Study on cleaning of residual from magnetorheological finishing (MRF) of potassium dihydrogen phosphate(KDP) crystal surface

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Abstract: KDP is a type of excellent nonlinear single crystal electro-optical material, which is used in high-energy laser systems. However, KDP is extremely difficult to be finished for its particular physical and chemical properties. Magnetorheological finishing (MRF) is considered to be an effective processing method for finishing KDP to high precision at present. But the performance of KDP would decrease obviously if the residual contamination remained after finishing. An active cleaning agent with high-frequency ultrasound technology was put forward to solve the problem. Microscope and white-light-interferometer were employed to assess the effects of cleaning process on KDP surface. The results show that the residual contamination is removed availably, and the surface roughness is improved as well. Additionally, the Raman spectral and laser interferometer analysis indicates that crystal structure and surface accuracy of KDP remain unchanged after cleaning. These conclusions prove that the cleaning method achieves desired result and exhibits a promising prospect of KDP cleaning.

Key words: optical crystal; cleaning; megasonic; KDP; MRF

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KDP 晶体表面残留磁流变抛光液清洗技术研究

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摘 要: 磷酸二氢钾(KDP)晶体是一种非常优良的非线性光学晶体材料,在强激光系统中有着重要应用。但其理化性质特殊,加工高表面质量的元件难度很大。目前研究认为磁流变抛光技术能够获得高精度的 KDP 晶体表面,但是该加工过程带入的表面残留如不清除,会大大降低元件的性能。提出了一种针对磁流变抛光后续的 KDP 的清洗技术,设计了含水活性清洗液,结合超高频超声作用共同去除 KDP 表面残留。光学显微镜和白光干涉仪分析表明,该技术较好地去除了表面残留,并且晶体表面粗糙度有所降低。拉曼光谱和激光干涉仪测试表明,清洗前后 KDP 表面化学结构和面形精度一致。该研究获得了较为满意的结果,说明该方法是一种有应用前景的 KDP 超精密清洗方法。

关键词: 光学晶体; 清洗; 兆声; KDP; MRF

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0 Introduction

KDP is a significant optical material in high-density laser systems. In the past decades, it has been widely used as frequency converters and photoelectric switches in the National Ignition Facility (NIF) at Lawrence Livermore National Laboratory (LLNL). As we know, KDP is difficult to be polished due to its low hardness, temperature sensitivity, high brittleness, and water solubility, which raise a huge challenge in regards to its fabrication for high precision. Researchers have been promoted to improve the surface quality of KDP. Currently, single-point diamond turning(SPDT) is the most reliable method for KDP precise manufacturing[1-5]. But SPDT tool marks comprise a set of surface flaws, sub-surface defects that shorten the life of optics if used in high-fluency laser applications. Magnetorheological finishing (MRF) is a novel technology which has been proved that it can wipe out turning grooves and relieve sub-surface defects. But iron powder and organic liquid from MRF fluids are left inevitably, particularly the iron powders are easily embedded into the soft surface of KDP crystal, resulting in that the laser-induced damage resistance would be greatly decreased. Therefore, the cleaning of residue from KDP after MRF is of vital importance[6-8].

Wang et al. have demonstrated different methods to clean the KDP surface which had been machined with lapping and polishing processing, and the results showed that wiping with lens paper, alcohol cotton and ultrasonic cleaning destroyed the surface at a certain extent[9]. Yuan et al. put forward to use ion beam figuring (IBF) to bombard KDP surface after MRF, which indicated that the iron powder on the surface had been almost cleaned on the whole, whereas inefficiency of this method restricts its practical application[10]. LLNL proposed that ultrasound as well as solvent atomizing with toluene could be an efficient way to remove the KDP scrap and particles from circumstance after SPDT. Though the cleaning technology of surface residue is a rewarding problem to improve the machined surface and expand its engineering application, fewer studies have focused on the KDP cleaning after MRF.

The finishing of fragile materials generally causes a defected layer in the near surface region of optical parts, including sub-surface and contamination which are found to the the governing factors of damage performance for optics when being subjected to intense irradiation. Regarding the research of fused silica (FS) surface process, acid etching is the most mature technology that can reduce the defects after MRF. Although the chemical etching usually deteriorates the surface shape and roughness, the cleanliness of optical element is evidently improved. Extraordinarily, the ultrasonic vibration compared with static etching is found to play a catalytic role which allows acid to enter the narrow micro-cracks. As a result, the sub-surface defects are removed and the damage threshold is improved[11-14].

Ultrasound cleaning is widely used in manufacturing industry by vibration and cavitation to enhance the cleaning process. But common ultrasound frequency is from 20 kHz to 100 kHz and a great quantity of cavitation occurring in this range would destroy the surface of fragile material. Till the 1980s, megasonic came up in the semiconductor cleaning due to the higher frequency may cause the fewer cavitation bubbles which will reduce the damage of surface. Hence, high-frequency megasonic vibration cleaning is employed to thoroughly remove the residual particles and chemicals.

Similarly to the cleaning of FS with etching under the assistance of ultrasound, an idea is proposed for KDP cleaning. In the study, a novel active cleaning agent combined with megasonic vibration process is attempted to clean out the residual of KDP after MRF.

1 Experiment

1.1 Principle of cleaning agent

To etching KDP surface, "etching" agent and solvent should be selected appropriately, otherwise the optical component may lose its high precision or have cracks on the surface.

Deionized water is considered as an ideal constituent in the active agent. Because the solubility of KDP in water is about 26 g and a small amount of water is enough for the cleaning. Moreover no new impurities would be introduced during the process. The effect of solvent is to regulate and control the reaction rate between solvent and KDP interface, and dissolve the remaining organic liquid from MRF fluids. Lower molecular mass and poor hygroscopicity are under consideration too. The organic solvent containing hydroxyl and short hydrocarbon chains is preferable for cleaning agent according to similar dissolve mutually theory[15].

1.2 Experiment section

The KDP crystal samples with the dimensions of 70 mm×70 mm are finished with self-developed MRF apparatus. After MRF, the samples are immediately rinsed and soaked in the cleaning agent with the water content of 3%, and then cleaned via megasonic (200–1 300 kHz) for 3 minutes by two times to absolutely remove the residuals. Finally, the samples are transferred to accomplish the vacuum drying for 36 hours to release the residual stress. The cleaning process is conducted in an acceptable humidity range.

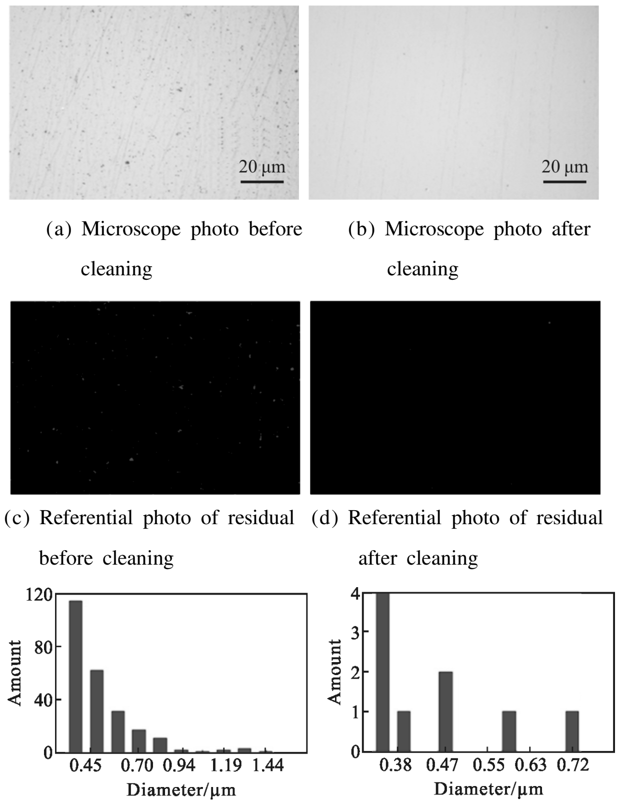
Microscope and white light interferometer are used to analyze the performance of residual removal, while Raman spectroscopy and laser interferometer are conducted to distinguish surface structure and accuracy before and after the cleaning progress.

2 Results and discussion

2.1 Cleaning effectiveness

During the MRF process, there are a certain extent amount of embed particles and remaining organics on the surface which cover the real machined surface and would affect the optical performance of the KDP crystal. Because organics are easier to be cleaned, we paid more attention to the removal of surface particles. The results are analyzed with Olympus MX40 and particle statistical analysis software.

The surface of uncleaned KDP is shown in Fig.1(a), 1(c) and 1(e). The microscope photo indicates that there are many micron-sized particles distributed randomly on the surface, as well as some superficial scratches left by finishing. However, the appearance of KDP surface after megasonic cleaning as shown in Fig.1(b), 1(d) and 1(f) show that the residual on the sample are significantly reduced and the scratches are ameliorated. Compared with the sample without megasonic cleaning, the statistic number of residual particles is decreased from 244 to 9, and the particles which are larger than 0.72 μm have been cleared completely.



(a) Microscope photo before cleaning
(b) Microscope photo after cleaning
(c) Referential photo of residual before cleaning
(d) Referential photo of residual after cleaning
(e) Residual statistics before cleaning
(f) Residual statistics after cleaning

Fig.1 Particles distribution before and after megasonic cleaning

The probable cleaning process may be illustrated as below. KDP is a typical solubility material and its crystal structure would be destroyed at the existence of water. Corresponding ions such as K^+, H^+ and HPO_4^{2-} are ionized from the KDP surface as illustrated in Eq.(1). With the assistance of megasonic, these free ions transfer to the cleaning agent quickly until the residual embedded particles are exposed completely and moved away from KDP surface. According to Fig.1, it also declares that the brightness and non-crazing KDP surface is attributed to accurate control of water content and parameters of megasonic vibration.

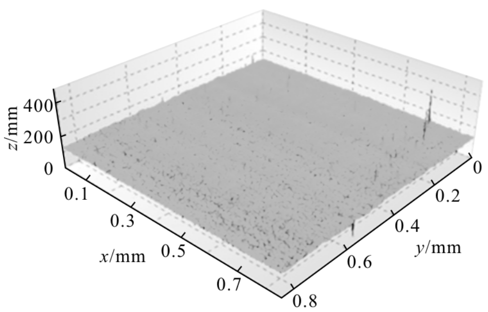
$$KH_2PO_4 \leftrightarrows K^+ + H_2PO_4^- \leftrightarrows K^+ + H^+ + HPO_4^{2-} \leftrightarrows K^+ + 2H^+ + PO_4^{3-} \quad (1)$$

2.2 Surface roughness

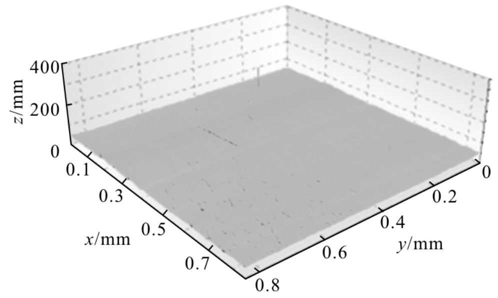
Talyor white light interferometer is applied for the analysis of roughness. Each sample has tested for 5 times and the data are shown in Tab.1. The average values of surface roughness before and after cleaning are 5.969 nm and 3.286 nm respectively, with the notable decreasing rate of more than 44%. We can also get the corresponding surface texture in the 3D morphology. It is found that there are plenty of convex particles on the original finished surface (Fig.2(a)), while the surface turns to be more neat and clean with adequate cleaning (Fig.2(b)) which is consisted with the roughness decreasing. The disappearance of particles and improvement of roughness clearly demonstrates the cleaning ability of above method.

Tab.1 Surface roughness of KDP

Roughness/nm	Point 1	Point 2	Point 3	Point 4	Point 5
Before cleaning	5.67	5.936	5.593	6.435	6.211
After cleaning	3.121	3.887	2.904	2.841	3.676



(a) RMS=5.670 nm



(b) RMS=3.121 nm

Fig.2 3D morphologies of KDP before (a) and after cleaning (b)

2.3 Figure accuracy and structure

As we know, KDP is very sensitive to external forces, so it is necessary to affirm whether there are alterations of surface accuracy when megasonic vibration cleaning has been completed. ZYGO laser interferometer is employed to accomplish the measurement. The cleaned sample is placed statically for more than 36 hours to release the stress adequately before the test. The results of Fig.3 indicate that the initial figure accuracy for a 70 mm×70 mm KDP is 0.657λ while the final figure accuracy is 0.663λ. The slight difference of data demonstrates that there is little changes in surface accuracy, and the cleaning process is feasible for KDP.

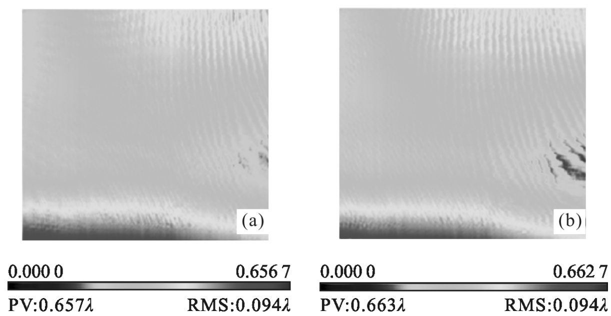


Fig.3 Figure accuracy before (a) and after cleaning (b)

In Fig.4, the peaks of the two spectra before and after cleaning are highly overlapped at 350, 480, 920 cm^{-1} which are the characteristic Raman spectra of KDP crystal [16]. Furthermore, no new Raman peaks are shown in the spectrum which also means no pollutant or deposition is produced on the surface either. This phenomenon indicates that the procedure could be an applicable way for removing residual without variation of KDP crystal structure.

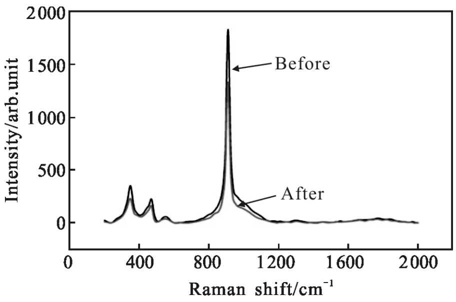


Fig.4 Raman spectra of KDP

2.4 Cleaning mechanism

A nano-indentation experiment is carried out in order to evaluate the feasibility of cleaning mechanism. A sample with indentations which is prepared to simulate the marks of embed particles is soaked in the cleaning agent for 1 h. The surface morphology is shown in Fig.5, the depth of indentations are about 600 nm as shown in the inserted figure of Fig.5(a).

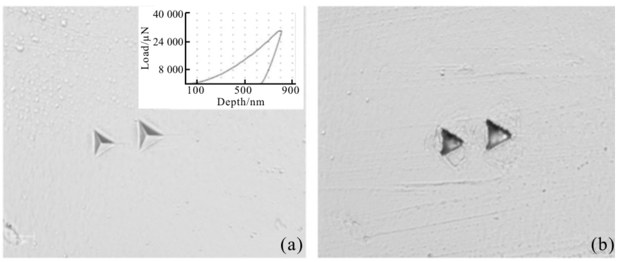


Fig.5 Microscope photo of KDP before (a) and after soaking (b)

The two photos in Fig.5 show that the whole surface of KDP is generally neat and clean, and there is no evident change except for the indentations. It can be seen that the sharp edge of the indentations are turned to be blurred and wrinkles are growing around. These phenomenon imply that the unstable state of indentations where defects concentrated provide more sites for chemical reaction than the other areas. The faster the reactions are, the more crystal is dissolved at the interspace between particles and KDP surface. Therefore the particles are easier to be exposed from the extending interspace.

The accumulation nearby also indicates the diffusion of dissolved KDP is not quickly enough to be removed, so the assistance of megasonic vibration is necessary to achieve more precision surface.

According to the analysis above, the cleaning mechanism is deduced as Fig.6. Firstly, the residuals deposited on the finished KDP surface, then a thin layer of KDP is slowly dissolved in cleaning agent with a little water and the particles are exposed. Finally, the particles and residual organics from magnetorheological fluids are removed and the redeposition is suppressed under the assistance of megasonic vibration.

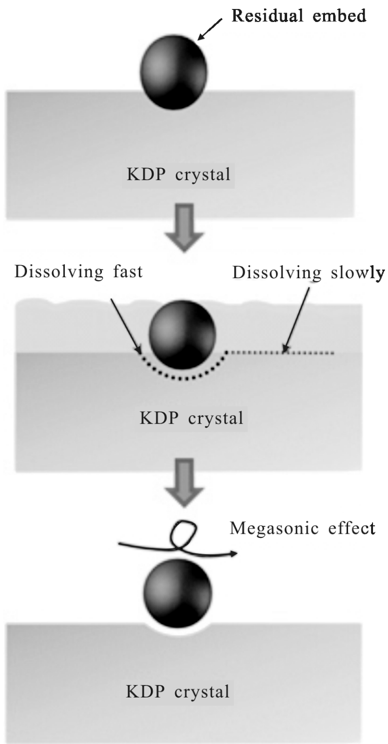


Fig.6 Schematic depiction of cleaning mechanism

3 Conclusion

The cleaning of residual arising from magnetorheological finishing process is a difficult work due to the unique physical and chemical properties of KDP. In this paper, KDP crystal is cleaned by a novel active agent assisted with megasonic vibration. Some essential analysis technologies have been explored to investigate the effects of the cleaning process.

The KDP surface is inevitably stained by residue such as particles after MRF. The cleaning agent containing a little water could dissolve and extend the microcosmic interface between the embedded particles and KDP surface under controlled reaction rate, which is benefit for the removal of above residual. Statistic result and 3D morphology indicate that most of the residual contaminations have been eliminated after cleaning, while the crystal structure and surface accuracy remain unchanged.

The above conclusions confirm that an appropriate KDP cleaning method may achieve the desired result and exhibit a promising prospect in KDP engineering application.

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