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Full Length Article

Simple and low-cost nanofabrication process of nanoimprint templates for high-quality master gratings: Friction-induced selective etching

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ABSTRACT

Gratings are core components in biochemical analysis, ultra-precision machine tools, and astronomy. However, the high complexity and cost of conventional fabrication processes severely prevent its wide commercial application. Here, we report a simple and inexpensive fabrication process based on friction-induced selective etching. A series of crucial technological issues in the fabrication process were systematically investigated and optimized to obtain high precision and practical master gratings. Compared with the conventional surface cleaning method, the magnetic stirring-assisted KOH etching process can nondestructively and completely remove chemical sediments. Moreover, the sharp and smooth diamond tip is less sensitive to the tip profile and can easily fabricate high-quality grating structures compared with the blunt diamond tip. Particularly, the line width/density and micromorphology of grating structures can be effectively controlled by the scratching load and etching time. By integrating the mature UV nanoimprint lithography technology, the master gratings fabricated by this improved process can be excellently replicated on the polymer surface. This work presents a solid progress toward the simple and inexpensive fabrication of Si-based master gratings and also provides detailed reference for fabricating intractable blazed and circular gratings.

1. Introduction

Gratings are critical component in many fields, such as spectroscopy, laser systems, synchrotron radiation, fiber-optic telecommunications and metrological applications [1,2]. Usually, commercial gratings are replicas of master gratings [3], the fabrication methods of which can be generally classified into mechanical ruling, holographic interference, holographic ion beam etching, electron beam lithography, laser direct writing, and anisotropic etching [4–8]. Among them, mechanical ruling and holographic interference are the main fabrication approaches for actual commercial applications, whereas the others are rarely applied due to expensive equipment, low efficiency and limited fabrication area [9]. The holographic interference approach can fabricate high-linear-density gratings in a short time, whereas the as-fabricated gratings have low profile accuracy and small scale. Fortunately, the conventional mechanical ruling method covers these shortages, but it needs harsh environmental conditions and complicated fabrication processes. For instance, the room temperature must be controlled within ± 0.02 °C and vibrational frequency must be less than 3 Hz [10]. These drawbacks undoubtedly lead to the low fabrication efficiency and high cost of master gratings, thereby increasing the price of replica gratings. Hence, it is highly desired to fabricate cheap and high-quality master gratings by simple fabrication process.

Recently, friction-induced selective etching has been proved to be a robust way of fabricating various nanostructures because of its simplicity, flexibility, and controllability [11–15]. Compared with the conventional mechanical ruling method, friction-induced selective etching divides the fabrication process into two steps: mechanical scanning and chemical etching. The whole process can be proceeded under normal pressure and temperature [13]. Therefore, friction-induced selective etching avoids the complicated process and dependence of harsh environmental conditions for master gratings. However, the present results on friction-induced selective etching are mainly concentrated on the fabrication mechanism study of a single structure. In this regard, it is necessary to further improve this fabrication process to promote its industrial application in large-scale master gratings.

In this work, we presented a very simple and low-cost fabrication process based on friction-induced selective etching for Si-based master gratings. For a systematical study, a series of processing approaches was detailed investigated to control the surface roughness and profile of large-scale master gratings. To accelerate its commercial application, UV-assisted nanoimprint lithography was employed to replicate the master gratings, and it demonstrated good replication ability. This work presents a solid progress toward the practical applications of friction-

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induced nanofabrication in large-scale Si-based master gratings and replica gratings.

2. Materials and methods

2.1. Experimental details

All the grating samples were performed on p-type Si(1 0 0) wafers with a thickness of 0.5 mm (MEMC Electronic Materials, Inc., USA). Before tip scanning, the samples were dipped into 10 wt.% HF solution for 2 min to remove the superficial native oxide layer to shorten etching time [16]. Using an atomic force microscope (AFM; SPI3800N, Seiko, Tokyo, Japan), the root mean square roughness of the H-passivated Si samples was measured to be 0.08 nm over a 5 × 5 μm area. In addition, the tip used in the scanning process was a conical diamond tip with a nominal radius of 10 μm and normal spring constant of ~40 N/m. Moreover, all the AFM images were scanned in a vacuum with silicon nitride tips (MLCT, Veeco Instruments Inc., Plainview, NY, USA) with a spring constant of 0.1 N/m. During the nanoimprint lithography process, the liquid dielectric used in our experiment is an UV-curable prepolymer (NOA65, Norland Products Inc., USA). The transfer substrates are transparent slide glasses. For easy demolding, the as-fabricated grating templates were treated in a 1.0 wt% ethanol solution of heptadecafluorodecyltrimethoxysilane ($CF_3(CF_2)_7CH_2CH_2Si\text{-}(OCH_3)_3$, FAS) for 4 h and subsequently baked at 70 °C for 10 min. Moreover, the replicas of master gratings are measured by laser scanning confocal microscopy (LSCM, Olympus OLS4000) with a resolution of 0.01 μm at the Z axis and 0.12 μm at the X and Y axes. The scanning electron microscopy (SEM) images are obtained using a FEI Quanta FEG 250 SEM instrument.

2.2. Fabrication and nanoimprint process of master gratings

Fig. 1a shows the schematic illustration of the fabrication process of master gratings. First, uniformly spaced scratches were conducted on a H-passivated Si(1 0 0) substrate under low load with 100 μm/s scanning speed by using a multi-probe micro-fabrication apparatus [17], as shown in stage I. Next, the patterned sample was immerged in a mixture solution (20 wt.% KOH solution: isopropyl alcohol = 5:1) at 25 ± 1 °C for 10–20 min (stage II). Finally, the as-fabricated sample was taken out from the solution and cleaned in ethyl alcohol and deionized water for 5 min in sequence. As shown in stage III, uniformly spaced grating structure was perfectly fabricated on Si(1 0 0) substrate.

Furthermore, the as-fabricated master grating sample was replicated by UV-assisted nanoimprint lithography process, [18,19] as clearly demonstrated in Fig. 1b. First, a FAS film was deposited on the as-fabricated master gratings for easy demolding after cleaning in ethyl alcohol and deionized water for 5 min in sequence, as shown in stage IV. Then, the fluidic UV-curable prepolymer was slowly filled into the mold cavities of the dried master gratings along with the previous scanning direction. Afterward, a transparent glass substrate was coated on the prepolymer surface and cured by UV irradiation for 10 min through the glass substrate (stage V). Finally, the polymer replica grating was transferred onto the glass substrate after separating from the master grating (stage VI).

3. Results and discussion

The surface roughness and profile are the crucial properties of master gratings, which directly affect the signal-to-noise ratio and diffraction efficiency of all replica gratings. Hence, for better application of this new fabrication approach of master gratings, a series of processing methods was investigated to control the surface roughness and profile of master gratings. Moreover, to further reduce its processing cost, UV-assisted nanoimprint lithography was employed to replicate the master gratings.

3.1. Control methods of surface roughness

As shown in Fig. 2a, during KOH etching process of grating

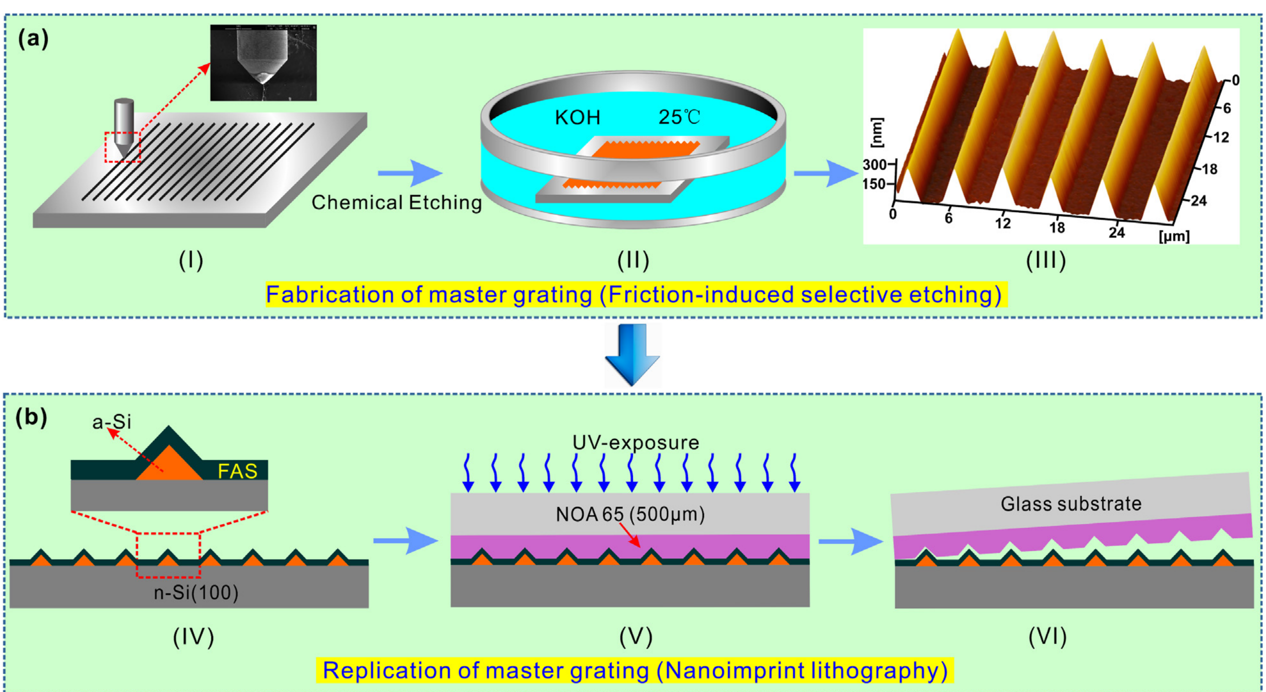

Fig. 1. Schematic diagrams (a and b) showing the fabrication and nanoimprint process of master gratings. (I) Mechanical scratching with a conical diamond tip. (II) Post-etching in KOH solution for fabricating grating structures. (III) Master grating sample fabricated by friction-induced selective etching. (IV) Depositing FAS film on the master gratings. (V) Filling the UV-curable prepolymer and cured by UV irradiation through glass substrate. (VI) Separation of the glass substrate from the master grating.

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