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CMP (Chemical Mechanical Polishing) characteristics of langasite single crystals for SAW filter applications

Min Chul Jang*, Kyung Joo and Keun Ho Auh
Ceramic Processing Research Center (CPRC), Hanyang University, Seoul 133-791, Korea

Langasite is a promising new piezoelectric material for SAW filter application. Little was known until recently about the methods needed to mechanically polish and chemically polish/etch this material. In this experiment, polishing, slurry chemistry and chemical wet etching for langasite will be described. Conventional quartz and LN ($LiNbO_3$) polishing methods did not produce satisfactory polished surfaces, and polishing with a colloidal silica slurries has so far shown to be most effective. And the optimum condition was found by changing the slurry chemistry. As the planarization effect is very important in SAW filter applications, the examination of the effective particle number effect and the particle size effect was carried out. Z-cut langasite surface which had been polished with the colloidal silica slurries was etched in a variety of etchants. Conventional quartz etchants destroyed the polished surface. Other etchants formed a thin film on the surfaces. In this experiment, the reaction between langasite and a few etching solution was analysed. And an appropriate selective etchant solution for analyzing the defects was synthesized.

Key words: Polishing, Langasite ($La_3Ga_5SiO_{14}$), Et- ching, Planarization.

Introduction

Recently, the evolution of electronic technology towards higher frequencies and larger baud rates had led to the interest in finding new piezoelectric materials, which realize filters with larger shifts or larger frequency stability. For the designation of the above mentioned devices, the necessity has arisen in discovering new piezoelectric materials having intermediate properties between those of quartz and lithium tantalate ($LiTaO_3$). $La_3Ga_5SiO_{14}$ (LGS) has been a current leading candidate to satisfy those requirements because of its unique acoustic characteristics [1].

When the wideband filter is produced using quartz crystal, it must be designed as so called a discrete type, because the electro-mechanical coupling coefficient of quartz crystal is small. In this case, since it uses many crystal units and transformers, it requires larger outer dimensions and weight resulting in poor yield, and requiring worrisome adjustment in manufacturing [2, 3].

Though langasite displayed superior piezoelectric properties, problems with single crystal growth and etching have hindered its' characterization. Little was known until recently about the methods needed to mechanically polish and chemically polish/etch this material. Conventional quartz polishing methods, using such polishing agents as rare earth slurries or ruby powder, did not produce well polished surfaces. But when colloidal silica slurries was used, satisfactory polishing results were obtained.

Z-cut langasite surfaces which had been polished with the colloidal silica slurries were etched in a variety of etchants. The conventional quartz etchants destroyed the polished surfaces, while other etchants formed a coating on the surfaces.

Langasite's acoustic attenuation has been reported to be three to five times lower than that of quartz. This suggests that devices with high Qs (Quality factor) should be possible, however, the Qs of langasite resonators have been reported to be significantly lower than the Qs which can be achieved with quartz resonators of the same frequency. This discrepancy has been attributed to the lack of good polishing methods for langasite [3-12].

Therefore, the experiments described below were undertaken with the primary goal of developing polishing and etching methods which are capable of producing defects-free langasite surfaces. A secondary goal was to obtain the planarization conditions in polishing process for SAW filter applications.

Description of Experiments

Polishing

Langasite polishing experiments were performed on a Z-cut langasite wafers, each having an area of approximately 21 mm × 15 mm. The wafers were lapped on the Buehler Model Automat II and a self-designed

*Corresponding author:
Tel: +82-2-2290-0567
Fax: +82-2-2299-2884
E-mail: mcjang@hymail.hanyang.ac.kr

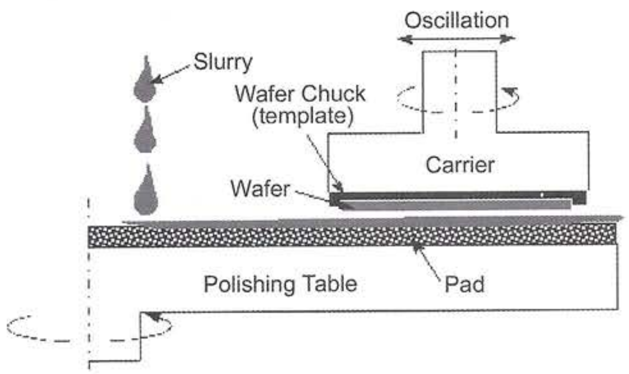


Fig. 1. The schematic drawing of the polishing apparatus.

polishing plate. Figure 1 shows the schematic diagram of the polishing apparatus. 10 μm, 8 μm and 3 μm SiC slurries were prepared for the lapping process, And, the wafers were polished on the same machine and chem-cloth.

First, the conventional mechanical polishing process was adapted by using a 1 μm diamond slurry, cerium oxide and 0.25 μm diamond paste in separate operations. Also, CMP (Chemical Mechanical Polishing) process was applied by using colloidal silica slurries. To find out the optimum conditions for obtaining the defect-free wafers, each method was compared.

4 lbs of pressure was applied at a 90 rpm rotation rate on an 8 inch wheel. The surfaces polished with the four different slurries were investigated by an OM (Optical Microscope) and an AFM (Atomic Force Microscope), where the surface roughness and morphology was observed to obtain the optimum slurries.

Colloidal silica slurries generally used in the CMP process consists of discreet submicron amorphous silica particles dispersed in water, and usually, some additives are also added for controlling the pH level. The particles are nearly spherical in shape and the particle size are very small, typically on the order of 50 nm [13, 14]. Figure 2 shows the particle size distributions for the colloidal silica used in this research.

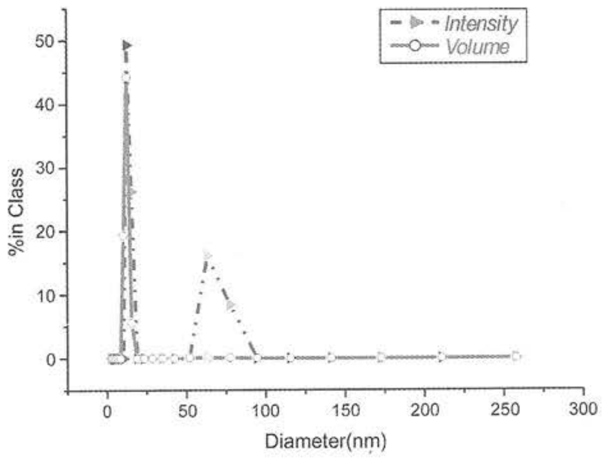


Fig. 2. Particle size distribution measured by a Zeta Sizer 3000.

Colloidal silica used in here are the order of 0.014 particles. The discrepancy in particle size will be explained later in this paper.

However, use of the colloidal silica slurries resulted in a lower polishing rate than the other slurries. So, in this experiment, the relationship between the KOH in the colloidal silica slurries and the polishing pad was investigated to explain this phenomena. And the relationship between wafers and chemical properties of slurry was also investigated by controlling the pH level of slurries. For such pH level control, CH_3COOH and NH_4OH was used.

Planarization

The most important factor in applying langasite single crystals to SAW filter devices is planarization. Because the surface acoustic wave travels on the surface of the crystal, the surface properties are very important in determining the insertion loss. The factors influencing the planarization include the pad's mechanical property, pad's roughness, slurry particle size, slurry chemistry and effective particle number [14, 16]. So in this experiment, slurry particle size and effective particle number effect was investigated to determine the optimum particle size and effective particle number in colloidal silica slurries. To measure the planarization, langasite wafers were prepared as in Fig. 3 1000 Å SiO_2 was deposited by a sputter and PR (PhotoResistive) was coated on langasite wafers. The wafer was then etched with a 1 : 10 volume mixture of HCl : H_2O. HCl was used due to its high etching rate on langasite. The removal rate was measured with an Alpha-step 500 surface profilometer by measuring the depth of the etched grooves before and after the polishing surface.

Wet etching

A variety of etchants were used for etching langasite. A various ratios of HCl : H_2O, HF : H_2O and HNO_3 : H_2O was investigated at various temperatures. HF and

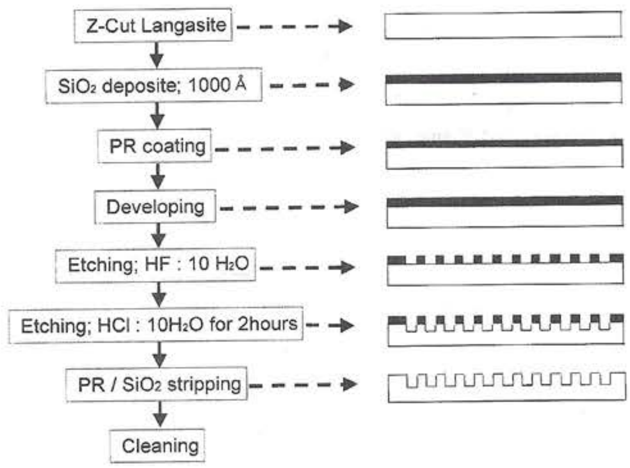


Fig. 3. Patterning process used as a re-treatment procedure before the measurement of planarization.

HNO_3 solutions resulted in the formation of a thin film on the surface, and this thin film was analyzed by an AES (Auger Electron Spectroscopy) and an SEM. And the selective etchant for langasite crystal was found to improve the crystal quality while some aided in the analysis of its defects. In this experiment, H_3PO_4 : H_2SO_4 was used to analyze the defects in the crystals.

Annealing effect

In the polishing process, annealing and etching was added to the conventional method to remove the damaged layers, relax the stress during polishing process and improve surface morphology [17, 18].

To investigate the annealing effect, the conventional polishing, polishing/etching, polishing/annealing and polishing/etching/annealing methods were performed by investigating the surface morphology using an AFM. In this experiment, the annealing process was performed 1200°C (elevated at 5°C/min) for 8 hrs. And, langasite wafers were etched at room temperature for 30 minutes.

Results and Discussion

Polishing Slurries and Slurries Chemistry

The surfaces polished with 1 μm diamond slurry, cerium oxide, 0.25 μm diamond paste and colloidal silica slurries were observed by an OM (Optical Micro-

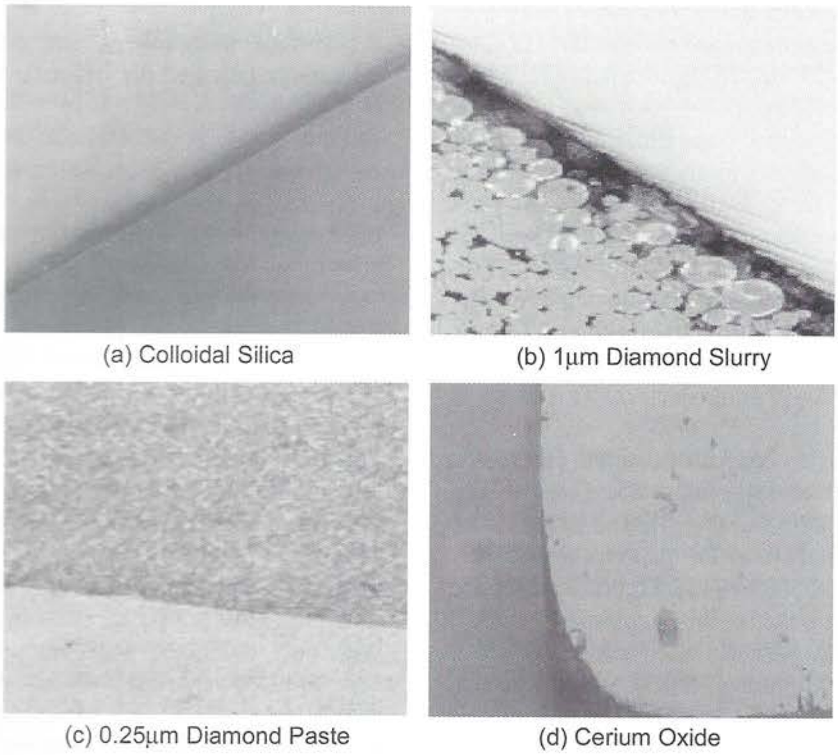


Fig. 4. Optical microscope image of surface polished with: (a) colloidal silica slurry (b) 1 diamond slurry (c) 0.25 diamond paste (d) cerium oxide.

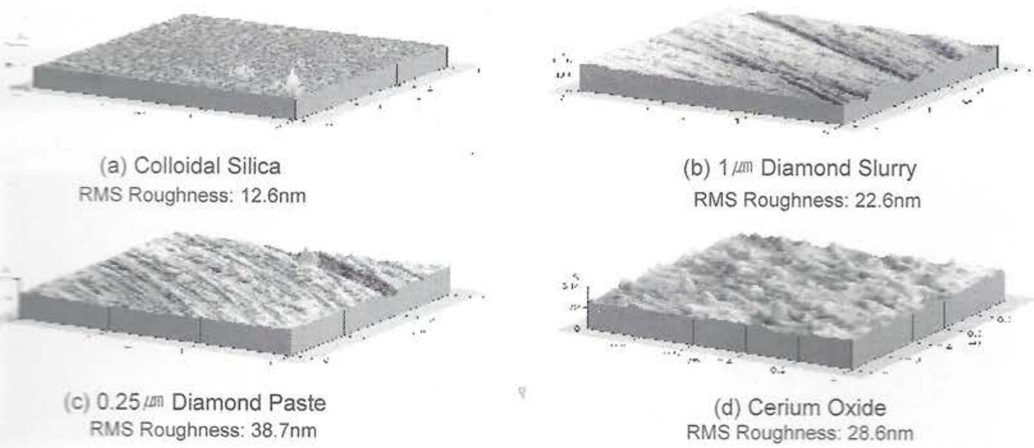


Fig. 5. AFM images of surface polished with: (a) colloidal silica slurry (b) 1 diamond slurry (c) 0.25 diamond paste (d) cerium oxide.

Table 1. Effects of the four slurry types on langasite

Abrasive materials	Results
1 p diamond slurry	Many scratches, edge rounding Average Roughness 19.7 nm
0.25 p diamond paste	Insufficiently polished surface Average Roughness 28.8 nm
Cerium oxide	Many scratches Average Roughness 18.8 nm
Colloidal silica	Sufficiently polished surface Average Roughness 11.2 nm

scope) and an AFM (Atomic Force Microscope). Figure 4 shows the OM (Optical Microscope) images of the surfaces polished with the four slurries. Cerium oxide resulted in many scratches and 1 diamond slurry produced many scratches and edge rounding. The 0.25 μm diamond slurry resulted in an insufficiently polished surface, owing to the low polishing rates. The best results were obtained with the colloidal silica slurries, and the surface roughness was then analyzed with an AFM. The roughness factor is important for SAW filter applications, as the average roughness must be between 10~15 nm for suitable insertion loss. In Fig. 5, colloidal silica slurries resulted in a smooth surface after polishing, with the surface roughness below 13 nm. Therefore, langasite wafers polished with colloidal silica could be applied for SAW filter devices. This result is summarized in Table 1.

Colloidal silica slurries consist of discrete submicron amorphous silica particles dispersed in water, and usually, some additives for controlling the pH. The particles are nearly spherical in shape and the particle size is very small, typically around 50 nm [13]. Figure 2 shows the particle size distributions for this particular colloidal silica. Colloidal silica particles used here was about 0.014 μm. In this CMP process, reaction layers formed between the wafer surface and the slurries and this layers was removed by the discrete submicron amorphous silica particles. Therefore, interaction between the mechanical and chemical reactions seems to improve

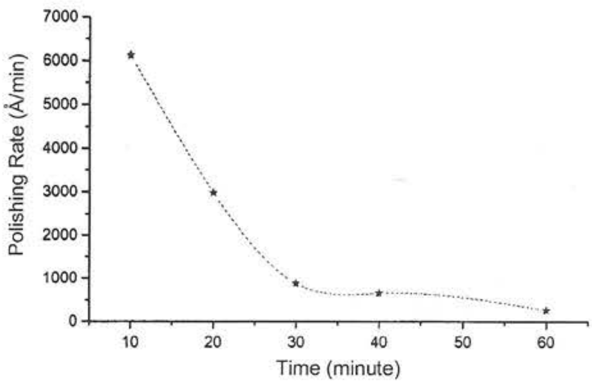


Fig. 6. Polishing rate (0.014 μm colloidal silica slurry) of langasite crystal by using colloidal silica slurries as a function of polishing time.

the surface morphology, and colloidal silica slurries effectively polished the langasite wafer.

Polishing rate and KOH effect

Polishing rate was measured to obtain the optimum polishing process for langasite. The graph in Fig. 6 illustrates the polishing rate for langasite, when polished with colloidal silica.

As Fig. 6 shows, the polishing rate decreases with time. This attributes to a low polishing rate when colloidal silica is used.

Two possibilities can be theorized to explain such a result. First, a physical distortion of the pad from the polishing pressure might hinder the polishing process. Second, the KOH might induce a chemical reaction with the pad, resulting in a decrease in polishing rate [15-16, 18].

In this experiment, we examined the pad surface condition after polishing with only a KOH solution and with only deionized water containing no abrasives to compare the respective effects of the KOH and the polishing pressure. The polishing pad was observed with a SEM. It can be seen in Fig. 7 that the formation of flat defective regions on the polishing pad surface is mainly due to the interaction of the pad surface with

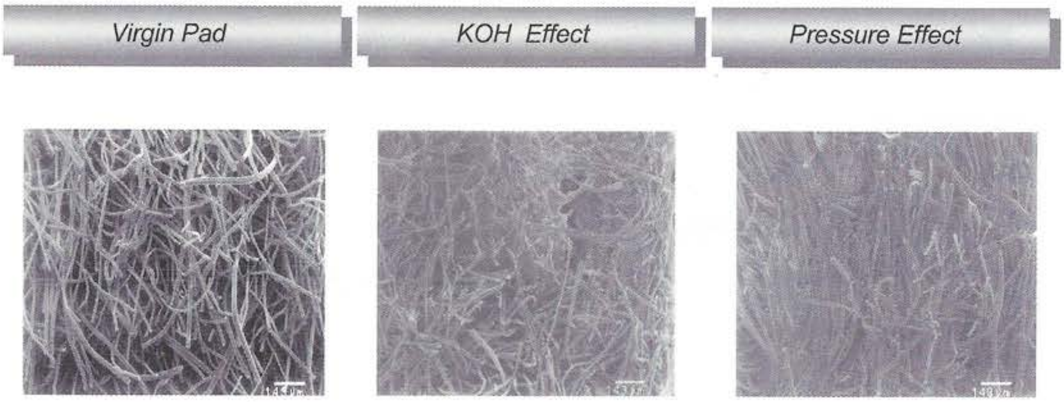


Fig. 7. SEM image of the polishing pad after polishing in different conditions: (a) vergin pad (b) KOH effect (c) pressure effect.

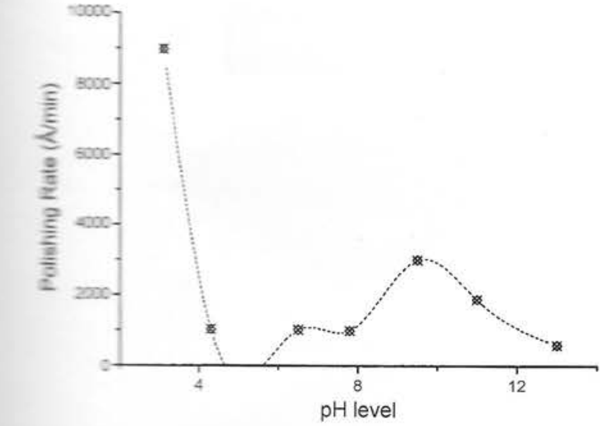


Fig. 8. Polishing rate of langasite crystal (0.014 colloidal silica slurry) by using colloidal silica slurries as a function of pH level of colloidal silica slurries

the KOH solution, and not the physical deformation of the pad surface caused by the shear forces applied during the polishing process. This damage to the polishing pad surface by the KOH solution increases the net contact area between the polishing pad and the wafer, reducing the effective polishing pressure for a constant polishing load. In addition, this damage reduces the number of channels available for slurry transport to the center of the wafer being polished, resulting in a reduced polishing rate.

To overcome this disadvantage, the recondition methods of polishing pads have been generally used [19]. In this experiment, we examined slurry chemistry to try to increase the polishing rate of the colloidal silica slurries. The relationship between the pH of slurries and the polishing rate was investigated to examine the polishing rate of the colloidal silica slurries.

pH of slurries was controlled by using CH_3COOH and NH_4OH. Figure 8 represents the polishing rate change according to the different pH levels. As Fig. 8 shows, low pH slurries results in higher polishing rate in case of langasite wafers. Therefore, pH of slurries must be acidic for effective use in langasite CMP process.

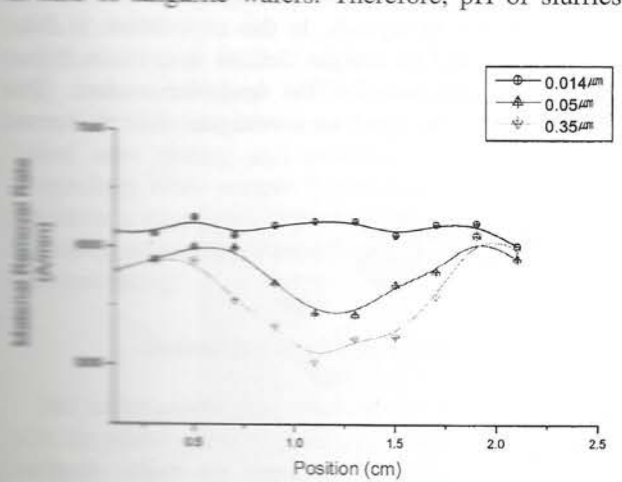


Fig. 9. Polishing rate of colloidal silica slurries according to particle size

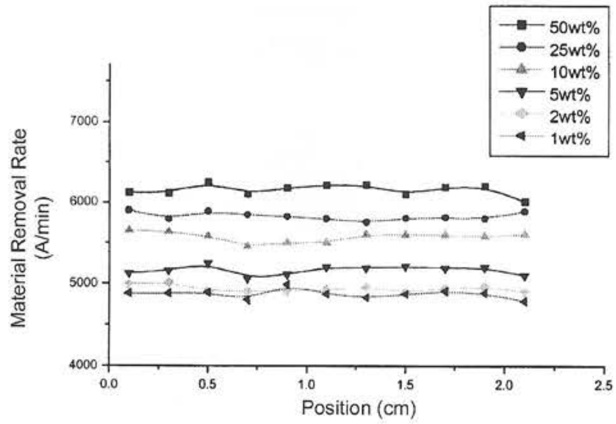


Fig. 10. Polishing rate of colloidal silica slurry having different particle concentrations.

Table 2. Polishing conditions for langasite

Parameters	Conditions
Relative rotation speed	80~90 rpm
Polishing Pressure	2~4 lbs
pH of slurry	2~3
Particle size	0.014~0.025 p
Particle concentration	1~2 wt%

Planarization

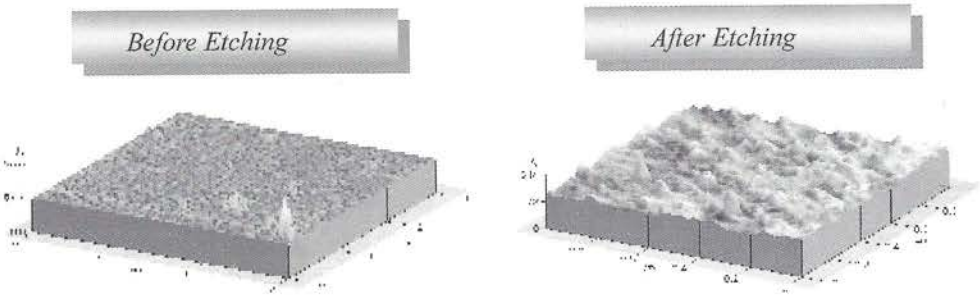
The most important factor in applying langasite single crystal to SAW filter device is planarization. Because surface acoustic wave travels on the surface, the surface properties are very important in determining the insertion loss.

Figure 9 shows the particle size effect of slurries. This data was measured after 10 minutes of polishing. In Fig. 9, 0.35 μm and 0.05 μm resulted in a "bull's eye" effect. Bull's eye effect refers to an insufficiently polished wafer center due to a reduction in the number of channels for the slurry to be transported to the center of the wafer being polished. However, 0.014 μm particle size resulted in the best planarization, as the minute size of the particle enables it to be easily transported to the center of the wafer.

Figure 10 shows the effective particle number effect of slurries. As is shown in Fig. 10, 1~2 wt% of submicron amorphous silica particles improved the planarization of langasite wafers. The optimum conditions of slurries is summarized in Table 2.

Wet etching

A variety of etchants were used for etching langasite. A various ratios of HCl : H_2O, HF : H_2O and HNO_3 : H_2O was investigated at various temperatures. HF and HNO_3 solutions resulted in a thin film formation on surface, and this thin film was analyzed with an AES



(a) RMS Roughness: 13nm

(b) RMS Roughness: 33nm

Fig. 11. AFM images of surface etched with HCl solution: (a) before (b) after.

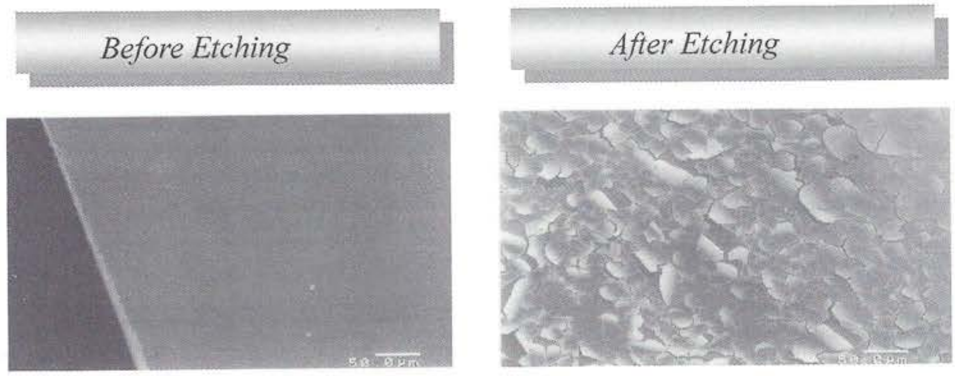


Fig. 12. SEM image of surface etched with an HF solution.

(Auger Electron Spectroscopy) and a SEM.

It can be seen in Fig. 11 that although HCl showed a high etching rate, it results in rough surface, making it inappropriate as a langasite etchant. However, the high etching rate of HCl was useful for patterning, and was used to measure the planarization.

HF solutions produced a thin film on the surface. Figure 12 shows an SEM microscopy of such a film. AES (Auger Electron Spectroscopy) revealed this film to consist of langasite's cations and etchant's anions, as can be seen in Fig. 13 This data was measured by AES (Auger Electron Spectroscopy).

HNO_3 also produced a thin film on the surface. HCl was added to HF and HNO_3 in hopes of preventing this phenomena, but a thin film was again observed on the surface.

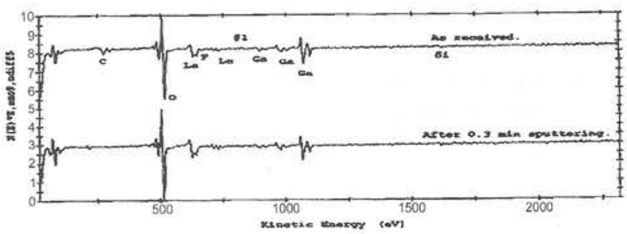


Fig. 13. AES data of surface etched with an HF solution.

Selective etchants of langasite crystal was found to improve the crystal quality and enabled the observation of defects in crystals. In this experiment, H_3PO_4 : H_2SO_4 was used to analyze defects in crystals. Figure 14 reveals etch pits on the langasite surface. This etchant could be used to investigate defects formed

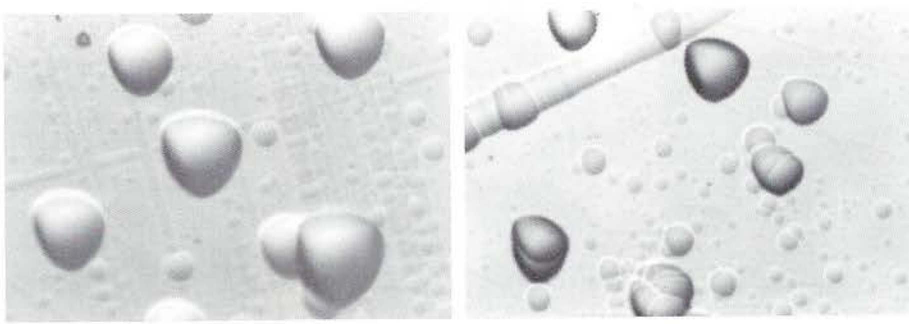


Fig. 14. Image of etch pit after etching.

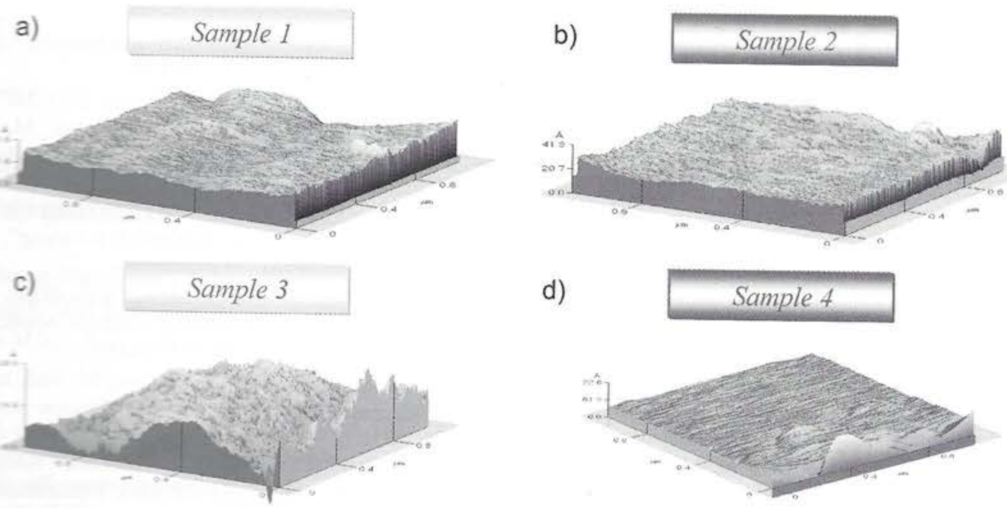


Fig. 15. AFM Image of surface polished by: (a) conventional polishing (b) polishing/annealing (c) polishing/etching (d) polishing/etching/annealing method.

Table 3. RMS roughness and average roughness of langasite under different polishing conditions

	RMS Roughness	Ave. roughness
Sample 1	14.76 nm	13.45 nm
Sample 2	12.52 nm	11.84 nm
Sample 3	14.46 nm	13.61 nm
Sample 4	9.5 nm	8.13 nm

during crystal growing and polishing process.

Annealing effect

In the polishing process, annealing and etching was added to the conventional method to remove the damaged layers, relax the stress during polishing process and improve surface morphology.

To investigate the annealing effect, the conventional polishing method, polishing method including etching, polishing method including annealing process and polishing method including both etching and annealing was performed by investigating the surface morphology with an AFM.

Figure 15 and Table 3 shows the surface morphology measured with an AFM. In Fig. 15. and Table 3, sample 2 (polished after annealing) and sample 4 (polished after etching and annealing) produced the best morphology, while sample 3 (polished after etching) did not show a significant improvement, indicating that the annealing process does have favorable effects on langasite processing.

Summary and Conclusions

The experiments described above were undertaken with the primary goal of developing polishing and etching methods which are capable of producing defect-free langasite surfaces. A secondary goal was to obtain the planarization conditions in polishing process for SAW filter applications.

In this experiment, langasite was polished by using the CMP (Chemical Mechanical Polishing) process. In the CMP process, colloidal silica slurry was used in polishing langasite crystals. The most important factor in applying langasite single crystals to SAW filter devices is plana- rization. The factors influencing the surface morphology include slurry particle size, slurry chemistry, effective particle number, polishing pressure, pH level of slurries and relative rotation speed of polishing plate. Table 2 summarizes optimum conditions in polishing langasite. However, the relationship between polishing pad and slurries was not investigated, so, this factor needs to be investigated.

In the etching process, the relationship between langasite and etchants was investigated and from these results, selective etchant were synthesized.

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