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RESEARCH ARTICLE

Analysis of surface quality and processing optimization of magnetorheological polishing of KDP crystal

Shaoshan Chen [1,2] · Shengyi Li [1,2] · Hao Hu [1,2] · Guipeng Tie [1,2] · Chaoliang Guan [1,2] · Qi Li [1,2]

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Abstract A new non-aqueous and abrasive-free magnetorheological (MRF) polishing method is adopted for processing KDP crystal due to its low hardness, high brittleness, temperature sensitivity, and water solubility. Water content in magnetorheological fluids has an important effect on polishing surface quality. In addition to affecting the removal efficiency and fogging, the recrystallization of KDP can produce great influence on surface quality. This paper analyzes the formation of recrystallization process, chemical composition, influence factors and finds a method to restrain recrystallization. Results indicate the surface roughness of KDP crystal by MRF is 0.624 nm (Ra), 0.809 nm (RMS). We get neat, non-nick and ultra smooth surface after MRF polishing. Besides the improvement of surface roughness, the tool marks imported by single-point diamond turning (SPDT) are eliminated clearly. This has significant meaning on KDP crystal processing. Large size KDP crystal is widely used in high power laser system. The cutting tool marks will affect the laser induced damage threshold (LIDT) clearly. This means after MRF polishing the KDP crystal has better performance in high power laser system without recrystallization, obvious scratch, and tool marks.

Keywords KDP crystal · Non-aqueous magnetorheological · Water content · Recrystallization · Surface roughness

✉ Shengyi Li
492478230@qq.com

1 College of Mechatronics Engineering and Automation, National University of Defense Technology, Changsha, China

2 Hu'nan Key Laboratory of Ultra-precision Machining Technology, Changsha, Hunan 410073, China

Introduction

A KDP crystal (Potassium Dihydrogen Phosphate, abbreviated: KH_2PO_4) is a kind of typical soft brittle crystals. It has a large nonlinear optical coefficient, high laser damage threshold, and high transmittance from the near infrared to the ultraviolet band. Today, a KDP crystal is the only material can be used in the inertial confinement fusion (ICF), and in a strong laser weapon as a frequency converter and electro-optical switch [1]. It is primarily considered as one of the most difficult optical materials to process due to its low hardness, high brittleness, temperature sensitivity, and water solubility [2].

Today, the common process for finishing a KDP crystal is precision grinding, single-point diamond turning (SPDT), MRF polishing, and ion beam polishing (IBF). Traditional precision grinding will inevitably cause embedded grits, which will reduce the surface quality [3]. Philippe Lahaye at Lawrence Livermore National Laboratory (LLNL) in the United States achieves a super smooth surface with a surface roughness of 1.1 nm (RMS) using SPDT [4]. ShuYi at national university of defense technology achieves a smooth surface with a surface roughness of 1.95 nm (RMS) using ion beam machining in 2011 [5]. S.R. Arrasmith and others achieve a super smooth surface with a surface roughness of 1.6 ± 0.3 nm (RMS) using a non-aqueous magnetorheological fluid, adding a certain amount of diamond polishing powder in 1999 [6]. J.A.Menapace at LLNL laboratory proposed a new MRF process to finish a KDP crystal in 2010. They achieve the surface roughness of 0.65 nm (RMS), surface accuracy of λ/9.3 (PV) [7]. Besides this, MRF polishing can increase the LIDT, for it can remove diamond turning marks [8].

These results validate the feasibility and practicability of MRF polishing KDP crystal. While the KDP surface quality cannot satisfy the demand of high power laser system due to embedded iron powder, surface recrystallization and other

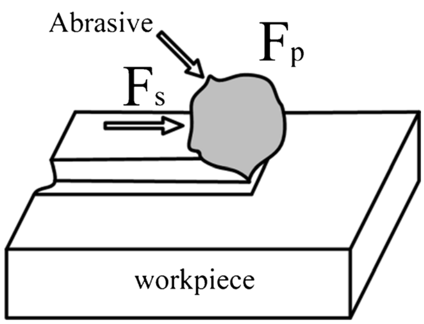

Fig. 1 Surface scratches after traditional MRF

factors. This paper will mainly analyse the surface recrystallization effects containing the formation of recrystallization process, chemical composition, influence factors, and the method to restrain recrystallization.

Removal mechanism of non-aqueous MRF polishing KDP crystals

The removal mechanism of traditional MRF polishing is utilizing shear stress to complete material removal when abrasive particles are embedded in the surface under pressure (Fig. 1). Traditional MRF fluid is mainly of carbonyl iron (CI) powder, abrasive particles, stabilizing agent and carrier liquid (deionized water). Traditional MRF polishing is easy to generate obvious scratch because of low hardness of KDP crystals. The abrasive particles are easy to embed into the surface (Fig. 2). The picture is measured with a KEYENCE-VHX-600 digital microscope (×500). The median particle size of CI particle is 3 μm. It is difficult to clean up and will affect the surface quality. For these reasons traditional MRF polishing reduces the LIDT of KDP crystals greatly. So traditional MRF polishing is unsuitable for processing KDP crystal [9].

KDP crystal is soluble in water. 1 g water can dissolve about 0.33 g KDP crystals equal to 141 mm^3 in volume at the temperature of 25 °C [10]. Basing on the water-solubility of KDP crystal, we add a small amount of deionized water

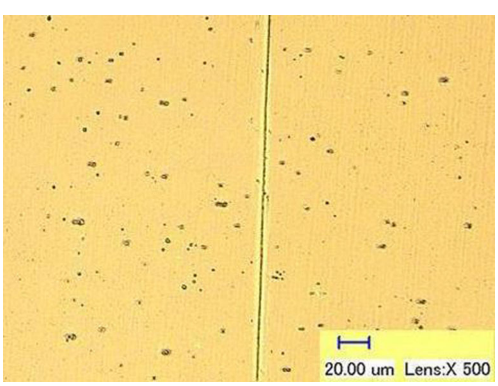

Fig. 2 Abrasive particles and CI powders embed into the surface

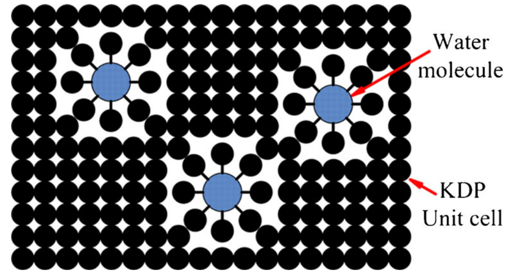

Fig. 3 Material removal of non-aqueous MRF

(about 4.5 wt.%) in magnetorheological fluid to replace the traditional polishing abrasive particles. Non-aqueous MR carrier liquid is nonvolatile hydrocarbon based. The CI powders are close to the polishing wheel while pressing the little water molecules in the magnetic field. These water molecules distribute in the outermost layer of the magnetorheological fluid ribbon. Different with traditional MRF polishing, non-aqueous MRF achieves material removal through water solution effect under much lower pressure (Fig. 3) [11]. The removal efficiency is stable when water content, polishing pressure, relative speed, and temperature are stable (Fig. 4). The MRF polishing spots are finished in 7 h at hourly intervals. The change of volume removal efficiency is <6.1 %. Thus, we can achieve a deterministic process basing on controlling dwell time. The carrier liquid with stable chemical properties constitutes a great proportion of MRF polishing fluid, and water accounts for only a small proportion. Thus, there is only a few amounts of water molecules contacting with KDP, and they are taken away quickly to avoid the fogging of the KDP crystal. We have carried out the experiment of polishing KDP crystals using the MRF equipment developed by our laboratory (Fig. 5).

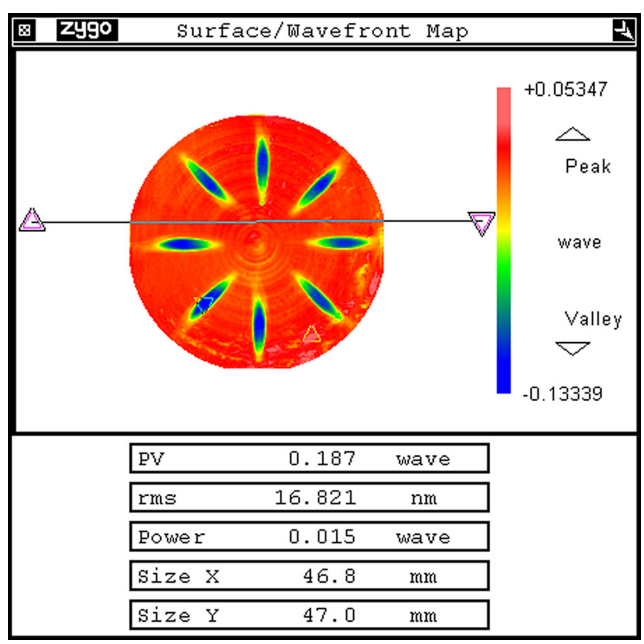

Fig. 4 The MRF polishing spot finished at hourly intervals

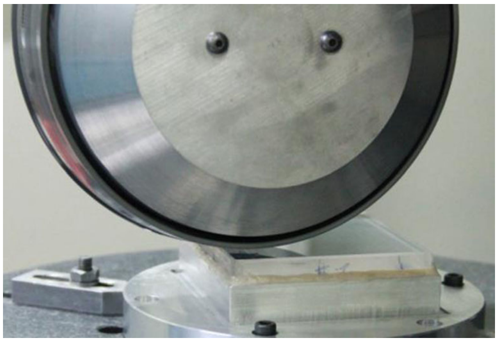

Fig. 5 Photo of MRF polishing KDP

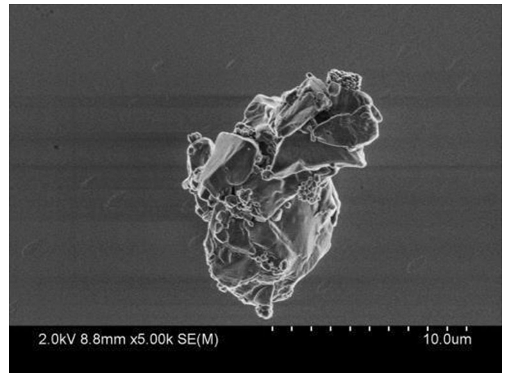

Fig. 7 The configuration of typical raised particles on the surface (under SEM)

Analysis of recrystallization and surface quality after MRF polishing

Analyze the principle of recrystallization process

The KDP crystal surface usually appears large number of small white raised particles after MRF polishing (Fig. 6a). We consider the raised particles are recrystallization of KDP. First we will analyze the principle of recrystallization process.

MR polishing fluid in contact with KDP crystals will form the solution of KH_2PO_4 on the KDP crystal surface because of water-solubility during MRF polishing of KDP. Commonly the polishing wheel of stays for a period of time from the polishing area. The residue MR fluid carrier will not be taken away quickly on the area of the polished KDP crystal surface due to adsorption effect. It remains there until the polishing wheel moves away entirely. Then the concentration of solution of KH_2PO_4 gradually increased. If dwell time is too long, there will be supersaturated solution on the KDP crystal surface. If the supersaturated solution is not stable the KH_2PO_4 will re-crystallize in areas within MRF polishing zone. If the supersaturated solution is meta-stable solution, the KH_2PO_4 will also re-crystallize for the solution contacting with KDP crystals that can be regarded as crystal nucleus. The polishing wheel mixing the solution will accelerate the recrystallization. The recrystallizations attach to the crystal surface and become part of the crystal, making the surface uneven with large number of small white raised particles which are observed under a MZDR0850 3D microscope (Fig. 6b). Thus, the surface quality of MRF polished KDP crystal is always relatively poor. The atomic force microscope (Bruke

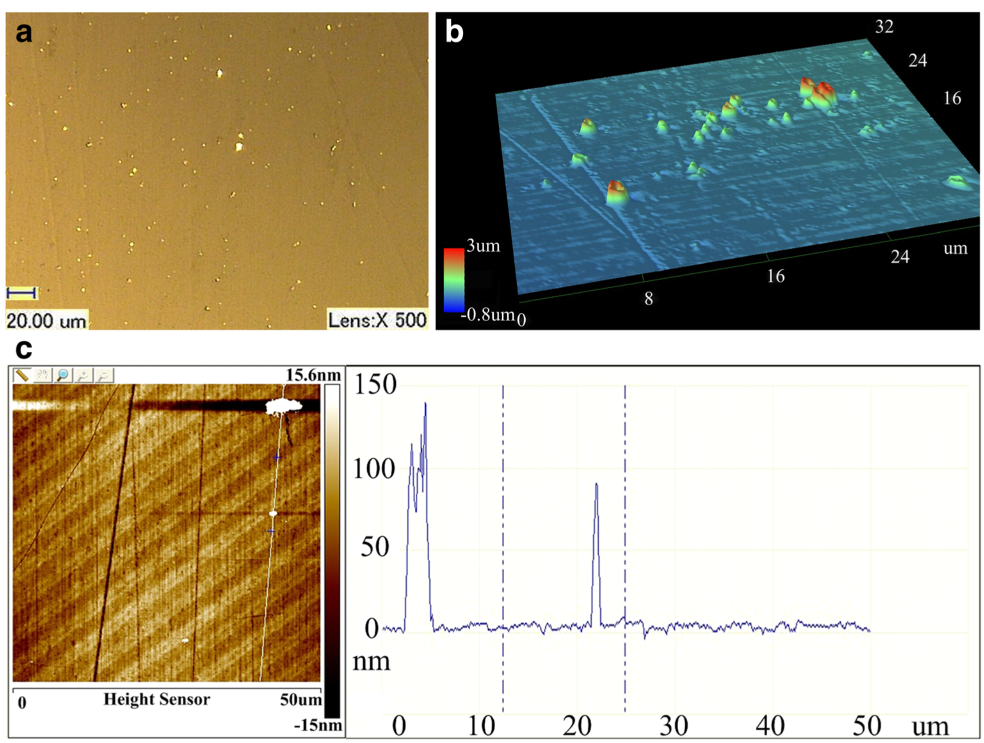

Fig. 6 **a** Recrystallization particles on the surface after MRF through a microscope (×500). **b** 3D microscope photo of the recrystallization (×8600). **c** The morphology of recrystallization particles under atomic force microscope

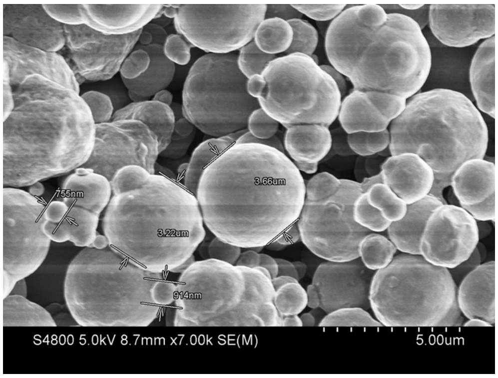

Fig. 8 The configuration of carbonyl irons powder (under SEM)

Dimension Icon) observation shows the diameter of these particles are distributed in the range 1–10 μm, the raised height difference is measured probably within 150 nm (Fig. 6c).

Analyzis of the chemical composition of recrystallization

MRF polishing KDP crystal is in weak alkaline environment. We use solid *KOH* to adjust PH of the MR fluid. The MR carrier fluid is non-volatile organic alcohol hydroxyl with stable chemical performance. So the main introduced impurity elements can only be Fe. That means the raised particles on the surface may be embedded CI particles. Results show that the small white raised particles are less than 10 μm in diameter, less than 0.15 μm in height, and have irregular shape as observed by a KYKY-EM3900 scanning electron microscope (SEM) (Fig. 7). The size of CI particle is in the range 2–10 μm and the CI particle have nearly inerratic spherical shape (Fig. 8). They are in the same range just consider the diameter. But the raised particles on the surface and CI particle have obvious difference according to the appeared shape and raised height.

In order to ascertain the chemical composition of the raised particles, we use an ICP-AES spectrometer to measure the content of various elements on the surface of KDP crystals before and after MRF polishing (Table 1). There are two samples which have been measured. We have measure its element content before and after MRF polishing. P is the referenced element. The results show the impurity content *Fe* is very low and we cannot find obvious change. If the raised particles are embedded CI particle it's easy to measured the change of impurity content of*Fe*because the diameter of CI particle can reach 10 μm. So we conclude that the small raised particles cannot be CI particle (Table 1).

Table 1 The content of various elements on the surface of KDP crystals before and after MRF polishing by ICP-AES spectrometer

	Element	Intensity of #1(mg/L)	Intensity of #2(mg/L)
Before MRF	Fe	0.0011	0.0019
	P	4.4063	4.1416
After MRF	Fe	0.0038	0.0017
	P	4.1179	4.2180

Based on the above analysis we conclude that the raised particles are not embedded CI particle. It's the recrystallization products of KDP in the solution of KH_2PO_4.

The ions in KDP aqueous solution are mainly K^+, H^+, OH^-, PO_4^{3-}, HPO_4^{2-}, and $H_2PO_4^-$ and H_3PO_4, as well as small amounts of other impurity ions. The solution PH can affect the ionization equilibrium of KH_2PO_4 [7]. The concentration of PO_4^{3-}, HPO_4^{2-}, $H_2PO_4^-$, H_3PO_4 changes along with the change of solution PH. The concentration of $H_2PO_4^-$ is the highest at PH 3.5–4.5 theoretically. It accounts for about 95 % proportion. The concentration of HPO_4^{2-} increases greatly and probably accounts for more than 60 % and the concentration of PO_4^{3-} increased to a considerable proportion when PH values are increased to ten. This variation tendency is shown in Fig. 9 [12].

MRF polishing KDP crystal is finished in weakly alkalescency environment at PH 9. Based on above analysis and Fig. 8 we conclude that the recrystallization products on the surface mainly are K_3PO_4, K_2HPO_4 and KH_2PO_4. K_2HPO_4 accounts for the most proportion. The crystallization process can be displayed by the following chemical equation.

$$KH_2PO_4 \overset{OH^-}{\rightleftharpoons} K^+ + H^+ + PO_4^{3-} + HPO_4^{2-} + H_2PO_4^- \overset{OH^-}{\rightleftharpoons} K_3PO_4(\downarrow) + K_2HPO_4(\downarrow) + KH_2PO_4(\downarrow)$$

Research of cleaning out recrystallization process technique

Based on the above analysis, the decisive factor of recrystallization is the formation of supersaturated solution of KH_2PO_4 when MRF polishing KDP crystal. The formation of supersaturated solution is related to the dwell time. So maybe we can control the polishing time to affect the formation of supersaturated solution and then control the recrystallization.

We have finished a few experiments in temperature 20 °C, humidity 35 % using a self-developed equipment. The workpiece comes from single-point diamond turning (SPDT), size parameters of 50 × 50 mm^2. The initial surface is clean

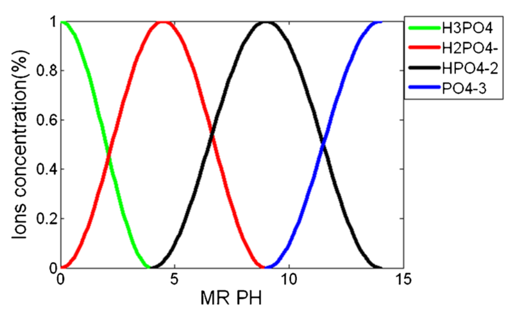

Fig. 9 The relation between various ion concentrations and solution PH values

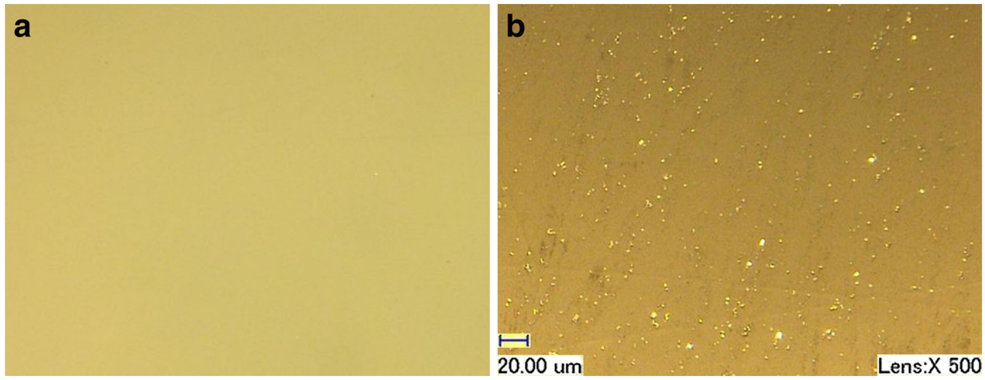

Fig. 10 Surface quality before and after MRF polishing (×500)

without recrystallization (Fig. 10 a). The polishing parameters of these experiments are: polishing wheel speed 340 rpm, penetration deep 0.2 mm, polishing time 180 min, water content 4 wt.%, and the peak removal efficiency is about 1 μm/min. The results show that the surface appeared to accommodate a lot of recrystallization particles after polishing. So the surface quality greatly worsened (Fig. 10b).

According to the previous analysis, main components of the recrystallize particles are K_3PO_4, K_2HPO_4 and KH_2PO_4. These three kinds of products have similar physical properties, all are soluble in water. This means these recrystallize particles can be removed away through appropriate polishing parameters. This can improve the surface quality, which had been worsened already.

We use the previous experiment workpiece to conduct a new experimental under identical condition. Polishing parameters are adjusted for: polishing wheel speed 340 rpm, penetration deep 0.1 mm, polishing time 50 min, water content 2.8 wt.%,

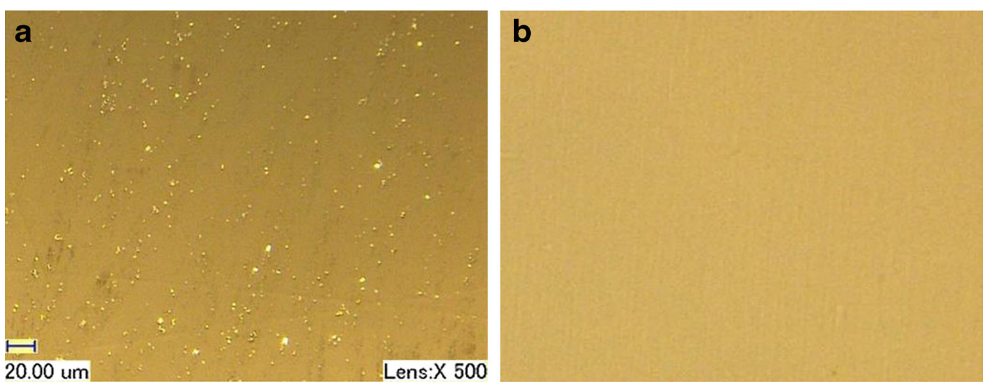

Fig. 11 Surface quality before and after changing MRF polishing parameters (×500)

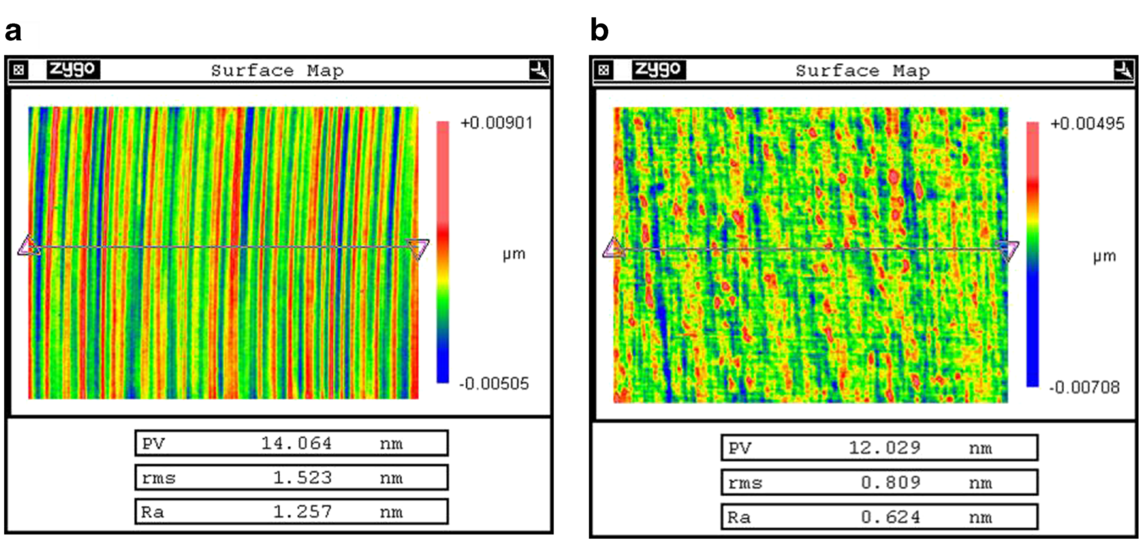

Fig. 12 Surface roughness before and after MRF polishing

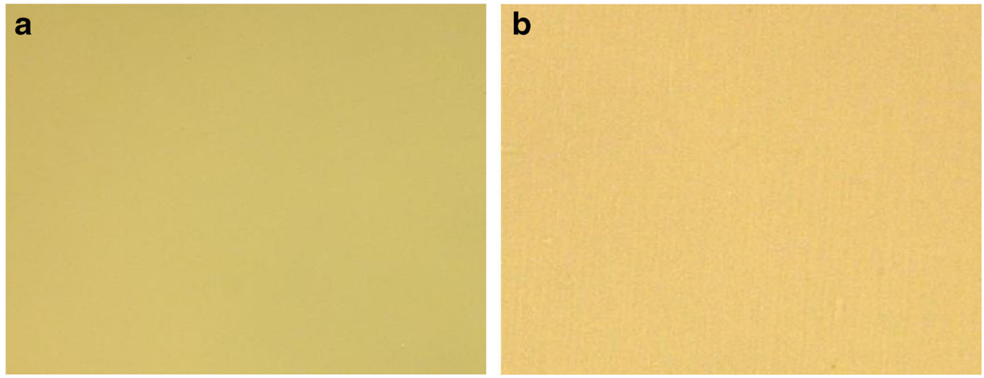

Fig. 13 Surface quality before and after MRF polishing (×500)

and the removal efficiency is about 0.2 μm/min. The surface quality improved significantly after three times polishing, and the material removal is about 0.6 μm (Fig. 11). We can clearly see the surface recrystallization particles have all been disappeared compared to with the surface before.

MRF polishing results and discussion

It's possible to get very good surface quality via controlling polishing parameters based on the above analysis. We have finished correlative experiments and achieved ideal effect. The workpieces come from SPDT with a surface roughness of 1.523 nm (RMS), 1.257 nm (Ra) (Fig. 12). The surface roughness is measured with a NewView-700 white light interferometer, a 10× objective that would make the field of view 940 × 700 μm^2. The surface is clean and has no obvious scratch before MRF polishing. The surface roughness is 0.809 nm (RMS), 0.624 nm (Ra) after three processes of MRF polishing with material removal about 1 μm. The surface is clean and has no obvious scratch after MRF polishing (Fig. 13).

From the results we can clearby see that the surface roughness is improved a lot. Large size KDP crystal is usually used in high power laser system. The high frequency error will induce the diffuse waste of laser power and reduce the LIDT [8, 13]. Besides the improvement of surface roughness, the tool marks imported by SPDT are eliminated. This has significantly effect on KDP crystal processing. The tool marks will lead to residual stress which will affect the LIDT clearly. By controlling the recrystallization we can get high quality KDP crystal surface with better surface roughness, no obvious scratch and no cutting tool marks that means better performance in high power laser system [14, 15].

Conclusions

We have finished several experiments of MRF polishing of KDP crystals. Analyzed how water content affects the surface quality after MRF polishing. This paper mainly analyzes the formation of recrystallization process, chemical composition, influence factors and finds a method to restrain recrystallization. We succeed to get high quality surface of KDP crystal. The surface is clean and has no obvious scratch with surface roughness of 0.809 nm (RMS), 0.624 nm (Ra). Beside the improvement of surface roughness, the tool marks imported by SPDT are eliminated. This means after MRF polishing the KDP crystal will have better performance in high power laser system without recrystallization, obvious scratch and tool marks.

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