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REVIEW

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Recent advances in microcontact printing

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Abstract Microcontact printing is a remarkable surface patterning technique. Developed about 10 years ago, it has triggered enormous interest from the surface science community, as well as from engineers and biologists. The last five years have been rich in improvements to the microcontact printing process itself, as well as in new technical innovations, many designed to suit new applications. In this review, we describe the evolution of microcontact printing over the past five years. The review is categorized into three main sections: the improvements made to the technique, new variations, and new applications.

Keywords Microcontact printing · PDMS · Polydimethylsiloxane · Surface patterning · Self-assembled monolayers · Soft lithography

Introduction

Surface interactions are of prime importance to all natural and artificial phenomena. It is therefore essential to be able to control the characteristics of surfaces in order to design surfaces with specific chemical properties. Over the past twenty years, studies of phenomena such as molecular interactions and miniaturization of technical devices has created the need for spatially-controlled chemical modification of surfaces on reduced scales.

Soft lithography [1, 2, 3, 4, 5] was developed in order to control specific properties of surfaces at micro- and nanoscale levels through the use of a parallel fabrication process for surface patterns. Soft lithography encompasses a family of techniques based on the process of molding a soft polymer using hard masters. Pioneering work in soft lithography was performed by G.M. Whitesides and co-workers [2, 6–40], followed by many others [41–62], resulting in both application development and studies of the parameters involved in the process.

Varying the way that the molds are used produces different techniques, the main ones being replica molding (REM) [33], micromolding in capillaries (MIMIC) [63, 64], microtransfer molding (μTM) [65], solvent-assisted microcontact molding (SAMIM) [66] and microcontact printing (μCP) [67]. The present review will focus on the latter.

In the original version of μCP, the micrometer-scale patterned chemical modification of a large surface area is obtained by transfering different types of compounds using a soft polymer stamp (Fig. 1). Polydimethylsiloxane (PDMS) is the material most frequently used to make the stamps, since it can be molded using a master and it results in a soft polymer, which allows for a conformal contact between the stamp and the surface to be modified. The stamp is subsequently soaked in a molecular "ink" that is imprinted on the surface.

As simple and efficient as it is, μCP does nevertheless present some problems. The use of the soft polymer is at the origin of the main problems of μCP. Swelling of the stamp during "inking" often results in the pattern increasing in size. Moreover, an excess of ink results in enhanced diffusion of the imprinted molecules on the patterned surface. Diffusion of non-covalently-bound molecules occurs after the printing as well. Finally, the

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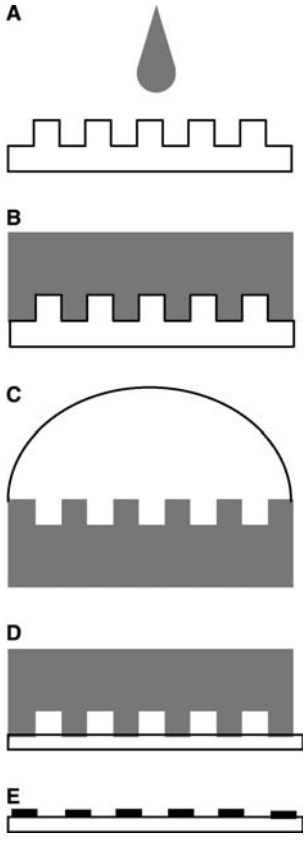

Fig. 1 Schematic representation of the microcontact printing process. PDMS is applied to a masterdesign (**a**) and allowed to cure (**b**), forming a mold/stamp. After peeling the stamp from the master, "ink" is applied (**c**), and the ink is transferred to a substrate (**d**) by stamping. After removal of the stamp, the ink is patterned on the substrate (**e**)

hydrophobicity of PDMS is a problem, if combined with polar inks. Deformation of the soft polymer stamps due to their elastomeric natures, such as pairing, buckling or roof collapse of structures during contact with the surfaces, is a problem that results in distorted patterns [38]. Such deformations are illustrated in Fig. 2. These phenomena are enhanced when the size of the corrugations reaches the submicron- or nanoscale.

Another major drawback of soft lithography is the contamination of the patterns with unpolymerized low molecular weight siloxane from the stamp. Peeling the stamp from the master is also a concern in stamp fabrication in general, and with nanometer-scale corrugations in particular. In addition, the forces exerted on the stamp during contact with the surfaces also influence the

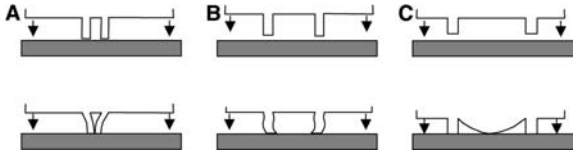

Fig. 2 The most commonly observed stamp deformations: **a** pairing, **b** buckling, and **c** roof collapse

pattern reproduction. These problems have limited the size of the patterns to the micron scale. In recent years, efforts were made to shrink the size of the patterns to the nanoscale. To overcome the obstacles described above, stamp production optimization has been crucial. Furthermore, improvements in printing conditions have enabled the possibility of patterning with nanoscale dimensions.

While μCP allows for lateral control of chemical modifications, vertical control has also been crucial to the development of micro- and nanotechnology. Self-assembled monolayers (SAMs) [12, 13] play an important role in spatially controlled chemical modification, since our understanding of their formation mechanism has allowed us to control the vertical dimensions and bulk structures of chemical coatings. Micropatterned SAMs on surfaces have been used to build structures [18, 26, 46] of diverse compositions.

Although initially mainly used as a method for patterning self-assembled alkylthiol monolayers onto gold surfaces [13, 14, 17, 44], μCP was extended to alkylsiloxanes on silicon oxide [12], and this resulted in numerous biotechnology applications, such as patterned growth of a variety of cells and fabrication of microarrays for biosensor purposes. The range of ink molecules has been extended from alkylsilanes and alkylthiols to various particles and organic molecules with higher molecular weights, ranging from Langmuir-Blodgett films [68] to DNA [69, 70] and proteins [71].

Subsequently, numerous techniques derived from the same principles have been used to produce patterns not only by transferring various molecules onto various surfaces, but also by transferring metals [15, 16]. Electromagnetically-patterned surfaces have been produced [72], and patterned electrochemical reactions have been performed [73, 74] in a similar way. Several new applications of μCP have been developed that will be discussed in more detail below.

This highlights the important role μCP plays in many fields requiring surface modifications. Numerous reviews have already been devoted to soft lithography techniques [1–4, 9, 10, 34, 45, 75, 76] and, more specifically, μCP [10, 45, 75]. In this review, we describe the evolution of the field of μCP over the past five years, categorizing our review into three main sections: the improvements made to the technique to solve some of the problem issues discussed above, new variations of the technique, and new applications.

Improvements

Inking process

Pompe et al [77] developed a "stamp pad" method, where the PDMS stamp was placed in contact with a surface wetted with the "ink", thereby adsorbing a minimal amount of solution. This method reduces the swelling of the stamp and the diffusion of the molecules

after patterning. A similar result was achieved by Libioulle et al [78] using an elastomer soaked in ink as an inker pad to localize the inking to the stamp corrugations. Diffusion of the printed molecules during and after the printing process is another problem, and is size-dependent. Bass and Lichtenberger [79] showed that a higher molecular weight alkylthiol such as octadecanethiol diffuses less on a gold surface compared to hexadecanethiol. Diffusion of non-covalently-bound molecules occurs after the printing, and this was investigated by Workmann and Manne [80]. They demonstrated the influence of ambient conditions—temperature and air relative humidity—on the diffusion.

Depending on the molecules to print, different degrees of wettability are desired. Li et al [81] used dendrimers, highly branched polymers with a globular architecture, as ink to imprint nanometer-scale patterns onto silicon (Fig. 3) using a "Material A" stamp, a modified PDMS polymer with a high elastic modulus allowing for transfer of patterns down to 100 nm [82]. The advantage of using dendrimers is the possibility of drying the PDMS stamp, which cancels out all of the detrimental effects of an excess solution.

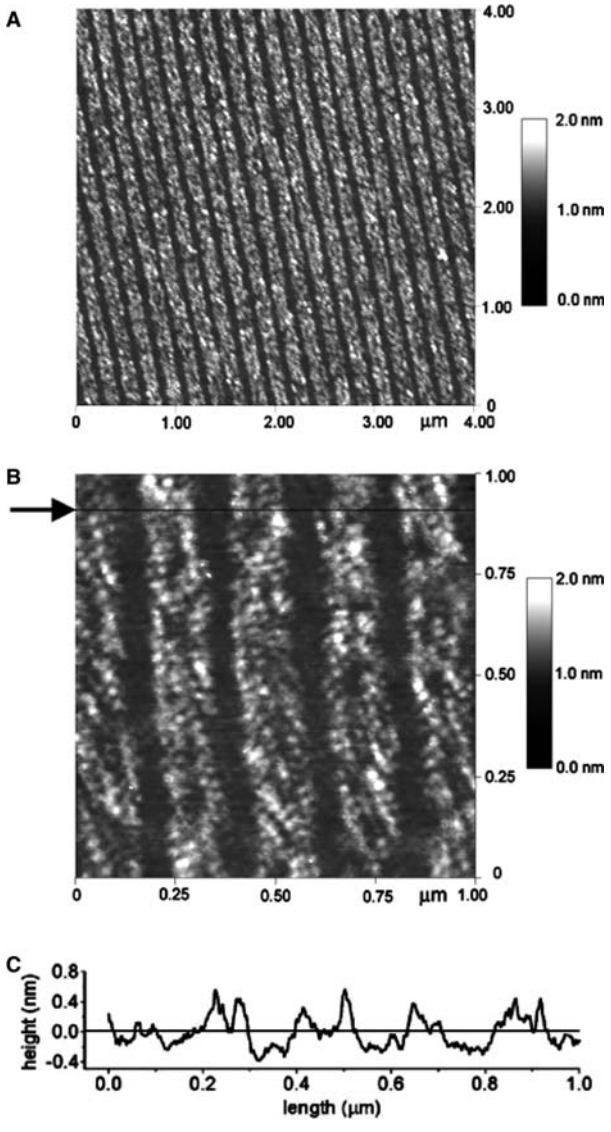

Fig. 3 **a** and **b** AFM images of printed dendrimer lines on a silicon surface. Periodicity is 210 nm, line width is 140 nm. **c** The cross-section of dendrimer lines in **b** at the position indicated by the *arrow* (reprinted with permission from Li et al [81]. Copyright (2002) American Chemical Society)

Certain experimental conditions require the use of hydrophilic ink, for which the hydrophobicity of the PDMS is a drawback. As an alternative to microwave plasma treatment [83], Trimbach et al [84] developed a hydrophilic stamp based on poly(ether-ester) to overcome this problem. A new type of inking process, resulting in micro-fluid contact printing [85], is based on an ink film on the surface of the stamp, which, when dewetted, results in droplets of ink smaller than the corrugations on the stamp surface. Instead of modifying the inking procedure or the stamp itself, the approach of Zhang et al [86] proceeds by whittling stamped microscale structures down to the nanoscale using electrochemical desorption.

Stamp deformation

A thorough theoretical analysis of the parameters influencing stamp deformation was performed by Hui et al [87]. They introduced novel constrains in stamp design and forces to be applied. He et al [88] performed an angular evaluation to study planar distortion of planar PDMS stamps, which supported the use of thinner stamps on a rigid backing and an auto-mechanical printing system. Recently, deformation-proof stamps were developed, to prevent distortion of the patterns resulting from buckling and roof collapse (see Fig. 2). Suh et al [89] developed a PDMS stamp with corrugations that were reinforced by chemical vapor deposition polymerization of poly(*p*-xylylene) on the structure's side walls. Modified PDMS "Material A" [82] or block copolymer poly(styrene-*block*-ethylene-*co*-butylene-*block*styrene) [90] also proved to be good candidates for the fabrication of deformation-proof stamps.

The use of a flat stamp patterned with inks that have high affinities for PDMS and low diffusion from a patterned inker pad is another way of avoiding the deformations previously mentioned, as was shown in studies performed by Geissler et al [91].

Siloxane contamination

To overcome this problem, the cured PDMS stamp can be briefly washed with heptane, a method used by Kumar et al [6] since their introduction of microcontact printing. After investigating the transfer of siloxane from stamps to surfaces, Graham et al [92] determined that a week-long wash was necessary to bring the contamination below the detection level. Siloxane contamination was recently more extensively investigated by Glasmaestar et al [93],

giving a detailed overview of the phenomenon. Their finding is that UV/ozone treatment of the stamps significantly decreases the amount of contamination.

Separation of stamp from master

Different methods can be employed to avoid adhesion to the master. They include coating of InP masters with self-assembled monolayers of perfluorinated thiols [81], passivation of silicon masters with hydrogen fluoride [73], or treatment with sodium dodecylsulfate [94].

Forces

Finally, the ultimate refinement of μCP is to thoroughly control the forces exerted on the stamp during the contact. Burgin et al [95] studied the use of a contact aligner to optimize submicron pattern printing, and showed that it is possible to transfer the pattern over inch-wide areas with a high precision.

New variations

Modified stamps

Some new variations, such as organic smart pixels [96] and optical devices with wide fields of view, which are performed through patterning of curved surfaces, require modified stamps. Rhee et al [97] described a method for producing non-planar stamps that satisfied this demand. The need to produce gradients on surfaces using μCP was addressed by Choi and Zhang Newby [98]. They showed that gradients could be produced by applying a pressure to stamps with different shapes, which resulted in various contact times. Paul et al [99] investigated the use of an elastomeric membrane as a mask to pattern a spherical surface. In patterning experiments that require a high pressure for the transfer process, it is necessary to use silicon hard stamps [100, 101] instead of deformable elastomers.

Transfer of metals

The transfer of patterned metals instead of organic molecules [15, 16] was introduced in 1996. More recently, Kind et al [102] transferred Pd patterns onto a titanium layer using an organometallic form of Pd solubilized in ethanol. Yang et al [103] investigated the transfer of metal ions such as nickel and copper from a PDMS stamp. Patterns of colloidal metals such as gold were also successfully transferred by Schmid et al [104]. Alternatively, μCP was used to deposit organic inks containing phosphine groups that bind a colloidal catalyst that initiates electroless metallization [105], a low-cost approach to selectively depositing films of nickel and copper.

Solid metal can be used as an alternative to ''wet'' ink. Kim et al [101] showed that it was possible to transfer metal patterns from a metallized hard Si stamp to a metallized surface by cold welding (by applying a pressure to the Si stamp sufficient to fuse the metal layers). Loo et al [106] described the nanotransfer printing (nTP) of metals based on the brief contact and simultaneous condensation reaction between oxidized surfaces and metallized PDMS stamps.

Electromagnetic patterning

Magnetic signals were successfully duplicated using μCP by Nikitov et al [72]. Using external magnets and patterned Fe dots as a master, they duplicated magnetic information by contact printing to a magnetic slave-film. Similar results were obtained by Presmanes and Tailhades [107], also using magnetized iron patterns. This method can be used to duplicate bits on harddisks or to write bits on floppy media.

Jacobs and Whitesides [108] established the use of conductive metallized stamps to perform the transfer of patterns of charges to a surface. A more recent study by Schmid et al [104] showed that this process could be applied to the patterning of organic photoluminescent and fluorescent surfaces. The most recent variation of μCP, using a conductive PDMS stamp to apply an electric field locally, uses current flow from a conductive surface to the stamp corrugations, allowing the creation of an optical waveguide by modulating the local refractive index of a doped polyvinylphenol film [109].

Electrochemical transfer

Other examples of the use of a conductive PDMS stamp include the transfer of patterns to surfaces through electrooxidation of the top-most atoms of an organic monolayer. Such a process has been described by Hoeppener et al [74], who used a copper transmission electron microscope grid as a ''hard'' conductive stamp to create carboxyl groups in an alkylsilane self-assembled monolayer. This method allows for controlled hydrophobicity/hydrophilicity of the surface. Pavlovic et al [73] developed a similar method based on the use of a metallized PDMS stamp, which enables the electrochemical oxidation of thiol groups on the silicon surface to reactive groups, allowing patterned covalent binding of thiolated molecules and particles (Fig. 4). The stamp functions as both reference and counter electrode. This method has the advantage of forming stable covalent bonds instantaneously with high efficiency at neutral pH in water solutions. The covalent disulfide bond is reversibly reduced by using dithiothreitol (DTT), followed by subsequent regeneration of the thiol surface.

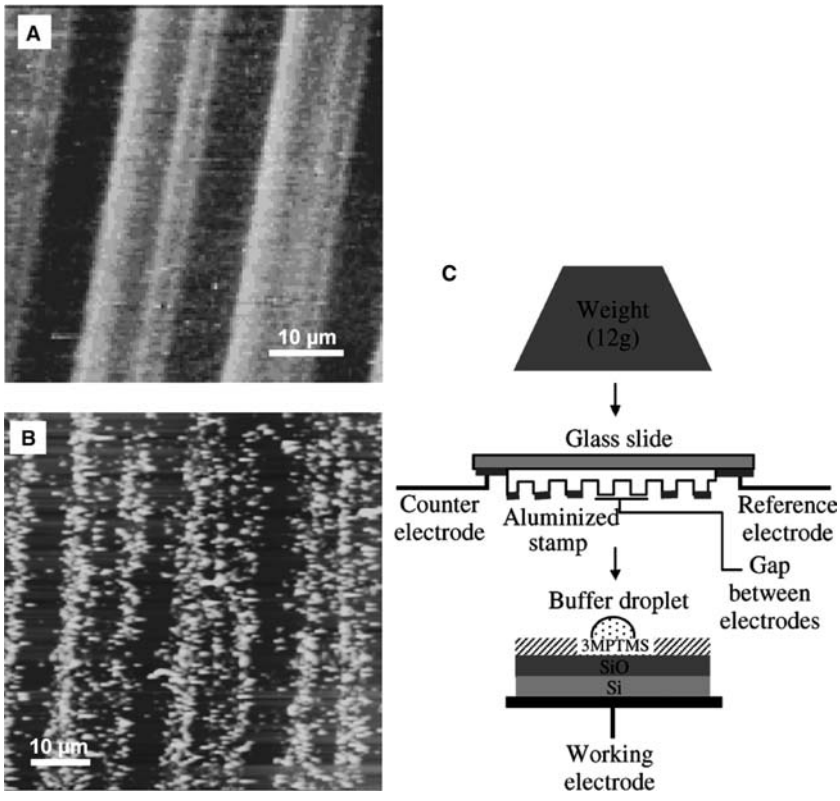

Fig. 4 **a** Friction AFM image of a thiolated surface electroactivated at a potential difference of 0.65 V versus Ag/AgCl; **b** tapping mode AFM image of polystyrene particles immobilized on the linear electroactivated pattern, with a height scale of 40 nm; **c** principle of electro-oxidation through microcontact printing. The stamp functions as both reference and counter electrode (reprinted with permission from Pavlovic et al [73] Copyright (2003) American Chemical Society)

Affinity contact printing

A new μCP technique, in which the corrugations of the PDMS stamp are inked with antibodies as ''capture molecules'', allows binding of selective proteins from a mixed solution to the stamp. The proteins are subsequently transferred to the surface to pattern (Fig. 5). This technique, named ''affinity contact printing'' (αCP) was developed by Bernard et al [110], and enables patterning of surfaces by proteins after their simultaneous separation and concentration. Microarrays of proteins were successfully produced by Renault et al [111] using this process.

Lift-off μCP

The μCP process transfers a substance from the surface to the stamp or from the stamp to the surface. In lift-off μCP, a PDMS stamp is used to locally withdraw

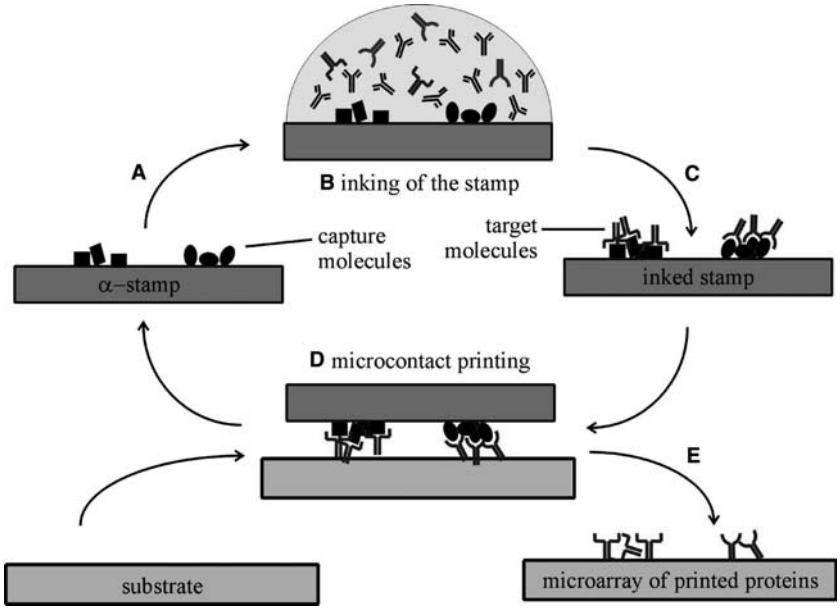

Fig. 5 Microarrays of proteins on surfaces can be fabricated using an α-stamp derivatized with various capture sites that can extract target biomolecules from a complex solution and release them onto a surface in a single microcontact-printing step. The α-stamp can be reused for several inking and printing cycles (reprinted with permission from Renault et al [111]. Copyright (2002) Wiley-VCH)

material from a surface, thus creating patterns. Cold welding also allows a metal layer lift-off process, as demonstrated by Kim et al [100]. This is the reverse case from the one described above [101], since the peeling of the stamp locally removes the metal layer from the surface to pattern. Recently, Yao et al [112] showed that it is possible to peel off silica microspheres from a silicon surface after heating and pressing, then cooling and peeling a PDMS stamp. A lift-off process achieved through simple adhesion of porous silicon to a PDMS stamp enabled Sirbuly et al [113] to pattern thin layers of porous silicon onto a silicon surface.

Hybrid dip-pen

A new variation resulting from a hybrid dip-pen lithography [114] and microcontact printing, named "scanning probe contact printing" or SP-CP, was studied by Wang et al [115]. An atomic force microscope tip is fitted with an elastomeric shaped end, enabling the transfer of an "ink" solution to a surface. Patterns are drawn by bringing the tip into successive contacts with the surface. This technique loses the main advantage of classic μCP, which is the patterning of large areas.

New applications

Sensors

One of the most potent applications of μCP is the fabrication of microchips for use in bio- or chemical sensors. Urbanowska et al [116] describe the fabrication of a protein microarray for the detection of rheumatoid arthritis biomarkers. A chemical array permitting the determination of the enantiomer purity of L- and D-aminoacid mixtures has been investigated by Korbel et al [117]. Xiao et al [83] describe a method for transferring patterns of oligonucleotide synthesis reactants to glass slides, enabling in situ synthesis of oligonucleotides. μCP was also used to print proteins onto a Au/Ta_2O_5 surface to produce a surface plasmon resonance chip with internal reference [118].

Covalently printed fluorophore molecules (Fig. 6) were patterned onto amino-terminated SAMs [119], resulting in a ion sensor that has the advantages of label-free analytes and binding groups, easy analysis and high throughput screening. Shim et al [120] used μCP to pattern silicon surfaces with aminopropyltriethoxysilane (APS) and covalently attached chemically modified liposomes to the micropatterns, with potential applications to chemosensor technology. APS was also used to pattern Co particles onto surfaces [121]. Patterned pyrrole-terminated alkylthiol SAMs were used to create patterns of electropolymerized pyrrole on gold [122]. The resulting polypyrrole/polymethylene patterned surface may be used for sensor applications and light emitting diodes.

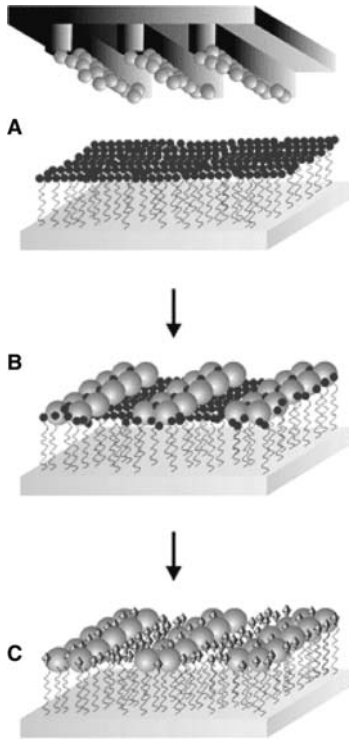

Fig. 6 Generation of patterned, sensitive monolayers using μCP. **a** An amino-terminated monolayer on a glass surface is brought into contact with a PDMS stamp inked with a fluorophore. **b** This results in the covalent attachment of the fluorophore to the amino-terminated monolayer, yielding a patterned fluorescent monolayer. **c** Subsequent immersion of the sample into a solution of reactive molecules allows the functionalization of the remaining free amino groups (reprinted with permission from Basabe-Desmonts et al [119], Copyright (2004) American Chemical Society)

Catalytic surfaces

Recently, cytochrome C has been patterned onto gold surfaces by Kwak et al [123] using direct μCP as well as indirect dip-pen lithography. Micrometer arrays of Cytochrome C were obtained by using Cytochrome C as ink in direct patterning, while submicron (200 nm) patterns were obtained by first patterning 16-mercaptohexadecanoic acid using dip pen lithography, and subsequent exposure of these patterns to a Cytochrome C solution. Active enzymes were successfully patterned using SAMs on gold surfaces [124]. High local enzyme activity of thiolated horseradish peroxidase was found after direct patterning of the enzyme onto gold surfaces [124]. Such direct patterning is beneficial in creating multi-enzyme-patterned surfaces.

Polymers and biomolecules

Hydroxylated surfaces were patterned with dendrimers of polyamidoamine (PAMAM) [81], resulting in 140 nm wide lines of a single dendrimer layer (Fig. 3). Dendrimers, due to their compact size and monodispersity, may prove suitable especially in nanotechnology applica-

tions. Patterns of amine-terminated PAMAM were used as stabilizers for the growth of photoluminescent CdS nanoparticles, simultaneously functioning as media between the particles and the silicon surface [125]. Amine-terminated PAMAM was also used to pattern reactive dendrimers on activated SAMs on gold [126]. Organic dendrimers may function as nanoreactors and as such act as a host for the growth of (luminescent) nanoparticles. The deposition of dendrimer multilayers on several substrates by μCP, and the effect of ink concentration, contact time and inking method have also been recently studied [127].

Surface-initiated polymerization resulting in covalently-bound, dense polymer layers is an important step in the fabrication of integrated systems. μCP of an unsaturated alkylsilane SAM derivatized with a Ru catalyst onto a silicon surface resulted in ring-opening metathesis polymerization of norbornene onto catalyst patterns [128]. This may be developed further in order to pattern polymer films with high resolution.

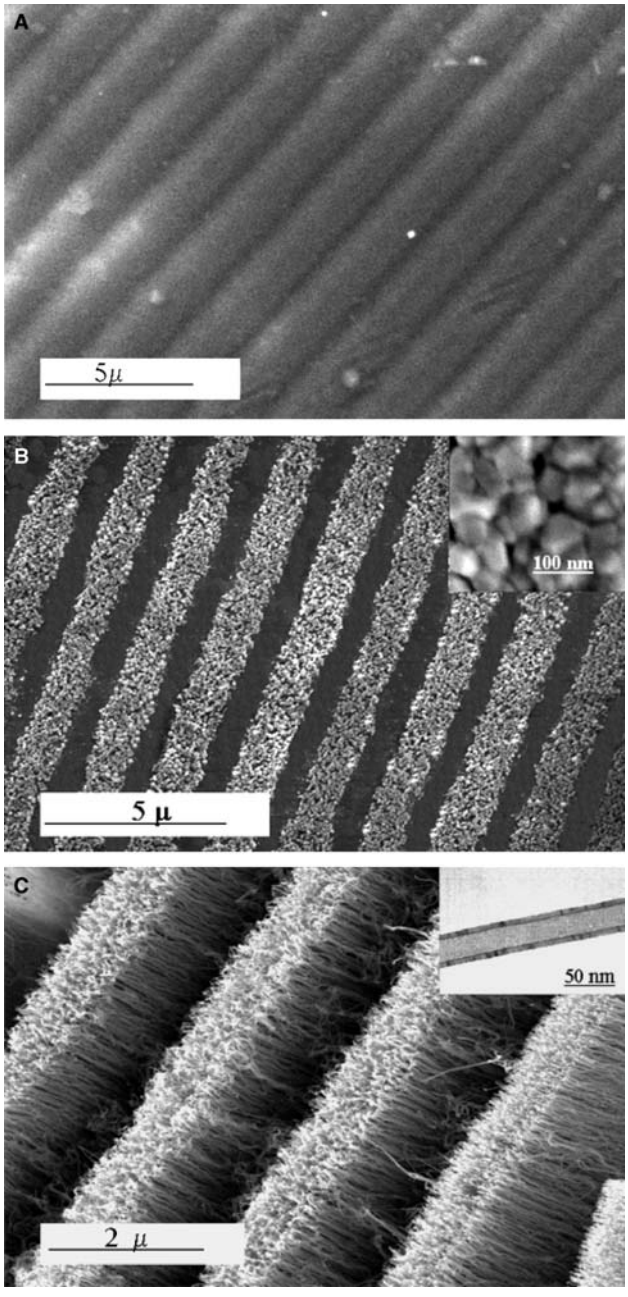

Fig. 7 SEM images of: **a** parallel line octadecyltrichlorosilane (*OTS*) pattern on a quartz substrate from micro-contact printing (μ *CP*); **b** parallel line silver pattern formed by selective deposition of silver on OTS-free regions by the silver mirror reaction (*inset* high magnification of SEM image showing the densely-packed silver nanoparticles); **c** aligned CNTs grown on silver-free regions by pyrolyzing iron(II) phthalocyanine (FePc) (*inset* TEM image of individual nanotubes). (Reprinted with permission from Huang et al. [129]. Copyright (2003) American Chemical Society)

Nano-electronics

Carbon nanotubes (CNT), which are involved in state-of-the-art nanotechnology and find applications in nano-electronics, can be grown by the pyrolysis of iron(II) phtalocyanine. Huang and Mau [129] and Huang et al [130] (Fig. 7) demonstrated such growth on silver-patterned SiO surfaces, prepared using μCP of octadecyltrichlorosilane (OTS) and subsequent exposure to Tollens solution ($Ag(NH_3)_4OH$). Such methods to create patterned and oriented CNT with high resolutions will be useful for CNT applications such as field emission and electrochemical CNT modifications. The same authors also studied the possibility of obtaining laterally-patterned CNT by directly patterning the catalyst onto surfaces using μCP [131]. Other ways to obtain a patterned and controlled growth of CNTs include the stamping of a catalyst polymeric precursor [132] for subsequent catalytic chemical vapor deposition (CCVD).

μCP-patterned SAMs used as resists and templates

Tightly-packed SAMs of alkylthiols on gold have been created as working resists to use with selective wet etching processes on gold [2, 45], as well as silver and copper [75]. A similar use of alkyloxysilanes on silicon is not possible due to the disorder in alkyloxysilane SAMs [2]. More recently, in order to overcome this problem, Finnie et al [133] investigated the use of docosyltrichlorosilane on silicon surfaces as a resist for wet etching applications. Carvalho et al [134] showed that eicosanethiol SAMs on Pd can also be used as resists for wet etching.

Self-assembled monolayers were also used as templates for patterning metals on surfaces [18], as well as inhibitors of metal nucleation during chemical vapor deposition, forming metallic patterns on areas not covered by the SAMs [135]. This phenomenon was used by Park et al [136] to pattern gas-phase deposited TiO on silicon surfaces.

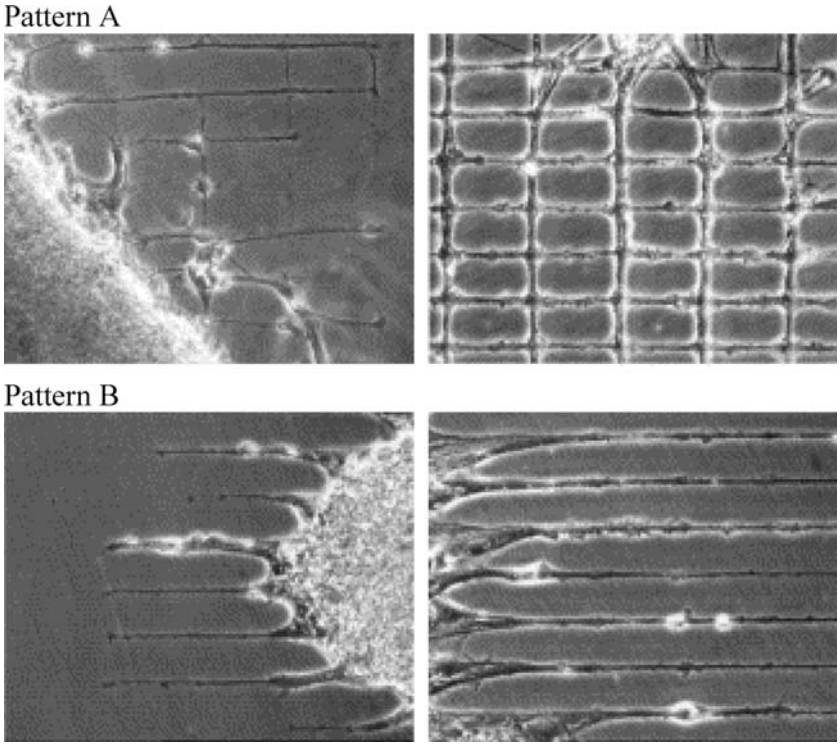

Fig. 8 Evaluation of a variety of extracellular matrix protein patterns. During the early days of the culture (left, day 3), the migrated neurons and growing processes are clearly visible with little overlapping on the patterns. These cultures continued to mature, and by ten days or more (*right*) the original shapes of the laminin stamped patterns were identifiable (reprinted from Yeung et al [143], with permission from Elsevier)

Cell biology

Classic microcontact printing engineered surfaces had a large impact on the study and control of cell growth. The possibility of patterning cells on surfaces [51, 56, 76, 137, 138] and the influence of patterning on cell physiology [25, 27] has been studied by several groups. The most popular method to achieve spatially-controlled cell growth is to pattern specific cell adhesion molecules such as the RGD peptide [139] onto surfaces, which can also be used in association with patterns of protein-repellent PEG molecules [140]. Patterning of protein-repellent PEG molecules onto Si surfaces using μCP was performed by Jun et al [141]. The efficiency of PEG as a non-permissive substrate in long term experiments (29 days) was reported by Branch et al [142]. Applications in patterned neural growth [94, 143–145] open up possibilities for viable neuronal networks. Figure 8 shows the guided neurite growth demonstrated by Yeung et al [143] using μCP patterned surfaces. Recently, cell motility was studied using micropatterned surfaces [146] and it was shown that cells can sense limits of areas patterned with extracellular matrix. As a new application of μCP, electrochemical μCP also results in novel applications. Bovine endothelial cells were released from octadecanethiol patterns by electrochemically desorbing the underlying SAM [147].

Conclusion

There is constant interest in microcontact printing, and in recent years many new applications have appeared in a wide variety of fields ranging from electronics to optics, chemical sensing, and cell biology. Such applications make use of some of the great benefits of microcontact printing. The technique is extremely flexible in terms of the shapes of the patterns obtained. Furthermore, it enables us to control the chemistry at the molecular level. In recent years, many studies have been performed that were aimed at minimizing some of the main drawbacks of microcontact printing, such as stamp deformation and ink-transfer issues. Such advances will allow the technique to become even more applicable to many fields, and in particular they should enhance the possibilities of patterning nanometer-scale structures with high precision.

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