Effect of Chemical Additive on Fixed Abrasive Polishing of LBO Crystal with Non-water Based Slurry

Jun Li, Wenze Wang, Zhanggui Hu, Yongwei Zhu, Dunwen Zuo

International Science Index Vol:9, No:5, 2015 waset.org/Publication/10001187

***Abstract*—**Non-water based fixed abrasive polishing was adopted to manufacture LBO crystal for nano precision surface quality because of its deliquescent. Ethyl alcohol was selected as the non-water based slurry solvent and ethanediamine, lactic acid, hydrogen peroxide was added in the slurry as a chemical additive, respectively. Effect of different additives with non-water based slurry on material removal rate, surface topography, microscopic appearances, and surface roughness were investigated in fixed abrasive polishing of LBO crystal. The results show the best surface quality of LBO crystal with surface roughness Sa 8.2 nm and small damages was obtained by non-water based slurry with lactic acid. Non-water based fixed abrasive polishing can achieve nano precision surface quality of LBO crystal with high material removal.

***Keywords*—**Non-water based slurry, LBO crystal, Fixed abrasive polishing, Surface roughness.

I. Introduction

NONLINEAR optical (NLO) crystals are mainly used for frequency conversion of lasers. Lithium triborate (LiB_3O_5 or LBO) crystal is a very important and also the most widely applied NLO crystal [1], [2]. 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 [3]. A high surface quality of LBO crystal with nanometer precision is urgently needed because of its applications in high energy laser systems [4].

Abrasive particles are fixed in the polishing pad and chemically active slurry that contains no abrasives flows between the pad and the wafer in fixed abrasive process. One of the advantages of fixed abrasive process is that it is extremely selective to wafer topography. The polishing process stops once planarity is achieved and ensures that any residual material on the wafer surface is removed. This helps not only in achieving total planarity, but also in reduce the overfilling needed, thereby lowering the costs and increasing throughput. Fixed abrasive polishing has the potential to reduce demand on waste treatment that no abrasives adsorbed on the wafer surface because of no abrasives in the slurry [5]. Then the costs are lowered and surface quality of the wafer is enhanced. Another advantages include polishing efficiency, high speed process, temperature stability, cost of consumables, and compatibility with computer numerically controlled generating machines [6], [7]. Owing to the advantages of processing technology, fixed abrasive polishing technology can achieve nanometer precision surface quality of LBO crystal with high processing efficiency [8], [9].

However, water based slurry may leave little water on the crystal surface, which is not good for the application of the crystal because LBO crystal has weak hygroscopy. Li et al. employed fixed abrasive polishing with water based slurry to polish of LBO crystal, and slurry pH regulator, pH value, matrix hardness and polishing powder concentration of fixed abrasive pad were optimized [10], [11]. Wang et al. studied chemical mechanical polishing of $CsLiB_6O_{10}$ crystal by selecting polyethylene glycol as the water-free slurry solvent and organic amine as pH value conditioner [12]. Based on the above, new method of non-water based fixed abrasive polishing was attempted to fabricate LBO crystal for super smooth surface.

A combined mechanical and chemical action leads to material removal in fixed abrasive polishing process. In chemical action, chemical reactions between wafer materials, slurry, particles and the pad play an important role in making the wafer surface flat. And chemical action could be controlled by the chemical additive [13], [14]. In this study, ethyl alcohol was selected as the slurry solvent and ethanediamine, lactic acid, hydrogen peroxide was added in the non-water based slurry as a chemical additive, respectively. Effect of different additives on material removal rate, surface topography, microscopic appearances and surface roughness were investigated for non-water based fixed abrasive polishing of LBO crystal. Interaction mechanism of the additives and LBO crystal was discussed.

II. Experiments

All experiments were conducted on the specific lattice plane (θ=90°, φ=13.8°) of LBO crystal in this study. Fixed abrasive polishing experiment was performed on Nanopoli-100 smart precision lapping/polishing machine and a fixed abrasive pad is introduced as the polishing pad, which has a regular intersecting orthogonal channel pattern with 1 μm CeO_2 [15]. Non-water based slurry mainly contains with ethyl alcohol, surfactant OP-10 agent ($C_{34}H_{62}O_{11}$, a nonionic surfactant) and corrosive agent. The surfactant is used to further reduce the

Jun Li is with College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, 29 Yudao Street, Nanjing, 210016, P. R. China (corresponding author: 86-25-84891686; fax: 86-25-84891501; e-mail: junli@ nuaa.edu.cn).

Wenze Wang is with College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, 29 Yudao Street, Nanjing, 210016, P. R. China (e-mail: xhcszz@163.com).

Zhanggui Hu is with Key Laboratory of Functional Crystals and Laser Technology, TIPC, Chinese Academy of Sciences, Beijing, 100190, P. R. China (e-mail: hu@mail.ipc.ac.cn).

Yongwei Zhu and Dunwen Zuo are with College of Mechanical and Electrical Engineering, Nanjing University of Aeronautics and Astronautics, 29 Yudao Street, Nanjing, 210016, P. R. China

International Science Index Vol:9, No:5, 2015 waset.org/Publication/10001187

surface tension of the pad, which makes the slurry quickly insinuate into the working site. Ethanediamine, lactic acid, hydrogen peroxide (H_2O_2) was added in the slurry as a corrosive agent, respectively. The concentration of each additive was optimized in previous experiments. Table I shows the optimal concentration of each additive was applied in the polishing experiments. Before each polishing, the lapping process is used to uniform morphology, and polishing parameters are listed in Table II.

TABLE I
ADDITIVE CONCENTRATION IN POLISHING EXPERIMENTS

Additive	Concentration (Vol. %)
Ethanediamine	30
Lactic acid	25
Hydrogen peroxide	5

TABLE II
POLISHING PARAMETERS

Polishing Parameters	Parameter Setting
Pad rotation speed (r/min)	70
Eccentric distance (mm)	82
Slurry flow rate (ml/min)	30
Process duration (min)	20
Pressure (kPa)	14

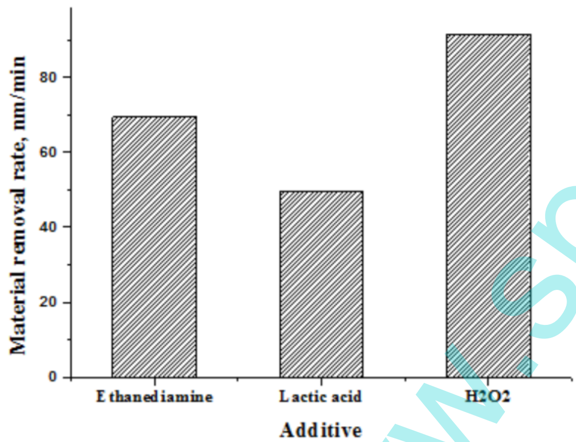


Fig. 1 *MRR* of fixed abrasive polishing of LBO crystal by different non-water based slurry

The mass of LBO crystal is determined by Sartorius BS224S precision balance with a resolution of 0.0001 g. The thickness is measured by electronic digital display micrometer with a resolution of 0.01 mm, which is distributed at eight different locations of each workpiece. The mean values are calculated from three repeated measurements. Material removal rater (*MRR*) is defined as the reduction of thickness per unit time, and calculated using (1):

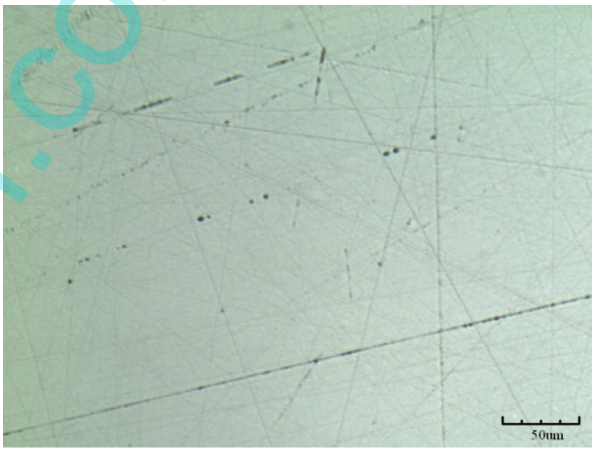
$$MRR = \frac{(m_0 - m_i)\times h_0}{m_0 \times t}\times 10^6 \quad (1)$$

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 (min), and the unit of *MRR* is nm/min. Surface topography of polished surface is observed with a metallographic microscope (XJX-200, Nanjing Jiangnan Novel Optics Co., Ltd., P. R. China). An atomic force microscopy (AFM, CSPM4000, Beijing Nano-Instruments LTD., P. R. 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.

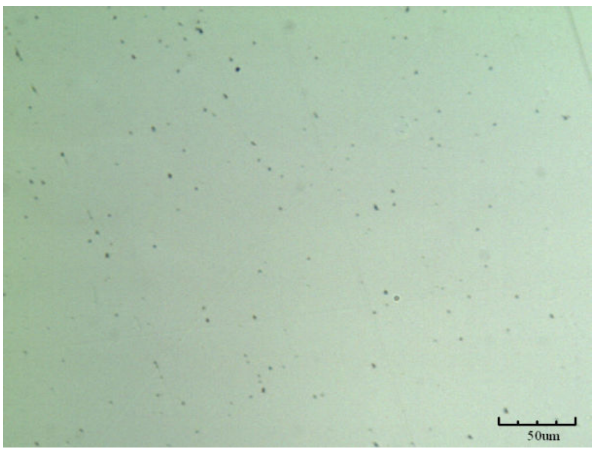
III. RESULTS AND DISCUSSION

Material removal rate of LBO crystal by different slurry in non-water based fixed abrasive polishing process is showed in Fig. 1. The maximum *MRR* of LBO crystal is 92 nm/min polished by H_2O_2 slurry, and the minimum is 50 nm/min by lactic acid slurry.

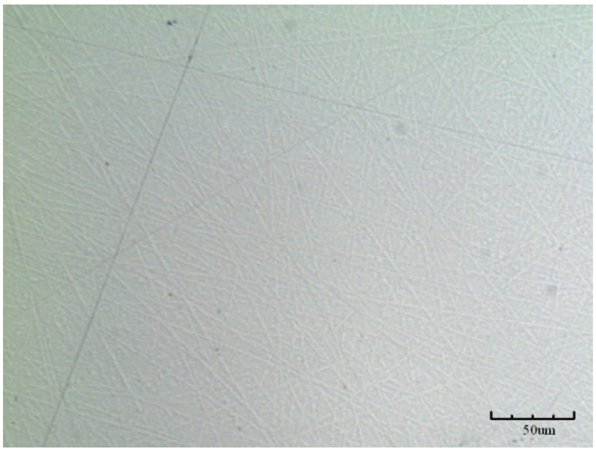
Fig. 2 shows surface topography of LBO crystal after polshing with different slurry was tested by Microscope. There are some big scratches and pits on LBO crystal surface polished by ethanediamine slurry and it is the worst surface quality. There are some traces of corrosion by H_2O_2 slurry. And the best surface quality with only little and small pits is the one by lactic acid slurry.



(a) Ethanediamine

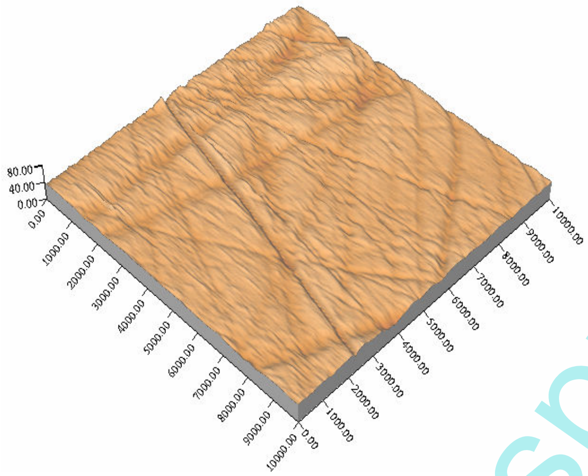


(b) Lactic acid

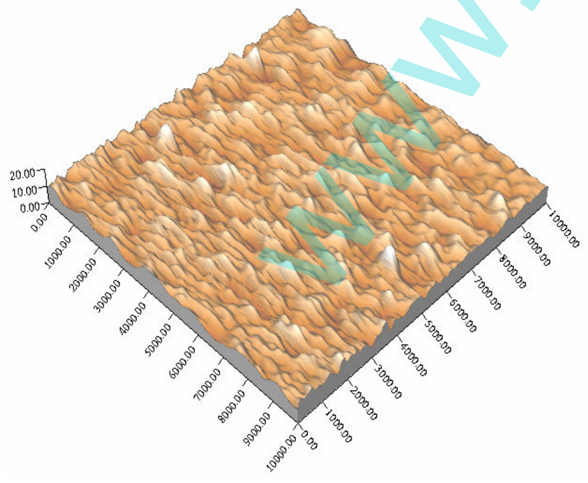


(c) H_2O_2

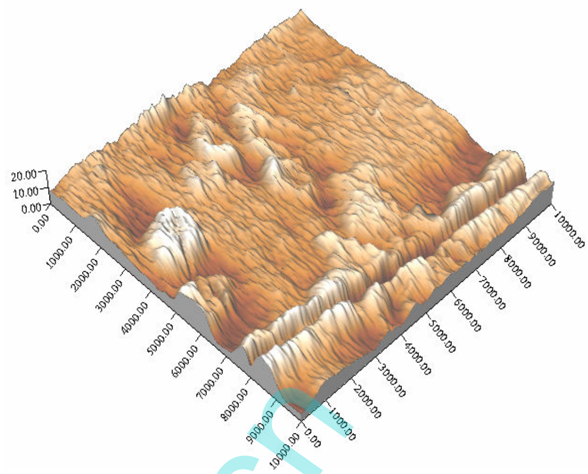
Fig. 2 Surface topography of LBO crystal after non-water based fixed abrasive polishing



(a) Ethanediamine



(b) Lactic acid



(c) H_2O_2

Fig. 3 Microscopic appearances of LBO crystal after fixed abrasive polishing

Microscopic appearances of LBO crystal by AFM were showed in Fig. 3. There are some big micro scratches on LBO crystal surface polished by ethanediamine slurry. And there are some micro scratches and pits on the surface polished by H_2O_2 slurry. And the best surface quality of LBO crystal is the surface polished with the uniform micro pits by lactic acid slurry.

Surface roughness of LBO crystal polished by different slurry is showed in Fig. 4. The worst surface roughness Sa is 11.4 nm polished by ethanediamine slurry. And the other two Sa are nearly, 8.2 nm and 8.7 nm by lactic acid and H_2O_2 slurry, respectively.

Fixed abrasive polishing is a complex mechanical and chemical process, and material removal is a result of combined mechanical and chemical action. The better surface can be obtained only when there is a balance between the two functions. If the mechanical action is oversupplied, the mechanical action can make abrasive particles to scratch the crystal surface. And if the chemical action is larger than the mechanical action, the transition soft layer that the chemical action generates is not promptly removed by the mechanical action [16]. When other polishing parameters are the same, the mechanical action is considered as the same and the chemical action is dependent on the additive.

For LBO crystal, there are two $–NH_2$ in the molecular structure of ethanediamine. And there are a weak chemical action between ethanediamine and LBO crystal, and the abrasives in fixed polishing pad remove the entire transition soft layer and scratch the crystal surface. Some big scratches and pits or some big micro scratches are formed from Fig. 2 (a) or 3 (a) by ethanediamine slurry, which leads to the worst surface roughness from Fig. 4.

International Science Index Vol:9, No:5, 2015 waset.org/Publication/10001187

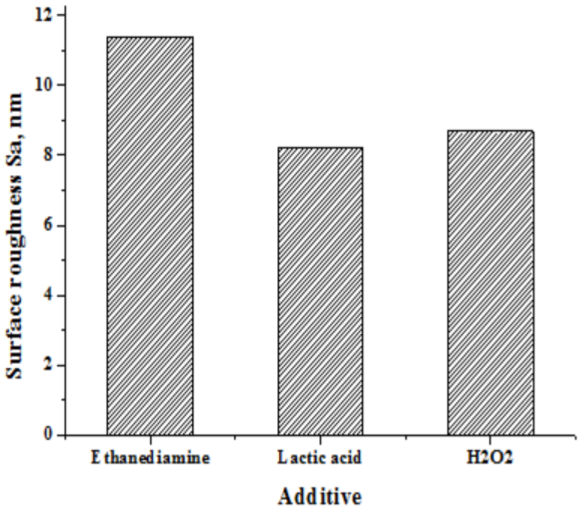


Fig. 4 Surface roughness of LBO crystal after fixed abrasive polishing

Hydrogen peroxide solution is easy to ionize H^+ ion and generate water. LBO crystal encounters water easy hygroscopic. When H_2O_2 was added in the slurry, the corrosion to LBO crystal is strong. The material removal by the chemical action is large and the maximum *MRR* was obtain among the three slurries from Fig. 1. The corrosion layer that the chemical action generates is not promptly removed by the mechanical action and some traces of corrosion are retained from Fig. 2 (c). Because surface quality is better generated by the chemical action than that of the mechanical action, surface roughness is better by H_2O_2 slurry than that of ethanediamine slurry from Fig. 4.

Lactic acid is a weak organic acid and its corrosion to LBO crystal is moderate. When lactic acid is added in non-water slurry, the mechanical and chemical actions are nearly. There are only little and small pits on LBO crystal surface from Figs. 2 (b) and 3 (b) and the best surface roughness was obtained from Fig. 4. The best surface quality was achieved by lactic acid slurry in non-water based fixed abrasive polishing process for nano machining LBO crystal.

IV. Conclusions

Non-water based fixed abrasive polishing with different additive, ethanediamine, lactic acid, hydrogen peroxide, was studied for nano precision surface quality of LBO crystal. Effect of different additives with non-water based slurry on material removal rate, surface topography, microscopic appearances and surface roughness was discussed in fixed abrasive polishing LBO crystal. Lactic acid is effective for non-water based fixed abrasive polishing of LBO crystal, obtaining a fine surface quality with surface roughness Sa 8.2 nm with rather slight surface damage and *MRR* of 50 nm/min. The nanometer precision surface quality with high material removal was obtained in fixed abrasive polishing of LBO crystal with non-water based slurry.

Acknowledgment

This project is supported by Key Program of National Natural Science Foundation of China (51132005) and National Natural Science Foundation of China (50905086, 51175260 and 51375237). This research is supported by Priority Academic Program Development of Jiangsu Higher Education Institutions (PAPD). The authors are also grateful for support from China Postdoctoral Science Foundation (2014M551586) and Postdoctoral Science Foundation of Jiangsu province (1302069B).

References

[1] C. T. Chen, Y. C. Wu, A. D. Jiang, B. C. Wu, G. M. You, R. K. Li and S. J. Lin "New nonlinear-optical crystal: LiB3O5." *J. Opt. Sco. Am.*, (B6), pp. 616-621. (1989).

[2] Z. Hu, Y. Zhao, Y. Yue and X. Yu "Large LBO crystal growth at 2 kg-level." *Journal of Crystal Growth*, vol. 335, (1), pp. 133-137. (2011).

[3] Z. G. Hu and Y. C. Wu " Review of the research on the nonlinear optical crystal: LiB3O5." *Mater. China*, vol. 29, (8), pp. 12-17. (2010).

[4] D. Liao, R. Xie, J. Hou, X. Chen and B. Zhong "A polishing process for nonlinear optical crystal flats based on an annular polyurethane pad." *Appl. Surf. Sci.*, vol. 258, (22), pp. 8552-8557. (2012).

[5] W. Huang, S. Tamilmani, C. Anderson, and S. Raghavan "Material removal and articulate generation during abrasion of copper films using a fixed abrasive pad." IEEE Trans. Semicon. Manu,. vol.17, pp. 525-530. (2004).

[6] B. Gillman and S. Jacobs "Bound-abrasive polishers for optical glass." *Appl. Opt.,* vol. 37, pp. 3498-3505. (1998).

[7] X. Wang and X. Zhang "Theoretical study on removal rate and surface roughness in grinding a RB-SiC mirror with a fixed abrasive." *Appl. opt.,* vol. 48, pp.904-910. (2009).

[8] B.-J. Cho, H.-M. Kim, R. Manivannan, D.-J. Moon and J.-G. Park "On the mechanism of material removal by fixed abrasive lapping of various glass substrates." *Wear*, vol. 302. (2013).

[9] J. Li, P. Gao, Y. W. Zhu, B. Li, Y. L. Sun and D. W. Zuo "Research on subsurface damage after abrasives and fixed-abrasive lapping of K9 glass." *Key Eng. Mater.*, vol. 487, pp. 253-256. (2011).

[10] J. Li, B. Li, Z. Hu, Y. Zhu and D. Zuo "Optimization of FAP in Nano Machining Process." *Integrated Ferroelectrics*, vol. 152, (1), pp. 43-50. (2014).

[11] J. Li, B. Li, Z. Hu, Y. Zhu and D. Zuo "Optimization of Fixed Abrasive Polishing Slurry for LiB3O5 Crystal." *J Chin Ceram Soc*, vol. 41, (6), pp. 789-796.(in Chinese). (2013).

[12] S. Wang, Y. Liu, X. Niu and B. Tan "Slurry and processing technique of CLBO crystal." *Trans. Nonferrous Met. Soc. china*, vol. 16, (Supplement 2), pp. s692~s695. (2006).

[13] Y. Tian, Z. Zhong, S. Lai, Y. Ang "Development of fixed abrasive chemical mechanical polishing process for glass disk substrates." *Int J Adv Manuf Technol,* vol. 68, (5-8), pp. 993-1000.(2013).

[14] J. Li, Y. Liu, Y. Pan, X. Lu "Chemical roles on Cu-Slurry interface during copper chemical mechanical planarization." *Appl Surf Sci,* vol. 293, pp. 287-292.(2014).

[15] J. Li, Y. W. Zhu, D. W. Zuo, K. Lin and M. Li "Fixed abrasive lapping and polishing of hard brittle materials." *Key Eng. Mater.*, vol. 426-427, pp. 589-592. (2010).

[16] J. Li, Y. W. Zhu, D. W. Zuo, Y. Zhu and C. T. Chen "Optimization of polishing with Taguchi method for LBO crystal in CMP." *J. Mater. Sci. Technol.*, vol. 25, (5), pp. 703-707. (2009).