

Optical Engineering

OpticalEngineering.SPIEDigitalLibrary.org

Radial-quality uniformity investigations of large-area thick Al films

Haigui Yang
Zizheng Li
Xiaoyi Wang
Zhenfeng Shen
Jinsong Gao
Shanwen Zhang

SPIE.

Radial-quality uniformity investigations of large-area thick Al films

Haigui Yang,^{a,*} Zizheng Li,^{a,b} Xiaoyi Wang,^a Zhenfeng Shen,^a Jinsong Gao,^{a,b} and Shanwen Zhang^a

^aChinese Academy of Sciences, Changchun Institute of Optics, Fine Mechanics and Physics, Key Laboratory of Optical System Advanced Manufacturing Technology, Dong Nanhu Road 3888, Changchun 130033, China

^bUniversity of Chinese Academy of Sciences, Daheng Academy, Yuquan Road 19A, Beijing 100039, China

Abstract. The fabrication of high-quality large-area thick Al films with a thickness around 10 μm or even more is one of the most important factors to realize high-performance large-size echelle gratings. During the deposition process of large-area Al films, Al film quality generally exhibits a different behavior along the radius (R) direction, which seriously affects the performance of echelle gratings. In this study, for the first time, we investigate the radial-quality uniformity of large-area ($R = 400$ mm) thick (>10 μm) Al films in detail. We not only analyze the radial-quality difference of Al films prepared by the traditional electron-beam evaporation process, but also significantly improve the radial-quality uniformity of large-area thick Al films by using a coevaporation process. By comparing two kinds of film coating processes, we clarify the origin of the radial-quality difference of Al films, and prepare large-area thick Al films with excellent radial-quality uniformity. © 2015 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: [10.1117/1.OE.54.4.045106](https://doi.org/10.1117/1.OE.54.4.045106)]

Keywords: radial-quality uniformity; large-area thick Al films; shadowing effects; echelle gratings.

Paper 150078 received Jan. 19, 2015; accepted for publication Apr. 7, 2015; published online Apr. 28, 2015.

1 Introduction

It is well known that echelle gratings have the advantages of high resolution and high dispersion because they work only at high spectral orders and high diffraction angles.^{1–3} Large-size echelle gratings are the heart of large-size optical spectrometers, which have very important applications in many engineering projects such as large astronomical telescopes and laser fusion.^{4–6} In order to achieve large-size high-performance echelle gratings, one of the most important factors is to prepare a thick, homogeneous, and flat large-area Al film coating on the glass blanks in which the grooves are made. So far, Al film coating has a long history and large-area thin Al films for reflecting mirrors have been widely reported using a thermal evaporation technique.^{7,8} For large-area Al film coating, Al film quality generally exhibits a different behavior due to the difference in deposition conditions such as incident angle and substrate temperature along the radius direction. Because Al films for reflecting mirrors are relatively thin, usually several tens or hundreds of nanometers, the influence of the radial-quality difference on its reflectance could be ignored.

For large-area thick Al films used for large-size echelle gratings, its thickness is usually several micrometers or even up to 25 μm .³ With an increase in Al film thickness, its radial-quality difference becomes more obvious. In this case, the radial-quality difference of large-area thick Al films will seriously affect the mechanical ruling and the performance of echelle gratings. First, the hardness difference of thick Al films along the radius direction directly affects the groove shape and consequently reduces the diffraction efficiency of echelle gratings. Second, the surface roughness difference induces the difference in diffraction efficiency along the radius direction. Therefore, the problems of radial-quality

difference must be resolved to produce a high-performance echelle grating. However, so far the related investigations have not been reported.

In this study, we focus on the investigations on improving radial-quality uniformity of large-area thick Al films. We first prepared large-area thick Al films by the traditional electron-beam evaporation process, and analyzed the radial-quality difference by means of surface reflectance, roughness, and hardness measurement. Then we prepared films by a double electron-beam coevaporation process, by which the radial-quality uniformity of large-area thick Al films is significantly improved. When comparing the two kinds of film coating processes, we discuss the origin of the radial-quality difference of Al films in detail, and prepare large-area thick Al films with excellent radial-quality uniformity.

2 Experimental Details

In order to deposit large-area Al films, in this study, we used an electron-beam evaporation coating machine with a chamber diameter up to 1800 mm. Figure 1 shows the schematic structure diagram. In the chamber, two electron-beam guns from the Telemark Company are symmetrically configured as evaporation sources, which are on a diameter through the axis of a rotated plane fixture. Here, it is noted that we choose a rotated plane fixture instead of a planetary substrate because large-area thickness uniformity can be conveniently achieved. Although an improved thickness uniformity can be achieved by a planetary substrate, it requires much more room for large-area Al films. During Al film deposition, K9-glass substrates are fixed on the center of a rotated plane fixture, by which the deposited Al films exhibit a central symmetry behavior.

*Address all correspondence to: Haigui Yang, E-mail: yanghg@ciomp.ac.cn

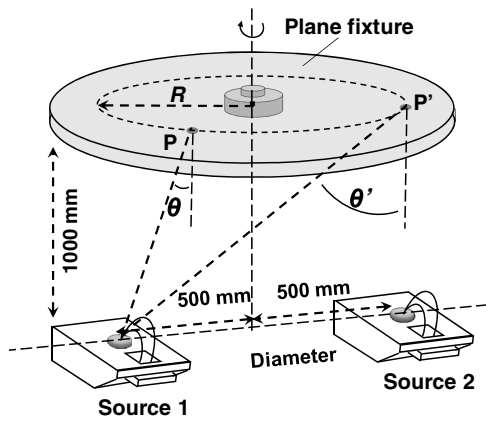


Fig. 1 A schematic structure diagram of the electron-beam evaporation coating machine.

Al films are deposited with a rate of 50 Å/s at a pressure of $5 \times 10^{-4} \text{ Pa}$ and room temperature. In order to investigate the radial-quality uniformity of large-area thick Al films, we used two kinds of film coating processes, e.g., traditional single electron-beam evaporation and double electron-beam coevaporation. For both processes, we guarantee the thickness uniformity within 1% or less by adjusting the plane fixture height. Moreover, we evaluate the inner structure, surface reflectance, surface roughness, and hardness of Al films by means of scanning electron microscope (SEM) (JEOL, JSM-6510), spectrophotometer (PerkinElmer, Lambda 1100), white-light interferometer (ZYGO, 7200), and nano indentation (Agilent, Nano Indenter G200), respectively.

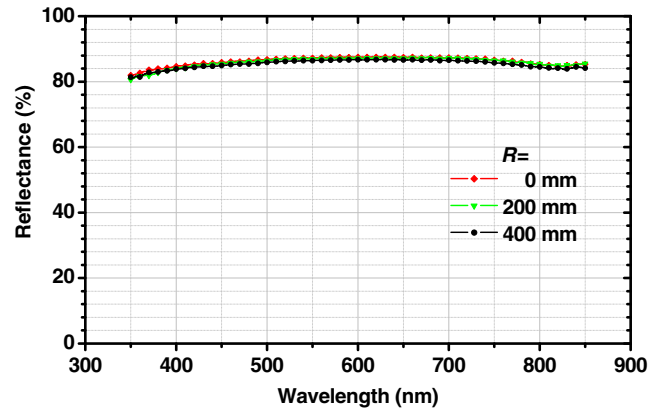
3 Results

3.1 Investigations of Large-Area Thick Al Films Fabricated by Traditional Single Electron-Beam Evaporation Process

Generally, people utilize traditional single electron-beam evaporation to deposit thin Al films around several tens or hundreds nanometers, which are generally used as the metal electrode or reflecting mirror.^{7,8} Figure 2(a) shows 500 nm-thick thin Al films with the radius (R) = 400 mm deposited on K9 substrates by the traditional single electron-beam evaporation process. By direct observation, we found that the film surface was very shiny and had no any difference along the radius direction. Figure 2(b) shows the corresponding reflectance spectra from 350 to 850 nm at the position of $R = 0, 200, 400 \text{ mm}$, respectively, which were measured at an oblique angle of 45 deg. Obviously, the reflectance has less dependence on the position of thin Al films along the radius direction. Therefore, the influence of the radial-quality difference on the reflectance can be ignored. Figure 3(a) shows 12 μm -thick Al films with $R = 400 \text{ mm}$ deposited on K9 substrates by the same evaporation process under the optimized conditions. Compared with Fig. 2(a), it is clear that the brightness of Al films obviously decreases with an increase in the Al film thickness. In particular, it was found from Fig. 3(a) that the brightness was seriously dependent on the Al film position. The brightness gradually decreases from the center of $R = 0 \text{ mm}$ to the edge of $R = 400 \text{ mm}$, which is consistent with the corresponding reflectance spectra from 350 to 850 nm as shown in Fig. 3(b).



(a)



(b)

Fig. 2 (a) 500 nm-thick thin Al films with $R = 400 \text{ mm}$ deposited on K9 substrates by traditional single electron-beam evaporation process; (b) The corresponding reflectance spectra from 350 to 850 nm at the position of $R = 0, 200, 400 \text{ mm}$, respectively, which were measured at an oblique angle of 45 deg.

For the reflectance at 600 nm, it decreases from 44.7% at the center of $R = 0 \text{ mm}$ to 34.7% at the edge of $R = 400 \text{ mm}$, which quantitatively reflects that the radial-quality uniformity of large-area thick Al films is low when deposited by the traditional single electron-beam evaporation process. Therefore, the radial-quality difference cannot be ignored for the fabrication of large-size echelle grating, and must be resolved by changing the deposition process.

3.2 Investigations of Large-Area Thick Al Films Fabricated by Double Electron-Beam Coevaporation Process

In order to improve the radial-quality uniformity of large-area thick Al films, we introduced a double electron-beam coevaporation process. As shown in Fig. (1), two electron-beam guns are symmetrically configured as evaporation sources, by which Al materials are simultaneously evaporated on substrates during Al deposition. Figure 4(a) shows 12 μm -thick Al films with $R = 400 \text{ mm}$ deposited on K9 substrates by the double electron-beam coevaporation process. By direct observation, we found that the brightness of the Al film surface obviously improved compared with that in Fig. 3(a). In particular, we cannot distinguish the brightness difference from the center of $R = 0 \text{ mm}$ to the edge of $R = 400 \text{ mm}$ along the radius direction. Figure 4(b) shows the reflectance spectra from 350 to 850 nm measured at an oblique angle of 45 deg. Compared with that in Fig. 3(b), it is obvious that both the reflectance and its uniformity were significantly improved by the double electron-beam coevaporation process. For the reflectance at 600 nm, it changes from 54.7% at the center of $R = 0 \text{ mm}$ to 51.9% at

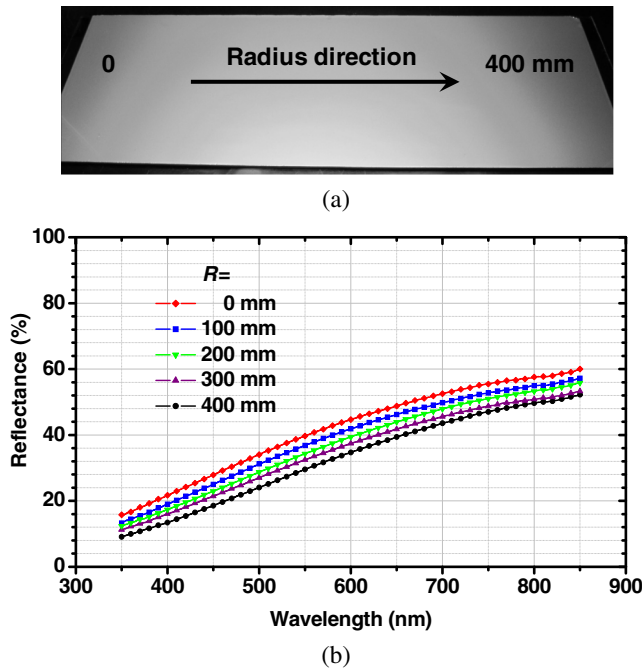


Fig. 3 (a) 12 μm -thick Al films with $R = 400$ mm deposited on K9 substrates by traditional single electron-beam evaporation process; (b) The corresponding reflectance spectra from 350 to 850 nm, which were measured at an oblique angle of 45 deg.

the edge of $R = 400$ mm. In other words, it decreases only by 3%.

It is known that surface brightness and reflectance are very closely related to surface roughness. Figure 5 shows a comparison of the surface roughness average (Ra) of 12 μm -thick Al films between the two kinds of film coating

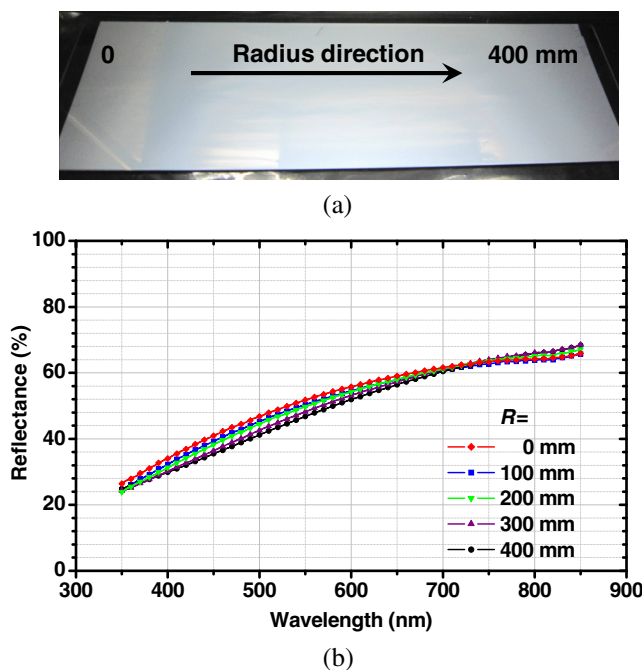


Fig. 4 (a) 12 μm -thick Al films with $R = 400$ mm deposited on K9 substrates by the double electron-beam coevaporation process; (b) The corresponding reflectance spectra from 350 to 850 nm, which were measured at an oblique angle of 45 deg.

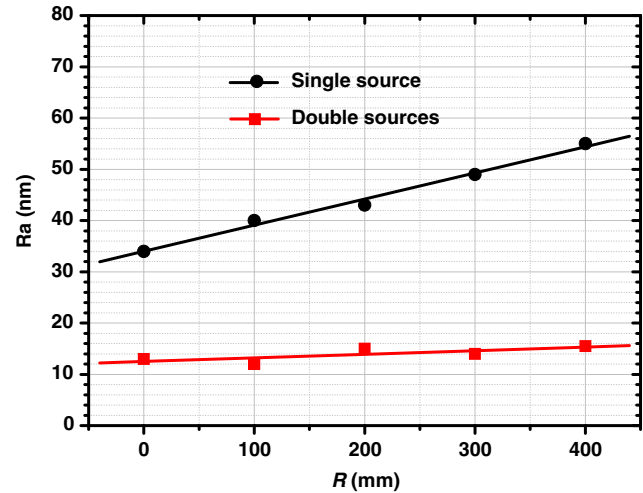


Fig. 5 A comparison of the surface roughness average of 12 μm -thick Al films between the two kinds of film coating processes.

processes. The roughness of Al films fabricated by the traditional single electron-beam evaporation process is not only much higher than that by double electron-beam coevaporation process, but gradually increases with an increase in R . Contrarily, the roughness of Al films fabricated by the double electron-beam coevaporation process maintains a low value of around 14 nm. These results are completely in agreement with that shown in Figs. 3 and 4. Thus, we concluded that the double electron-beam coevaporation process not only improved the quality as a whole, but also significantly reduced the radial-quality difference of large-area thick Al films.

4 Discussions

At first, it is concluded from Fig. 6 that the influence of thickness uniformity on the radial-quality difference should be excluded because the thickness uniformity is less than 1% within $R = 400$ mm for both deposition processes. In order to clarify the influence of different film coating process on the quality of large-area thick Al films, in this section, we discussed the growth dynamics of thin films. Recently, it has been recognized that in order to better explain the growth dynamics of thin films deposited by using thermal

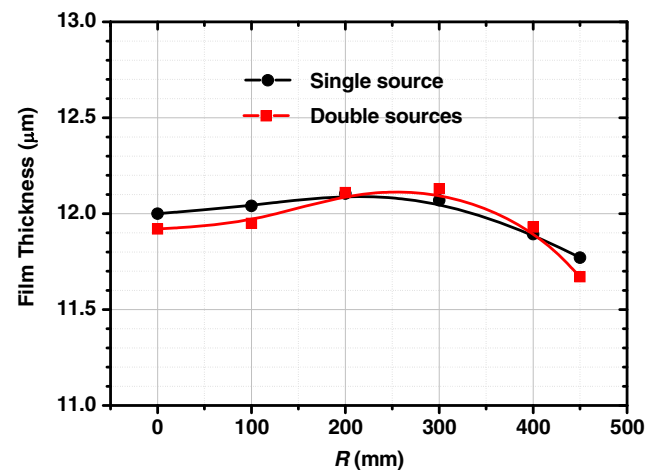


Fig. 6 Al film thickness versus radius for both deposition processes.

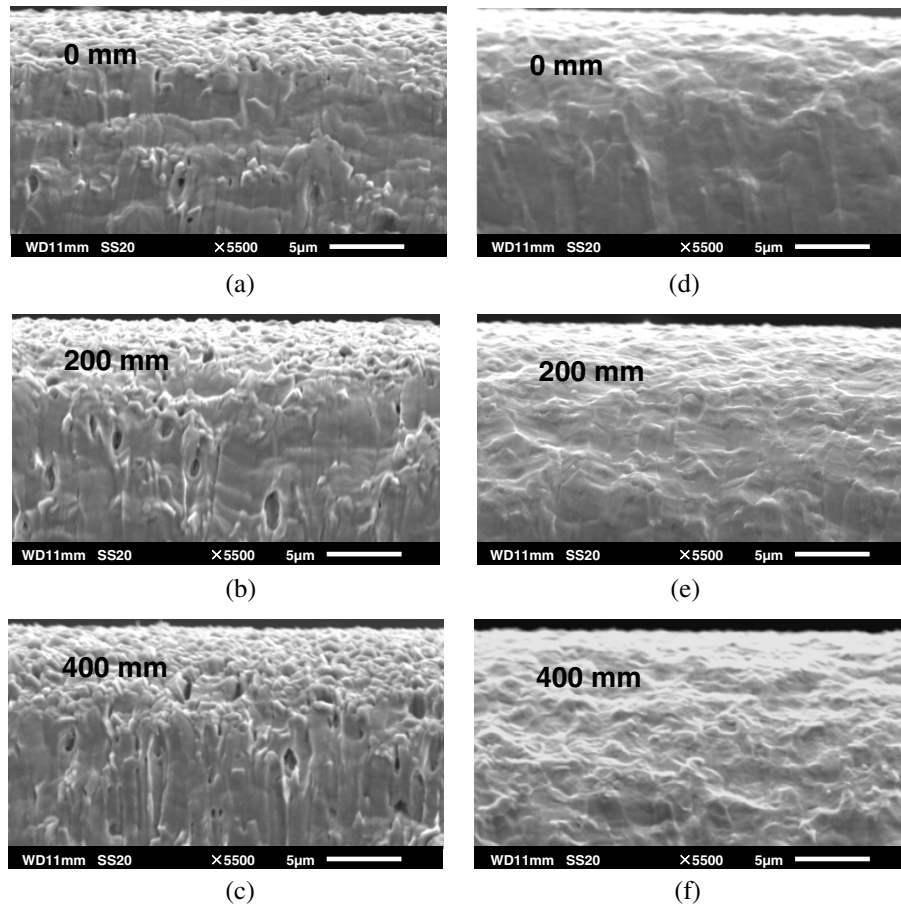


Fig. 7 The cross-sectional scanning electron microscope (SEM) images of thick Al films at $R = 0, 200$, and 400 mm fabricated by (a)-(c): traditional single electron-beam evaporation process; (d)-(f): double electron-beam coevaporation process.

evaporation or sputtering, one should take into account the shadowing and re-emission effects.^{9–11} They have been proven to be more dominant over the local effects such as surface diffusion, and act as the main drivers of the dynamical surface growth front. The shadowing effect originates from oblique incident atoms being preferentially deposited on the hills of the surface, which leads to the formation of rougher surfaces with columnar structures. Under extreme shadowing conditions, such as oblique incident angles larger than 60° , it can even produce nanostructured arrays of rods and springs through a physical self-assembly process.^{12–14} Compared with the shadowing effect, the re-emission effect is a result of nonsticking atoms, which can bounce off from hills and deposit on valleys of the surface. Therefore, re-emission has a smoothing effect, while shadowing has a roughening effect.

From the above-mentioned viewpoints, for large-area Al film deposition, we consider that the shadowing effect must be taken into account due to the large oblique incident angle. In Fig. 1, we showed the movement track of a random point (P), where θ is its corresponding incident angle. During Al film deposition, θ always varies with P position due to its circular motion. In the case of the traditional single electron-beam evaporation process, although θ is small when P rotates to the same side as the evaporation source, it (θ') becomes large when it rotates to the contrary side (P'). In particular, it becomes much larger when P' is far from the

center. For example, it changes from the minimum value of 6° to a maximum value of 42° at $R = 400$ mm. Consequently, the shadowing effect with a roughening function increases with an increase in R . Figures 7(a)–7(c) show the cross-sectional SEM images of thick Al films at $R = 0, 200$, and 400 mm fabricated by the traditional single

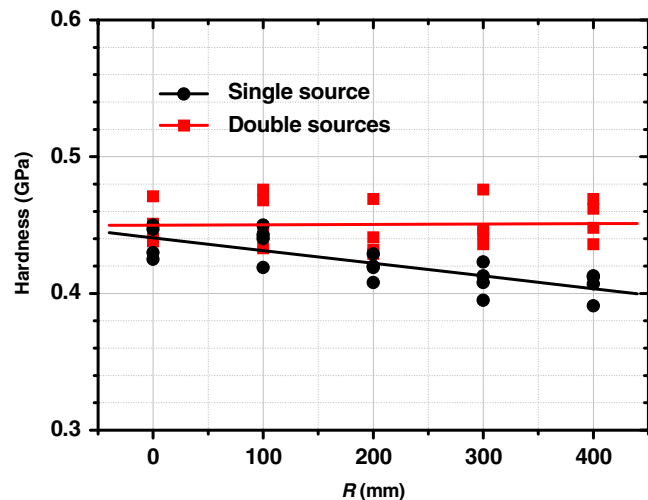


Fig. 8 A comparison of Al film hardness between two kinds of film coating processes.

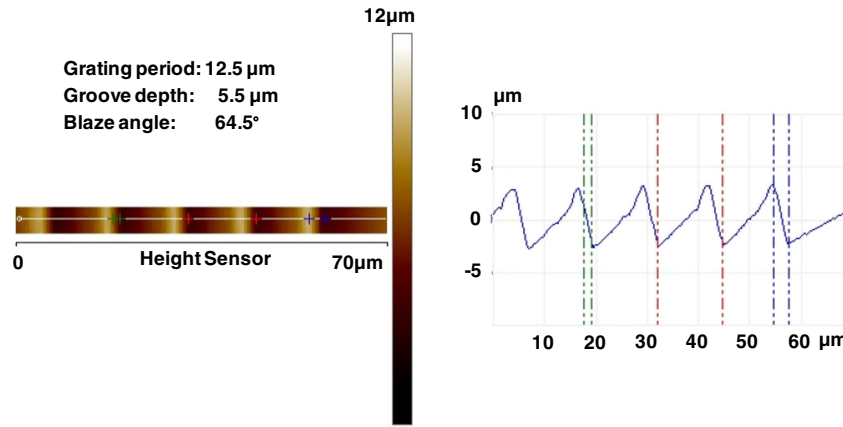


Fig. 9 Atomic force microscope (AFM) measurement results of echelle grating ruled on thick Al films.

electron-beam evaporation process. Because particles are preferentially deposited on the hills of the surface, internal hole defects induced by the shadowing effect were clearly observed in thick Al films. In addition, when R increases the defect density also increases, which should be attributed to the increasing shadowing effect due to a larger incident angle. As a result, surface brightness and reflectance in Fig. 3 and surface roughness in Fig. 5 of thick Al films fabricated by the traditional single electron-beam evaporation process exhibit a big difference along the radius direction.

In the case of the double electron-beam coevaporation process, most of the important difference is the θ change compared with the traditional single electron-beam evaporation process. As shown in Fig. 1, when P rotates to right side (P'), while θ' is very large and results in large oblique-angle evaporation for the left evaporation source, it is small-angle evaporation for the right evaporation source. Therefore, the double sources effectively compensate each other for the large oblique incident angle and diminish the influence of the shadowing effect on the Al film quality. This can be proven by the cross-sectional SEM images of thick Al films at $R = 0, 200$, and 400 mm fabricated by the double electron-beam coevaporation process, as shown in Figures 7(d)–7(f). Obviously, hole defects disappeared for the whole Al films, which indicates that the double electron-beam coevaporation process is effective for suppressing the shadowing effect, and is especially more effective for the edge area. As a result, it improves not only the surface brightness and roughness, but also the radial-quality uniformity of large-area thick Al films as shown in Figs. 4 and 5. Figure 8 shows a comparison of the Al film hardness between the two kinds of film coating processes, which is in accordance with the results in Fig. 7. Because the hole defects induced by the shadowing effect decrease, the hardness increases and almost maintains a constant value for thick Al films fabricated by the double electron-beam coevaporation process.

Finally, we attempted to rule an echelle grating on the thick Al films fabricated by the double electron-beam coevaporation process. Figure 9 shows atomic force microscope measurement results of the echelle grating, which has a period of $12.5 \mu\text{m}$, a groove depth of $5.5 \mu\text{m}$, and a blaze angle of 64.5° . The good groove shape indicates that the thick Al films fabricated by the double electron-beam coevaporation process are suitable for echelle grating ruling.

5 Conclusion

In summary, we clarified the origin of the radial-quality difference of large-area thick Al films fabricated by a traditional single electron-beam evaporation process, and found that increasing the shadowing effect caused by large oblique incident angle led to low surface brightness and high surface roughness with an increase in R . By using the double electron-beam coevaporation process, we effectively suppressed the shadowing effect, and significantly improved the radial-quality uniformity of large-area Al films. Finally, we successfully obtained large-area ($R = 400$ mm) thick ($>10 \mu\text{m}$) Al films with a low surface roughness of $R_a = 14$ nm and an excellent radial-quality uniformity. This study provides a useful approach to prepare high-quality large-area thick Al films applied in a high-performance large-size echelle grating.

Acknowledgments

Project supported by the National Key Scientific and Research Equipment Development Foundation of China (No. ZBY2008-1), the Ministry of National Science and Technology for National Key Basic Research Program of China (No. 2014CB049500), the National Natural Science Foundation of China (Nos. 61306125 and U1435210), the Science and Technology Innovation Project (Y3CX1SS143) of CIOMP, the Science and Technology Innovation Project of Jilin Province (Nos. Y3293UM130, 20130522147JH, and 20140101176JC).

References

1. G. R. Harrison, "The production of diffraction gratings: II. The design of echelle gratings and spectrographs," *J. Opt. Soc. Am.* **39**, 522–527 (1949).
2. E. G. Loewen and E. Popov, *Diffraction Gratings and Applications*, Marcel Dekker, New York (1997).
3. E. W. Palmer et al., "Diffraction gratings," *Rep. Prog. Phys.* **38**, 975–1048 (1975).
4. J. Ge, R. Angel, and J. Shelton, "Optical spectroscopy with a near single-mode fiber feed and adaptive optics," *Proc. SPIE* **3355**, 253–263 (1998).
5. Y. Zhu, "High resolution spectrographs for 8 ~ 10 m class optical/IR telescopes," *Prog. Astron.* **19**, 336–345 (2001).
6. B. W. Shore et al., "Design of high-efficiency dielectric reflection gratings," *J. Opt. Soc. Am. A* **14**, 1124–1136 (1997).
7. A. P. Bradford et al., "Preparation of mirror coatings for the vacuum ultraviolet in a 2-m evaporator," *Appl. Opt.* **8**, 1183–1189 (1969).

8. J. T. Cox, G. Hass, and J. E. Waylonis, "Further studies on LiF-overcoated aluminum mirrors with highest reflectance in the vacuum ultra-violet," *Appl. Opt.* **7**, 1535–1540 (1968).
9. T. Karabacak, "Thin-film growth dynamics with shadowing and re-emission effects," *J. Nanophotonics* **5**, 052501 (2011).
10. M. Pelliccione, T. Karabacak, and T.-M. Lu, "Breakdown of dynamic scaling in surface growth under shadowing," *Phys. Rev. Lett.* **96**, 146105 (2006).
11. M. Pelliccione et al., "Mound formation in surface growth under shadowing," *Phys. Rev. B* **74**, 125420 (2006).
12. Y.-J. Liu, H. Y. Chu, and Y.-P. Zhao, "Silver nanorod array substrates fabricated by oblique angle deposition: morphological, optical, and SERS characterizations" *J. Phys. Chem. C* **114**, 8176–8183 (2010).
13. C. Guo et al., "Microstructure-related properties of magnesium fluoride films at 193 nm by oblique-angle deposition," *Opt. Express* **21**(1), 960–967 (2013).
14. C. Jaing et al., "Residual stress in obliquely deposited MgF₂ thin films," *Appl. Opt.* **47**(13), C266–C270 (2008).

Haigui Yang is an assistant professor at the Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences. He received his BS and PhD degrees in physics from the Jilin University of China in 2002 and 2007, respectively. He is the author of more than 50 journal papers in optical, optoelectronics, and microelectronics fields. His current research interests include optical thin films and microstructure functional thin films.

Biographies for the other authors are not available.