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REVIEW ARTICLE

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Research progress on ultra-precision machining technologies for soft-brittle crystal materials

Abstract Soft-brittle crystal materials are widely used in many fields, especially optics and microelectronics. However, these materials are difficult to machine through traditional machining methods because of their brittle, soft, and anisotropic nature. In this article, the characteristics and machining difficulties of soft-brittle and crystals are presented. Moreover, the latest research progress of novel machining technologies and their applications for soft-brittle crystals are introduced by using some representative materials (e.g., potassium dihydrogen phosphate (KDP), cadmium zinc telluride (CZT)) as examples. This article reviews the research progress of soft-brittle crystals processing.

Keywords brittle, soft, functional crystal, ultra-precision machining

1 Introduction

Functional crystal materials can achieve energy conversion because of their unique physical characteristics. With developments in science and technology, functional crystal materials have become important in many fields. For instance, potassium dihydrogen phosphate (KH_2PO_4, KDP) single crystal is a multifunctional material that possesses excellent nonlinear optical characteristics, effective photoelastic coefficients, and acousto-optic figures. KDP crystal is the first choice for multi-dimensional acousto-optical device [1,2] and currently the only material suitable for laser frequency conversion and electro-optic switch applications in high-power laser systems [3]. Mercury cadmium telluride (HgCdTe, MCT) single crystal is the most significant material for infrared optoelectronic devices and MCT-based high performance infrared devices, and is widely used in the fields of aeronautics and astronautics [4]. Cadmium zinc telluride (CdZnTe, CZT) single crystal is the most promising material for the fabrication of room temperature radiation detectors [5,6] and is also widely used as the perfect substrates for growing epitaxial layers of MCT crystal [7]. CaF_2 single crystal is extensively applied in deep ultraviolet photolithography, solid-state lasers, and high-energy radiation detection [8]. BaF_2 single crystal with high transparency for infrared and visible light is often used in CO_2 laser hatch and infrared optical systems [9]. Ammonium dihydrogen phosphate ($NH_4H_2PO_4$, ADP) single crystals whose crystal structure is similar to KDP also possess excellent nonlinear optical characteristics [10].

The demand for high-quality functional crystal elements increases with the progression of optical technology. For instance, the high-power laser system for the American National Ignition Facility has 192 identical beam lines that use more than 560 large KDP elements (above 300 mm$\times$ 300 mm$\times$ 10 mm) with low surface roughness ($\leqslant$5 nm), high surface figure accuracy (transmission wave-front distortion$\leqslant\lambda/6$ Peak-Valley), and high laser-induced damage threshold ($\geqslant$15 J/cm^2) [11]. The extreme quality requirements pose a severe challenge to ultra-precision machining. The hardness of some functional crystal materials is significantly lower than that of conventional hard and brittle materials, e.g., single crystal silicon (Mohs hardness 7), sapphire crystal (Mohs hardness 9), and silicon carbide ceramics (Mohs hardness 9.5). Although the ultra-precision machining methods for hard and brittle materials are relatively mature, these methods may not be suitable for soft materials. In this article, the material characteristics and machining difficulties of soft-brittle crystals are reviewed. Moreover, this article also introduces the research progress of machining technologies for soft-brittle crystal materials using some representative materials (e.g., KDP, CZT) as examples.

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2 Material characteristics and machining difficulties of soft-brittle crystals

Functional crystals with a Mohs hardness below 5 are defined as soft-brittle crystals to distinguish them from hard-brittle materials in this article. These materials have low hardness (the Mohs hardness of the common soft-brittle crystals is shown in Table 1), high brittleness, low fracture toughness (cracks and pits can be induced when loaded beyond the elastic limit), strong anisotropy, and low resistance to thermal shock.

Table 1 Mohs hardness of several kinds of soft-brittle crystals

Crystal name	Mohs hardness
KDP crystal	2.5
CZT crystal	2.3
MCT crystal	3.0
BaF_2 crystal	3.5
ADP crystal	2.0
CaF_2 crystal	4.0
NaCl crystal	2.5
LiF crystal	3.0
BBO crystal	4.0
ZnS crystal	3.5 – 4.0

Moreover, water can dissolve some soft-brittle crystal materials, such as KDP, ADP, and LiF [12,13]. A machined surface can be destroyed by water absorbed from air if exposed to the atmosphere. Additional challenges are encountered because of water solubility during the ultra-precision machining process for such soft-brittle crystals.

Given the special characteristics of soft-brittle crystals, conventional ultra-precision methods (e.g., grinding, lapping, and traditional polishing) may not be suitable for processing these crystals. Abrasives and chips are easily embedded into the surface as a result of low hardness and will thus affect surface quality. Meanwhile, most soft-brittle crystal materials have a high thermal expansion coefficient and low thermal conductivity; therefore, the heat generated during machining can easily fracture these crystals. Many ultra-precision machining technologies are developed to process soft-brittle crystal materials because obtaining a damage-free surface by traditional methods is difficult.

3 Ultra-precision machining technologies for soft-brittle crystals

3.1 Precision cutting technology

The first step in manufacturing soft-brittle crystal elements is to cut a bulk crystal into slices along a certain angle and orientation. A band saw is the most common tool for slicing a large size crystal that exceeds 1 m in size, such as KDP. However, a wide kerf produces more material wastes, and fractures are easily generated during the cutting. Moreover, surface and subsurface cracks may be developed during the cutting, leaving chipping on the machined surface.

Gao et al. [14] and Teng et al. [15] proposed a diamond wire sawing technology that uses water dissolution from the emulsified cutting fluid. The mechanism of water dissolution in diamond wire sawing is shown in Fig. 1 [15]. As a result of the shear force at the contact point between diamond cutter and KDP optical crystal, micro water droplets are released from the water nuclei in fluid to dissolve the KDP crystal. The cutting force is reduced with the assistance of water dissolution, which helps suppress

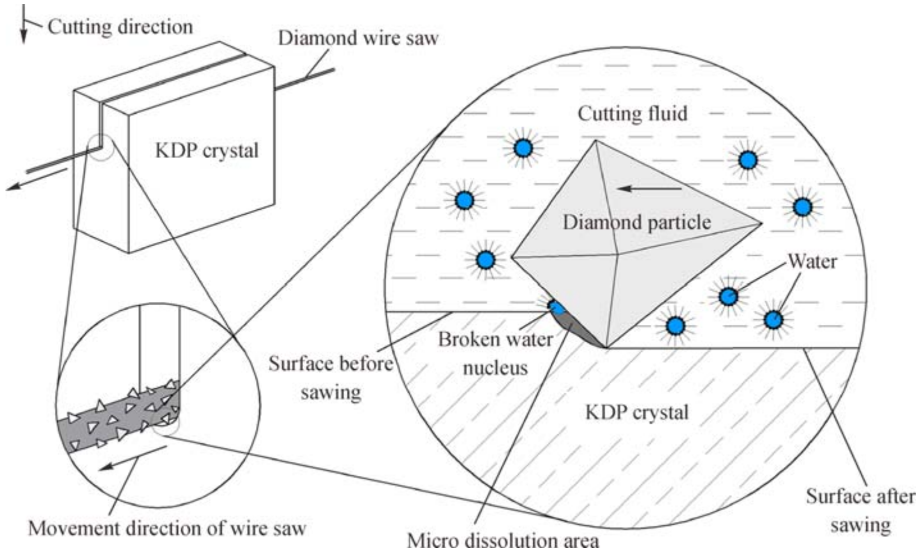


Fig. 1 Mechanism of diamond wire sawing assisted with water dissolution [15]

crack and damage generation. The new method using water dissolution obtains a surface (300 mm × 300 mm) with higher quality and improves cutting efficiency by 15%–20% compared with sawing the diamond wire using conventional cutting oil.

Deng et al. [16,17] developed a laser beam separation technology for KDP crystals. The mechanism of the technology is to pretreat a KDP crystal by using an ultrafast laser to achieve artificial adjustment of optical absorptivity and binding capacity inside the crystal. Tensile stress or microscopic tensile stress inside the crystal is then induced by using continuous laser to achieve crystal separation. The experimental setup for laser beam separation of KDP crystals is shown in Fig. 2 [17]. A damage-free mirror-separated sidewall could be achieved on KDP crystals with a thickness of 12 mm by using this method; the separating speed is higher than that of traditional mechanical cutting.

3.2 Single-point diamond turning (SPDT)

SPDT is currently the most important ultra-precision machining for large optical elements, especially KDP crystal lenses. As shown in Fig. 3, a diamond tool is installed on a flying cutter head. A workpiece is fed along the radial direction of the rotational motion of the diamond tool. The cutting depth is small, and the cutting velocity is high. A super smooth surface can be obtained by the SPDT method without grinding and polishing.

SPDT was developed in the 1960s. Fuchs et al. [18] at the Lawrence Livermore National Laboratory (LLNL) first introduced SPDT into the manufacturing of KDP optical lenses. A super smooth surface with a surface root-mean-square (rms) roughness of 0.8 nm was obtained by adopting a diamond tool with negative rake (−45°). Small cutting depth, large edge radius, and low feed rate were beneficial to the surface quality. However, Kozlowski et al. [19] found that KDP crystal surface fogging was associated with several parameters of diamond turning and subsequent cleaning processes. The use of a new oil noticeably reduced the occurrence of fogging on freshly turned crystals. The influence of humidity on crystals was minimized in a laser system by applying a protective silicone coating.

In China, An et al. [20], Liang et al. [21,22], and Chen et al. [23] at Harbin Institute of Technology developed an SPDT equipment (as shown in Fig. 4 [23]) and presented the design philosophy of an ultra-precision fly cutting machine tool based on machining material properties and processing requirements. The gantry-type structure has excellent dynamic performance and is suitable for producing flat half-meter scale optics. The influence of the main components on tool-workpiece structural loop was analyzed, and the weak structural component was optimized to further improve the dynamic performances of the machine tool. The complete angular displacement formula of aerostatic bearing spindles and a 3D surface profile simulation program were constructed and verified to

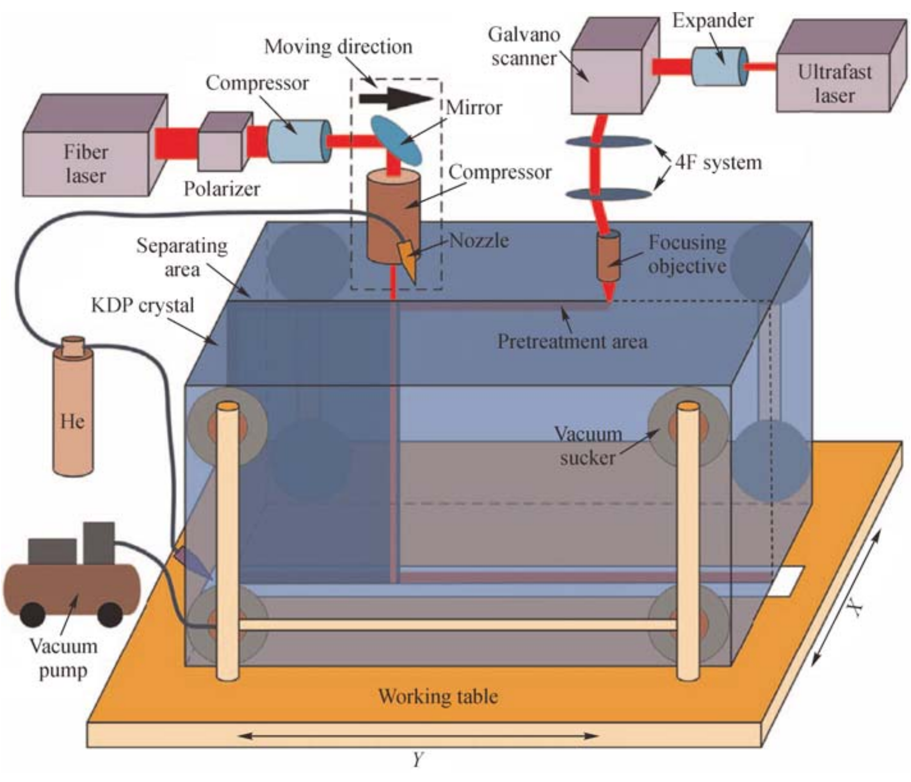


Fig. 2 Schematic diagram of the experimental setup for laser separation of KDP crystals [17]

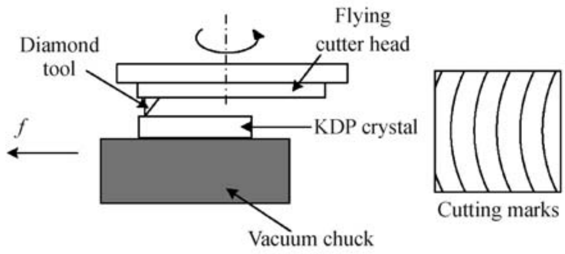


Fig. 3 Schematic diagram of the SPDT setup

study the tilting motions of spindles. The inertia tensor criterion that decreases the waviness errors of machining surface was represented. Machining results further proved and validated that the design philosophy proposed by Liang et al. can ensure high performance of the developed SPDT machine tool. In addition, Wang et al. [24] studied the degenerative layer thickness in an SPDT process through ANSYS analysis and nano-indentation. They found that cutting speed has the greatest effect on the thickness of the degenerative layer, followed by depth of cut and feed rate. Moreover, Wang et al. [25] optimized the distribution of suction holes to improve surface accuracy; the optimized structure is suitable for fixing soft and fragile parts. Their study showed that the vacuum chuck with small suction holes has minimal adsorption deformation, and the increase in vacuum ratios of vacuum chuck reduces the vacuum degree of adsorption.

Many studies were carried out to investigate the influence of material anisotropy on ductile cutting of soft-brittle crystals. Chen et al. [26] proved that cutting forces and surface roughness vary significantly with different crystallographic orientations on the (001) plane of a KDP crystal; and the amplitude variation of cutting forces and surface finish is closely related to the cutting parameter of maximum undeformed chip thickness. With the maximum undeformed chip thickness below 30 nm, the amplitude variation of cutting force is minimized, achieving a super-smooth surface with a consistent surface finish in all crystallographic orientations. In a different study, Tie et al. [27] used the spiral scratch method and obtained the critical brittle-ductile transition depth on the tripler plane of a KDP crystal, as well as the ductile turning parameters. With the use of these parameters, a super smooth surface (surface roughness 1.3–1.7 nm) with no distinct anisotropic distribution of surface morphology was manufactured. Wang et al. [28] carried out cutting experiments on the (001) doubler and tripler planes of a KDP crystal. They analyzed the change law of cutting force and the brittle ductile-transition depth related to cutting direction, as well as established accurate theoretical models for calculating the cutting force and conditions to achieve a crack-free surface by using a circular edge cutter. Their study served as a guideline for studying the fly cutting mechanism of other soft-brittle crystals.

Chen et al. [29] and Zong et al. [30] manufactured CaF_2 and ZnS crystal samples with high quality surface, respectively. SPDT is suitable for processing large size soft-brittle crystal elements; SPDT-machined elements have low roughness and high surface accuracy. However, SPDT causes the formation of cutting marks (microwaviness, as shown in Fig. 5 [31]). Cutting marks adversely affect the optical performance of a KDP element, especially the laser-induced damage threshold. Subsurface layer damage, which can adversely affect the optical

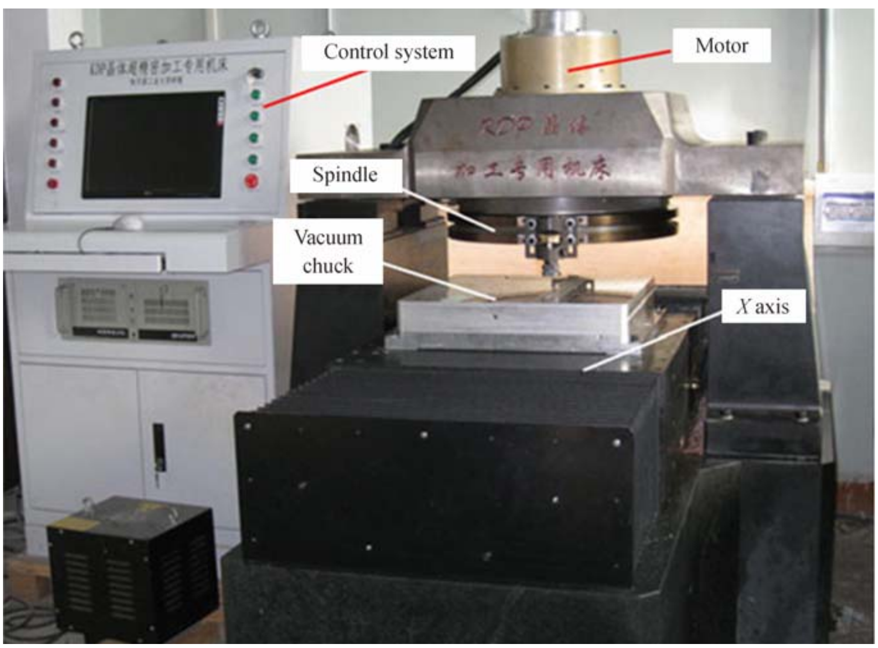


Fig. 4 Ultra-precision SPDT equipment [23]

properties of a KDP lens, is inevitable when conducting a process that mechanically removes material from a workpiece [32]. The results of Chen et al. [33,34] showed that the modulation effect of surface microwaviness and subsurface cracks are the two important mechanisms that damage a KDP crystal. The modulation degrees increase linearly when waviness amplitude and subsurface crack depth increase; the laser-induced damage threshold decreases with increasing amplitude and subsurface crack depth.

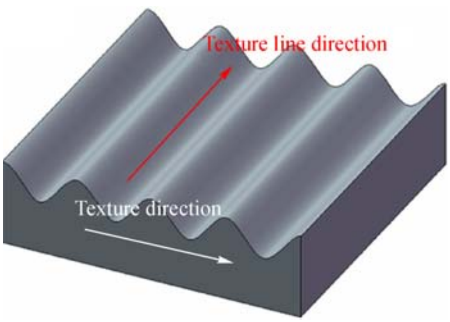


Fig. 5 Schematic diagram of the micro waviness on the surface after SPDT process [31]

3.3 Ultra-precision grinding

Ultra-precision grinding is a popular method to obtain high quality surface and is widely used in the fields of large size wafer thinning and optical element manufacturing.

Namba et al. [35] demonstrated the possibility of obtaining an optical surface on a KDP crystal by using the ultra-precision grinding method. They optimized the grinding parameters and obtained a high-quality KDP surface with rms roughness of 0.553 nm by using a mixture of 10 Pa·s silicon oil and 5 wt% heptanol as grinding fluid. They also found that laser-induced damage threshold increases with decreasing surface roughness.

To investigate the deformation of CZT single crystals under nanogrinding, Zhang et al. [36] used cross-sectional transmission electron microscope (TEM). Their results revealed that only dislocations and nanocrystals with sizes of 5–20 nm exist on the subsurface of CZT crystals generated by nanogrinding with wheels of different grit sizes. The deformation mechanism of CZT is different from the formation of damage layers in hard-brittle materials like silicon. Wu et al. [37] investigated the deformation nature of CZT crystals induced by nanoscratching using cross-sectional TEM; the TEM characterization provided insights into the formation of crystallite defects induced by high-pressure mechanical loading. The formations of twins, stacking faults, and nanocrystals were the dominant deformations induced by nanoscratching on CZT single crystals. Significant pile-up was observed, but no amorphization phases existed.

Zhang et al. [38] adopted cup wheels to machine CZT wafers, as shown in Fig. 6. A comparative research on a surface processed with different diamond grinding wheels (experimental results are shown in Fig. 7 [38]) found that plastic deformation was small and the scratches were subtle on the wafer surface ground with a #5000 grinding wheel. Li et al. [39] investigated the effect of mechanical anisotropy on grinding of CZT (110) and (111) planes. They found that the optimum grinding directions are $\langle\bar{1}10\rangle$ directions on the (110) plane and $\langle 11\bar{2}\rangle$ directions on the (111) plane.

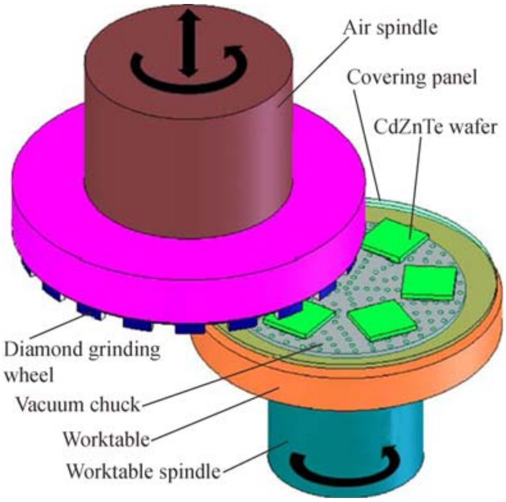


Fig. 6 Schematic diagram of precision grinding of CZT wafers [38]

3.4 Magnetorheological finishing

Magnetorheological finishing (MRF) is a newly emerging technology with good prospects for development and application value. The magnetic fluid disperses the micro-nanoscale magnetic particles (commonly carbonyl iron particles) into the insulating carrier liquid. The rheological properties of magnetic particles can significantly change into a high-intensity gradient magnetic field. In normal state, the rheological property of a magnetic fluid is similar to that of Newtonian fluid. However, the magnetic fluid loses its mobility and transforms into a solid state within a millisecond when subjected to a high-intensity magnetic field. During MRF, the magnetic fluid is sprayed onto a buffing wheel and then changed to viscoplastic Bingham media as a flexible polisher with increased viscosity and hardness under strong magnetic field. The polisher is pressurized into the workpiece at a certain depth, and the shear force formed between the tool and workpiece removes the material. MRF technology can obtain good surface quality and can also obtain quantitative material removal of a certain area through computer-controlled path planning. Therefore, MRF is suitable for manufacturing ultraprecision optics with a large aperture.

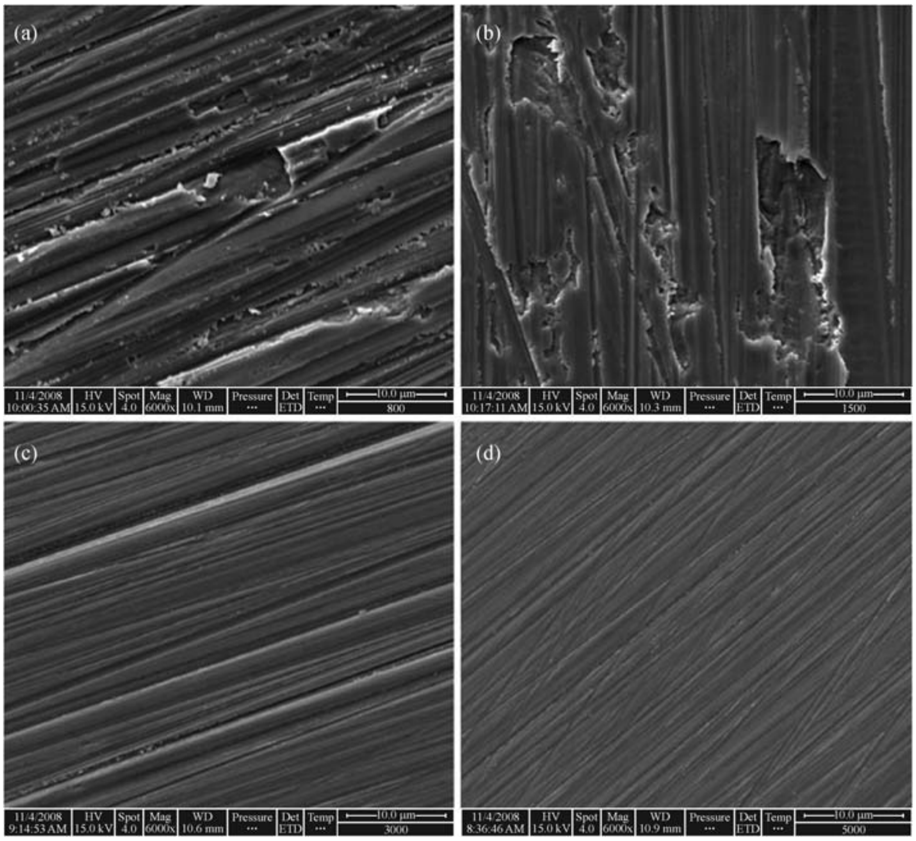


Fig. 7 SEM surface topography of CZT wafers after precision grinding by diamond grinding wheels with different diameter abrasives: (a) #800, (b) #1500, (c) #3000, and (d) #5000 [38]

Kordonski and Jacobs applied MRF technology to aspherical optics manufacturing [40,41]. In polishing water soluble materials, such as KDP crystal, the conventional water-based magnetorheological fluid is no longer applicable. Arrasmish et al. [42] developed a new MRF polishing liquid using carbonyl iron magnetic particles, glyoxylic acid ester, and a small amount of nano-diamond abrasive. This new formula solved the fogging problem on the KDP crystal surface in the polishing process. Later on, Jacobs et al. [43] improved the MRF fluid for KDP crystal polishing and selected chemicals with less environmental pollution. The method successfully removed the surface microwaviness of KDP crystal (50 mm$\times$50 mm) on a Q22 MRF machine using SPDT (as shown in Fig. 8). The surface rms roughness was reduced to 2 nm or less with a slight improvement on laser damage threshold.

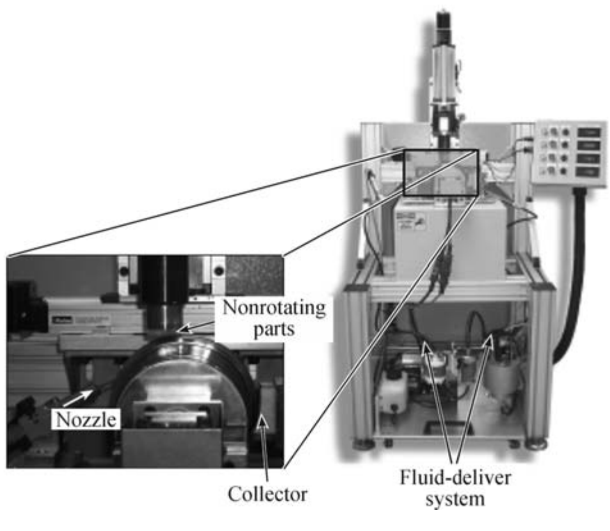


Fig. 8 MRF machine (Q22, QED Company)

In China, the National University of Defense Technology developed MRF equipment independently and conducted substantial research work on MRF polishing for ultra-precision optics. Ma et al. [44] used silicone oil, carbonyl iron, surface active agents, and aluminum oxide abrasive to develop a nonaqueous-based MRF fluid for machining KDP crystal, which has a good effect. However, some residual pollutants, such as silicone oil and carbonyl iron, remain after polishing. Peng et al. [45] continued to improve the MRF fluid and the polishing process, and tried to replace the abrasive in the MRF fluid with deionized water. Finally, a precision KDP surface with 0.809 nm rms roughness was achieved.

However, in the MRF process of soft and brittle crystal, carbonyl iron powder may be embedded into or scratch the machined surface, thereby affecting the optical performance of the crystal elements. To solve this problem, Ji et al. [46] coated iron particles (CIPs) with methoxyl poly (ethylene glycol) (MPEG), and the elastic modulus of CIP was reduced from 90 to 75 GPa after coating with less than 1.1 times the value of KDP. Polishing experiments on KDP samples were carried out with the coated CIPs in MRF fluid. The roughness was lowered from 4.521 to 2.748 nm with complete removal of turning grooves, as well as reduced processing stress (as shown in Fig. 9 [44]).

3.5 Ion beam figuring

Ion beam figuring (IBF) is an advanced deterministic manufacturing technology for optics. IBF utilizes an ion beam to bombard the workpiece surface and then transfers energy to the surface atoms through massive collisions. When enough energy is gained to break away from the binding energy, the atoms will escape from the surface, thereby obtaining atomic removal of material on the workpiece surface by physical sputtering. When integrated with computer numerical control technology, this method can achieve quantitative material removal of a local area. Figure 10 [47] shows the ion beam figuring process, which is a non-contact manufacturing technology. In the polishing process, a workpiece is not subjected to pressure, and no subsurface damage that has usually been seen in conventional machining will be generated. Theoretically, ion beam figuring can achieve a damage-free ultra-precision surface.

Wilson and Mcneil [48] first introduced IBF technology into the processing of optical lenses in the 1980s; they improved the surface figure of a fused silica optic (300 mm× 300 mm). Meanwhile, Allen and Keim [49] built an IBF system for large optics up to 2.5 m in diameter. Research on IBF technology in China started late compared with that in Western countries; the researchers at the National University of Defense Technology have been studying IBF technology since 2005 and independently developed an IBF equipment KDIFS-500. To avoid the cracks caused by high thermal stress in IBF, Li et al. [47] established the temperature field model of KDP by IBF technology and studied the change law of surface roughness related to IBF processing parameters. Chen et al. [50] introduced IBF technology into the manufacturing of KDP crystals to remove the iron powders left from the MRF process; they proved that no iron contamination was found using ion mass spectrometry. Yin et al. [51] presented a compound machining technology, and they reported that using chemical mechanical polishing improves surface roughness and using IBF technology improves surface figure. A high-quality CaF_2 crystal surface with a surface roughness of Rq 0.207 nm and a surface-figure peak-to-valley of 13.1 nm was obtained by this compound machining method.

However, the IBF method has limitations. First, the vacuum condition is necessary for IBF, the structure of IBF equipment is complex, and the cost of IBF machining is high. Second, the material removal rate of IBF technology is relatively low (generally below hundreds of nanometers per minute). Therefore, IBF technology is more suitable for improving the surface figure of workpieces after precision machining.

3.6 Micro water dissolution machining

Some soft-brittle crystal materials can be dissolved in water. These materials are easily damaged by water in the environment during manufacturing and storage because of their water solubility. In general, such soft-brittle crystals

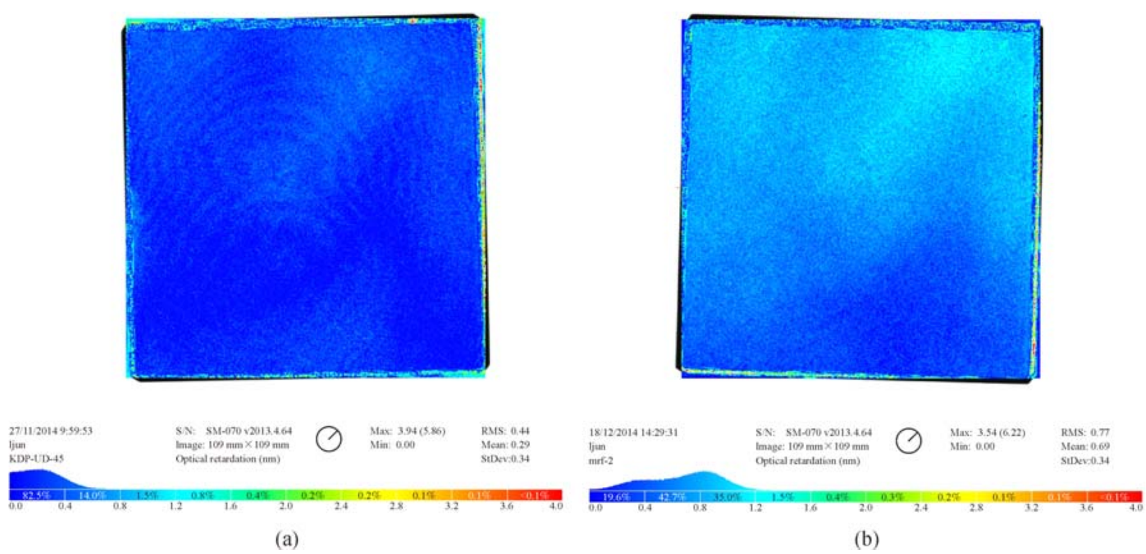


Fig. 9 Stress in the KDPs after (a) SPDT and (b) MRF [44]

Fig. 10 Experimental process of KDP 100 mm×100 mm polished using IBF method [47]

should be strictly protected from being in contact with water during ultra-precision machining. Nevertheless, no mechanical stress is observed during the dissolution of a water-soluble crystal in water. The subsurface damage often caused in mechanical machining does not exist. Therefore, researchers have attempted to incorporate water into the machining of water-soluble crystals.

A KDP crystal is a typical water-soluble, soft-brittle material with a water solubility of 33 g per 100 g of water at 25 °C. Teng [52] used a Raman spectrometer to analyze the composition of the deliquesced surface (results are shown in Fig. 11 [52]); the absence of any new characteristic peaks revealed no chemical reaction between KDP crystal and water. Teng proved the feasibility of machining KDP crystal with water. Menapace et al. [53] of LLNL added water to the MR fluid to eliminate KDP removed from the sample through precipitation and recrystallization, and successfully improved the laser-induced damage threshold to 98.5 J/cm^2. Zhang et al. [54,55] polished KDP samples by using a mixed solution of water, alcohol, and surfactant, and obtained a super smooth surface. However, this treatment introduced a new problem; alcohol absorbs water from the atmosphere, but the volatile nature of alcohol was not mentioned.

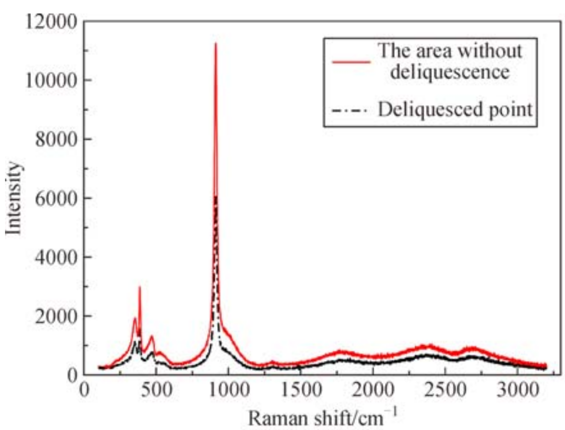


Fig. 11 Raman spectra of KDP crystal surfaces with and without deliquescence [52]

Recently, the authors' group conducted numerous studies on developing a KDP machining technology based on water-soluble characteristics. Gao et al. [56] and Wang et al. [57] invented a type of water-in-oil (W/O) micro-emulsion fluid that can be used as an abrasive-free polishing fluid. As shown in Fig. 12 [56], micro water droplets are wrapped in a non-ionic surfactant, where one end is a hydrophilic group and the other end is a hydrophobic group; these droplets form water micelles in an oil-based solvent. During polishing and at the points of contact between the polishing pad and a KDP crystal, the water micelles are deformed by shear force between the polishing pad and KDP crystal. The water droplets are released and dissolve the asperities of the KDP crystal. The dissolution layer is removed by the mechanical friction of polishing pad and the flow of polishing fluid. By contrast, no direct contact between the polishing pad and the crystal was observed in the valleys of the crystal surface, and the structure of water micelles remains intact. They obtained a scratch-free polished KDP surface (18 mm×18 mm) with rms 1.69 nm using this W/O micro-emulsion fluid as polishing fluid. Wang et al. [58] proposed a new machining method that combines the micro water dissolution polishing with the computer-controlled optical surfacing technique to improve the surface quality of the SPDT-machined KDP crystals. A small tool was used to polish the large KDP samples after the initial SPDT. The surface microwaviness and surface roughness were significantly reduced (as shown in Fig. 13 [58]), and the laser-induced damage threshold of the sample was also improved.

Although micro water dissolution machining is not established as a novel technology, this method is increasingly being recognized by researchers because of its advantages, i.e., it imposes no mechanical stress during polishing and does not damage a polished surface. The micro water dissolution machining method has great potential for finishing water-soluble materials.

4 Conclusions

Soft-brittle functional crystals are widely used because of their excellent physical properties. However, the unique characteristics of these crystals make them extremely difficult to machine and pose challenges in producing quality surfaces through conventional machining methods. This review introduces several new technologies and their applications in the manufacturing of soft-brittle crystals, and draws the following conclusions:

1) SPDT is currently the most important and widely used

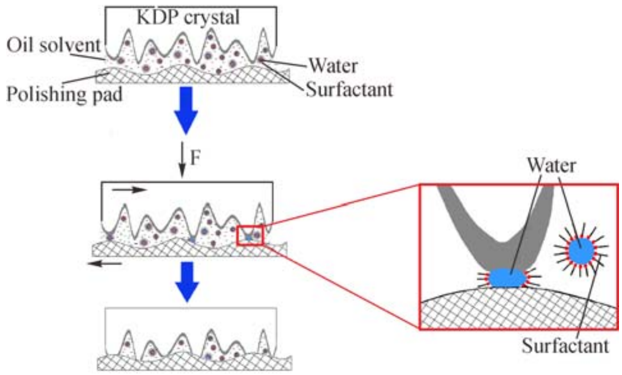
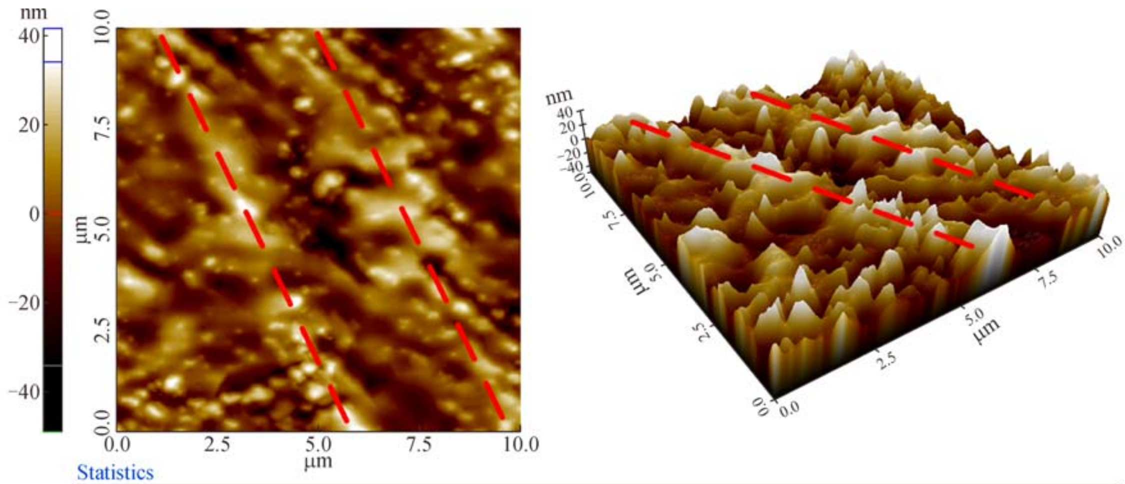
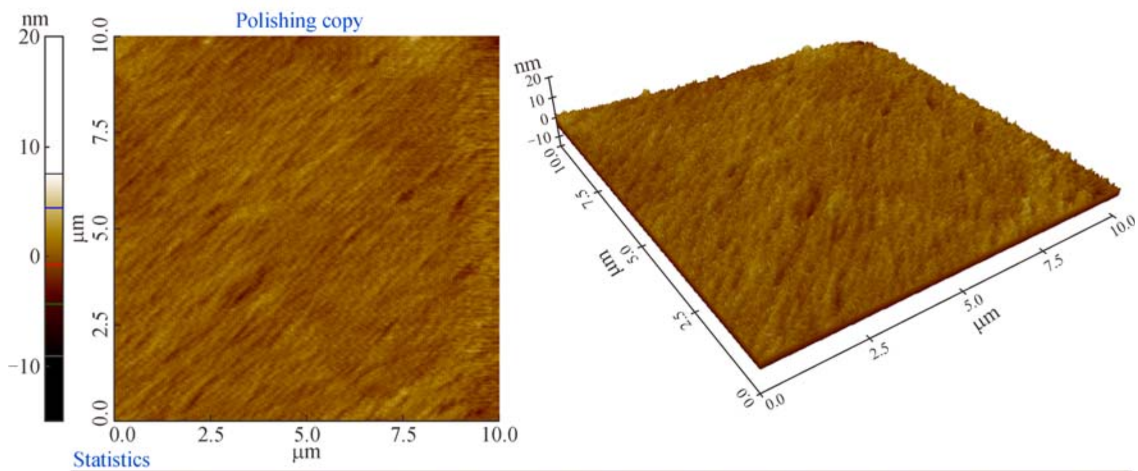


Fig. 12 Schematic diagram of material removal of KDP crystal during water dissolution polishing [56]



Region	Min/nm	Max/nm	Mid/nm	Mean/nm	Rpv/nm	Rq/nm	Ra/nm	Rz/nm	Rsk	Rku
Red	−49.018	41.610	−3.704	0.000	90.628	12.160	9.747	84.957	−0.049	2.887

(a)



Region	Min/nm	Max/nm	Mid/nm	Mean/nm	Rpv/nm	Rq/nm	Ra/nm	Rz/nm	Rsk	Rku
Red	−4.357	4.415	0.029	−0.003	8.773	0.842	0.664	8.283	−0.057	3.372

(b)

Fig. 13 Atomic force microscopy measurements of the KDP surface (a) before and (b) after polishing. The dashed lines in (a) indicate the locations of the maximum microwaviness [58] technology for processing soft-brittle optics, especially for precision manufacturing of a KDP element with a large aperture. However, the turning grooves and subsurface damage left in the KDP element after SPDT may compromise the optical performance of a machined KDP element and should thus be eliminated in a subsequent process.

2) Ultra-precision grinding may also leave grinding marks and subsurface damage on the machined surface; these marks and damage are detrimental to the optical performance of a KDP element.

3) The MRF technology shows evident advantages in removing turning grooves and improving surface accuracy. However, the problems of iron powder embedment, surface scratch, and iron contamination should be solved.

4) The ion beam figuring technology can achieve a damage-free ultra-precision surface. This method can be widely adapted if the cost is reduced and the material removal rate is increased.

5) The water dissolution machining technology based on the water solubility of a crystal removes micro ripples left by SPDT and produces a damage-free surface with no mechanical stress. This method has a broad prospect for further development.

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