FLOAT POLISHING OF CALCIUM FLUORIDE SINGLE CRYSTALS FOR ULTRA VIOLET APPLICATIONS

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Abstract

Calcium fluoride single crystals for ultra violet applications have been finished to flat by the float polishing process with 7nm-diameter silicon dioxide powder, pure water and a tin lap. The 42nm P-V and 5.81nm rms flatness were obtained on a sample of 100mm in diameter. Atomically-smooth surfaces were also obtained on the surface. The surfaces were measured with optical surface measuring apparatus, atomic force microscope and transmission electron microscope. There is no damage layer on float-polished surface due to machining.

Keywords: Calcium fluoride; Float polishing; Atomic surface structure; Lithography; Transmission electron microscopy

1. Introduction

The light sources for micro-lithography has been changed from the g-line, i-line of mercury lamp to excimer lasers of Deep Ultra Violet (DUV) region, and will be planed to use ArF excimer and F_2 excimer in Vacuum Ultra Violet (VUV) region. However, there are some problems on optical components for using such shorter wavelength lasers. The problems are listed as limitation of transparent materials for such regions, no established machining technology for such materials, lack of knowledge on interaction between the materials and lasers, and lack of data of laser induced damage thresholds on the materials. DUV/VUV lithography-grade calcium fluoride (CaF_2) is the only candidate for such applications. The demands on the surface are high precision in figure and smoother surface roughness, though the crystal is soft and relatively high thermal expansion coefficient, as well as higher laser-induced damage threshold, higher uniformity in index and higher transparency. The float polishing has being finished various single crystals and optical glasses ultra-finely without deformed layer [1-3]. We will try to use this technology to get DUV/VUV lithography-grade surface on CaF_2 single crystals. The finished surface geometry will be also measured in the range of macro to atomic scale.

2. Experimental Procedure

DUV-grade CaF_2 single crystal samples of 35mm×35mm×10mm in dimensions were cut oriented in the (111) plane. The samples were ground, optically polished and finally float-polished with SiO_2 powder of 7nm in nominal diameter and pure water on a tin lap as shown in Fig. 1. The float polishing machine [3] has a hydrostatic oil bearing of high rigidity and damping as well as high rotational accuracy. The tin lap having many concentric grooves of 2mm in pitch and 1mm in depth was cut into flat with a single point diamond tool. The temperature of polishing fluid was controlled in a range of $\pm$ 0.01K. Optically polished samples were also obtained from the market. One sample has dimensions of 100mm in diameter and 20mm in thickness. This sample was float-polished to get a large plane.

The finished surfaces were observed under a Nikon's Nomarski differential interference microscope, and measured with ZYGO's GPI-XP, WYKO's TOPO-2D, Seiko's Atomic Force Microscope (AFM). Subsurface damage was observed by an etching technology with the Nikon's Nomarski microscope.

The cross-sections of polished surfaces were observed with JEOL's JEM-2010 Transmission Electron Microscope (TEM) operated at 200kV. For the TEM observations, the optically polished and float-polished crystal samples were sectioned oriented normal to the (111) surface. The samples were then ground and polished with Al_2O_3 powder of about 50 nm in diameter. These samples were finally ion-polished with Fischione's Model-1010 ion mill.

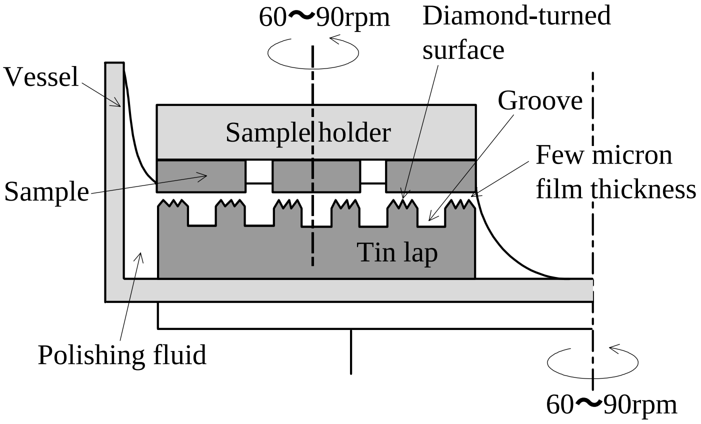

Fig. 1 Principle structure of a float polishing machine.

3. Experimental Results and Discussion

Figure 2 shows the flatness of the float-polished CaF_2 sample of 100mm in diameter and 20mm in thickness, measured with ZYGO's GPI-XP phase measuring interferometer system. The surface flatness of 5.81nm (λ/109;λ=633nm) rms and 42nm (λ/15) P-V was obtained on a float-polished CaF_2 sample of 100mm in diameter. By controlling polishing conditions, especially temperature, 3.16nm (λ/20) P-V flatness will be obtained very near future by using the float polishing

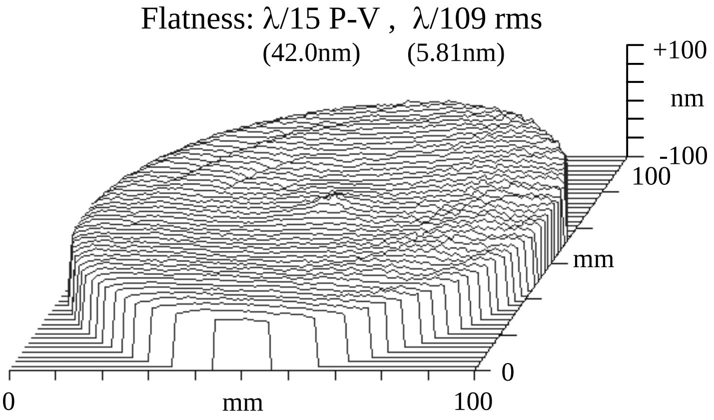

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Fig. 2 Surface flatness of a float-polished CaF_2 sample of 100mm in diameter, measured with GPI-XP.

Figure 3 shows the surface roughness of a float-polished CaF_2 sample, measured with TOPO-2D at 40X. The surface roughness numbers are 0.228nm rms, 0.183nm Ra, 1.33nm P-V in the measuring length of 320μm. This number is about the same number in float-polished BK-7 and Zerodur [3], however this number is the smallest among many samples that we have measured optically polished CaF_2 samples from the market.

Figure 4 shows the surface roughness of a float-polished CaF_2 sample, measured with an AFM in 10μm by 10μm area. The surface roughness numbers are 0.162nm rms, 0.131nm Ra, 1.10nm P-V. In the case of polishing CaF_2 single crystals for infrared applications, the surface roughness of 0.235nm rms, 0.188nm Ra, 1.65nm P-V was obtained. From this result, it is clear that the polished surface roughness depends upon the purity of materials. From the simulation of 3D surface roughness on ideal atomic structure [4], the ideal surface roughness of CaF_2 may be estimated 0.033nm rms. Therefore, the surface roughness less than 0.1nm rms can be obtained on CaF_2 samples.

Various (111) CaF_2 samples such as float-polished, cleaved and ultra-precisely ground were prepared and etched with hydrochloric acid to reveal the dislocation on the surface. The float-polished sample shows etch pits of triangular pyramid in shape. The distribution density of such pits is same as those on well-cleaved surface. From the observation of etch pits, it is clear the float-polished surface is free from dislocation due to machining.

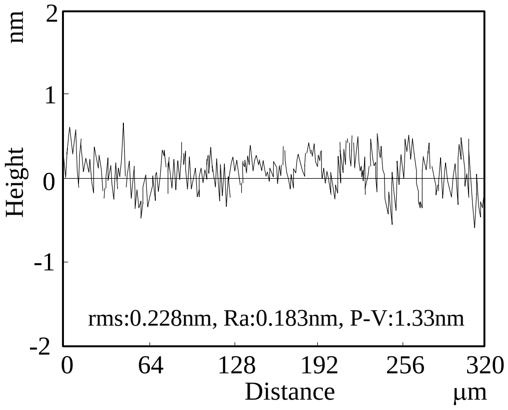


Fig. 3 Surface roughness of a float-polished CaF_2 sample, measured with TOPO-2D.

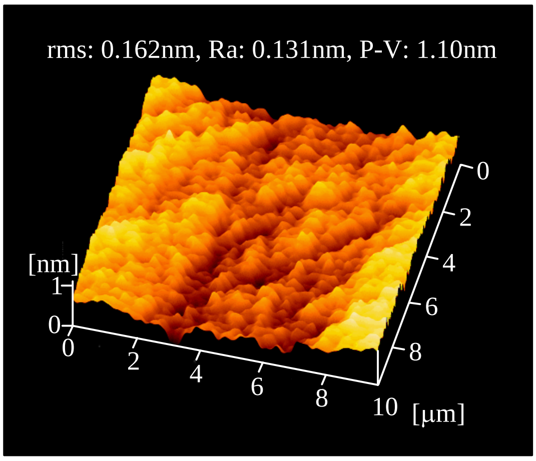
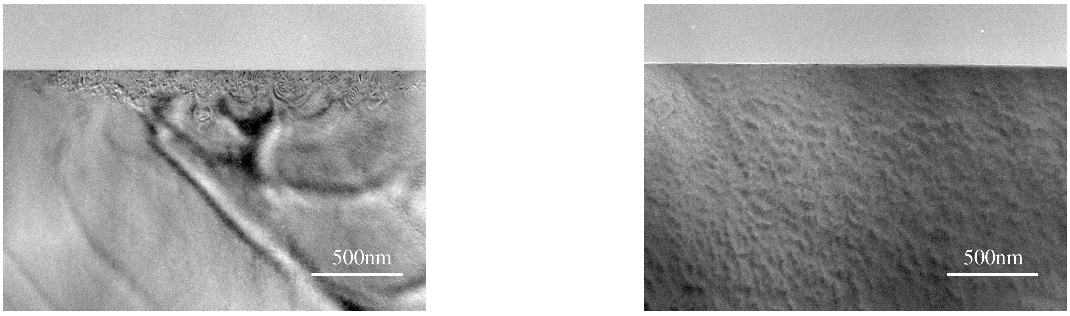


Fig. 4 Surface roughness of a float-polished CaF_2 sample, measured with SPA270.

Figure 5 shows cross-section TEM images of optically polished and float-polished CaF_2 crystal surface, obtained with an incident electron beam parallel to the (111) plane. The images show high-density strain field contrast at the subsurface of optically polished specimen. This indicates higher dislocation density exist at the subsurface of the sample. On the other hand, there is no such strain contrast attributed to dislocations due to machining on float-polished surfaces. These TEM observation results are consistent with those obtained by the etching experiments.



(a) Optically polished surface

(b) Float polished surface

Fig. 5 Cross-section TEM images of polished CaF_2 samples.

Figure 6 is a high-resolution TEM image of float-polished surface taken with the same electron beam incidence as Figure 5. The image shows fine fringes of (111) plane with a lattice spacing of 0.32 nm. This profile-view image shows the float-polished surface consists of fine lattice steps of the (111) plane. In a small area on the terrace of the surface, an atomically smooth planes parallel to the (111) lattice are achieved, though the surface roughness derives from steps of a few nm between (111) planes. These step profiles are considered due to mismatch of the sample surface orientation from the (111) lattice plane. And thus, the result shows that if we can obtain a sample surface oriented parallel to the (111) plane in ultra high precision, the surface roughness less than 0.1nm rms may be obtained by float polishing.

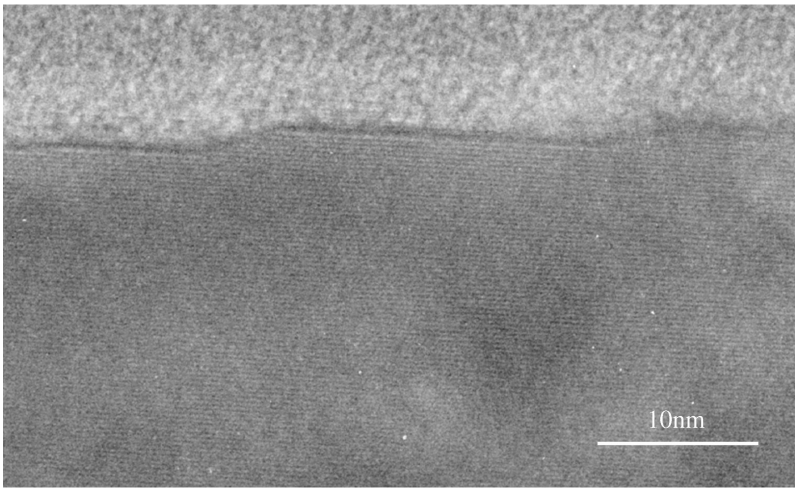


Fig. 6 TEM image of a float-polished CaF_2 surface, indicating the (111) lattice fringes.

4. Conclusions

Calcium fluoride single crystals were finished to flat by the float polishing process with 7nm-diameter silicon dioxide powder, pure water and a tin lap. The 42nm P-V and 5.81nm rms flatness were obtained on a sample of 100mm in diameter. Surface roughness of 0.235nm rms, 1.33nm P-V in measuring length of 0.32mm was obtained on the float-polished surface, measured with TOPO-2D at 40X. The surface roughness of 0.162nm rms, 1.10nm P-V was obtained on the float-polished sample, measured with an AFM in 10μm by 10μm area. The polished surface roughness depends upon the purity of materials. The etching technology makes clear that the float-polished surface is free from dislocation due to machining. Transmission electron microscopy shows the float-polished surface is atomically smooth on (111) surface, however there are lattice steps due to miss orientation of the sample from (111) surface.

Acknowledgements

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