High power laser antireflection subwavelength grating on fused silica by colloidal lithography

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Abstract
In this study we report on an efficient and simple method to fabricate an antireflection subwavelength grating on a fused silica substrate using two-step reactive ion etching with monolayer polystyrene colloidal crystals as masks. We show that the period and spacing of the obtained subwavelength grating were determined by the initial diameter of polystyrene microspheres and the oxygen ion etching duration. The height of pillar arrays can be adjusted by tuning the second-step fluorine ion etching duration. These parameters are proved to be useful in tailoring the antireflection properties of subwavelength grating using a finite-difference time-domain (FDTD) method and effective medium theory. The subwavelength grating exhibits excellent antireflection properties. The near-field distribution of the SWG which is directly patterned into the substrate material is performed by a 3D-FDTD method. It is found that the near-field distribution is strongly dependent on the periodicity of surface structure, which has the potential to promote the ability of anti-laser-induced damage. For 10 ns pulse duration and 1064 nm wavelength, we experimentally determined their laser induced damage threshold to 32 J cm\textsuperscript{−2}, which is nearly as high as bulk fused silica with 31.5 J cm\textsuperscript{−2}.

Keywords: subwavelength gratings, antireflection, finite-difference time-domain, near-field distribution, laser induce damage threshold

(Some figures may appear in colour only in the online journal)

1. Introduction
Over the last decades, steady efforts have been made to obtain broadband antireflection (AR) [1–3]. Various antireflective technologies have been investigated [2], such as thin film antireflective coatings [4–6] and subwavelength grating (SWG) [7, 8]. Unfortunately, even though AR coatings using single layer or multilayer thin-film stacks are widely used, there still exist some problems associated with band limitation material selection and thermal mismatch at the interface between the film and the substrate [5, 8]. The moth’s eye use arrays of non-close-packed surface relief of sub-micrometer size as antireflective structure to suppress reflection [9]. The surfaces relief with a subwavelength size scale shorter than...
the wavelengths of visible light can reduce the surface reflection. The periodic surface relief structures are also called an antireflective SWG layer [10, 11]. The distinct advantages of the SWG layer can be divided into these categories compared to coatings. First, the SWG layer exhibits higher laser induced damage thresholds (LIDTs) [12] and better durability when applied in a high power laser range because no foreign materials are introduced. Second, the material of the SWG layer is the same as that of the substrate, while coating materials with appropriate refractive index for reducing the reflection are rare in nature [1, 5, 6]. Last, the SWG layer has many tunable factors, such as the period, spacing, height and geometry profiles [13, 14]. Similar to the surface relief on the moth’s eyes, the SWG layer with pillar-like shape has a graded effective reflective index profile from air to substrate. Therefore, they can dramatically suppress the reflection losses at the interface of different medium [15]. Indeed, SWG layers have been used to improve diffractive gratings, diffractive lenses, and antireflective technology on solar cells [3, 16–18]. The SWG layer is commonly produced by top–down technologies, such as interference lithography [19] and electron beam lithography [10]. However, these methods may be expensive and time-consuming. Masuda and coworkers reported an effective creation of high performance AR SWG layer by nanoimprinting lithography [20]. But self-assembly monolayer colloidal crystal technology may provide a much simpler and cheaper alternative in creating SWG layers [21–23]. However, most of these works lay emphasizes on silicon wafer. In the previous reports, the SWG layer on silica was produced by one-step fluorine chemistry reactive ion etching (RIE) using colloidal monolayer colloidal crystals of polystyrene as a mask [24]. A few of them involve methods to control the other parameters such as the spacing and height of the nanopillars on fused silica. Whereas these parameters are very important for high performance optical properties. Additionally, even though previous works have focused on the SWG, the near-field distribution which is adjusted by the 3D periodicity structure has not been reported actively. To the best of our knowledge, a few of the researchers focused on the LIDT of SWG on the fused silica substrate. In particular, it is very important for high power laser application.

In this paper, at first, we demonstrate fused silica SWG with high performance antireflective behavior from visible (VIS) to near-infrared (NIR), which is created by two-step RIE, using monolayer colloidal crystals of polystyrene as a mask. The two-step etching processes minifying the close-packed polystyrene spheres and then etching the underlying fused silica substrate are reported in this study which can control the spacing and height of SWG on fused silica structures. The height and spacing of SWG were optimized to achieve high transmittance over a wide wavelength range using a finite-difference time-domain (FDTD) method as well as by effective medium theory (EMT). And then, the near-field distribution adjusted by 3D SWG structure was modeled by the 3D-FDTD method using the commercial software package FDTD solutions (Lumerical). Due to the change of near-field distribution and fabrication with the same material, SWG has a high damage threshold, as we will show in this paper. We investigate the optical reflectance and transmission spectroscopy of such a fused silica SWG.

2. Materials and method

2.1. Manufacturing process

Monodisperse polystyrene (PS) microspheres were synthesized by emulsifier-free emulsion polymerization technique [25]. Experimental procedures for synthesizing the colloidal crystals assembly are as follows: first, a suspension of the polystyrene microsphere was diluted to a definite concentration by deionized water. Before the self-assembly process, polystyrene microspheres were modified with sodium dodecyl sulfate (SDS) so as to terminate them with negative charged groups and prevent aggregation. Then, a clean hydrophilic fused silica substrate was immersed vertically into the dispersion and lifted up with a constant speed, which was precisely controlled by a motor. The temperature for the experiment was set at 20 ± 2 °C. The oxygen RIE process was performed using oxygen gas at RF power 50W, 40 SCCM, 10 Pa, for different time duration. The fluorine-RIE process was performed using a mixture of Ar (150 SCCM) and CHF3 (15 SCCM) as the etching gas, with chamber pressure of 1.2 Pa and RF power of 100W. Finally, these templating polystyrene microspheres at the tips of silica nanopillars can be selectively removed by wet etching with brief tetrahydrofuran to generate clean silica nanopillar arrays.

2.2. Characterization

The diameter of the spheres was estimated using a Mastersizer 2000 Laser particle sizer. The morphology of the colloidal crystals template and SWG were examined using FEI Nova NanoSEM 450 scanning electron microscopy (SEM). Transmission and reflection measurements were performed using a Lambda 950 spectrometer (the specular reflection was measured at an incidence angle of 8°). The LIDT was tested by the Q-tuned Nd:YAG laser, which was used to provide a near-Gauss-type pulse beam (spatially and temporally) at a 1064 nm wavelength. A fixed energy attenuator was installed in the beam path to provide energy control. The maximum output energy was up to 1 J. The diameter of the laser spot on the testing sample was ~1 mm. (the width at 1/e2 of the pulse intensity), and the pulse width was ~10 ns. The R-on-1 testing procedure was carried out at 20 locations that were arranged in to a 4 × 5 array. According to ISO standard 11254, the distance between any two closer irradiated spots was 3 mm, which was long enough to avoid the over lapping of damage regions. By increasing the energy irradiated on the sample at an increment of 0.3 mJ and a time interval of 3 s, the samples were shot until breakdown damage (plasma flash) occurred, and this energy was recorded at once. The final LIDT of SWS and the substrate were the root mean square of 20 breakdown damage energies.

3. Results and discussion

A schematic diagram of the fabrication process for the SWG surfaces by the monolayer polystyrene colloidal crystal template and RIE on the fused silica substrate is shown in figure 1.
First, the dip-coating technique is used to generate colloidal monolayers of hexagonally ordered polystyrene microspheres on the fused silica substrate. The polystyrene microsphere monolayers’ colloidal crystals were prepared by dip coating, which was developed from Nagayama’s method [26]. Second, the diameter and spacing of the polystyrene microspheres were tuned by selective oxygen chemistry RIE of polystyrene. Third, fused silica nanopillars were produced by fluoroine chemistry selective and anisotropic RIE of the fused silica using a mixture of Ar and CHF₃. Polystyrene microspheres protect fused silica immediately underneath them, resulting in the formation of pillar-like arrays directly on the fused silica.
surface. The patterns of the polystyrene spheres were transferred to the fused silica substrates, the period and spacing of the silica nanopillars were determined by the initial polystyrene particle size and oxygen plasma duration. After the polystyrene microsphere on the top of the pillar was removed, SWG was obtained.

In the process of monolayer colloidal crystals’ template formation, the wettable fused silica substrate was dipped into the liquid dispersion of polystyrene spheres, a meniscus water–substrate–air interface system formed, which was caused by wetting effect. The polystyrene spheres are organized into a hexagonal array in a thin film of liquid supported on a flat substrate due to the attractive capillary forces among that spheres [26]. In the experiment, a liquid dispersion of polystyrene spheres is spread onto the hydrophile surface of fused silica substrate. When the liquid evaporates slowly under a controlled constant rate, these polystyrene spheres are fabricated into a hexagonal and closely packed array.

The detailed organization of the spheres was investigated by scanning electron microscopy (SEM) (figure 2). The array structure of polystyrene microspheres is hexagonal close-packed monolayer on the surface of the fused silica, as shown
in figure 2(A). Figure 2(B) shows a tilted-view SEM image of the self-assembled monolayer. The sample is 2 cm × 2 cm. Inset of figure 2(A) shows a digital camera picture of a 2 cm × 2 cm fused silica spread with a monolayer of 260 nm diameter polystyrene microspheres. The high quality of the colloidal crystals can be judged by uniformly brilliant yellow that extends to the entire substrate.

Remarkably, the polystyrene microspheres arrays etched by oxygen reactive ions are hexagonally non-close-packed colloidal crystals (figures 3(A)–(H)). Each microsphere was reduced to a nonspherical shape resembling a biconvex micro-lens due to the preferential etching in the direction normal to the polystyrene microspheres. Moreover, the periodic nature of the previously polystyrene microsphere colloidal crystals was preserved during the etching process, because oxygen RIE transformed the polystyrene microspheres into nonspherical particles without affecting the original periodic and ordered arrangement of these microspheres. Figures 3(A)–(H) show SEM images of the periodic non-close-packed array of the nonspherical polystyrene particles created after different etching duration. After O₂–RIE etching of 30 s, we can see that the microspheres exhibit nonspherical profile resembling a biconvex microlens with 240 nm in diameter from figures 3(A) and (B). Figures 3(C) and (D) shows the SEM images of polystyrene microspheres after etching of 60 s. The polystyrene microspheres’ diameter reduced to 220 nm. When the etching duration is 90 s, the diameter reduced from 260 to 180 nm. For 120 s etching, the polystyrene microspheres change into nonspherical profile with 120 nm in diameter (figures 3(G) and (H)). In order to trace the morphological evolution of the SWG, we prepared the non-close-packed polystyrene microsphere array with different etching time. Figure 4 shows the time dependency of the diameter of the etched PS particle with 260 nm in period. As the etching time increases, the diameter of PS particles decreases. Through careful study of the change of the PS particles’ diameter with different RIE time, the relationship between the oxygen RIE time and the size of the etched microspheres measured at the top view was determined by theoretical simulation from a previous report [27].

Pillar-like arrays were formed on the surface of the fused silica substrate by the second fluorochemical RIE, as shown in figures 5(A)–(I). The fused silica substrate with the hexagonally non-close-packed polystyrene microsphere array, previously etched 90 s by pure O₂ RIE, was then subjected to the second fluorochemicals RIE process. The fused silica pillar-like arrays are obtained after the polystyrene microspheres are completely removed by tetrahydrofuran.

By changing the etching time, we have prepared SWG with different height: from 125 to 227 nm. Figure 8 shows the morphology evolution of SWG with 260 nm in period as etching time increasing. As shown in figures 5(A), (D) and (G), silica pillar-like arrays were etched into the substrate with 125 nm in height after RIE etching of 4 min. Figures 5(B), (E) and (H) show the SEM images of SWG which etched for 6 min. We can see that the SWG exhibit a truncated cone-shaped profile vertical to the substrate with 165 nm in height. Figures 5(C), (F) and (I) depicts the SEM image of SWG etched for 8 min, the morphologies show a truncated cone-shaped profile with 227 nm in height, and the surface of the pillar became rougher than those of 6 min. During the RIE process, the PS microspheres are like masks, but they can also be etched by the reactive ions. From figures 5(A), (D) and (G), it is clearly seen that with 4 min etching the silica pillar-like arrays are formed after removing the left spheres. During the fluorochemicals etching procedure, the diameter of the PS microspheres reduces leading to the formation of truncated cone-shaped profile arrays, as shown in figures 5(C), (F) and (I). For the polystyrene microspheres’ monolayer mask, the tops of the spheres become rough owing to the etching of the ions during the RIE process, which leads to the formation of the pillar profile with relatively rough edges in figures 5(A)–(C).

The numerical calculation was performed using the program FDTD Solutions purchased from Lumerical Solutions, Inc. (Vancouver, Canada). The incidence optical source was set as plane waves. Periodic boundary conditions were set around a unit cell, while perfectly matched layer (PML) absorbing boundary conditions were used at the top and bottom boundaries of the cell. The unit cell was designed in rectangle lattices \( D = \sqrt{3}D \), as shown in figure 6, where \( D \) is the diameter of the nanostructures.

In our case, to calculate the transmittance of SWG on the fused silica substrate, a 3D FDTD simulation method was employed. In this calculation, the grating period of SWG was fixed to 260 nm, the height of SWG was set to 300 nm, and the diameter of nanopillar structure was varied from 0 to 260 nm. A six-fold hexagonal symmetry was used and the fused silica was taken into account.

Figure 7(A) shows the color map of the calculated total transmittance of fused silica SWG as a function of the diameter of the nanopillar (0 to 260 nm) and wavelength (300 to 1400 nm) at normal incidence for a period of 260 nm. The flat surface (diameter = 0 nm) of the fused silica substrate shows the transmittance of approximately 96% as expected. It is observed that the fused silica substrate with 180 nm diameter nanostructures exhibits a maximum transmission of >99% at normal incidence. From the EMT, the SWG here can be

![Figure 4. Time dependency of the diameter of PS particles with 260 nm in period.](image-url)
equivalent to an inserted layer with effective refraction index \( n_{\text{eff}} \), which is determined by the fused silica filling factor \( f_{\text{silica}} \). The SWG contains a fraction \( f_{\text{silica}} \) of fused silica with complex refractive index \( \approx 1.46 \) and fraction \( 1 - f_{\text{silica}} \) of air with refractive index \( n_{\text{air}} \). In order to obtain this optimisation in reducing reflection for an incident light, the optimal refractive index for a SWG should be \( n = [n_{\text{silica}} \cdot n_{\text{air}}]^{1/2} \approx 1.21 \). The effective refractive index \( n_{\text{eff}} \) of the SWG can be approximated by \( n_{\text{eff}} = [f_{\text{silica}} \cdot n_{\text{silica}}^q + (1 - f) \cdot n_{\text{air}}^q]^{1/q} \), where \( q = 2/3 \) [28]. The hexagonal structure of the pillar arrays leads to the formula \( f_{\text{silica}} = 2\pi r^2/\sqrt{3}D^2 \) where \( r \) is pillar radius and \( D = 260 \) nm is the array period (initial nanoparticle size). The calculated value of \( f_{\text{silica}} \) was 0.49 and \( r \) was 90 nm. As a result, the SWG with 80 nm in spacing of the nanopillar is optimal for high performance AR. From the numerical calculations, we used the 90 s oxygen etching times for the polystyrene microsphere with 180 nm in diameter to fabricate optimal AR structures, as shown in figures 3(E) and (F). Figure 7(B) shows optical transmittance of the SWG fused silica as a function of wavelength and height of the nanopillar. At each wavelength, there are fluctuations in transmittance as the height increases due to the interference of light reflected at the top and bottom of the layer. In fact, the optical property of the nanopillar with a sub-wavelength period is quite similar to that of a single layer AR coating, because the nanopillar acts as an effective medium that approximates a single layer thin film.

Figure 5. (A)–(C) SEM image of the top view of the nanopillar array; (D)–(F) cross-sectional view SEM image of the nanopillar array; (G)–(I) tilted-view SEM image showing the pillar-like profiles. The durations of the reactive ion etching are 4 min ((A), (D) and (G)), 6 min ((B), (E) and (H)), and 8 min ((C), (F), and (I)), respectively. All scale bars are 500 nm.

Figure 6. The unit cell of the numerical calculation was designed in rectangle lattices \((D * \sqrt{3}D)\), where \( D \) is the diameter of the nanostructure. (A) Top view of the calculation model; (B) tilted-view of the calculation model.
The heights of SWG are 125, 165 and 227 nm for etching durations of 4, 6 and 8 min, respectively. The specular transmission of different heights is measured, as indicated in the experimental section. Figure 8(A) shows the transmission spectrum of the fused silica SWG in the wavelength from 300 to 1400 nm. For the sample with 125 nm in height, the transmission is above 99% from 540–720 nm (green line in figure 8(A)). For the sample with 165 nm in height, the
maximum transmission is above 99% in 560–950 nm (red line in figure 8(A)). After etching time of 8 min, the transmission of the bare fused silica substrate increases from 93% to above 99% from 820 to 1250 nm due to the higher height of SWG (black line and blue line in figure 8(A)). Figure 8(B) shows the SWG and the bare fused silica substrate exposed to sunlight. The background below the SWG area displays a high degree of transparency and low reflection loss (bottom), but the bare area of fused silica reflects incandescent lamp (top).

The optical property of the fused silica SWG was estimated by wavelength dependent and reflection measurements. As shown in figure 8(C), the improvement of reflection is ~0.5% in 550 nm for the 4 min etching sample. We can see that the reflectivity of the 6 min etching sample was decreased to 0.5% in 700 nm. The minimum reflectance in 1000 nm is ~0.5% after RIE of 8 min (the blue line in figure 8(C)), and the reflectance of bare fused silica substrate is ~7% (the black line in figure 8(C)).

The experimental reflectance measurements are complemented by theoretical calculations using a 3D-FDTD method. As shown in the morphology of SWG, we assume the SWG has a shape truncated cone profile with 90 nm in the base diameter. The heights of nanostructures are 125, 165 and 227 nm, respectively. And apex diameters estimated from the measured SEM pictures are 80, 75 and 70 nm, respectively. Figure 8(D) displays the reflectance spectra of SWG having heights ranging from 125 to 227 nm. When the height of the SWG increases from 125 to 227 nm, the reflectance decreases significantly (~6%) at wavelengths of 300–1400 nm, and the periodicity of the reflectance spectra also changes like a single AR layer. The regular move of the reflectance of the SWG surface can be explained as follows. The etch depth, , is 1 quarter wave in optical thickness, , where is the wavelength at which the reflection minimum is to occur [28]. According to the equation, the minimum reflectance wavelength (λ) moves from a short wavelength to a longer wavelength with an increase in the etching depth if is held constant as shown in figure 8(D). The simulated spectra agree reasonably well with the optical measurements for SWG on the fused silica substrate.

The damage threshold of optical elements used in high power laser systems is of special interest. In order to interpret the high LIDT properties, the near-field distributions of SWG are simulated numerically by the 3D-FDTD method at normal incidence. The height of SWG was set to 220 nm. The calculation model has been used widely to analyze the near-field distributions of SWG displayed in figure 9. A plane wave was propagated from the air to the SWG surface in the presence or absence of pillar structure. Figure 9 depicts the different near-field distribution at the 355, 532 and 1064 nm, respectively. In the simulation, the SWG was set on the back side of the substrate because the laser induced damage always occurred on the back side of the substrate. We can see that the near-field distribution is strongly dependent on the periodicity of surface structure for incidence wave. The strong electric field region is distinctly distributed in the groove of SWG, which has the potential to promote the ability of high LIDT. Furthermore, it is evident that the enhancement of electric field with the incident wavelength of 355 nm shown in figures 9(B), (C) and (D) is also distributed in the pillar of SWG. It is so important for high power laser application that the property of fused silica has the high laser damage threshold. According to the quantitatively calculated near-field distribution shown in figure 9 for the incident wavelength of 1064, 532 and 355 nm, respectively, the LIDT at 1064 nm was investigated in our work. The results indicate a damage threshold for the fused silica window with the SWS of 32 J cm−2, and a damage threshold for the untreated fused silica substrate of 31.5 J cm−2. These results show that our SWS in fused silica are nearly as stable as the unstructured plain surface and are much more resistant to high power densities. This is because the LIDT of fused silica was limited by a damage precursor formed in the subsurface during the polishing [29].

4. Conclusions

In conclusion, we have prepared high performance optical surfaces by colloidal lithography and two step RIE. How changes in the spacing and height of SWG affect the optical properties of surface has been studied by the 3D-FDTD method and experiment. From the result of numerical calculations, the SWG with 80 nm in the spacing of the nanopillar is proved to be desirable for high performance AR properties. Then, the near-field distribution in the SWG structure performed by the FDTD method exhibits a strong dependence on the surface period. The SWG with a different height was
fabricated upon a fused silica substrate. It was shown that the transmission of the SWG had been increased to about 99\% while the reflectivity reduced to 0.5\% at the same time. Regarding the constant spacing of SWG, the incident wavelength from which the maximum transmittance was obtained, moved from a short wavelength range to a longer wavelength range with increasing the height of SWG, suggesting the maximum transmittance can be controlled by etching duration. In particular, it is expected that the antireflective SWG structure directly built into the substrate material can be applied to high power laser systems due to its promising higher LIDT. We found the LIDT of SWG to be nearly as high as for plain bulk fused silica.

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