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Effects of scratching parameters on fabrication of polymer nanostructures in atomic force microscope tapping mode

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Abstract

The nano scratching with an oscillating Atomic Force Microscopy (AFM) tip in tapping mode is called as the dynamic ploughing. The tip is vibrated in a high frequency and scratches the surface which is similar to the conventional vibration-assistant machining process. In the present study, the dynamic ploughing technique is utilized to scratch PolymethylMethacrylate (PMMA) polymer surfaces forming nanostructures with a commercial AFM system and two kinds of cantilevers. Effects of scratching parameters of the dynamic ploughing including scratching velocity, driving amplitude, pitch and the cantilever's elastic constant on the machined results are studied in detail. Finally nano ring structures with different radius are achieved successfully.

Keywords: Atomic force microscopy; Tapping mode; Ploughing; Nanostructures; Polymer

1. Introduction

Nanostructures are widely used in many research fields because of their unique optical, electrical or mechanical properties. Polymer nanostructures as masks or the isolated part are of importance in the nano manufacturing processes of nano imprint, wet etching and nanolithography etc. How to fabricate such kind of structure is a major concern for the manufacturing engineers. Recently, the Atomic Force Microscopy (AFM) tip-based nano machining method is a low cost and a potential way to fabricate polymer nanostructures. Up to now, there are many kinds of AFM tip-based nano machining methods which can modify the polymer surface directly, including the Dip-Pen Nanolithography [1], the thermal effect technology [2], the nano mechanical machining [3], the field emission method [4] etc..

Among these nanofabrication methods, the AFM tip-based nano mechanical machining method is investigated widely. There are two different kinds of tip's states: static and dynamic states which are in contact mode and tapping mode of AFM correspondingly. Using the tip-based static ploughing method, complex structures can be fabricated [5-6]. The tip-based dynamic ploughing method with the tip oscillating with a high frequency can also be employed to modify the polymer surfaces effectively [7-10]. This process is similar to the conventional vibration-assistant machining process. More recently, by the dynamic ploughing approach and the thermal-annealing treatment techniques, functional nano devices with a lateral feature size of 100 nm to an etching depth of 70 nm are demonstrated based on the wet etching with the scratched PolymethylMethacrylate (PMMA) nanostructures as the mask [11]. This method is also used to modify graphene on silicon dioxide substrates [12]. All these works show that it is an easy way to fabricate soft materials like polymer and soft metals which are widely used as masks in the conventional nano fabrication process. However, previous studies mainly focus on the static ploughing process and little researches are carried out on the dynamic ploughing process and the effects of scratching parameters including the driving amplitude, scratching velocity, the

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elastic constant of the cantilever and the pitch on the fabrication process on the polymer surfaces. Therefore, in the present study, effects of these parameters are studied in detail in order to achieve optimized scratching parameters and obtain perfect nano polymer structures with a high vibration tip.

2. Experimental details

The commercial AFM system (Dimension Icon) is provided by Bruker Corporation, USA. The two kinds of AFM silicon probes are TAP525 and TAP300 provided by the Budget Sensors Company, Bulgaria. The elastic constant and the resonance frequency of the TAP525 probes are 200 N/m and 450 KHz, respectively. The elastic constant and the resonance frequency of the TAP300 probes are 40 N/m and 325 KHz, respectively. All imaging and fabrication tests are carried out in AFM tapping mode. The driving amplitude of the probe is changed by the software to achieve the transition from the imaging to the fabrication process. The driving amplitude in the fabrication is about 10-50 times larger than that in the imaging process. The scratching direction is perpendicular to the long axis of the cantilever. The complex motion traces of the tip are programmed by the Nanoman module of the system. The detail probe and scratching parameters are summarized in Tab. 1. The injected molded PMMA polymer plate is used as the sample. Before tests, the sample is washed with ethanol to get rid of the large dirty particles on the surface. Then it is immersed in ethanol, and cleaned in ultrasonic bath for 3 min. After drying in air, it can be used for following tests.

Table 1. Probe and scratching parameters

AFM mode	Tapping mode		
Probe material	Silicon		
Parameters of probes	Model	Elastic constant	Resonance frequency
	TAP525	200 N/m	450 KHz
	TAP300	40 N/m	325 KHz
Sample material	Polymer PMMA		
Scratching direction	Perpendicular to the long axis of the cantilever		
Driving amplitude	The ratio of the driving amplitude in the fabrication and that in the imaging process is 4-50.		
Scratching velocity	0.1 μm/s-1000 μm/s		
Pitch	10 nm, 30 nm, 50 nm and 100 nm		

3. Effect of the scratching velocity on the dynamic ploughing process

The TAP300 probe is used in the tests. In the imaging test, the driving amplitude is 120.8 mV. The amplitude setpoint of the control system is 341.1 mV. In the nano fabrication process, the driving amplitude is 6000 mV and the amplitude setpoint is fixed at the value of 341.1 mV. Figs. 1 (a) and (b) show the AFM image and the sections of the nano grooves scratched by the dynamic ploughing with different scratching velocities. As shown in Fig. 1 (a), the scratching velocities are 0.1 μm/s, 1 μm/s, 10 μm/s, 100 μm/s and 1000 μm/s. The corresponding scratched depths of the grooves are 16.6 nm, 9.5 nm, 2.7 nm, 2.6 nm and 0 nm, respectively. As shown in Fig. 1 (b), an increase in the scratching velocity results in a shallower scratched depth. For the scratching velocity of 100 μm/s, only uneven grooves can be found. When the scratching velocity increases to 1000 μm/s, the groove is only formed at the beginning of the tip's trace. This indicates that larger scratching velocity leads to failure of the fabrication because the materials can not be removed effectively.

The TAP525 probe is used in the tests. In the imaging test, the driving amplitude is 65.0 mV. The amplitude setpoint of the control system is 377.2 mV. In the nano fabrication process, the driving amplitude is 3000 mV and the amplitude setpoint is kept at the value of 377.2 mV. As shown in Fig. 1 (c), the scratching velocities are 0.1 μm/s, 1 μm/s, 10 μm/s, 100 μm/s and 1000 μm/s. In Fig. 1 (d), when the scratching velocity is less than100 μm/s, the corresponding scratched depths of the grooves are 21.1 nm, 21.5 nm, 18.3 nm and 13.1 nm, respectively. When the scratching velocity is 1000 μm/s, the groove is only formed at the beginning of the tip's trace. Under this condition, it is the same situation with using the TAP300 probe.

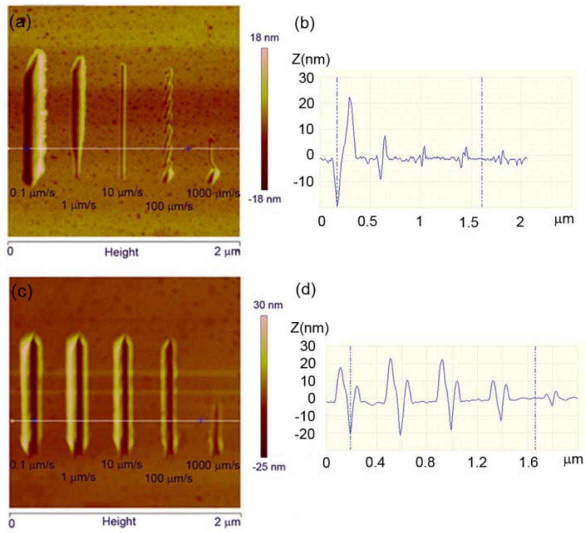

Fig.1. (a) and (b) are two dimensional AFM image and the section data of the scratched groove with the TAP300 probe; (c) and (d) are two dimensional AFM image and the section data of the scratched groove with the TAP525 probe.

As shown in both conditions, the scratched depth increases with the decrease in the scratching velocity in the dynamic ploughing process. The reasons are as follows: In AFM tapping mode, the vibrating frequency is constant, that is the tapping times per second on one location of the sample is fixed. If the scratching velocity is small, tapping times on one location become more which results in a deeper scratched depth. For the probes with different resonance frequencies, the critical scratching velocities of the stable process are different. In the present work, the critical scratching speed is 10 μm/s achieving the perfect groove for the probe of TAP300 with the resonance frequency of 352 KHz. While, when the scratching speed is 0.1-100 μm/s, the topographies of the scratched grooves are stable and perfect for the probe of TAP525 with the resonance frequency of 450 KHz. Moreover, due to the different elastic constants for both probes, the scratched depth using the TAP525 probe with the elastic constant of 200 N/m is larger than that using the TAP300 probe with the elastic constant of 40 N/m, as shown in Fig. 1 (b) and (d).

4. Effect of the driving amplitude on the fabrication of nano groove

The TAP300 probe is employed in the tests. In the measurement process, the driving amplitude is 120.8 mV and the amplitude setpoint of the control system is 341.1 mV. In the nano machining process, the scratching velocity is 0.1 μm/s. The driving amplitudes are 500 mV, 1000 mV, 2000 mV, 4000 mV and 6000 mV and the amplitude setpoint is kept constant. As shown in Fig. 2 (a) and (b), when the driving amplitudes are 500 mV, 1000 mV and 2000 mV, there are no obvious scratches on the PMMA surface and only elastically deformation happens. This is because that the normal load applied by the vibrating tip is less than the value which can deform the PMMA sample plastically. When the driving amplitude is 4000 mV and 6000 mV, the scratched depths of the grooves are 10.5 nm and 19 nm in Fig. 2 (b). This indicates that when the plastically deformations happen, an increase in the driving amplitude results in a deeper scratched depth. Moreover, using the TAP300 probe to dynamic plough the injected PMMA surface, the driving amplitude is at least 2000/120≈17 times larger in the fabrication than the measurement process to ensure the reliable fabrication process.

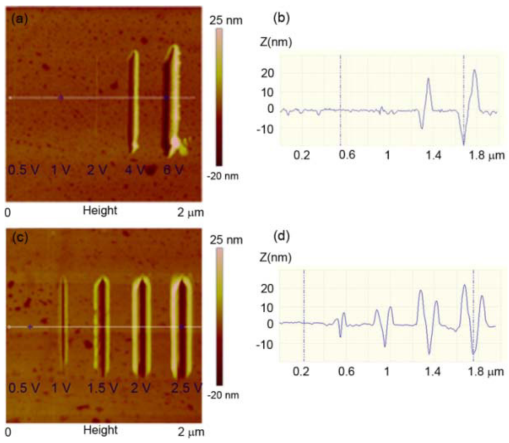

Fig.2. (a) and (b) are two dimensional AFM image and the section data of the scratched groove with the TAP300 probe; (c) and (d) are two dimensional AFM image and the section data of the scratched groove with the TAP525 probe.

Figs. 2 (c) and (d) show the AFM image and the section data of the nano grooves with the TAP525 probe. In the measurement process, the driving amplitude is 65.0 mV and the amplitude setpoint of the control system is 377.2 mV. In the nano machining process, the scratching velocity is 1 μm/s. The driving amplitudes are 500 mV, 1000 mV, 1500 mV, 2000 mV and 2500 mV and the amplitude setpoint is kept constant. As shown in Fig. 2 (c), when the driving amplitude is 500 mV, there are no remarkable marks on the sample surface. When the driving amplitude is 1000 mV, the sample is deformed plastically. Thus, using the TAP525 probe to dynamic plough the injected PMMA surface, the driving amplitude is at least 1000/65≈16 times larger in the fabrication than the measurement process to ensure the reliable fabrication process. When the driving amplitudes are 1000 mV, 1500 mV, 2000 mV and 2500 mV, the scratched depths are 7.6 nm, 12.9 nm, 17.2 nm and 17.5 nm, respectively. It is the same variation trend with that using the TAP300 probes besides that the scratched depth is deeper.

5. Effect of the driving amplitude on the fabrication of nano pockets

In the measurement process, the driving amplitude is 65.0 mV and the amplitude setpoint of the control system is 377.2 mV. In the nano machining process, the driving amplitudes are 1000 mV, 2000 mV, 3000 mV, 4000 mV, 5000 mV and 6000 mV and the amplitude setpoint is kept constant. The tip pitch is 10 nm and the scratching velocity is 1μm/s. Fig. 3 shows the AFM images of the machined results. The scratching and feeding directions are from down side to up side and from left side to right side, respectively.

As shown in Figs. 3 (a), (b), (c) and (d), when the driving amplitude is from 1000 mV to 4000 mV, the removed materials are mainly piled up on both sides of the pockets. When the driving amplitude is from 5000 mV to 6000 mV in Figs. 3 (e) and (f), the removed materials are piled up at the up side of the pockets. This result may be explained as follows: when the driving amplitude is small (1000 mV to 4000 mV), the tapping force on the PMMA sample is also small with the vibrating tip. Thus, the materials are not removed completely like belt-type chips. Simultaneously, the tip traces are overlapped each other leading to poor surface quality due to the pitch. When the driving amplitude is large (5000 mV to 6000 mV), the tapping force is large enough to remove the materials effectively each time. Therefore, the removed materials are piled up at the end of the scratch trace (the up side of the pocket in Figs. 3 (e) and (f)).

Fig. 4 is the corresponding section data of the figure in Fig. 3. As shown in Fig. 4 (a), (b) and (c), the surfaces of the bottom are all uneven. Only in Fig. 4 (d), the pocket with the depth of 20 nm is formed which are similar to the states of Figs. 4 (e) and (f) although the materials are not removed completely. The surfaces of the pockets in Figs. 4 (e) and (f) are better and the depths are 40-70 nm and 50-100 nm, respectively. Under this condition, the depth of the pocket also increases with the driving amplitude.

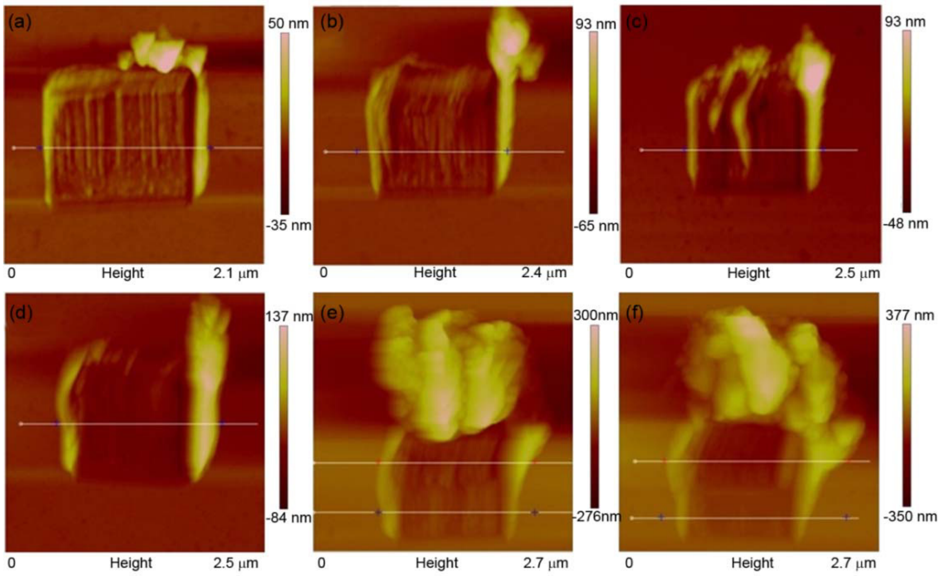

Fig. 3. Two dimensional AFM images of the pockets fabricated by the dynamic ploughing with the TAP525 probe. The driving amplitudes are (a) 1000 mV, (b) 2000 mV, (c) 3000 mV, (d) 4000 mV, (e) 5000 mV and (f) 6000 mV, respectively.

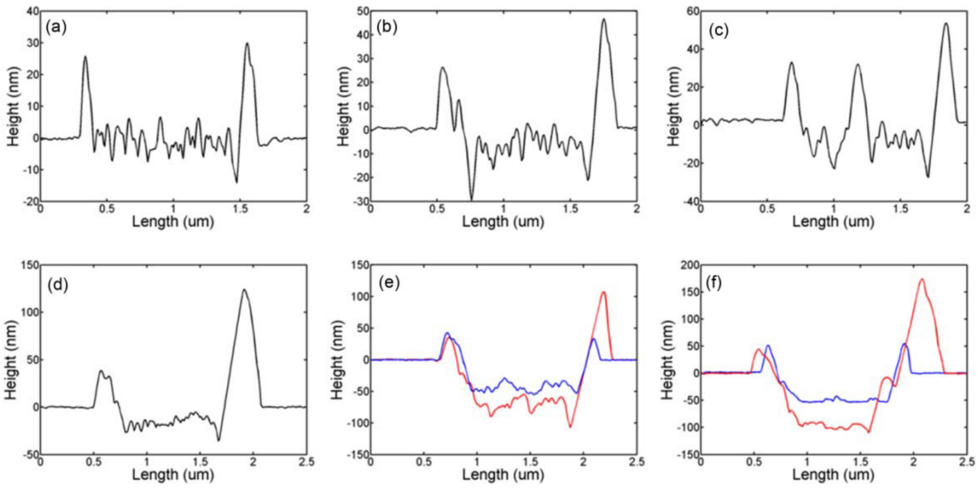

Fig. 4. The section data of the corresponding pockets in Fig. 3. The blue and red lines of (e) and (f) correspond to the lower and upper sections in Figs. 3 (e) and (f).

6. Effect of the tip pitch on the fabrication of nano pockets

The TAP525 probes are utilized to study the effect of the tip pitch. The driving amplitude is fixed to the value of 6000 mV. The amplitude setpoint of the control system is 378 mV. The scratching velocity is 1 µm/s. The pitches are 10 nm, 30 nm, 50 nm and 100 nm. The size length of the pocket is 1 µm. Figs. 5 and 6 are the AFM images and the corresponding sections, respectively. In Fig. 5 (a) and Fig. 6 (a), it can be found that when the pitch is 100 nm, no perfect pocket can be machined and projections at the bottom are apparent. When the pitch is reduced to 50 nm, perfect pockets can be achieved. With the pitch reducing, the depth of the pocket is deeper and the surface quality is better. The reason is explained as follows: A small pitch leads to high line density and more processing times at the same area. Thus the depth of the pocket is becoming deeper. Simultaneously, the materials are removed effectively and the prefect surface can also be obtained.

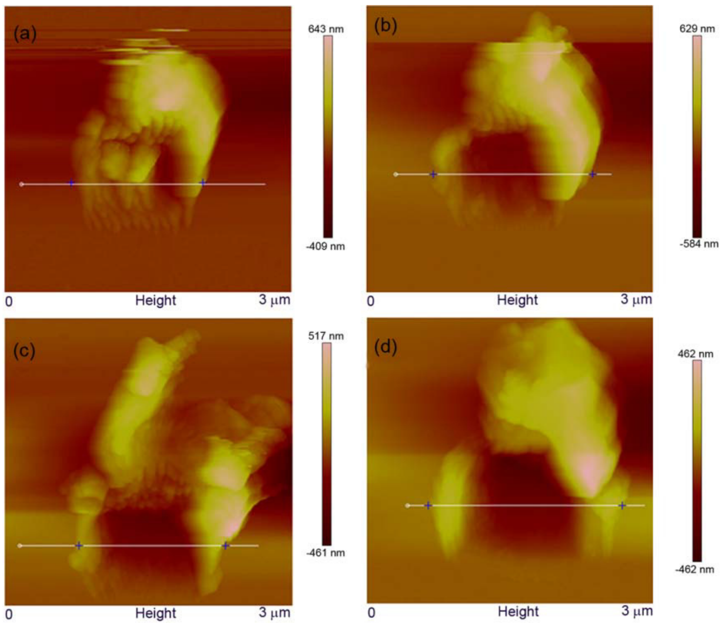

Fig. 5. AFM images of the machined pockets with different tip pitches in tapping mode. (a) 100 nm, (b) 50 nm, (c) 30 nm and (d) 10 nm.

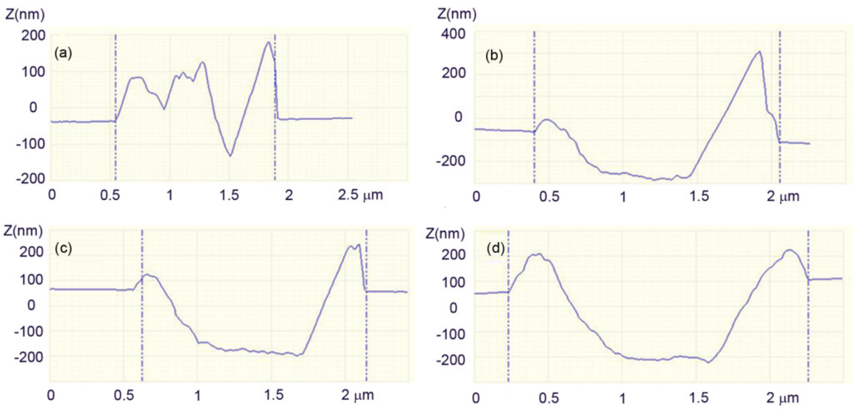

Fig. 6. Sections of the corresponding pockets in Fig. 5

In order to show the ability of the dynamic ploughing technique, a complex circular structure are fabricated on the PMMA film which is spin coated on the silicon plate. The driving amplitude is 4000 mV. The pitch is 100 nm. The radiuses of the circles are 1.5 µm, 2.5 µm and 4 µm from inner to outer sides. Previous studies showed that when the driving amplitude is not big enough, some regular projections may be formed and the pockets can be formed with larger driving amplitude [3, 10]. Fig. 7 shows the machined result. From the section, it can be found that it is also a protruding structure although the driving amplitude is not so small. It does not agree well with the previous studies. The mechanism of this phenomenon is being studied now by us.

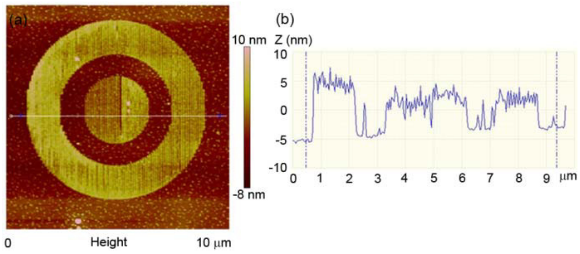

Fig. 7. Circular microstructure example on PMMA surface

7. Conclusions

The vibrating AFM tip in tapping mode is used to dynamic plough the polymer PMMA surface. The effects of the scratching velocity, the driving amplitude, the pitch and the elastic constant of the cantilever on the nano groove and the pocket are studied. Also the regular circular structures are successfully fabricated by this technique. The following conclusions are obtained.

(1) With the scratching velocity increasing, the depth of the nano groove becomes shallow. For some kind of probe, there exists a critical value which can realize the nanomachining process. In the present study, the critical velocities of the TAP300 and TAP525 probes on the polymer PMMA surface are 100 μm/s and 1000 μm/s, respectively. Simultaneously, the depth scratched by the stiffer TAP525 probe with a higher resonance frequency is larger than that scratched by the softer TAP300 probe with a lower resonance frequency.

(2) Under the same conditions, the higher driving amplitude leads to a deeper groove. The critical driving amplitudes of plastically deformation are different for different probes. The ratio of the driving amplitude in machining and that in measuring must be larger than at least 16-17 in the present study.

(3) With the same driving amplitude, a small pitch results in a deeper pocket and better surface quality. The materials can be removed effectively with a smaller pitch and larger driving amplitude. Thus a good surface and regular pocket can be achieved correspondingly.

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