) (PP: Moplen SM 6100), polyoxymethylene copolymers (POM-C: 511P Delrin NC010), cyclic olefin copolymers (COC: Topas 8007X10), polyvinylidenfluoride (PVDF: Kynar 720 Arkema) and liquid crystal polymer (LCP: Vectra A 390).

2.4. Microinjection mold processes

Up to 160 °C, the tool temperatures were controlled by heated water. For the higher process temperatures up to 260 °C oil served as heat transport medium. The other process parameters are summarized in Table 1. As the foil mentioned above 100 μm-thick polycarbonate (PC: Bayer Makrofol ID 6-2) and 25 μm-thick PEEK (Aptiv 2000 series) were inserted. While PC with a glass transition temperature of 148 °C only allows molding polymers with rather low process temperatures, PEEK, which has a comparative glass transition temperature of 143 °C was considered as higher temperature alternative because of its excellent demolding properties. The PC and PEEK foils were hot embossed in a Jenoptik HEX 03 machine for a period of 10 min using temperatures of 160 and 175 °C and forces of 15 and 4 kN, respectively. As the molds for hot embossing, either surface patterned silicon wafers or replicas in Ormostamp both with anti-sticking layer were used [26,27].

Table 1
Injection molding process parameters for the selected polymer materials including all grades of PEEK.

	COC	PP	PEEK	POM-C	LCP	PVDP
Melt temperature [°C]	240	200	400	220	300	220
Tool temperature [°C]	77	40	225	120	150	120
Mold insert temperature [°C]	77	40	260	120	150	120
Injection speed [cm^3/s]	30	9	10	10	10	10

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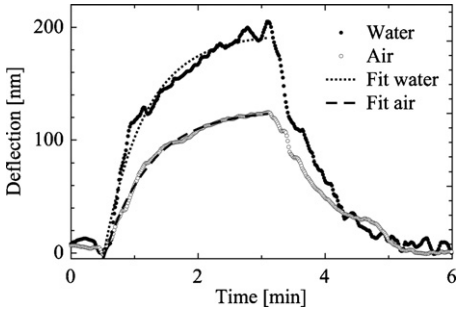

Fig. 3. Real time monitoring of injection molded PVDF μC deflection: heat test of 30 μm-thick μCs with a temperature increase from 25 to 35 °C at a heating rate of about t_{heat} = 0.5 min and a temperature decrease back to 25 °C at a cooling rate of about t_{cool} = 3.2 min.

2.5. Cantilever finishing

The injection molded μCs were coated on the mirror side with 20 nm-thin gold films using a thermal evaporator (Balzers BAE250). This film guarantees sufficient laser beam reflectivity to use the Cantisens® research system (Concentris GmbH, Basel, Switzerland) for measuring the deflection and the resonance frequency of the μC. Replication quality was analyzed using scanning electron microscopy (SEM: Supra 55 VP, Carl Zeiss NTS GmbH, Oberkochen, Germany), after coating with a thin layer of PdAu.

3. Results

Complete filling of the mold cavities was observed for all polymers using isothermal μIM (Fig. 2, bottom micrograph for PP) with the exception of the high-performance polymer PEEK, which requires mold temperatures of up to 320 °C and processing temperatures higher than 260 °C. We have not observed any degradation for PP, COC, POM-C, and PVDF. However, with PEEK, which is more sensitive to longer residence time, visible signs of degradation were observed. Also with the patterned foil-like molds, using standard isothermal μIM process parameters, a complete filling of high-aspect-ratio micro-cavities was achieved for PP (see Fig. 5). The mold temperature was low enough to use the polymeric foils for several hundreds replications without degradation of the surface relief.

With the exception of PEEK, the cantilevers reveal the expected thermal behavior as demonstrated in the diagram in Fig. 3 for the gold-coated PVDF cantilever under atmospheric conditions, i.e. in air, and in liquid (water). The heat tests included a temperature cycle with an increase from 25 to 35 °C and a subsequent decrease back to 25 °C within a time of about 4 min. The heat tests prove the sensitivity of the cantilevers that corresponds to deflections of the order of 10 nm. The deflection signal exhibits an exponential, asymptotic behavior as confirmed by the fits in Fig. 3. For the temperature difference of 10 K the maximal deflection for PVDF μCs in air corresponds to (95 ± 16) nm and (55 ± 5) nm for thicknesses of 30 μm and 40 μm, respectively. In water, these values should be similar but gave higher values, namely (127 ± 17) nm and (154 ± 55) nm. Note the larger scattering of the data in liquid, which indicates less stable experimental conditions and reduced reproducibility in liquid compared to air.

The Cantisens® Research system permits the experimental determination of resonance frequencies f_{res} and quality factors Q for the polymeric μCs. Table 2 summarizes the mean values and related standard deviations of the resonance frequency measurements for the μCs in air and water. The deviations of the experimental data from the estimated ones are reasonably explained accounting for dimensional variations as well as the frequency dependence on E.

Table 2
Mean values and related standard deviations of the resonance frequency f_{res} as well as quality factor Q in air and water.

μC thickness and polymer	30 μm PP	40 μm PP	30 μm PVDF	40 μm PVDF	30 μm POM-C
f_{res} in air (experimental) [kHz]	48 ± 3	50 ± 1	60 ± 3	79 ± 5	60 ± 4
f_{res} (theoretical, Eq. (4))	38	46	66	88	78
f_{res} in water (experimental) [kHz]	37 ± 8	33 ± 27	43 ± 5	52 ± 5	36 ± 7
Q-factor in air	28	46	38	19	33
Q-factor in water	20	11	10	9	19

The drop in resonance frequency in water results from the damping, which lowers the Q-factor of the μCs as given in Table 2. The Q-factors were estimated directly from the frequency spectra [1,2,4].

The stiffness of the μCs was determined by nanoindentation of the injection molded PP μCs. The measurements were carried out using a nanoindenter (MTS XP® with a Berkovich tip (XPT-12761-0)). The unloading segment of the measured load–displacement curve in nanoindentation permits an estimation of the cantilever's Young's modulus, by defining the elastic stiffness as the slope of the unloading segment [28]. The obtained value of 2.4 GPa is close to the value (1.9 GPa) mentioned in the technical datasheet from the PP supplier.

As a first attempt towards biosensing, the chemisorption of thiols on gold coated μCs was recorded by means of the Cantisens® Research system. The data of six PVDF 60 μm-thick μCs from an injection molded array are shown in the diagram of Fig. 4. The deflection results from the surface stress, that is generated during the self-assembly of thiol molecules on the gold-coated substrate. Using the Stoney formula (1), the surface stress values can be determined to derive the sensitivity of the individual μC sensors. Although the curves in Fig. 4 exhibit the expected characteristic behavior, the maximal amplitudes differ by up to a factor of three.

3D corrugation patterns have been applied to enhance the stability of membranes and their stiffness against bending [21]. Therefore, 5 μm-wide stripes as presented in Fig. 5 were introduced into the mold and tested for cantilevers, too. Preliminary experiments show that 5 μm-deep trenches, when oriented parallel to the beam, enhance the resonance frequency, and also serve as a means to stiffen cantilevers against torsion (see Fig. 6). For separation of different patterns, only a rough alignment of the stripe patterns needs to be ensured. However, as can be seen in Fig. 6, due to the softening of the polymer mold during injection, the venting channels can be closed, leading to an incomplete filling of the mold cavities. This seems more likely with 1 μm-deep stripes in Fig. 6, particularly if they are oriented perpendicularly and not parallel to the beam.

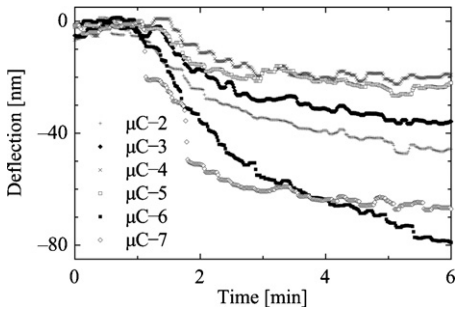

Fig. 4. Real-time monitoring of injection molded PVDF μC deflection in static mode. Formation of mercaptohexanol self-assembled monolayers on gold-coated 60 μm-thick μCs.

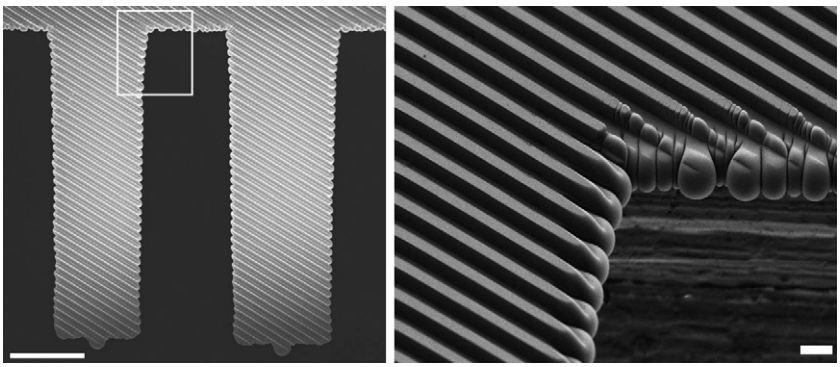

Fig. 5. SEM micrographs of the line pattern (period 10 μm, depth 5 μm, width 5 μm) transferred during the μIM process from a foil-like mold to the surface of two molded μC (left side). In contrast to the non-patterned original beams, the surface patterned beams are slightly (10%) wider due to high injection pressure and the softness of the PC foil (see extract at right side). The scale bar for the left micrograph corresponds to 100 μm and right micrograph corresponds to 10 μm.

4. Discussion

The incorporation of a foil-like mold with well-defined microstructures into the molding tool is a relatively simple approach to build microstructures on the polymer cantilevers. One can easily change the design, the size and the orientation of the pattern as demonstrated by the micrographs in Figs. 5 and 6. The orientation of the lines along the μCs is controlled without changing the device dimensions and outlines. Deep longitudinal stripes aligned within 5 μm precision promote the complete filling of the mold cavities and, hence, give rise to fully molded cantilevers. Moreover, these longitudinal channels are preferred for the contractile cell force measurements [29] as cells generally orient themselves along the ridges. Deep trenches with directions perpendicular to the beams can lead to a slight broadening of the cantilever, since material can flow in the trenches of the softened foil-like mold during injection. Different orientations, depths and patterns will be tested in future.

The differences of the heat tests in water and air are associated with an artifact, which results from the optical refraction at the air–water interface in the Concentris system. This also explains the larger data scattering. However, the main reason for the large tolerances observed in the experimental data is due to the fact the current mold exhibits large dimensional variations and a large surface roughness. The latter may be responsible that during demolding, high demolding forces induce intrinsic stress and distortion. This will be improved by using molds with reduced surface roughness.

The thiol adsorption measurements elucidate the necessity of calibration before reproducible experiments can be performed. As noted before, the deflection variations between the cantilevers originate mainly from the discrepancies in the μCs geometry. Consequently, the processing has to be improved or, alternatively, the obtained μCs have to be precisely calibrated in a more or less individual fashion.

5. Conclusions and outlook

μIM permits the fabrication of polymeric μC arrays with fair properties for biomedical applications. The choice of polymer material and geometry allows tailoring the sensor characteristics. The thiol-gold binding tests demonstrate that the prepared polymer μCs are highly sensitive surface stress monitors. Recent studies have demonstrated the applicability of μCs as olfactory sensors [30,31]. Last year one of the first clinical studies was published applying standard silicon μCs for the detection of diseases [31]. In addition, polymer μCs can be used to measure contractile cell forces [29]. By modifying its surface morphology or chemistry one can mimic implant surfaces and can compare the influence on the cell response. Thus, the microstructured μC array sensors will support the selection of advanced surface-modified substrates and medical implant surfaces.

Initial mechanical and functional tests imply that these polymer μCs are mechanically compliant for use in biochemistry and biomedicine. An additional advantage is that the polymer cantilevers can be modified adding micro- and nano-patterns to the mold cavities [32–34]. It is expected that by choosing appropriate sizes and orientations of the surface microstructures, the mechanical properties of individual μCs with identical outlines can be modified, e.g. by softening (line ridges perpendicular to beam) or stiffening (line ridges along beam) of the beam. Surface structuring can also tailor cell locomotion, adhesion and spreading, which are closely related to the contractile cell forces to be quantified. The

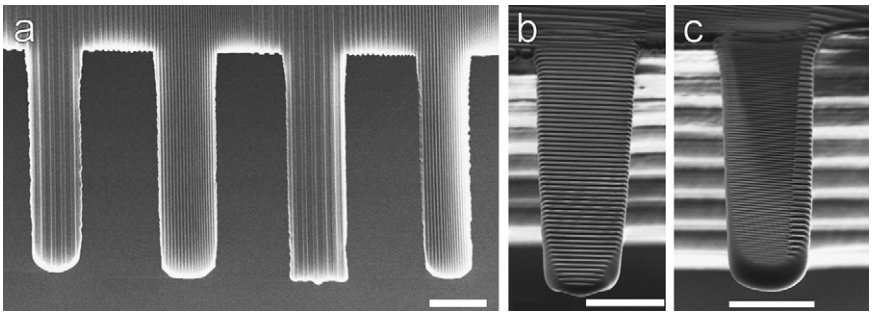

Fig. 6. SEM micrographs of PP μCs line patterns (depth 1 μm) (a) with different periods in the direction of beams and (b), (c) perpendicular to beams (right side), (c) with two patterns on one beam. The scale bars corresponds to 100 μm.

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μCs patterning can be established for a range of cantilever designs. Once successfully established, the polymer-based μC systems will permit to gain major cost reductions and to address further applications in the field of biomedicine.

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