Optical and Physical Characterization of SiO_{2-x}–Al Thin-Film Polarizer on x-Cut $LiNbO_3$ Substrate

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***Abstract*–For the purpose of a mechanical evaluation of a metal-cladding polarizer, a precise characterization of SiO_{2-x}-Al thin-film succession fabricated on a $LiNbO_3$ substrate was made as well as an experimental optimization of the SiO_{2-x}-Al polar-izer for the Ti: $LiNbO_3$ waveguide. A 10-nm-thick SiO_{2-x}-Al was selected as the optimized underlay of a SiO_{2-x}-Al polarizer for the Ti: $LiNbO_3$ waveguide using a wavelength of λ = 1.55 μm. Results of scratch testing show that the adhesive strength of SiO_{2-x}-Al films was almost the same level as that of Ti-Au films on a thick SiO_2 layer, commonly used for metallic underlay of Au-plated electrodes. From observing SiO_{2-x}-Al film using a transmission electron microscope, it was confirmed that the 10-nm-thick SiO_{2-x} underlay stratified well without serious thickness fluctuation.**

***Index Terms*–Adhesive strength, metal-cladding polarizer, SiO_{2-x} underlay, thin-film structure, Ti : $LiNbO_3$ waveguide.**

I. INTRODUCTION

METAL-CLADDlNG structure on an optical waveguide is known as a TE-mode pass polarizer and is commercially applied to various optical devices [1]-[5]. In order to improve the TE/TM extinction ratio of the polarizer, a thin-film underlay of dielectric material, such as Y_2O_3, is generally inserted be-tween the optical waveguide and cladding metal [3]. In such a configuration, the TM mode interacts with a surface polariton supported by a metal surface through the evanescent field in the underlay and is absorbed into the clad-metal, whereas absorp-tion of the TE mode is negligibly small [2]-[4]. We have been attracted to the application of metal-cladding polarizer for an x-cut $LiNbO_3$ optical waveguide modulator because it is easy to integrate it on the modulator chip through the wafer process. In our investigation using A1 as cladding-metal and SiO_{2-x} as dielectric thin-film underlay, the highest extinction ratio was achieved when a 10-nm-thick SiO_{2-x} was inserted between the Al and Ti: $LiNbO_3$ Optical waveguide.

However, in the case of applying such thin-film succession to practical modulators, we worry about fluctuation of the extinction ratio caused by thickness variation of SiO_{2-x} and/or mechanical peeling of the polarizer caused by degradation of adhesive strength. Nevertheless, there is little investiga-tion concerning adhesive strength and the microstructure of metal-cladding polarizer, as well as its experimental optimiza-tion for the Ti : $LiNbO_3$ optical waveguide. For the purpose of mechanical evaluation of the metal-cladding polarizer, we tried to make a precise characterization of SiO_{2-x}-Al thin-film succession fabricated on a $LiNbO_3$ substrate. The results of scratch testing show that adhesive strength of SiO_{2-x}-Al films was almost the same level as that of Ti-Au films on a thick SiO_2 layer, commonly used for metallic underlay of Au-plated electrodes. From the observation by transmission electron microscope (TEM) of the SiO_{2-x}-Al film, we also confrrmed that the 10-nm-thick SiO_{2-x} underlay stratified well without serious thickness fluctuation.

Manuscript received October 6, 2000; revised March 30, 2001.

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Publisher Item Identifier S 0733-8724(0l)05312-9.

II. OPTICAL PERFORMANCE OF SiO_{2-x}-Al POLARJZER

At first, we engaged in experimental optirnization of the SiO_{2-x}-Al polarizer for the Ti : LiNbO3 optical waveguide to achieve higher extinction ratio and lower excess loss. The thickness of the SiO_{2-x} underlay and cladding length of the polarizer were selected as experimental parameters because they were critical ones as shown in the references about theoretical and experimental investigations on a metal-cladding polarizer [4]-[6].

To prepare a Ti : $LiNbO_3$ waveguide, photoresist patterns of a straight waveguide having a width of 7 μm were formed on an x-cut $LiNbO_3$ wafer by photolithography. A 90-nm-thick Ti layer was deposited on the wafer using electron beam evapora-tion, and Ti stripes were fabricated by the conventional lift-off technique. The wafer sample with Ti stripes was treated in the electric furnace at 1000 ˚C for 15 h in a wet O_2 atmosphere to diffuse Ti into the wafer. After the Ti-diffusion process, the wafer was diced into rectangular blocks with frve waveguides each, and the optical end-surface was polished. Before fabrica-tion of SiO_{2-x}-Al film, TE-mode insertion loss of the waveg-uides was measured at a wavelength of λ = 1.55 μm to know the initial waveguide loss without the polarizer. Then, wave-guide blocks were masked by a polyimide film having a tri-angular opening and the SiO_{2-x} and Al films were succes-sively sputter-deposited on the waveguides. In the sputtering process of SiO_{2-x}, only Ar gas was introduced in the sputtering chamber because oxygen-starved SiO_{2-x} seemed to be more ef-fective than SiO_{2-x} for achieving higher adhesive strength of the interfaces between $LiNbO_3$ and/or A1. According to the shape of the opening, cladding lengths of SiO_{2-x}-Al film to each wave-guide ranged from 0.25 to 3.5 mm. The thickness of SiO_{2-x} underlay was set to be 10 nm, 20 nm, 50 nm, and 0 nm as a reference, and that of A1 was fixed to be 100 nm.

After fabrication of SiO_{2-x}-Al film on waveguides, the in-sertion loss of the waveguides was measured at a wavelength of λ = 1.55 ftm in TE and TM modes. Results of measurement are shown in Figs. 1 and 2. The excess loss of the TE-mode,

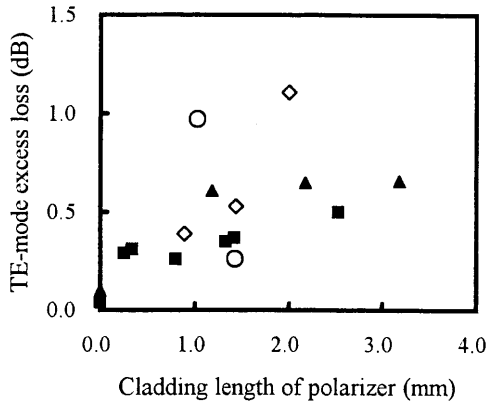

Fig. 1 . Influence of SiO_{2-x} thickness and cladding length on TE-mode excess loss of SiO_{2-x}-Al polarizer. Symbols shown in the graph are corresponding to the thickness of SiO_{2-x} underlay as 10 (■), 20 (▲), 50 (○), and 0 nm (◇), respectively.

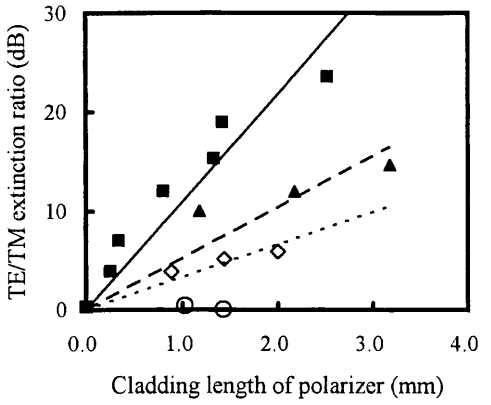

Fig. 2. Influence of SiO_{2-x} thickness and cladding length on the TE/TM extinction ratio of the SiO_{2-x}-Al polarizer. Symbols shown in the graph are corresponding: to the thickness of the SiO_{2-x} underlay as 10 (■), 20 (▲), 50 (○), and 0 nm (◇), respectively.

defined as subtraction of insertion loss values measured be-fore and after fabrication of the polarizer, seems to depend only on cladding length and no effect from thickness variation of SiO_{2-x} underlay is recognized as shown in Fig. 1. On the contrary, the TE/TM extinction ratio is influenced significantly by the thickness of the SiO_{2-x} underlay as well as cladding length as shown in Fig. 2. It is obvious that the 10-nm-thick SiO_{2-x} underlay was optimum to achieve the highest TE/TM extinction ratio. From these results, a thin-film succession of 10-nm-thick SiO_{2-x} and 100-nm-thick A1 was selected as the optimized SiO_{2-x} -Al polarizer for a Ti : $LiNbO_3$ waveguide using a wavelength of $\lambda = 1.55$ pam.

III. Mechanical Performance of SiO_{2-x} -Al Thin Film

A. Fabrication of Test Samples

In the process of integrating the SiO_{2-x} -Al polarizer on a high-speed x-cut $LiNbO_3$ optical modulator, two fabrication procedures are applicable for cladding a SiO_{2-x} -Al polarizer on a waveguide. They are different methods to make a window in a thick SiO_2 buffer layer which is formed on a waveguide to achieve velocity-matching between lightwave and microwave for high-speed modulation. One is an "aperture method" in which SiO_2 deposition on a $LiNbO_3$ wafer is partly prevented by a physical mask employed during buffer layer fabrication process. The other is a dry-etching method in which a conventional plasma etching technique using fluorocarbon gas is employed in a partial removal of the SiO_2 buffer layer. Although the latter is superior to the former in precise productivity and reproducibility, there is a problem in that the plasma etching process using fluorocarbon gas causes chemical deterioration of Al film as we have already noted [7]. As a result of optimizing the dry-etching process, we found that the Ar-plasma treatment was effective in preventing chemical deterioration of Al film. In order to simulate these actual fabrication processes, four types of samples were prepared for the characterization of the SiO_{2-x}-Al thin films. The fabrication processes of test samples are schematically summarized in Fig. 3. The samples referred to as Types I and II are corresponding to the samples fabricated by the process of the "aperture method" and that of the dry-etching method, respectively. Sample Type I-A was fabricated by sputter deposition of SiO_{2-x}-A1-SiO_{2-x} films having thickness of 10, 100, and 100 nm, respectively, on the entire surface of an x-cut $LiNbO_3$ wafer without waveguide. The purpose of forming the upper 100-nm-thick SiO_{2-x} on Al was for protection of the A1 film from chemical attack in the following wafer process. Sample Type I-B was fabricated using almost the same procedure as Type I-A except that the former did not have the 10-nm-thick SiO_{2-x} underlay to evaluate the effect of its ab-sence, as a reference to Type I-A. On the other hand, the fab-rication process of sample Types II-A and II-B was different from that of Type I in wafer treatment before deposition of SiO_{2-x}-Al-SiO_{2-x} films. To sirnulate the surface condition after the plasma etching process, a virgin x-cut $LiNbO_3$ wafer was exposed to Ar gas plasma for 10 min using an electron cyclotron resonance (ECR) plasma etching apparatus and subsequently annealed at 600 °C for 5 h in an O_2 atmosphere to compensate for the O_2 vacancy caused by the Ar-plasma attack. After these treatments, the SiO_{2-x}-A1-SiO_{2-x} films for Type II-A and A1-SiO_{2-x} films for Type II-B were sputter-deposited on the wafers in the same manner as samples Types I-A and I-B, respectively.

After fabrication of SiO_{2-x}-Al-SiO_{2-x} films and SiO_{2-x} films, metallic films consisting of a 50-nm-thick Ti and a 35-nm-thick Au were successively deposited on the surface of the upper SiO_{2-x} film by vacuum evaporation. For the purpose of simulating the chemical etching process applied in the actual electrode-forming process, metallic films were chemically removed using an alcoholic solution of I_2 and KI for Au and a mixture of NH_4OH and H_2O_2 for Ti. After fabrication, the wafer samples were diced into small pieces and used for evaluation of adhesive strength and structural analysis.

B. Adhesive Strength of Films

For the purpose of mechanical evaluation of a metal-cladding polarizer, adhesive strengths of the SiO_{2-x}-Al-SiO_{2-x} and Al-SiO_{2-x} films were examined in as-deposited, high-temperature stored, and heat-cycled samples. At first, small pieces

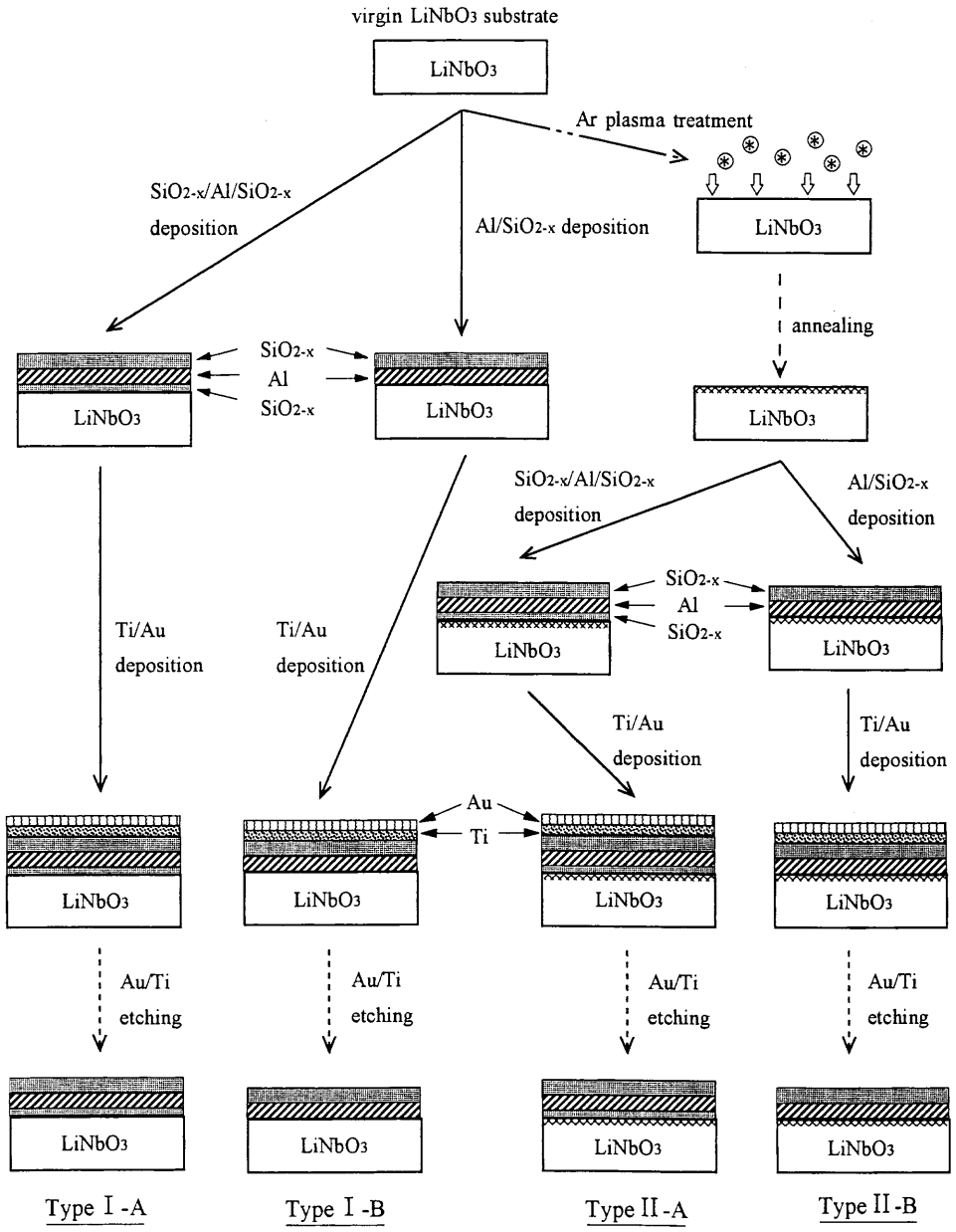

Fig. 3. Schematic diagram showing fabrication processes of test samples.

of Type I and II samples were divided into three groups. The first group was of as-deposited samples, which were stored in ordinary atmosphere until the adhesive strength test. The second group, as samples of high-temperature storage, was kept at 100 ˚C for 100 h without intentional humidity control. A heat-cycle of -50 ˚C to 100 ˚C was given 100 times to the third group.

The adhesive strength of each sample was measured by a scratch tester consisting of an inclined stage for attaching the sample and pressure sensor. The sensor had the same structure as an audio-cartridge, in which a diamond stylus, pressed onto the sample, traced the surface while being vibrated perpendicularly to the tracing direction. The magnitude of frictional force between the stylus and the sample was picked up as the amplitude of electric signal and the applied load to the sample Was increased gradually by moving the inclined sample toward the stylus. When the applied load reached critical point, vibration generated by destruction was detected as a scratch noise. In actual measurement, the adhesive strengths were estimated from the applied load at the abrupt destruction point of the substrate because peeling in the interfaces between SiO_{2-x}-Al-SiO_{2-x} or Al-SiO_{2-x} films and $LiNbO_3$ substrates were not clearly identified in all the samples.

Results of measurernents are shown in Fig. 4. The adhesive strength was highest in sample Type I-A with SiO_{2-x}-A1-SiO_{2-x} films, although it tended to be lower in the samples of high-temperature stored and heat-cycled than that of as-deposited. The adhesive strengths of the remaining

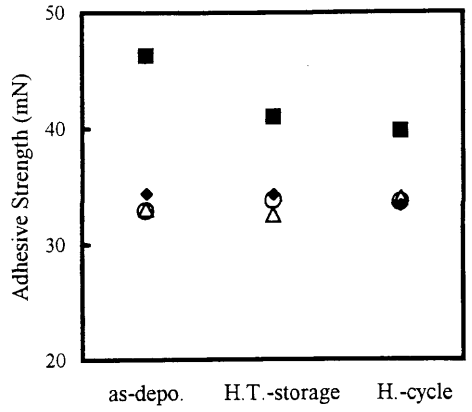

Fig. 4. Scratch test results of the SiO_{2-x}-Al-SiO_{2-x} and Al-SiO_{2-x} films fabricated on $LiNbO_3$ substrate. Symbols shown in the graph correspond to the samples of Type I-A (■), Type I-B (◆). Type II-A (△), and Type II-B (○), respectively.

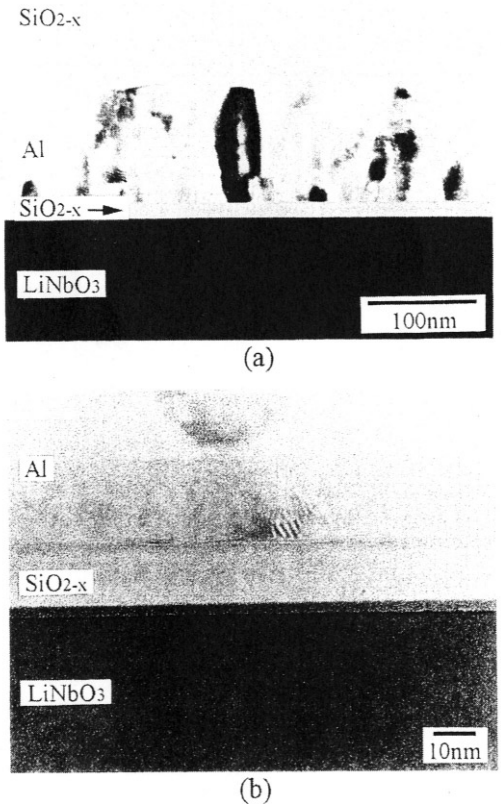

Fig. 5. Cross-sectional TEM ima_ges of the sample Type I-A fabricated by "aperture method" showing (a) stratified structure of SiO_{2-x}-Al-SiO_{2-x} films on the $LiNbO_3$ substrate and (b) magnifled image around the SiO_{2-x} underlay.

three samples, i.e., Types I-B, II-A, and II-B, were almost the same level and did not show degradation in adhesive strength by exposure to heat-stressed conditions such as high-temperature storage and heat-cycle. These results suggest that the existence of the very thin underlay would not be a defect causing mechanical peeling of the polarizer. In addition, the

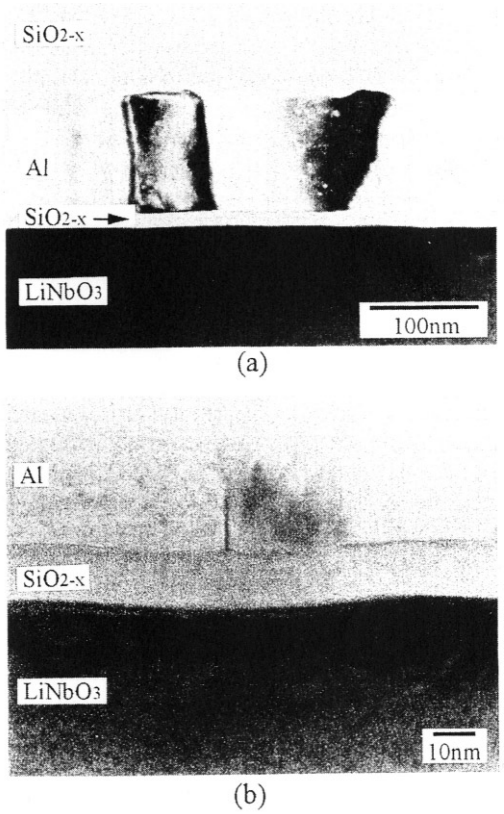

Fig. 6. Cross-sectional TEM images of the sample Type II-A fabricated by dry-etching method showing (a) stratified structure of SiO_{2-x}-Al-SiO_{2-x} films on the $LiNbO_3$ Substrate and (b) magnified image around the SiO_{2-x} underlay.

adhesive strength of evaporated Ti-Au films on thick SiO_2 layer measured by the same scratch tester was almost the same level (32~46 mN) as that of SiO_{2-x}-Al films. Considering that the Ti-Au films are commonly used for metallic underlay of Au-plated electrodes, we conclude that SiO_{2-x}-Al films are reliable enough for application to practical modulators.

C. TEM Analysis of Films

Structural analysis of SiO_{2-x}-A1-SiO_{2-x} films was performed by a cross-sectional transmission electron microscope (TEM) observation on the samples of Types I-A and II-A. Figs. 5 and 6 are cross-sectional TEM images of an as-deposited sample corresponding to Types I-A and II-A samples, respectively. According to these TEM images, it was confirmed that the 10-nm-thick SiO_{2-x} underlay of both samples stratified well without serious thickness fluctuation. although actual thickness of SiO_{2-x} underlay was much thicker than planned. In the bottom of the SiO_{2-x} underlay, a very thin dark-contrasted layer was observed. To characterize this thin layer, components of the dark and bright contrasted layer in SiO_{2-x} underlay were examined by energy dispersive X-ray spectroscopy (EDX) performed observing the guidelines of TEM. Data from TEM-EDX, shown in Fig. 7, suggest that this thin layer might be an intermediate phase between $LiNbO_3$ and

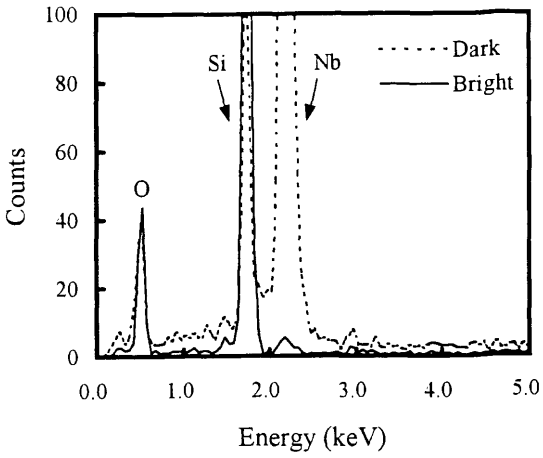

Fig. 7. TEM-EDX spectra of the dark and bright contrasted layer in the SiO_{2-x} underlay of the sample Type I-A.

SiO_{2-x} . On the other hand, Al film of both samples showed a columnar-like structure and no amorphous phase was identified. Electron-diffraction patterns of Al film could fit to any one of the indexes of Al. From these results, it is concluded that A1 film deposited on a SiO_{2-x} underlay had a polycrystalline nature and the growth of amorphous compounds, as we reported in our previous paper [7], did not occur in both samples.

In the sample Type II-A, a slight undulation derived from Ar-plasma treatment of the $LiNbO_3$ Substrate was observed in both the interface of $LiNbO_3$-SiO_{2-x} and SiO_{2-x}-AI and the lattice of the $LiNbO_3$ crystal seerned to be deformed in the surface area of the $LiNbO_3$ Substrate. The interface between $LiNbO_3$ and SiO_{2-x} underlay was smoother than that between $LiNbO_3$ and Al film deposited on CF4-etched surface [7]. The difference in these interfaces might be derived from that of gas species in plasma because Ar-plasma is chemically inactive and does not generate a precipitation such as LiF and fluorocarbon polymers.

Besides these samples, the high-temperature stored sample of Type I-A was also observed so as to examine the cause of lowering adhesive strength by heat-stressed history. From the TEM image shown in Fig. 8, no defect identified as the cause of the lowering adhesive strength was observed, except that the thickness of the SiO_{2-x} underlay seemed to be slightly decreased. The reduction of underlay thickness may suggest that the underlay was densified by an annealing effect from high-ternperature storage. In general, the densification causes an increase of internal stress in thin film generated by reduction of its volume. Thus, we consider that the increase of internal stress derived from the heat-stressed history might be one of the causes of the lowering of the adhesive strength of SiO_{2-x}-Al-SiO_{2-x} films to the $LiNbO_3$ Substrate. However, this speculation seems to be not consistent with the fact that the adhesive strength of the films fabricated by a dry-etching method did not show the degradation of adhesive strength by exposure to the heat-stressed conditions as mentioned in the previous section. Therefore, the cause of the lowering of the adhesive strength by heat-stressed history is obscured and further investigation is required to determine it.

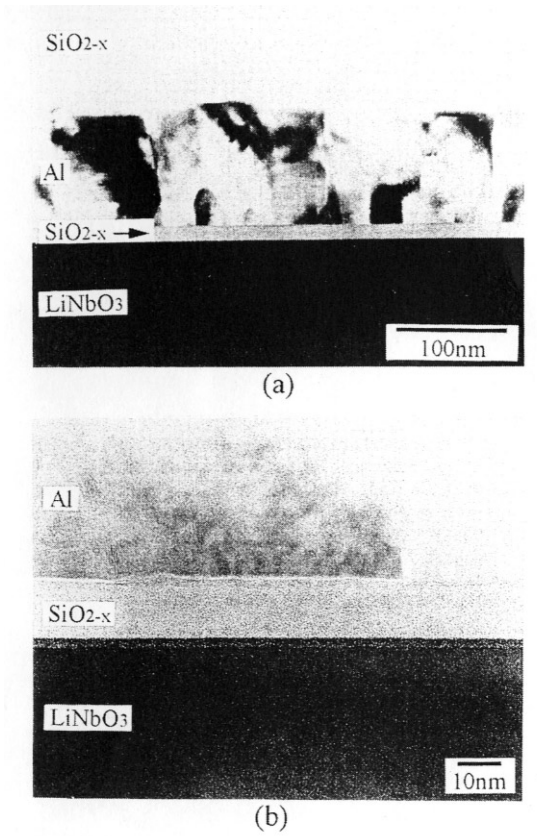

Fig. 8, Cross-sectional TEM images of the sample Type I-A after high-temperature storage showing (a) stratified structure of SiO_{2-x}-Al-SiO_{2-x} films on LiNbO3 substrate and (b) magnified image around the SiO_{2-x} underlay.

IV. SUMMARY

For the mechanical evaluation of a metal-cladding polarizer, a precise characterization of SiO_{2-x}-Al thin-film succession fabricated on the $LiNbO_3$ substrate was made, as well as experimental optimization of SiO_{2-x}-Al polarizer for the Ti : $LiNbO_3$ waveguide. Thin-film succession of the 10-nm-thick SiO_{2-x} and 100-nm-thick Al was selected as the optimized SiO_{2-x}-Al polarizer for Ti : LiNbO3 waveguide at a wavelength of λ = 1.55 μm. Results of scratch tests of as-deposited, high-temperature stored, and heat-cycled samples show that adhesive strength of SiO_{2-x}-AI film was higher in the sample fabricated by the "aperture method" than that of the dry-etching method, and their values were almost the same level as that of evaporated Ti-Au films on the thick SiO_2 layer. From the observation of the TEM of the SiO_{2-x}-Al film, it was conflrmed that the 10-nm-thick SiO_{2-x} underlay stratified well without serious thickness fluctuation.

ACKNOWLEDGMENT

The authors would like to thank the staff of NSG Techno-Research Co., Ltd., for scratch tests and the Foundation for Promotion of Material Science and Technology of Japan for TEM

observations. They also are grateful to the staff of the optoelectronics research division and the optoelectronlcs division of Sumitomo Osaka Cement Co., Ltd., for their help in fabricating the samples.

REFERENCES

[1] Y. Suemcrtsu, M. Hakuta, K. Furuya, K. Chiba, and R. Hasumi, "Fundamentill transverse electric field (TE_0) mode selection for thin-film asymmetric light guides." *Appl. Phys. Lett*., vol. 21 , no. 6. pp. 291-293, 1972

[2] J. Polky and G. Mitchell, "Metal-clad planar dielectric waveguide for integrated optics." *J. Opt. Soc. Amer. A*, vol. 64, no. 3. pp. 274-279, 1974.

[3] F. Tian, C. Harizi, H. Herrmann, R. Ricken, U. Rust, W. Sohler, F. Wehrmann, and S. Westenhofer, "Polarization-independent integrated optical acoustically tunable double-stage wavelength filter in $LiNbO_3$," *J. Lightwave Technol*., vol. 12, pp. 1192-1197, July 1994.

[4] M. Sletten and S. Seshadri. "Thick metal surface-polariton polarizer for a planar optical waveguide." *J. Opt. Soc. Amer. A*, vol. 7, no. 7. pp. 1174-1184. 1990.

[5] ——, "Experimental investigation of a thin-film surface polariton polarizer," *J. Appl. Phys*., vol. 70, no, 2. pp. 574~579, 1991 .

[6] C. Chen and L. Wang, "Design of finite-length metal-clad optical waveguide polarizer," *IEEE J. Quantum. Electron*., vol. 34, pp, 1089-l097, July 1998.

[7] H. Nagata, Y. Miyama, N. Mitsugi, and K. Shima, "Chemical deterioration of Al film prepared on CF_4 plasma-etched $LiNbO_3$ surface," *J. Mater Res.*, vol, 15. no. 2, pp. 476-482, 2000.

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