Study on ultra-precision water dissolution polishing of KDP crystals

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Potassium dihydrogen phosphate (KDP) crystals have attracted much attention in the field of laser systems due to their excellent nonlinear optical properties, high laser damage threshold, and outstanding transparency and birefringence across the near-infrared to ultraviolet wavelength range. However, KDP crystals are soft, brittle, and easily soluble in water, making it difficult to obtain high-quality surfaces. In this study, a polishing solution composed of ethanol and water was utilized. Through a two-step abrasive-free water dissolution polishing technique, a high-quality KDP crystal surface with a surface roughness of 2.78 nm in Sa was obtained. The effectiveness of the cleaning method was experimentally verified, and the influence of ethanol content in the polishing solution on surface roughness and material removal rate was also investigated. This work provides theoretical basis and technical support for the ultra-precision machining of KDP crystals.

1. Introduction

Potassium dihydrogen phosphate (KDP) crystals exhibit exceptional optical properties, high laser damage thresholds, and the capability to grow large single-crystal ingots [1], making them a high-quality nonlinear optical material with broad application potential in fields such as communications, medicine, and scientific research [2]. In the 1960s, with the advent of laser technology, KDP crystals were widely employed in solid-state lasers for frequency conversion and electro-optic switching, owing to their outstanding electro-optic properties [3]. Furthermore, KDP crystals are indispensable in critical components such as electro-optic modulators and high-speed photographic shutters [4]. Consequently, research and exploration into high-quality KDP crystal processing techniques have become a key focus in the scientific community, attracting considerable interest from the research community.

In laser systems, the surface quality of optical components is subject to stringent requirements. To achieve a high laser damage threshold, it is crucial to strictly control their surface roughness and minimize subsurface damage (SSD) to the greatest extent possible [5]. However, processing KDP crystals poses significant challenges. With a Mohs hardness of only 2.5, KDP crystals are soft and brittle, which makes them highly susceptible to damage during machining processes. Additionally, KDP crystals exhibit strong anisotropy, are highly sensitive to temperature changes, and are easily soluble in water. Their surfaces are prone to absorbing moisture from the air, resulting in deliquescence. These characteristics present considerable challenges in both the processing and preservation of KDP crystals.

Currently, conventional KDP crystal processing techniques mainly consist of single-point diamond turning (SPDT), ultra-precision grinding, magnetorheological finishing (MRF), and ion beam polishing [6]. These techniques can yield favorable processing outcomes; however, each method has certain limitations. For instance, surfaces processed by SPDT often exhibit small-scale cutting textures that are unavoidable. Grinding and MRF may cause surface scratches and abrasive particle embedding [7]. Although ion beam polishing provides high precision, it is associated with complex equipment, low processing efficiency, and potential thermal effects that can damage the material [8].

Gao [9] et al. proposed an abrasive-free jet polishing technique that effectively removes turning grooves on KDP crystals generated by SPDT, however, the use of oily polishing solution may contaminate the polished surface. Liu [10] et al. introduced a polishing technique for KDP crystals based on gas-liquid two-phase flow, which can reduce the surface roughness (Ra) to 4 nm. These polishing methods based on water dissolution provide new approaches for the processing of KDP crystals. However, the challenge of quickly removing macroscopic pre-treatment damage and obtaining a high-quality surface without complex cleaning steps remains unresolved.

In order to solve the above problems, this study utilizes a mixture of ethanol and water as the polishing solution. This method effectively polishes KDP crystals without the use of abrasives or other chemical contaminants. By employing an ethanol rinse followed by drying, the issue of polishing solution residue affecting surface quality is effectively mitigated. The study investigates the effect of ethanol concentration in the polishing solution on surface roughness and material removal rate with a self-constructed polishing apparatus. Ultimately, a two-step polishing process results in a high-quality surface with a surface roughness of 2.78 nm in Sa.

2. Principles of Material Removal and Surface Finishing

As shown in Fig. 1, during the dissolution process of KDP crystals, the concentration of the solution near the surface gradually increases, forming a high-concentration solution layer. Due to the relatively slow diffusion of substances at the valley positions of the crystal, these areas are more prone to forming high-concentration solution layers, which slow down the further dissolution of the crystal. This variation in dissolution rates provides some surface-flattening capability. Due to the instability of the solution flow and defects on the crystal surface, this flattening effect is often inadequate for achieving a high-quality surface. Moreover, it is difficult to obtain an ideal surface profile solely through dissolution. However, when the polishing pad is in use, as illustrated in Fig. 1c, the high-concentration solution layer in the peak regions of the crystal surface is effectively removed by the polishing pad, thereby enhancing the dissolution rate in these peak regions. In contrast, the valley regions, because they have fewer opportunities to contact the polishing pad, are protected by the high-concentration solution layer, resulting in a reduced dissolution rate. This differential material removal rate facilitates the finishing of the KDP crystal surface.

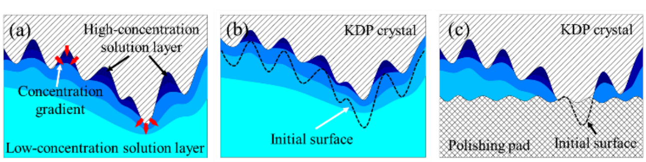

Fig. 1 Schematic diagram of material removal and surface finishing principles. (a) The dissolution process of KDP crystals. (b) Comparison of surface morphology before and after KDP crystal dissolution. (c) The material removal process of KDP crystals under the action of polishing pad

3. Experimental Approach

3.1 Materials and equipment

All experiments and measurements were conducted in a Class 1000 cleanroom, with a controlled relative humidity of 30% and a maintained temperature of 22°C. This controlled environment prevents deliquescence of KDP crystals.

The KDP crystals, oriented along the (001) crystal plane, were provided by the Shanghai Institute of Optics and Fine Mechanics (SIOM). The crystals were cut using a diamond wire saw to a size of 15 mm × 15 mm × 15 mm. A white high-density polyurethane polishing pad was used. The polishing solution was a mixture of deionized water and ethanol (analytical grade) at a specific ratio. To ensure consistent initial conditions, the crystal surfaces were pretreated with 4000 mesh sandpaper. The surface morphology after pre-processing is shown in Fig. 2. To ensure the reliability of the experimental results, each experiment was repeated three times.

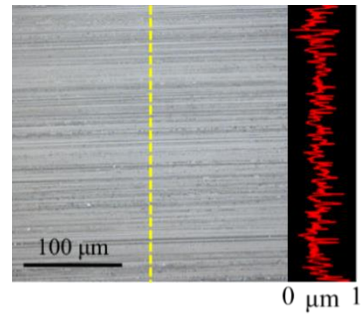

Fig. 2 Surface morphology of KDP crystals after polishing with 4000 mesh sandpaper, with a surface roughness of 99 nm in Sa

The polishing equipment used in this experiment, as shown in Fig. 3. The polishing apparatus primarily consists of a top disc, a bottom disc, an electric Z-axis displacement stage, and a recovery unit. The polishing pad is fixed on the bottom disc, while the KDP crystal is secured in the fixture. The fixture's height is adjusted via the electric Z-axis displacement stage to ensure optimal contact between the KDP crystal and the polishing pad. The recovery unit efficiently collects excess polishing solution. Under computer control, the polishing pad revolves with the lower plate at a specified speed, while the KDP crystal rotates in unison with the polishing pad. The distance between the centers of revolution and rotation is 25 mm. The polishing solution is uniformly applied to the polishing pad in a spray pattern based on the Bernoulli principle.

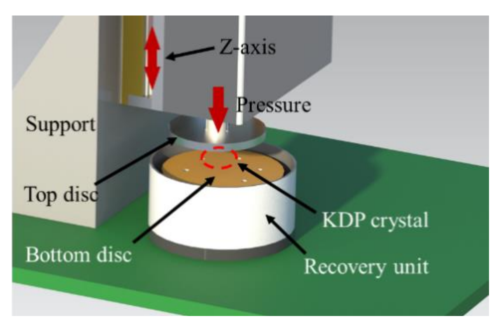

Fig. 3 Schematic diagram of the polishing system

The surface morphology of the KDP crystal was observed using a laser scanning confocal microscope (Olympus, OLS 4000). Surface roughness information of the KDP crystal was obtained using a white light interferometer (Veeco, NT9300) with 50× magnification and 1× field-of-view multiplier in phase-shift interferometry mode. The material removal rate (MRR) was calculated using an electronic balance with a measurement accuracy of 0.1 mg, and a drying oven (Huamai, 101-1B) was used to treat the polished crystals.

3.2 Experimental investigation

Residue from the polishing solution can adversely affect the surface quality and optical performance of the crystal, making it necessary to clean the processed crystals. In this experiment, anhydrous ethanol was used to rinse the crystals. The cleaned crystals were then placed in a drying oven to ensure the surface was fully dried. To verify the effectiveness of the cleaning method, comparative experiments were conducted. The surface morphology of a KDP crystal was initially measured using a white light interferometer. Then, polishing solution was applied to the crystal surface to simulate the presence of residual polishing solution. The crystals were subjected to in-situ measurements after both immediate cleaning and delayed cleaning (1 minute delay), and the results were compared to the initial surface morphology.

To investigate the effect of water content in the polishing solution on polishing efficiency and surface roughness, a single-variable experimental design was employed. Under constant conditions of a polishing pressure of 600 g, a polishing time of 10 minutes, and a lower plate revolution speed of 30 rpm, the ethanol-to-water volume ratio in the polishing solution was systematically varied. The ratios tested were 1:1, 2:1, 3:1, 4:1, and 5:1.

Table 1 Parameters for two-step polishing process

Factors	First step	Second step
Volume ratio of ethanol to water	3:1	4:1
Polishing pressure (g)	600	600
Revolution speed (rpm)	30	30
Polishing time (min)	10	20

Based on the single-variable experiments, a two-step polishing process was applied to the KDP crystals, with the detailed parameters provided in Table 1. The first polishing step utilized a low-concentration polishing solution to rapidly remove the initial surface texture. In the second polishing step, a higher concentration polishing solution was employed to further decrease the surface roughness of the KDP crystals.

4. Results and Discussion

4.1 Precision cleaning of KDP crystals after processing

Ethanol, a common organic solvent, does not introduce additional chemical contaminants. Thus, anhydrous ethanol was chosen as the cleaning agent in this study. The crystals were rinsed with anhydrous ethanol. The cleaned samples were then placed in a drying oven to remove any residual ethanol. Fig. 4b shows the surface after the polishing solution was immediately cleaned off, exhibiting surface morphology and roughness consistent with the initial surface (Fig. 4a). No dissolution pits or precipitated KDP were observed, indicating that the cleaning method effectively and thoroughly removes residual polishing solution, thereby supporting subsequent processing.

KDP crystals exhibit significant hydrophilicity, resulting in residual polishing solution remaining on the surface and around the crystal after polishing. This residual polishing solution may continue to dissolve the crystal, and upon evaporation, dissolved KDP can precipitate onto the crystal surface, severely affecting surface quality. Fig. 4c shows the surface after delayed cleaning, where the precipitated KDP is distributed in a dot-like pattern, which significantly increases surface roughness.

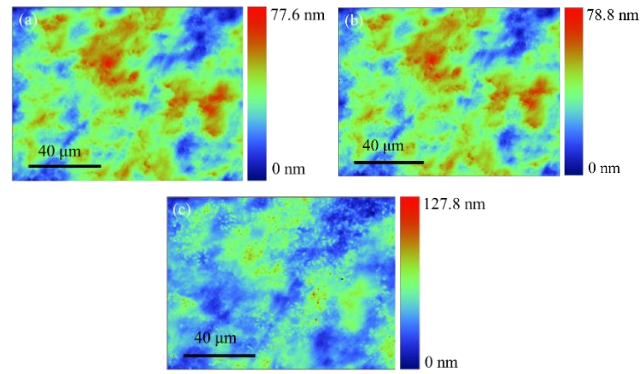

Fig. 4 White light interferometer measurement results of the KDP crystal before and after cleaning: (a) Initial surface with a surface roughness of 7.65 nm in Sa; (b) Surface immediately cleaned after the addition of the polishing solution, with a surface roughness of 7.67 nm in Sa; (c) Surface cleaned 1 minute after the addition of the polishing solution, with a surface roughness of 8.82 nm in Sa

4.2 Impact of water content in polishing solution

Fig. 5 demonstrates how varying ethanol content in the polishing solution influences both surface roughness and material removal rate. Experimental findings show that as the ethanol-to-water volume ratio in the solution increases from 1:1 to 3:1, there is a corresponding decrease in the material removal rate and surface roughness. At lower ethanol concentrations, the KDP dissolution rate is relatively high, which leads to less effective surface finishing. With higher ethanol content, the polishing solution more easily forms a high-concentration layer on the surface of the sample. This layer slows down the material removal rate and enhances the selectivity of the removal process. However, at an ethanol-to-water ratio of 4:1, the lower material removal rate prevents the full elimination of the initial surface features within a 10-minute polishing period. While increasing the polishing duration might improve surface quality, overly long polishing times are not efficient. When the ethanol-to-water ratio is further increased to 5:1, the resulting surface roughness is nearly the same as the initial surface, and the material removal rate is almost negligible. This indicates that when the water content in the polishing solution is too low, the solution becomes ineffective at dissolving the material, causing both material removal and surface finishing processes to halt.

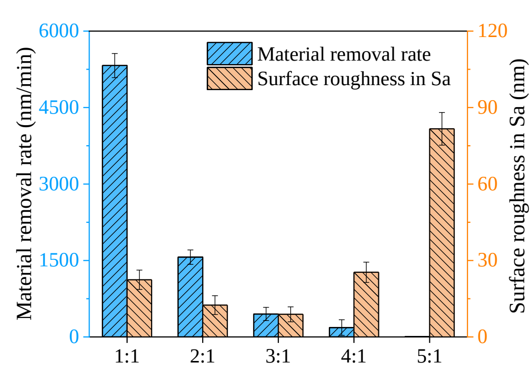

Fig. 5 Impact of the volume ratio of ethanol to water in the polishing solution on surface roughness in Sa and MRR of KDP crystals

4.3 Two-step polishing

Based on this, a two-step polishing process was applied to the KDP crystal blanks. In the first step polishing, a low-concentration polishing solution was used to quickly eliminate macroscopic structures and existing defects. As shown in Fig. 6a, after 10 minutes of the first step polishing, the regular scratches on the initial surface were completely removed, and the surface roughness in Sa rapidly decreased from 100 nm to below 10 nm, with a material removal rate of approximately 450 nm/min. This laid a solid foundation for the second step polishing. In the second step polishing, by increasing the concentration of the polishing solution, the surface quality of the KDP crystals was further improved. The material removal rate decreased to 183 nm/min, and after 20 minutes of the second step polishing, a final surface roughness of 2.78 nm in Sa was achieved, as shown in Fig. 6b.

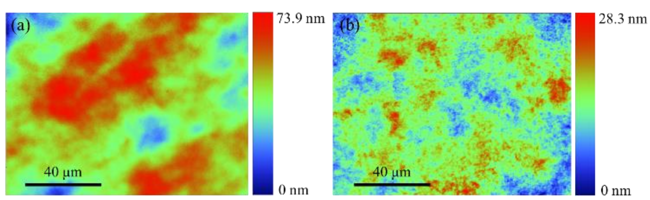

Fig. 6 Surface morphology of the KDP crystal after different polishing steps: (a) After the first step, with a surface roughness of 8.53 nm in Sa; (b) After the second step, with a surface roughness of 2.78 nm in Sa

5. Conclusions

In this study, a mixture of ethanol and water was used as the polishing solution to achieve high-quality KDP crystal surfaces. The experiment validated the effectiveness of the cleaning method, investigated the impact of ethanol content in the polishing solution on surface roughness and material removal rate, and preliminarily explored the effect of a two-step polishing process on surface quality. The main conclusions are as follows:

(1) The ethanol content in the polishing solution influences polishing efficiency and quality. Lower ethanol content results in higher polishing efficiency but poorer surface quality, whereas higher ethanol content yields better surface quality but lower processing efficiency. If the ethanol content is too high, the process becomes ineffective.

(2) The two-step polishing process can improve processing efficiency while ensuring surface quality, resulting in a high-quality surface with a surface roughness of 2.78 nm in Sa from an initially sanded surface in under 30 minutes.

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