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Sputtering Power on the Microstructure and Properties of MgF_2 Thin Films Prepared with Magnetron Sputtering

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Abstract: To reduce the F deficiency defect in MgF_2 thin films deposited with magnetron sputtering, SF_6 was added to the working gas Ar_2 as the reactive gas, and MgF_2 thin films were prepared on quartz glass substrates with radio frequency (RF) magnetron sputtering. The effects of sputtering power on the chemical compositions, microstructure and optical properties of MgF_2 thin film were investigated. The results show that with sputtering power increase from 115 to 220 W, the atomic ratio of F to Mg increased continuously, and reached 2.02 at 185 W, close to ideal stoichiometric ratio of 2 : 1. The crystallinity of MgF_2 film improved first, then decreased, and finally changed into amorphous state. Profile of particles composing MgF_2 film became clearer at first, and finally became blurred. Refractive index of MgF_2 film decreased firstly and then increased, and got the lowest value at 185 W, 1.384 at 550 nm wavelength which is very close to that of MgF_2 bulk crystal. The integral transmittance of the coated glass within 300–1100 nm (hereinafter referred to as the transmittance of the thin film) increased first and then decreased, and reached 94.99% at 185 W, higher than that of the bare glass substrate by 1.79%.

Key words: MgF_2 thin film; F deficiency; transmittance; antireflection; sputtering power; magnetron sputtering

Solar cell is one of the main power sources of various space equipment, such as satellite, space workstation, planet lander, *etc*. The service environment of space solar cell is rather harsh, where the temperature fluctuates greatly, and there are also many cosmic ray radiations and high-energy particle impacts[1]. Therefore, radiation resistance and light weight are the important characteristics of space solar cells which are different from the ground solar cells. Mars exploration is one of the important goals of human space exploration. Due to the scattering of a great deal of dust suspending on the surface and the absorption of carbon dioxide atmosphere, the scattering degree of the infrared light (long wave) part of the solar spectrum on the surface of Mars is far greater than that of the blue light (short wave) part, so the solar spectrum has a certain degree of blue shift compared with that on the earth's surface[2]. Accordingly, the designs of both the solar cell and its glass cover for Mars also need "blue shift", such as adjusting the main absorption wave band of solar cell and high transmittant wavelength band of the cover to 300–1100 nm.

One of the reasons for the loss of solar cell conversion efficiency is that the photovoltaic glass or the cover glass has a reflection loss of nearly 10% to the incident sunlight. The most direct and convenient way to increase the conversion efficiency of the solar cells is to reduce the surface reflectivity of the cells and increase the absorption of sunlight. One of the most commonly used methods of antireflection is to coat a layer of antireflection film on the surface of the glass cover, that is, to coat one or more layers of optical film on the surface of the optical element, to reduce the light reflection and increase the light transmittance through the interference of light[3]. The antireflection film can be divided into single-layer, double-layer and multi-layer antireflection films.

MgF_2 film is one of the most widely used antireflection films because of its wide transmission spectrum, good chemical stability, radiation resistance and mechanical properties[4]. There are many methods to be used to prepare MgF_2 thin films, including vacuum evaporation,

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Sol-Gel and magnetron sputtering, *etc*. Andenet, *et al.* [5] deposited MgF_2/BN bilayer film on the surfaces of GaAs and Si solar cells with thermal evaporation, which reduced the reflection loss to less than 5%, and effectively improved the transmittance of solar cells. Cid, *et al.* [6] deposited MgF_2/ZnS bilayer film on the surface of Si solar cell with vacuum evaporation, and successfully prepared broadband antireflection film. Jahanbakhsh, *et al.* [7] deposited MgF_2 on the glass substrate with electron beam evaporation and found that the measured spectrum of the sample was relatively consistent with that of the theory, and the film had the effect of increasing transmittance in the band of 400–1000 nm. Hannes, *et al.* [8] prepared MgF_2 sol at low temperature, and deposited the sol on glass to get porous MgF_2 film, of which refractive index measured by ellipsometry was 1.38, in good agreement with that of the MgF_2 bulk crystal. Nock, *et al.* [9] also prepared porous MgF_2 thin film with Sol-Gel method, which had the smallest reflectivity at 600 nm wavelength, 0.2%. The biggest advantage of Sol-Gel method to prepare MgF_2 thin film is that it can effectively suppress F deficiency defect. Its biggest disadvantage is that the film is porous, and its chemical stability, mechanical properties and radiation resistance need to be improved. Lee, *et al.* [10] prepared CeO_2/MgF_2 thin film with magnetron sputtering, of which reflectivity decreased to 1.87% in the wavelength range of 400–1100 nm, and the conversion efficiency of solar cells was improved successfully. Mertin, *et al.* [11] deposited MgF_2 film on glass substrate with direct current (DC) reactive magnetron sputtering, where Ar_2, O_2 and CF_4 gas were introduced into the reaction chamber. And MgF_2 thin film's reflectivity at 760 nm decreased to 5.2%, while the average transmittance reached 93.4% when applied to solar energy equipment.

The principle of magnetron sputtering is to bombard the target with high energy ions. The target materials are sputtered to the substrate surface in the form of ions, atoms or molecules to form thin films. Its advantages are obvious, include the high density of film, the strong adhesion between the film and the substrate, and the high preparation efficiency. However, its disadvantages are also apparent. The main one is that in the sputtering of fluoride, oxides and other compounds, most of the target materials are not sputtered out and deposited on the substrate surface in molecular form, and the film has some chemical composition deviation. The main defect of MgF_2 thin film prepared by magnetron sputtering is the F deficiency. The atom ratio of F to Mg is difficult to reach the ideal stoichiometric ratio of 2 : 1. There are a few of factors that affect the extent of F deficiency, including the quality of target material, the composition and ratio of sputtering gas, the sputtering processing parameters, such as the gas pressure, the substrate temperature, the sputtering power and so on. When the target and working gas are determined, the sputtering power has an important influence on the F: Mg atomic ratio of the film. Kawamata, *et al.* [12] investigated the effects of sputtering power and substrate temperature on the structure and properties of the film and found that MgF_2 could be sputtered out and deposited on the glass substrate in the form of molecules at high power of 600 W and high temperature of 670 ℃, which significantly inhibited the F deficiency.

In this work, SF_6 was added to the working gas Ar_2 as the reactive gas, and MgF_2 thin films were deposited on quartz glass substrates *via* RF magnetron sputtering with high pure MgF_2 target. The effects of sputtering power on the structure and optical properties of MgF_2 thin films were investigated.

1 Experimental

1.1 Film preparation

The light transmission process in the system of Air/MgF_2 thin film (refractive index 1.38)/quartz glass substrate (refractive index 1.46, thickness 1.0 mm) was simulated with G-solver software. The optimal thickness of MgF_2 thin film was determined to be 70 nm, and the highest integral transmittance within 300–1100 nm, *i.e.*, the film transmittance, to be 94.51%.

MgF_2 thin films were deposited on quartz glass (99.9%, 50 mm×25 mm×1 mm) *via* RF magnetron sputtering with MgF_2 target (Φ101.6 mm×3 mm, 99.99%, Nanchang Guocai Technology Co., Ltd., China). Before sputtering, the quartz glass pieces were cleaned with ultrasonic 15 min with decontamination powder, acetone, anhydrous ethanol and deionized water in turn, and then dried in a drying oven for standby; the working gas was high-purity Ar_2 (99.999%), SF_6 (99.999%) with a flow ratio of 5% was added as the reactive gas, and the background vacuum degree was 6.0×10^{-4} Pa; the sputtering working pressure was 2.0 Pa; the RF power supply was set to 115, 150, 185, and 220 W, respectively; the target was pre-sputtered for 10 min before deposition to clean the target surface, stabilize the working pressure and RF power.

1.2 Characterization

X-ray photoelectron spectroscopy (XPS, K-alpha, Thermo-fisher, USA) was used to qualitatively and quantitatively analyze the chemical composition of the film. X-ray diffraction (XRD, D8 Discover, Bruker, Germany) was used to analyze the crystal structure of the film, and the measuring angle ranged from 10° to 80°. The surface morphology of the film was observed with field emission scanning electron microscope (FE-SEM, S4800, Hitachi, Japan). The thickness and refractive index of the film were determined with a spectrum ellipsometer (COSE-DVN-D-C, Syscos Instrument Technology (Shanghai) Co., Ltd, China). The film's transmittance spectrum within 300–1100 nm was reordered with an ultraviolet-visible near infrared spectrophotometer (UV-Vis-NIR, UH4150, Hitachi, Japan).

2 Results and discussion

2.1 F: Mg molar ratio of thin films

The XPS spectra of MgF_2 films are shown in Fig. 1. From Fig. 1 (a-d), the film mainly contains F, Mg, C, Si elements. Among them, C may be from the CO_2 adsorbed from the atmosphere during the sample storage and transfer process or the adhered organic matter from the transparent plastic sample bag; Si should come from the glass substrate. It can be found that the height of F1s peak in Fig. 2(b) is significantly higher than that in Fig. 2(a); the height of F1s peak in Fig. 2(c) or (d) is much higher than that in Fig. 2(b). This means that the relative content of F in the film increases with the sputtering power.

The molar ratios of F to Mg in the films prepared at different sputtering powers were determined to be 1.65 (115 W), 1.88 (150 W), 2.02 (185 W), and 2.44 (220 W), respectively, with calculation according to the intensities of Mg and F peaks in each figure and an appropriate sensitivity factor. It can be seen that with the increase of sputtering power, the molar ratio of F: Mg increases continuously, and 2.02 at 185 W is the closest to the ideal stoichiometric ratio of 2∶1. This is mainly because with the increase of sputtering power, the ionized F^- ratio from SF_6 increases gradually, so the molar ratio of F to Mg in the film also increases.

Tuszewski, *et al.* [13] investigated Ar_2/SF_6 plasma discharge with optical emission and mass spectrometry. They found that the SF_6 gas was decomposed into lighter SF_x ($x = 0–2$) and S_2F_x ($x = 0–1$) neutral species, F^- and other positive ion species, such as S, SO, SF, S_2, SOF, SF_2, S_2F SiF_3, SF_3, SOF_3, SF_5, *etc.* When the RF power increased, the total percentage of positive ion species SF_3 and SF_5 decreased obviously, correspondingly the total percentage of other positive ion species S, SO, SF, S_2, SOF, and SF_2 increased, which meant that a single SF_6 molecule could contribute more F^-. When the sputtering power was 185 W, the amount and concentration of F^- ion ionized in the vacuum chamber just made the combination of F^- ions and Mg^{2+} ions basically meet the ideal stoichiometric ratio of 2∶1. When the sputtering power continued to increase, the concentration of F^- ion ionized in the chamber was too high, resulting in excessive F^- ions in the film, and the molar ratio of F to Mg reached 2.44, far exceeding the ideal stoichiometric ratio 2∶1.

2.2 Micro structure

Fig. 2 shows the XRD patterns of the prepared MgF_2 films.

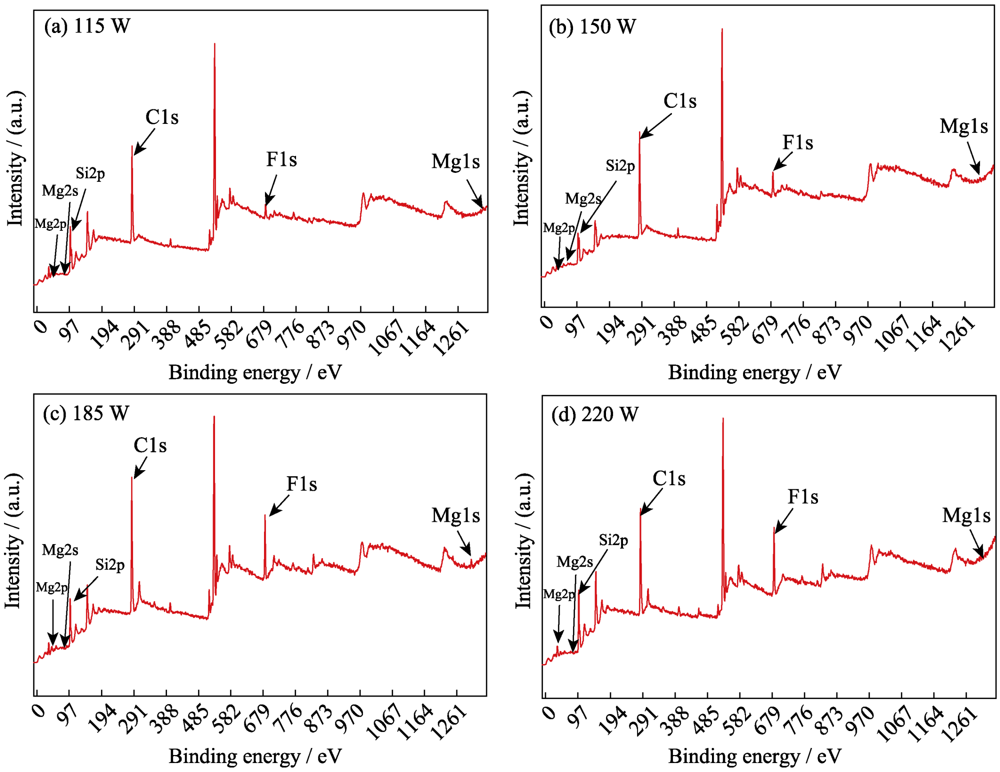

Fig. 1 Effect of sputtering power on XPS spectrum of MgF_2 film

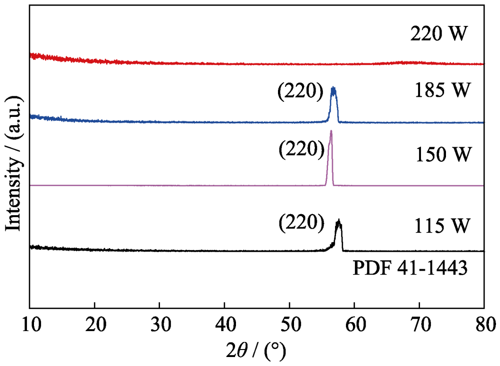

Fig. 2 Effect of sputtering power on XRD pattern of MgF_2 thin film

It can be seen from the figure that, in the first three diffraction patterns of the thin films with power of 115, 150, and 185 W, only the characteristic diffraction peak of (220) crystal plane appears near 2θ=56°, the main characteristic diffraction peak of (110) crystal plane and other characteristic diffraction peaks of (111) crystal plane and (211) crystal plane do not appear; while in the diffraction pattern of the thin film with power of 220 W, there is no diffraction peak. Secondly, when the power increased from 115 W to 185 W, the characteristic diffraction peak of (220) crystal plane firstly became sharper then widened, and its intensity firstly increased greatly then decreased rapidly; when the power reached 220 W, this diffraction peak disappeared. It indicates that with the increase of sputtering power, the diffraction peak intensity firstly increases and then decreases, while the half peak width firstly decreases and then increases.

The intensity, position angle and FWHM (Full width at half maximum) of the diffraction peak of (220) crystal plane of each film, as well as the molar ratio of F to Mg, are listed in Table 1.

According to the Scherrer formula $D = K\lambda/\beta\cos\theta$ (where D is the grain size, K is the Scherrer constant, conventionally set at 0.89, λ is the X-ray wavelength 0.154056 nm, β is the corrected FWHM of (222) plane diffraction peak, and θ is Bragg's diffraction angle), the calculated average grain sizes of these three samples are 9.6, 17.2 and 10.4 nm, respectively, which are also listed in Table 1. In addition, the lattice constants of the crystal were refined with Jade analysis software, which are also listed in Table 1. With the sputtering power increase from 115 W to 150 W, the interplanar spacing of (220) crystal plane of the film increases from 0.1593 nm to 0.1628 nm, and then decreases to 0.1615 nm at 185 W. The grain size also changes with the increase of sputtering power. When the sputtering power increases from 115 W to 150 W, the grain size increases from 9.6 nm to 17.2 nm. As the power further increases to 185 W, the grain size decreases to 10.4 nm. Both the FWHM change of the diffraction peak of (220) crystal plane and the that of the grain size confirm the change of the crystalline state of the film with the sputtering power.

The absence of characteristic diffraction peaks of (110), (111) and (211) crystal planes may be due to the preferred orientation of the sputtered particles during deposition and crystallization. Compared with (110), (111), (211) crystal planes, the interspacing between (220) crystal planes is smaller (PDF 41–413), and the <220> crystal orientation is in the direction of non-dense arrangement of atoms, so it is easier for particles to adhere along the <220> crystal orientation. When the sputtering power was low, the crystallinity of the film was also low. Firstly, the molar ratio of F to Mg was only 1.65 : 1, far away from the ideal chemical ratio of 2 : 1, as described in Section **2.1**. Secondly, the energy of the sputtered particles was low, which was not conducive to the combination and crystallization of particles. When the sputtering power increased, the molar ratio of F to Mg was closer to the ideal chemical ratio of 2 : 1, and the sputtered particles also obtained higher energy, which was conducive to the combination and crystallization of particles. When the sputtering power was too high, firstly, the molar ratio of F to Mg was again far away from the ideal chemical ratio; secondly, the output of the sputtered particles, the energy of the particles and the deposition rate of the film were greatly increased, and the combination and reaction time between the particles were shortened; thirdly, the high-energy particles might impact the deposited particles, resulting in the secondary sputtering. All of these were not conducive to the combination and crystallization of particles, so the crystallinity of the film seriously decreased, and finally became amorphous.

2.3 Surface morphology

The surface micro morphologies of MgF_2 films are shown in Fig. 3. It can be seen that the sputtering power

Table 1 Molar ratio of F to Mg and XRD analysis results of MgF_2 films

Sample	n(F) : n(Mg)	2θ/(°)	Intensity/(a.u.)	FWHM	Interplanar spacing/nm	Grain size/nm
115 W	1.65	57.832	127	0.95	0.1593	9.6
150 W	1.88	56.447	36086	0.534	0.1629	17.2
185 W	2.02	56.968	158	0.874	0.1615	10.4
220 W	2.44	Amorphous	–	–	–	–

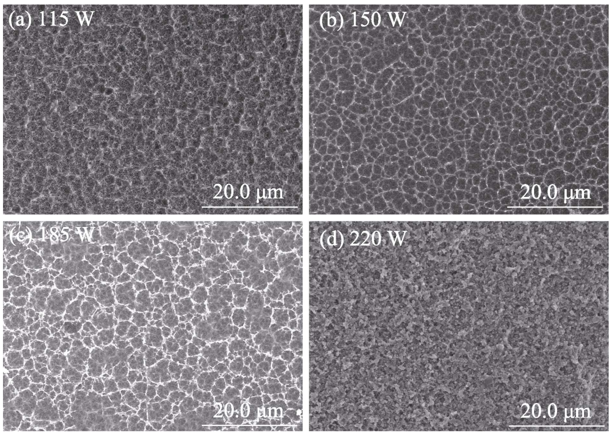

Fig. 3 Effect of sputtering power on the surface micro morphology of MgF_2 film

has a significant effect on the surface morphology of MgF_2 film. First, all film surfaces are very compact. Comparing Fig. 3(a) with Fig. 3(b), we can see that after the sputtering power increased from 115 to 150 W, the profile of particles on the film surface became clearer, and particle size increased slightly. When the sputtering power increased from 150 W to 185 W, the particle size continued to increase, forming a white network between the particles, as shown in Fig. 3(c). When the sputtering power continued to increase to 220 W, the profile of the particles on the film surface became blurred, as shown in Fig. 3(d). The change of the film surface micro morphology corresponds to that of the film crystallinity. However, we can see that the particle size shown in Fig. 3 is much larger than the grain size in Table 1, it's due to the fact that a single particle should be composed of many grains that aggregated together.

2.4 Optical properties

Fig. 4 shows the refractive index spectra of MgF_2 films. It can be seen from Fig. 4 that, as the incident wavelength increases from 300 nm to 1100 nm, the refractive index of the film decreases gradually. Secondly, with the increase of sputtering power, the refractive index of the film decreases first and then increases; when the sputtering power is 185 W, the refractive index of the film is the lowest. Table 2 lists the thicknesses of the films, the refractive indexes within 300–1100 nm and the refractive indexes at 550 nm of the films prepared at different sputtering powers. It can be seen that with the increase of sputtering power, the refractive index at 550 nm decreases from 1.501 to 1.384, which is very close to that of the MgF_2 bulk crystal, 1.38, and then increases to 1.484.

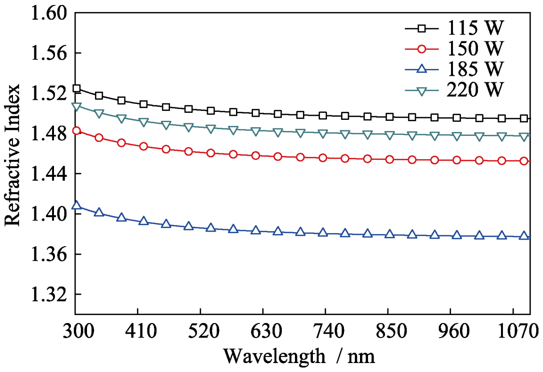

Fig. 4 Effect of sputtering power on refractive index spectra of MgF_2 thin film

The change of refractive index is mainly due to the fact that molar ratio of F: Mg. MgF_2 is the lowest refractive index substance in nature. Once the molar ratio of F to Mg deviates from the ideal chemical ratio 2 : 1, the refractive index of MgF_2 will become larger. Secondly, it comes from the change of crystallinity state. When the crystallinity of the film increases, the defects in the film reduces, so does the interference to the optical transmission, thus the refractive index decreases. But the effect of the latter is far less than that of the former. Therefore, when the sputtering power is 185 W, the ratio of F to Mg in the film is 2.02, closest to the idea stoichiometric ratio of 2 : 1, the refractive index is the lowest.

Fig. 5 shows the transmittance spectra of the glass cover coated MgF_2 films (hereinafter referred to as the film transmittance spectrum) and that of the bare glass substrate within 300–1100 nm. It can be seen from the figure that, firstly, both the film transmittance and the glass substrate transmittance increase with the increase of wavelength. Secondly, all the film spectra intersect with the substrate spectrum. The wavelength of the junction firstly decreases and then increases with the increase of sputtering power. The junction wavelengths are about

Table 2 Thickness, refractive index and integral transmittance of MgF_2 film

Sputtering power/ W	Thickness/ nm	Refractive index within 300–1100 nm	Refractive index at 550 nm	Integral transmittance within 300–1100 nm/%
115	65	1.525–1.494	1.501	92.489
150	63	1.483–1.452	1.459	93.433
185	67	1.408–1.377	1.384	94.990
220	76	1.508–1.477	1.484	92.925
Glass	0		1.46	93.20

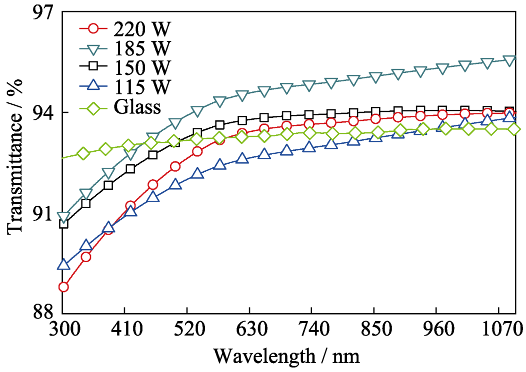

Fig. 5　Effect of sputtering power on transmission spectra of MgF_2 thin film

930, 580, 430 and 500 nm, respectively. That is to say, the junction first moves to the left and then to the right. At the right of the junction, the transmittance of film is higher than that of the substrate; at the left of the junction, the transmittance of film is lower than that of the substrate. According to the visual inspection, the integrated transmittance of 115 W film should be lower than that of the glass substrate, and the transmittance of other films may be higher than that of the substrate. Moreover, the cross point of 185 W film spectrum with that of the substrate is on the left of other cross points, and the transmittance of 185 W film on the right is far higher than that of other films, so the integrated transmittance of 185 W film should be the highest.

To integrate all transmittance spectra, the specific transmittance values are also listed in Table 2. It can be seen that with the increase of sputtering power from 115 W to 220 W, the transmittance of the thin film first increases from 92.498% to 94.99%, and then decreases to 92.925%; only the transmittance of the thin film prepared at 150 or 185 W power is higher than that of the glass substrate, 93.2%, realizing the antireflection function. Fig. 6 is based on the data in Table 2, shows more intuitively that the transmittance (300–1100 nm) of the film has a good corresponding relationship with the refractive index at 550 nm, that is, the transmittance increases with the decrease of the refractive index, and decreases with the increase of the refractive index. As stated in the experimental section, the optimal thickness of MgF_2 film is 70 nm, and the integral highest transmittance calculated with G-solver software is 94.51%. The experimental results show that the transmittance of film prepared at 185 W is 94.99%, which is about 0.5% higher than the calculated value, and about 1.79% higher than that of the bare glass substrate.

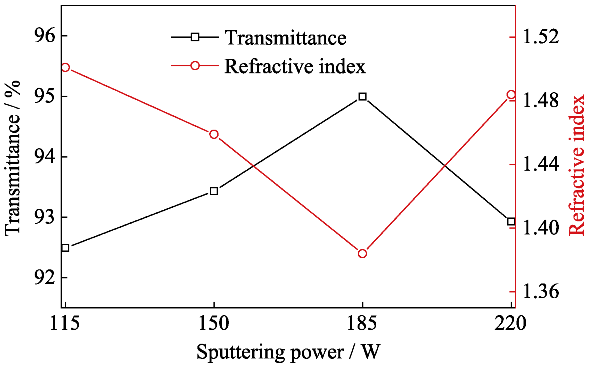

Fig. 6　Relation between integrated transmittance within 300–1100 nm and refractive index of thin film at 550 nm

3 Conclusions

F deficiency is a common defect of MgF_2 thin films prepared with sputtering and evaporation. In order to reduce the F deficient defect in MgF_2 thin films prepared by magnetron sputtering, SF_6 was added to the working gas Ar_2 as the reactive gas, MgF_2 thin films were prepared on quartz glass substrates with RF magnetron sputtering. The chemical composition of the film was quantitatively analyzed with XPS, the microstructure of the film was analyzed with XRD, and the surface micromorphology of the films was observed with SEM, the transmittances of MgF_2 film and glass substrate were measured with UV-Vis-NIR spectrometer, and the thickness and refractive index of the film were measured by spectrum ellipsometer. The effects of sputtering power on the structure and properties of MgF_2 films were investigated. The main conclusions are as follows:

1) With the sputtering power increase from 115 to 220 W, the atomic ratio of F to Mg increases continuously; at 185 W, the atomic ratio of F to Mg reaches 2.02, closest to the ideal stoichiometric ratio of 2 : 1.

2) The crystallinity of MgF_2 film increases first, then decreases, and finally changes into amorphous state with the increase of sputtering power; when the power is 150 W, the crystallinity is the highest. The particle profile of MgF_2 film surface becomes clearer at first, and the particle size increases slightly, finally the particle profile becomes blurred.

3) The refractive index of MgF_2 film decreases firstly and then increases with the increase of sputtering power. When the power is 185 W, the lowest refractive index at 550 nm of MgF_2 film is 1.384, close to that of MgF_2 bulk crystal.

4) The integral transmittance of the coated glass at 300–1100 nm reaches 94.99% when the sputtering power is 185 W, which increases the transmittance of the glass substrate by 1.79%.

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溅射功率对磁控溅射法制备 MgF_2 薄膜组织和性能的影响

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摘　要: 为了减少磁控溅射法沉积 MgF_2 薄膜的 F 贫乏缺陷, 在工作气体 Ar_2 中加入 SF_6 作为反应气体, 在石英玻璃衬底上用射频磁控溅射法制备了 MgF_2 薄膜, 研究了溅射功率对 MgF_2 薄膜化学成分、微观结构和光学性能的影响。结果表明, 随着溅射功率从 115 W 增加到 220 W, F: Mg 的原子比不断增加, 185 W 时达到 2.02, 最接近理想化学计量比 2 : 1; 薄膜的结晶度先提高后降低, 最后转变为非晶态; MgF_2 薄膜的颗粒尺寸先是有所增加, 轮廓也变得更加清晰, 最后又变得模糊。MgF_2 薄膜的折射率先减小后增大, 在 185 W 时获得最低值, 550 nm 波长的折射率 1.384 非常接近 MgF_2 块体晶体; 镀膜玻璃在 300~1100 nm 范围内的透光率(以下简称薄膜透光率)先增大后减小, 185 W 时达到 94.99%, 比玻璃基底的透光率高出 1.79%。

关　键　词: MgF_2 薄膜; F 贫乏; 透光率; 减反射; 溅射功率; 磁控溅射

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