Study of Reactive Ion Etching for Reverse Tone Nanoimprint Process

Y Tsuji, M Yanagisawa, H Yoshinaga, and K Hiratsuka
Fiber-optics Core Devices R&D Department, Transmission Devices R&D Laboratories, Sumitomo Electric Industries, LTD.
1, Taya-cho, Sakae-ku, Yokohama, Kanagawa, 244-8588 Japan
E-mail: tsuji-yukihiro@sei.co.jp

Abstract: We have used reverse nanoimprint for fabricating diffraction gratings of distributed feedback laser diodes. Generation of residues in the etching process of resin is a serious issue leading to poor line edge roughness of the grating patterns. We have found that the residues are composed of oxide products from Si-containing resin. We have successfully suppressed the generation of the residues by optimizing oxygen partial pressure of reactive ion etching (RIE). We have also succeeded in effectively removing the residues by utilizing sputtering effect of RIE.

1. Introduction
Nanoimprint lithography (NIL) has been studied to fabricate diffraction gratings of distributed feedback laser diodes (DFB LDs) for optical communication network [1]. Because compound semiconductor wafers such as GaAs and InP for device fabrication have a certain undulation, thickness uniformity of imprinted residual layer is affected. Then, reverse tone nanoimprint process, for example SFIL/R™ process proposed by Molecular Imprints, Inc. [2], has been applied in order to improve the uniformity of residual layer thickness. There are two kinds of resin layers stacked on the wafer and the two-step reactive ion etchings (RIE) have been used for pattern transfer. The upper layer is a Si-containing resin, which is used for an etching mask in the second RIE.

In this paper, we demonstrate the optimized etching condition for fabricating diffraction gratings in view of eliminating resin residues.

2. Experimental Procedure
Reverse tone nanoimprint process is shown in figure 1. We prepared 2-in-diameter InP epitaxial wafers including InGaAsP multi-quantum-well (MQW) layer, the function of which is not only an active layer but also a grating layer. Grating patterns with 200-240 nm pitch have been transferred on the wafers by a serial process described in below. An 80 nm-thick film of adhesion layer is spin-coated on the wafer [figure 1(a)]. An 8 mm x 6 mm template is pressed on dispensed UV curing resin at a force of 4 N [figure 1(b) and 1(c)]. After demolding, the spin-coating of a 200 nm-thick Si-containing resin is followed by the etch-back by first RIE to reveal the tops of the imprinted feature, which is selectively etched down to the wafer by subsequent RIE using oxygen [figure 1(d), 1(f), 1(g) and 1(h)]. These grating patterns are used as masks for the etching of the wafer [figure 1(i)].
The second RIE is carried out using the inductively coupled plasma (ICP) RIE system. The etching rate of the adhesion layer is approximately 200 nm/min and the etching selectivity ratio is more than 30. The etching time is 3 minutes. We used a mixture of oxygen and nitrogen or CF$_4$ as the etching gas. Fabricated patterns are observed by scanning electron microscope (SEM, Hitachi, S-4800) after the second RIE.

3. Results and Discussion

When we used our conventional etching condition of second RIE, many resin residues were found on grating patterns. The condition was with the bias power of 100 W, with the reactor pressure of 0.8 Pa, and with the oxygen flow rate of 117 sccm. Chemical composition of the generated reaction products have been analyzed using energy dispersive X-ray spectroscopy (EDX, Horiba, EX-320). EDX spectra after a conventional etching condition are shown in figure 2(b) and 2(c). Oxygen composition in residues area is larger than that in no resin residues area. Thus, we guesse resin residues are oxide-silicon. Figure 3 is a SEM image of grating patterns with a 200 nm pitch and shows there are undesirable residues in the grooves of the gratings. These residues appear when oxygen is exclusively supplied as the etching gas for second RIE. Thus, it is considered that the residues originate in reaction of Si-containing resin and oxygen. We guess that the residues are mainly composed of SiO$_2$, because the residues are not removed but increased by extenting the etching time of second RIE.
Figure 2. EDX analysis after conventional etching condition from the point of (b) resin residues and (c) no residues area which are indicated in (a) SEM.

Figure 3. SEM images of grating patterns with resin residues. Resin residues are shown by arrow symbols in the photographs.

Figure 4 is a SEM image of cross sectional view of the grating pattern after the second RIE using both oxygen and nitrogen gas. When oxygen flow rate is 4 sccm and nitrogen flow rate is 6 sccm, there are few residues in the grooves. So, it is considered that the reaction between the Si-containing resin and oxygen is suppressed because of less oxygen supplied.

Figure 5 is a SEM image of cross sectional view of grating pattern after the second RIE with the bias power raised to 250 W. It is found that the residues in the grooves are completely disappeared, because residues are mechanically removed by sputtering effect because of the bias power increased.

Figure 6 is a SEM image of cross sectional view of grating pattern after the second RIE with a mixture of oxygen and CF₄. When oxygen flow rate is 50 sccm and CF₄ flow rate is 4 sccm, there are also no residues in the grooves, because the chemical reaction between the resin residues and radicals are increased. However, a large undercut is found in the lower mask layer. In these etching conditions, etching selectivity ratio decreased to more than 20.
4. Conclusion
Several RIE conditions have been examined for transferring nanoinprinted grating patterns to DFB LDs. We have found that the residues are eliminated by using RIE conditions with the mixture of nitrogen and oxygen or CF$_4$ and oxygen. This is because of the three reasons: (1) generation of resin residues is suppressed by lowering oxygen ratio for the second RIE, (2) generated residues are removed by sputtering effect due to increasing the bias power of second RIE, and (3) generated residues are removed with enhanced chemical reaction by using a mixture of oxygen and CF$_4$ gas.

We consider that the large undercut found in (3) condition is undesirable problem, so we conclude that (1) and (2) are the most suitable conditions for fabricating fine grating patterns with the pitch of 200-240 nm.

References
[1] Yanagisawa M, Tsuji Y, Yoshinaga H, and Hiratsuka K 2008 21st International Microprocesses and Nanotechnology Conference 29D-9-125 pp 400-401
[2] Miller M, Doyle G, Stacey N, Xu F, Sreenivasan S V, Watts M, and LaBrake D L 2005 Proc. SPIE 5751 994.