Microelectronic Engineering 141 (2015) 6–11

Contents lists available at ScienceDirect

Microelectronic Engineering

journal homepage: www.elsevier.com/locate/mee

Three-dimensional nanometrology of microstructures by replica molding and large-range atomic force microscopy

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ARTICLE INFO

Article history:
Received 24 October 2014
Accepted 28 November 2014
Available online 27 December 2014

Keywords:
High-aspect ratio microstructures
Atomic force microscopy
Replication molding
X-ray optics

ABSTRACT

We have used replica molding and large-range atomic force microscopy to characterize the three-dimensional shape of high aspect ratio microstructures. Casting inverted replicas of microstructures using polydimethylsiloxane (PDMS) circumvents the inability of AFM probes to measure deep and narrow cavities. We investigated cylindrical deep reactive ion etched cavities in silicon wafers and determined the radius of curvature (ROC) of the sidewalls as a function of depth. Statistical analysis verified the reliability and reproducibility of the replication procedure. The mean ROC was determined as (6.32 ± 0.06) μm, i.e., with 1% accuracy, while the ROC linearly increases by (0.52 ± 0.03) μm from the top to the bottom of the sidewalls. Nanometer sized surface defects are also well replicated. In addition, the method allows combining multiple features from differently processed wafers into a single sample, accelerating characterization in process optimization tasks. To access the sidewall shape samples needed to be cleaved. The method was applied to study X-ray refractive optics, whose performance is crucially affected by their three dimensional shapes.

1. Introduction

Techniques such as deep reactive ion etching (DRIE), SU-8 processing and micro-electro-discharge machining allow fabrication of high-aspect ratio microstructures [1–3]. The engineering tolerances of such geometries approach the nanometer range, meaning that measuring their detailed full three-dimensional (3D) topography is crucial for proper operation as well as process development, process optimization and quality control. Optical microscopes, scanning electron microscopes (SEM) and atomic force microscopes (AFM) are essential characterization tools in any microfabrication facility. However, when characterizing 3D microstructures, these instruments are often limited in terms of range, resolution and throughput [4,5]. Unless equipped with a software package for 3D reconstruction, electron microscopy is inherently 2D, while optical profilers are limited by the maximum detectable slope and have relatively poor lateral resolution. In contrast, AFM is an inherently 3D technique with nanometer resolution. Nowadays, AFMs may have large scan fields (e.g., 100 × 100 μm^2) and height ranges exceeding 10 μm, making them promising tools for tasks beyond nano-scale measurements such as more traditional profiling.

High-aspect ratio microstructures often possess geometries that cannot be directly measured by common AFMs, such as cavities, undercuts, bottoms of trenches or holes, and sloped or vertical sidewalls. Several attempts have been made to overcome these limitations: Custom-made probes enabled the characterization of sidewalls of high-aspect ratio micro-structures [6,7], but such probes are not yet commercially available. Oscillation and servo control of flared probes in both the lateral and vertical direction allowed true 3D metrology of nanostructures [8,9], but such scanning modes are not supported by conventional AFMs. Tilting the sample stage [10,11] or rotating the scanner head [12] allowed probes to scan sidewalls effectively, but required shallow structures such that the probes could approach the surface.

Clearly, the finite widths of commercially available AFM cantilevers and the limited lengths of their tips compromise the scanning of deep and shallow cavities. Protrusions, on the other hand, do not pose such difficulties. By polymer casting an inverted replica of a given specimen, indentations are turned into protrusions. Thus,

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http://dx.doi.org/10.1016/j.mee.2014.11.026
0167-9317/

replication molding for the sake of characterizing the master is an effective strategy whenever a challenging specimen is at hand [13,14]. However, to the authors' knowledge no assessment of the accuracy of a 3D replication method of microstructures has been detailed in the literature to date.

In this study, we characterized the three-dimensional shape of deep reactive ion etched cavities in silicon with diameters and depths of approximately 100 μm. An array of such cavities may be used to create a focused line beam of hard X-rays at a synchrotron radiation facility (Fig. 1a) [15,16]. To achieve an X-ray line beam with optimal focusing, the shape of the cavities must be close to ideal parabolic cylinders [17]. Any deviation from the ideal shape causes aberration and reduced performance (e.g., blurring). Most importantly, we need to optimize the uniformity of the radius of curvature of the parabolic shape along the depth while minimizing surface defects and roughness. Therefore, we developed a technique to characterize the complex shape of X-ray lenses with a large-range AFM. Since the apexes of the lenses are too narrow to be scanned with an AFM probe directly, inverted replicas of lens cross sections were cast in polydimethylsiloxane (PDMS). The quality of the replication procedure was assessed and the results are presented.

2. Methods and experimental details

2.1. Lens fabrication

The refractive X-ray optics investigated here were fabricated at DTU Danchip, the cleanroom facility at the Technical University of Denmark. The manufacture included standard UV-lithography and pattern transfer by reactive ion etching into a thermally grown silicon oxide layer, which served as a hard mask for subsequent deep reactive ion etching of the silicon (Fig. 2a–c). In addition, we performed a surface smoothening step by the thermal growth of a 1 μm thick silicon oxide layer followed by its removal in buffered hydrofluoric acid.

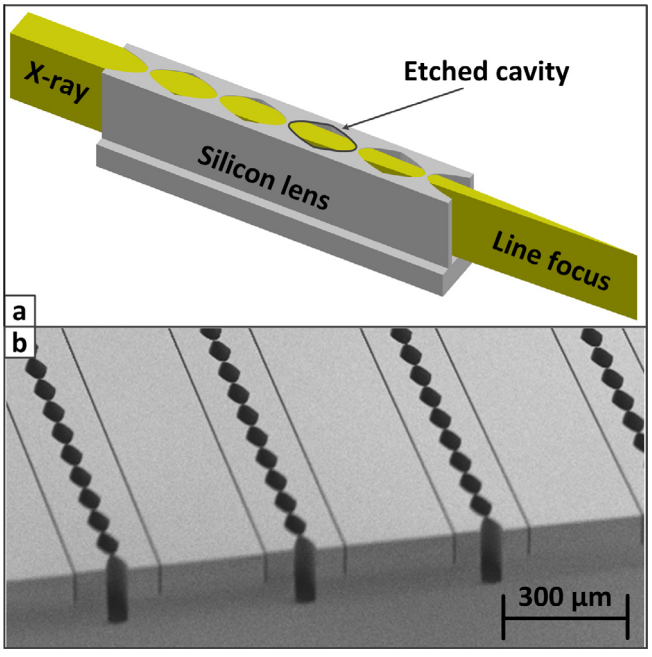

Fig. 1. Features of interest in this study. (a) Conceptual drawing of a silicon compound refractive lens focusing hard X-rays into a line. (b) Scanning electron micrograph of arrays of parabolic cavities in silicon.

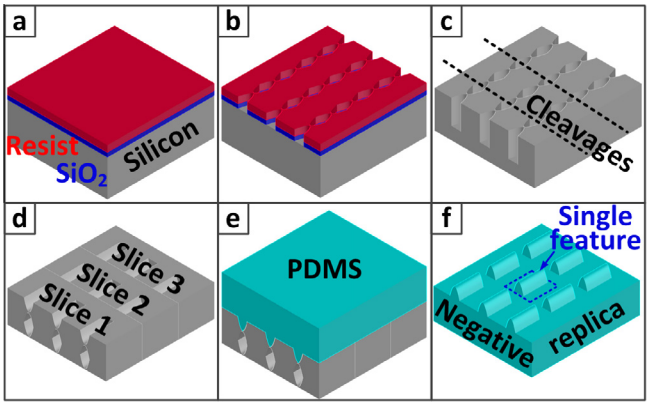

Fig. 2. A typical fabrication process for the manufacture of silicon compound refractive optics. (a) Thermal silicon oxide growth and resist spinning. (b) Lithography and pattern transfer by reactive ion etching. (c) Deep reactive ion etching of silicon using the structured silicon oxide layer as a hard mask. For characterization, the wafers were cleaved into slices of ~1 cm width. (d) Stacking of the slices and tight clamping to obtain a mold. (e) Casting the cross sections with PDMS. (f) Release of the thermally cured PDMS.

The geometry of one cavity is defined by a pair of adjacent parabolas with radii of curvature of 6 μm. The cavities have a lateral dimension of approximately 100 μm and were etched to a depth of 120 μm (Fig. 1b). Adjacent cavities are separated by 2 μm from each other. Thus, the local aspect ratio at the apex is significantly larger than 10.

2.2. Fabrication of silicon V-grooves

To confirm the AFM linearity and calibration, we fabricated reference structures comprising silicon V-grooves fabricated by anisotropic etching of (100)-oriented silicon wafers using aqueous potassium hydroxide (KOH). A silicon nitride layer deposited on a silicon wafer by LPCVD was patterned by standard UV-lithography and reactive ion etching. The patterned wafers were etched in aqueous KOH at 80 °C until the {1 1 1} crystal planes had met and essentially stopped further etching. The silicon nitride mask was subsequently removed in phosphoric acid at 180 °C, resulting in V-grooves of 160 μm lengths, 15 μm widths and 11 μm depths.

2.3. Inverted replication

To obtain inverted replicas of cross sections of etched cavities, we manually cleaved finished processed wafers perpendicular to the axis of the lens arrays into slices of ~1 cm widths (Fig. 2c–f). These slices were stacked and fixed with a purpose-made clamp. To facilitate release of the polymer cast, the surface of this unit of stacked and clamped slices was functionalized with a self-assembled monolayer of perfluorodecyltrichlorosilane (FDTS) using molecular vapor deposition (MVD). We used polydimethylsiloxane (PDMS) as a cast material. PDMS is known for its unique flow properties in its uncured state and its low elastic modulus in its cured state, and is widely used in soft-lithography of e.g., microfluidic circuits [18,19]. We combined ~50 g of monomer and hardener from our PDMS kit (Sylgard 184, Dow Corning) in a weight ratio of 10:1 and manually mixed it with a spatula for 10 min. The mass was degassed in a desiccator evacuated to 2×10^{-3} mbar until air bubbles were removed [20]. The outgassed mixture was poured into the prepared mold, and again degassed at 2×10^{-3} mbar. After 30 min of degassing, we removed the casted mold from the desiccator and cured the PDMS in an oven at 60 °C and atmospheric pressure for 6 h. The cured and cooled PDMS (room temperature) was released from the mold, ready to