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Disposable Polymeric Micro-Cantilever Arrays for Sensing

Prabitha Urwyler^{a,b,*}, Oskar Häfeli^c, Helmut Schiff^a, Jens Gobrecht^{a,c}, Felice Battiston^d,
Bert Müller^b

^aPaul Scherrer Institut, Laboratory for Micro- and Nanotechnology, CH-5232 Villigen PSI, Switzerland

^bBiomaterials Science Center, University of Basel, c/o University Hospital, CH-4031 Basel, Switzerland

^cUniversity of Applied Sciences Northwestern Switzerland, School of Engineering, CH-5210 Windisch, Switzerland

^dConcentris GmbH, Davidsbodenstrasse 63, CH-4012 Basel, Switzerland

Abstract

To fabricate low-cost polymeric cantilever arrays, we have applied injection molding. For polymers, including polypropylene and polyvinylidene fluoride, cantilever dimensions in the micrometer range with an aspect ratio as large as 10 were successfully manufactured. The cantilevers show a performance similar to the established silicon cantilevers. Combined with functionalization, the cantilever arrays show a great potential in biomedical applications.

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1. Introduction

Micro-Cantilevers (Cs), similar to those used in scanning force microscopes, have become increasingly popular as transducers in chemical and biological sensors. Various detection methods are introduced to measure the bending of the Cs, which is in the range of few nanometers resulting in extremely high sensitivity. A compelling feature of Cs sensors is that they can operate in air, vacuum, or liquid [1]. In the field of biomedicine, silicon-based Cs are typically applied, but for single use they are often too expensive, due to the need of clean-room based micro-machining processes. Polymer materials offer tailored physical and chemical properties that can be combined with low-cost mass production. Polymeric Cs are preferred over silicon-based, because of their tunable properties including biocompatibility, low-cost and rapid prototyping along with fitting mechanical properties, which make them particularly sensitive. Despite their advantages over silicon-based cantilever arrays, polymeric C arrays are not yet commercially available.

Cantilevers convert forces acting on them into a deflection [2], thereby relying on their deflection to indicate sensing. Forces in the pN-range can be detected which correspond to a sub-nanometer deflection of the apex of the C sensor. The detectable forces comprise expansions or contractions acting on one side of the cantilever surface,

* Corresponding author. Tel.: +41 56 310 2430; fax: +41 56 310 2646.

E-mail address: prabitha.urwyler@psi.ch

since they cause bending [2]. Both dynamic and static modes are used in sensing applications. In the static mode it is the surface stress generated when molecules selectively adsorb onto one surface of the cantilever that is measured. Cantilever free-end deflection due to surface stress is often quantified using the Stoney formula [1]. The sensitivity of the sensor depends on Young's modulus E and the cantilever thickness. In order to fabricate polymeric Cs with a sensitivity comparable to silicon ones, they should be one order of magnitude thicker to compensate the lower E .

Polymeric Cs can be fabricated in a variety of ways. The type of polymer often determines the fabrication method [1]. Polymeric Cs previously reported were prepared using photolithography, so only a limited number of materials are suitable and their fabrication is rather expensive [3]. Molding of micro-components from thermoplastic polymers has become a routinely used industrial production process and is one of the most promising fabrication techniques for non-electronic micro devices [4]. Fabrication costs of molded micro-parts are hardly affected by the complexity of the design. Once a mold insert is available, several thousand parts can be molded with little effort. The cost of the raw material in most cases is negligibly low, because only small material quantities are required for micrometer-sized components. Therefore, parts fabricated by micro-molding, even from high-end materials, are suitable for applications requiring low-cost and disposable components [4]. Several molding processes such as hot embossing, micro-injection molding (μ IM), reaction injection molding, injection compression molding and thermoforming (IM) give rise to thermoplastics micro-parts [5,6]. Polymeric replication techniques based on nano imprinting and polymer casting can be used to produce polymeric nanometer-sized structures with high precision and repeatability. The hot embossing and the μ IM seem to be the most industrially viable processes for molded micro-parts [5]. Polystyrene (PS) cantilever beams of thicknesses between 2 to 40 μm with a stiffness ranging from 0.01 to 10 Nm^{-1} were produced using IM [1]. The acceptance of C sensors in research crucially depends on the robustness, the ease of use, the reproducibility and finally the price. The question arises if disposable polymeric micro-cantilever arrays can be fabricated on the basis of standard thermal IM using precisely machined metal molds. It is the aim of the present scientific activity 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 polymeric μ Cs with a performance comparable or even better to the presently used silicon-based arrays.

2. Experimental details

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), polyvinylidene fluoride (PVDF: Kynar 720 Arkema) and liquid crystal polymer (LCP: Vectra A 390).

High quality steel (Polmax Uddeholm) mold inserts were fabricated using laser ablation (Fig. 1, left micrograph) and fixed in the three-plate molding tool 'handy mold'. The Arburg 320 Allrounder (ARBURG, Lossburg, Germany) with a maximum clamping force of 600 kN served for injection molding. The tool temperatures were varied up to 160 $^{\circ}\text{C}$ with water heating and to 260 $^{\circ}\text{C}$ with oil heating. Further process parameters are summarized in Table 1. Complete filling of the mold cavities was observed for all polymers (Fig. 2 left micrograph for PP) with the exception of PEEK, which needs higher processing temperatures than 260 $^{\circ}\text{C}$.

The injection-molded Cs were coated with 20 nm-thin gold films using an evaporator (BALZERS BAE250). This film guarantees sufficient laser beam reflectivity to use the Cantisens[®] Research system (Concentris GmbH, Basel, Switzerland) for measuring the resonant frequency and the deflection of the C.

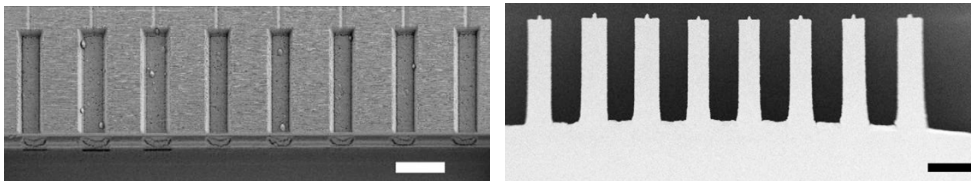


Fig. 1. The left SEM micrograph shows the array of eight laser ablated cantilever cavities in the steel mold insert. The cavity width varies from 80 to 130 μm . The scale bar corresponds to 200 μm . The micrograph on the right is an image of an injection-molded PP micro-cantilever array.

Table 1. Injection molding process parameters for different polymers. The same parameters are valid for all grades of PEEK.

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

3. Results and Discussion

With the exception of the high-performance polymer PEEK, which requires mold temperatures of up to 320 °C, the cantilevers reveal the expected thermal behavior as demonstrated in the left diagram of Fig. 2 for the gold-coated PVDF cantilever in air and water. The heat test included a temperature increase from 25 to 35 °C at about $t = 0.5$ min and a decrease back to 25 °C at about $t = 3.2$ min. The heat test indicates the sensitivity of the cantilevers that corresponds to deflections of the order of 10 nm. The deflection signal exhibits an exponential, asymptotic behavior. For the temperature difference of 10 K the maximal deflection for PVDF μ Cs in air corresponds to (95 ± 16) nm and (55 ± 5) nm for thicknesses of 30 μ m and 40 μ m, respectively. In water, these values increase to (127 ± 17) nm and (154 ± 55) nm as the result of the refraction at the interfaces.

The Cantisens[®] Research system permits the experimental determination of resonance frequencies f and quality factors Q for the polymeric μ Cs. Table 2 summarizes the mean values and related standard deviations of the resonance frequency measurements for the μ Cs in air and water.

The resonance frequency expected is estimated using the μ C-length L , the μ C-thickness t , the polymer density ρ and the bulk elastic modulus E of the polymer. 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 . The drop in resonance frequency in water results from the damping, which lowers the Q -factor of the μ Cs as given in Table 2. The Q -factors were estimated directly from the frequency spectra.

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

Polymer	30 μ m PP	40 μ m PP	30 μ m PVDF	40 μ m PVDF	30 μ m POM-C
Frequency f in air [kHz]	48 \pm 3	50 \pm 1	60 \pm 3	79 \pm 5	60 \pm 4
Frequency f in water [kHz]	37 \pm 8	33 \pm 27	43 \pm 5	52 \pm 5	36 \pm 7
$f = 0.162 \frac{t}{L^2} \sqrt{E/\rho}$ [kHz]	38	46	66	88	78
Q -factor in air	28	46	38	19	33
Q -factor in water	20	11	10	9	19

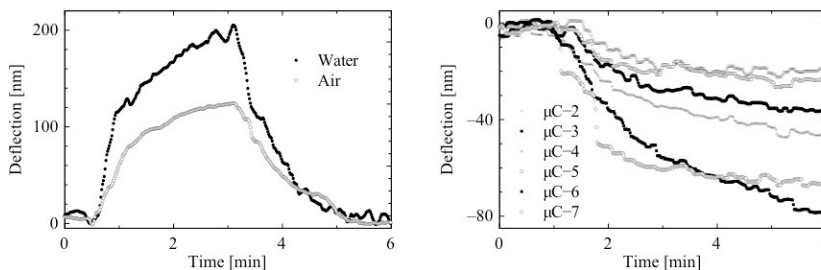


Fig. 2. (a) heat test of 30 μ m-thin injection-molded PVDF μ Cs; (b) deflection curve of 60 μ m-thin PVDF μ Cs during the binding with thiol molecules on the gold-coated side

As a first attempt towards biosensing, thiol's bonding at the gold-coated surface of the μ Cs was recorded by means of the Cantisens[®] Research system. The measured 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, the surface stress values can be uncovered to derive the sensitivity of the cantilever sensor. Figure 2 represents deflection curves of the six central cantilevers of a 60 μ m-thin PVDF array. Although the curves have the expected characteristic behavior, the maximal amplitudes differ by up to a factor of three, which is mainly due to the variations of the μ Cs-geometry. Note the cantilever width, for example, varies from 80 and 130 μ m.

4. Conclusions and Outlook

Injection molding permits the fabrication of polymeric micro-cantilever arrays with reasonable properties for biomedical applications. The choice of polymer and geometry allows tailoring sensor characteristics. The preliminary thiol binding tests demonstrate that polymeric μ Cs are highly sensitive surface stress monitors. The resonance frequencies of the polymeric μ Cs are higher than their silicon counterparts making them especially suitable for gas sensing. Recent studies have demonstrated the applicability of μ Cs as olfactory sensors [7,8]. Last year one of the first clinical studies was published applying standard silicon μ Cs for the detection of two diseases [8].

In addition, polymeric μ Cs can be used to measure contractile cell forces as described by Köser et al. [9]. Modifying its surface morphology or chemistry one can mimic implant surfaces and can compare the influence on the cell response in physical manner. Thus, the disposable μ C array sensors will support the selection of advanced surface-modified substrates and medical implant surfaces, along with opening more applications in the field of biomedicine.

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