

# The Effect of Slurry pH and Particle Size on LiTaO<sub>3</sub> Polishing

Kazuki Moriyama, Akira Ozeki, Shinichi Haba, Matsunori Mori  
Nitta Haas Incorporated  
3-17-1, Kannabidai, Kyotanabe-Shi, Kyoto, 610-0333, Japan  
kmoriyama@nittahaas.co.jp

*Abstract –The demand of LiTaO<sub>3</sub> (LT) wafer mainly used as substrate of SAW (Surface Acoustic Wave) filter device has been quite increasing in recent years. From the viewpoint of LT wafer's productivity in the polishing process, high removal rate and good recycle stability are required for slurry to eliminate generated damages in the previous process. In this study, the effect of pH and particle size in the slurry on the removal rate and the polishing stability of LT wafer were investigated. We found out that acid slurry containing large particle can achieve high removal rate with good recycle stability. The balance of pH and particle size would be taken advantage of polishing other hard brittle or chemically stable materials such as sapphire, silicon carbide and gallium compounds.*

## INTRODUCTION

LT is one of the ceramics materials mainly used as the substrate of SAW filter device that can distinguish an electric signal of specific frequency. In recent years, in addition to the market growth of smartphones, demand is rapidly expanding due to the large increase in the number of devices mounted on each smartphone because of high speed and large capacity of transmission. Under such circumstances, improvement of productivity is desired. LT wafer should be polished as well as typical substrates like silicon, sapphire and silicon carbide in order to eliminate the damage occurred at previous process. The features of the LT wafer are different from others, for example, thinner thickness, brittleness and high chemical stability.<sup>[1]</sup> As these features, polishing condition of LT wafer is generally so mild that removal rate can not be raised easily. One of the key factors to improve the removal rate effectively is polishing slurry. Typical one is composed of a normal colloidal silica with a pH adjuster,<sup>[2]</sup> but it can't realize a sufficient polishing performance with respect to both a high removal rate and good stability. In this study, the effect of pH and particle size in the slurry on the removal rate and the polishing stability of LT wafer were investigated, and we propose a suitable area that can keep the balance of pH and particle size with respect to high removal rate with good stability.

## EXPERIMENTS

The effect of slurry pH on removal rate was evaluated by using slurries containing 20wt% silica abrasives. Mean particle size was 122nm and slurry pH was controlled by hydrochloric acid and potassium hydroxide from 3 to 9. Removal rate was calculated by weight loss of 15 minutes polishing. Polishing conditions are shown in Table 1. The effect of particle size on

Table 1. Polishing conditions for pH dependence test.

Polisher	12inch single side polisher
LiTaO <sub>3</sub> wafer	4inch 42° Y-X Black Yellow LiTaO <sub>3</sub>
Pad	SUBA800™ (XY-Groove)
Slurry	Colloidal Silica Slurry
Pressure	3psi
Table/Head Speed	120rpm/120rpm
Polishing time	15min
Slurry flow rate	400mL/min
Recycle amount of slurry	1500mL

Table 2. Polishing conditions for slurry recycle test.

Polisher	12inch single side polisher
LiTaO <sub>3</sub> wafer	4inch 42° Y-X Black Yellow LiTaO <sub>3</sub>
Pad	SUBA800™ (XY-Groove)
Slurry	Colloidal Silica Slurries
Pressure	3psi
Table/Head Speed	120rpm/120rpm
Polishing time	30min
Slurry flow rate	400mL/min
Recycle amount of slurry	1500mL

removal rate and recycle stability was evaluated by using three different sizes of silica abrasives. Mean particle sizes were 36nm, 90nm and 122nm respectively. Abrasive concentration of all slurries was prepared to 20wt% and pH was controlled at 4 by hydrochloric acid and potassium hydroxide. The 30 minutes polishing was repeated 4 times to evaluate recycle stability for each slurry. Polishing conditions are shown in Table 2.

## RESULTS AND DISCUSSION

Fig. 1 shows the effect of slurry pH on removal rate. Though the removal rate in alkali atmosphere was very low, it increased gradually with the decrease of slurry pH, and became maximal at pH4. Fig. 2 shows the zeta potential titration curves of LT wafer and silica abrasives. LT wafer is charged negatively in pH 6.2 or more and positively in pH 6.2 or less. Silica abrasive is charged negatively in the range of pH 3 to 9. Fig. 3 shows the image of relationship between LT wafer and silica abrasives in acid and alkali atmospheres. LT wafer and silica abrasives repulsed each other because both of them are charged negatively in alkali atmosphere. It is thought that the repulsion

between LT wafer and silica abrasives caused weak mechanical polishing and lower removal rate. On the other hand, LT wafer and silica abrasives are attracted each other because LT wafer is charged positively and silica abrasives are charged negatively in acid atmosphere. It is thought that the attraction between LT and silica abrasives caused strong mechanical polishing and higher removal rate.

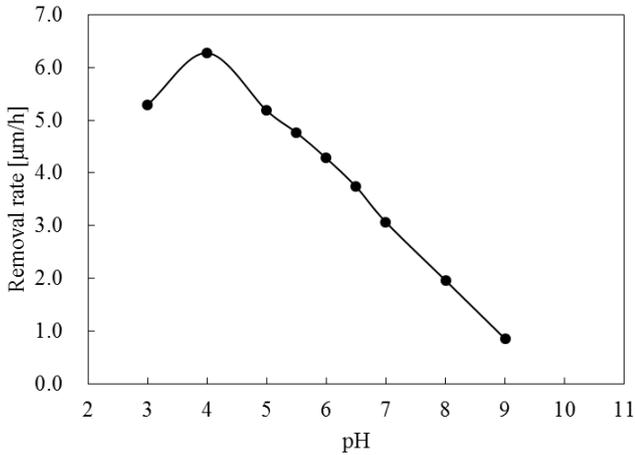


Fig. 1. Effect of slurry pH on removal rate of LiTaO<sub>3</sub> wafer.

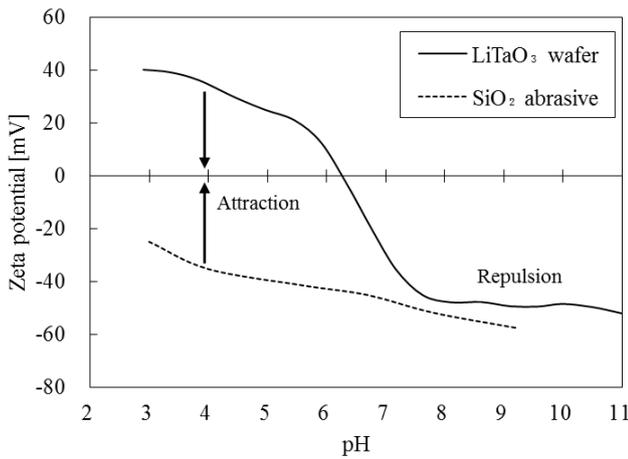


Fig. 2. Zeta potential titration curves of LiTaO<sub>3</sub> and silica abrasive.

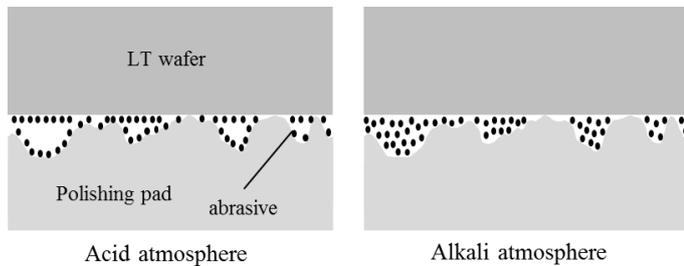


Fig. 3. Relationship between LT wafer and silica abrasives at acid and alkali atmospheres.

The effect of particle size on recycle stability in acid slurry was evaluated by using three different sizes of silica abrasives as shown in Fig. 4.

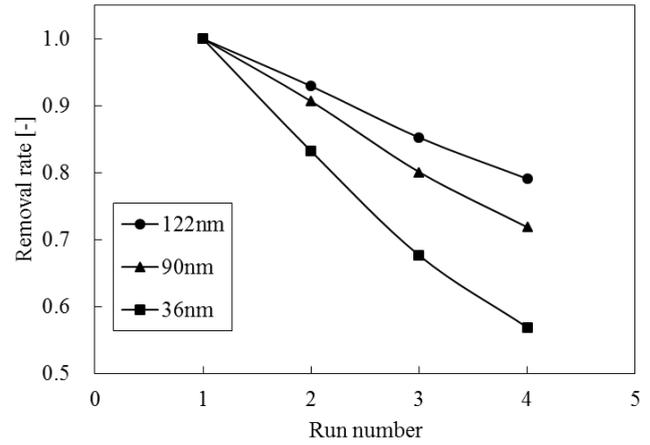


Fig. 4. Removal rate reduction with various particle sizes.

The recycle stability of slurry containing larger particle is better than smaller one. Fig. 5 shows the particle size increase of the abrasives after 4 runs of 30 minutes polishing to the particle size before polishing.

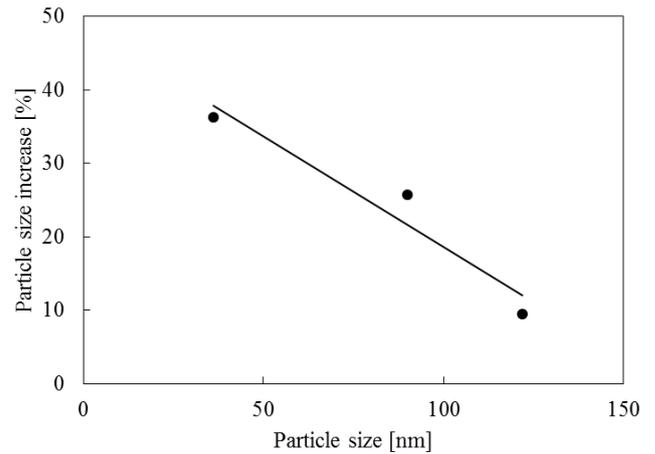


Fig. 5. Particle size increase after 4 runs of polishing to the particle size before polishing.

It is obvious that the larger particle aggregate hardly compared to smaller one though all particles are aggregated after polishing regardless of particle size. The reason causing aggregation of particles by polishing is thought that the concentration of positively charged contaminant generated by polishing of LT wafer increases gradually during polishing. As a result, positive ions as counter ions surrounding the surface of abrasive charged negatively are pushed to the surface of the abrasive. In other words, abrasives are attracted each other within intermolecular force working range without repulsion between counter ions because the electric double layer thickness becomes thinner with the increase of the concentration of the positively charged contaminant in the slurry as shown in Fig. 6.<sup>[3][4]</sup>

## SUMMARY

The Polishing performance of LT slurry was evaluated from the point of view of particle size and slurry pH to achieve high removal rate and superior recycle stability simultaneously. It is revealed that acid slurry achieves high removal rate compared with alkali slurry and large particle is superior to small one regarding recycle stability in acid atmosphere. With increasing demands for improvement of productivity in the polishing process, it is important to achieve both high removal rate and superior recycle stability. The technique shown in this research would be taken advantage of polishing other hard brittle or chemically stable materials such as sapphire, silicon carbide and gallium compounds.

## REFERENCES

- [1]S. Koga, T. Doi, S. Kurokawa, O. Ohnishi, M. Uneda and Y. Matsukawa "Polishing Mechanism on Oxide Optical CrystalCMP" The Japan Society For Precision Engineering, Autumn 2011. pp.171-172.
- [2]K. Tamai, A. Yasui, M. Serikawa, H. Morinaga, T. Doi, S. Kurokawa, O. Ohnishi and M. Uneda: "Analysis of Polishing Friction Factor for Material Removal Rate Improvement in CMP" Journal of the Japan Society of Advanced Production Technology Vol.29 (1) (2011) pp.53-58.
- [3]C. Takai, M. Fuji, M. Takahashi: "Control and characterization of nanoparticle dispersability in aqueous and non-aqueous slurries" Annual report of the Ceramics Research Laboratory Nagoya Institute of Technology, 6:32-29
- [4]J. Brant, H. Lecoanet, M. R. Wiesner: "Aggregation and deposition characteristics of fullerene nanoparticles in aqueous systems" Journal of Nanoparticle Research (2005) 7 :545-553.
- [5]H. Kamiya, M. IJIMA "Dispersion Behavior Control of Nanoparticles and its Applications" The MICROMERITICS No.55 2012 pp.12-18.

## AUTHOR BIOGRAPHY

**Kazuki Moriyama**, is in charge of the development of polishing slurry at R&D department, who is focusing on new application fields such as  $\text{LiNbO}_3$ , gallium compounds and resin materials.

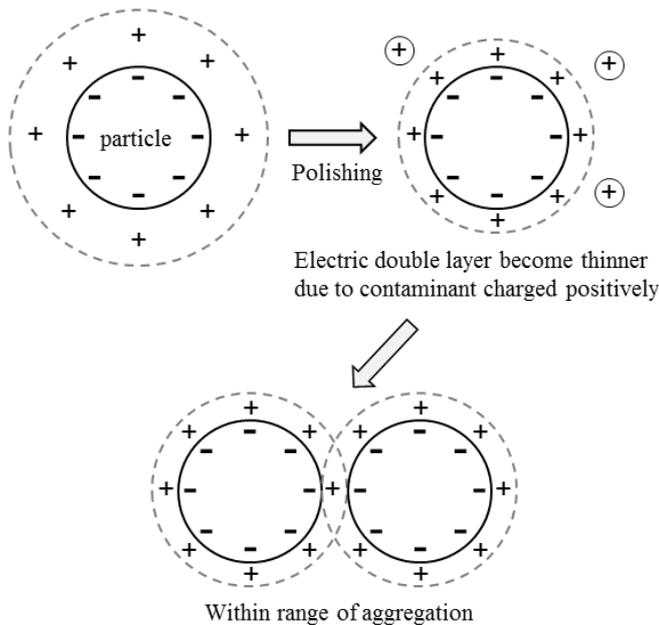


Fig. 6. Image of aggregation mechanism by polishing.

Fig. 7 shows slurry pH increase of each slurry with the number of polishing. As slurry pH increased by polishing, it is thought that positively charged contaminants are increased in the slurry as described above.

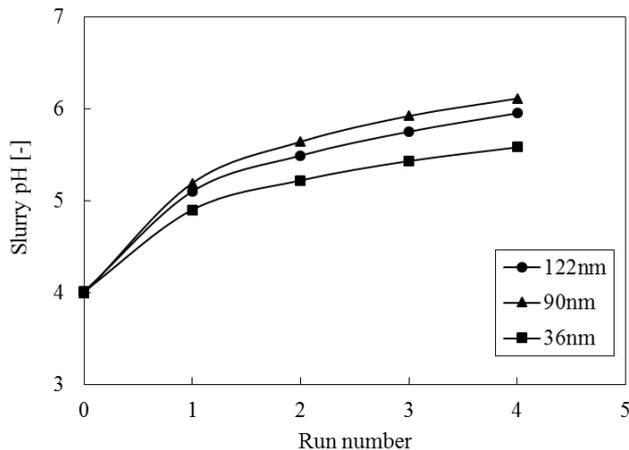


Fig. 7. Slurry pH change with the number of polishing.

On the other hand, the reason smaller particle tends to aggregate easier than larger one is that the distance between smaller particles is shorter than that of larger particles at the same particle concentration. Therefore, the collision frequency between smaller particles is higher than that of larger particles.<sup>[5]</sup> As a result, it is considered that a bigger change in particle size of smaller particle compared to larger particle during polishing caused bigger reduction of removal rate. From a series of evaluation results, acid slurry containing large particle is good for obtaining both high removal rate and good recycle stability.