Direct Observation of Nanofabrication using the Optical Microscope combined with Nanopipette/QTF-AFM System

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ABSTRACT

We demonstrated direct observation of nanofabrication using the optical microscope (OM) combined with nanopipette/QTF-AFM (Quartz Tuning Fork-Atomic Force Microscopy) system. Various solutions are ejected onto the substrate through the nano-/micro-aperture of the pulled pipette, and the nano-/microscale objects were formed in on-demand spot of the surface with the patterning system. Lithography with various materials can be realized with any liquid solution and sample, such as nanoparticles, PDMS, nanowires by monitoring the fluid phenomena on the substrate with the home-made OM. After forming of capillary condensation between apex of the pipette tip and the surface, the electric field is applied to extract out the inside liquid to the substrate and the nano-microscale objects are fabricated. The nanoscale patterning size can be controlled by the aperture diameters of the pulled pipette.

Keywords: Nanopipette/QTF-AFM, Nano-Patterning, liquid delivery, PDMS

1 INTRODUCTION

Nanofabrication is highlighted as an important research field in nano-biomedical science and engineering which has a potential in application of human life, and also offers a breakthrough of the limitation of the improvement of technology and science [1-2]. There have been several methods to realization of the fabrication, such as photolithography, E-beam lithography [3], physical/chemical vapor deposition (PVD/CVD). In particular, the scanning probe microscope-based nanofabrication gives a nanoscaled critical resolution of the fabricated objects [4]. However, the output results can be confirmed by a scanning electron microscope (SEM), AFM, and so on, after fabrication process is done in the experiment. In this work, we demonstrate the in situ direct observation of the nanofabrication process using the OM combined with the nanopipette/QTF-AFM system [5,6] which define the accurate position of substrate and nanoscale phenomena. Nanolithography and liquid fluids through the nano-aperture of the pulled pipette facilitated on the QTF-AFM sensor head were presented with in situ capturing the OM images. The liquid solution filled nanopipette is glued on the edge of one prong of QTF with commercial epoxy as a tip of force sensor, and approaches closely on the substrate within 10 nm to form a nanoscale object by ejection of the solution while the extrusion phenomena were in situ observed by OM. In addition, the PDMS is directly patterned, and aggregated nanowires were fabricated as a connector of between two electrodes. With this technique, one can define the exact situation of nanofabrication process, additionally any liquid solution can be used to fabricate nanoscale object on the desired region. The motion of the system can be interpreted by the electrical signal of the QTF sensor as a viscoelasticity [7,8].

2 EXPERIMENT SETUP

Nanofabrication was performed with various solutions of material, such as nanoparticles, NaCl, dye molecules using in situ observation of the phenomena of small systems from the optical microscope facilitated under the nanopipette/QTF-AFM.

2.1 OM combined with Nanopipette/QTF-AFM system

Fabrication of the various aperture diameters of the pulled pipette were performed by mechanical puller (P-2000, Sutter Inst.). Filling of the liquid solutions into the nanopipette is complete by capillary filament which is inserted inner side wall of the pipette. Figure 1(a) shows the schematic of the experiment. The liquid filled pipette is attached one prong of the QTF sensor (< 1 nN) for sustaining the narrow distance between the tip and the substrate within 10 nm. The experimental procedure is consisted of three steps: approaching, applying electric field, and liquid election. After the tip approach the surface, the electric field is applied to eject the liquid solution from the nano-aperture of the pipette. The current signal is measured by using *IV*-converter (10^7 gain). While the process is continued, the phenomena on the substrate are in situ captured by OM located under the QTF-AFM system. The substrate was coated by sputtering with Au on the glass substrate with 20 nm thickness which is thin enough to observe the fabrication and fluid phenomena under skin depth. Figure 1(b) shows real picture of the proposed nanopipette/QTF-AFM system. The 3 μm grating was captured by OM to give a information of standard length for the experiment results.

2.2 Interpretation of the fabricated nanomaterial with viscoelasticity

We employed the non-contact, small-modulation (< 1 nm) shear-mode QTF-AFM system, because it easily detect only shear motion force, which can be formed by confined condensed nano-scaled water meniscus between apex of nanopipette and the surface [9]. The QTF can be used as a high sensitive force sensor and as a simple harmonic oscillator. The resonance frequency of used quartz tuning fork was about 32,768Hz, and the oscillation amplitude was about 0.5 nm. As this tip approach to the sample, the resonance frequency of QTF changed due to the perturbation of nucleation of water meniscus. With the equation of simple harmonic motion, the interacted force can be interpreted by effective elasticity (k_{int}) and viscosity (b_{int}) [10]. With this theoretical tool, we can study about mechanical properties of the fabricated objects.

2.3 Experiment conditions

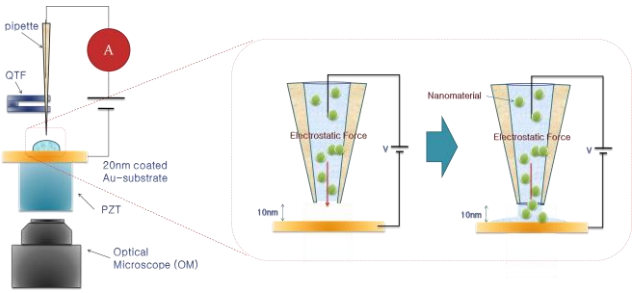
Figure 1(c) shows real picture of the proposed nanofabrication system. To form a well-shaped nanoscale objects which result from the stable confined nanoscale water meniscus, temperature stable/humidity control chamber, and the anti-vibration system (Bench-top / Sponge) are needed. The humidity variation leads to the change of the resonance frequency of QTF and the temperature variation can affect the whole system due to thermal drift. To maintain the temperature and humidity within the required control range, a double chamber setup was employed by inserting an acryl chamber inside the metal chamber for shielding against electromagnetic noise. In particular humidity is more important. At low humidity condition / small aperture diameter of pipette, the patterning size can be small. On the contrary, at high humidity condition / big aperture diameter of pipette, the patterning size can be large. Approach speed, voltage level also influence to the size of patterning. The commercial piezoelectric transducer (PZT) is facilitated, which has a capability of 50 μm (x-direction), 50 μm (y-direction), and 50 μm (z-direction) for movement of the pipette tip.

3 EXPERIMENT RESULTS

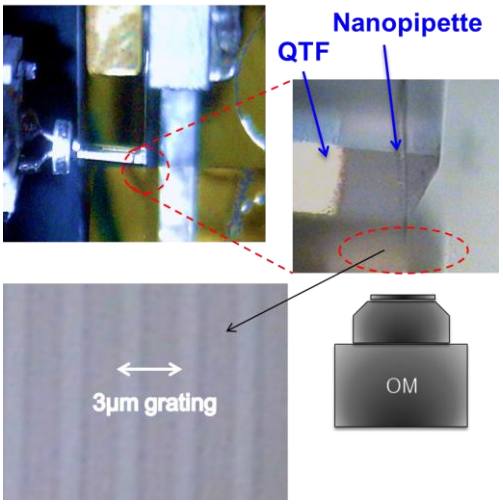
The electric field induced nano-/microscale liquid ejection, transport of the materials, and formation a certain nano-/microscale shape on the substrate are investigated. Any kind of nano-materials can be adjusted in this system.

3.1 Liquid solution delivery and Nano-patterning

Figure 2(a) shows the approach image of the liquid solution filled pulled pipette, which is captured by OM. When the tip is approach the surface, the signal is suddenly changed due to the formation of the confined nanoscale



(a)



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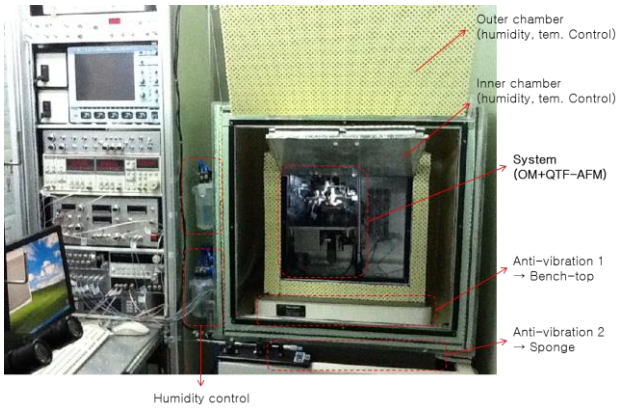


(c)

Figure 1: (a) Diagram and (b) real images of the proposed nanofabrication system. The solution filled pipette was attached on the one side of QTF for precise distance control between the tip and surface for escaping the percussion of the tip. The 3 μm grating captured by OM gives a information of standard lenth for the experiment results. (c) Acryl inside metal chamber was employed to maintain the temperature and humidity, and anti-vibration systems are facilitated.

water meniscus formed between the apex of the tip and the substrate. Figure 2(b) shows the time evolving oscilloscope signals of the QTF sensor and the electrical current. Sudden changed of the signals informs tip location within 10 nm distance and the ejection of the inside liquid of the pipette. After the liquid solution is ejected, the tip is retracted immediately to the opposite direction to form a nano-/microscale objects. Figure 2(c) shows liquid solution delivery phenomena (i) and the fabricated nano-patternings (ii). After the inside liquid is ejected onto the substrate, the fluids are continue to spread on the surface by electro-osmosis effect by application of the electric field in the case of the Au electrode coated 'on' the surface. By calculation of the spread area and speed, the flow rate through the nano-/microscale aperture of the pulled pipette can be derived. On the contrary of the fluids case, the ejected amount of the liquid volume can be suppressed by using the Au electrode coated under the surface, which electrostatic field is only exerted without electro-osmosis effect with simple flipping method of the Au coated substrate (ii). field in the case of the Au electrode coated 'under' the surface Fig. 3 (b) shows the results of the nano-patternings on the clean Au-coated glass substrate with Au nanoparticles liquid solution. After ejection of liquid solution on the substrate, the solution was evaporated onto the air, and nanoparticles form a nano-patternings.

3.2 Nano/microfabrications (PDMS / Nanowires)

Using this technique, any target nanomaterial can be taken. In the case of PDMS, the air pumping system with a commercial micro-injector is used to eject the inside PDMS solution onto the substrate instead of an electric field (Fig. 3(a)). The 7 μm aperture pipette was used to fabricate the microscale patternings, while the nanoscale patternings are still challenging issue due to the effectively high viscosity of the PDMS solution. The patterned lateral width can be varied by the pumping rate and writing speed. The depth information of the patternings can be derived using the calculation of the color image using the optical interference physical property of the transmission image. The 1 μm width of the patterning could be fabricated at high speed of tip movement.

The nanopipette tip filled with the nanowire solution approaches the surface, and moves lateral direction with sustaining the distance between the tip and the surface in a gap of 500 nm while the solution is ejected. Figure 3(b) shows the SEM image of the result.

4 CONCLUSION

We demonstrated direct observation of nanofabrication based on the optical microscope (OM) combined with nanopipette/QTF-AFM system. The liquid solution delivery and nano-/micropatterning were performed. Also PDMS and nanowires are shown with nano-/microscale fabrication

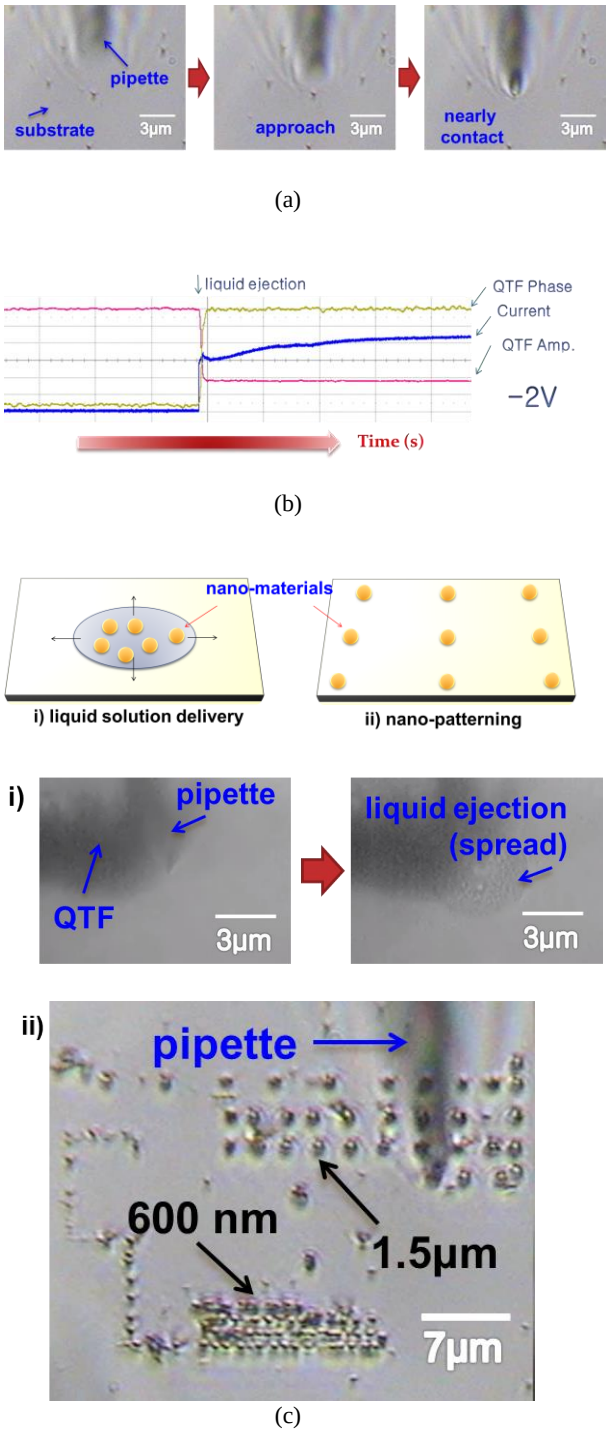
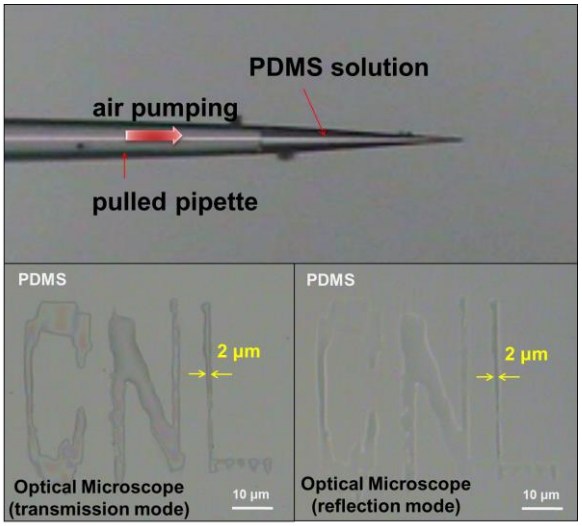
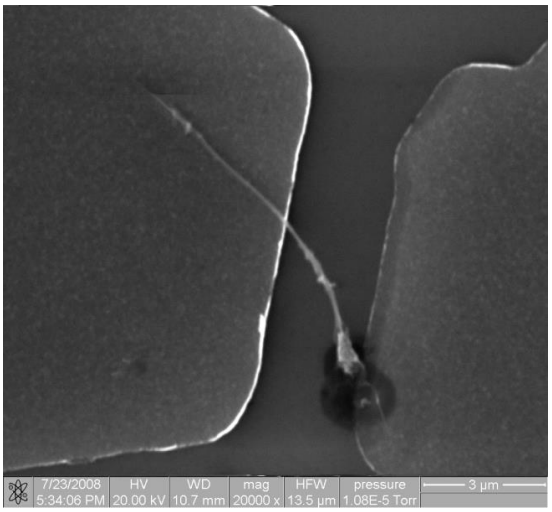


Figure 2: (a) OM image (z-axis) of the pulled pipette tip which approach the surface. (b) The signal of the QTF's amplitude and phase (mechanical) and the current (electrical). (c) In situ observing of the nano-/microscale fabricated objects. The pattern size can be controlled by varing the aperture diameters of the pulled pipette).

of the objects. We have to progress further more small and more stable work about nano-patternings with various materials, such as biomolecules, Organic/inorganic materials with this technology. We expect that this system can be adjusted and applied on a variety research field of bio-nanotechnology.



(a)



(b)

Figure 3: (a) Transmission/reflection OM images of the patterned PDMS. The PDMS solution can be ejected onto the glass surface and by pumping method with micro-injector and directly patterned. The lateral width of the pattern can be controlled by the flow rate of the pumping and the writing speed. (b) SEM image of the nanoscale fabricated object using nanowire solution, which object connects the electrical path between two electrodes.

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