Development of high-throughput silicon lens and grism with moth-eye antireflection structure for mid-infrared astronomy

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\textbf{ABSTRACT}

We have been developing high-throughput optical elements with the moth-eye structures for mid-infrared optical systems. The moth-eye structures are optimized for the wavelength of 25–45 \textmu m. It consists of cones with a height of 15–20 \textmu m arranged at an interval of 5 \textmu m. They are formed on silicon substrate by electron-beam lithography and reactive ion etching. As a verification of the usefulness of moth-eye, a double-sided moth-eye silicon plane was fabricated. It shows a transmittance increase of 60\% compared with the unprocessed silicon plane. As the first trial of the moth-eye optical element, two silicon lenses with single-sided moth-eye were fabricated. One is a plane-convex lens with the moth-eye on the convex surface. The size of the moth-eye formed region is 30 mm \times 30 mm. Its focal length is 186 mm. The other one is a biconvex lens with moth-eye formed region of \phi 33 mm and a focal length of 94 mm. Uniform moth-eye pattern was fabricated especially for the second lens sample. Imaging test with the first sample showed that neither image degradation nor focal length variation was induced by the moth-eye fabrication. As a step to grism with moth-eye, a moth-eye grating sample was fabricated. The grating pattern (Grating constant: 124.9 \textmu m, Blaze angle: 4 deg) was successfully fabricated with anisotropic etching. Moth-eye patterns were fabricated on the grating surface. Although the resulted moth-eye was successfully fabricated in the most regions, some non-uniformity was found. It can be attributed to unevenness of resist coating, and improvement of coating method is needed.

\textbf{Keywords:} mid-infrared, moth-eye, antireflection, lens, grism, silicon, electron beam lithography, reactive ion etching

\textbf{1. INTRODUCTION}

Mid-infrared (MIR) optical systems are cooled down to cryogenic temperature to suppress the instrumental thermal emission and dark current of detector. Cryogenic environment is achieved by refrigerant or coolers, and their cooling capability is limited. Therefore, compact system is preferred for MIR optics. At this point, refractive optics is preferable because it can make the system compact. The IRC (Infrared Camera) onboard the infrared astronomical satellite AKARI\textsuperscript{1} employed refractive optics and realized very compact system.\textsuperscript{2} The weak point of the refractive optics is ghost image caused by the internal reflection. Anti-reflection (AR) can reduce the reflection and ghost, and perfect AR is highly demanded for refractive optical systems. Actually, the

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IRC had some ghost which arises from imperfect AR coating on the lens. It strongly indicates the importance of AR.

Refractive optical system has another issue. It is throughput. Transparent materials are required for high-throughput optical systems. Although such materials are heavily limited in the MIR region, silicon, germanium, CsI, KRS-5, diamond, etc. are available. However, some materials have difficulties. CsI is deliquescent. KRS-5 has toxicity. Diamond is highly transparent, but it is very expensive. Then, the MIR-transparent materials with good availability and workability are limited to silicon and germanium. However, these materials also have a problem. The MIR refractive indices of silicon and germanium are about 3.4 and 4.0, respectively. These relatively high refractive indices are useful to make optical systems compact, however, they cause throughput decrease due to high surface reflectance. The surface reflectance can be written as $R = \left( \frac{n_1 - n_2}{n_1 + n_2} \right)^2$, where $n_1$ and $n_2$ are the refractive indices of materials contact at scoped surface. Then, the surface reflectance between silicon and vacuum is about 30% ($n_1=3.4, n_2=1.0$). That of germanium is about 36% ($n_1=4.0, n_2=1.0$). This reflection occurs at the both side of material. Therefore, the total transmittance of silicon and germanium amount to about 50% and 44%, respectively. It is severe problem for astronomical instruments. AR can improve them and is important also in this sense.

AR technology for long-wavelength MIR (LW-MIR; $\lambda = 30–50\ \mu m$) and far-infrared (FIR) region has not been established. The most common AR is multi-layer interference coating, but it has two issues in the wavelength regions. The first one is the requirement of the various optical materials. The coating consists of some layers of the transparent materials with different refractive indices. In the LW-MIR and FIR region, the optical materials are limited as described above, and there is a difficulty to design AR coatings. The other issue is fragility against cryogenic environment required for LW-MIR and FIR optical systems. The thickness of each layer is usually about a few quarter of the targeted wavelength. Then, in the wavelength regions, AR coating becomes thick (several tens/hundreds of micrometers). In addition, AR coating consists of materials with different coefficients of thermal expansion (CTE). Such structure is not durable against cryogenic environment. Therefore, AR multi-layer coating is not always good for the LW-MIR and FIR optical systems.

Another method for AR is employing sub-wavelength structure (SWS). SWS means the structures whose space scale is smaller than the scoped wavelength. Material with small voids is a good example of SWS material. For the light whose wavelength is longer than the void size, such material appears to have dielectric constant and refractive index different from the material without voids. The apparent refractive index can be tuned by varying the porosity. Then, SWS can vary the refractive index of a material freely by processing. The same structure as the interference multi-layer AR coating can be formed with SWS by stacking up the layers with different porosities. SWS capability for LW-MIR and FIR optical elements are recently shown by Wada et al. Moth-eye structure, the scope of this paper, is another technology for AR. Moth-eye is named after eyes of the insects ‘Moth’. On the surface of their eyes, many bumps exist, and they work as AR. The moth-eye structure means the surface with many bumps like moths’ eye (e.g. Fig. 1-(b)). The cause of reflection is steep

Figure 1. (a) Top view of moth-eye pattern. (b) Birds-eye view of moth-eye pattern. (c) Schematic diagram of transmittance of the moth-eye.
The change of refractive index of medium. As with SWS, the bumps of the moth-eye structure appears as smooth gradient of refractive index for the light whose wavelength is longer than the height of the bumps. This smooth gradient does not cause surface reflection, and AR is realized with the bumpy structures. Moth-eye structures have been already utilized for AR on the solar-panels, and they also have potential for AR in the LW-MIR and FIR region.

We have been developing silicon optical components with moth-eye structure to realize high-throughput LW-MIR optics. The targeted wavelength is set to 25–45 μm for application in the next-generation MIR instruments like MIMIZUKU/TAO and MCS/SPICA. Initial developments and tolerance discussions are reported by Imada et al. in this conference. In their works, moth-eye structure is formed on one side of silicon wafers. The moth-eye layer achieved a transmittance of >98%, and their capability for LW-MIR optics is shown. In this paper, as its application, development of a both-sided moth-eye wafer, a silicon lens and grism with moth-eye AR structure is reported. In Sec. 2, design and fabrication process of moth-eye pattern are shown. In Sec. 3, the development of the double-sided moth-eye wafer is shown. In Sec. 4, manufacturing and evaluation of the moth-eye lens is reported. In Sec. 5, as the first step for the moth-eye grism, fabrication of the moth-eye grating is reported. Section 6 summarizes these results.

2. DESIGN AND FABRICATION OF MOTH-EYE

In this paper, the structure of moth-eye is defined as aligned cones as shown in Fig. 1-(a) and (b). Structure of the cones must be optimized to the targeted wavelength because it defines the wavelength region where the moth-eye works as AR. The parameters to be optimized are pattern pitch \( D \) and height of the cones \( L \). The wavelength region is \( nD \sim nL \), where \( n \) is the refractive index of the moth-eye material (Fig. 1-(c)). Here, the material is silicon, and \( n \) is about 3.4. Our target wavelength is around 25–45 μm. Then, \( D \) and \( L \) are set to 5 μm and 15–20 μm, respectively.

The fabrication process is shown in Fig. 2. The moth-eye pattern is created by electron-beam (EB) lithography and reactive ion etching (RIE). At the first step, resist is coated on the silicon substrate, and mask pattern is developed with electron beam. Metal film is deposited on it. Then, metal masks were formed by liftoff. After forming the masks, the substrate is etched by RIE. The cones of moth-eye pattern are formed by controlling the energy of the etching ion gases. Finally, the masks are removed, and the moth-eye pattern is completed.

The design and fabrication process described above are applied to the all samples shown below.

3. DOUBLE-SIDED MOTH-EYE PLANE

Fabrication and optical performance of single-sided moth-eye plane samples are reported in Imada et al. in this conference. As their expansion, a double-sided moth-eye plane sample was fabricated and examined. They are shown in this section. Appearance of the sample is shown in Fig. 3-(a). The sample is an wafer with a diameter of 100 mm and a thickness of 0.6 mm. Moth-eye pattern was formed in the central square region with a size of
20 mm × 20 mm, seen as black region in the figure. The region surrounding the moth-eye region with a width of 7.5 mm is thinly (about 18 μm) etched silicon region, distinct in color from non-processed region around it. Electro micrographs of the moth-eye pattern are shown in Fig. 4-(b). The cones of moth-eye on both sides are well uniformly fabricated. The pattern pitch and cone height for both sides were measured 5.0 ± 0.1 μm and 18.2–18.5 μm, respectively.

The transmittance spectra of the sample were measured at some points to verify the performance of the moth-eye. Measured points are shown in Fig. 4-(a). Five points in the moth-eye formed region and two points in the non-moth-eye region were measured. The diameter of each measured region was 5 mm. A Fourier Transform InfraRed spectrometer (FT-IR) BOMEM DA8 was used for the measurement. The light source was a mercury lamp. The beam-splitter was a 3 μm-thick Mylar film. A deuterated triglycine sulfate (DTGS) detector was used for the detector. The resolution was set to 4 cm⁻¹. The measurement was conducted in vacuum. The measured transmittance spectra are shown in Fig. 4-(b). The data in the wavelength region of 10–15 μm are not real because they cannot be measured due to the property of the beam splitter. The transmittance spectra of the Out-1 and Out-2 are the lower ones. Those of the moth-eye formed regions are the higher ones. The latter ones are almost alike. It shows that uniform moth-eye pattern was successfully formed. The highest transmittance of >80% can be seen around the wavelength of 20–60 μm as expected from the design. On the other hand, the transmittance of the non-moth-eye region is around 50%. The transmittance increase by 60% was confirmed. It shows that the moth-eye structure works well as AR in this wavelength region. The transmittance of single moth-eye layer can be derived by correcting the effect of the reflection and absorption in the silicon substrate as described in Ref. [11]. The derived value is 95 ± 2% around 30 μm. It means that the fabricated moth-eye has performance close to the ideal one (100%). The optical elements with double-sided moth-eye are expected to have high performance in the LW-MIR region.

4. MOTH-EYE LENS

Lens is a fundamental optical element of refractive optics. Its throughput is very important because it decides the overall throughput of the system. For high-throughput MIR lens, silicon lenses with single-sided moth-eye
Two moth-eye lens samples were fabricated. One is a spherical convex-plane lens of which curvature radius is 450 mm (focal length: 186 mm). Its appearance and cross-section view are shown in Fig. 5-(a). The moth-eye was formed in the central square region (30 mm $\times$ 30 mm) on the convex surface. The sag of the edge of the moth-eye region is 0.25 mm. The electro micrograph of the moth-eye is shown in Fig. 5-(b) and (c). The cones of the central region and those of the outer region a little bit differ from each other. The largest difference is the top of the cone. This difference is attributed to non-uniform mask formation. The metal masks are formed with EB lithography as described in Sec. 2. If the mask pattern is drawn on a curved surface, deformation occurs due to the defocusing effect, and the different mask patterns are formed depending on the position. They can create non-uniform moth-eye structures. The other moth-eye lens sample is a spherical biconvex lens of which curvature radius is 450 mm (focal length: 94 mm). Its appearance and cross-section diagram are shown in Fig. 6-(a). The moth-eye pattern was formed on one side with a diameter of 33 mm. The sag of the edge of the moth-eye formed region is 0.30 mm. The micrograph of the moth-eye is shown in Fig. 6-(b) and (c). The pattern of this sample is more uniform than that of the first sample. The uniformity was realized by optimizing the position of the EB focal plane relative to the lens surface. Enough uniformity was obtained and better optical performance is expected for the second lens sample. The same moth-eye pattern can be formed on the other side by the same way. Therefore, this result opens up the possibility of high-throughput lenses with relatively small F-numbers (~3) and common diameters (~30 mm).

An imaging test to check the moth-eye fabrication effect on the focal length and image quality was conducted. The setup is shown in Fig. 7. A wedge-shaped slit illuminated by a blackbody furnace of which temperature is 1000 °C was set as an object. The images and the lens positions were examined when the slit image was focused on a bolometer camera by the first moth-eye lens and an unprocessed silicon lens whose parameters are the same

Figure 5. (a) Appearance and schematic cross-section view of the first moth-eye lens. (b) Electro micrograph of the moth-eye pattern around the central region. (c) Same as (b), but at the outer region.

Figure 6. Same as Fig. 5, but for the second lens.
Figure 7. (a) Setting of the imaging test. (b) Image focused by unprocessed silicon lens. (c) Image focused by moth-eye silicon lens.

as the moth-eye lens. A MIR band-pass filter (BPF) was inserted after the slit to check the MIR properties. The transparent wavelength was 6.8–13.8 μm.

Figure 7-(b) and (c) are the slit image focused by the unprocessed lens and the moth-eye lens, respectively. The circular pattern at the center is not real but detector defects. The bottom-right linear pattern is the focused slit image. Their appearance is not so different. It means that remarkable image degradation does not exist. The difference of the focal length of the unprocessed lens and the moth-eye lens was derived by using the lens equation and the positions of the slit, lens, and imager. The result is that the focal length of the moth-eye lens is shorter than that of the unprocessed silicon lens by 0.8 ± 0.2 mm, although the lenses were fabricated to have the same parameters. The difference is 0.4% of the designed focal length of 186 mm. The silicon lens can usually have the same order errors in the focal length. The focal length of the moth-eye lens is missed to be measured before the moth-eye fabrication. Therefore, the measured difference cannot be definitely attributed to the effect of the moth-eye fabrication. However, it can be said that significant focal length change was not induced by the moth-eye fabrication. For the second moth-eye lens, the focal length was measured before the moth-eye fabrication. The effect will be examined by the same experiment with the second moth-eye lens in future.

5. MOTH-EYE GRATING

As the first trial for the moth-eye grism, a grating sample with moth-eye was fabricated. The grating surface was formed by anisotropic etching with alkaline etchant. The way of forming grating pattern is shown in Fig. 8. Silicon has the characteristics that the (100) surface is easier to be etched than the (111) surface and that the etching speed differs depending on the crystal axis. This difference creates the saw-edged structure. Desired blaze angle can be obtained by tilting the (100) surface against the wafer surface. Here, the blaze angle was set to 4 deg. Hence, the silicon wafer was cut out as the (100) surface lines at 51 deg to the wafer surface, and it was etched with KOH after creating SiN masks.

The first result is shown in Fig. 9. Figure 9-(b) shows an electro micrograph of the sample. Its surface consists of rough regions and smooth regions, and it differs from the expected structure. Figure 9-(a) shows the schematic cross-section view. The region drawn with the dashed lines is the expected structure which cannot be confirmed in the sample. The cause is that the corrosion did not stop because of the steepness of the edge under the mask. The mask and the region under it seem to be corroded. The rough region was made by this improper corrosion, and the smooth region is the residual (111) surface which avoided the corrosion.

In the next trial, we made stopper structure to avoid the unexpected corrosion. The stopper is a wall made with a material resistive against the etching. The processing sequence is shown in Fig. 10-(a). At first, the masks are formed with EB lithography. Then, the stopper is created by using RIE, and the etching is conducted to form the grating pattern. After the grating pattern formation, the masks and the stoppers are removed.

The result is shown in Fig. 10-(b). The gradient pattern was almost successfully fabricated by using the stopper. Signature of the removed stopper is seen as dark regions. The gradient of the grating pattern can be seen at the edge of the pattern. The pattern pitch was measured 124.9 μm, although the designed value was 124.2 μm. The roughness of the grating pattern was measured by atomic force microscope (AFM). It was less than 20 nm, enough smaller than the targeted wavelength of 25–45 μm.
The moth-eye pattern was created after the completion of the grating pattern by the same method as described in Sec. 2. The designed pitch and height of the moth-eye structure are the same as the previous ones. Figure 11-(a) shows the resulted moth-eye pattern formed on the grating surface. In the most regions, the moth-eye pattern was successfully formed. Figure 11-(b) shows the magnified image of the successful moth-eye structure. The pitch of the cones was measured 4.93–4.99 μm, almost the same as the designed value of 5 μm. Regions with the successful moth-eye pattern are found in the midsection of the grating gradient. On the other hand, around the both edges of the grating gradient, the moth-eye pattern was not properly formed. At the bottom region of the gradient, there are non-uniform thick cones. At the top region, the plane and deep trough without the moth-eye pattern are found. These unsuccessful patterns are thought to be caused by unevenness of the resist coating. It is illustrated in Fig. 11-(c). The resist was coated by spin coating. Spin coating tends to cause the unevenness due to the centrifugal force especially when it is applied to complicated structures. Both too thin and too thick resist can lead the failure of the pattern formation, and the unsuccessful patterns are seen in such regions where the coating becomes too thin and too thick. To solve this problem, introduction of spray coating for resist coating is under consideration because it is more suitable to make uniform coating on complex structures. After the completion of the sample with more uniform moth-eye pattern, mid-infrared optical performance, especially about the throughput, of the sample will be examined.

6. SUMMARY

Optical elements with moth-eye AR structures have been developed for high-throughput MIR optical systems. As the first trial, the double-sided moth-eye plane sample was fabricated. The uniform optical performance
and high transmittance (>80%) in the wavelength of 20–60 μm were confirmed. Moth-eye lens and grism are under development for the advanced applications. The moth-eye pattern on the curved surface of the lens was successfully formed. The focused image and the focal length of the moth-eye lens were examined. As the result, neither image degradation nor focal length change was confirmed, however, more detailed performance verification is needed. The moth-eye on the grating surface was almost successfully fabricated, but it showed some wrong features. Those are attributed to the unevenness of the resist coating coated by spin coating. Spray coating is expected to improve the moth-eye pattern by forming more uniform resist coating. Further developments are required for the moth-eye grism.

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