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Experimental analysis of lithium niobate CMP for room temperature bonding

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Lithium niobate (LN, $LiNbO_3$) is a type of artificial crystal exhibition piezoelectricity, pyroelectric and ferroelectricity, which has been widely used in electronic components. To use in electronic components, a LN wafer has to be bonded to a Si substrate. Bonding of Si and LN allows for ease of micromachining a high quality electrically-sensitive thin film, as well as creating mechanically-rigid diaphragms. For this reason, Si combined with LN is desirable for microelectromechanical system (MEMS) devices such as a pressure sensors, microfluidic devices and optical data storage systems. However, the large difference in thermal expansion coefficient between Si and LN causes serious thermal stresses during the thermal-pressure bonding process which has generally been used. Therefore room temperature bonding would be the best candidate for making a strong and stress-free interface between Si and LN. Room temperature bonding requires a lower surface roughness and lower defect contact on the LN wafer surface than thermal bonding does. A chemical mechanical polishing (CMP) process produces a thin LN wafer with a high quality surface suited for room temperature bonding and with a suitale thickness that affects the sensitivity of the devices. Here LN wafers were polished using a colloidal silica slurry, resulting in a high material removal rate (MRR) and a fine surface quality under a condition of low pH, high abrasive concentration and low flow rate. The polishing mechanism of LN was investigated by mechanical, chemical and thermal analysis.

Key words : Lithium niobate, CMP, Room temperature bonding

Introduction

Lithium niobate (LN) has been widely used as a material inthe fabrication of modulators for optoelectronic devices and MEMS devices because of its high electro-optic coefficients, piezoelectricity, pyroelectricity and ferroelectricity. Its high Curie temperature (1,100-1,180 °C) allows the fabrication of low-loss optical waveguides through the diffusion of metals, by ion exchange or proton exchange [1-4]. A very small index change in the crystal is induced using these methods in waveguide fabrication. These waveguides are used in high-speed electro-optical modulators or in nonlinear wave mixing devices such as periodically poled lithium niobate waveguide parametric oscillators and amplifiers [5]. LN is also thermally, chemically and physically stable and compatible with conventional integrated-circuit processing technology. LN has to be bonded to Si substrates to apply in devices such as pressure sensors, microfluidic devices and data storage systems. [6, 7] However, the large difference in the thermal expansion coefficient between the LN wafer and substrate causes serious thermal stress during the thermal bonding process. The thermal expansion coefficient of Si is 2.6*10-6/K at room temperature, while that of LN is $7.5*10^{-6}$/K [8]. Because the thermal coefficient of LN is at least three times than that of Si, thermal stress will occur at the bonded interface and will separate the bonded interface. The mismatch of the thermal expansion coefficient is solved by using a room temperature surface activated bonding (SAB) process. For the SAB process, a high surface quality of the LN wafer is needed. A rough surface and defects induce a weak adhesive force between the LN wafer and substrate [9]. The practical limit for making more sensitive detectors is dependent on the ability to make thinner plates, because the current generated by a pryoelectric detector is inversely proportional to the plate thickness. [10-13]. To obtain a strong adhesive force between the LN wafer and substrate and more sensitive detectors, a chemical mechanical polishing (CMP) process, which has been widely applied to the wafer planarization process and the wiring process, was used [14]. In the CMP process, the wafer surface moves across a polishing pad under pressure in the presence of a slurry. As shown in Fig. 1, mechanical motion and a downward force are imparted to the wafer by the polishing head. The polishing pad surface provides the rough points or asperities, which come in contact with the wafer. The slurry provides the abrasive particles and the appropriate chemistry for the CMP process to proceed [15]. In this paper, the polishing mechanism of LN is discussed based on the results of an integrated analysis which considering mechanical, chemical and thermal factors.

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Experimental

To analyze the LN CMP mechanism according to

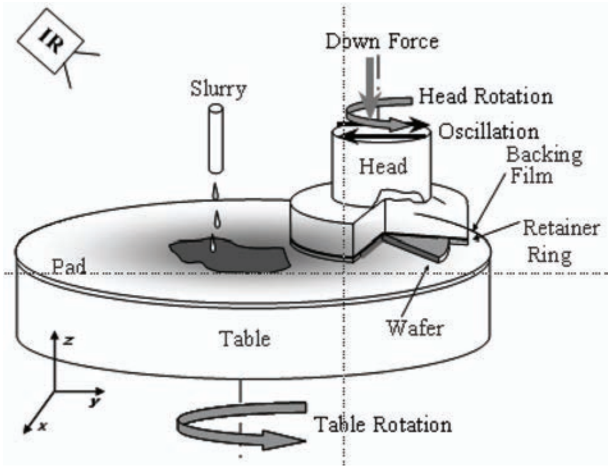

Fig. 1. Schematic of CMP system.

chemical, mechanical and thermal factors, the variables shown in table 1 were fixed, except for the slurry pH, the abrasive concentration, and the slurry flow rate. Research and development CMP equipment, POLI-400 (G&P Technology Co.), was used in the experiments. The slurry was formulated by adding colloidal silica abrasive (average ~120 nm diameter, pH 10.7, Ace Hitech Co.) and a foamed cross-linked polymer pad, IC 1400 k-groove (Rohm & Hass Corp.), was used. The MRR was achieved by using the thickness data converted from the weights measured by a precision scale. The surface roughness (Ra) was measured by a Nano-view (Nano system Co.) system using white-light scanning interferometry. The variation of slurry temperature was obtained by an IR camera at the exit point of the slurry every 2 minute. The reaction of the wafer surface was examined by XPS (X-ray photoelectron spectroscopy), and the hardness of the wafer surface was measured by a nano-indenter XP (MTS Co.). Conditions used in these experiments are shown in table 1.

Results and Discussion

Slurry pH

To find the chemical effect on the MRR and surface roughness, slurries were made with different pH. The slurry was a colloidal silica abrasive (20 wt%) based on KOH. H_3PO_4, and HNO_3 was added to adjust the pH of the slurry (pH 3 and 7) from the standard pH 10.7 slurry. The other conditions were the same. Figs. 2 (a) and (b) show the MRR and surface roughness according to the pH, H_3PO_4 and HNO_3. The MRR increased with the lower slurry pH in both cases with H_3PO_4 and HNO_3. HNO_3 had a stronger effect on the MRR than H_3PO_4. High quality surfaces (Ra < 2 nm) were obtained with pH 3 and 7. These results are suitable for the room temperature bonding process.

Table 1. Experimental condition

Pressure	400 [g/cm^2]
Velocity	Head and Platen : 60 [rpm]
Pad	IC 1400^{TM} (k-groove)
Slurry pH	3, 7, 10.7
Slurry flow rate	150 [cc/minute]
Wafer	4 [inch] $LiNbO_3$ wafer(128RY-cut)
Temperature	23 [°C]
Conditioning	Diamond conditioner, Ex-situ (1minute)
Surface roughness [Ra]	Measured when 7, 17, 27, 37, 47 [minute] passed after polishing starts

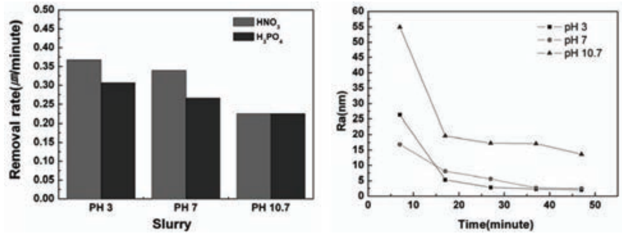

Fig. 2. (a) Removal rate according to the slurry pH, H_3PO_4 and HNO_3. (b) Roughness of surface according to pH 3, 7, 10.7 as a function of time.

Abrasive concentration

To evaluate the mechanical effect on the MRR, the abrasive concentration was changed from 5 wt% to 20 wt% (increments of 5 wt%). The pH of slurry was fixed at 3 and the other conditions were the same. The effect of the abrasive concentration on the MRR is shown Fig. 3. The higher the abrasive concentration induced the higher MRR. It is thought that the number of abrasives depends on the frequency of wear from mechanical abrasion. The same result was found in different material CMP processes such as a metal CMP and silicon oxide CMP. This result showed that the LN removal rate was largely affected by the mechanical action.

Flow rate and temperature

The slurry, which is transported to the polishing zone, helps the abrasives disperse uniformly across the platen. The flow rate of the slurry is closely related with the

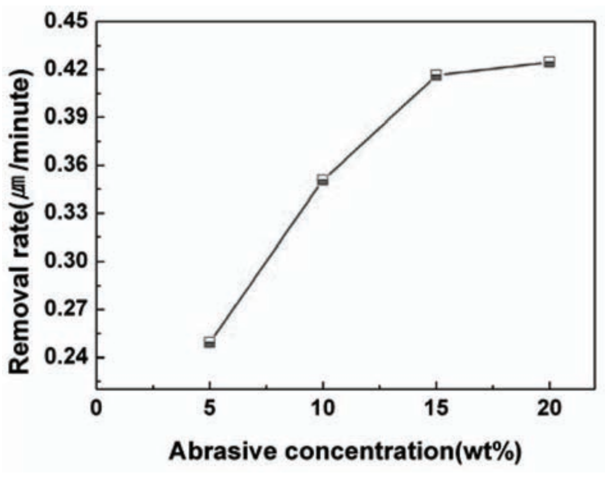

Fig. 3. Change of removal rate according to the abrasive concentration.

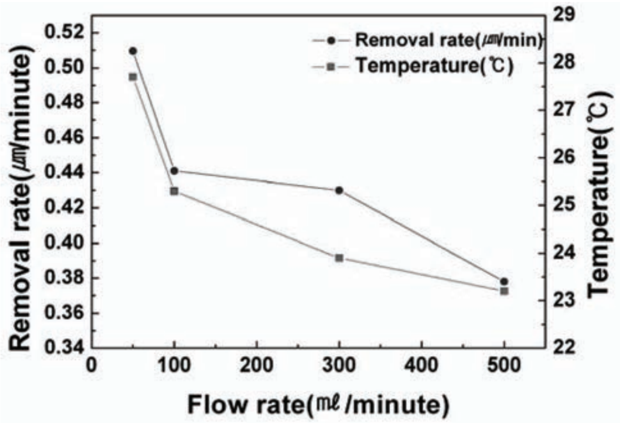

Fig. 4. Changes of removal rate and temperature in the exit-point according to the slurry flow rate.

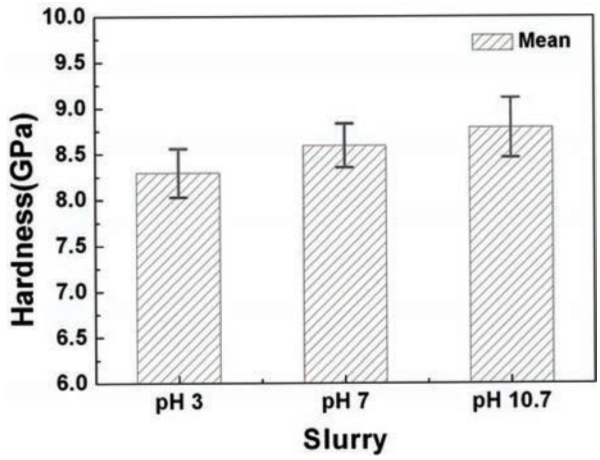

Fig. 6. Hardness variation with slurries.

temperature of the slurry on the platen. The cooling effect of the slurry is very important in CMP. The removal rate is proportional to the temperature of the slurry because of the increase of chemical activity [16]. The flow rate was controlled from 50 cc/minute to 500 cc/minute (50, 100, 300, 500 cc/minute) in the experiment. The exit slurry temperature was measured by an IR camera 1 minute after the start of polishing. Fig. 4 shows the change of the removal rate according to the slurry flow rate. As the flow rate decreased, the removal rate increased. It is thought that the lower flow rate reduced the cooling effect of the slurry and increased the chemical activity at higher temperatures. This result explains the thermo-chemical reaction effects on the LN CMP.

Analysis

The LN wafers polished at different pHs may have different reaction layers on the surface. The reaction layer protects the mechanical abrasion or can be easily removed by abrasion. It is important to know the chemical modification and the hardness of the polished surface. Information about the atomic composition of the surface was obtained by XPS. Fig. 5 shows the chemical effects obtained from the XPS study using the Nb3d spectra lineshape. The intensity peaked

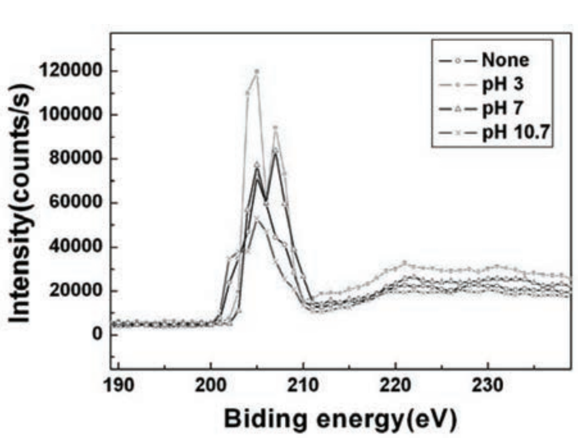

Fig. 5. XPS results with pH.

at 205 eV. According to the database for XPS, the binding energy of NbO_2 is 205.5 eV, which is very close to the peak mentioned above. A new intensity peak was induced at 207 eV at pH 3 and 7. There were no peaks at pH 10.7 and no polished wafer. According to the database fir XPS, the binding energy of Nb_2O_5 is at 207.2 eV, which was very close to the new peak mentioned above. The pH 3 and 7 slurries had high MRR relatively in the previous experiments. It was thought that the MRR was related to the difference of the heights of the peaks. Ultimately, NbO_2 and Nb_2O_5 were closely related with the LN CMP process.

Fig. 6 shows the variation of hardness of the LN with pH. The reaction layers produced different hardness of the surface. All the hardness values were obtained using the CSM mode and considering the indentation size effect (ISE). The MRR was inversely proportional to the hardness. Eq. 1 is the wear rate equation for a non-metallic substance. The hardness result matches with Eq.1.

$$W = kL/H \text{(W : wear rate, K : coefficients, L : load, H : hardness) [17-19]} \quad (1)$$

Conclusions

(1) The LN wafer could be polished to a high surface quality (Ra ~2 nm) under a low pH CMP condition, which is suitable for room temperature bonding.

(2) A high removal rate could be obtained with the combined conditions of low pH slurry and a high abrasive concentration. Therefore, chemical and mechanical energy affect the LN CMP.

(3) A low flow rate increased the MRR because it induced frictional heat, the thermal energy, and increased the chemical activation energy. It was thought that the chemical effects acted on the LN CMP.

(4) The reaction layer formed on the LN surface was investigated by XPS and nano-indenter analysis, which showed that NbO_2 and Nb_20_5 were closely related with the process.

(5) The reaction layer showed a variation of hardness

with NbO_2 and Nb_2O_5, and changed the MRR.

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