Fast patterning and dry-etch of SiN_x for high resolution nanoimprint templates[*]

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Abstract: We developed a simplified nanofabrication process for imprint templates by fast speed electron beam lithography (EBL) and a dry etch technique on a SiN_x substrate, intended for large area manufacturing. To this end, the highly sensitive chemically amplified resist (CAR), NEB-22, with negative tone was used. The EBL process first defines the template pattern in NEB-22, which is then directly used as an etching mask in the subsequent reactive ion etching (RIE) on the SiN_x to form the desired templates. The properties of both e-beam lithography and dry etch of NEB-22 were carefully studied, indicating significant advantages of this process with some drawbacks compared to when Cr was used as an etching mask. Nevertheless, our results open up a good opportunity to fabricate high resolution imprint templates with the prospect of wafer scale manufacturing.

Key words: SiN_x templates; nanoimprint; NEB-22; electron bean lithography; reactive ion etch

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1. Introduction

Diffraction and sub-wavelength gratings can find broad applications in optics, opto-electronics, communications, nanophotonics, and nanobio-sciences. Modern optical systems often require high reliability, robustness, and functional integration of the gratings[1]. Recently, nanoimprint lithography (NIL) has been applied to the fabrication of gratings and other structures[2–7]. In the development of this technique, one of the challenges is to fabricate imprint/hot embossing templates with a large area, comparable to that of a wafer. Although electron beam lithography is able to meet the high resolution requirement[8], its serial writing nature results in a very long e-beam exposure time; thus, leading to a low throughput[9]. Therefore, high speed patterning with a high resolution by electron beam lithography (EBL) is urgently needed. The other challenge of fabricating a large area and high density sub-wavelength gratings is the separation of the template from the substrate, which becomes significantly more difficult when the template size increases to size of a wafer. Taking this into account, SiN_x was used as a template material due to its hardness and its low surface energy[10].

In an attempt to achieve a large area patterning by EBL, the highly sensitive, negative tone CAR resist, NEB-22, was applied to define the grating pattern by EBL. The patterned NEB-22 was then used as an etch mask in the pattern transfer into SiN_x by dry-etch RIE. The immediate advantages of such a process are that the e-beam exposure time is shortened by a factor of 50 due to the high sensitivity of the NEB-22 resist to the e-beam radiation compared to the sensitivity of conventional PMMA. In addition, the process of metallization/lift-off could be omitted. The etching properties of SiN_x with a NEB-22 resist as the etch mask were studied. By this process, both 100 nm and 150 nm half pitch SiN_x templates were fabricated. Further evaluation of this developed process was carried out by comparison with a Cr film used as the etch mask. The advantages and disadvantages of the NEB-22 technique were discussed. Finally, the fabricated 100 nm half pitch SiN_x mold was applied to nanoimprint lithography for sub-wavelength gratings of SU-8 on a Pyrex substrate without any anti-sticking agent.

2. Experiments and analysis

2.1. EBL process for forming grating patterns in a NEB-22 resist on SiN_x

The properties of EBL on NEB-22 were first carefully characterized by contrast curves at various post exposure bake (PEB) temperatures ranging from 90 to 120 °C. The process details were as follows: on a silicon wafer pre-coated by a 500 nm thick SiN_x grown by the PECVD method, the NEB-22 resist was spin coated with a thickness of 210 nm. The soft bake was undertaken on a hot plate at 100 °C for 2 min. Exposure of the resist using an electron beam was undertaken at 100 keV. Before each exposure, the beam spot size was characterized by scanning the e-beam across a gold wire. A 15 nm spot size

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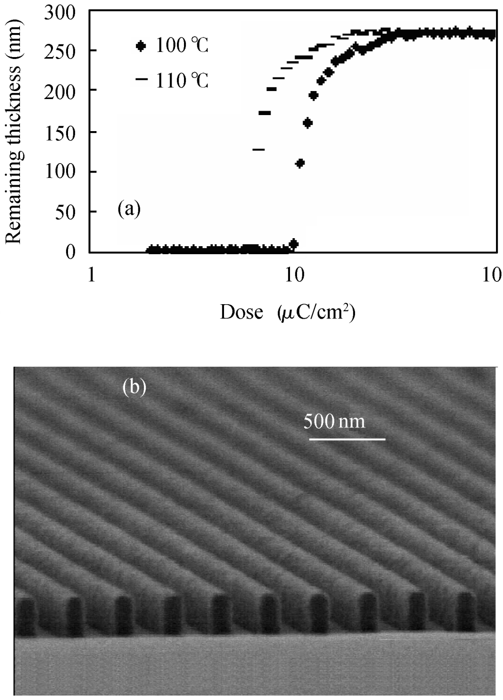

Fig. 1. (a) Contrast curves at two different post exposure bake (PEB) temperatures, 100 ℃(bar) and 110 ℃(diamond dots). A significant increase of the sensitivity can be achieved by increasing the PEB temperature; (b) SEM image of a 200 nm pitch gratings patterned in NEB-22 resist by EBL with a height of 210 nm.

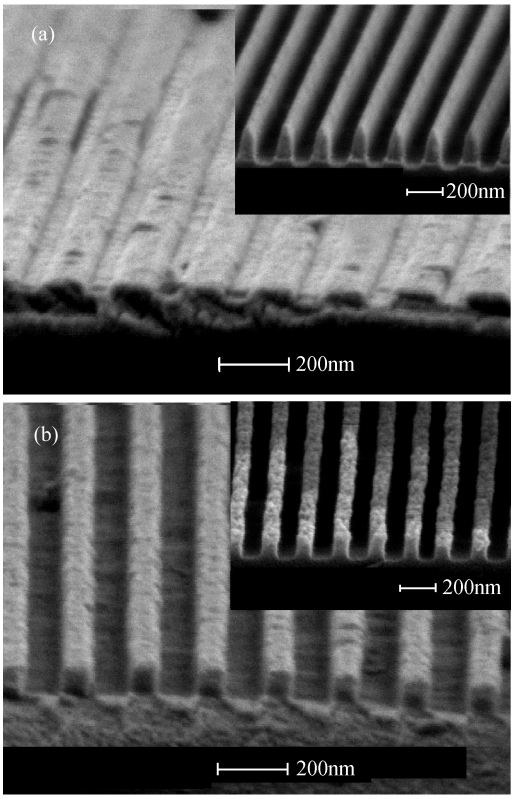

Fig. 2. SEM images of the cross-section of a 100 nm half pitch SiN$_x$ grating etched in the RIE system: (a) For an RF power of 50 W, an etching time of 25 min, and a CHF$_3$ gas flow rate of 30 sccm, the height of the SiN$_x$ gratings is 50 nm. The inset image is the view of the pattern before stripping off the NEB-22 resist; (b) For a RF power of 80 W and an etch time of 25 min, the height of the SiN$_x$ gratings is 80 nm.

was measured and kept for all the exposures in order to achieve a high consistency in the lithography conditions. A dose range was applied during the exposure to form a dose matrix on each site. Such an exposure strategy was repeated on a number of sites on one wafer to study how the lithography conditions influence the sensitivity of the NEB-22. In general, the sensitivity of an electron beam resist can be affected by many factors. A great deal of work has been done on varying the pre-bake temperatures[11]. In this work, we focus on the post exposure bake temperature, trying to achieve as high a sensitivity as possible. Our initial question was whether a good pre-bake temperature is essential for maintaining a good hardness of the resist in order to be able to sustain the subsequent dry etch process. However, the post-bake temperature provides us with a good opportunity to tune the sensitivity of the resist, achieving a fast patterning speed without losing its resolution. Figure 1(a) presents two contrast curves of NEB-22, post baked at 100 and 110 °C, respectively. It was found that the sensitivity can be readily increased by raising the PEB temperature. However, the PEB temperatures beyond 110 °C caused an unresolved pattern in the gratings in the adopted dose range. Therefore, in our work, the 110 °C was used as the PEB temperature, and the baking time was 2 min on a hot plate. The exposed resist was developed in CD26 for 60 s, followed by thorough rinsing in de-ionized water.

The optimized dose to form 100 nm lines/spaces in the NEB-22 was found to be 25 mC/cm^2, which is 3.5 times faster than when UVIII was applied (typical dose: 90 mC/cm^2) for the same pattern and 50 times faster than when PMMA was used (typical dose: 1250 mC/cm^2). During writing a 4 inch wafer, for example, our NEB-22 technique only requires 13 , which is possible for a beam writer. However, the exposure time for the UVIII technique is 2 days, requiring a very stable beam current of the beam writer. If PMMA was to be used, the writing time would be 27 days, which is too long for it to be used in a production process.

Figure 1(b) presents the scanning electron microscope (SEM) images of the gratings of the NEB-22 with a pitch of 200 nm and a height of 210 nm. A well defined resist profile without the residual resist can be observed.

2.2. Etch selectivity of NEB-22 over SiN$_x$

The patterned SiN$_x$ was subsequently etched in a CHF$_3$ plasma in a Samco RIE-10NR reactor. The purpose of using the plasma etching was to achieve the highest aspect ratio (height/width) with a vertical sidewall in the nanoimprint templates of the SiN$_x$. The processing parameters used for controlling the etched profiles included the RF power and the etch time at a fixed pressure of 4.0 Pa and a flow rate of 30 sccm. The etch depth of both the SiN$_x$ and the NEB-22 resist were monitored by a high resolution scanning electron microscope

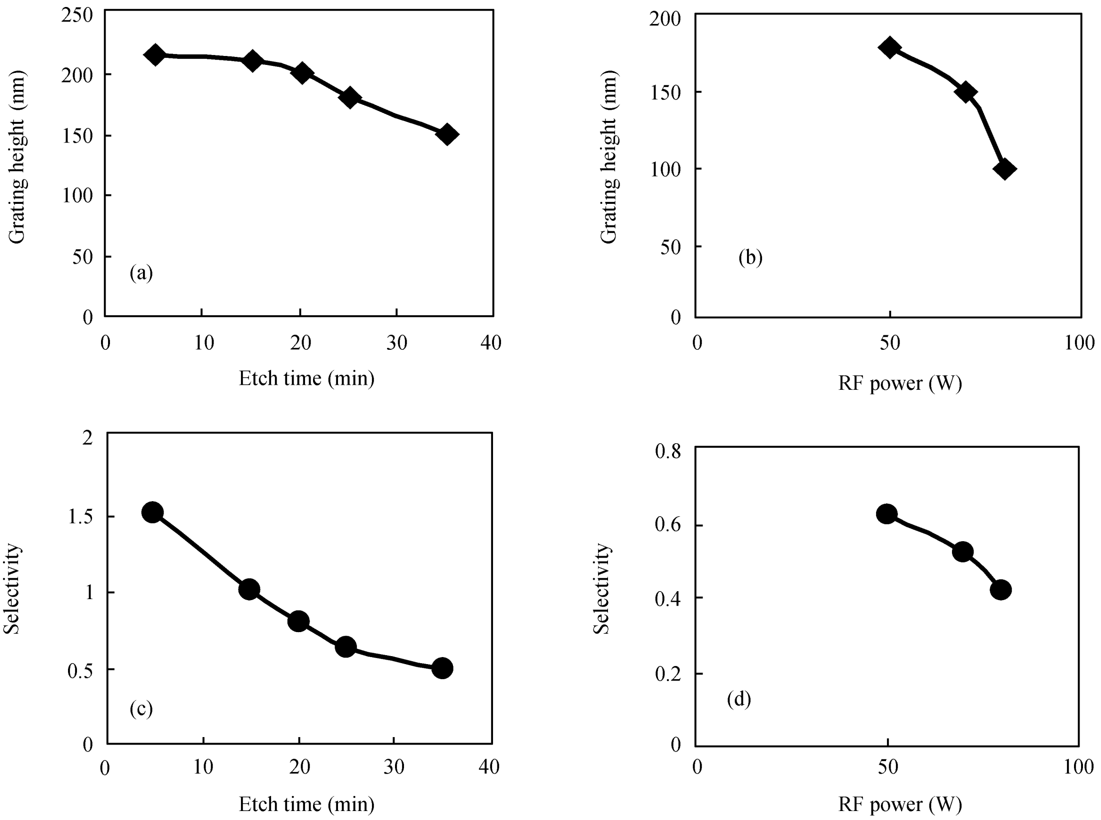

Fig. 3. Height of the etched depth and the selectivity of NEB-22 over SiN_x with respect to the etch time and the RF power: (a) Variation of the grating height with respect to the etch time (RF power is fixed at 50 W); (b) Variation of the height with respect to the RF power (time is fixed at 25 min); (c) Change of the etch selectivity of the NEB-22 resist to SiN_x with etch time; (d) Change of the selectivity with respect to the RF power.

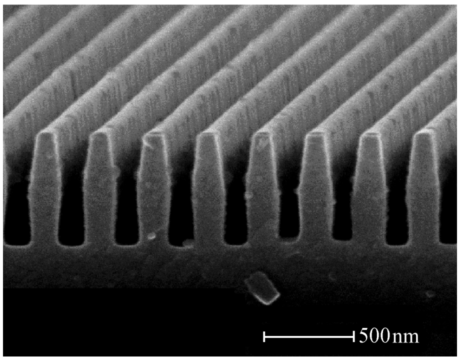

Fig. 4. Best profile for 150 nm half pitch gratings of SiN_x using Cr as the etch mask. The height of the gratings is 600 nm.

(SEM). Due to the poor contrast between the SiN_x and the NEB-22 resist, it was very difficult to distinguish the profiles between SiN_x and NEB-22. Hence, two groups of samples were tested: one used the SiN_x gratings with NEB-22 and the other used the SiN_x gratings with NEB-22 being removed by the Nanostrip for 30 min by ultrasonic agitation. Figure 2 shows two typical etched profiles of 100 nm half pitch SiN_x gratings, etched with a RF of 50 W for 25 min and a RF of 80 W for 25 min, respectively. The etched results demonstrated that the height of the etched SiN_x gratings was quite low: 50 nm when etched at 50 W for 25 min and 80 nm when etched at 50 W for 25 min. The optimized profile was obtained when the RF was 80 W and the etch time was 25 min. After etching using such conditions, NEB-22 resist was almost completely etched away. The rough surface on top of the gratings was caused by the remaining scum of NEB-22.

In order to analyze the etch selectivity of NEB-22 over SiN_x quantitatively, the depth of both SiN_x and NEB-22 were carefully measured by SEM. The etched depth of the gratings against the etch time (when the RF power was fixed at 50 W) and against the RF power (when the etch time fixed at 25 min) were measured as shown in Figs. 3(a) and 3(b). It can be seen that increasing the RF power or the etching time could not help increase the trench depth of the gratings; it even rather reduced it instead. The etch selectivity of NEB-22 over the SiN_x against the etch time and RF power was also deducted from the etch rates measured. Figures 3(c) and 3(d) show the effect of the etch time and the RF power on the selectivity, respectively. One can see that the higher the power and the longer the time, the lower the selectivity. The maximum selectivity, which is about 1.5, happens at the lowest RF power. This is unfortunately a weak point in this technique. With such a low selectivity, the fabricated SiN_x templates will not have a high aspect ratio.

2.3. RIE on SiN_x using a Cr film as a etch mask as a comparison

For comparison, Cr was also used as an etching mask for the templates in SiN_x. In this case, conventional PMMA was

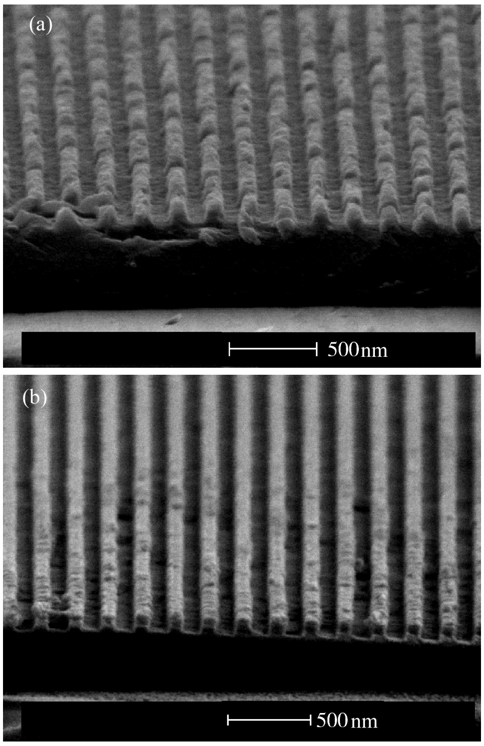

Fig. 5. (a) SEM image of the imprinted gratings in the SU-8 resist by the 100 nm half pitch SiN_x template with a depth of 60 nm; (b) SEM image of the SiN_x template with a depth of 80 nm.

applied during EBL to form a 150 nm lines/spaces. Physical vapor deposition (PVD) was then carried out, followed by a lift off process to form the Cr mask with a thickness of 40 nm. Figure 4 presents the etch profile of the gratings under the optimal condition of 100 W for the RF power, a 35 sccm CHF3 flow rate, and a 40 minutes etching time. The selectivity of Cr to SiN_x was as high as 15, which was at least 7.5 times that of the NEB-22 resist.

2.4. Imprinting a SiN_x template into the SU-8 resist

The fabricated SiN_x template with 1×1 mm^2 in area, 200 nm in pitch, and 80 nm in height was used to nanoimprint in a 100 nm thick SU-8. In this process, no anti-sticking agent was used for the de-molding process. The original template was etched in the Samco RIE-10NR system and had a uniform depth over the whole grating area; meanwhile, the depth of the transferred gratings in SU-8 resist was 60 nm and it had a variance of about 5–10 nm at the two different ends of the imprinted patterns with an area of 1×1 mm^2. The depth difference of the imprinted patterns was not due to the template, but due to the pressure un-uniformity during the imprinting process, which can be mitigated by adding paper or PDMS as a buffer layer between the torque and the imprinted system. There is also a ball joint in the imprinting machine to assure the uniformity of the imprinted pressure. Figure 5 shows the cross section of the imprinted SU-8 gratings and the corresponding SiN_x template we used. Clear grating structures have been created by the imprint process. However, compared with the SU-8 gratings imprinted with silicon templates[12], the imprinted SU-8 grating is more defective. This could be caused by the roughness and the defects in the templates when removing the NEB-22 resist in the nanostrip solution by an ultrasonic treatment.

3. Conclusions

In this paper, a nanofabrication technique involving fast speed EBL and dry etch was demonstrated for the fabrication of high resolution SiN_x templates. It has been proven that wafer scale manufacturing of templates is feasible when a highly sensitive resist is applied. Using NEB-22 as a pattern definition and etch mask layer, 100 nm half pitch SiN_x gratings were fabricated. However, due to the limited resistance to the fluorine-based plasma of NEB-22, the fabricated templates do not have a high aspect ratio, compared to when Cr is used as the etch mask. A thicker NEB resist may be the effective solution for achieving templates with high aspect ratios. Our work certainly has shown that wafer scale templates with high resolution are feasible when a CAR resist is used during electron beam lithography.

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