Study of key processes in the fabrication of high quality diffraction gratings by nanoimprint lithography

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Diffraction grating is very critical component of distributed feedback laser diode (DFB LD) and its quality has a strong influence on the performance of final device. Nanoimprint lithography (NIL), as a very promising technology for nanostructure fabrication, has been applied to fabricate diffraction grating recently. In this paper, key processes to achieve high quality gratings by NIL are discussed, including the soft stamp applying, the removal of residual layer and the etching of substrate. Large area and high quality diffraction gratings are demonstrated at the end, laying the foundation for industrial-level mass production of DFB LD diffraction gratings by NIL.

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1. Introduction

Distributed feedback laser diode (DFB LD), because of its narrow linewidth, high side mode suppression ratio and wavelength stability, has always been the most attractive and competitive optical source of optical communication networks since it was invented. Along with the extention of optical fiber communication from long-haul networks to metro and even access networks, reducing the cost of optical networks components, especially the optical source, is highly desired.

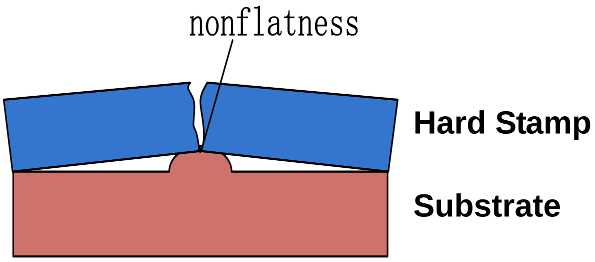
Besides epitaxial process, performances of DFB LD mainly depend on the quality of embedded diffraction gratings. The final device yield, then the cost of single device, is also mainly dependent on the fabrication yield of diffraction gratings. Conventional fabrication methods of diffraction gratings, such as interference exposure and electron beam lithography (EBL), have their own drawbacks respectively. Interference exposure can be used to fabricate uniform gratings with low cost, however it is not suitable for gratings with phase-shift, which give better device performance than uniform gratings, or gratings with different periods on single chip. EBL suffers from high cost and low throughput, even though it has sufficient resolution and the capability of fabricating arbitrary patterns.

Nanoimprint lithography (NIL) was proposed first by Stephen Chou in 1995 [1]. Since then NIL has found extensive applications in many fields due to its promising characteristics of high resolution, low cost and high throughput, including photonic crystal [2], metal grating [3], micro circuit [4], nanofluidic channels [5] and other structures. Recently NIL was developed to fabricate diffraction gratings for DFB LD and demonstrated high potential [6~8]. To push forward industrial-level mass production of DFB LD diffraction gratings by NIL, in this paper, key processes of fabricating high quality gratings by NIL are discussed, including the choice of mother stamp type, the soft son stamp applying, the removal of residual layer and the etching of the substrate. Through the optimization of every single process step, large area and high quality diffraction gratings which have period of about 240nm and phase-shift in the center are demonstrated at the end.

2. Experimental

Different from conventional lithographic approaches that define patterns through the modification of photoresist properties caused by either photons or electrons and following development in specific developing solution, NIL is based on direct mechanical deformation of the resist under imprinting. This principle determines the resolution of NIL is beyond limitations set by light diffractions or electron beam scattering but mainly limited by the stamp. Actually, sub 10nm features patterned by NIL were reported [9~10].

However, because InP epitaxial wafers have always undulations and we can never make sure the wafer has not any dust particle, several problems as illustrated in Fig.1 could happen when a hard stamp is used to press against a hard substrate. Stamp and epitaxial wafer which are both expensive could be damaged and big hollow areas that result in big area patterning defects could be formed.



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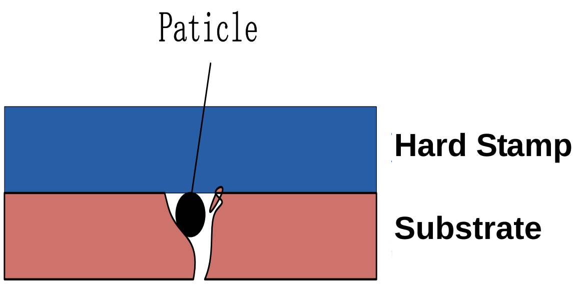

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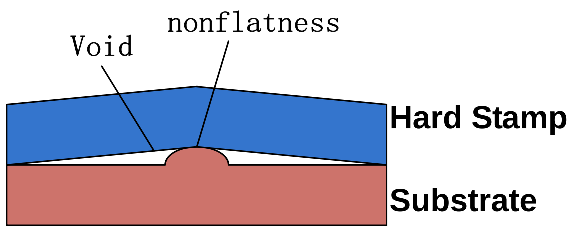

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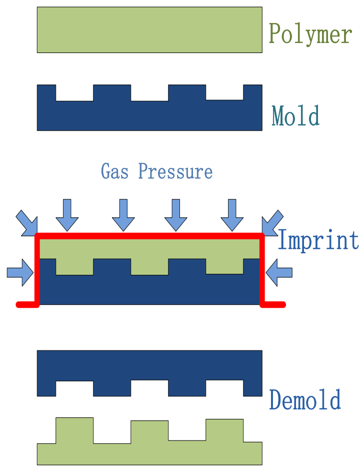
Fig.1. Schematic diagram of problems that may occur in NIL with a hard stamp: (a) Damage to stamp, (b) Damage to substrate, (c) Large incomplete filling defect area

To overcome the problems above-mentioned, soft stamp scheme is applied here. As shown in Fig.2, first, soft son stamp is fabricated by pressing a hard mother stamp against the polymer film through thermal NIL at a high temperature above T_g of such polymer, and then, soft son stamp is used to transfer patterns onto a final substrate by simultaneous thermal and ultraviolet (STU) imprint process at a temperature far below the T_g of such polymer. In our experiments, both this two steps are performed with the Etire3 machine from Obducat Ltd.



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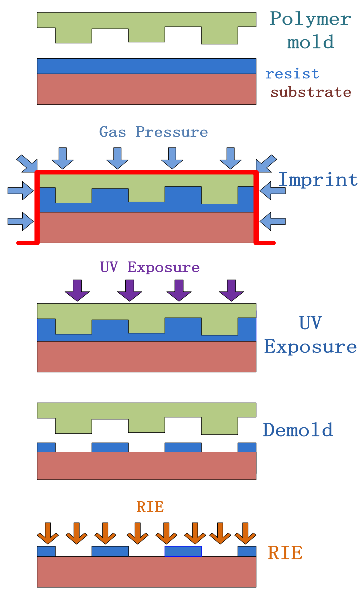

(b)

Fig. 2. Schematic diagram of process flow of soft stamp scheme: (a) Fabrication of a soft son stamp, (b) Imprint with the soft son stamp and STU process

The soft stamp used here has several obvious advantages compared with a hard one. (1) As shown in Fig.3 (a), particles sticking on the mother stamp will be pulled off by the polymer film during mold replication, then the mother stamp will be cleaned and newly defined soft son stamp will be much better. (2) The soft son stamp can self-adapt to the substrate even though there are undulations or particles on the substrate surface, largely reducing incomplete filling defect areas and preventing probable damages to the mother stamp or substrate, thus improving imprint result as shown in Fig.3 (b). (3) The soft stamp is ideally anti-adhesive to the imprint resist, hence demoulding process is very easy without destroying patterns.

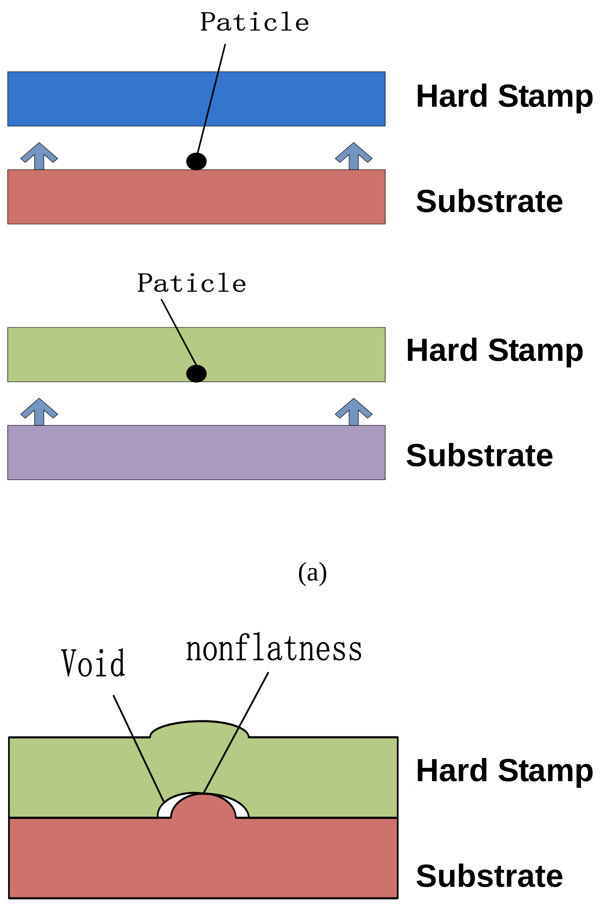

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Fig.3. Schematic diagram of main advantages of soft stamp compared with a hard one: (a) Stick off particles and clean the mother stamp, (b) Self-adapt to the substrate

Mother stamps are usually defined by a high-resolution electron beam writer on silicon wafer. Before imprinting, they are rinsed in acetone, isopropanol, and deionized water and then dried with N_2 flow. Afterwards, they are baked at 140 °C for 5 minutes on a hot plate. The new vacuum packaging InP substrate wafer can be used without washing. It is baked on a hot plate, then spin-coated with TU2 (a UV-curable resist, developed by Obducat Ltd.) and baked at 95 °C for 3 minutes.

To demonstrate advantages of soft son stamp in handling above-mentioned issues, a thermal NIL process directly using a hard silicon stamp imprinting against substrate is carried out as a comparison. The pattern of this silicon stamp is almost the same as that of the mother stamp used in the soft stamp scheme and this experiment is also performed with the Etire3 machine. A thermoplastic polymer imprint resist named mr-7020E, produced by Micro Resist Technology Ltd., is used here. During the imprint, the 7020E resist is spin-coated on a badly cleaned 2 inch diameter single-side polished silicon substrate to a thickness of 200nm. In order to minimize the defect area, the thermal NIL is carried out at 155 °C, and a pressure of 40 Bar for the same long time as the soft stamp scheme.

Usually there are two pattern types for the mother stamp, positive and negative. For the positive type, patterns are convex that means the top surface of patterns is higher than areas with no patterns. While for the negative type, patterns are concave that means they are beneath the stamp surface. These two types are both widely used in NIL. Here experiments using these two kinds of stamps as mother stamps in the soft stamp scheme respectively are conducted to make a comparison to find out which type is more suitable for NIL technology. These two stamps have nearly the same patterns, which are both diffraction gratings for DFB LD. Only difference is that the depth of the negative stamp is about 130nm while that of the positive stamp is 60nm.

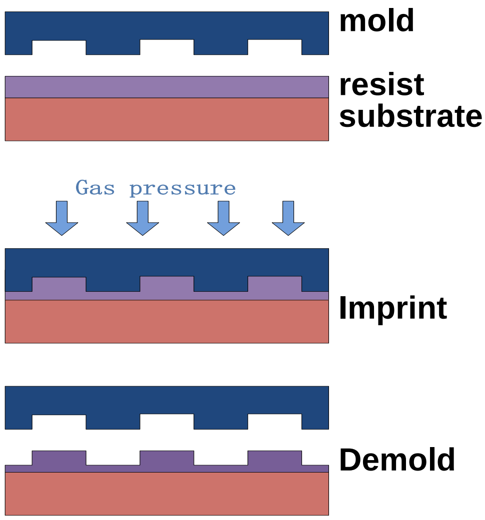
After imprinting, removal of the residual layer and etching of the substrate are two following critical steps of pattern transferring. In this paper, experiments using Inductive Coupled Plasma Reactive Ion Etching (ICP-RIE, Oxford 100 system) are carried out to optimize parameters.

The inspection of pattern quality is done by AFM (Veeco NanoScope MultiMode), SEM (Quanta 200) and optical microscope (Olympus BX51).

3. Results and discussion

3.1 Advantage of soft stamp scheme

Fig. 4(a) shows the process flow of thermal NIL directly using a silicon stamp. Fig. 4(b) shows a typical picture of particle influence in direct hard stamp thermal NIL process, gradually changed resist color forming a radiating shape around the particle as described in[11~12]. It's in fact caused by the particle that makes the space variation between the two pressing plates as shown in Fig.1(c). Although high pressure, high temperature and thick resist can improve the flow of resist, large hollow space between two pressing plates is still very difficult to fill up. The defect center looks black, seeming as that the substrate is damaged when such high pressure is applied on the small particle.



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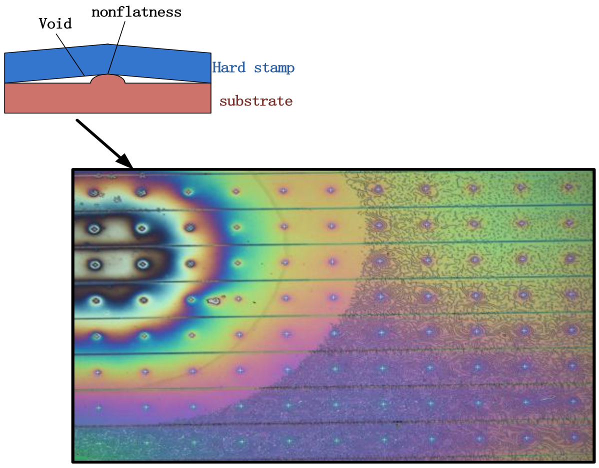


(b)

Fig.4 (a) Schematic diagram of thermal NIL process flow, (b) Microscope image of particle influence in direct hard stamp thermal NIL process

In contrast, for soft stamp scheme process, influence of particles with the diameter as large as about 10 μm is shown in Fig.5. Even though particles are very close to the grating pattern, there is nearly not bad influence. This should be attributed to the self-adapting feature of soft stamp. This demonstrates the great ability of soft stamp to minimize influences of dust and non-flatness and improve yield of large area imprinting.

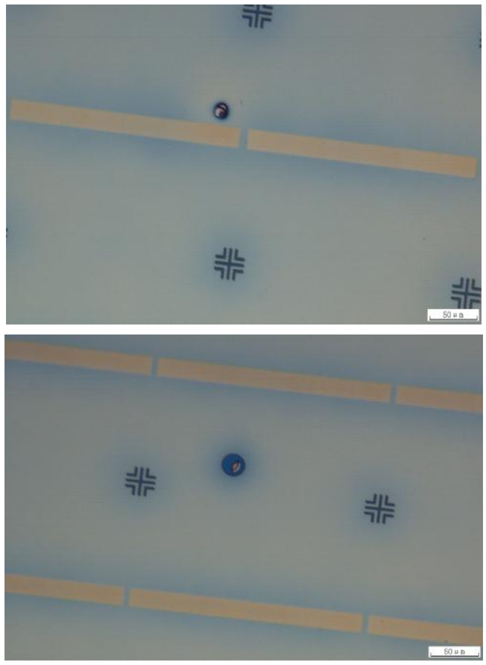
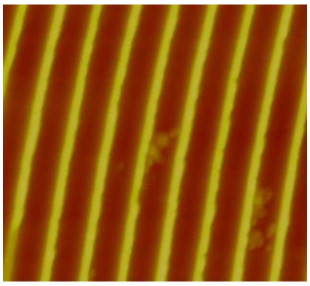


Fig.5. Microscope images of particle influence in soft stamp scheme process

3.2 Comparison of positive stamp and negative stamp

Using positive stamp and negative stamp as mother stamps respectively, we have done experiments with the same process and other parameters but different imprinting time. Fig.6 shows the imprinting results. We can see that for negative mother stamp patterns could be completely filled only in 3 minutes to get very good grating quality. However, it takes positive mother stamp 30 minutes to get the same quality of grating as the negative one and patterns could not be fully filled within 10 minutes. Negative mother stamp is more effective in pattern filling than positive mother stamp. One point that should be emphasized is that you should always keep in mind the special soft stamp scheme used here. Positive mother stamp gives negative soft son stamp and negative mother stamp means positive soft son stamp. In the first step of soft stamp scheme pattern can be always completely filled in the polymer film because the polymer film is thick enough. Difference is located in the second step, namely the pattern transferring from soft son stamp to substrate. So we can also draw the conclusion in other words that positive soft son stamp is more effective in pattern filling than negative soft son stamp.



(a)

(b)

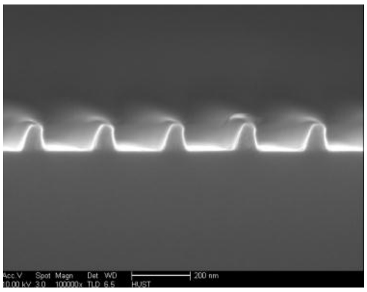
(c)

Fig.6. AFM 2D images of imprinting results: (a) Positive mother stamp with 10 minutes. (b) Positive mother stamp with 30 minutes. (c) Negative mother stamp with 3 minutes.

3.3 Removal of residual layer

For the aim of protecting stamp and substrate, there is always residual resist layer that remains underneath imprinted structures. This residual layer has to be removed to expose the substrate prior to following etching step. However, erosion of resist mask happens along with the removal of residual layer, and actually resist mask is eroded faster than residual layer is etched because the residual layer is at the bottom of the imprinted structures. Hence parameters must be chose carefully to etch the residual layer effectively while maintaining the resist mask as fine as possible.

Here we use O_2 and SF_6 as etching gases, and their flow rates are 20sccm and 5sccm respectively. Pressure is 7mTorr. Three different RF power schemes are applied to make a comparison, 90W for 85 seconds, 80W for 100 seconds and a two steps scheme with 100W for 30 seconds following 80W for 55 seconds. Fig.7 shows SEM results. The little bit decreased duty cycle can be easily solved by reducing the etching time. From the aspect of resist mask structure maintaining, the two steps scheme gives the best result. The verticality is very good and the structure is most similar to rectangle. This could be explained that at the beginning of removing residual layer, resist mask structure is very deep so that 100W RF power can generate more vertical plasma particles to etch residual layer more effectively. When the resist mask structure turns to relatively shallow, 80W RF power is sufficient to etch the residual layer effectively but also decreases the erosion of resist mask.



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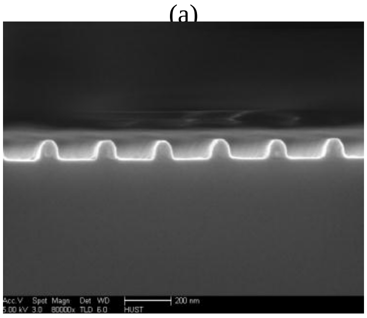

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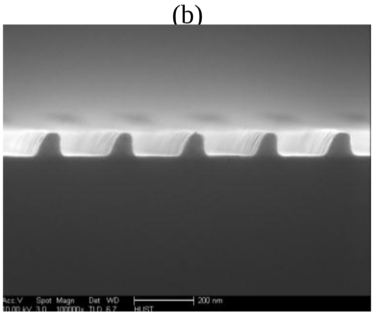


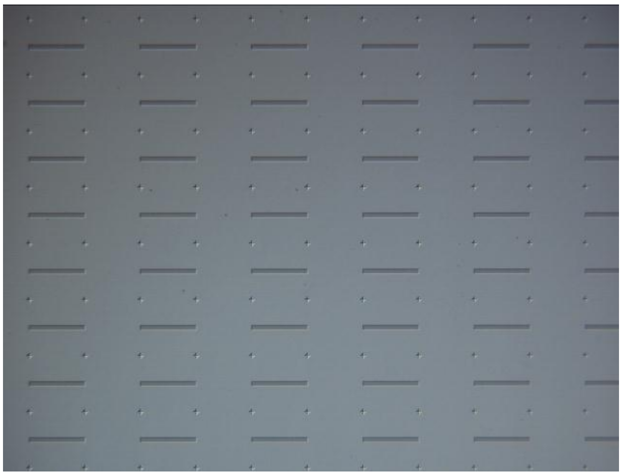
(c)

Fig.7. SEM images of after removing residual layer with different RF power scheme: (a) 90W for 85s, (b) Two steps, 100W for 30s following 80W for 55s, (c) 80W for 100s.

3.4 Etching of substrate.

After removing the residual layer, etching of InP substrate is performed with CH_4/H_2 chemistry. Temperature is 55 °C . RF power, pressure and flow rate ratio of CH_4 and H_2 are varied. Finally we get the best result when RF power is 80W, pressure is 10mTorr, and flow rate ratio of CH_4 and H_2 is 40sccm:10sccm, shown in Fig.8. This is very different from results of reference

[13], which said InP surface began to undergo polymer deposition rather than etching when CH_4 concentration exceeded 25%. Fig. 8(a) shows large area etched patterns on InP substrate under optical microscope, and actually it is just part of 2 inch wafer. Each stripe is for a DFB LD, 250 μm long and 20 μm wide. We can see it is very uniform. Figure 8(b) gives SEM images of detailed grating structure, which is uniform and perfect rectangle with depth of 60nm that is enough for DFB LD application.



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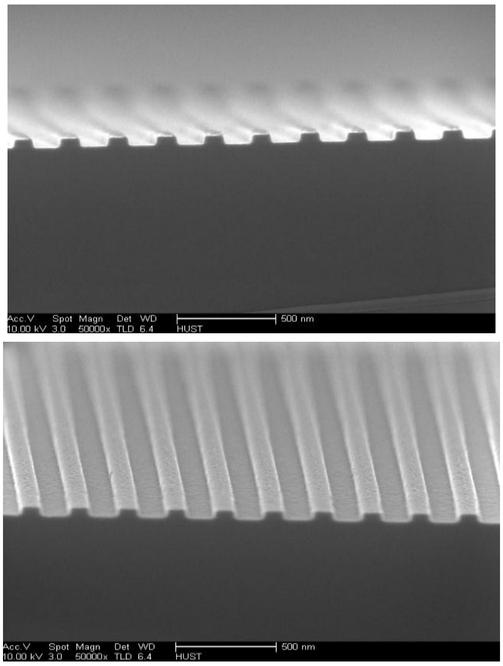

(b)

Fig.8. (a) Microscope image of large area patterns on InP substrate, (b) SEM images of detailed grating structure.

4. Conclusions

Large area and high quality diffraction gratings for DFB LD are fabricated by NIL. Key steps of the process are investigated carefully, including the choice of mother stamp type, the soft son stamp applying, the removal of residual layer and the etching of the substrate. Experimental results show that negative mother stamp is more effective in pattern filling than positive one and soft stamp scheme has several advantages over imprinting directly using hard stamp. Also in removing of residual layer, etching parameters are optimized to etch residual layer effectively while maintaining good resist mask structure. Finally, perfect rectangular grating structures are obtained with optimized etching parameters. Results of this study lay the foundation for industrial-level mass production of DFB LD diffraction gratings by NIL.

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