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Abstract

Morpho-Mechanical Characterization and Removal Strategy of Pile-Ups in AFM-Based Nanolithography †

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Abstract: Nowadays, mechanical AFM-based nanolithography has emerged as the most promising nanolithography technique, allowing the patterning of nanostructures on polymer layers with a sub-nanometer resolution. In such a stimulating context, we developed the Pulse-AFM method to obtain continuous structures with a controlled depth profile, either constant or variable, on a polymer layer. However, those nanostructures are contoured by polymer pile-ups that limit their integration into high-tech devices. Since pile-up removal is still an open challenge, AFM force–distance curve analysis was performed to characterize the stiffness of bulges, and an effective strategy to easily remove pile-ups while preserving the shape and morphology of nanostructures was then developed.

Keywords: Pulse-Atomic Force Nanolithography; Atomic Force Microscopy; pile-up characterization; nanomechanics

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1. Introduction

In the panorama of Tip-Based Nanofabrication (TBN) approaches, mechanical lithography (m-TBN) plays a prominent role since it permits the manipulation of materials by applying a force to the sample surface with many different operation modes [1], securing versatility, flexibility, low cost, accuracy, and nanoscale resolution [2]. However, the obtained 2D, 2.5D, and 3D nanostructures are affected by edge irregularities and high pile-up materials. To overcome these limitations, we have developed an innovative AFM-based nanofabrication method based on nanoindentation, named Pulse-Atomic Force Lithography (P-AFL) [3,4], with which we can realize nanogrooves on a thin polymer layer with high accuracy and reproducibility. Unfortunately, the formation of high pile-ups is still observed at the nanogroove edges. Supposing pile-ups are composed of less stiff material, we performed a complete characterization of pile-up morpho-mechanical properties and developed an easy and very effective protocol for completely removing them.

2. Materials and Methods

All the P-AFL tests and characterizations were carried out with AFM NTEGRA (NT-MDT Spectrum Instruments, Moscow, Russia). The P-AFL process and the Force-Distance curves, for the evaluation of the stiffness (in terms of Young's modulus), were performed with DCP20 tips, while the morphology of pristine and patterned PMMA was carried out using NSG01 probes All the probes were purchased from NT-MDT (NT-MDT Spectrum Instruments, Moscow, Russia). To optimize the removal protocol of pile-ups, samples were immersed in a 1:4 (v/v) solution of Methyl Isobutyl Ketone (MIBK) and 2-propanol (IPA) for different exposure times (0 s, 10 s, 30 s, 60 s, and 90 s).

3. Discussion

With P-AFL lithography, we obtained nanochannels with a highly regular V-shaped geometry, constant depth and width equal to (20.64 ± 0.25) nm and (82.0 ± 1.2) nm, respectively, and roughness at the bottom of the channels (Rq of 0.65 nm). The nanogrooves are asymmetrically contoured by PMMA bulges, whose heights were (9.12 ± 0.65) nm and (19.62 ± 0.28) nm (Figure 1a,b). Based on the experimental findings and aiming for pile-up removal, we hypothesized that PMMA was not compact in the bulges but internally cracked. FD analysis confirmed that two different Young's moduli characterize pile-ups and pristine PMMA, i.e., (4.01 ± 0.36) GPa and (5.11 ± 0.33) GPa, respectively. Based on the nanomechanical characterization results, we thought that immersion into a standard EBL development solution could be effective in removing the pile-ups.

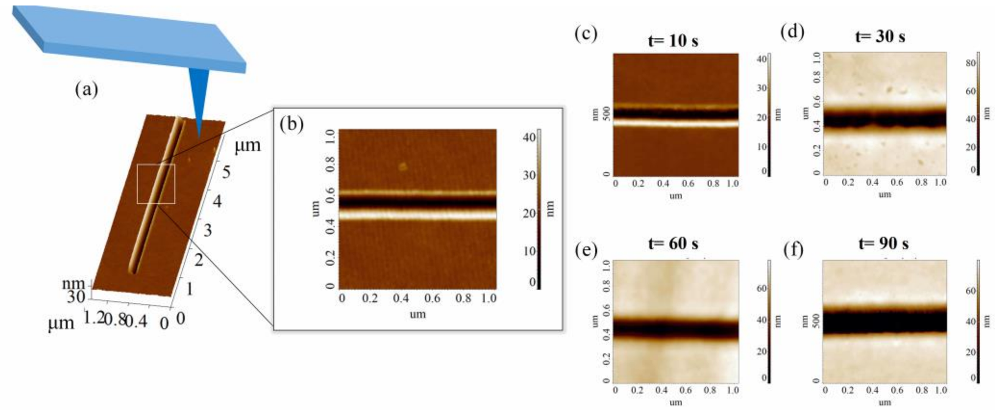

Figure 1. (**a**) P-AFL-patterned nanochannels; (**b**) details of PMMA pile-ups contouring the nanochannel; (**c**–**f**) effect of the pile-up removal for increasing times.

The use of different time points of immersion in MIBK/IPA solution had the following different effects: up to 60 s, the removal process is ineffective; at 60 s, pile-ups are completely dissolved and the shape of the grooves preserved; and for longer immersion times, increased channel width was observed (Figure 1c–f). We concluded that the P-AFL technique proved useful for the fabrication of 2D and 2.5D nanostructures. In addition, the proposed strategy of pile-up removal is very effective for etching the undesired bulges, thus strengthening the power of the proposed nanolithography technique.

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