

## Investigation on the cleaning of KDP ultra-precision surface polished with micro water dissolution machining principle

CHEN YuChuan, GAO Hang<sup>\*</sup>, WANG Xu, GUO DongMing & TENG XiaoJi

*Key Laboratory for Precision and Non-Traditional Machining Technology of Ministry of Education, School of Mechanical Engineering, Dalian University of Technology, Dalian 116023, China*

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A potassium dihydrogen phosphate (KDP) optical crystal was machined to an ultra-precision surface with water-in-oil (W/O) micro emulsion polishing fluid. The micro water dissolution principle utilized in the machining process is discussed, its planarization mechanism is illustrated, and an ultra-precision polished surface with 2.205 nm RMS roughness is obtained. However, a substantial quantity of residual contamination remained on the polished surface after machining. This can seriously impact the optical performance of the crystal, and so it must be removed. Fourier transform infrared (FTIR) spectroscopy was used to conduct an investigation into the composition of the surface residue, and the results showed that the residue was comprised of organic chemicals with hydrocarbon chains and aromatic ether, i.e., mostly the polishing fluid. The cleaning method and the principle on which the KDP ultra precision surface investigation is based are discussed in detail, and the cleaning experiments with selected KDP-compatible organic solvents were then performed. FTIR transmittance spectra measurement and microscopic observations were employed to assess the effects of the cleaning process on the surface of the KDP crystal. The results showed that toluene cleaning achieved the most desirable results. This cleaning method produced a surface roughness of 1.826 nm RMS, which allows the KDP crystal to be applied to subsequent engineering applications.

**KDP optical crystal, micro water dissolution machining, micro emulsion fluid, ultra-precision surface, FTIR spectra, surface residue, cleaning**

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### 1 Introduction

Optically fabricated glasses and substrates have been widely used in applications within the semiconductor industry and in high power laser systems due to their excellent and unique electronic or electro-optical characteristics. However, over 50% of the yield losses in integrated circuit fabrication and optical glass manufacturing are generally accepted to be attributable to micro contaminations and residue left over as a result of inadequate cleaning [1]. As the performance and

reliability requirements increase for silicon wafers and optics in the field of very large-scale silicon circuit technology and mega joule laser facilities, the importance of cleanliness and very clean substrate surfaces acquisition becomes paramount. This reality has been acknowledged since in the preliminary research stage of electronic or optical crystal parts fabrication [2].

High-power laser facilities have been home to popular areas of research for a long time and great progress has been made over the past few decades [3]. In particular, an appreciable amount of time and effort has been invested into minimizing the amount of contamination that laser optics may be susceptible to. Such contamination may degrade the

<sup>\*</sup>Corresponding author (email: hanggao4187@126.com)

optics and lower the damage thresholds of the components after frequent exposure to intense laser light [4]. The National Ignition Facility (NIF) in the US [5] and SHENGU-ANG programme of China [6] are two examples of large facilities that utilize laser optics. There are tens of thousands of ultra-precision machined optics, such as fused silica and neodymium-doped glass, employed in inertial confinement fusion (ICF) research [7,8]. And it is hard to keep all of the equipment clean, and cleanliness is the largest single problem any optics project has to contend with [9]. Due to the performance threshold and the requirements of NIF optical components, the optics, their surrounding beam path, and the supporting utility system must be fabricated, cleaned, assembled, and commissioned for precision cleanliness. NIF cleanliness requirements are designed to maintain the pristine conditions of the high-performance laser optics [10]. Since contamination-induced optical damage is a significant factor in reducing laser system reliability and lifespan, techniques that avoid contamination and generate clean optical surfaces are of critical importance in the production of high quality optical elements.

A KDP optical crystal with large aperture and high surface quality is currently the only nonlinear optical material available for mega joules-class inertial confinement fusion [11]. In addition, the ultra-precision processing of KDP optical crystals is widely acknowledged to be extremely difficult due to its moisture absorption, deliquescence property, low hardness (Moh's hardness 2.5) and thermal sensitivity. Single point diamond turning (SPDT) [12] and magnetorheological finishing (MRF) technologies [13] are considered to be state-of-the-art for the ultra-precision machining of this type of crystal. However, the SPDT process can introduce micro-waviness tooling marks on the machined surface [14], and MRF may lead to undesirable embedding of carbonyl iron particles into the polished surface [15]. Precision surface cleaning methods for the KDP crystal after SPDT were thoroughly studied. Toluene solvent was used as the cleaning agent and the corresponding cleaning procedures were created to maximize the removal of turning oils, which influence fogging, and also to remove contaminants that affect the quality of the subsequent anti-reflection coating [16]. The ion beam cleaning method was employed to remove the embedded iron powder on the polished crystal surfaces caused by MRF in the laboratory. It had proved to be an effective method for removing the embedded iron powder while improving the surface quality of the KDP optical crystal [17]. Nonetheless, this technique of ion beam cleaning seems more like a remachining process than a cleaning method. Thermal annealing at 160°C results in a clean surface, but introduces changes in the chemical structure of the KDP crystal surface, which were characterized by X-ray absorption experiments [18]. Therefore, the ultra-precision machining technology of the KDP optical crystal and its subsequent cleaning methods are still under further research.

Micro water dissolution ultra-precision polishing, based on the hygroscopic characteristic of a KDP crystal, takes a kind of W/O micro emulsion as the polishing fluid [19]. This novel machining technology has attracted much attention because it can remove the micro scale ripples left by the SPDT process, and can achieve super smooth surface almost without any subsurface damage [20,21]. However, the crystal's surface cleanliness directly impacts the optical output quality and the service life of the components [22]. Wang attempted to use alcohol to clean the well-polished KDP surface manually [23]. However, unexpected deliquescence and micro scratches can be easily introduced due to the absorption of ambient moisture and the brute force required for cleaning. Few studies have reported on the cleaning method involved with this new processing technology. Therefore, studying surface residue and cleaning technology in order to develop better methods for removing the contaminants on the machined surfaces of KDP crystals machined with micro water dissolution principle becomes an essential step for expanding its engineering application.

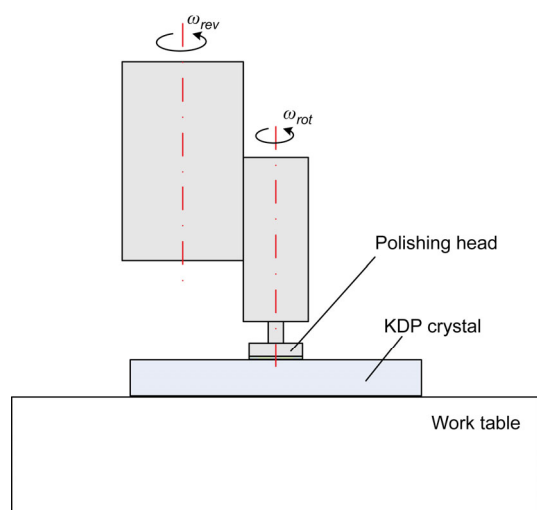
For surface cleanliness observation of optics machined by traditional methods, optical microscopic instruments such as the microscope and white light interferometer are frequently used [24]. For organic residue on machined surfaces, FTIR spectroscopy has been demonstrated as a useful and powerful tool for detecting the structural variations of IR-active groups [25]. It has been used in the analysis of surface constitution [26] and pollution detection [27]. Therefore, this study attempts to use FTIR spectroscopy to investigate the chemical composition of surface contaminants on the optics of the machined KDP crystal.

In this study, the micro water dissolution principle was used to machine KDP optical crystal to an ultra-precision surface. Polishing experiments were conducted as well. The surface residue on the polished crystal surface was studied with FTIR absorption spectroscopy in order to characterize the chemical components of organics. Cleaning experiments with ultrasonic aided vibration using different organic solvents were conducted and the cleaning effects for different cleaning solvents were also assessed. The feasibility of acquiring a clean and ultra-precision surface using a toluene solvent was verified, and its success laid a technical foundation for the industrial application of the micro water dissolution ultra-precision polishing technology for KDP optical crystals.

## 2 Experimental

### 2.1 Polishing experiment

The KDP crystal samples were cut to dimensions of 30 mm× 30 mm in preparation for the polishing process. The schematic for the experimental setup is shown in Figure 1. During the polishing process, a KDP sample was fixed by a



**Figure 1** (Color online) The schematic of the experimental setup for polishing the KDP crystal samples.

vacuum chuck on the work table. Polishing tool with a polishing head rotates along its axis at  $\omega_{rot}$  and the machine spindle rotates at  $\omega_{rev}$ . This motion allows the polishing tool to perform planetary motion and finally achieves planarization of the entire surface. In this experiment, the polishing medium was a kind of W/O micro emulsion fluid as mentioned in ref. [19], and was directed to the polishing area on the crystal surface through the center hole on the polishing tool.

## 2.2 Cleaning process

Novac<sup>TM</sup> 711PA, Novac<sup>TM</sup> 7100 (well-engineered hydrofluoroether fluids widely used in the advanced cleaning industry) and toluene solvents were selected as the cleaning detergents in this experiment. The cleaning experiments were carried out to determine the performance of these fluids. The cleaning procedures used in this experiment are described as follows. After micro water dissolution polishing, the KDP crystal samples were wiped with filter paper to blot solutions off of the surface, and then carefully cleaned with lens tissues. Afterwards, they were immediately soaked in different solvents to be ultrasonically cleaned for several minutes. After precision cleaning and thorough drying, the samples were wrapped in ultra-low outgassing plastic films. The cleaning process was conducted in an air-tight environment to prevent the crystal from deliquescing.

## 2.3 Instruments

The residual contamination left on the KDP crystal surface after ultra-precision polishing were observed with an OLYMPUS MX40 metallographic microscope and a ZYGO 5022 white light interferometer. A NICOLET 6700 FTIR spectrometer operating in reflectance mode was then used to analyze the composition of the residue and assess the effect

that the polishing process had on the surface of the KDP crystal as well as the cleaning performance of the solvents. An XE-200 (manufactured by Park Systems) was employed for atomic force microscopy (AFM).

## 3 Results and discussion

### 3.1 Ultra-precision polishing of the KDP crystal

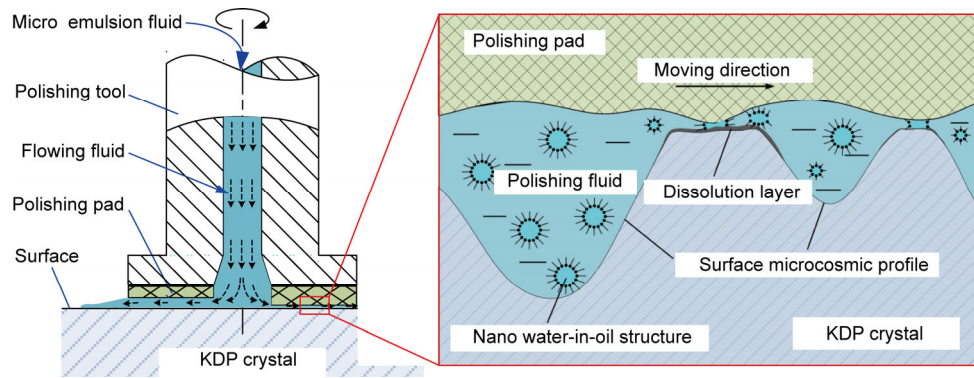
#### 3.1.1 Polishing principle and planarization mechanism

The basic principle for the KDP crystal ultra-precision polishing process shown in Figure 2 [28] is the same as what is depicted in ref. [20]. The polishing medium is a kind of W/O structured micro emulsion developed peculiar to KDP ultra precision polishing. The mechanical friction created by the relative motion of the polishing pad and the crystal helps to remove the dissolved material, and acts as the driving force in the polishing process.

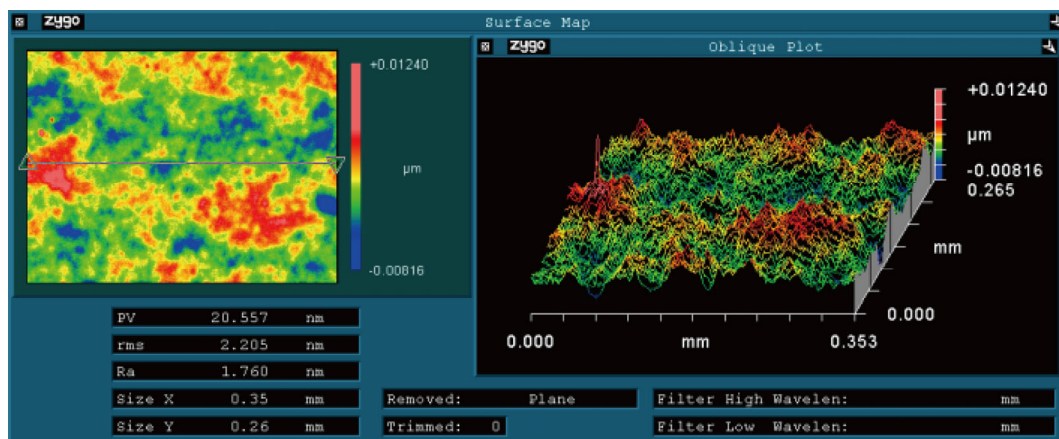
In this fluid, nano-scale water droplets are caged in non-ionic surfactant, forming water micelles that are evenly distributed in an oil-based solvent. Micro emulsion dispersion is a rather stable three-phase system that is not subject to changes to some extent. During this polishing process, micro emulsion fluid is introduced to the polishing area. The W/O structures in the valleys retain their shape in the absence of any external force. Meanwhile, the water micelles are dispersed in the organic solvent, and they neither dissolve nor react with the KDP surface. Therefore, the KDP surface is protected by the organic solvent from coming into direct contact with the water droplets, which are trapped in the micro-micelles. In the polishing area, at the contact points between the polishing pad and the KDP crystal, the water micelles are deformed by the shear force applied between the polishing pad and the KDP crystal. The water droplets are then released and are able to dissolve the asperities of the KDP crystal. The dissolution layer is then removed by the mechanical friction of the polishing pad and the flow of the polishing fluid. Meanwhile, in the valleys of the KDP surface where the polishing pad does not directly contact the KDP crystal, the water micelles are stable and no dissolution should occur. Thus, the selective removal of material is achieved by ensuring that the micro water dissolution behavior only occurs at the asperities of the crystal surface. Finally, the planarization of entire crystal surface is realized.

#### 3.1.2 Polishing result

In this polishing process, the size of the water nuclei is minimized to a nano-scale and the oil-based fluid does not react with the KDP crystal, so precise control of the material removal in polishing can be achieved, thus realizing the ultra-precision machining of KDP optical crystal. Surface cleanliness after precision polishing was treated with simply wiping, and the surface morphology is shown in Figure 3.



**Figure 2** (Color online) Micro emulsion fluid polishing of a KDP crystal surface and its planarization mechanism [28].



**Figure 3** (Color online) The morphology of a crystal surface after ultra-precision polishing.

An ultra-precision polished surface with 2.205 nm RMS and 1.760 nm Ra was obtained, which indicates a significant improvement in the surface quality of the crystal. However, a polished precision surface without adequate cleaning will inevitably stain the optics with polishing contamination. This can potentially cause optical failure of KDP crystal in the subsequent engineering application.

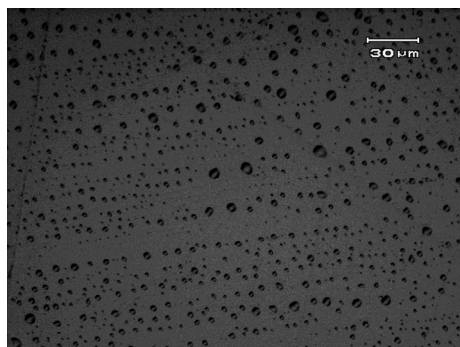
### 3.2 Cleaning investigation of the polished KDP surface

#### 3.2.1 Surface residue analysis

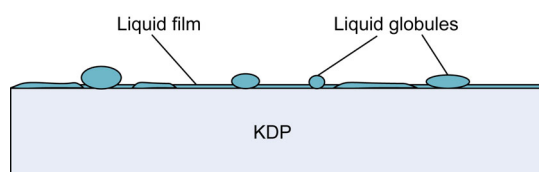
During the micro water dissolution polishing process, the sample surfaces were all soaked in the polishing fluid. Therefore, there was a substantial amount of polishing solution on the machined surface after the polishing process was completed. The machining for KDP crystal are results from micro scale water dissolution reaction which is a physical change in the polishing zone. The friction force provided by the relative motion between the pad and the crystal help to release water from the micelles in order to allow the dissolution process to occur. This process removes the dissolved crystal materials. In addition, the experiment was conducted in a clean room. Therefore, the polished surface was free of particles of all solid types, and the residue was comprised of

solutions of chemical compounds. Figure 3 is a ZYGO image tested after the polished surface was carefully wiped with lens tissue. The surface roughness value is low, while the peak to valley (PV) value stays at 20.557 nm which is higher than expected. It can be attributed to the residue attaching to the machined surface. This is concluded from the spikes and stains in a particular region of the ZYGO oblique plot. This indicates inadequate cleaning of non-volatile residue on the ultra-precision polished crystal surface. The residual pollution covers the real machined ultra-precision surface and will affect the optical performance of the KDP crystal. Hence, further investigation and rigorous cleaning of the polished surface must be conducted.

In a microscopic view, the distribution of residue on the polished KDP crystal surface is shown in Figure 4. Due to the subtle friction force applied from wiping the surface with filter paper and lens tissue, along with the strong hydrophobic property of the oil-based polishing solution, the residual polishing solution broke down into tiny liquid globules and was randomly distributed along the entire surface of the crystal. By reason of viscosity and surface tension, the residual solution will automatically aggregate into larger liquid droplets, leaving a thin fluid film across the entire surface (Figure 5). The sizes of small residue droplets



**Figure 4** Distribution of residual droplets on the polished surface.

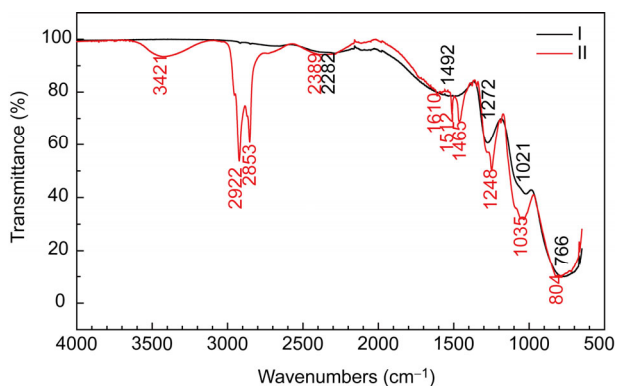


**Figure 5** (Color online) Schematic of residue on the surface of a polished KDP crystal.

can be measured in micrometers in diameter and they are difficult to completely eliminate. Inadequate cleaning and the existence of small droplets of residue on a crystal's surface can seriously impact the surface accuracy and optical performance, making it impossible in subsequent applications.

An FTIR spectrometer was used to analyze the chemical composition of the residue on the surface of the polished KDP crystal. The spectrometer was operated in reflectance mode and a spectra measurement on a clean crystal surface in the mid IR region was conducted for comparison purposes, the results are shown in Figure 6.

In the crystallographic structure of a single KDP crystal, which is comprised of both ionic bonds and covalent bonds, there exists a tetragonal system. In the case of a unit cell of KDP crystal,  $\text{H}_2\text{PO}_4^-$  ions form a three-dimensional cubic framework. In a  $\text{H}_2\text{PO}_4^-$  framework,  $\text{H}_2\text{PO}_4^-$  ions connect with adjacent ones through hydrogen bonding (P—O—H...

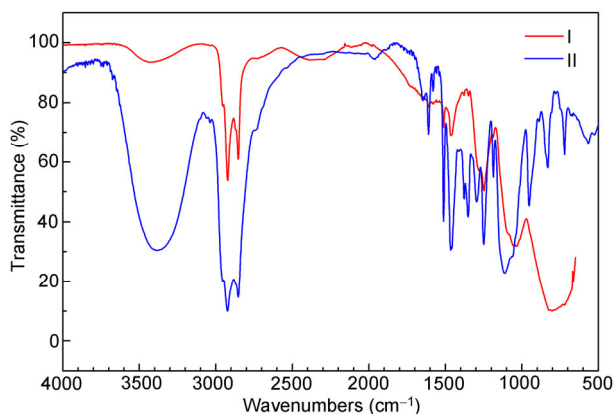


**Figure 6** (Color online) Surface FTIR spectra of KDP crystal, I-clean surface, II-polished surface.

O—P) and form a linear ionic chain along  $a$  ( $b$ ) and axis direction. Along the  $c$  axis direction,  $\text{H}_2\text{PO}_4^-$  ions likewise connect with each other by hydrogen bonding, however, forming a zigzag ionic chain. While K cations are inset into the  $\text{H}_2\text{PO}_4^-$  framework via 8 K—O bonds with O of  $\text{H}_2\text{PO}_4^-$  [29]. In the KDP crystal lattice, hydrogen bonding only forms between  $\text{H}_2\text{PO}_4^-$  groups. The chemical bonding calculation shows that a perfect KDP crystal and its family crystals appearances exhibit a tetragonal prism ended with two tetragonal pyramids. Therefore, the useful information from the IR spectroscopy is a reflection of the covalent bonds (P—O or P—O—H), while the P—O—H...O—P hydrogen bonds with the structural characteristics of the crystal are often used to characterize the crystallization behaviors of KDP.

The KDP spectrum (Figure 6) shows strong absorption bands at 1272 and 1021  $\text{cm}^{-1}$ , which could be assigned to P=O stretching and P—O stretching mode of vibration in the  $(\text{PO}_4)^{3-}$  groups. The absorption band at 2282  $\text{cm}^{-1}$  is attributed to O—H (band B) stretching vibration in the KDP lattice [30], which is an indication of hydrogen bonding between  $\text{H}_2\text{PO}_4^-$  groups. P—O—H stretching vibrations are responsible for the absorption bands appearing at 1492 and 766  $\text{cm}^{-1}$ . These absorption peaks at their characteristic positions indicated clearly the typical of KDP crystal characters. While in the FTIR spectrum of the polished crystal surface (Figure 6II), the intense broad absorption peak at between 3100 and 3700  $\text{cm}^{-1}$  corresponding to the stretching vibration mode of hydroxyl groups (O—H) is clearly observed, which has probably resulted from the ambient moisture or free water molecule enclosed in the residues since the polishing fluid is a kind of W/O micro emulsion, so as to form hydroxyl groups attaching to the surface of KDP crystal. The major absorption bands at 2922 and 2853  $\text{cm}^{-1}$  are assigned to the asymmetric and symmetric  $\text{CH}_2$  stretch, respectively. The stretching vibration of the benzenoid group can be clearly identified by the intense bands at 1610 and 1512  $\text{cm}^{-1}$ . The absorption bands at 1248 and 1110  $\text{cm}^{-1}$  are owing to the asymmetric stretching of aromatic ether. The absorption band at 2282  $\text{cm}^{-1}$  is still the O—H (band B) stretching vibration which is affected by other bonds in its molecule and shift to higher wavenumbers. Therefore, the spectra difference between the polished surface and the clean surface indicates that the polished crystal surface is contaminated with organic polishing pollution. It was also noted that organic residue is not subject to the crystal surface since the characteristic peaks of KDP crystal are almost in the right positions.

In order to further study the composition of the residue, a comparison with the spectrum of the polishing fluid was conducted. As shown in Figure 7, two spectra are identical at high wavenumbers and vary at low wavenumbers. This is because the characteristic peaks in low wavenumbers of the polished surface spectrum are mainly the reflection of KDP

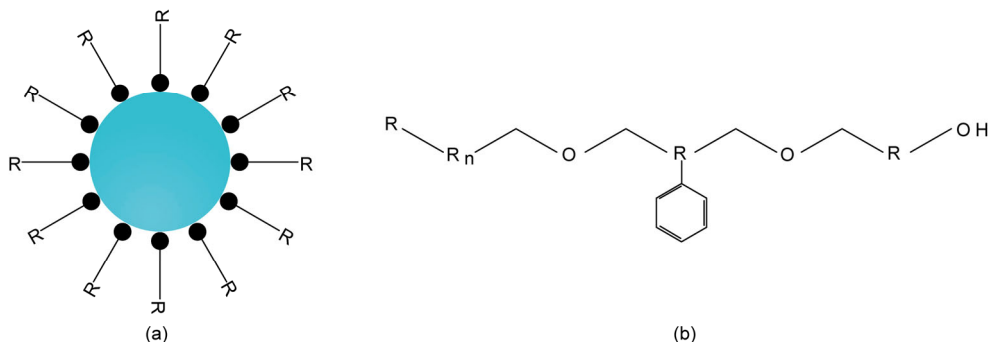


**Figure 7** (Color online) FTIR spectra of polished surface I and polishing fluid II.

composition. The amount of residue that remains on the polished surface is relatively rare in the KDP substrate. The W/O polishing fluids are rich in hydroxyl groups and methyl groups. As a result, the characteristic bands of the uncleaned surface were found to be the superposition of the clean crystal surface spectrum and the residue constituent spectrum. The results of FTIR measurements indicate that the residue attached to the polished surface are organic chemicals with hydrocarbon carbon chains and aromatic ether, i.e., mostly the polishing fluid. Thus, the surface is stained and needs to be further cleaned for the subsequent application.

### 3.2.2 Surface residue composition

The stubborn non-volatile residue on the crystal surface was qualitatively analyzed and determined to be chemical compounds of polishing fluids and organics. For the micro emulsion polishing fluid, the micro structures (Figure 8(a)) disperse in the continuous phase of the oil based carrier. The surfactant is a kind of polyfunctional compound with a hydrophilic group on one end and a hydrophobic group on the other. The non-aqueous carrier liquid is a long carbon chain alcohol species, and is compatible with KDP in a neat arrangement and takes up the most proportion. Based on the analysis, it was concluded that the composition of the organics were organic chemicals with several characteristics.



**Figure 8** (Color online) Chemical structure of micro emulsion fluid (a) and organic residues (b).

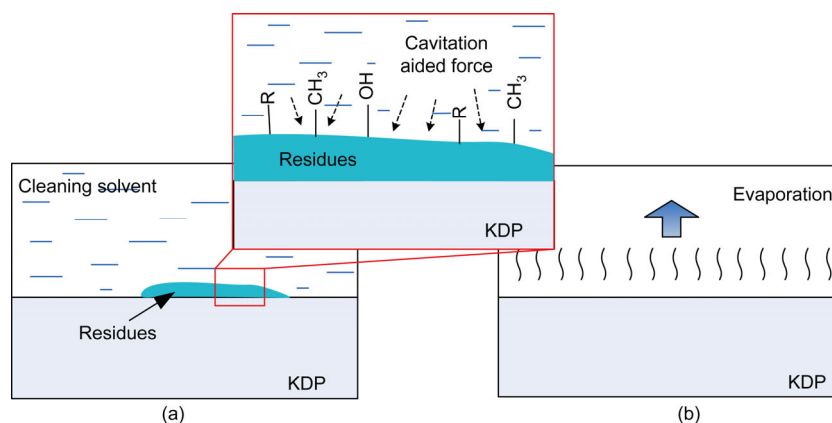
Examples of these characteristics include: long hydrocarbon chains, hydrophilic and hydrophobic groups, and aromatic ether. This is shown in Figure 8(b). Cleaning solvents could be selected and cleaning experiments can be carried out based on the results of the investigation.

### 3.2.3 Surface cleaning mechanism

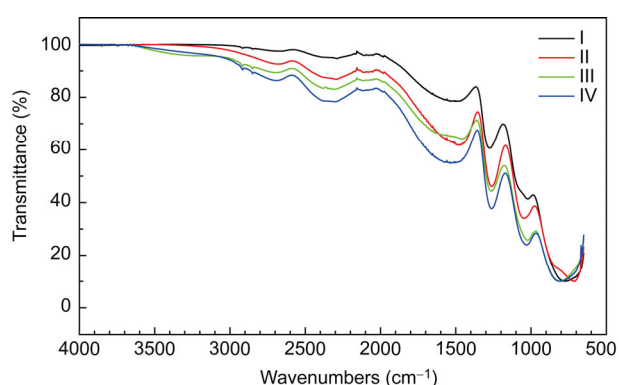
According to the principle of similitude between solutes and solvents, a kind of detergent with a chemical structure similar to that of the surface residue must be selected. Organics with low molecular mass and short hydrocarbon chains are preferable because viscosity which is detrimental to cleaning increases with chain length and molecular weight. In addition, the candidates should all be strong organic solvents with good volatility and compatibility. This means that they have the ability to dissolve organic chemicals, but do not affect or react with the optical surface of the KDP crystal. Ethanol and acetone are not suitable because they cause rapid evaporation and ambient water condensation on the crystal surface leading to staining and fogging after cleaning. However, the fluid used was micro emulsion dispersion and the whole process was conducted in the clean room, so no abrasive or dust was found adhering to the crystal surface. Solvent wiping may not be as effective here for precision cleaning because organic residue with long hydrocarbon chains are so hydrophobic and polar that they can be glued onto the polished precision surface by the Van der Waals force. Therefore, high-frequency ultrasonic-aided vibration cleaning was employed in order to overcome the intermolecular forces and hydrogen bonding present at the interface between the KDP crystal and the residual chemicals. A schematic of the cleaning process with the selected cleaning solvents is shown in Figure 9. The stubborn contamination was finally fully eliminated. The FTIR transmittance spectra observation was then conducted to assess the cleaning effect for the ultra-precision KDP surface polished with micro water dissolution principle.

### 3.2.4 Cleaning effect analysis

The FTIR spectra comparison for the polished crystal surface cleaned with different solvents is shown in Figure 10. In addition, a comparison spectrum for a clean crystal sur-



**Figure 9** (Color online) Schematic of the cleaning process with selected solvents. (a) Ultrasonic aided cleaning; (b) evaporation.



**Figure 10** (Color online) FTIR spectra comparison of the polished crystal surface cleaned with different solvents. I-clean surface, II-toluene, III-7100, IV-71IPA.

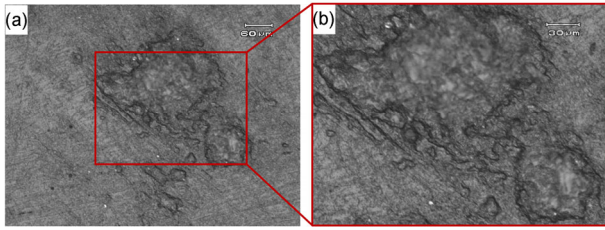
face was added to the curves group. From the spectra comparison, it can be determined that the tendencies of the four curves are all identical, and the number of corresponding characteristic peaks on the curves is the same to their generation positions. This indicates a good cleaning effect on the surface homogeneity. Furthermore, there are no evident  $\text{CH}_2$  absorption bands, and at the same time, hydroxyl ( $\text{O—H}$ ) peaks at approximately  $3500\text{ cm}^{-1}$  disappear in the spectra. Compared to the spectrum of the clean crystal, there is no newly generated peak, which indicates the surface is clean and without contaminants. In other words, there is no polishing solution or cleaning solvent residue present on the cleaned crystal surface as far as the spectra analysis can determine. The increased transmittance indicates a progressively cleaner KDP optical surface. Therefore, toluene cleaning minimized the obscuration on the optical surface and had a better cleaning effect superior to other solvents from the spectral comparison aspect.

A microscope was used to investigate the 71IPA cleaned surface, and it was determined that the surface was free of residue across the entire aperture, except for a few local areas. Some deteriorated regions were observed and are shown in Figure 11. Large areas of black and white and

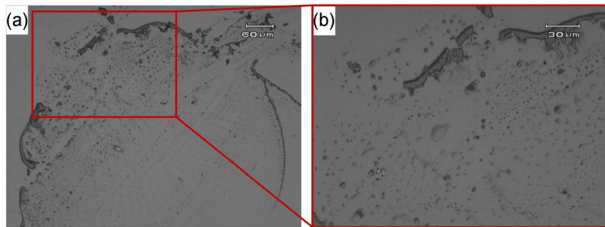
stained spots appeared on the crystal surface. The black area was a collection of dense pitting, which was the consequence of corrosion. This pitting corrosion was predominantly focused in the scratch-concentrated and interlaced areas. An equal concentration of caput mortuum was also observed, and deliquescence and corrosion also occurred in the surrounding areas. By analyzing the constituency of the solvents, these could be attributed to isopropyl alcohol ingredient in the 71IPA solution. Isopropyl alcohol is water soluble and volatile, and deliquescence on the surface was caused by the absorbed moisture from the ambient air during its evaporation process. Due to poor quality and wide contact area with the air, it is most probable to be deliquescent in those areas where scratches present and surface deteriorates. In addition, caput mortuum was the consequence of solvent evaporation caused water condensation.

The microscopic picture of the polished surface cleaned with 7100 is illustrated in Figure 12. Dust-like substances attached to the crystal surface in a large semi-closed shape. The size of the shape was measured at approximately hundreds of micrometers. The outer periphery was clean and free of pollution, while the inner area was covered in ashes. The enlarged view of the circular areas showed that these substances were hydrophobic liquid marbles (Figure 12(b)). A careful observation of the droplets showed that they were not covering on the surface but self-gathered in various sizes and. However, the crystal surface beneath these droplets was not deliquesced or corroded. The analyzed results showed that they were probably non-volatile residue from the cleaning solvent after evaporation. The hydrophobic liquid marbles could be the packed cleaning solutions that were not broken away from the surface during the ultrasonic aided cleaning process. Although this residue can be easily eliminated by wiping without damaging the polished surface accuracy, it is still an undesirable side effect of cleaning with the 7100 detergent.

The surface topography of the polished KDP crystal cleaned with toluene solvent is shown in Figure 13. No non-volatile residue or corrosion was found anywhere on



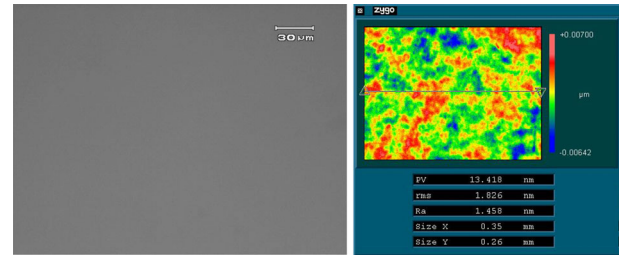
**Figure 11** (Color online) Polished surfaces cleaned with 71IPA solvent.



**Figure 12** (Color online) Polished surfaces cleaned with 7100 solvent.

the entire polished surface. The sample presented a neat and clean appearance, and meets the cleanliness requirement for laser optics. The ZYGO image showed that the surface RMS and Ra roughness were 1.826 and 1.458 nm respectively, which indicated that the polished crystal surface after cleaning was a super smooth and ultra-precision surface.

The PV value decreased remarkably from 20.557 to 13.418 nm. These improvements in the surface quality specifications revealed that the polished surface was actually covered with residue and the real ultra-precision surface

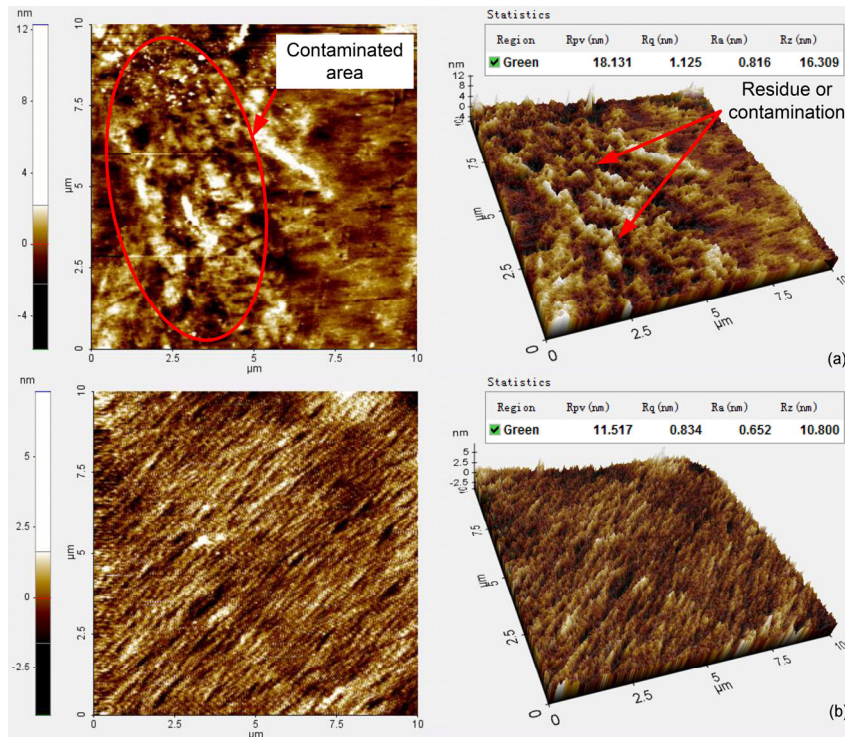


**Figure 13** (Color online) Surface topography of polished KDP crystal cleaned with toluene.

beneath came out after effective cleaning with toluene solvent. Figure 14 shows a comparison between the atomic force microscopy pictures for the surface topography of the polished KDP optical crystal before and after toluene solvent cleaning. It is apparent that there were residue and contamination on the polished surface and that they severely impacted the surface quality of the KDP optical crystal. Through proper cleaning with ultra-pure toluene solvent using the ultrasonic assisted method, the residue was driven off and the crystal surface became flat and uniform. Finally, an ultra-precision surface without residue and chemical contaminants was acquired, which can be applied directly to the coming engineering preparation process.

### 4 Conclusions

Machining of KDP optical crystal to an ultra-precision sur-



**Figure 14** (Color online) AFM topography of KDP crystal surface before (a) and after cleaning (b).



face for engineering application is tough work due to its unique physical and chemical properties (softness, brittleness, and water solubility). In this study, a KDP optical crystal was machined to an ultra-precision surface, and an investigation into the effects of the subsequent cleaning process was conducted. The following conclusions can be drawn from the study.

The micro water dissolution based ultra-precision machining principle was discussed and its planarization mechanism was illustrated. The polishing medium used was a kind of W/O micro emulsion and an ultra-precision polished surface with 2.205 nm rms roughness was achieved.

The investigation of the polished KDP optical surface showed that without adequate cleaning, the optics were inevitably stained with residue.

FTIR spectra indicated that the residue on the polished crystal surface was comprised of hydrocarbon compounds containing benzenoid groups, ether, and other functional groups, i.e., mostly the polishing fluid.

Cleaning experiments were performed in order to remove the residue from the polished ultra-precision surface. These experiments were based on the mutual adsorption mechanism of similar organics. Ultrasonic aided vibration was also used to enhance the cleaning effect. The results showed that toluene solvent cleaning can completely eliminate the residue on the polished surface with favorable cleaning effects. A clean and real polished surface with 1.826 nm RMS roughness was achieved, which laid a technical foundation for industrial application of the micro water dissolution ultra-precision polishing technology for KDP optical crystals.

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