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Understanding the friction and wear of KDP crystals by nanoscratching

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ABSTRACT

Potassium dihydrogen phosphate (KDP) is an important nonlinear optical crystal material, and has been widely used in large-aperture high-power laser systems. This paper aims to explore the friction and wear mechanisms of KDP by nanoscratching using Berkovich and conical indentation tips. Two scratching loading modes, the 'Constant' and the 'Continuous', were applied. During the experiment, the moisture and temperature in the testing chamber were controlled. The study found that the adhesive force plays an important role when the scratching load is low. The lateral force or the friction coefficient fluctuates due to the non-uniform deformation of the KDP in front of the indentation tip. During a constant loading scratching, the frequency and amplitude of the fluctuation vary with the level of the scratching load and the type of the indenter, while the velocity effect seems to be trivial. The slip in the KDP crystal, which is very much affected by contact stress, could be a main mechanism of plastic deformation. The Berkovich tip produces more obvious wavy groove walls, while the conical tip brings about more ploughing deformation along the wear track. The strain rate in the range investigated in this study seems to have a little effect on the micro-plastic deformation.

1. Introduction

Potassium dihydrogen phosphate (KDP) is an attractive crystal material for its superior photoelectric and piezoelectric properties. In the past decades, KDP has been widely used as frequency converters and Pockels photoelectric switches in high-density laser systems, as well as electro-optic modulation and piezo-electric transducers in sophisticated applications [1–4]. However, KDP is well known as a difficult-to-handle material for its high sensitivity to external stresses and environmental conditions, such as contact stresses, temperature variation and moisture invasion. It is also a soft material yet easy to fracture. Because of these problems, traditional methods for obtaining optical surface finish through abrasion wear, such as polishing and lapping, are not suitable for making KDP components with satisfactory surface integrity. This is because an abrasive particle can be easily embedded into a machined surface and an abrasion-induced plastic deformation in KDP could seriously downgrade the optical performance of the material (e.g., the remarkable reduction of the laser-induced damage threshold [5]). To overcome these difficulties, single point diamond fly-cutting processes have been developed to generate the optical surfaces of KDP [6]. However, it has been found that the abrasion wear associated with the fly-cutting still downgrades significantly the optical performance of the material [7,8]. For example, Chen et al. [9] reported that the cutting thickness significantly influenced the quality of a machined surface of a KDP crystal. A uniform surface could not be obtained if the thickness was large. Likewise, the geometry of a tool tip, especially its rake angle, plays an important role [10]. Different tool rake angles and cutting thicknesses would produce different external stresses while machining, and hence would affect the friction and wear deformation of KDP.

To understand the relationship between the deformation and optical performance of a KDP crystal, and thus to facilitate its applications, extensive investigations have been carried out to explore the mechanical properties of KDP and $K(D_xH_{1-x})PO_4$(DKDP) crystals. Nevertheless, the studies to date have been mainly on the hardness, Young's Modulus and its anisotropy in different crystalline planes and orientations [11–20]. For instance, Wang et al. [17] reported that the periodic fluctuation of the Young's Modulus E and shear modulus G is related to the microstructural symmetry of the material. However, the stress distribution in an indentation test is significantly

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different from that in a machining process, and hence the results of the investigations cannot be used to reveal precisely the deformation mechanisms in the surfacing of KDP crystals during a single point fly-cutting in which the friction and wear and friction-induced heating play a key role. In addition, the friction properties, which have considerable effect on the machining stability and surface qualities, cannot be exploited by nanoindentation. It has been considered that nanoscratching could be more suitable for investigating the friction properties and wear deformation of KDP associated with machining. Lu et al. [21] studied the microscratching of KDP under a range of normal loads from 1.8 N to 6.8 N, and found that the material mainly experienced brittle fracture. They reported that the coefficient of friction was affected by the crystallographic orientation of KDP. However, the friction and wear of KDP in a surface finishing process is nanoscopic, and the mechanisms are still unclear.

Nanoscratching was found to be an appropriate method for characterising the wear and friction properties of a sliding system at the nanoscale. With the high resolution and high precision sensors available nowadays, many important parameters in a contact sliding process can be quantified, including the normal force, frictional force, scratching depth and microstructural effects of materials. Nanoscratching has therefore been applied to explore the wear resistance of coatings [22,23], near-surface wear mechanism of composites [24], deformation in the neighbourhood of a scratching tip [25], and adhesion properties between a thin film and its substrate [26].

The objective of this work is to examine the friction and wear properties of KDP, with the aid of nanoscratching, to provide some fundamental understanding for a better design and implementation of ultra-precision surfacing of KDP.

2. Material and methods

The samples upon investigation in this work were as-grown KDP crystals produced by the State Key Laboratory of Crystal Materials, Shandong University, China, using a rapid growth method. The details of the crystal growing process can be found in [27]. The crystallographic orientation of the sample was (001) with a misorientation less than 0.5°, which was confirmed by a commercially available X-ray diffractometer, Philips Panalytical MRD.

To eliminate the effect of any initial subsurface damage, a damage-free specimen surface before nanoscratching is necessary. To this end, a mechanical stress cleavage method was used in the sample preparation. This method, as illustrated in Fig. 1, had two steps. First, a machined KDP block was sectioned to a small piece perpendicular to the machined (001) surface. This piece was then cleaved in parallel to the machined (001) surface to break it into two sections. The cleaved surfaces were damage-free and could be used for the nanoscratching experiments conducted on a Triboindenter (Hysitron TI950, Hystron Inc., Minneapolis, USA) which is equipped with a transducer with a normal force/displacement sensor and two lateral force/displacement sensors. Two types of tips, a Berkovich tip and a conical tip, were used for the nanoscratching tests. The former had the radius of curvature of ~150 nm and the included angle from its perpendicular axis to one face 65.35°. The latter had an apex angle of 60° (conical indenter I) and 90° (conical indenter II), and the tip radii of 0.5 μm (conical indenter I) and 1.22 μm (conical indenter II), respectively. Two scratching loading modes, the constant and continuous, were tested individually. The procedure of a nanoscratching could be described as follows. At the beginning of each scratch, an indenter tip was brought into contact with a sample surface, and this contact point was defined as the middle of the nanoscratching track to be conducted. Then, the transducer was moved horizontally by half of the nominated scratching displacement (10 μm in this study) under an extremely small contact force of about 2 μN. After that, the transducer was moved to the other end of the nominated scratching displacement. A scratching motion along the above defined track was then started after a normal force was fully applied vertically to the sample surface. Under the constant load mode, the normal forces were 500 μN, 1000 μN, 2000 μN and 4000 μN, respectively, and under the continuous load mode, the normal force was varied linearly from zero at the beginning of the nanoscratching to 2000 μN at the end of the scratching track defined above. The scratching speeds were 0.5 μm/s and 5 μm/s, respectively. When reaching the end of the track of the defined 10 μm, the normal force was fully released vertically. During the experiment, the moisture and temperature in the testing chamber were controlled. The scratched surface topography was measured by an in-situ scanning probe microscopy (SPM) imaging function of the nanoindentation device. The resultant SPM images were analysed using the SPM image processing software Triboview installed with the manufacturer.

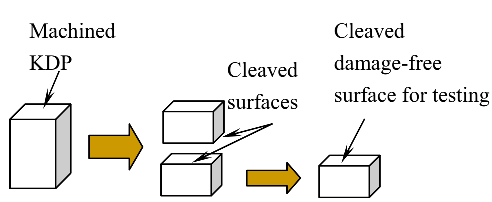

Fig. 1. Schematics of the preparation procedure of damage-free KDP surfaces for nanoscratching tests.

3. Results and discussion

3.1. Nanoscratching with the constant loading mode

3.1.1. Coefficient of friction

Fig. 2 shows the variations of the coefficient of friction (COF) using the conical and Berkovich indenters under constant loading mode with various normal forces/loads of 500 μN, 1000 μN, 2000 μN and 4000 μN, respectively, at a given scratching speed of 0.5 μm/s. It can be seen that the COF fluctuates in a similar manner in general. However, the effects of tip geometries and normal forces on the COF are obvious. At a lower normal force, the COF fluctuates at a higher frequency but a smaller magnitude, see Fig. 2(a and b). At the same lower normal load (e.g., at 500 μN or 1000 μN), the COF with the Berkovich tip varies more severely than those with the conical tips. When the load increases to 2000 μN, such tip geometry effect vanishes, but the COF starts to fluctuate at a lower frequency and increasingly larger magnitude, as shown in Fig. 2(c and d). It is interesting to note that when the normal load is 500 μN, the average COF corresponding to conical indenter II is the smallest, as shown in Fig. 2(a). At the normal load of 4000 μN, however, the average COF with conical indenter I becomes bigger than that of the Berkovich and conical indenter II. Such COF variations are caused by the change of the contact stresses and the extent of plastic deformation under the indenters when the normal force increases.

According to Wong et al. [28], assuming a Hertzian contact condition, the stresses induced by a conical indenter with a round tip are

$$P_e = \frac{F}{\phi[h+(\sqrt{2}-1)r]^2} \tag{1a}$$

$$P_s = \frac{3F}{2\phi a^2}\left(1-\frac{x^2}{a^2}\right)^{0.5} \tag{1b}$$

where P_e and P_s are the vertical components of stress exerted on the tapered and round/spherical surfaces of the indenter, respectively; ϕ represents the ability of the material recovery elastically; r and a are, respectively, depicted as the radius of the spherical tip of the indenter