Fabrication of high-efficiency and low-stray-light grating by inductively coupled plasma(ICP) etching-polishing method

X. Tan,[*] Q. B. Jiao, X. D. Qi, and H. Bayan

Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences, Changchun 130033, China

[*]*xintan_grating@163.com*

Abstract: Gratings with stray light of 4.99×10^{-7}-5.67×10^{-7} and efficiency of 93%-95% in a wavelength range of 1592 nm-1632 nm on Si-surface-modification SiC, fused silica and BK7 have been fabricated by the method of ICP etching-polishing. The CHF_3 and SF_6 plasma were used to etch a preliminary grating profile. Ar and O_2 plasma with low energy were then used to polish the grating to acquire low surface roughness and groove profiles closer to the ideal profiles. The morphologies of the gratings were characterized by AFM. The efficiencies and stray light were measured quantitatively by self-developed equipment. These results show that the ICP etching-polishing method is a promising candidate for production of good quality gratings into common optical materials.

OCIS codes: (050.1950) Diffraction gratings; (290.2648) Stray light.

References and links

1. R. E. Collin, "Electromagnetic scattering from perfectly conducting rough surfaces," IEEE Trans. Antenn. Propag. **40**(12), 1466–1477 (1992).
2. R. D. Kodis, "A note on the theory of scattering from an irregular surface," IEEE Trans. Antennas Propagat. AP **14**(1), 77–82 (1966).
3. D. P. Nichoils, "Shape deformation in rough surface scattering improved algorithms," Opt. Soc. Am. A **21**(4), 606–621 (2004).
4. L. X. Guo, Y. H. Wang, and H. S. Wu, "Study on the shadowing effect for optical wave scattering from randomly rough surface," Chin. Opt. Lett. **2**(7), 431 (2004).
5. W. R. Hunter, M. P. Kowalski, J. C. Rife, and R. G. Cruddace, "Investigation of the properties of an ion-etched plane laminar holographic grating," Appl. Opt. **40**(34), 6157–6165 (2001).
6. H. Lin, L. Zhang, L. Li, C. Jin, H. Zhou, and T. Huo, "High-efficiency multilayer-coated ion-beam-etched blazed grating in the extreme-ultraviolet wavelength region," Opt. Lett. **33**(5), 485–487 (2008).
7. B. Sheng, X. Xu, Y. Liu, Y. Hong, H. Zhou, T. Huo, and S. Fu, "Vacuum-ultraviolet blazed silicon grating anisotropically etched by native-oxide mask," Opt. Lett. **34**(8), 1147–1149 (2009).
8. E. Ishiguro, K. Yamashita, H. Ohashi, M. Sakurai, O. Aita, M. Watanabe, K. Sano, M. Koeda, and T. Nagano, "Fabrication and characterization of reactive ion beam etched SiC gratings," Rev. Sci. Instrum. **63**(1), 1439–1442 (1992).
9. S. P. Neck and M. Volz, "NASA's earth science missions overview," SPIE **7474OB**, 7474OB (2009).
10. R. Mager, W. Fricke, J. P. Burrows, J. Frerick, and H. Bovensmann, "Sciamachy: a new-generation of hyper spectral remote sensing instrument," Proc. SPIE **3106**, 84–94 (1997).
11. H. H. Aumann, M. T. Chanine, C. Cautier, and M. D. Goldberg, "AIRS/AMSU/HSB on the Aqua mission: design, science objectives, data products, and processing systems," IEEE T. Geosci., Remote **41**(2), 253–264 (2003).
12. B. Q. Wu, A. Kumar, C. Cautier, and S. Pamarthy, "High aspect ratio silicon etch: A review," J. Appl. Phys. **108**(5), 051101 (2010).
13. F. Gaboriau, M. C. Fernandez-Peignon, G. Cartry, and C. Cardinaud, "Etching mechanisms of Si and SiO_2 in inductively coupled fluorocarbon plasmas: Correlation between plasma species and surface etching," J. Vac. Sci. Technol. A **23**(2), 226–233 (2005).
14. International Intellectual Group, Inc., "Accurate electromagnetic theories," http://www.pcgrate.com/etestlab.
15. Shimadzu Corporation, "Low Stray Light Diffraction Gratings," http://www.shimadzu.com/opt/products/dif/o-k25cur0000007dvj.html.

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16. M. P. Kowalski, T. W. Barbee, Jr., and W. R. Hunter, "Replication of a holographic ion-etched spherical blazed grating for use at extreme-ultraviolet wavelengths: efficiency," Appl. Opt. **45**(2), 322–334 (2006).

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1. Introduction

Diffraction gratings with low surface roughness can produce lower stray light [1–4]. They have been widely used in high-resolution, high-precision spectrometers for trace element determined in atmospheric, medicine, agriculture, environment, et al. To achieve low stray light, there are several methods. William R. Hunter et al. [5] shown an ion-etched method to fabricate the plane laminar holographic grating with the roughness about 1.0 nm-1.8 nm (rms). Hui Lin et al. [6] developed an Ar and O_2 mixed-gas ion beam etching technique to reduce the roughness in a fused silica substrate. Using this method, the surface roughness of fabricated grating is about 0.56 nm ± 0.09 nm(rms). Bin Sheng et al. [7] described a wet etching method to fabricate the blazed grating using an off-cut silicon (111) wafers. The roughness of the grating is about 0.2 nm (rms). E. lshiguro et al. [8] fabricated a holographic grating on a SiC substrate with low surface roughness by means of reactive ion beam etching to reduce the grating surface roughness. Most of these gratings mentioned above have been deeply investigated. Optimizing etching parameters, choosing substrates with low roughness and utilizing the particular properties of the substrates are the common points to all of the methods to reduce the surface roughness of the fabricated gratings.

However, these methods are all aimed at the fabrication process or the substrate itself to control the roughness. If these gratings are fabricated by dry or wet etching completely, there will be no more method to reduce the surface roughness. In addition, most of these gratings are used in the EUV and UV wavelength range. But the effect of surface roughness of gratings on the stray light is still a great problem needed to be solved in the visible-infrared wavelength region.

In this paper, we have developed a grating ICP etching-polishing method to reduce the roughness of grating applied in the visible-infrared wavelength region. The method mainly includes two steps: firstly, the photoresist grating mask was etched using the mixed gas consisting CHF_3 and SF_6. Secondly, the etched grating without photoresist was polished using the mixed gas consisting Ar and O_2. It is known that the measured precision of Space-based atmospheric CO_2 spectrometer is approximate 1×10^{-6}-2×10^{-6} [9–11]. In order to achieve the precision, as the core device, the stray light of grating with 190mm × 160mm in area should be lower than 1×10^{-6} (the ratio of standardized light intensity in the middle position of the order 0 and order + 1 at wavelength 1610 nm with incident angle about 38.86°) and TM polarization diffraction efficiency should be above 85% in the range between 1592 nm and 1632 nm. In this paper, as an example of this grating, we describe the ICP etching-polishing process and present our measured results of stray light and efficiency results of several kinds of substrates, such as Si-surface-modification SiC, fused silica and BK7.

2. Experiment and measurement

The grating specimens were designed for the 1st order whose wavelength range is between1592 nm and 1632 nm with incident angle about at 38.86°. According to the technical indicators direction, the diffraction efficiencies of blazed grating is slightly lower than trapezoid grating in the range of 1592nm-1632nm. In addition, compared with the blazed grating fabricated by ion beam etching, the trapezoid grating fabricated by ICP etching [12,13] with higher etching rate ratio could reduce the requirement of mask depth. In view of technical parameters and difficulty coefficient, we found that the trapezoid grating fabricated by ICP etching is appropriate for the requirement to the grating used in Space-based atmospheric CO_2 spectrometer.

The diffraction efficiency of the trapezoid grating is sensitively dependent on the grating parameters of duty cycle, groove depth, sidewall angle, grating constant and wavelength range. With the PCGrate-SX 6.4 [14], there is only one variable in the optimization process of

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the diffraction efficiency at a time. So, we choose one grating parameter as a variable each time when the diffraction efficiency was calculated. An iterative method was adopted to calculate the relation of the optimum efficiencies and grating parameters. Because of the diffraction efficiency of the grating change slightly in wavelength range 1592 nm-1632 nm, wavelength 1612 nm is selected as the calculated wavelength. Concrete scheme as follows: step 1, the relation of the optimum efficiencies and the duty cycle, the groove depth was calculated when the sidewall angle is an invariant. Eleven relation schemas of the optimum efficiencies and the duty cycle, the groove depth, like Fig. 1, were drawn for the sidewall angle from 68° to 78°. Step 2, contrast the eleven relation schemas. Figure 1 is one of the eleven relation schemas, in which the diffraction efficiency is higher and the tolerance ranges are larger. The diffraction efficiency is highest at the black point in Fig. 1. Then, the relation of the optimum efficiencies and the grating sidewall angle is calculated as functions of wavelength when the duty cycle and the groove depth are at the black point in Fig. 1, shown in Fig. 2.

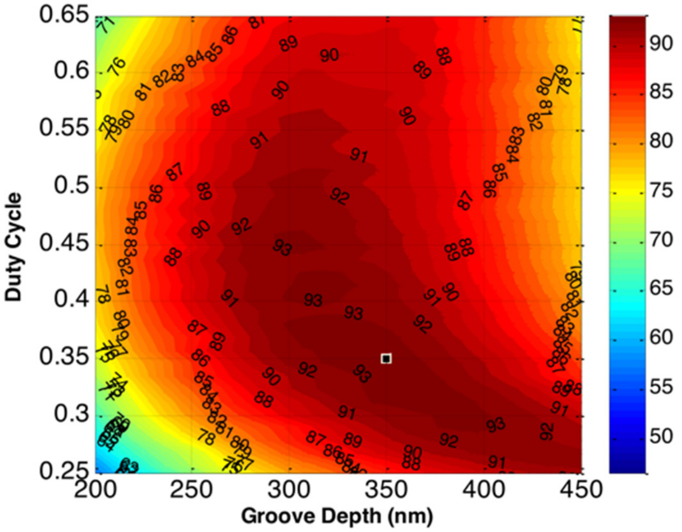

Fig. 1. The relation of the optimum efficiencies and the duty cycle and the groove depth with sidewall angle 73°.

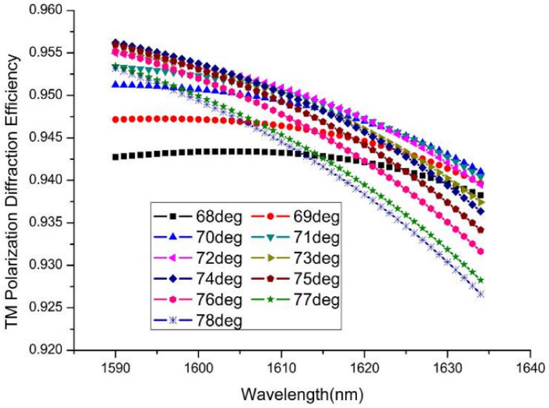

Fig. 2. The relation of the optimum efficiencies and the grating sidewall angle.

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The grating parameters in the rectangle will achieve 90% diffraction efficiency as shown in Fig. 1. Figure 2 shows the relation of the optimum efficiencies and the grating sidewall angle. According to Fig. 2, the efficiencies will be higher at sidewall angle 72°-74° in the range of 1592nm-1632nm. According to Fig. 1 and Fig. 2, the target grating groove parameters deduced from the trapezoid condition are shown in Table 1.

Table 1. Target Grating Groove Profile Parameters

Parameter Name	Groove Depth(nm)	Duty Cycle(%)	Sidewall Angle(°)	Roughness (nm, Rq)	Roughness (nm, Ra)
target grating groove profile parameters	350 ± 10	35 ± 2	73 ± 1	<1.5	<1.5

We first spin coated the photoresist on a finely polished surface with 190mm × 160mm in area and 21mm in thickness. The process parameters of the three kinds of substrates are just the same. Then we followed the conventional holographic exposure and development steps to form a 967 gr/mm grating mask. We got an etched mask whose ridges are approximately rectangular. After that, we did etching with CHF_3 and SF_6 mixed gas by inductively coupled plasma(ICP) etching. For different substrates, we choose different optimized etching parameters as shown in Table 2. According to the characteristics of ICP etching, it is difficult to obtain a grating sidewall angle whose value is equal to 73°. For that reason, the grating sidewall angle was about 76°-78°after ICP etching process. The changing of diffraction efficiency of the grating between sidewall angle with 76°-78° and with 73° is lower than 1%-1.5%. We found that ICP polishing using Ar^+ and O^+ mixed plasma with low energy not only can be used to smooth the surface but also can be used to reduce the grating sidewall angle. A lot of ICP polishing experiments proved that the grating sidewall angle will be reduced 2°-9° after polishing, which is closer to the ideal grating sidewall angle, and the surface roughness will be reduced also after ICP polishing. As the target surface roughness <1.5nm, the grating is polished by Ar^+ and O^+ mixed plasma using the same ICP equipment. The optimized polishing parameters are shown in Table 3.

Table 2. ICP Etching Parameters

Substrate	ICP Source Power (W)	RIE Source Power (W)	SF_6 (sccm)	CHF_3 (sccm)	Stress (Pa)
modified SiC	600	330	50	120	3
fused silica	300	800	40	200	3
BK7	300	750	60	210	3

Table 3. ICP Polishing Parameters

Substrate	ICP Source Power (W)	RIE Source Power (W)	Ar (sccm)	O_2 (sccm)	Stress (Pa)
modified SiC	400	350	60	30	3
fused silica	400	450	40	40	3
BK7	400	450	40	30	3

Then, the Au coating was deposited on the gratings by magnetron sputtering at the National engineering research center for diffraction gratings manufacturing and application of China.

The morphologies of the gratings were characterized by AFM. The performance of the gratings cannot be directly calculated with the groove profiles. So, the efficiency and stray light were measured quantitatively by self-developed equipment.

Efficiency measurements were made at the National engineering research center for diffraction gratings manufacturing and application of China. It is made up including TLB-

6730 tunable lasers with a mode-hop free tuning range of 1550 nm-1630 nm (Newport Corporation, USA), 818-IR/DB photodetector (Newport Corporation, USA), a two-dimensional scan stage which can hold the specimen and scan in X-Y plane. We placed a precision linear polarizer (05LP-NIR, Newport Corporation, USA) at the emission aperture of the laser to ensure that the radiation for TM polarization. We first measured the intensity by the photodetector of the beam through the linear polarizer. Then we set the grating specimen on the scan stage at incident angle 38.86°, and the photodetector in the direction of + 1 diffraction beam. The intensity of + 1 diffraction beam can be measured. Twelve locations in each grating specimen were measured at one wavelength. The diffraction efficiency of one location is the ratio of the 1st-order diffraction beam intensity and the incident beam intensity. The diffraction efficiency at one wavelength is the arithmetic average value of twelve different even-distributed locations in the specimen. The measurements were executed at discrete wavelengths with an interval of 2 nm.

Stray light measurements were made at the National engineering research center for diffraction gratings manufacturing and application of China. The stray light is the ratio of the stray light intensity to the first order light intensity of 1592 nm-1632 nm laser. And the stray light intensity is detected of mid-point between zero- and first-order light at the incident angle about 38.86°. This is similar to the measurement method of Shimadzu Corporation [15]. The measurements is made up including TLB-6730 tunable lasers, 918D-IR-OD3R with which linearity of photodiode response is 10^{-12}W-10^{-3}W optical power(Newport Corporation, USA), a rotary stage which can hold the grating specimen, a beam expander system and a focusing system with aperture larger than grating aperture. The grating specimen is placed on the rotary stage at incident angle 38.86°. First, the 1st order light intensity is measured without beam expander and focusing system. This method can eliminate the influence of grating stray light. Second, the stray light intensity is detected of mid-point between 0 and 1st-order light with beam expander and focusing system. At this point, all of the grating aperture is irradiated and full aperture stray light intensity of the grating can be detected. Third, the stray light can be calculated.

3. Results and discussion

The groove profiles of the three kinds of samples before and after polishing are measured by AFM. Figure 3 shows the modified SiC grating groove profile before polishing. Figure 4 shows the modified SiC grating groove profile after polishing. Figure 5 shows the fused silica grating groove profile before polishing. Figure 6 shows the fused silica grating groove profile after polishing. Figure 7 shows the BK7 grating groove profile before polishing. Figure 8 shows the BK7 grating groove profile after polishing

Here we choose Ar^+ with low ion energy to play down the removing rate of the substrate in order to smooth the surface and reduce the impact of the groove profile. Since Ar + ions can bombard the tip by a higher removing rate, the surface roughness of gratings can be significantly reduced. There is an advantage to use O^+ ions that their ashing effect can remove some irregularities of the photoresist mask, which could make the profile smoother.

We controlled the removing rate by total pressure, RF power and mixing ratio of the gas. We also noticed that the surface roughness and the groove profile were approximately proportional to the removing rate and removing time. The measured profile parameters of ICP etching-polishing grating, listed in Table 4, indicate that the polishing groove profiles meet our design goal well.

To get a lower roughness of the grooves, the ICP etching parameters and the ICP polishing parameters were optimized for the different substrates. From the experimental results, it is found that the groove surface quality of the modified SiC grating is smoother than the other two whether or not the three kinds of substrates are polished. And, the groove surface quality of the BK7 grating is roughest.

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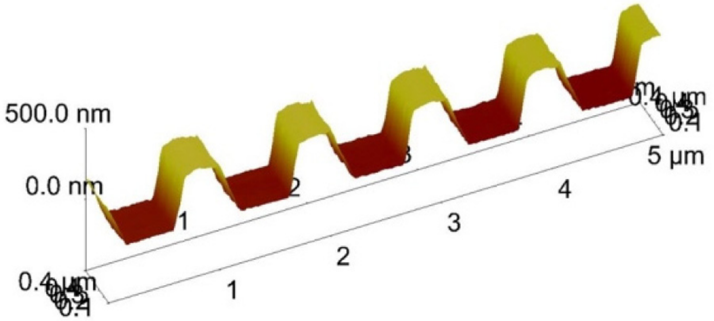

Fig. 3. AFM images of the modified SiC grating groove profiles before polishing.

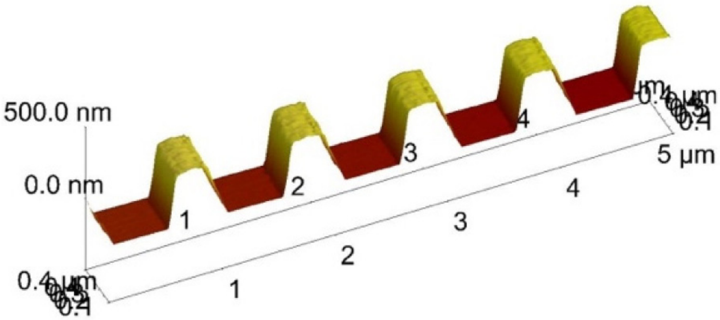

Fig. 4. AFM images of the modified SiC grating groove profiles after polishing.

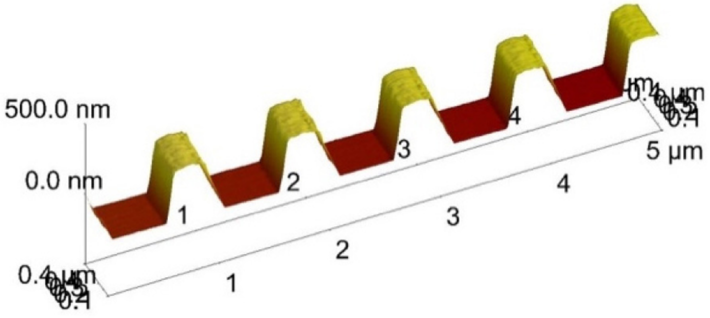

Fig. 5. AFM images of the fused silica grating groove profiles before polishing.

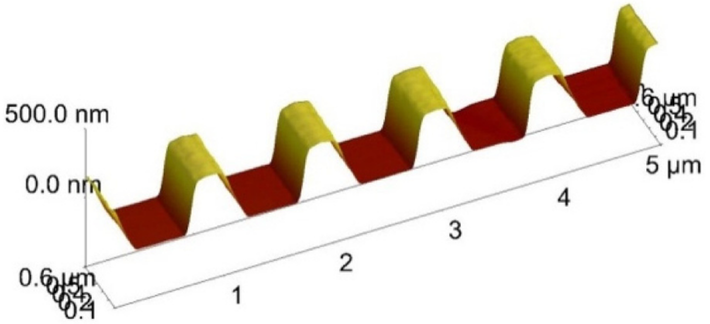

Fig. 6. AFM images of the fused silica grating groove profiles after polishing.

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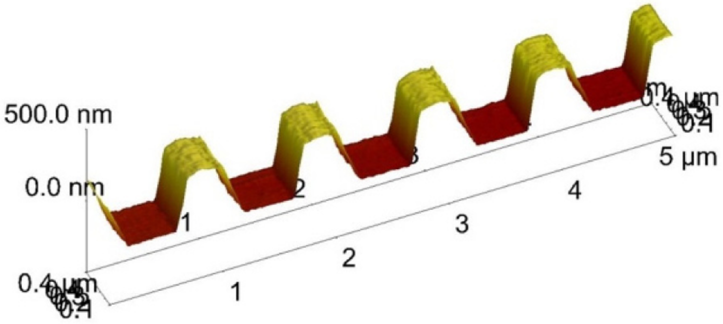


Fig. 7. AFM images of the BK7 grating groove profiles before polishing.

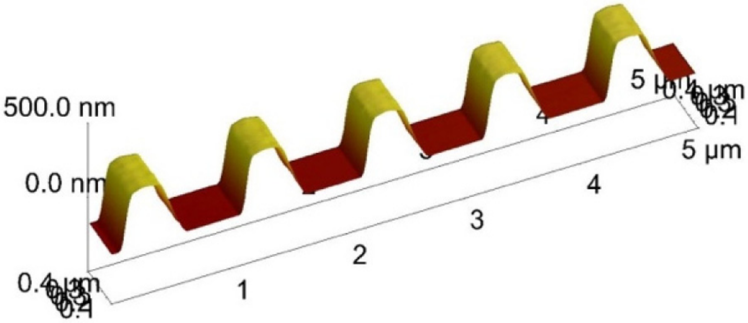
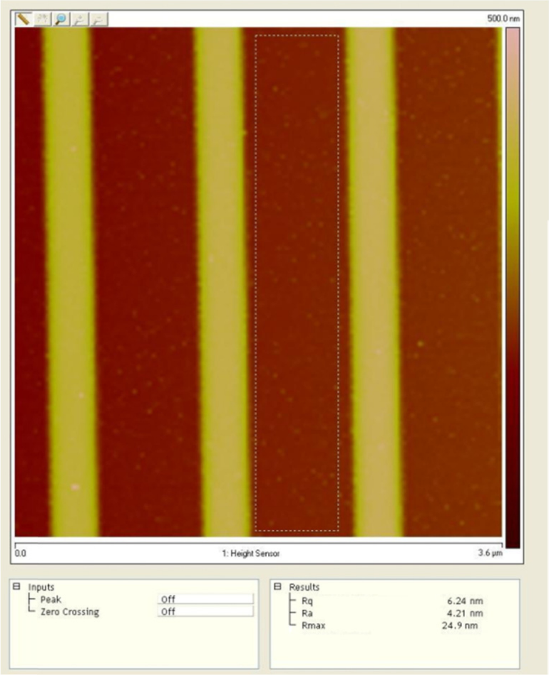


Fig. 8. AFM images of the BK7 grating groove profiles after polishing.

The groove roughness of the three kinds of samples before and after polishing are also measured by AFM. The groove roughness measured results of the modified SiC grating before polishing are shown in Fig. 9. The groove roughness measured results of the modified SiC grating after polishing are shown in Fig. 10. The groove roughness measured results of the fused silica grating before polishing are shown in Fig. 11. The groove roughness measured results of the fused silica grating after polishing are shown in Fig. 12. The groove roughness measured results of the BK7 grating before polishing are shown in Fig. 13. The groove roughness measured results of the BK7 grating after polishing are shown in Fig. 14.

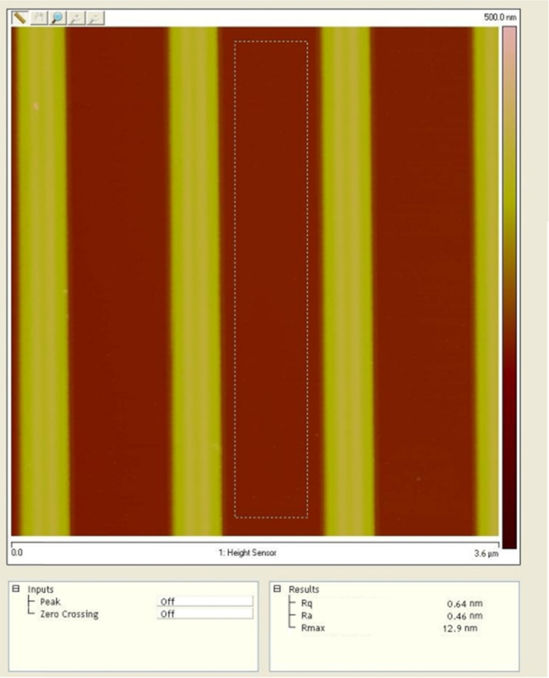
According to Fig. 9 – Fig. 14, the Rq and Ra of the three kinds of samples after polishing are reduced to below 1 nm. The Rq and Ra of the BK7 grating after polishing are larger. And, the Rq and Ra of the modified SiC grating after polishing are smallest. The differences of the groove roughness of the samples before and after polishing are due to the properties of the substrates. It is difficult to improve the groove roughness of the BK7 grating to the groove roughness of the modified SiC grating with by ICP polishing process. This is because of differences in material properties.



Inputs
Peak Off
Zero Crossing Off

Results
Rq 6.24 nm
Ra 4.21 nm
Rmax 24.9 nm

Fig. 9. AFM image of the modified SiC grating groove roughness before polishing.



Inputs
Peak Off
Zero Crossing Off

Results
Rq 0.64 nm
Ra 0.46 nm
Rmax 12.9 nm

Fig. 10. AFM image of the modified SiC grating groove roughness after polishing.

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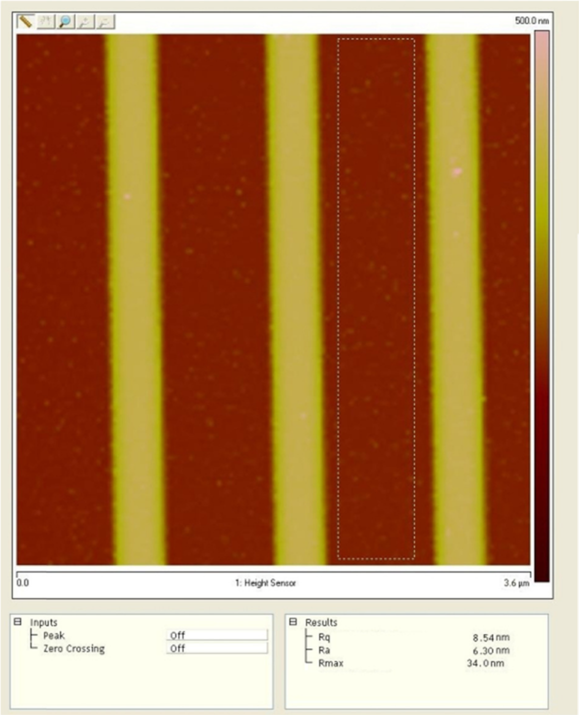
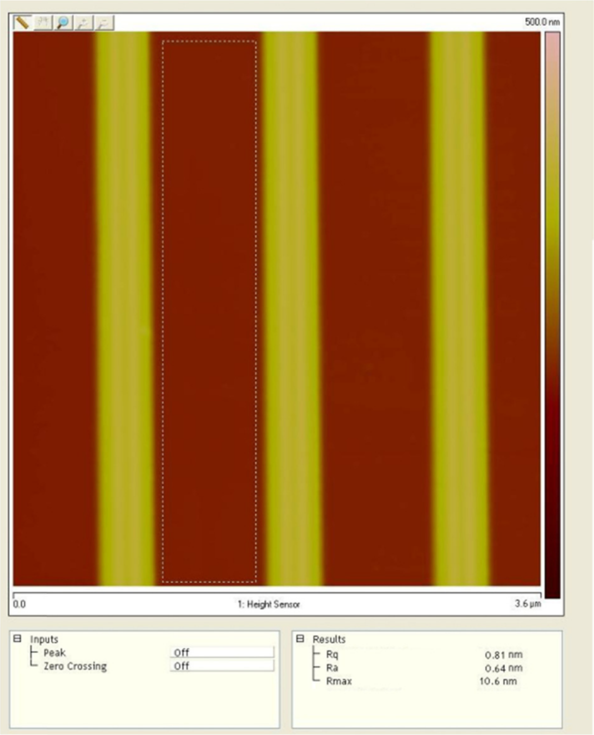


Inputs
Peak: Off
Zero Crossing: Off

Results
Rq 8.54 nm
Ra 6.30 nm
Rmax 34.0 nm

Fig. 11. AFM image of the fused silica grating groove roughness before polishing.



Inputs
Peak: Off
Zero Crossing: Off

Results
Rq 0.81 nm
Ra 0.64 nm
Rmax 10.6 nm

Fig. 12. AFM image of the fused silica grating groove roughness after polishing.

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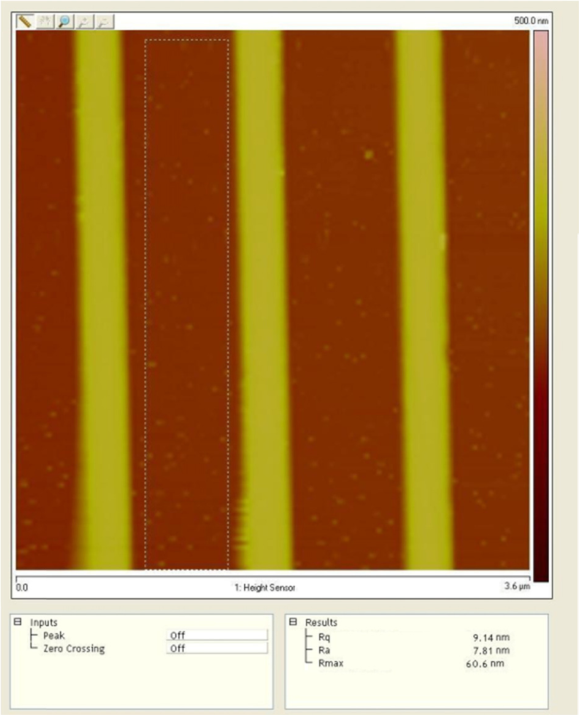
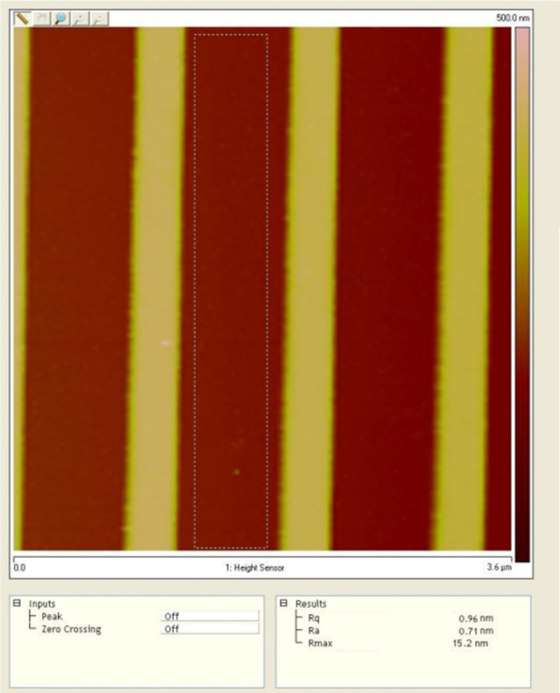


Inputs
- Peak: Off
- Zero Crossing: Off

Results
- Rq: 9.14 nm
- Ra: 7.81 nm
- Rmax: 60.6 nm

Fig. 13. AFM image of the BK7 grating groove roughness before polishing.



Inputs
- Peak: Off
- Zero Crossing: Off

Results
- Rq: 0.96 nm
- Ra: 0.71 nm
- Rmax: 15.2 nm

Fig. 14. AFM image of the BK7 grating groove roughness after polishing.

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AFM was used to measure the grating profile before and after polishing. Twelve locations in or around the specimen center were measured. We used the algorithm introduced in literature [16] to deduce grating parameters from the AFM data. The groove depth, duty cycle and sidewall angles were computed from a statistics algorithm. The parameters averaged over all locations are shown in Table4.

Table 4. Designed and Measured Grating Parameters of modified SiC, fused silica and BK7 substrates

substrates	Parameter Name	Groove Depth(nm)	Duty Cycle(%)	Sidewall Angle(°)	Roughness (nm, Rq)	Roughness (nm, Ra)
	Design	350	35	73	<1.5	<1.5
modified SiC	before polishing	347.8 ± 0.4	36.1 ± 1.4	78.28 ± 0.52	6.21 ± 0.06	4.21 ± 0.07
	after polishing	343.5 ± 0.3	35.4 ± 1.2	72.94 ± 0.46	0.69 ± 0.06	0.45 ± 0.04
	Au coating without polishing	347.8 ± 0.4	36.5 ± 1.3	77.71 ± 0.56	7.13 ± 0.04	4.13 ± 0.06
	Au coating with polishing	344.2 ± 0.4	36.2 ± 1.2	72.46 ± 0.61	0.67 ± 0.07	0.48 ± 0.06
fused silica	before polishing	354.2 ± 0.4	36.7 ± 1.4	77.08 ± 0.59	8.45 ± 0.10	6.38 ± 0.09
	after polishing	352.6 ± 0.3	36.1 ± 1.2	73.87 ± 0.42	0.77 ± 0.06	0.57 ± 0.04
	Au coating without polishing	353.8 ± 0.3	36.9 ± 1.3	77.21 ± 0.60	8.44 ± 0.04	7.32 ± 0.07
	Au coating with polishing	352.5 ± 0.4	36.2 ± 1.3	73.57 ± 0.60	0.81 ± 0.07	0.64 ± 0.06
BK7	before polishing	357.2 ± 0.4	37.8 ± 1.4	78.18 ± 0.48	9.14 ± 0.06	7.81 ± 0.09
	after polishing	354.8 ± 0.3	36.3 ± 1.0	73.87 ± 0.51	0.79 ± 0.05	0.67 ± 0.04
	Au coating without polishing	355.8 ± 0.6	37.9 ± 1.4	78.06 ± 0.71	10.13 ± 0.06	8.67 ± 0.08
	Au coating with polishing	351.4 ± 0.7	36.2 ± 1.3	73.56 ± 0.59	0.96 ± 0.07	0.71 ± 0.04

Table 4 illustrates the changing rule of the roughness of the grating before and after polishing, before and after Au coating. The roughness before polishing is about 6-10 nm (Ra, Rq). Because of the leftover of the ICP etching and the photoresist grating mask's roughness being about 15 nm generally. The ICP etching parameters is optimized to reduce the roughness, but the lowest roughness is about 7-10 nm before polishing. The roughness after Ar^+ and O^+ mixed plasma polishing is about 0.4-0.6 nm (Ra, Rq). Because of the deposited uniformity and the granule, Au coating can increase the roughness to about 0.67-1.0 nm (Ra, Rq), but the polished roughness after Au coating can still meet the requirements.

According to Table 4, we can get two conclusions. One, the ICP polishing process can reduce the groove roughness. Two, the physico-chemical properties of substrates limit the improvement degree of the groove roughness by ICP polishing.

Figures 15, Fig. 16, and Fig. 17 illustrate the changing rule of the measured and calculated + 1 order diffraction efficiencies versus the wavelength of the three kinds of substrates. The calculated + 1 order diffraction efficiency is about 94%-96% from 1592 nm to 1632 nm. The measured + 1 order diffraction efficiency of the grating before polishing is about 92%-94%, and the measured + 1 order diffraction efficiency the grating after polishing is about 93%-95%. Because of the lasers' tuning range is 1550 nm-1630 nm, the TM polarization diffraction efficiency at 1632 can't be measured.

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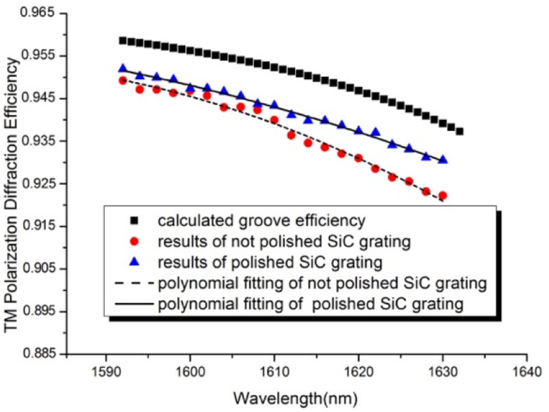

Fig. 15. Measured and calculated grating TM polarization efficiencies of the modified SiC grating. The solid curves and the short dash dot curves are fitted by polynomial fitness method.

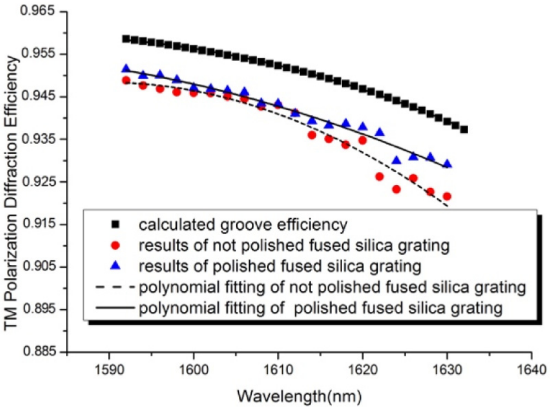

Fig. 16. Measured and calculated grating TM polarization efficiencies of the fused silica grating. The solid curves and the short dash dot curves are fitted by polynomial fitness method.

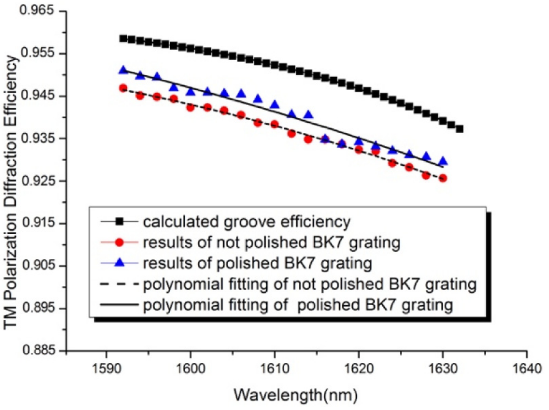

Fig. 17. Measured and calculated grating TM polarization efficiencies of the BK7 grating. The solid curves and the short dash dot curves are fitted by polynomial fitness method.

The measured diffraction efficiencies of the gratings are lower about 1%-1.5% than the calculated diffraction efficiencies of the target grating parameters on average. The groove parameters of the gratings are different from the parameters used in the calculation. Also, the edges and corners of the groove profiles are not sharp as the ideal groove profiles. The diffraction efficiency variety direction of the polished grating and the grating without polishing are the same to the calculated diffraction efficiency.

The diffraction efficiencies of the three kinds of substrates are increased after polishing. The modified SiC grating diffraction efficiencies increased by 1%-2% after polishing, the fused silica grating diffraction efficiencies increased by 1%-2%, and the BK7 grating diffraction efficiencies increased by 1%-1.5%. The reason is the reducing of the sidewall angle from 76°-78° to 73° after polishing which have already been shown in Table 4. The variety difference of groove profiles in Table 4 can be attributed to the polishing. But the change of groove depth and duty cycle in Table 4 is not enough to cause the change of efficiency as shown in Fig. 1. These results indicate that ICP polishing process can reduce the roughness as listed in Table 4, and increase the diffraction efficiencies at the same time in all the three kinds of substrates.

According to Figs. 15-17, the diffraction efficiency of the BK7 grating is higher. This is not due to the differences in the physico-chemical properties of the substrates. The diffraction efficiency is sensitively dependent on the grating parameters of duty cycle, groove depth and sidewall angle. It is insensitivity to the roughness. The diffraction efficiency of the BK7 grating is higher owe to the groove profile of the BK7 grating is closer to the target grating groove profile.

Figure 18, Fig. 19, and Fig. 20 illustrate the measured results of stray light of the gratings versus wavelength. These indicate the ratio of the stray light intensity to the incident intensity of TLB-6730 tunable laser. The stray light is detected of mid-point between zero- and first-order light at the incident angle which is equal to 38.86°. The arithmetic average analysis results of stray light are shown in Table 5.

According to Figs. 18-20 and Table 5, the arithmetic mean value of stray light of the three kinds of gratings after and before polishing dropped in nearly two orders of magnitude. And the standard deviation dropped in nearly one order of magnitude. This shows that the stray light uniformity become better. For polished gratings, the roughness and the stray light reduces, and the stray light uniformity is better. There are slight differences in the roughness and the stray light of the three kinds of gratings. There are two reasons, one is the sensitive to Ar^+ plasma, the other is the physico-chemical properties of the substrates. The modified SiC's Si layer is characterized by high purity, single-component, and the milling tip effect is more obvious. So the roughness and the stray light of the modified SiC is lower than the other two substrates. The fused silica has higher purity and less component than BK7, so the roughness and the stray light is also lower. These results suggest that the smooth effect of the ICP polishing process is slightly different for different materials. However, through the optimization of ICP polishing parameters, the results are satisfactory.

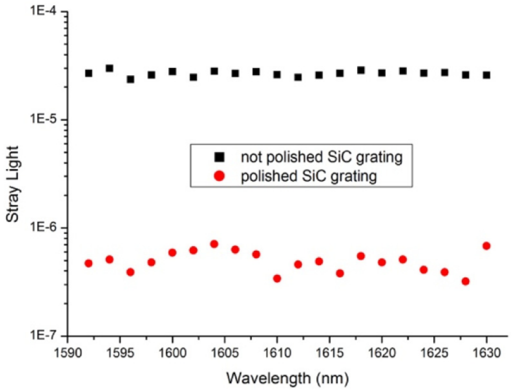

Fig. 18. Measured stray light of the modified SiC grating versus wavelength.

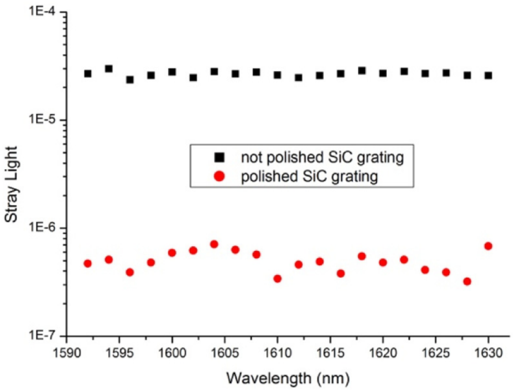

Fig. 19. Measured stray light of the fused silica grating versus wavelength.

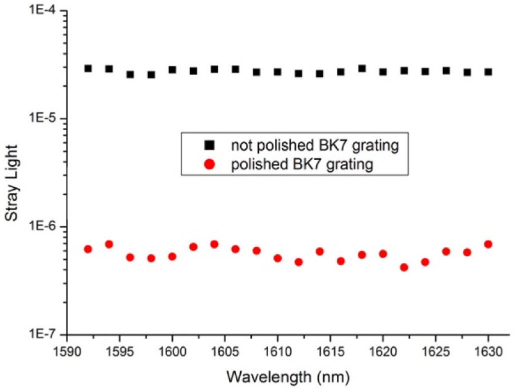

Fig. 20. Measured stray light of the BK7 grating versus wavelength.

Table 5. Arithmetic average analysis of the stray light

Substrate	Mean	Standard Deviation	Minimum	Median	Maximum
not polished modified SiC	2.671×10^{-5}	1.48922×10^{-6}	2.36×10^{-5}	2.68×10^{-5}	2.98×10^{-5}
polished modified SiC	4.99×10^{-7}	1.10401×10^{-7}	3.2×10^{-7}	4.85×10^{-7}	7.1×10^{-7}
not polished fused silica	2.7265×10^{-5}	1.35929×10^{-6}	2.46×10^{-5}	2.74×10^{-5}	2.91×10^{-5}
polished fused silica	5.2195×10^{-7}	1.02543×10^{-7}	3.4×10^{-7}	5.3×10^{-7}	7.3×10^{-7}
not polished BK7	2.744×10^{-5}	1.12876×10^{-6}	2.55×10^{-5}	2.72×10^{-5}	2.91×10^{-5}
polished BK7	5.67×10^{-7}	7.90137×10^{-8}	4.2×10^{-7}	5.7×10^{-7}	6.9×10^{-7}

We have fabricated 190mm × 160mm trapezoid gratings by using a method of ICP etching with a CHF_3 and SF_6 mixed gas and polishing with a low ion energy Ar^+ and O^+ mixed plasma by ICP in Si-surface- modification SiC, fused silica and BK7 substrates through a photoresist grating mask. The ICP etching-polishing process is very effective for improving the diffraction efficiency and reducing the stray light. Owing to the well-controlled groove profile, the peak TM polarization diffraction efficiency of the 1st order has reached 95% at a wavelength of 1592 nm and the lowest TM polarization diffraction efficiency is higher than

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93% in wavelength range of 1592 nm-1632 nm. The roughness of the groove profile is about 0.5nm, and the stray light is about 5×10^{-7}.

4. Conclusion

We introduced a new, so called ICP etching-polishing method to produce good quality gratings with high efficiency and low stray light on Si-surface- modification SiC, fused silica and BK7, and the diffraction efficiency and stray light is 89.5%-93.5% and 4.99×10^{-7}-5.67×10^{-7} respectively. Compared with the roughness before polishing, lower roughness after polishing can scatter lower incident radiation to suppress the stray light to a lower level. It was also demonstrated that the beneficial reduction of sidewall angles after polishing closer to the ideal groove profiles and making the diffraction efficiency increased. Gratings fabricated by ICP etching-polishing method could be used in the atmospheric CO_2 spectrometer. These results show that the ICP etching-polishing method is a promising candidate for production of good quality gratings into common optical materials.

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