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Roughness Reduction of Large-Area High-Quality Thick Al Coatings for Large-Size Echelle Gratings by Chemical Mechanical Polishing

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Large-area high-quality thick Al coating is one of the most important factor which always acts as a ruling substrate in realizing high-performance large-size echelle grating ruling process. Based on our previous work, we proposed use of precise mechanical polishing methods to further reduce surface roughness of thick Al films. Surface roughness Rq of 12 μm-thick Al film can be effectively controlled under 25 nm by early reported multi-step deposition process. The chemical mechanical polishing method was chosen in this study to improve the surface quality of thick Al coating. Rq of Al film was significantly reduced to 5.26 nm as a consequence. The surface morphology, cross section and film hardness of the polished samples were also investigated in detail. Finally, the echelle grating was fabricated on the polishing sample and the diffraction efficiency was 50% at 632.8 nm laser and 36th order. This work has great potential for both high-quality thick Al film research and large-size echelle grating performance enhancement.

Keywords: Thick Al Coatings, Echelle Grating, Chemical Mechanical Polishing.

1. INTRODUCTION

Echelle grating was first invented by Harrison in 1949[1] and it has nowadays been applied extensively in spectral detection and analysis fields due to its high diffraction order, broad spectrum range, high dispersion, and excellent resolution.[2–4] Large-size echelle grating can achieve a super-high spectrum resolution up to 106 as an extension to conventional echelle grating, owing to its extremely high diffraction order and large aperture.[5,6] It has currently become an essential component of various optical devices applied in astronomical observation, element detection and high power lasers.[7,8] When the size of echelle grating is larger than 300 mm × 300 mm, it can only be currently prepared by grating ruling engine, and[9,10] its fabrication method involves extruding and polishing of Al films on a grating substrate using diamond graver, causing the Al coatings to generate previously designed deformations and exhibit periodic nanostructure. The fabrication method for the echelle grating is a long-time Al film ruling process, so controlling the surface roughness of Al film is very important.[11,12]

When the designed ruling depth of an echelle grating is around 5 μm, the thickness of Al film should be 2–2.5 times the ruling depth, which is in the range between 10–15 μm, according to grating ruling experience.[13] For ensuring plasticity and shaping ability of such thick Al films during the long-time ruling process, fabricators mostly use thermal evaporation and electron-beam evaporation method to prepare Al films for ordinary gratings. We proposed a novel multi-step deposition method for high-quality and large-area thick Al film in our previous research.[11] Compared with conventional thermal deposition method, the multi-step deposition process effectively suppressed the growth of large-size grains by controlling deposition temperature via cooling per time interval. We successfully prepared Al film with surface roughness Rq less than 25 nm.

We in this paper proposed using direct mechanical polishing method for further reduction of surface roughness. As known, the history of Al mirror mechanical polishing can be traced to the last century. Direct polishing process has been already successfully used to improve the Al mirror coating surface quality. Single point diamond turning (SPDT) method is the first option, when considering

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the polishing method, which is widely used to achieve good surface quality and accuracy in aluminum alloy.[14] Tool marks are sometimes left on the polishing substrate surface after SPDT during the polishing process. The periodical inevitable scars are fatal for conventional Al reflection mirror but the influence of SPDT on thick Al coating for echelle grating should be measured and investigated. It is essential to find another direct polishing method for surface roughness further reduction of thick Al film if it doesn't work. The chemical mechanical polishing (CMP) method is thus proposed to be the alternative option.[15–17] There are no reports until now on the use of direct polishing method to reduce surface roughness of thick Al coating, which shows its high difficulty in processing from different angles.

We in our previous work expounded the difference between Al reflector coating and echelle grating Al ruling coating.[11] For echelle grating, controlling the quality uniformity, defect quantity, film hardness and inside compactness of thick Al film is of great importance. All above factors should be considered when the mechanical polishing process is introduced. Unexpectedly large surface roughness is still a fatal problem for thick Al films as mentioned, and it is the research emphasis of this study. Superiority and effectiveness of direct mechanical polishing method was discussed and demonstrated through comparison experiments. First, we present the influence of SPDT and CMP process on the thickness change, surface reflectance and surface roughness of Al films by experimental measurements. Then, on the basis of measurement results, better treatment method was confirmed. We finally discuss the change of inner layered structure and coating hardness before and after mechanical polishing. These two factors are significantly important for echelle grating performance. Optimized polishing parameters were in the end obtained and surface roughness of thick Al film for echelle grating has a remarkable improvement.

2. EXPERIMENTAL DETAILS

In this study, a piece of 520 mm × 420 mm × 200 mm neoceramic glass was used as substrate material that has an extremely low expansion coefficient, lower than conventional glass materials. Thick Al films were deposited on the electron-beam evaporation coating system with a chamber diameter of 1800 mm, which was equipped with two electron guns (TeleMark II). Figure 1 shows schematic structure diagram of the deposition system. The substrate was loaded on a rotated plane fixture instead of planetary fixture after chemical cleaning in acetone and ethanol, because large-area thickness uniformity was more conveniently obtained by adjusting fixture height. In order to ensure the radial-quality uniformity of Al films, the double electron-beam co-evaporation reported in our previous work was performed to deposit thick Al films at a pressure of 2×10^{-4} Pa. The deposition rate was controlled at 100 Å/s, which was monitored by quartz crystal.

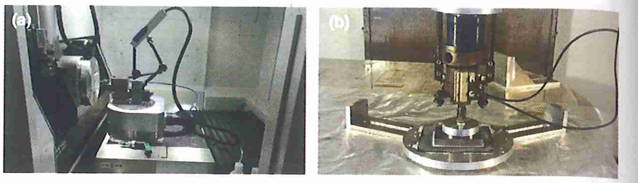

Fig. 1. Direct polishing instruments (a) SPDT, (b) CMP.

Here we utilized two mechanical polishing methods to reduce the surface roughness of thick Al coatings through single point diamond turning. In other words, we used vacuum chuck to fix and cut the part as a rotational symmetry mirror as shown in Figure 1(a). Another method involved chemical mechanical polishing. In this case, the polishing tool was made on a thick aluminum pack plate attached with a polishing cloth. Al coating substrate was pressed onto a polishing pad by a wafer carrier while polishing powder was provided to the coating and pad interface. SiO_2 polishing powder (abrasive particle size was 50 nm) was applied in the polishing operation with 100 rpm orbital motion and 0.05 MPa pressure under pH 8 condition as shown in Figure 1(b). The uniformity of Al coating within 700 mm diameter was less than ±2% after the CMP procedure. Besides, we evaluated coating thickness, inner structure, surface reflectance, surface roughness and hardness of thick Al films by using stylus profiler (Nanomap 500LS of CAEP), scanning electron microscope (SEM, JSM-6510 of JEOL), spectrophotometer (Lambda 1100 of PerkinElmer), atomic force microscope (AFM, EDG of Bruker) and nano indentation (Nano Indenter G200 of Agilent), respectively.

3. RESULTS AND DISCUSSION

The SPDT method was preferred for its sophisticated and stable process at the beginning of our polishing experiment. The required thick Al coating for echelle grating had exact film hardness and it was rather soft, different from other Al reflection mirror coating and Al alloys. So, polishing pressure was controlled as low as possible during the SPDT process. The surface roughness of Al coatings prepared by multi-step deposition method had a 30.4 nm Sq. in our previous work as shown in Figure 2(a). Figures 2(b), (c) show experimental results from AFM of Al coating surface after SPDT method. As seen, periodical polishing marks were left after the SPDT process, showing single-line marks (Fig. 2(b)) or multi-line marks (Fig. 2(c)). The Al coating treated with SPDT method could not be used as echelle grating ruling substrate for the polishing marks although the surface roughness Rq of thick Al coating was extremely lowered to 3.84 nm, because the marks directly influence the blazed face flatness of echelle grating.

The SPDT method was abandoned and chemical mechanical polishing method was put forward as the new

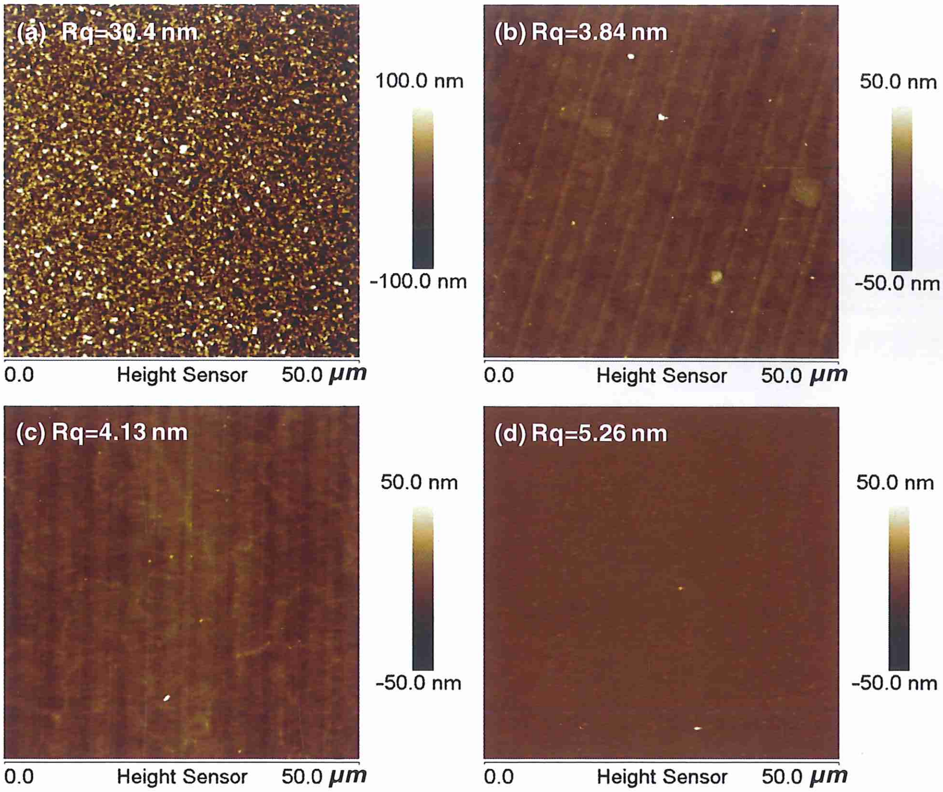

Fig. 2. Top surface AFM image of thick Al coating prepared by multi-step deposition method (b), (c) Top surface AFM images of thick Al coating after SPDT process. (d) Top surface AFM image of thick Al coating after CMP process.

approach. Figure 2(d) shows the top surface AFM image of thick Al coating after the CMP process. Surface roughness exhibited novel reduction from 30.4 nm to 5.26 nm. It can therefore be concluded that, for thick Al coating, the polishing effect of SPDT was a little better than that of CMP but the surface quality of treated Al coating after CMP was just good. There were no obvious polishing marks left after the CMP process. The CMP process was thus decided as the final polishing method for surface roughness further reduction of Al coating.

Thick Al coating was applied as the echelle grating ruling substrate in our experiments, with strict thickness requirement. As known, mechanical polishing method causes thickness reduction, so the thickness change should therefore be investigated. Figures 3(a), (b) show the Al coating thickness measurement before and after the CMP process. In the microscope photographs of Figures 3(a) and (b), the left halves are thick Al coatings and right halves are glass substrates. The thickness of the Al coatings was measured by calculating the altitude intercept of two areas. The thickness of Al coating prepared by multi-step deposition technique was 10.6385 μm as shown in Figure 3(a). The thickness changed to 10.4671 μm as shown in Figure 3(b). 171.4 nm-thick Al film was lost during the polishing process, and such thickness attrition had nearly no influence on the echelle grating ruling.

Besides the thickness, surface reflectance is a key parameter to evaluate the thick Al coating quality.[18,19] Figure 4 shows the surface reflectance of thick Al coating before and after the CMP process. The surface reflectance of thick Al coating had nearly 40% improvement after the CMP process. We also demonstrated in our last study that the radial-quality uniformity, as surface reflectance difference along the radial direction, should be analyzed in detail, to assess which has great impact to diffraction efficiency uniformity of large-size echelle grating. The surface roughness is determined by surface roughness and internal compactness. The surface reflectance of Al coating increased remarkably when introducing the CMP process. The radial-quality uniformity has also great improvement by surface reflectance enhancement of whole Al coating.

As known, the inner quality should also be investigated adequately besides the surface quality of the Al coating. The thick Al coating prepared by multi-step deposition method has a special four-layer micro-structure unlike the conventional thick Al coating. For the polishing technique is a process of exerting pressure on the Al coating, the inner structure and multi-layer interface possibly changed. Figures 5(a) and (b) show the cross-sectional SEM images of Al coating before and after the CMP process. By comparing the photographs, the multi-layer structure had no obvious change, and by surface morphology comparison, the Al coating after the CMP process had a

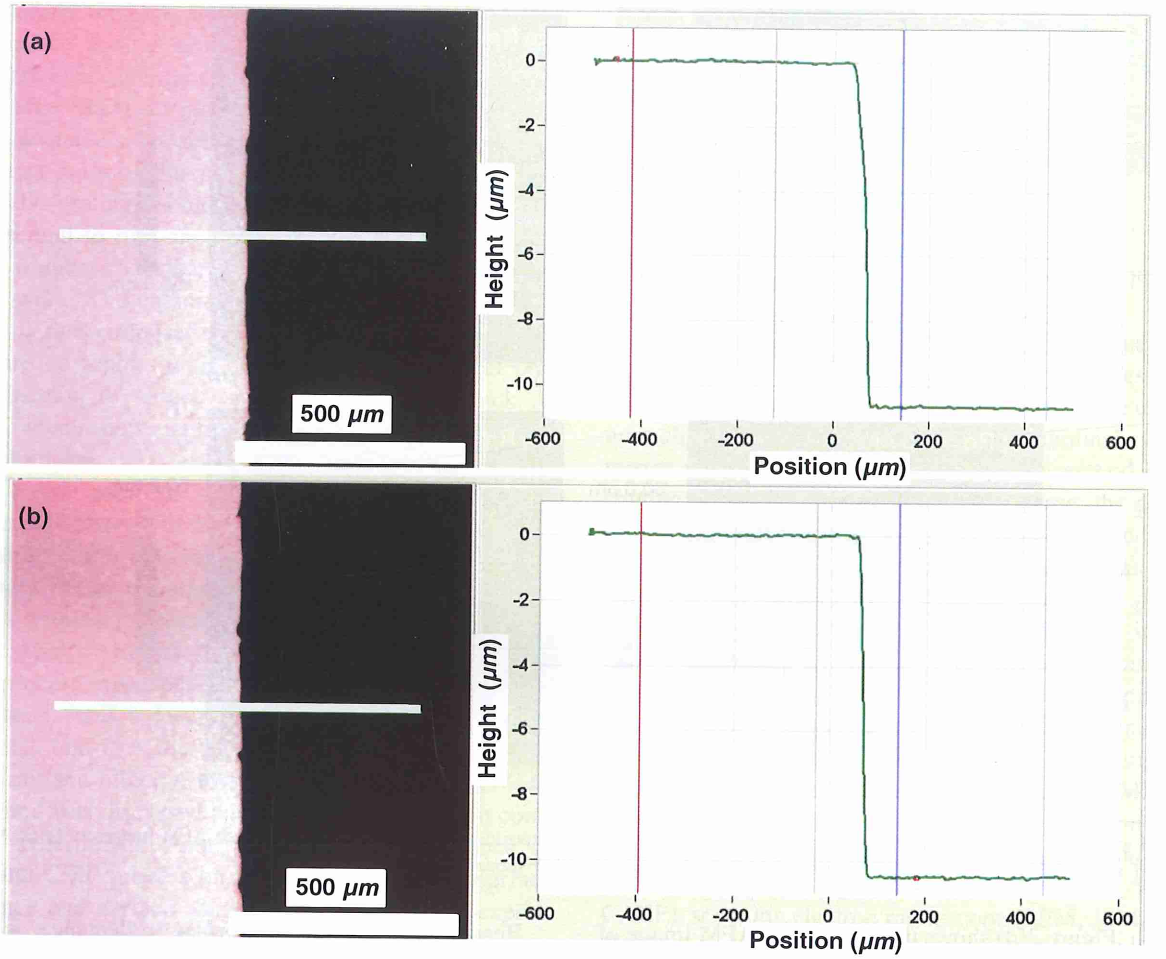

Fig. 3. Al coating thickness measurement (a) before and (b) after CMP process.

much flatter surface. In this way, it can be concluded that the CMP process could not only achieve further reduction of surface roughness, but also guaranteed stability of inner micro-structure.

Figure 5(c) shows the enlarged SEM image of Figure 5(a) where different component parts of multi-layer thick Al structure are shown. The black area represents the upper surface of the first Al layer. The SEM image in Figure 5(c) is a cross-sectional image. The perfect cross-sectional sample was very difficult to prepare because of high ductility of Al material. Sometimes the prepared cross-sectional sample presents overlapped layer behavior, while sometimes the cross section presents a separated-layer behavior as shown in Figure 5(a). In this case the cross-sectional image shows a dark area between two single Al layers.

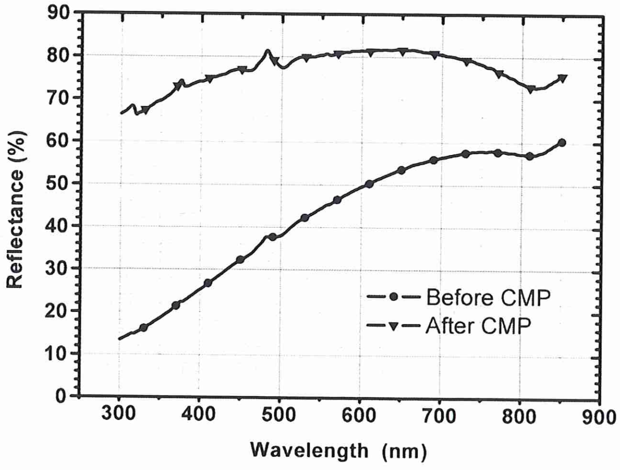

Fig. 4. Al coating surface reflectance before and after CMP process.

Lifetime of an aluminum coating oxidized by limited exposures to atomic oxygen can be substantially extended by periodic recoating of the oxidized aluminum surface with new aluminum coatings.[20] So the Al oxidation in the long-time deposition process should be considered. We proved in our last study that there was no redundant oxidized oxygen between the Al coating layers. Nevertheless, it was doubted that if an extremely thin aluminum oxide film could be formed or not formed between two Al layers during the long-time polishing process. To clarify this problem, we also did an energy dispersive X-ray (EDX) analysis on the interface between the two Al layers. The test results are shown in Figure 5(d). The

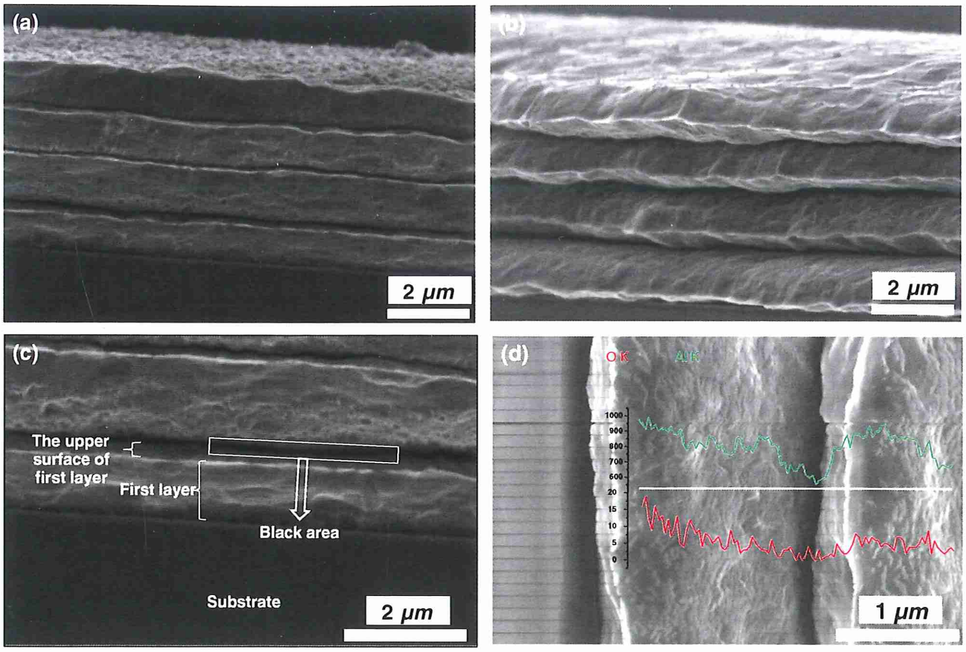

Fig. 5. Al coating cross-sectional SEM images (a) before and (b) after CMP process. (c) The enlarged image of Al coating cross-sectional SEM image. (d) Energy dispersive X-ray analysis on the interface between two Al layers.

scan mode of EDX is line scan with a scan length of 4 μm, shown by the white line. The green line and red line represent the Aluminum and Oxygen content, respectively. The signal intensity of Al was about 100 times higher than that of O. Although the intensity of Al dropped a little around the interface due to its loss in the shadow, it was still much higher than that of O. We therefore believed that there was no obvious oxidation effect of Al coating during the coating deposition and CMP process.

The film hardness of thick Al substrate is fatal to the final ruling consequence in the echelle grating ruling process. The film hardness of Al coating was appropriately around 0.5 Mpa and the used measurement instrument was nano indentation of Agilent. The measurement method was G-Series DCM CSM Standard Hardness, Modulus and Tip Cal. Figure 6 shows the Al coating hardness and modulus changing along with displacement into surface before and after the CMP process. The average hardness between 400 nm and 500 nm of displacement into surface before and after the CMP process were 0.483 GPa and 0.516 GPa, respectively, showing that the CMP process made the Al coating hardness a litter larger. But the film hardness was still in the allowed region.

Figure 7 shows the ruled echelle grating on the thick Al coating treated by CMP process. The torque equilibrium method was used in the ruling of this echelle grating. The scatter light results for the fabricated echelle gratings was 3.1×10^{-4} and used ruling machine was CIOMP-2 ruling engine. The scatter intensities and ghost of gratings ruled with CIOMP-2 were low and no Rowland ghosts were visible. CIOMP-2 can also produce varied-line-space, bend-line, and aberration-reducing gratings and echelle gratings. After analyzing the AFM image of echelle grating surface, the grating groove density was 79 gr/mm and the measured diffraction efficiency at 632.8 nm and 36th order was 50%, where the diffraction wave front was

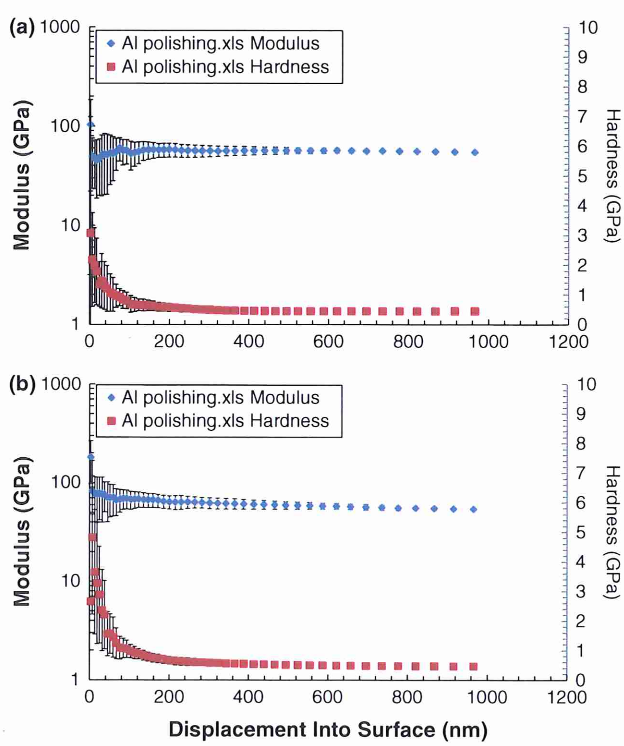

Fig. 6. Al coating hardness and modulus (a) before and (b) after CMP process.

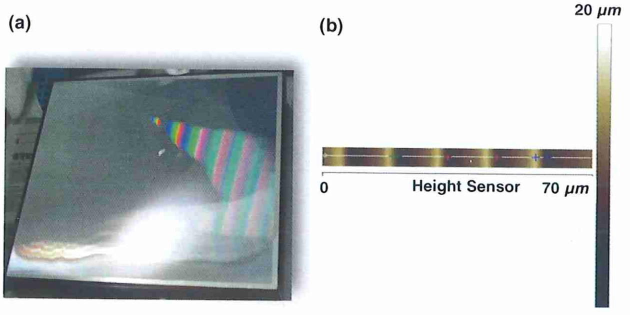

Fig. 7. (a) Fabricated echelle grating sample; (b) AFM image of echelle grating surface.

$\lambda/3$. Stray-light level was 1×10^{-4} during the measurement process. Finally, the grating groove depth was 5.5 μm and Blaze angle was 64.5°.

4. CONCLUSION

In summary, based on our previous investigation of Al coating surface roughness reduction by multi-step deposition method, we put forward mechanical polishing method to improve surface quality of echelle grating ruling substrate. The CMP method was chosen as the new approach which almost left no polishing marks on the soft thick Al coating, unlike the SPDT process. The thickness loss, surface reflectance, inner structure, oxidation problem and film hardness were investigated in detail respectively. The thicknesses loss of the CMP process was 171.4 nm by different measurement methods, and Al coating surface reflectance had nearly 40% increase. The inner four-layer microstructure and film hardness had no obvious change. Besides, there was hardly any oxidation effect introduced during the multi-step deposition and CMP process. A large-size echelle grating ruling Al coating substrate was finally obtained, and the Rq of Al coating was significantly reduced to 5.26 nm and the diffraction efficiency of ruled echelle grating was 50%.

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References and Notes

1. G. Harrison, *Phys. Today* 3, 6 **(1950)**.
2. J. Song, L. C. Chen, and B. J. Li, *Prog. Electromagn. Res.* 141, 369 **(2013)**.
3. S. Engman and P. Lindblom, *Appl. Opt.* 21, 4356 **(1982)**.
4. P. Lindblom, *Appl. Opt.* 42, 4549 **(2003)**.
5. K. G. Bach and B. W. Bach, *Proc. SPIE* 4014, 389091 **(2000)**.
6. I. S. Mclean, E. E. Becklin, O. Bendiksen, G. Brims, J. Canfield, D. F. Figer, J. R. Graham, J. Hare, F. Lacayanga, J. E. Larkin, S. B. Larson, N. G. Levenson, N. Magnone, H. I. Teplitz, and W. Wong, *Proc. SPIE* 3354, 566 **(1998)**.
7. T. W. Barnard, M. I. Crockett, J. C. Ivaldi, and P. L. Lundberg, *Anal. Chem.* 65, 1225 **(1993)**.
8. D. Nevejans, E. Neefs, E. V. Ransbeeck, S. Berkenbosch, R. Clairquin, L. D. Vos, W. Moelans, S. Glorieux, A. Baeke, O. Korablev, I. Vinogradov, Y. Kalinnikov, B. Bach, J. P. Dubois, and E. Villard, *Appl. Opt.* 45, 5191 **(2006)**.
9. G. R. Harrison, S. W. Thompson, H. Kazukonis, and J. R. Connell, *J. Opt. Soc. Am.* 62, 751 **(1972)**.
10. X. T. Li, H. L. Yu, X. D. Qi, S. L. Feng, J. C. Cui, S. W. Zhang, Jirigalantu, and Y. G. Tang, *Appl. Opt.* 54, 1819 **(2015)**.
11. Z. Z. Li, J. S. Gao, H. G. Yang, T. T. Wang, and X. Y. Wang, *Opt. Express* 23, 243509 **(2015)**.
12. J. Strong, *Astrophys. J.* 83, 401 **(1936)**.
13. Z. Z. Li, H. G. Yang, X. Y. Wang, and J. S. Gao, *Acta Phys. Sin.* 63, 157801 **(2014)**.
14. Z. Q. Yin and Z. Yi, *Appl. Opt.* 54, 7835 **(2015)**.
15. Y. Y. Lai, Y. P. Lan, and T. C. Lu, *Light: Sci. Appl.* 2, 76 **(2013)**.
16. D. X. Dai, J. Bauters, and J. E. Bowers, *Light: Sci. Appl.* 1, 1 **(2012)**.
17. K. Lee, J. Lee, B. A. Mazor, and S. R. Forrest, *Light: Sci. Appl.* 4, 288 **(2015)**.
18. H. G. Yang, Z. Z. Li, X. Y. Wang, Z. F. Shen, J. S. Gao, and S. W. Zhang, *Opt. Eng.* 54, 045106 **(2015)**.
19. H. A. Macleod, Thin-Film Optical Filters, Taylor & Francis, New York **(2010)**, Vols. 4–5.
20. J. I. Larruquert, J. A. Méndez, and J. A. Aznárez, *Opt. Commun.* 135, 60 **(1997)**.
21. X. T. Li, H. L. Yu, X. D. Qi, S. L. Feng, J. C. Cui, S. W. Zhang, Jirigalantu, and Y. G. Tang, *Appl. Opt.* 54, 7 **(2015)**.
22. Jirigalantu, X. T. Li, S. W. Zhang, X. T. Mi, J. X. Gao, Bayanheshig, X. D. Qi, and Y. G. Tang, *Appl. Opt.* 55, 28 **(2015)**.

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