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Fabrication of a Composite Film Optic with High Transmittance in Vis and IR Regions for an Optical System

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Abstract: In this study, a lightweight composite polyimide film optic with antireflective coating was prepared using an ion-beam-assisted reactive sputtering method. It exhibited high transmittance in visible (87%, 0.5~0.8 μm), short (86%, 1.8~2.7 μm) and middle (78%, 3.3~5.4 μm) wavelength infrared regions. Additionally, it also showed great optical homogeneity (0.006 λ in RMS and 0.034 λ in PV, λ = 632.8 nm) with smooth surfaces (RMS = 0.786 nm). The thermal stability of the polyimide film was also effectively maintained after the antireflective coating. At the same time, by utilizing the hydrophobic properties of coating materials (Ta_2O_5/SiO_2), the antireflective layer acted as an effective water barrier on the film surface to reduce the water absorption.

Keywords: composite polyimide film; Ta_2O_5/SiO_2 antireflective coating; high transmittance; visible; infrared

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1. Introduction

Its excellent thermal stability, high mechanical strength, chemical resistance and easy shape-controlled ability makes polyimide (PI) a fine candidate for a lightweight optical system. With a lot of application experience in space and on the ground, PI film optic has great application potential in a large diameter optical system. However, traditional aromatic PI films usually have low optical transmittance in both Vis and the infrared range because of their molecular structures [1–3]. Further, the PI films mostly show higher water absorption compared to conventional polymers due to the strong polar amide groups and other hydrophilic groups in the molecular structures [4]. Therefore, PI films for use as lightweight optical lenses with good optical properties and environmental suitability are needed.

Previous literature surveys have mainly focused on improving the optical properties of optical lenses using antireflective coatings. However, this is more about antireflective coatings on hard-base optics. These coatings offer many advantages, for example low reflection, high transmittance and good mechanical stability [5–8]. These antireflective coatings are generally formed by evaporation, rapid thermal oxidation, sputtering, plasma-enhanced chemical vapor deposition, electron cyclotron resonance chemical vapor deposition, etc. [9–14]. However, very few studies have focused on antireflective coatings on flexible polymer film optics. Additionally, some of the coating methods mentioned above are not applicable to polymer films because of their strict preparation conditions.

For the physical vapor deposition (PVD) deposition method, the selection of substrate materials is broader. Like in the flexible display field, PVD technology is often used for depositing active metal electrodes and functional films in the flexible active-matrix organic light-emitting diode (AMOLED) industry [15]. PVD technology, especially the sputtering deposition method, has shown its compatibility well with the deposition of polymer films and the deposition of other materials, such as the deposition of metal films and alloy films, and the deposition of compounds, ceramics and semiconductors. In this study, in order

to obtain high quality composite films at a low temperature, ion beam assisted reactive sputtering was put forward.

The materials to be chosen for an antireflective film are also very important. In this work, the purpose was to improve the transmittance of the polyimide film optic in both the Vis and IR regions. At the same time, we hope that it will act like a physical barrier to reduce the water absorption of the substrate polyimide film. Various materials can be used for preparing optical antireflective coatings, such as HfO_2, ZrO_2, TiO_2, Ta_2O_5, Nb_2O_5, YbF_3, SiO_2, Al_2O_3, CeO_2, MgF_2, ZnS, etc. Among them, Ta_2O_5 and SiO_2 have been the two most commonly used coating materials in visible and near-infrared bands [16–27]. Ta_2O_5, with a high refractive index, has a wide transmittance spectrum, low film absorption rate, stable chemical properties and good hydrophobicity. SiO_2, with a low refractive index, has good thermal stability, high light transmittance and good dielectric properties [27,28]. Therefore, Ta_2O_5 and SiO_2 have been used as coating materials in this work.

2. Materials and Methods

2.1. Preparation of PI film

Polyimide film (PI) was prepared referring to the previous method [29]. The synthesis details are as follows: 5.8844 g 3,3′,4,4′-Biphenyltetracarboxylic Dianhydride (BPDA, 98%, TCI) and 4.5452 g Diamine 4,4′-Diaminobenzanilide (DABA, 98%, TCI) (Tokyo Chemical Industry Co., Ltd., Tokyo, Japan) (n_{DABA}:n_{BPDA} = 1:1) were dissolved in 117.542 g N-Methyl-2-Pyrrolidinone (NMP, 99.5%, Aladdin reagents) (Shanghai Aladdin Bio-Chem Technology Co., Ltd, Shanghai, China) in a dry three-neck flask equipped with a mechanical stirrer and nitrogen flow at room temperature. The reaction mixture was stirred for 48 h under a N_2 atmosphere. The concentration of the solution was controlled at 8% (wt.). The poly (amic acid) (PAA) resin solution was de-aerated before being cast on the surface of the quartz glass plate by spin coating. At the end, it was heated by vacuum oven in stages (100 °C/1 h; 200 °C/1 h; 300 °C/1 h) to convert the amic acid functional groups to imides with a cyclodehydration reaction. The produced polyimide film (22–25 μm) was separated from the substrate. The synthesis of PAA resin and the preparation process of PI film are shown in Figure 1. The detailed thermal imidization procedure is shown in Figure 2.

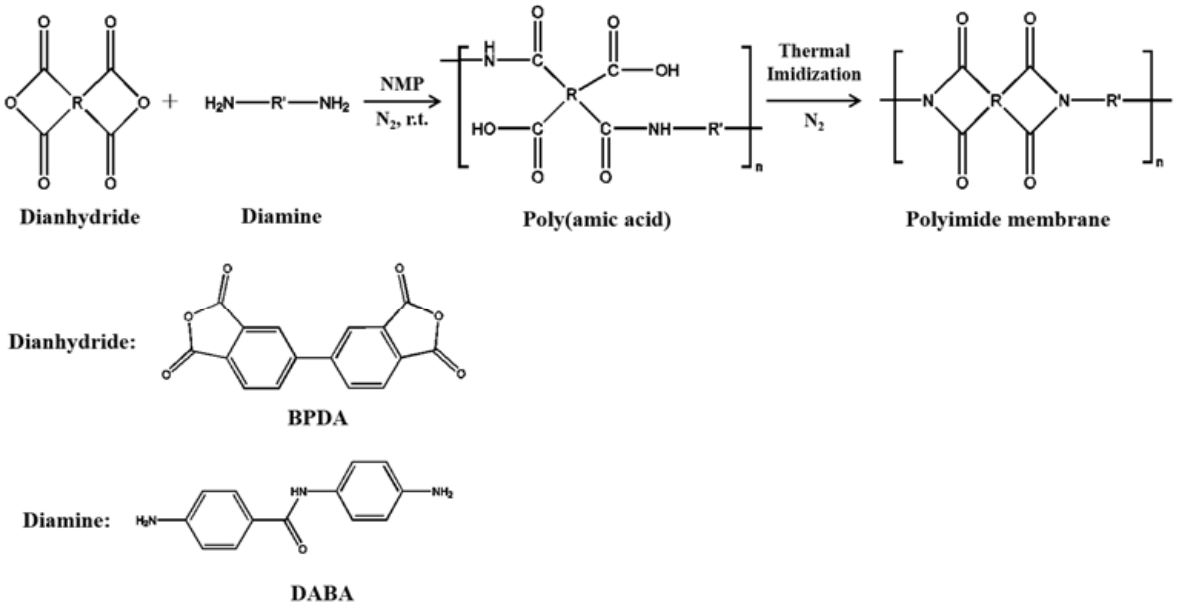


Figure 1. The synthesis of PAA resin and the preparation process of PI film.

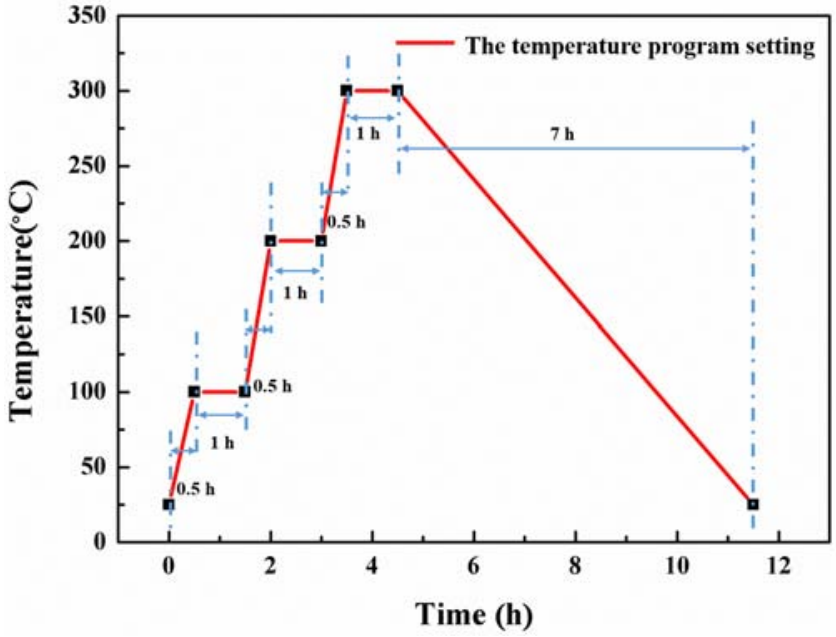


Figure 2. The thermal imidization procedure of PI film.

2.2. Preparation of the Composite Polyimide Film Optic

Tantalum oxide (Ta_2O_5, n = 2.0466, λ = 632.8 nm) and silicon oxide (SiO_2, n = 1.4500, λ = 632.8 nm) have been considered as antireflective coating materials. The characteristics of the PI film have been taken into account [30]. The multilayer antireflective film consisted of Ta_2O_5 and SiO_2 films, and the detailed film design is shown in Table S1 (Supplementary Materials). The deposition technology used must be suitable for the durable temperature and thermos-physical properties of polyimide film. The layers (Ta_2O_5 and SiO_2) were deposited on a φ50 mm PI film optic by ion beam assisted reactive sputtering. Before coating, the substrate had been washed with anhydrous Ethonal (99.8%). Figure 3a,b show the pictures of the ϕ50 mm film optic before and after being coated with antireflective film. The whole process was performed in a vacuum. The sputtering power was 150 W and the air pressure was 1.0 Pa. The temperature of the PI substrate was controlled at 40~50 °C. The total thickness of the antireflective film was 891.61 nm. The experiment was conducted in a vacuum magnetron coating machine (Chengdu Xingnan Yi Vacuum Equipment Co., Ltd., Chengdu, China).

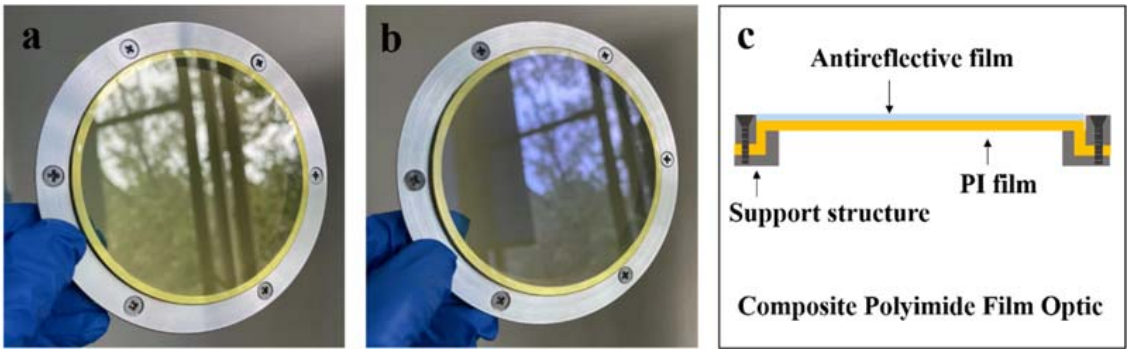


Figure 3. The pictures of ϕ50 mm PI film (**a**) before and (**b**) after being coated with antireflective film; (**c**) the structure illustration of the composite polyimide film optic.

After being coated with antireflective film, the composite polyimide film optic showed a blue reflection under sunlight, as shown in Figure 3c. The PI Film was mounted on the supporting structure to ensure the planar surface.

2.3. Characterization

The transmission spectra of film optics in the visible and short wavelength infrared regions were measured by an ultraviolet–visible–near infrared spectrophotometer (Perkin Elmer Lambda 1050, Perkin Elmer, Waltham, MA, USA) in the wavelength (λ) range of 0.2~2.5 μm. The transmission spectra of film optics in the middle wavelength infrared region were measured by infrared spectrophotometer (Perkin Elmer Spectrum^{TM} 3, Perkin Elmer, Waltham, MA, USA) in the wavelength (λ) range of 2.5~6 μm. The optical inhomogeneity of membranes was carried out by wave-front error using a Zygo laser interferometer (GPI XP, Middlefield, CT, USA), and the measurement wavelength was 632.8 nm. The surface roughness was characterized by laser interferometer optical microscope (Bruker Optics, Ettlingen, Germany). The water absorption test weighed the dry PI films (5 cm × 5 cm, three pieces) and composite PI films (5 cm × 5 cm, three pieces) to obtain the original weight (W_0), first. Then, soak them in water for 24 h, take them out, dry the water on the surface and weigh them again to obtain the weight (W_A). Calculate the water absorption according to the formula:

$$\text{Water absorption rate} = [(W_A - W_0)/W_0] \times 100\% \quad (1)$$

The glass transition temperature (T_g) of the PI film and composite PI film were regarded as the peak temperature of the tan δ curves, which was measured using dynamic mechanical analysis (DMA) performed on a TA Q800 instrument (TA Instruments, New Castle, DE, USA) at a heating rate of 5 °C/min in nitrogen with a load frequency of 1 Hz in membrane tension geometry. The thermal stability of the PI film and composite PI film were recorded with thermo gravimetric analysis (TGA, Netzsch, Selb, Germany), which was performed on a TG 209 F1 Libra at a heating rate of 20 °C/min in a nitrogen atmosphere (40 mL/min). The values of onset, 5% weight loss temperatures ($T_{5\%}$) and residue at 750 °C (Rw_{750}) were obtained from the TGA curves.

3. Results and Discussion

3.1. Transmittance Analysis

The expansion of the working band of polymer film materials from the visible region to the infrared region is conducive to the development of lightweight optical systems in the infrared field. Traditional infrared optical lenses are usually made from crystal materials, infrared glass and so on. These materials are heavy, brittle and costly, while lightweight polymer film materials solve the appeal problems. Transmittance studies were conducted to estimate the effectiveness of the antireflective coating on the PI film. Further, the recorded transmittance spectra were shown in Figure 4. The composite film optic coated with the designed antireflective film showed a higher transmittance in visible (0.5~0.8 μm), short (1.8~2.7 μm) and middle (3.3~5.4 μm) wavelength infrared regions. In the visible region (0.5~0.8 μm), the average transmittance of the composite film improved from 83% to 87% compared to the bare PI film. Similarly, an improvement in the average transmittance of the composite film was observed in the short wavelength IR region (1.8~2.7 μm) from 76% to 86% and the middle wavelength IR region (3.3~5.4 μm) from 73% to 78%, respectively. The average transmittance of the PI film before and after being coated with antireflective film can be seen in Table 1. The results indicate that the PI film optic coated with the designed antireflective film effectively reduced the surface reflectivity of the PI film, which helped to increase the transmittance in Vis, short and middle wavelength IR regions.

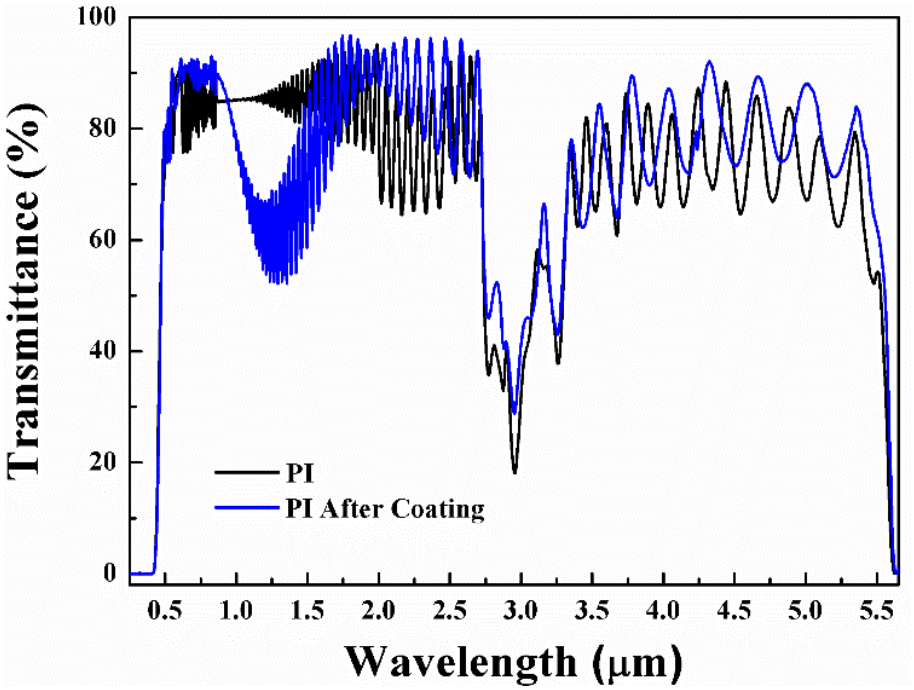


Figure 4. The transmittance spectra of PI film before and after being coated with antireflective film.

Table 1. Transmittance results of PI film before and after being coated with antireflective film.

Sample	Thickness	T_{AV} (0.5~0.8 μm) [a]	T_{AV} (1.8~2.7 μm) [b]	T_{AV} (3.3~5.4 μm) [c]
PI	22 μm	83%	76%	73%
Composite PI	22 μm + 891.61 nm	87%	86%	78%

[a] T_{AV} (0.5~0.8 μm): Average transmittance between 0.5 and 0.8 μm; [b] T_{AV} (1.8~2.7 μm): Average transmittance between 1.8 and 2.7 μm; [c] T_{AV} (3.3~5.4 μm): Average transmittance between 3.3 and 5.4 μm.

3.2. Optical Homogeneity Analysis

The optical homogeneity of a film is strongly dependent on the thickness uniformity and surface roughness. It is a quite important prerequisite for high precision optical films being used as optical lens and windows. The uniformity of the composite film thickness will directly affect the engineering application of film components. In order to evaluate whether the coating preparation process influences the overall thickness uniformity, the optical inhomogeneity of ϕ50 mm PI film before and after being coated with the antireflective film was characterized using a Zygo laser interferometer with an accuracy of λ/1000 (λ = 632.8 nm, λ is the measurement wavelength). The wave front error was tested as shown in Figure 5. The bare PI film (ϕ50 mm) acquired good optical homogeneity with a wave-front error value of 0.006 λ in RMS (root mean square) and 0.034 λ in PV (peak to valley), whereas PI film coated with antireflective film exhibited an enhanced optical homogeneity with a value of 0.009 λ in RMS and 0.052 λ in PV. Additionally, the antireflective film improved the average surface roughness (Ra) of the PI film from 1.686 to 0.768 nm, as shown in Figure 6 and Table 2. In summary, the results indicate that the ion-beam-assisted reactive sputtering method can successfully avoid the deformation of the PI substrate and successfully maintain the optical uniformity of the original PI film.

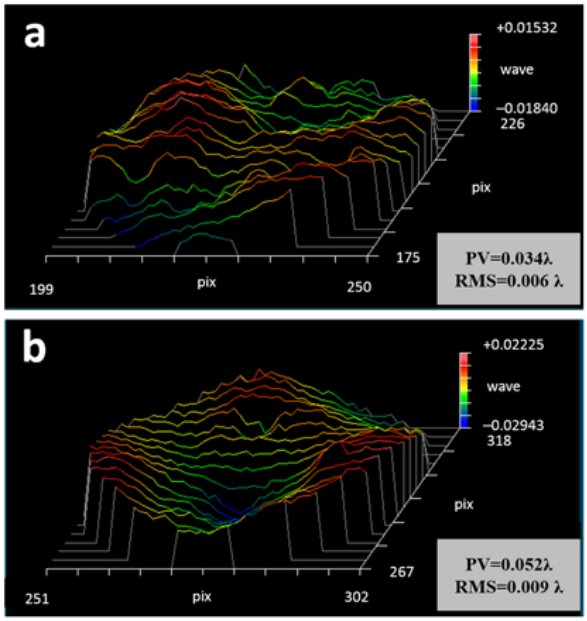


Figure 5. The wave-front error of ϕ50 mm PI film (**a**) before and (**b**) after being coated with antireflective film.

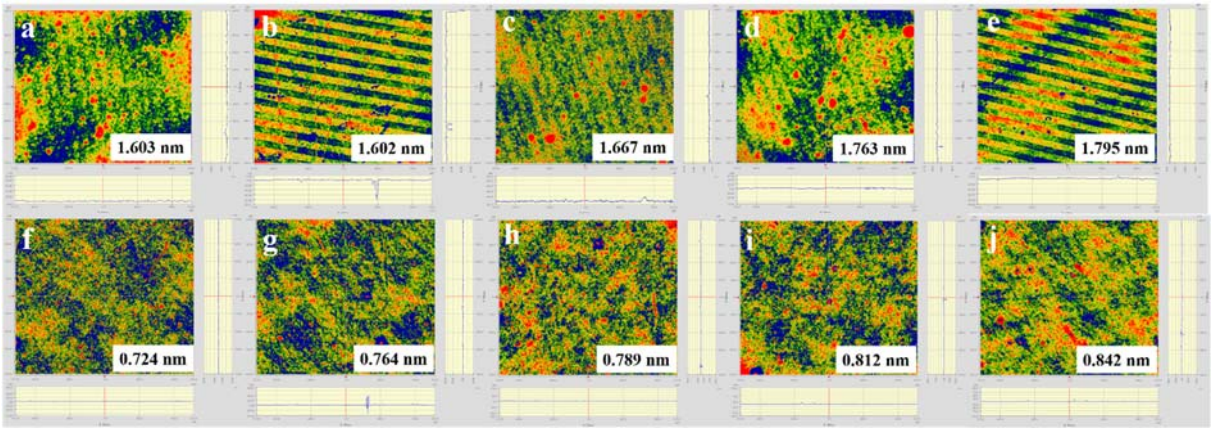


Figure 6. The surface roughness of PI film (**a**–**e**) before and (**f**–**j**) after being coated with antireflective film.

Table 2. Average surface roughness results of PI film before and after being coated with antireflective film.

Sample	Surface Roughness (Ra, nm)	Average Roughness (nm)	σ (nm)
PI	1.603/1.602/1.667/1.763/1.795	1.686	0.080
Composite PI	0.724/0.764/0.789/0.812/0.842	0.786	0.040

3.3. Water Absorption

The water absorption of the PI film will critically affect its morphology and optical properties, which is a crucial aspect to be considered when using it for application as an infrared optical lens and window material. Therefore, a good design of antireflective film material is essential, which can effectively act as water barrier. In this study, to understand the hydrophobic behavior of the PI film and composite PI film, the water absorption rate testing was evaluated. The results are shown in Tables 3 and 4. PI film after being coated

with antireflective film showed a lower water absorption rate, which was 2.9% compared with the bare PI film (3.2%). The results showed that the antireflective film can act as an effective water barrier on the PI film to reduce water absorption.

Table 3. The water absorption rate of PI film before being coated with antireflective film (the calculation formula refers to Formula (1)).

PI	Before Soak (W_0)	After Soak (W_A)	Water Absorption Rate (%)
#1	0.0992 g	0.1021 g	3.2%
#2	0.1090 g	0.1126 g	3.3%
#3	0.1282 g	0.1327 g	3.5%
Average water absorption rate			3.3%

Table 4. The water absorption rate of PI film after being coated with antireflective film (the calculation formula refers to Formula (1)).

PI after Coating	Before Soak (W_0)	After Soak (W_A)	Water Absorption Rate (%)
#1	0.1093 g	0.1130 g	2.9%
#2	0.1066 g	0.1094 g	2.6%
#3	0.1076 g	0.1111 g	3.3%
Average water absorption rate			2.9%

3.4. Thermal Stability Analysis

The thermal stability of PI is one of its most important advantages as a polymer material. The proper understanding of the thermal behavior of the composite film is essential in order to utilize it for better applications in different environments. Studies such as dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) were performed in this research to evaluate the glass transition temperature and thermal degradation behavior of PI film and composite PI film. As shown in Figure 7, the Tg value of composite PI dropped by 5.8 °C, bringing the Tg value of composite PI from 366.9 °C to 361.1 °C. Additionally, there were no significant changes noted for tan δ with the addition of the surface antireflective film. From the results of TGA (as shown in Figure 8 and Table 5), the decomposition temperatures of PI film and composite PI film at $T_{5\%}$ in nitrogen were 553 °C and 547 °C, respectively. The carbon yield decreased by a value of 3%, bringing down the value from 59% to 56% at 750 °C. The results indicate that the coating process can change slightly, and affect the thermal resistant property of polyimide. However, overall, a good inherent thermal stability of the PI film was maintained after being coated with the antireflective film.

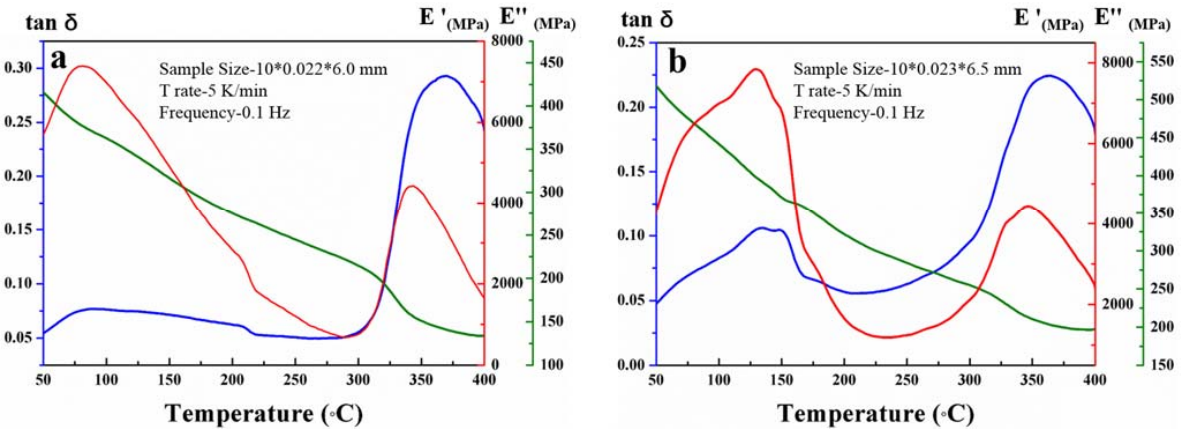


Figure 7. DMA results of (**a**) PI film and (**b**) composite PI film.

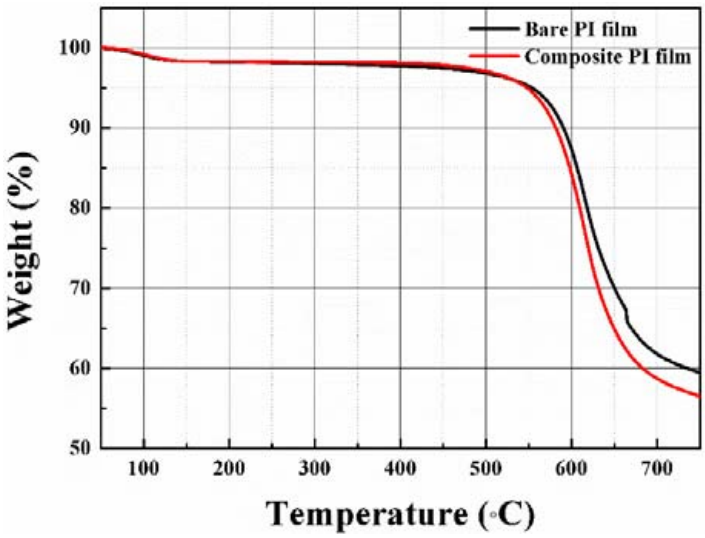


Figure 8. TGA curves of bare PI film and composite PI film.

Table 5. The water absorption rate of PI film before and after being coated with antireflective film.

Sample	[a] T_g	[b] $T_{5\%}$	[c] $R_{W\ 750}$
PI	366.9 °C	553 °C	59% wt.
Composite PI	361.1 °C	547 °C	56% wt.

[a] T_g: Glass transition temperature; [b] $T_{5\%}$: Temperature at 5% weight loss; [c] Rw_{750}: Residual weight ratio at 750 °C in nitrogen.

4. Conclusions

A composite PI film optic with an antireflective film (Ta_2O_5/SiO_2) on the surface was prepared using the ion-beam-assisted reactive sputtering method. The composite PI film optic with antireflective film showed a high transmittance in visible (87%, 0.5~0.8 μm), short (86%, 1.8~2.7 μm) and middle (78%, 3.3~5.4 μm) wavelength IR regions, and relatively low water absorption (2.9%). Further, it also showed a great optical homogeneity (Φ50 mm, RMS = 0.009λ, λ = 632.8 nm), smooth surface (Ra = 0.786 nm) and excellent thermal stability.

With the increasing popularity of the advanced infrared optical system, the aperture of infrared optical components is increasing and the requirement for manufacturing accuracy is becoming higher. A large aperture with good processability can be easily realized by composite PI films compared with crystalline materials or infrared glass materials. With all the exhibited features, such as high transmittance, good optical homogeneity, water resistance and thermal stability, the composite PI film optic is an excellent candidate for application in lightweight IR optical systems.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/coatings13050811/s1, Figure S1: The designed spectra; Table S1: Information of the antireflective coatings.

Author Contributions: Conceptualization, J.Y.; Methodology, B.F.; Resources, J.B.; Data Curation, J.Y.; Writing—Original Draft Preparation, J.Y.; Writing—Review and Editing, J.Y.; Supervision, J.D.; Project Administration, H.Y.; Funding Acquisition, J.B. All authors have read and agreed to the published version of the manuscript.

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