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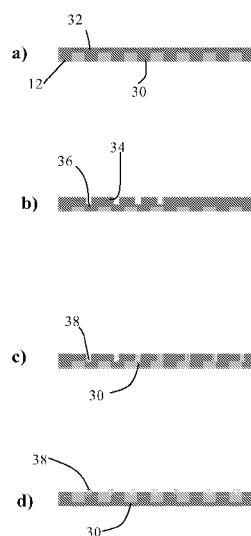
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Fig. 3



(57) Abstract: A method for creating nanotemplates using solvent assisted dip pen nanolithography is described. In this method a substrate is coated with a thin layer of polymer, the polymer is removed using a tip, for example the tip being coated with an ink consisting of either a solvent or a solution, resulting in nanotemplates.



MASKLESS MICRO- AND NANO - TEMPLATING

Field of the Invention

The present invention relates to the fabrication of micro- and nano-structures.

Background to the Invention

Nanostructures (on scales of less than 100 nm) and microsystems (on scales of less than 100µm) offer an interesting platform for the study of physical and chemical properties at single particle level. As the field of microsystems and nanotechnology is expanding, new ways of fabricating structures have been evolving. Traditionally, e-beam lithography involving the use of a high-energy electron beam has been used for fabrication on this scale, however this technique is very expensive. Scanning probe lithography and nanoimprint lithography have become popular over the last decade. These techniques are limited by the choice of ink material and substrate surfaces.

Dip Pen Nanolithography (DPN), which involves dipping a pointed tip into 'ink' and then 'writing' or depositing the ink onto a substrate, offers a desktop route to fabricate micro- and nano-structures and for micro- and nano-patterning of substrates. However one of the main requirements of this technique is compatibility between the ink used and the substrate onto which it is written. The substrates that can be used are mainly limited to gold, silicon and silicon dioxide. The nanopattern dimension in this case depends on dwell time, writing speeds, solubility and diffusion constant.

Nanotemplates formed by directly writing organic molecules such as mercaptohexadecanoic acid (MHA) and octadecanethiol (ODT) have been reported earlier. These templates allow controlled placement and configuration of single particles such as carbon coated iron nanoparticles (NPs) and carbon nanotubes. More recently Huang *et al.* (Huang, L.; Braunschweig, A. B.; Shim, W.; Qin, L. D.; Lim, J. K.; Hurst, S. J.; Huo, F. W.; Xue, C.; Jong, J. W.; Mirkin, C. A., Matrix-Assisted Dip-Pen Nanolithography and Polymer Pen Lithography. *Small* 2010, 6, (10), 1077-1081) reported the use of poly (ethylene glycol) (PEG) matrix assisted DPN and polymer lithography for patterning Au, Fe₃O₄ and C₆₀ nanoparticles. PEG here acts as a carrier of the nanoparticles and once deposited the PEG is removed by oxygen plasma treatment and subsequent washing steps. This approach addresses some limitations of the simple direct writing of molecules but it still necessitates

surface blocking for the next stage to allow specific interaction between the NPs and the molecules.

Summary of the Invention

The present invention provides a technique for fabrication of templates of micro- and nano-structures based on the selective removal of material by a tip carrying a suitable solvent, which may be in a solution comprising an organic solvent saturated with an aqueous phase.

According to one aspect of the invention there is provided a method of fabricating a template, the method comprising providing a substrate, providing a polymer layer on the substrate, and removing parts of the polymer layer selected using a tip so as to form the template. The tip may be a scanning probe microscopy tip. The template may be for a micro- or nano-structure. For example it may be less than 1mm in size. However the invention has particular application for templates of less than 100µm in size. The polymer layer may be spun on the substrate. The tip may carry a solvent, for example having been dipped in solvent or being connected to a reservoir of solvent, so that it can be used to apply solvent to the polymer layer. The parts of the polymer layer to be removed may then be dissolved, or softened, by the solvent.

The polymer may be a photoresist.

The solvent may be an organic solvent. The solvent may be in a solution or mixture comprising the solvent saturated with an aqueous phase. This may soften the polymer layer in the areas contacted by the solvent. In some cases this may form a liquid, or it may soften the polymer, and it may form a solution of the solvent and polymer. The solvent may be in the form of an organic phase solvent saturated with an aqueous phase e.g. Toluene saturated with water. However other aromatic solvents can be used.

The parts of the polymer may be removed, for example by removing the liquid or solution, to leave a template formed by the remaining polymer. The removal may also be performed using the tip. The polymer may be removed in solution in the solvent. The removal of the polymer by the use of solution in the solvent gives precise control of the dimension of the micro/nanotemplates.

The tip may be moved over the polymer to remove an area of the polymer. The tip may be arranged to apply the solvent to the polymer simultaneously with the removal of the polymer. The solvent may be arranged to facilitate swelling and dissolution of the polymer. The area of the substrate from which polymer is removed may have a width in the direction perpendicular to the direction of movement of the tip. The speed of movement of the tip relative to the polymer may be controlled so as to control the width of the area. The speed of movement may be varied during the fabrication of a template so that the template has areas of different widths, or the speed may be selected from a range of speeds and then kept constant during formation of a template.

According to a further aspect of the invention there is provided a method of forming a nanostructure comprising fabricating a template according to the first aspect and then applying metal, or other material, to the substrate, for example in at least one area of the substrate from which the polymer has been removed. For example, once the template is obtained the nanostructure can be elaborated by metallization, further etching of the exposed substrates, or dispensing a liquid onto the region of the template. For e.g. after the templates have been created, they can be metallised by e-beam evaporation or electroplating or introduction of nanoparticles solutions. The method may further comprise removing the template from the substrate. Once the metallic structures are formed the polymer layer (for example the photoresist) can be stripped off, for example using acetone, thereby leaving the nanostructures in place.

The present invention further provides apparatus for forming a template comprising a means arranged to control movement of the tip over the polymer. The control means may be arranged to control the speed of the tip as described above. The control means may be arranged to control the speed of movement of the tip relative to the polymer layer thereby to control the width of one of the parts of the polymer layer that is dissolved. The apparatus may comprise support means for the tip. The control means may be arranged to control movement of the support means. The control means may be arranged to control a dwell time for which the tip is in contact with the polymer layer thereby to control the size of a part of the polymer layer that is dissolved. The apparatus may comprise a support for the substrate.

The advantage some embodiments of the invention offer over micro- and nano-templates formed by direct writing is that they provide reservoirs (which may be of

femtolitre volumes) that can be filled with a liquid material the nature of which will depend on the process being performed, and so allow longer incubation times for reaction with the substrate thus enabling a wider range of combinations to be used. A further aspect of this technique is that it may remove the need for blocking agents to prevent non-specific adsorption. This may allow the development of methods to obtain high throughput micro- and nano-patterned structures for applications ranging from single cell assays to nanowires and nanosensors. The polymer may be a photoresist, in which case the templating method may further comprise photolithography, which may be used to form one or more features of the template, in a hybrid fabrication approach.

The method may further comprise any one or more of the steps, or the apparatus may comprise any one or more of the features, in any combination, of preferred embodiments of the invention, which will now be described by way of example only with reference to the accompanying figures.

Brief Description of the Drawings

Figure 1 is a schematic diagram of nanolithography system according to an embodiment of the invention;

Figures 2a to 2d show steps in a method of forming a nanopatterned device using the system of Figure 1;

Figures 3a to 3d show the nanopatterned device at each step of the method of Figure 2;

Figures 4a to 4f are LFM images of solvent assisted lithography on a polymer coated substrate;

Figure 5 is a graph showing the linear dependence of lithography line width on the square root of write time in the system of Figure 1;

Figures 6a to 6d are images of metal lines and dots obtained after metallization of a lithography pattern and removal of the polymer template;

Figure 7 is an image of a series of dots formed by solvent assisted lithography using an NLP 2000 lithography system; and

Figure 8 is a line analysis showing how the surface height of the sample of Figure 7 varies along a set of lines across its surface.

Description of Embodiments of the Invention

Referring to Figure 1, a nanolithography system according to an embodiment of the invention comprises a support table 10 on which a sample 12 can be supported, and a nib or tip 14, which is supported on a cantilever support arm 16. A drive mechanism 18 is provided to move the table 10 in a horizontal plane, so as to move the sample 12 in a horizontal plane relative to the tip 14, and a write control mechanism 20 is arranged to move the cantilever arm 16 vertically so as to move the tip 14 vertically into and out of contact with the sample 12. A controller 22 is arranged to control operation of the drive mechanism 18 and the write control mechanism 20 so as to bring the tip 14 into contact with the sample, move the table 10 to move the tip 14 across the sample if required, and then lift the tip 14 away from the sample. The controller 22 is programmable using appropriate software so that a pattern can be programmed into it, and after the tip has been dipped into an appropriate material, the system will then apply the material to the sample, and remove areas of polymer material from the sample, in the desired pattern.

Referring to Figure 2a and Figure 3a, the sample 12 comprises a substrate material 30 with a layer of polymeric material 32 coated onto one surface of the substrate. The material carried on the tip 14 is a solvent, which is selected so that it will dissolve or otherwise soften the polymeric material to which it is applied.

Referring to Figure 2b and Figure 3b, the system is then controlled by the controller 22 to apply the solvent from the tip 14 to the polymeric material 32 in a pattern, which is programmed into the controller. In the example shown the pattern comprises an array of dots, but typically it may comprise a combination of lines and dots. As it is applied, the solvent dissolves the polymeric material 32 into which it comes into contact, softening the polymeric material. The softened polymeric material is then removed to leave openings 34 in the polymeric material. In this embodiment the polymeric material is removed using the tip 14. This may be done simultaneously with the application of the solvent in a single 'writing' step, or it may be done subsequently in a second 'writing' step, in which the tip is moved in the same pattern as in the first writing step. Indeed, the writing step may be repeated several times, with the tip being arranged to apply solvent and remove the dissolved

or softened polymer in each writing step. This exposes areas 36 of the surface of the substrate 30 in the same writing pattern as the solvent was applied to the polymer, with the surrounding areas of the substrate still covered by the layer of polymeric material.

Where a dot is formed the tip is brought into contact with the polymer layer 32, then moved down through the polymer layer to the substrate 30, and then lifted off again without being moved across the sample. Where a line is formed the tip 14 is brought into contact with the surface of the polymeric material and then down through the polymer layer 32 to the substrate 30, then moved across the sample. As it moves across the sample solvent on the tip 14 softens the polymeric material 32, and the tip physically pushes the softened material away from its path. At the end of the line the tip is lifted off again. For a dot, the amount of solvent deposited, and hence the size of the dot formed, will depend on the 'dwell' time for which the tip 14 is held in contact with the surface. For a line, the width of the line will depend on the write speed, i.e. the speed at which the tip is moved across the sample.

Where a series of writing steps are performed on the same sample, each of them may follow the same writing pattern and therefore may include the same movements of the tip relative to the sample, so the tip re-traces the same movements in the same positions on the sample in each step.

Referring to Figure 2c and Figure 3c, the polymeric material then forms a template, and metal 38 is applied to the exposed areas 36 of the surface of the substrate 30, for example by deposition, electroplating or introduction of nanoparticle solutions by pipette followed by sintering.

Finally, referring to Figures 2d and 3d, the template of polymeric material 32 is removed leaving the metal micro- or nano-structure 38 on the surface of the substrate 30.

Rather than metal being applied to the exposed substrate, in some embodiments an etching material, such as an acid, is applied to the areas of the substrate exposed by the template. For example a silicon or silicon dioxide surface can be etched with a combination of nitric acid or hydrofluoric acid.

Examples

In one example, the polymer, commercially available as image reversal photoresist (AZ5214E) (Clariant GmbH, Germany), was thinned with Methoxypropyl Acetate (Microchemicals GmbH) in the ratio (1:3) and spun for 1 minute on gold/silicon substrate, followed by a soft bake at 90°C. The thickness of the polymer layers was measured using a Dek Tak profilometer to be 52 nm. As the 'ink' or solvent, toluene was used.

Use of a current sensing atomic force microscope (CSAFM) using toluene is known. In the known methods, toluene was used to apply voltages to electrochemically remove a layer of self assembled molecules (Octadecanethiol (ODT) in that case) mainly because it is a non-polar solvent with a low dielectric constant. In the present example toluene was used because of its low volatility (boiling point 110 °C) and ability to dissolve the photoresist polymer.

The DPN work was done using the NSCRIPTOR™ DPN System (NanoInk Inc., Skokie, IL, USA). The DPN probes used were single pen, Type A (Model: PEN-0011-04). The solvent ink was loaded onto the tip using a double dip coating method which involved 20 seconds dipping followed by a 10 seconds wait and a final 20 seconds dipping. During the dip pen lithography writing scans temperature was set at 18 °C and humidity was set at 45%. Temperatures of no more than 20 °C, and preferably at least 15 °C, and humidity of at least 30%, preferably at least 45% have been found to be optimal as they reduce the rate of evaporation of the toluene.

Different patterns such as lines and dots were written using the InkCAD software (version 3.4). Control writing scans were done by using the un-inked tips under similar conditions. Lines of 8 microns length were designed and written at write speeds of 0.05, 0.10, 0.20, 0.40 and 0.50 µm/s. The scans were repeated on each sample to ensure removal of the photoresist polymer from the pattern area was as complete as possible. For the dots dwell times were 2, 4, 8, 16 and 32 seconds respectively. All image files were processed using Nanorule+ (version 2.5.05) and ImageJ software. Figures 4a to 4f show AFM images after writing with a tip dipped in toluene. The images show residual photoresist, which was obtained during the reading scan (10 µm/s, 6 Hz). Figures 4a and 4b show forward and reverse LFM images of solvent assisted lithography of the London Underground map on a polymer-coated substrate. Figures 4c and 4d are forward and reverse LFM images of solvent assisted lithography of lines on a polymer-coated substrate. Figures 4e and

4f are forward LFM images of solvent assisted lithography of lines and dots on a polymer-coated substrate.

For the lines the width, in the direction transverse to the direction of movement of the tip relative to the polymer, and depth, of the lines decrease with increased writing speed. As seen from Figure 5, there is an inverse linear dependence of line width on the square root of writing speed. For a fixed line length this means there is a linear relationship between line width and the square root of the write time ($t^{1/2}$).

The nanotemplates were tested either by etching the exposed gold layer using a gold etch solution (HNO_3 : HCl) (1:3) or by controlled e-beam evaporation of Ti/Au (20 nm/20 nm). The nanostructures were imaged using a high-resolution microscope (1000X), Atomic Force Microscopy (AFM) (Veeco Di systems) or Scanning Electron Microscopy (SEM) (JEOL JSM840A) is represented by Figure 6. The polymer was seen to be stable and withstood conditions that facilitated etching of the exposed gold by aqua regia. Figure 6a is a SEM image of metal lines obtained after metallization by e-beam evaporation of a Ti/Au layer. Figure 6b is an optical microscope image showing dots, and Figures 6c and 6d are LFM images showing lines, after etching the gold layer with aqua regia.

DPN process usually includes a sequence of steps involving molecular deposition of the ink from the AFM tip to the substrate followed by lateral diffusion, substrate binding and final reorganization of the ink. In this case the Toluene solvent diffuses laterally thus swelling and dissolving the coated polymer layer; in the second writing step, or in some cases the first writing step, or in each of a series of writing steps, the tip removes the polymer giving the nanotemplates. The method offers good control of the size of the nanostructures. Compared to direct writing with high MP solvents we observed a RSD of >10%.

The straight line relationship between line width and writing time shows a clear relation between the writing speed and line width, and so the nano-erasing protocols described above can be followed to create nanotemplates which can then be used to obtain nanostructures without the need to protect the substrate surface from non-specific adsorption.

Referring to Figure 7, in a further experiment was carried out using the same conditions and materials as described above for the NSCRIPTOR™ DPN System,

but in this case using an NLP 2000 nanolithography system. Referring to Figure 8, a line analysis was carried out to test the consistency of the dots produced. As can be seen the dots are reasonably consistent and have a profile that is less than 1 μ m wide at the top and several hundred nm wide at the base.

The photoresist polymer as a coating layer and toluene as a solvent is just one example to represent an alternative route to formation of nanotemplates. Use of other coatings and solvents, and substrates other than Au and SiO_x can be used for a wide range of applications. By choosing the appropriate polymer and solvent this method will increase the applicability and versatility of DPN. The same polymer can be used with other solvents like acetone saturated with water, squalene and solvents with high boiling points (>110 °C). Other examples include lipids as a coating material and lipase enzymes as the solvent. Parylene can be used as a coating material and selectively removed using a combination of tetrahydrofuran, dichloromethane, and toluene solvents and after the nanostructures are obtained parylene can be removed by oxygen plasma or reactive ion etching (RIE). PMMA can also be used as the polymeric material and oxygen plasma and RIE can also be used to strip PMMA layer from the substrate. Similarly, the epoxy SU8 polymer can be selectively removed using acetone or acetone saturated with water.

Claims

1. A method of fabricating a template for a structure, the method comprising providing a substrate, providing a polymer layer on the substrate, applying a solvent to parts of the polymer layer using a tip so as to dissolve parts of the polymer layer, and removing the dissolved parts of the polymer layer to form the template.
2. A method according to claim 1 wherein the solvent is removed after it has dissolved said parts of the polymer layer.
3. A method according to claim 2 wherein said parts of the polymer layer are removed with the solvent.
4. A method according to any foregoing claim wherein said parts of the polymer layer are removed using the tip.
5. A method according to any foregoing claim wherein the tip is moved over the polymer to apply the solvent to an area of the polymer for removal.
6. A method according to claim 5 wherein the speed of movement is varied during the fabrication of a template so that the template exposes areas on the substrate of different widths.
7. A method of forming a nanostructure comprising fabricating a template according to the method of any foregoing claim and then applying material to areas of the substrate exposed by the template.
8. A method according to claim 7 wherein the material is selected from the group consisting of a metal, an etching material, and a liquid.
9. A method according to any foregoing claim wherein the structure is a nanostructure or a microstructure.
10. Apparatus for fabricating a template for a structure, the apparatus comprising support means for supporting a substrate having a polymer layer thereon, a tip carrying a solvent, control means arranged to control relative movement of the tip

over the polymer layer to apply the solvent to parts of the polymer layer thereby to dissolve parts of the polymer layer.

11. Apparatus according to claim 10 wherein the control means is arranged to control the speed of movement of the tip relative to the polymer layer thereby to control the width of one of the parts of the polymer layer that is dissolved.

12. Apparatus according to claim 10 or claim 11 further comprising support means for the tip, and wherein the control means is arranged to control a dwell time for which the tip is in contact with the polymer layer thereby to control the size of a part of the polymer layer that is dissolved.

13. A method of fabricating a template substantially as described herein with reference to any one or more of the accompanying drawings.

14. Apparatus for fabricating a template substantially as described herein with reference to any one or more of the accompanying drawings.

Fig. 2

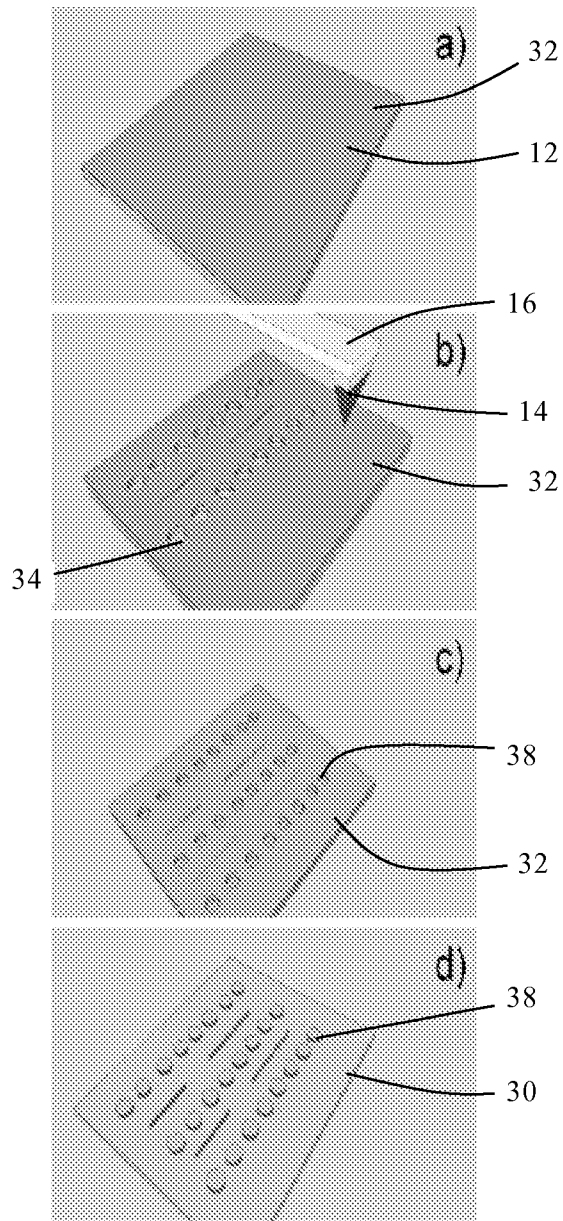


Fig. 3

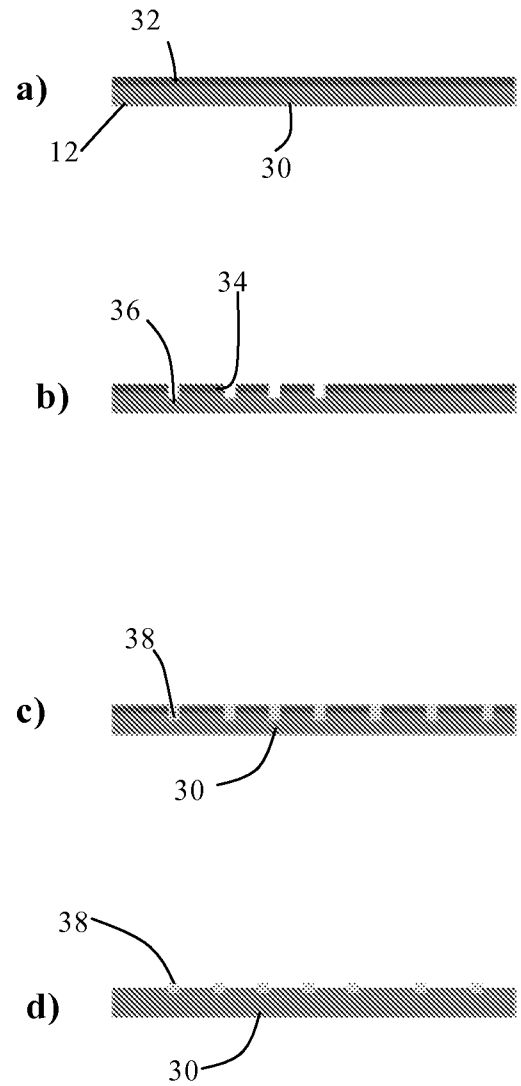


Fig. 1

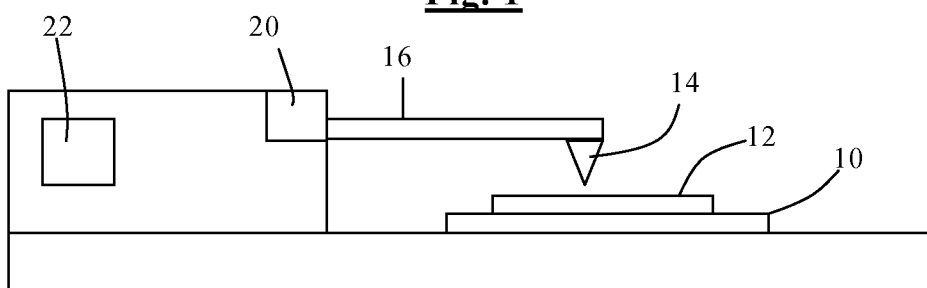
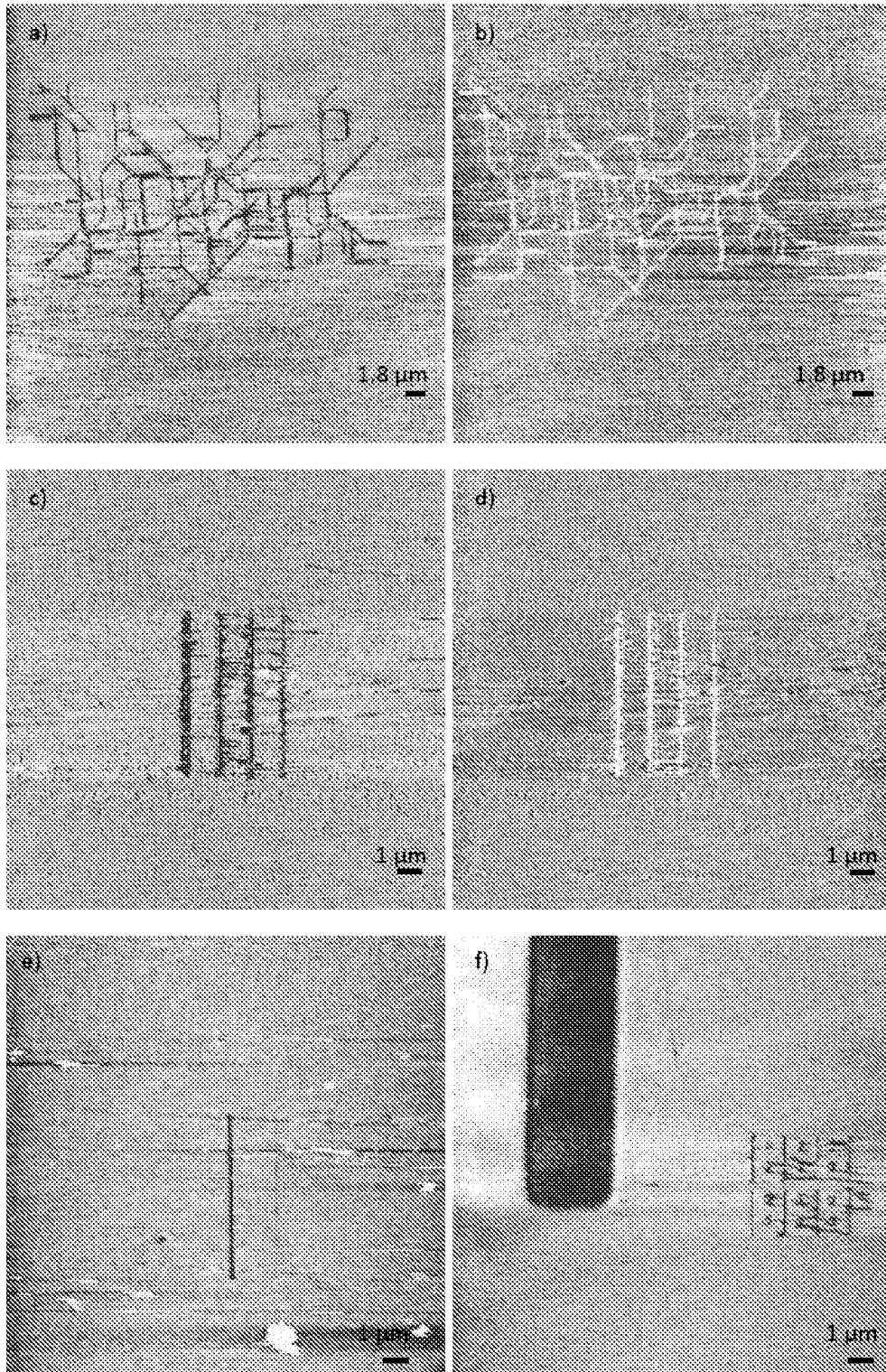


Fig. 4

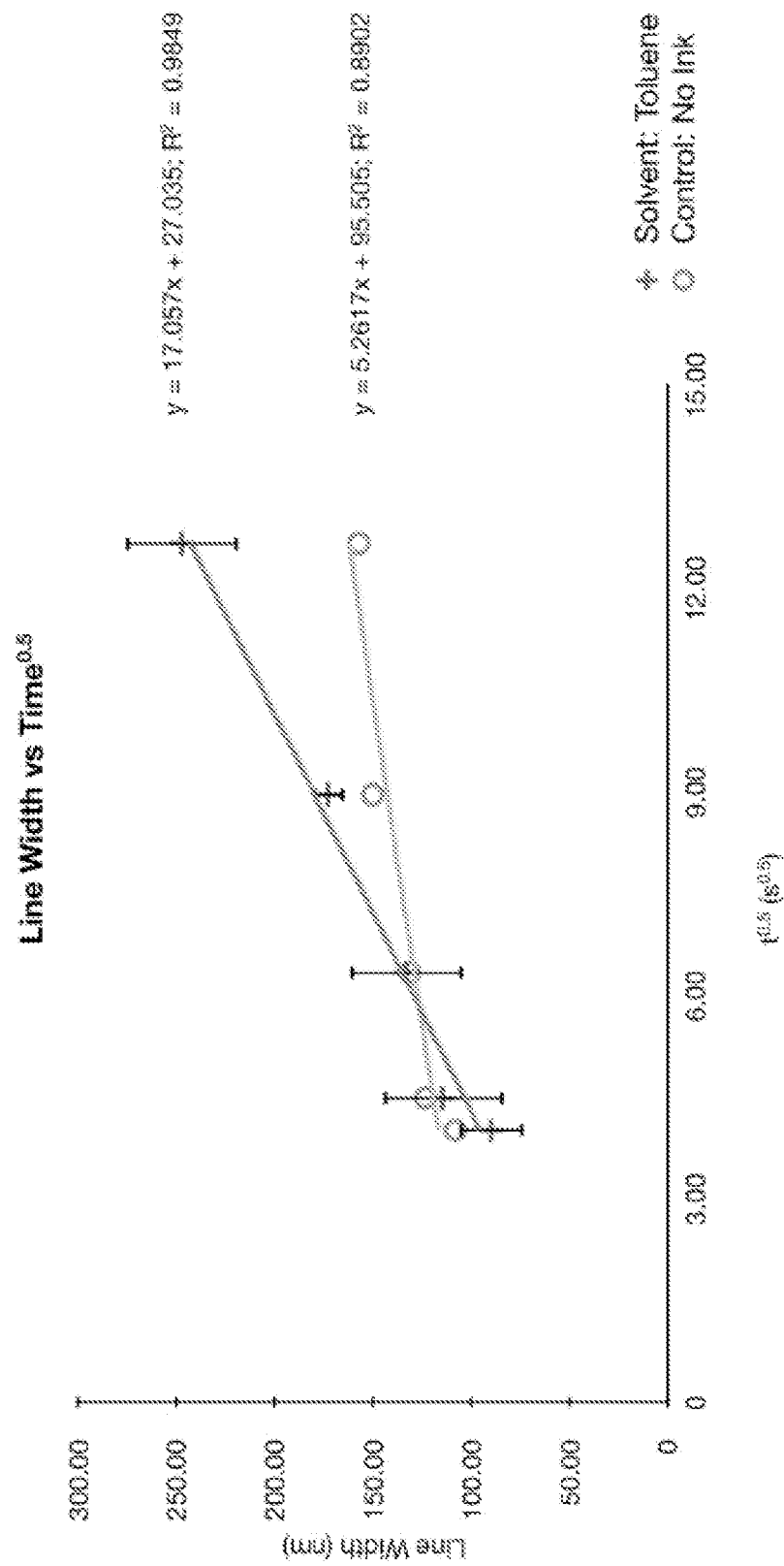


Fig. 5

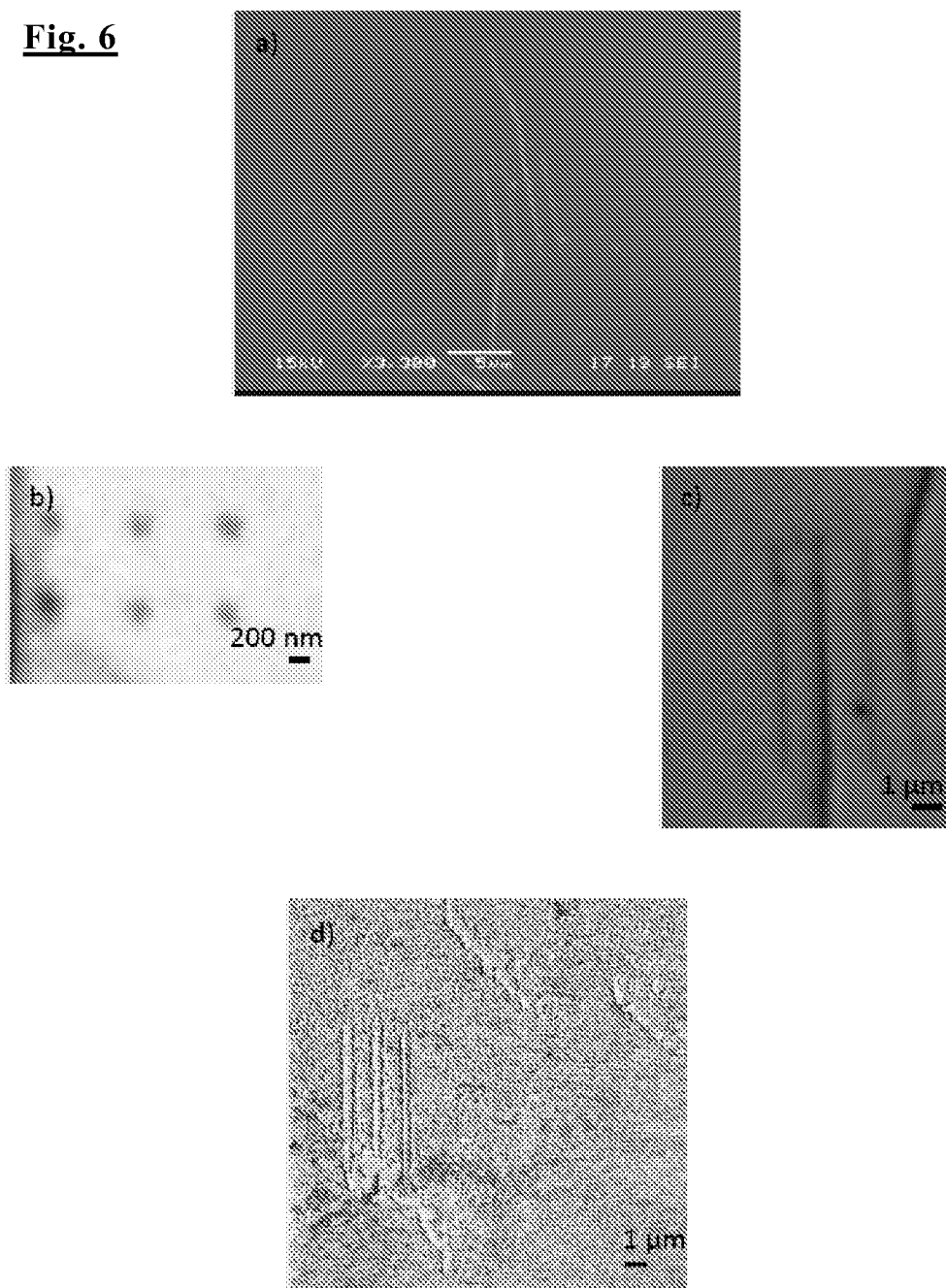
Fig. 6

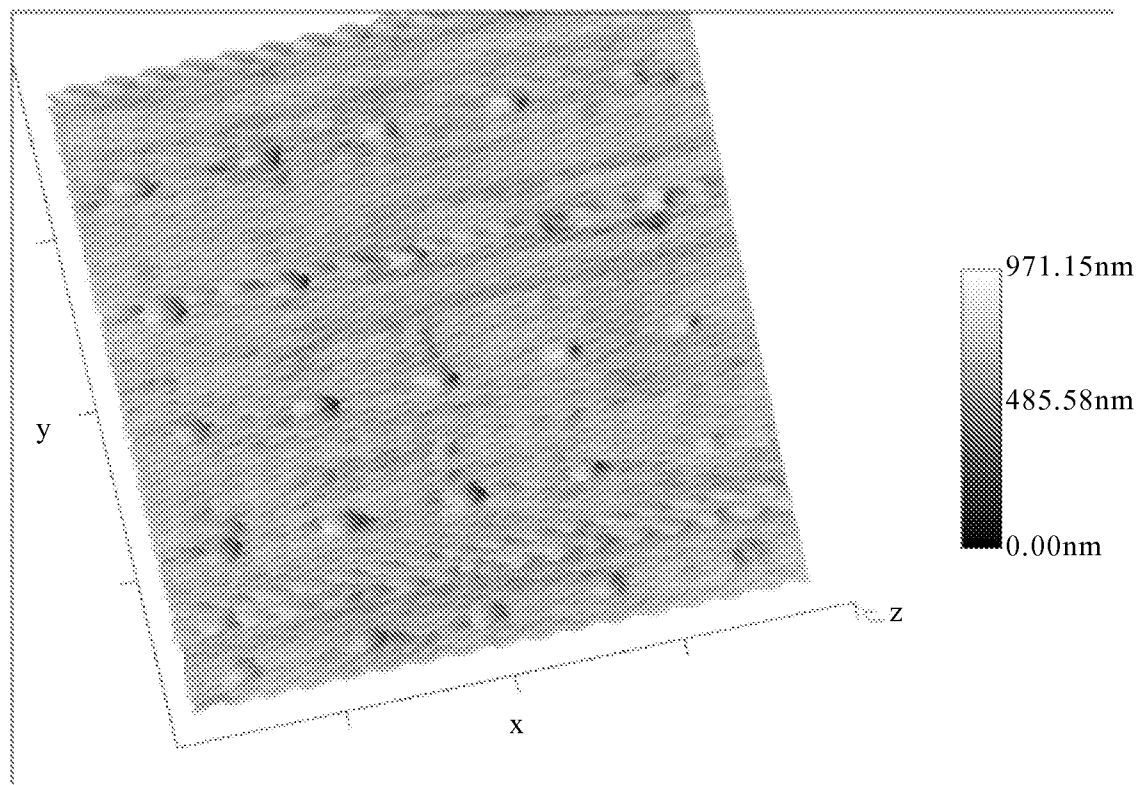
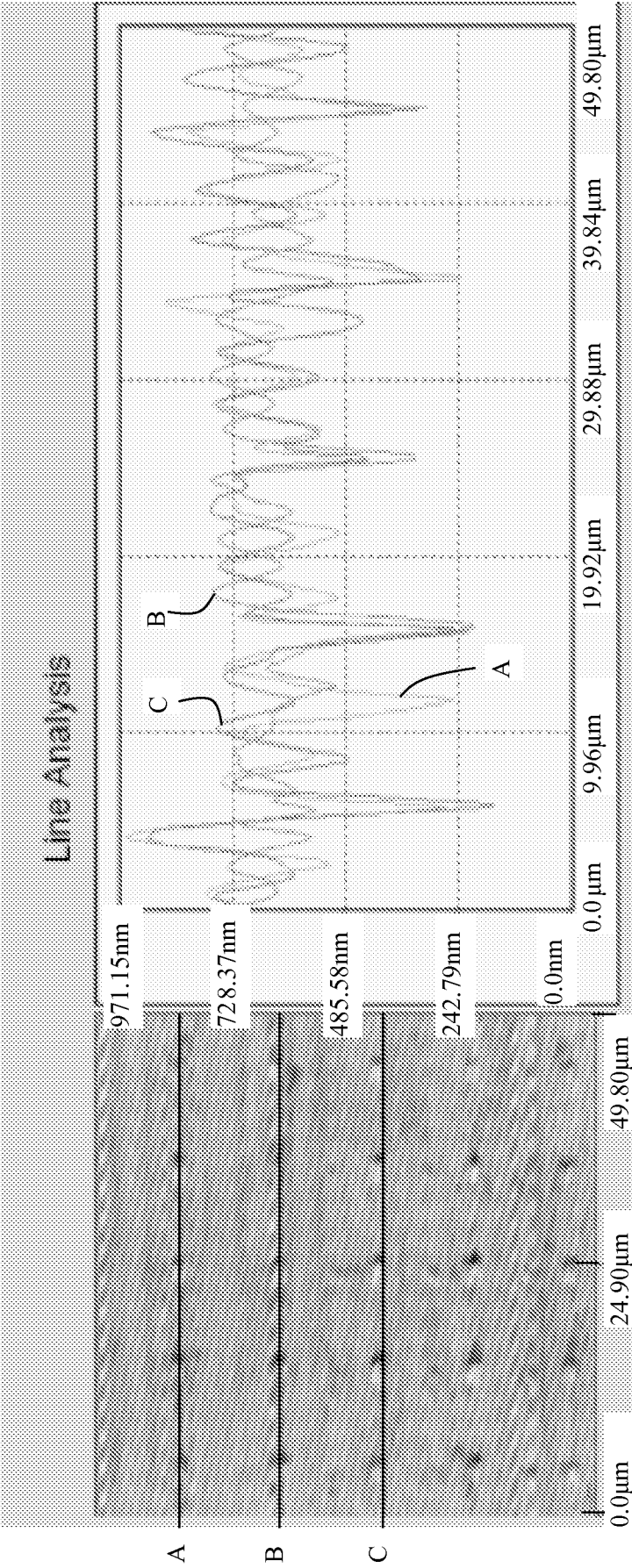
Fig. 7

Fig. 8



INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2012/051091

A. CLASSIFICATION OF SUBJECT MATTER
INV. G03F7/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
G03F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EP0-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A	US 2004/026007 A1 (HUBERT BRIAN [US] ET AL) 12 February 2004 (2004-02-12) abstract; claims 1,3,30; figures 1,3a-3c,4 paragraph [0064] - paragraph [0074] ----- -/-	1-14

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Date of the actual completion of the international search

19 September 2012

Date of mailing of the international search report

26/09/2012

Name and mailing address of the ISA/

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Paisdor, Bernd

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2012/051091

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2010/159229 A1 (SHIN CHAE-HO [KR] ET AL) 24 June 2010 (2010-06-24) abstract; figures 1,2,5 paragraph [0025] - paragraph [0026] paragraph [0028] - paragraph [0030] paragraph [0035] - paragraph [0042] paragraph [0046] - paragraph [0054] -----	1-14
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A	LING HUANG ET AL: "Matrix-Assisted Dip-Pen Nanolithography and Polymer Pen Lithography", SMALL, vol. 6, no. 10, 2 November 2009 (2009-11-02), pages 1077-1081, XP55035304, ISSN: 1613-6810, DOI: 10.1002/smll.200901198 cited in the application the whole document -----	1-14

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2012/051091

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