



Fabrication of inverse micro/nano pyramid structures using soft UV-NIL and wet chemical methods for residual layer removal and Si-etching

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ARTICLE INFO

Article history:

Available online 14 March 2013

Keywords:

Nanoimprint

Residual layer etching with wet chemical

Micro/nano inverse pyramids

ABSTRACT

In this study we present a novel and simple fabrication method for micro- and nano-scale inverse pyramidal structures by a combination of soft UV-NIL and wet chemical etchings. The unique feature of our method is the absence of a RIE process, which is usually applied for removal of residual layer. This is achieved by modifying the imprint resist with an O₂-plasma in ash and subsequent wet etching of residual layer by diluted HF. Combined with conventional anisotropic wet etching step with KOH/IPA, our method allows cost effective fabrication of inverse pyramidal structures on Si-substrate in batch process. Such structures in nano- and micro scale are particularly suitable for light management in photovoltaic, solid state lighting and silicon based photonic.

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1. Introduction

Inverse pyramidal patterns in micro or nano scales into silicon (Si) substrates are versatile pattern geometries as they can be applied in the area of light management for light harvesting components such as high efficiency solar cell [1], for light guidance in solid state lightning [2], SERS (Surface Enhanced Raman Spectroscopy) to analyze material properties [3] or surface texturing of thin-film crystalline silicon solar cells, where inverse nano scale pyramids are introduced to enhance the light absorption allowing a further reduction of silicon material usage [4].

Until today, transfer of inverse high density pyramidal patterns in micro scale into Si substrate is commonly done by a combination of optical photolithography using selective opened photo resist layer and underlying hard mask (SiO₂ e.g.) followed by anisotropic wet chemical etching. But for pattern transfer of inverted pyramids in nano scale, feasible pattern resolution of conventional contact photolithography comes to its limit and alternative lithography methods such as electron beam lithography or laser interference lithography, which are cost or time intensive, have to be used.

Another cost effective lithography method is nanoimprint lithography (NIL). Compared to conventional photolithography, nanoimprint lithography (NIL) offers tailored pattern definition and replication of micro- and nano scale structures at low cost [5]. Soft Ultra-Violet Nanoimprint lithography (Soft UV-NIL) in particular enables further cost reduction by using a cast-molded flexible mold for patterning over large surface in a single process step [6].

One of main drawbacks of NIL, however, is the presence of residual layer underneath the defined resist patterns, which needs to be removed before any pattern transfer process. A combined NIL and photolithography using hybrid mold by quartz mold with light blocking metal layer allows patterning without residual layer [7], but this method requires complicated quartz mold fabrication and is still bounded to resolution limit of photolithography. Hence NIL enables cost effective pattern definition for wide pattern resolution from micro to nano meter scale, but the removal of the residual layer still remains as pivotal issue and imposes an additional RIE (reactive ion etching) process step [8], which is cost intensive and hinders batch process for higher throughput.

Here we present a unique method for fabrication of periodic nano- and micro scale inverse pyramidal structures on Si substrates. Patterns were defined by soft UV-NIL. After pattern definition, imprint resists were treated with O₂-Plasma in barrel resist ash in order to increase chemical resistance during the subsequent wet etching process. Unlike common RIE, resist ash enables simultaneous treatment of multiple substrates in a single process step. Removal of residual layer and a selective etching of a SiO₂ hard mask layer, as well as following pattern transfer into Si substrates were carried out using only wet chemical etching process.

2. Experimental

2.1. Fabrication of master templates and flexible molds

Soft UV-NIL uses flexible mold as replication tool, whose fabrication requires prepatterned master templates. In this study, two master templates were used for the fabrication of flexible molds

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for micro- and nano scale pattern definition. For the fabrication of master templates, hole pattern with a $2.5\ \mu\text{m}$ and $500\ \text{nm}$ pitch were defined by using an optical stepper (Canon FPA3000) and laser interference lithography (LIL) respectively. Those patterns were etched into Si using standard SF_6 RIE processes to a depth of $150\ \text{nm}$ and $120\ \text{nm}$ followed by deposition of anti-Sticking layer for better mold separation during the cast molding process.

Flexible molds, having inverse patterns to master templates, were made from the templates with cast molding process. Polydimethylsiloxane (PDMS) from Dow Corning (Sylgard 184) was used for the fabrication of the molds. The mixture consisting of two components (10:1, base material: curing agent) was poured onto the respective masters, degassed in a vacuum chamber and were thermally cured on a hot plate with a temperature of $90\ ^\circ\text{C}$ for 2 h. The flexible molds were then separated from the master templates and used as replication tool for pattern definition on the target substrates.

2.2. Process flow for the fabrication of nano- & micro scale inverted pyramids

The complete process flow following the described mold fabrication is illustrated in Fig. 1.

In detail, $10\ \text{nm}$ silicon oxide layer was deposited on Si(100) substrates acting later as hard mask for wet chemical etching. AMONIL[®] Imprint resist (AMO GmbH) was spin coated on substrates and the imprint process using flexible mold was carried out (Fig. 1a) for pattern definitions of micro and nano scale inverse pyramidal structures. The thicknesses of imprint resist layers were defined to $200\ \text{nm}$ (AMONIL[®] MMS4, 3000RPM for 30 s) and $130\ \text{nm}$ (AMONIL[®] MMS10, 2000RPM for 30 s) respectively. Imprint processes for both patterns were carried out using a soft

UV-NIL tool (EVG620). An imprint pressure of $200\ \text{mbar}$ with a UV-exposure time of $10\ \text{min}$ was applied for the imprint process.

In order to allow a removal of residual resist and a selective wet etching of SiO_2 hard mask layer, the resist was modified with O_2 -Plasma in a resist ashers (Tepla Semi 300, O_2 : $800\ \text{ml/min}$, $1000\ \text{W}$) (Fig. 1b). The effect of resist modification on wet chemical based residual layer etching by HF was analyzed and optimized treatment time was determined. Those results will be discussed in Results and Discussion.

For removal of the residual layer and selective etching of the deposited SiO_2 layer, substrates were dipped in 2.5% hydrogen fluoride (HF) for $30\ \text{s}$ (Fig. 1c).

Pattern transfer for inverted pyramid structures into silicon substrates was carried out with potassium hydroxide (KOH) using selectively etched SiO_2 layer as a hard mask (Fig. 1d). For the mixture of 30% KOH, $70\ \text{g}$ of KOH pellets were mixed with $190\ \text{ml}$ deionized (DI) water at $70\ ^\circ\text{C}$. Additional $50\ \text{ml}$ of isopropyl alcohol were added for better surface contact of etchants. Micro scale holes were etched for $60\ \text{s}$ and nano scale holes for $45\ \text{s}$ with subsequent cleaning of samples in 5% HF in order to remove resist residue and SiO_2 hard mask layer (Fig. 1e).

3. Results/discussion

3.1. Pattern definition by soft UV-NIL

The results of the nanoimprint process in comparison to the masters for micro and nano patterns are shown in Fig. 2. Patterns of the both imprinted structures correspond well, within a few nanometers, to the original master templates. Holes with a period of $2.5\ \mu\text{m}$ and $1.15\ \mu\text{m}$ diameter were defined with a pattern height of $145\ \text{nm}$. Thickness of residual layer was measured to less than $10\ \text{nm}$ (Fig. 2a and b). Patterning results of holes with $500\ \text{nm}$

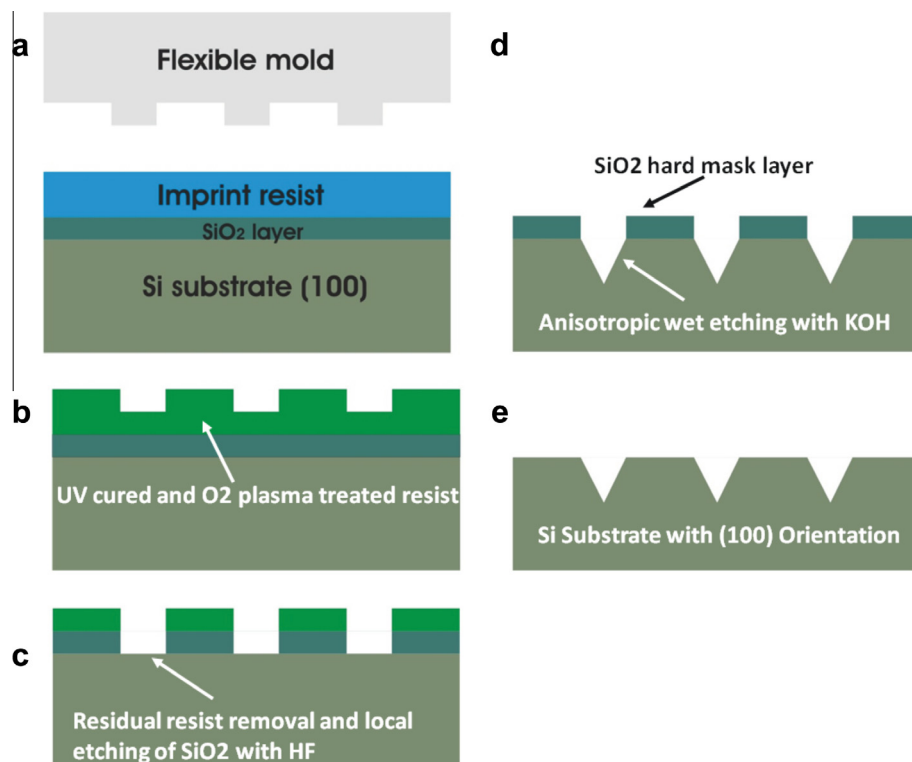


Fig. 1. Process flow for fabrication of inverse pyramids, SiO_2 layer was deposited as a hard mask layer and pattern definition step by soft UV-NIL was carried out (a), patterned imprint resist was treated with O_2 -Plasma (b), residual layer and SiO_2 layer was etched with diluted HF (c), anisotropic wet etching was carried out with KOH/IPA etchant (d), substrates were cleaned with HF in order to remove SiO_2 layer and resist residue (e).

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