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Dc drift reduction in LiNbO₃ optical Modulators by decreasing the water content of vacuum evaporation deposited SiO₂ buffer layers

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1 Introduction

With increasing demand for LiNbO3 (LN) optical intensity modulators for fiber communication systems, fine process Control of LN device fabrication has become important to increase the fabrication yield and to supply low-priced mental results concerning the quality of LN crystal wafers and device fabrication processes.1-5 For the fabrication of optical intensity modulators, especially, process control to obtain reduced dc drift device is needed. Our previous communication described such a technique, where the measurement of the refractive index of the SiO₂ buffer layer over the LN waveguides was found to be applicable to an in-process screening test.4 The buffer layer formed to have a higher refractive index tended to lead to inferior modulators showing larger dc drift behavior. Here, the chemical composition of the buffer layer is investigated, and H₂O (OH) content is also considered to influence the larger dc drift

2 Process Control by SiO2 Refractive Index

As described in our previous communication, the magnitude of the dc drift can be screened by measuring the re-

fractive index of the SiO2 buffer layer after the process.4For instance, the 1.44 measured at wavelength of 633 nm after the oxygen annealing process at 600°C can be chosen as the screening criterion, where the processed wafers showing a higher SiO₂ refractive index are predicted to result in inferior devices with larger drift behavior.6 Such a criterion was applied to both the in-process screening test and the process control for maintaining device fabrication repeatability. If the measured refractive index was higher, process be readjusted to decrease the refractive index. The previous investigation found that the initial stage of the dc drift behavior was mainly influenced by the nature of the SiO₂ buffer layer.^{1,3,7} A SiO₂ layer with a smaller refractive index provided a larger initial negative drift, canceling the undesirable positive drift, and ultimately suppressing the long-team dc drift. Such a negative drift was considered to be caused by the retardation in the effective application of the bias voltage to the LN waveguides though the buffer layer.⁷ Contrarily, the dense buffer laver, such as the sputtering deposited SiO₂ layer, had a high enough electric resistance to induce an occurrence of bias relaxation in the LN substrate; i.e., the intrinsic dc drift.78Such consideration was consistent with the observed results of a smaller dc drift for the lower SiO₂ refractive index device, and a larger drift for higher refractive SiO2 index devices.

3 Remaining Uncontrolled Drift Characteristics in Devices

As described, the dc drift behavior is strongly affected by the nature of the SiO₂ buffer layer. Especially, the vacuum evaporation deposited SiO₂ layers were not as dense as the sputtering deposited SiO₂, and their refractive indices were observed to change depending on the storage atmosphere. There was a tendency for the refractive index to increase slightly when the processed films were kept for a few hours (one night) in an ordinary room atmosphere. Therefore, the accurate measurement of the refractive index for the purpose of in-process screening of the SiO2 films was sometimes problematical. For instance, although the refractive index values were measured to be almost the same and less than 1.44 at = 633 nm for all the samples, the dc drift magnitude scattered slightly depending on fabricationbatch. There results mean that an uncontrolled on fabrication parameter remained, and the dc drift phenomenon could not be stabilized completely by controlling only the SiO₂ film density, which was known to result in a large difference in the film refractive index.

As a possible origin for the uncontrolled parameter, we considered the effect of OH in the prepared SiO₂ buffer layer, because a lower OH content in the LN wafer was found previously to suppress the dc drift.9 Further, the re-fractive index of the vacuum evaporation deposited oxide



Fig. 1 TDS measurement results for the as-deposited (solid curve) and the 600° C wet O₂ annealed (dotted curve) SiO₂ films : (a) the thermal dsorption of H₂O (mass number 18) and (b) the desrption of mainly O₂ (mass number 32) from the film samples.

films was reported to increase by absorbing humidity (H₂O), which was consistent with our observation in the process.¹⁰ To investigate the existence of H₂O (OH) in the SiO₂film, thermal desorption spectroscopy (TDS) was performed for the as-desorpted 500-nm-thick film and the annealed film. The annealing was done at 600°C for 5 h in flowing wet O2. The SiO2 film for the TDS analysis had been deposited on both sides of the LN substrate to exclued the effect from the substrate. Figures 1 (a) and 1 (b) show the TDS profile for the detected gases with mass number 18 (mainly H2O) and 32 (mainly O2), respectively, where the solid curves are for the as-deposited film and the dotted curves are for the annealed film. As seen, the asdeposited film included large amounts of H₂O (OH), and with the wet O₂ annealing, the H₂O (OH) content was reduced (to about 20 %). Further, the H₂O in the annealed film was found to evaporate below 250°C almost completely. At 280°C we also detected the degassing of O₂, which was possibly trapped in the film as excess O₂ molecules.

The result of Fig. 1 indicate that a vacuum annealing of the buffer layer at 250°C was effective in checking the influence of H₂O in the SiO₂ to the dc drift. TWO LN modulator devices for $= 1.55\mu$ m use were fabricated from two different wafer with an ~ 1- μ m-thick SiO₂ buffer layer prepared by the same vacuum evaporation deposition and

followed by a wet O₂ annealing process. Then, one wafer sample was baked at 250°C for about 15 h in a vacuum just before a sputtering deposition of the thin Si layer over the buffer layer, while the over was not baked before the Si deposition. This Si layer worked as a protection layer against humidity.¹¹ The following process, such as electrode formation, etc., were similarly performed for both wafers. Ultimately, two modulator devices with and without the SiO₂ baking were assembled into the conventional hermetically sealed packages.

Figures 2(a) and 2(b) reveal the results of the OH con-Tent measurements for the samples with and without the SiO₂ baking, respectively, by secondary ion mass spectros-Copy (SIMS). The horizontal axis denotes the depth from the Si surface. The contents of Si, O, Na (impurity), and Li (impurity outdiffusing from LN substrate) were shown to be almost identical for both samples. As expected, the H content was confirmed to be reduced in the baked SiO₂layer; it was ~ 0.5×10^{21} atoms / cm3 for the baked film and 1×10^{21} atoms / cm³ for the unbaked layer. Similar results were also obtained by hydrogen forward scattering spectroscopy (HFS) for the other pieces of the wafers, as shown in Fig. 3. The amount of H (H₂O) in the SiO₂ layer was reduced by the vacuum baking to half of that in the unbaked one.

Finally, the dc drift was measured for the two modulators at 80°C for 100 h with an initially applied bias voltage of 3.5 V. Figure 4 shows the chronological change in the dc voltages applied to maintain the optical output modulation signal at the initial state. As shown by the solid curve, the dc drift of the baked modul1 tor sample was successfully reduced compared with that of the unbaked one (dotted curve). Especially, the magnitude of the negative drift appearing in the first 5 h of measurement was significantly greater for the baked modulator. The measurements were performed for other modulators assembled from the same wafers and similar results were obtained.

4 Discussion

The reduction of the H₂O (OH) contents in the SiO₂ buffer layer was found experimentally to be effective in decreasing the magnitude of the dc drift. Other LN modulators with dry O₂ annealed and the vacuum baked buffer layers, in which the H₂O was possibly further reduced than in the modulators of Figs. 2 to 4, were also fabricated and a greater drift toward the negative direction was observed. The sama effect, the intensified negative drift, was buffer Layer was increased.¹ Seino et al.⁷ reported similarly a large negative drift at an early stage.

These experimental results were considered to come from changes in the electrical characteristics of the SiO₂film. for instance, the reduction of the absorbed H₂O molecules might relax the film structure via. decreasing the dopaing of the glass intermediate ions such as Ti might introduce a loose bonding in the tight SiO₂ network structure. The introduction of such external and internal relaxation in the SiO₂ film structure was expected to change the dielec-



Fig. 2 SIMS measurement results for (a) the vacuum baked SiO₂ layer on the LN and (b) the unbaked SiO₂ layer. Ion emissions corresponding to H, Li, O, Na, Si, and Nb, respectively, were detected. The amount of H detected from the films was quantitatively plotted in units of atoms per cublic centimeter on the left vertical axis of the figures.

tric characteristics (electrical permittivity) of the film. Further, suitable amount of H₂O (OH ions) and glass modifer ions such as In, were expected to largely change the electrical resistivity of the film, commonly decreasing the resistivity. The thickness of the total (effective) magnitude of these electrical parameters.

At this moment, the direct evidence for the preceding consideration of a possible change of the electrical parameters of the SiO₂ has not yet been found. The data experimentally shown here, however, should be useful for the design and fabrication of reduced dc drift LN modulator devices. Especially, the dc drift phenomenon could be sup-



Fig. 3 HFS measurement results for the vacuum baked (open circles) and unbaked (solid circled) SiO_2 layer on the LN substrate. The vertical axis denotes the signal intensity corresponding to the H amounts in the film.



Fig. 4 Results of dc drift measurements at 80° C for devices consisting of a vacuum baked SiO₂ buffer layer (solid curve) and of an unbaked buffer layer (dotted curve).

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Pressed by adjusting the H_2O amount in the SiO₂ at low levels in addition to the control of the film density. Vacuum baking at 250°C was a promising method to realize this, for instance.

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Refernces

- H. Nagata and J. Ichikawa, "progress and problems in reliability of Ti:LiNbO₃ optical intensity modulators, "*Opt. Eng.* 34 (11), 3284-3293 (1995).
- H. Nagata, S. Oikawa, and M. Yamada, "Comments on fabrication parameters for reducing thermal drift on LiNbO3 optical modulator," *opt. Eng.* 36 (1), 283-286 (1997).
- H.Nagata, N. Mitsugi, J. Ichikawa, and J. Minowa, "Materials reliabilisy for high-speed lithium niobate modulators, "*Proc. SPIE* 3006, 301-313 (1997).

- H. Nagata, T. Kitanobou, K. Shima, and M. Shiroishi, "process control for a SiO₂ buffer layer of LiNbO₃ modulators to obtain reduced dc drift performance, "*Opt. Eng.* 36 (12), 3478-3480 (1997).
- H. Nagata, "Evaluation of hydroxyl content in commercial x-cut LiNbO3 wafer for optical waveguides devices, "*Opt. Eng.* 37 (5), 1612-1617 (1998).
- H. Nagata, J. Minowa, M. Yamada, J. Ichikawa, J. Nayyer, S. Shimotsu, J. Ogiwara, M. Kobayashi, H. Honda, K. Kiuchi, T. Shinriki, and T. Sakamoto, "optical waveguide modulator having a reduced d.c. drift" US Patent No. 5,526,448 (June 1996).
- M.Seino, T. Nakazawa, S. Taniguchi, and M. Doi, "Improvement of dc-dfirt characteristics in Ti:LiNbO3 modulator, "Technical Report of IEICE OCS95-66, OPE95-82 LQE95-76, pp. 55-60 (1995).
- H. Nagata, H. Takahashi, H. Takai, and T. Kougo, "Impurity evaluation of SiO₂ films formed on LiNbO₃ substrates, "*Jpn. J. Appl. Phys.* 34 (2A), 606-609 (1995).
- 9. H. Nagata, J. Ichikawa, M. Kobayashi, J. Hidaka, H. Honda, K. Kiuchi, and T. Sugamata, "Possibility of dc drift reduction of
- Ti:LiNbO:modulators via dry O2 annealing process, "Appl. Phys. Lett. 64 (10), 1180-1182 (1994).
- 10. Thin films and their application in optoelectronics, Optronics Editorial Office, Eds., 269-275 Optronics, Tokyo (1992).
- M. Seino, T. Nakzawa, Y. Kubota, M. Doi, T. Yamane, and H. Hakogi, "A low dc-drift TiLiNbO3 modulator assured over 15 Years," in *Proc. OFC'92*, pp. 325-328, Optical Society of America, Washington, DC (1992).