Planarization of Lithium Niobate Surface Using a Thin Film Catalyst in Pure Water

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Abstract. A catalytically assisted etching method, named Catalyst-Referred Etching (CARE) was applied to the planarization of Lithium Niobate (LN) surface, which is widely used for optical waveguides, optical modulators, piezoelectric applications. The study demonstrates that an atomically smooth surface with less than 0.1 nm root-mean-square roughness could be achieved on a LN substrate using a thin metal film and pure water as the catalyst and etching solution, respectively. All residual stress and surface damage could be removed completely thanks to the removal mechanism of CARE.

1 Introduction

There is a growing demand for ultraprecision optical components for scientific and industrial applications, especially in extreme ultraviolet (EUV) and X-ray regimes. X-ray microscopy is one of the ultimately non-invasive observation techniques. Using short-wavelength light, scientific imaging of cutting-edge materials /biological samples has become possible thanks to the ultra-precision optical components [1,2]. Although a low figure error of several nm is required for the optics, a highly ordered surface with a root-mean-square (RMS) roughness at the level of several tens of picometer is greatly desired for the highest reflectivity and the lowest unwanted scattering [3].

Recently, Matsuyama proposed an X-ray mirror element for X-ray microscopy using piezoelectric material as a mirror substrate [4]. Thanks to the non-linear optical, electro-optical, piezo-electrical, and pyro-electrical characteristics, single crystal Lithium Niobate ($LiNbO_3$: LN) is the material of choice for the required mirrors [5]. This novel deformable mirror could correct the low spatial frequency mirror shape with nanometer precision with high stability. However, it is challenging to polish the mirror surface to a highly ordered surface.

Chemical mechanical polishing (CMP) is widely used as a final polishing method. In CMP, the state-of-the-art balancing between chemical modification and mechanical removal needs to be determined before a practical polishing. A recent study reported an effect of the chemical-mechanical balance on the polished SiC surface morphology [6]. Several types of step-and-terrace structures were produced depending on the roles of chemical modification and mechanical removal. Thanks to the high hardness of SiC, the mechanical factor is calibrated to avoid any serious subsurface damage and residual stress on the polished surface. However, it is difficult to completely avoid all mechanical damage, especially for a low-hardness material such as silica glass and LN. For such materials, chemical-based polishing methods have advantages in producing a smooth and mechanical damage-free surface.

A new polishing method using a catalyst has recently been developed named Catalyst-referred Etching (CARE) [7]. CARE has been successfully applied to the polishing of oxides and functional material surfaces, such as silica glass, SiC, GaN. Unlike CMP, chemical polishing is sensitive to the electronic and geometrical factors of the polished surface. Thus, chemical attacks at a step edge and a terrace site of a crystalline material would be different. The chemical etching prefers processing at a step edge that leads to the step-flow etching type, resulting a single step-and-terrace structure observed on the polished surface [8]. The removal mechanism of CARE was confirmed to be hydrolysis catalyzed by forming chemical bonds with O/OH at the interface, stabilizing the hypervalent state and promoting the chemical reaction at the interface [9].

In this study, we apply catalyst-referred etching (CARE), an abrasive-free polishing method, to the planarization of a crystalline LN surface, using Pt as the catalyst and pure water as the etching solution. An atomically smooth surface with sub-Angstrom root-mean-square roughness is achieved on the LN substrate.

2 Experimental procedure

To demonstrate the feasibility of CARE for planarization of a LN substrate, a 2-inch single crystal LN with Y-off-

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cut 36 degree was used in this study. The schematic of the apparatus used in this study is shown in Fig. 1.
A thin film of catalyst was deposited on a polishing pad, which has narrow grooves on its surface to supply etchant to the sample surface. The sample is put in a holder and pressed onto the catalytic pad with a pressure of 20 kPa by an airbag placed behind the sample. The sample and the pad are immersed in pure water during polishing. The planarization is promoted by rotating the sample and the pad on two parallel axes at approximately the same speed of ca. 10 rpm.

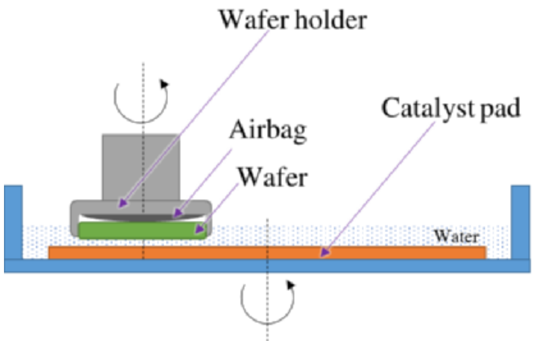

Fig. 1. Schematic of the apparatus

The experimental conditions are summarized in Table 1.

Table 1. The experimental conditions.

	Conditions
Applied pressure	20 kPa
Rotational speed	10 rpm
Catalyst	Pt
Etchant	Pure water

The surface before and after CARE were examined by an atomic force microscope (AFM; Scanning Probe Microscope, SPM-9700HT), phase-shift interference microscope (ZYGO NewView 9000).

3 Results and discussion

The phase shift interference microscopy images of the as-received (left) and CARE-processed (right) LN surfaces are shown in Fig. 2. The size of each image is 170 µm × 110 µm. After CARE processing, the surface roughness was improved from 0.15 nm RMS (as-received surface) to 0.1 nm RMS (CARE-processed surface).

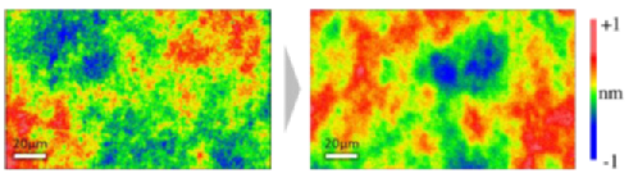

Fig. 2. Phase shift interference microscopy images of the as-received (left, 0.15 nm RMS) and CARE-processed (right, 0.1 nm RMS) LN surface.

AFM images of the as-received (left) and CARE-processed (right) LN surfaces are shown in Fig. 3. Size of each surface is 2µm × 2µm. The surface roughness is improved from 0.175 nm RMS (as-received (left)) to 0.064 nm RMS (CARE-processed (right)).
The surface roughness of the as-received surface is equivalent to previously reported the surface prepared by CMP [10]. After CARE processing, the surface is smoothened, evidenced in low surface roughness of 0.064 nm RMS.

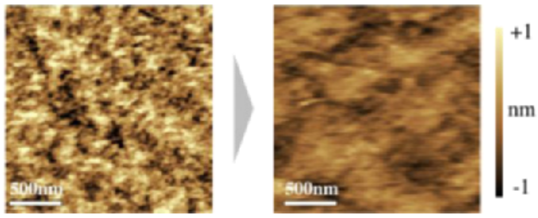

Fig. 3. AFM images (2µm × 2µm) of the as-received (left) and CARE-processed (right) LN surfaces

In CARE processing, the material removal is based on a chemical reaction that occurred at the interface of the catalyst and the substrate. Thanks to its removal mechanism, there is no subsurface damage or residual stress that could introduce on the processed surface. Thus, CARE method is a good choice as a final surfacing method to remove high spatial frequency error and surface damage.

4 Summary

The study demonstrated the feasibility of CARE as a final polishing method for LN substrates. The CARE processed surface is atomically smooth with RMS of less than 0.1 nm RMS over the whole surface. Thanks to its removal mechanism, a geometrically and crystallographically highly ordered surface can be produced.

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