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ORIGINAL ARTICLE

Influence of acid slurries on surface quality of LBO crystal in fixed abrasive CMP

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Abstract The chemical action, which is generally determined by acid or alkaline regulator in the slurry, plays an important role in fixed abrasive chemical mechanical polishing (CMP). Experiments were conducted to explore the influence of acid slurries on surface quality and material removal in fixed abrasive CMP of LBO (LiB_3O_5) crystal with soft and brittle, difficult-machining properties. Four organic acid regulators with pH 4.0 (acetic acid, lactic acid, citric acid, and oxalic acid) were screened by the polishing experiments. Further, pH value of the regulator was optimized to the most suitable chemical environment fit for polishing of LBO crystal using three indexes, i.e., material removal rate (MRR), surface topography, and surface roughness. The results indicated that citric acid leads to more robust polishing performance with surface roughness Sa (surface roughness average) 0.52 nm lower than the others. Additionally, MRR decreases with increasing pH value. Surface roughness initially decreases with increasing pH value, then it mutates to the maximum 9.37 nm after reach the minimum. The abrasive-free acid slurry with citric acid at pH 5.0 is effective for fixed abrasive CMP of LBO crystal, obtaining a MRR of 366 nm/min and fine surface quality with surface roughness Sa 0.32 nm with rather slight surface damage.

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1 Introduction

Owing to its relatively large nonlinear optical (NLO) coefficient, wide transparency, large angular bandwidth, small wake-off effect, and high laser-induced damage threshold, lithium triborate (LiB_3O_5, LBO) crystal is commonly used as frequency-conversion crystal in solid-state lasers [1]. Recently, LBO crystal has been applied in OPCPA (combined with optical parametric amplification and chirped pulse amplification), fast ignition in laser fusion, solid-state laser displays, etc. [2]. Surface quality and efficiency of processing NLO crystal are the main factors that restrict its high-performance and large-scale development. A high surface quality of LBO crystal is urgently needed because of its applications in high-energy laser systems, and chemical mechanical polishing (CMP) is an effective method to obtain a super smooth surface of LBO crystal [3, 4].

There are many factors that influence CMP process, and the chemical action is a significant one in achieving a super smooth surface. Much effort has been made to better understand the chemical effect of the slurry in CMP process, which is mainly composed of chemical additives such as acid [5–10], alkaline [11], oxidant [12], and inhibitor. Li et al. [5] studied the law of chemistry-enhanced mechanical effects dependent on pH value for copper CMP and compared the chemical mechanism under three different environments of acid, neutral and alkaline slurries. According to Cho et al. [6], there was only negligible formation of chemical oxides with an increase in citric acid in the H_2O_2 containing slurry, while they were formed continuously for β-cyclodextrin in CMP of polycrystalline $Ge_2Sb_2Te_5$ (GST) film. Chen et al. [7] indicated that the chemical removal rate of Ta was substantially enhanced and surface roughness from 32.52 nm could be decreased to 16.21 and 13.81 nm, respectively, after acid (CH_3COOH or H_3PO_4) was added into Ta-CMP in H_2O_2-base slurries. For stronger corrosion capacity, sulfuric acid is added to an H_2O_2 containing slurry, which results in a glossy mirror-like surface of polished stainless steel foil. Surface roughness can be reduced from the unpolished 13.6 nm to the polished 0.7 nm with the maximum material removal rate (MRR) of over 150 nm/min [8]. However, static etching experiments showed that GST film was etched faster in the alkaline region than the acidic region, both resulting in a porous island-like surface, which differs from conventions with acid having stronger corrosion than alkaline [9]. Jeong et al. [10] studied the additive's effect for higher MRR in lithium niobate CMP and the manufactured slurry consisting of H_2O_2-citric acid in the KOH-based slurry, and found that MRR by H_2O_2 at 2.0 wt% and citric acid at 0.06 M was higher than the other conditions. Wang et al. [11] found that ceria particles increase MRR before slurry pH reaches 10.0 in CMP of silicon wafers. Lu et al. [12] revealed that H_2O_2 could greatly increase the static etch rate and MRR of cobalt, and the corrosion potential difference between Co and Cu can be reduced to a very small value by using 2-MT in a glycine based slurry at pH 5.0.

Apart from the difficulty of growing NLO crystals, fabrication to meet high productivity and surface quality is necessary to realize the excellent properties of these crystals [13]. Problems in traditional machining such as scratch, chipping, cracking, stress concentration on the edge, and abrasive embedded normally happened on account of its soft and brittle characteristics [14–17]. In addition, little has been reported on LBO crystals with different lattice planes. Because of its rhombic structure, the anisotropy of LBO crystal leads to different properties of different lattice planes and needs different process conditions for obtaining a super smooth surface. It is difficult to machine a specified surface of LBO crystal, especially to fit its unique chemical sensitivity and machinability. Li et al. [4] compared (100) surface, (010) surface, and (001) surface in the same CMP process, and found that the anisotropy of LBO crystal has an effect on MRR and surface roughness, and in the scope of their experiment high MRR leads to better surface roughness. Especially for (001) surface, the best surface roughness reaches 0.197 nm RMS when pH is 4.0 using colloidal SiO_2 slurry, and Taguchi method was applied for optimization of its polishing parameters [14, 18]. Recently, Li et al. [15] reported that the abrasive-free alkaline slurry with ethylenediamine and pH 11.0 can be effective for fixed abrasive polishing of LBO crystal (110) surface, obtaining a high surface quality with surface roughness Sa (surface roughness average) 1.94 nm. Liao et al. [16] developed a new septum with a pre-strain and a carrier system in common CMP. The obtained P-V error of the LBO crystal is also less than $\lambda/8$ while surface roughness is approximately 0.68 nm.

Recently, fixed abrasive CMP has been introduced as a potential alternative method to abrasive polishing to improve geometrical accuracy, reduce cost, and improve machining efficiency with controlled surface and subsurface damage [19–21]. In a fixed abrasive pad, the abrasive particles are embedded in the pad uniformly and densely, which results in a uniform distribution of micro-cutting edges over the pad. Instead of traditional slurry, only deionized water and chemical additives are used as an abrasive-free slurry, which can make the polishing process less complicated, avoid random abrasives scratching the wafer surface, and reduce the cost of consumable slurry. Tian et al. [22] conducted a series of CMP experiments for optical silicon substrates using seven different chemical slurries and analyzed the polishing performances under different chemical environments. A deep fundamental understanding of chemical effects in LBO crystal CMP using a fixed abrasive pad can provide some insight into the optimization of the process and improve surface quality while maintaining a high MRR [6].

In this work, screening experiments were performed with four acid regulators (acetic acid, lactic acid, citric acid, and oxalic acid) to find represent to fit LBO crystal polishing by compounding abrasive-free slurries with fixed pH value at 4.0 [4, 14], and then pH value from 3.0 to 5.5 was optimized to achieve the most suitable chemical environment. The polishing performance was evaluated and compared in terms of material removal rate, surface topography, and surface roughness, which attempts to explore chemical effect and reveal the generation of super smooth surface.

2 Experimental

The specific lattice plane (θ=90°, φ=13.8°) of LBO crystal, which will be applied in fast ignition in laser fusion in the future, was adopted in the polishing experiments and was conducted by a Nanopoli-100 smart precision lapping/polishing machine (Wistates Precision Technology Group, People's Republic of China). The size of the workpiece is 33.2 mm×31.6 mm×7.1 mm. A hydrophilic fixed abrasive pad with 1 μm CeO_2 is selected as polishing pad [15, 23, 24], which has a regular intersecting orthogonal channel pattern. Abrasive-free slurry mainly contains with deionized water, surfactant OP-10 agent ($C_{34}H_{62}O_{11}$, a nonionic surfactant) and organic acid regulator. The surfactant is used to further reduce the surface tension of the pad, which makes the slurry quickly insinuate into the working site. Owing to their small volatility, decrease of metallic ion contamination, and weak acid properties, acetic acid (CH_3COOH), lactic acid

($C_3H_6O_3$), citric acid ($C_6H_8O_7$), and oxalic acid ($C_2H_2O_4$) are adopted in these experiments, respectively. The prepared slurry is sprayed by a peristaltic pump onto the pad surface and then transported to the working site. Before polishing, the lapping process is used to uniform morphology, and polishing parameters are listed in Table 1.

After every process, LBO crystal is demounted to soak in anhydrous alcohol with ultrasonic cleaning (GEN II ultrasonic wave apparatus) followed by drying, and then put into drying vessel for testing. The mass of LBO crystal is determined by Sartorius BS224S precision balance with a resolution 0.0001 g. The thickness is measured by electronic digital display micrometer with a resolution 0.01 mm, which is distributed at eight different locations of each workpiece and the mean values are calculated from repeated experiments. MRR is defined as the reduction of thickness per unit time, and calculated using Eq. 1.

$$\mathrm{MRR} = \frac{(m_0 - m_{\mathrm{i}}) \times h_0}{m_0 \times t} \times 10^6 \qquad (1)$$

Where m_0 and m_i is the mass before and after each experiment, h_0 is the thickness before each experiment, and t is the process duration (in minute), and the unit of MRR is in nanometer per minute. Surface topography of polished surface is observed with a metallographic microscope (XJX-200, Nanjing Jiangnan Novel Optics Co., Ltd., People's Republic of China). An atomic force microscopy (AFM, CSPM4000, Beijing Nano-Instruments LTD., People's Republic of China) is adopted to measure surface topography and surface roughness with contact mode, and AFM traces are taken in an area of 10 μm×10 μm.

3 Results and discussion

3.1 Lapping

Figure 1 shows the typical surface topography of a lapped surface after uniform morphology. There is no obvious corrosion trace generated on the surface, and the balance of

Table 1 Polishing parameters

Parameters	Polishing
Pad rotation speed	70 r/min
Eccentric distance	82 mm
Slurry flow rate	60 ml/min
Process duration	20 min
Pressure	14 kPa

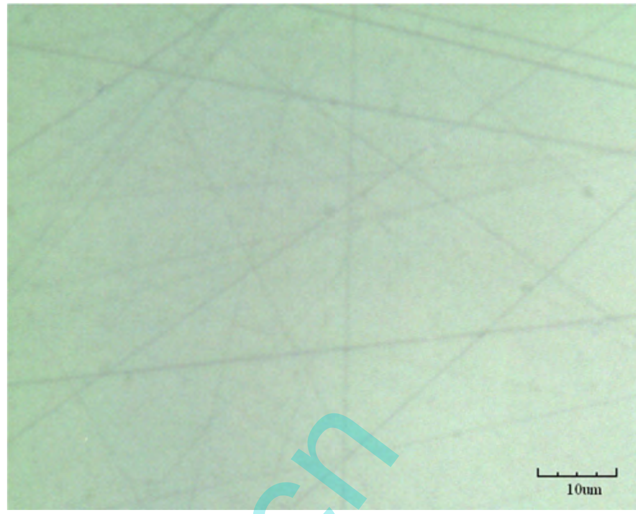


Fig. 1 Microscope surface topography of lapped surface

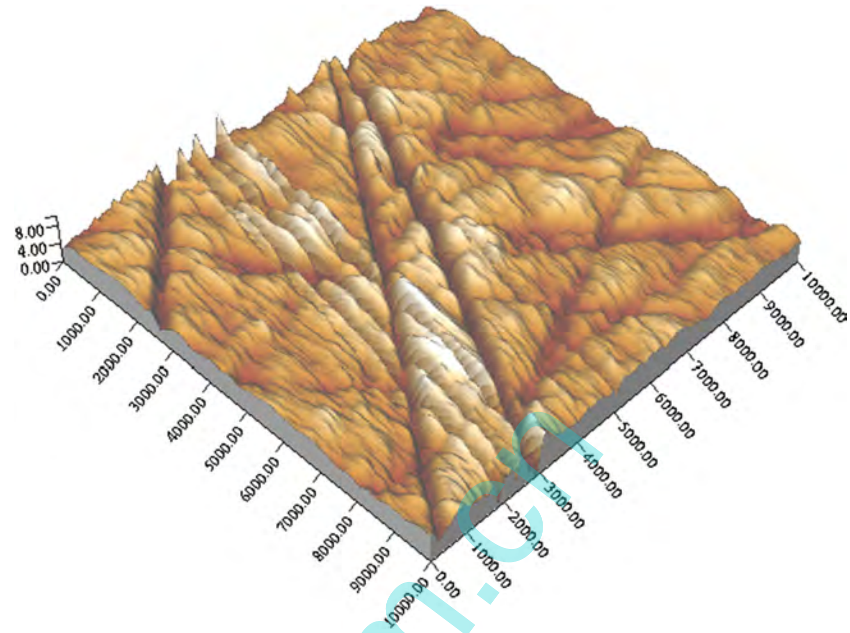
chemical effect and mechanical function results in ductile micro-scratches. The depth of micro-scratches in lapping depends to a great extent on the variation of cutting-in that attributes to the exposure of diamond and the contact mode between interfaces [25]. Surface roughness Sa is 0.97 nm, and AFM surface topography is shown in Fig. 2 with intersecting micro-scratches.

3.2 Polishing

3.2.1 Selection of acid regulator

As illustrated in Fig. 3, MRR varied from 369 to 628 nm/min with different acid regulators at pH 4.0, which almost follows the similar trend of the $\mathrm{pK_a}$ curve [26], except for the lactic acid slurry. Here, $\mathrm{pK_a}$ is the symbol for the acid dissociation constant at logarithmic scale, and generally, the smaller $\mathrm{pK_a}$ value represents the stronger acid. Because the first-stage ionization dominates multi-stage ionization process of polyprotic acid, the $\mathrm{pK_a}$ values of first-stage are selected for citric acid and oxalic acid, respectively. With the acid corrosion action, a softened layer is formed on crystal surface, whose hardness is lower than the crystal body, and it will be easily removed by the function of mechanical carving or scraping [27]. The stronger acid corrosion is, the thicker or softer the soften layer becomes, and the thicker softened layer made MRR increase steadily when mechanical removal was constant. Especially for lactic acid slurry with MRR break, MRR 608 nm/min is almost 180 nm/min higher than that of citric acid slurry. Material removal rate may have a certain relationship with the presence of the intramolecular hydrogen bridge between the hydroxyl and the carboxylate radical in

Fig. 2 AFM surface topography of lapped surface



acid. According to the pK_a curve, the acidity of citric acid is more than that of lactic acid but symmetrical structure of the three carboxylate radicals in citric acid would promote the formation of hydrogen bridge, which improved molecular stability (seen Fig. 4). Ultimately, ionizing of hydrogen ion is suppressed partly, which weakens its chemical activity, making it less capable of strongly generating a soften corrosion layer than that of lactic acid. For details, MRR by oxalic acid slurry, 628 nm/min, was slightly higher than by lactic acid slurry and the highest one of four slurries. Due to the strong acid role of oxalic acid, obvious corrosion pits were generated and the porous softened layer with pits had lower surface energy, which sharply increased the mechanical removal amount.

Figure 5 provides surface topography of polished surface with four acid slurries. Surface quality by acetic acid slurry is the worst because many disorderly grooves emerged, which indicates mechanical function dominates. For oxalic acid, the sharp cutting edge of the pad or its debris penetrated into the softened layer and left ploughing trace on the harder interface below the softened layer, which is presented as discontinuous lines different from plastic scratches. Lactic acid and citric acid slurries appeared with similar corrosion streaks; the latter was random but the former had fine continuity and smoother edges, which means a milder corrosive effect. Additionally, the direction of the corrosion streaks may reflect the grain boundary of LBO crystal, and citric acid molecule can easily run into the gap of LBO crystal surface. With its muti-carboxyl symmetric structure, citric acid shows the modest chemical activity when releasing hydrogen ions to resolve the crystal surface.

As shown in Fig. 6, surface roughness Sa varies within a range of 0.52 and 1.03 nm in the four acid slurries, and surface roughness by citric acid slurry is about half of the three others. It is indicated that citric acid slurry can decrease surface roughness sharply. AFM surface topography of surfaces polished by four acid slurries was plotted in Fig. 7. The surface polished by citric acid slurry is flatter and smoother than others, with surface roughness Sa 0.52 nm. Compared with the other slurries which also had corrosion marks, the stronger acid action of oxalic acid results in obvious corrosion pits which destroys the original undamaged surface, and on

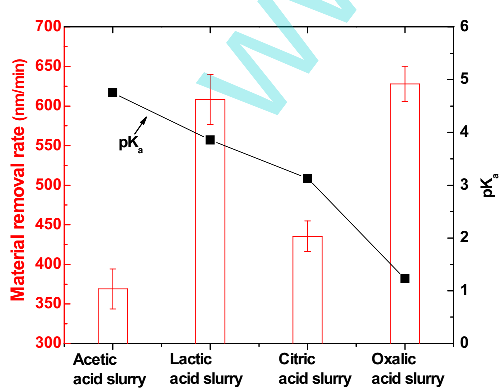


Fig. 3 pK_a curve and MRR of polishing process using four acid slurries with pH 4.0

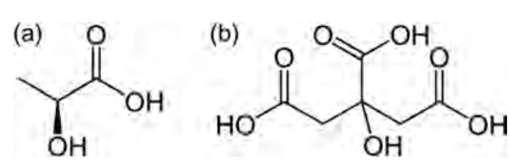


Fig. 4 Skeletal formula of **a** lactic acid and **b** citric acid

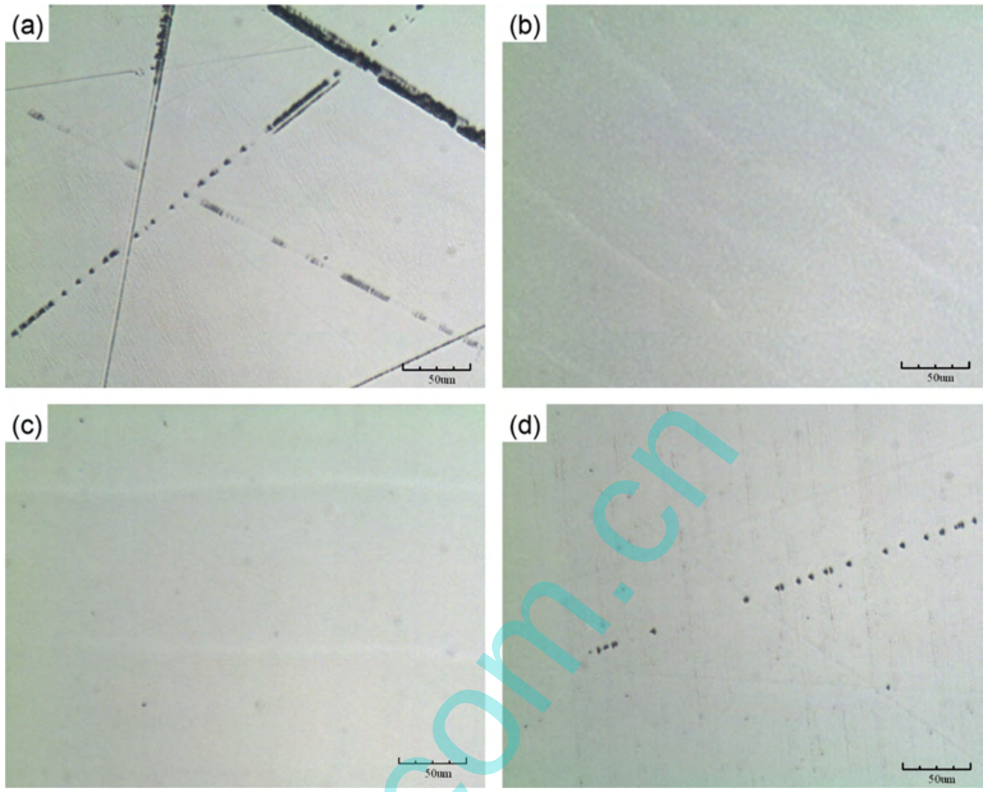


Fig. 5 Microscope surface topography of polished surface in four acid slurries with pH 4.0 **a** acetic acid; **b** lactic acid; **c** citric acid; **d** oxalic acid

the region of grain boundary of LBO crystal, ridgy zonal streaks were etched by lactic acid slurry. There is obvious scratch damage on the surface by the acetic acid slurry that mainly leads to the increasing of surface roughness Sa up to 1.03 nm. Both excessive mechanical function and chemical action would degrade surface quality seriously, but relatively mild chemical action could ensure better results.

Considering crystal surface topography and surface roughness, citric acid slurry achieves the best surface quality among four experiments conducted with the same pH value. In order to determine the capacity of organic acid to facilitate the best surface finish, citric acid is opted to optimize pH value.

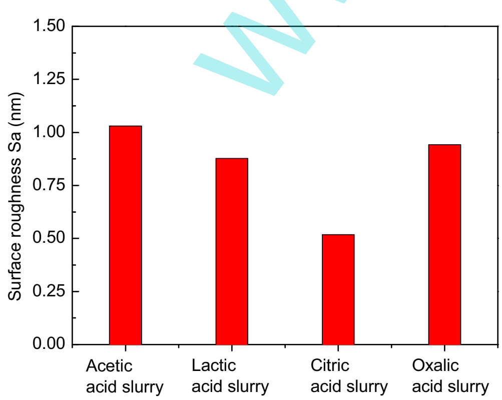


Fig. 6 Surface roughness of polished surface with four acid slurries

3.2.2 Optimization of pH value for acid regulator

Figure 8 exhibits MRR and surface roughness Sa versus pH value. MRR decreases from 673 to 208 nm/min with increasing pH value from 3.0 to 5.5. This result is quite fit the conventional concept thinking that the chemical reaction is enhanced by the intensification of the acid environment, which boosts CMP process and accelerates material removal. Meanwhile, surface roughness decreases along with increasing slurry pH value initially. After reach the minimum value of 0.32 nm at pH 5.0 with MRR 366 nm/min, surface roughness Sa mutated to the maximum 9.37 nm. Additionally, there is a platform from pH value 4.0 to 5.0 happened on both MRR and surface roughness, which indicates that there is a relative counterweight of the chemical action and mechanical function in a game.

Figure 9 shows surface topography of polished surface with different pH values by adjusting the additive amount of citric acid. Owing to the identical process parameters, the mechanical function is assumed to the same. The pH value adjustment activates the interface chemical reaction to varying degrees, which leads to different surface quality. At higher pH value, citric acid slurry is easier to obtain better surface with the decline of corrosion degree, but further increase of pH value results in the worst surface finish gradually, shown in Fig. 9d, with many big scratch damage and typical brittle peeling off. It is stronger the mechanical function than

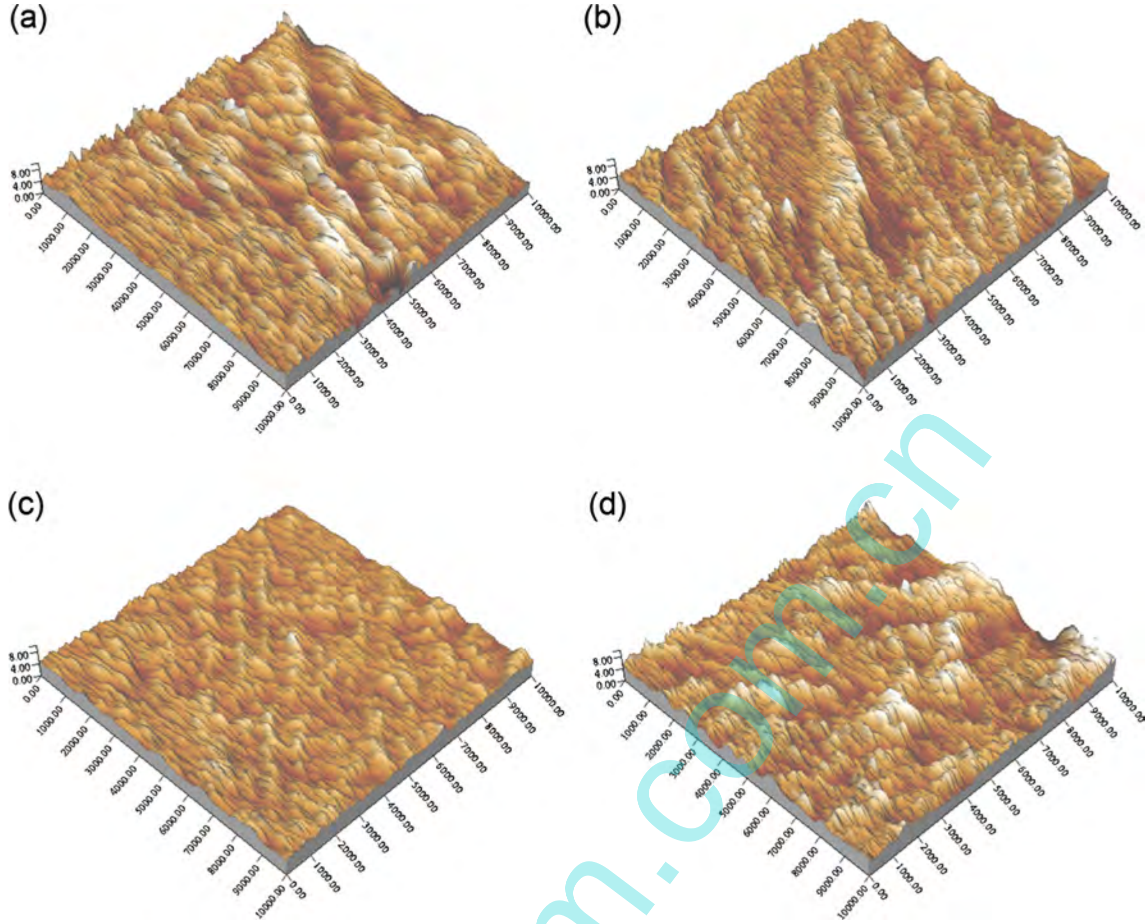


Fig. 7 AFM surface topography of polished surface in four acid slurries with pH 4.0 **a** acetic acid; **b** lactic acid; **c** citric acid; **d** oxalic acid

chemical action, which makes scratches increase. In addition, Fig. 10 describes the differences of the polished surface by different pH situations. There are micro-scratches emerged at pH 5.0, which indicates that the mechanical function

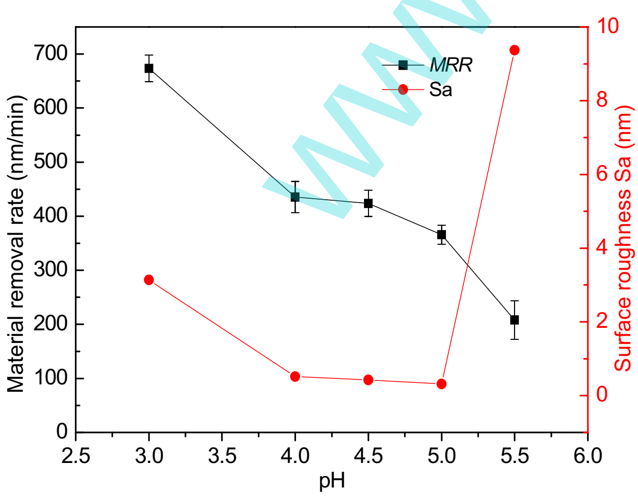


Fig. 8 MRR and surface roughness of polished surface with pH value variation

effectively begin to balance against excessive corrosion. At the highest pH value, the mechanical carving function dominates in CMP process, which results in broad and deep scratches with the evidence shown in Fig. 10d. Due to the release of brittle removal energy, the material removal drags the adjacent surface, which degrades the surface quality.

At a relatively opposite extreme, scratches weaken and acid corrosion strengthens gradually with pH value decreasing. From Fig. 9a, b, the ridgy zonal streaks remain to be the main surface characteristics, and the surface is bumpy with pits of uneven size shown in Fig. 10a, b. The mechanical function is surpassed by chemical action, which results in excessive corrosion and then raises surface roughness undesirably. There are already signs that the balance of the chemical action and mechanical function comes out at pH 4.5. Then, if the mechanical scribing function kept good consistency with the continuous forming of chemical softened layer, the surface finish is desirable, such as the smooth and intact surface shown in Fig. 10c at pH 5.0.

With intensifying acidic environment, the amount of corrosive ions (e.g., hydrogen ion) is growing. These ions impel the proceeding of "ions exchange" [28] in CMP process,

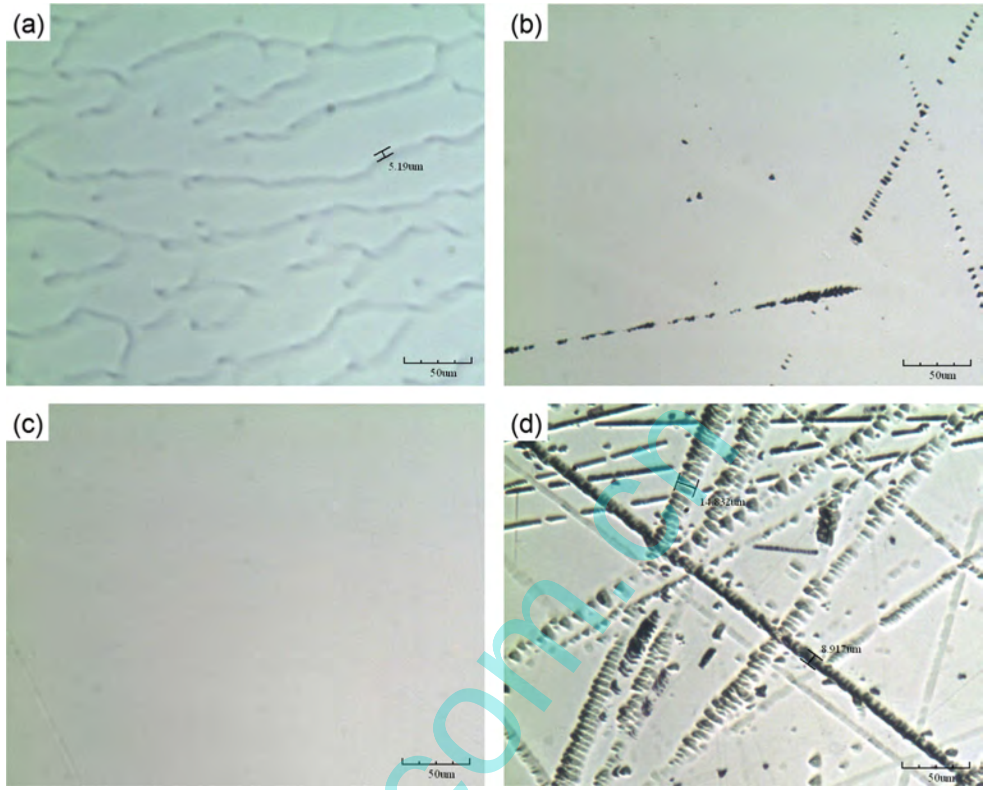


Fig. 9 Microscope surface topography of polished surface with different pH **a** pH 3.0; **b** pH 4.5; **c** pH 5.0; **d** pH 5.5. (Note: Fig. 5c shows pH 4.0)

which boosts the tribochemical reaction on the superficial layer of LBO crystal, and then the softened layer was generated continuously. On the premise of fixed process parameters, the removal amount increases with time going on. There are numerous "Generating-Removed" circles of the softened layer happened in CMP process. These experimental results indicated that:

1. Surface quality was influenced by the continuity of "Generating-Removed" circles on a certain extent. The mechanical function dominates when pH value is high. However, the generating softened layer cannot meet the demand of the mechanical removal amount, which leads to the continuity of circle breaking up partly. Therefore, the unnecessary mechanical scribing action scratches the crystal surface and makes surface roughness increase sharply shown in Figs. 9d and 8. Oppositely, the thickness of softened layer is higher over the timely mechanical removal amount when pH value is low enough. Therefore, the excessively corrosive surface deteriorates surface quality seriously shown in Fig. 9a.
2. MRR was determined by the rate of "Generating-Removed" circles. The chemical corrosion action dominates at the lower pH value, which makes the thickness of the generated softened layer guaranteed the mechanical removal amount against shortage. Thus, rate of circle is improved, resulting in high MRR. However, this circle is slowing down and MRR decreases with increasing pH value when the thickness of the generated softened layer is not enough for mechanical removal shown in Fig. 8.

From experimental results, both the continuity and rate of "Generating-Removed" circles tended to a relatively stable stage during pH band ranged from 4.0 to 5.0. Among these slurry pHs, the abrasive-free acid slurry with citric acid regulated at pH 5.0 is most effective for fixed abrasive CMP of LBO crystal in consideration of slight surface damage.

4 Conclusions

The effect of various acid regulators on LBO crystal was characterized in the fixed abrasive CMP process, focusing on the kinds of acid and the slurry pH. The following conclusions can be drawn.

a. Of the four acid regulators, citric acid is the most suitable chemical additive, with surface roughness Sa 0.52 nm and MRR 435 nm/min, which exerts more modest chemical activity than others.
b. In citric acid slurry, MRR decreases from 673 to 208 nm/min with increasing pH value from 3.0 to 5.5. Surface roughness Sa initially decreases with increasing pH value,

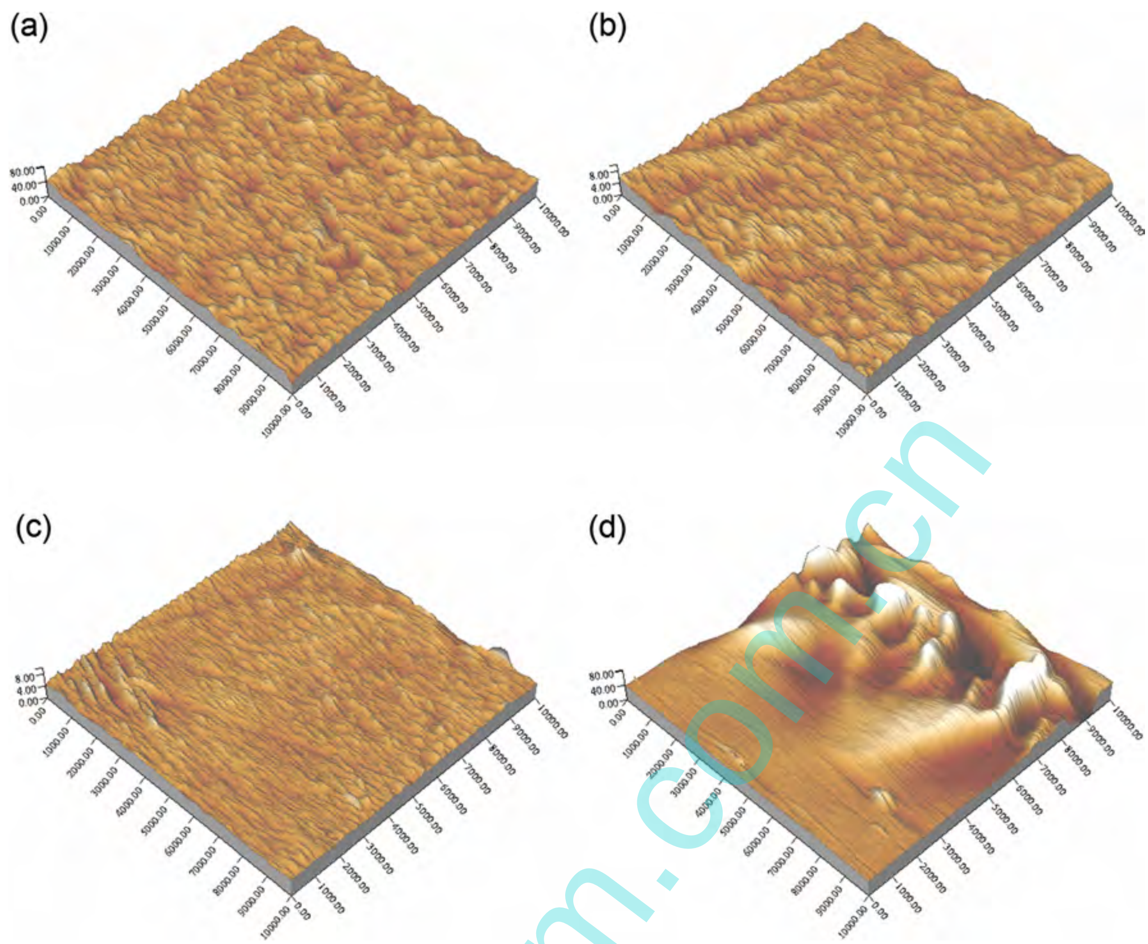


Fig. 10 AFM surface topography of polished surface with different pH values **a** pH 3.0; **b** pH 4.5; **c** pH 5.0; **d** pH 5.5. (Note: Fig. 7c shows pH 4.0)

then it mutates to the maximum 9.37 nm after reach the minimum 0.32 nm.

c. The abrasive-free acid slurry with citric acid regulated at pH 5.0 is effective for fixed abrasive CMP of LBO crystal, obtaining a high surface quality with surface roughness Sa 0.32 nm while maintaining a MRR of 366 nm/min.

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