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The study of high-efficiency four-layer photon-to-heat conversion film based on Ti-MgF_2 cermet

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Abstract: The optical constants for the absorbing layer material, satisfying high efficiency of the photon-to-heat conversion of a four-layer structure from ultraviolet to near-infrared wavelength range, are theoretically studied. By employing the effective medium approximation (EMA) model, the measured optical properties of the composite materials (cermet) show in great agreement with that of the simulated ones. Furthermore, a four-layer film structure with high photon-to-heat conversion efficiency using Ti-MgF_2 cermet as the absorption layer is proposed to have a high absorptance of about 95.1% achieved in the wavelength range of 300~1 600 nm. The research results provide a new method to realize high-efficiency photon-to-heat conversion devices and show excellent application prospects.

Key words: photon-to-heat conversion film, optical constants, the effective medium approximation model, Ti-MgF_2 cermet

基于Ti-MgF_2金属陶瓷的高效率四层光热转换薄膜的研究

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摘要:理论上研究了吸收层材料的光学常数,该常数满足从紫外线到近红外波长范围的四层结构的光子-热转换的高效率。通过使用有效介质近似(EMA)模型,复合材料(金属陶瓷)的光学性能与模拟材料的光学性能非常吻合。此外,提出了使用Ti-MgF_2金属陶瓷作为吸收层的具有高光子-热转换效率的四层膜结构,其在300~1 600 nm的波长范围内具有约95.1%的高吸收率。研究结果为实现高效率的光热转换器件提供了新途径,显示了优良的应用前景。

关 键 词:光热转换薄膜;光学常数;有效介质近似模型;Ti-MgF_2金属陶瓷

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Introduction

As one kind of sustainable clean energies, solar energy has been a research hot spot for decades. There are mainly two approaches for utilizing solar energy: (1) photon-to-electric conversion, which converts solar energy into electrical energy, and (2) photon-to-heat conversion, which converts solar energy into thermal energy. The photon-to-heat conversion has been significantly studied in recent years due to its full spectrum absorption matching to that of the solar radiation. In addition, the device tends to be simpler in structure, convenient and relatively low cost in production.

Up to date, the solar selective absorption film can be categorized into five distinct types, mainly: a) Intrinsic absorbing coatings. b) Semiconductor-metal tandem coatings[1]. c) Textured surface coatings[2-3]. d) Multilayer interference stacks[4-5]. e) Cermet composite coatings[6]. The preparation methods of selective absorption film can be divided into physical and chemical methods, including: Physical vapor deposition (PVD)[7-9] and chemical vapor deposition (CVD)[10-11].

The multilayer interference structure is a typical selective absorption film device consisting at least of a dielectric layer and a metal absorption layer with high absorptance, low thermal emittance and good stability at high temperature. Therefore, the multilayer interference stacks have been concerned widely in recent years. As is known, the solar radiation spectrum covers the band 300~2 500 nm. In theory, the photon-to-heat conversion film needs to have high absorptance in this band. However, due to the specific optical properties of the material, no photon-to-heat conversion film with such properties has been prepared yet. The absorption characteristics of the photon-to-heat conversion film with high absorptance shows the absorptance is significantly reduced in the band greater than 1 600 nm, and the solar spectrum radiation intensity distribution diagram shows that the solar radiation energy is more concentrated in the 300~1 600 nm band. Therefore, this paper focuses on the absorption characteristics of the photon-to-heat conversion film in the 300~1 600 nm band.

In 2012, a four-layer medal/dielectric film structure using Cr as the absorption layer was proposed and tested[12], which achieved photon-to-heat conversion efficiency higher than 95% in the 400~1 200 nm wavelength range. Subsequently, the six-layer film structure with absorption higher than 95.5% in the 250~1 200 nm wavelength range was realized in experiment[13]. In order to improve the absorption performance, the film structure with more numbers of absorption layers was suggested and tested. As the number of absorption layers increases, however, the difficulty in preparation of the device also increases. In this work, therefore, a new four-layer film structure is designed and fabricated to improve the absorption performance in the 300~1 600 nm wavelength range. Due to the intrinsic optical properties of absorbing materials limited in nature, the study of composite materials with specific optical constants of the absorption layer will be particularly required.

In this work, the ideal optical constants of the material working as the absorbing layer were theoretically simulated. Among many methods to analyze the optical constants, the effective medium approximation theory (EMA) was applied. Then, the optical constants of some common metal alloys were simulated with the result to show that the refractive index n and extinction coefficient k will have similar spectral dispersion features as that of metal and medium, respectively. Consequently, the optical constants of cermet materials consisting of the metal were calculated, especially for Ti-MgF_2 cermet to have the optical constants very close to the ideal ones. Therefore, we proposed and prepared a new four-layer film structure using Ti-MgF_2 cermet as the absorption layer, resulting in higher photon-to-heat conversion efficiency in the 300~1 600 nm wavelength range. The photon-to-heat conversion film has certain practical significance for the advantages of simple preparation, low price and high absorptance.

In this article, a new method for the design of multilayer film structure is proposed. The optical constant of the ideal absorption layer material is obtained firstly, then the material satisfying this optical property is searched, finally the high absorptance photon-to-heat conversion film is designed. This method has the guiding significance for the research of high absorptance photon-to-heat conversion film. When searching for the material of the absorption layer, it is no longer necessary to try randomly, but to search purposefully, which greatly improves the efficiency and accuracy.

1 Optimal optical constants

It is well known that the solar radiation is mainly distributed in the wavelength range of 250~2 500 nm, concentrated most in the visible range of 380~780 nm. Therefore, the photon-to-heat conversion of the film device is required to have the feature of high solar absorptance in the visible and near infrared region. The typical four-layer film structure used for photon-to-heat conversion is composed from the surface top to the substrate as that: (1) an optional antireflection layer utilizing a transparent dielectric material to enhance solar absorption; (2) a thin solar absorbing transition metal layer, designed with a thickness to achieve the effective internal absorptance of solar radiation; (3) a transparent dielectric layer with the proper optical constant and thickness matching to the phase and amplitude of solar light propagating in the film; (4) a metal reflecting layer, usually using a copper or aluminum layer deposited on the silicon or K9 glass substrate to reduce significantly the IR emittance of the device[12]. Figure 1 shows the structure diagram of a typical four-layer-based photon-to-heat conversion film structure.

In order to prepare a photon-to-heat conversion film with high absorptance in the broad wavelength range, the software (Film Wizard) was used to simulate the multilayer film structure with the optical feature of high absorptance in the design based on the transfer matrix method, which converts the Maxwell equation into an effec-

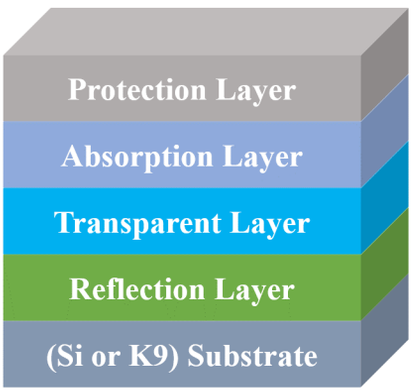

Fig. 1 Schematic diagram of the solar selective absorption film with a typical four-layer film structure
图1 典型的四层膜结构的太阳选择性吸收膜的示意图

tive way to solve the electric and magnetic fields of the light wave propagated at the interface of two adjacent layers. The simulated results will correctly show the optical characteristics of multilayered film structure as designed in advance.

The metal absorption layer plays the key role in the typical four-layer photon-to-heat conversion film. However, the four-layer photon-to-heat conversion film prepared with various natural metals as the absorbing layer could not satisfy the requirement with a high absorptance in the wide wavelength range. Therefore, a new material used as the absorption layer of the multilayer films is further studied, so that the absorption performance of the films can be significantly improved while maintaining the original number of four-layer film structure. In data simulation, the absorbing layer is regarded as unknown material. Its refractive index n and extinction coefficient k are set variable, so that they can be adjusted and changed in the specified wavelength range as needed. A reasonable dispersion model needs to be established in order to make the optical constants of this unknown material satisfy the KRAMERS-KRONIG relationship. The refractive index n and extinction coefficient k can be expressed according to the formula:

$$\varepsilon_1 = n^2 - k^2 \quad , \quad (1)$$

$$\varepsilon_2 = 2nk \quad . \quad (2)$$

The calculated ε_1 and ε_2 required to satisfy the formula:

$$\varepsilon_1(\omega) = 1 + \frac{2}{\pi} P \int_0^{\infty} \frac{\omega' \varepsilon_2(\omega')}{\omega'^2 - \omega^2} d\omega' \quad , \quad (3)$$

$$\varepsilon_2(\omega) = -\frac{2\omega}{\pi} P \int_0^{\infty} \frac{\varepsilon_1(\omega') - 1}{\omega'^2 - \omega^2} d\omega' \quad , \quad (4)$$

where

$$P\int_0^{\infty} d\omega' \equiv lim_{\delta \to 0}\left(\int_0^{\omega-\delta} d\omega' + \int_{\omega+\delta}^{\infty} d\omega'\right) \quad . \quad (5)$$

The Lorentz oscillator dispersion model is based on the theory of the damped oscillator approximation, which is suitable for insulators and semiconductors. The Drude dispersion model theory is based on the free electron gas approximation theory and is applicable to metals in the intraband transition region. The Lorentz-Drude model then is a generalization of the above two models. It not only describes the effect of free carriers on the dielectric function, but also reflects the effect of lattice scattering. In the Film Wizard software, the description of the Lorentz- Drude dispersion model is given by the following formula:

$$\varepsilon = \varepsilon_{\infty}\left(1 + \sum_{j=1}^{m} \frac{A_j^2}{E_{center}^{\ 2} - E(E - i\nu)} - \frac{\omega_p^2}{E(E + i\nu)}\right) . (6)$$

Then the metal absorbing layer in the original four-layer film structure is replaced by the new material layer with the optical feature presented by Lorentz-Drude model. Optimized in data simulation, the absorptance of the new four-layer film structure in the 300~1 600 nm wavelength range reaches nearly 100%. The absorption layer material is represented by X, the new four-layer film structure is [SiO_2 (14.41 nm) / X (132.76 nm) / SiO_2 (10.21 nm) / Cu (> 100 nm)]. The refractive index n and extinction coefficient k spectra of material X are shown in Fig. 2.

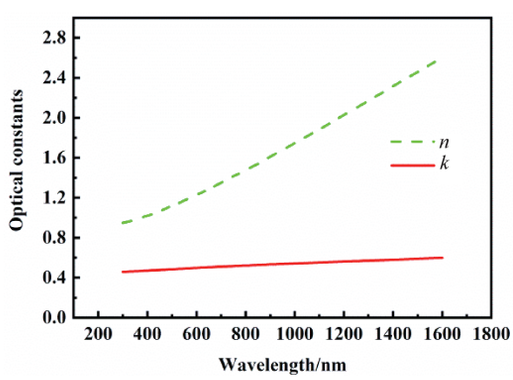

Fig. 2 The refractive index n and extinction coefficient k spectra of the simulated material X as the absorption layer
图2 作为吸收层的模拟材料X的折射率n和消光系数k的曲线图

Due to the intrinsic optical properties of the absorbing materials limited in nature, it will be hard to directly seek the metallic material with similar optical constants applied for the material X in simulation. The new approach, therefore, will be required in the study to look for a composite material with similar optical characteristics as mentioned above.

2 Effective medium approximation model

As for the composite material of the absorption layer, there are mainly two types of methods to make the new material: (1) metal-metal alloy; (2) metal-dielectric composition. In order to obtain the optical constants of composite materials, the effective medium approximation (EMA) model was applied, which can produce the optical feature of a composite nano-ceramic film with nano-metal particles dispersed in a ceramic (dielectric) matrix. The general formula describing the EMA model is expressed as:

$$\frac{\varepsilon - \varepsilon_h}{\varepsilon + Y\omega_h} = \sum_{i=1}^{m} f_i \frac{\varepsilon_i - \varepsilon_h}{\varepsilon_i + Y\varepsilon_h} \quad . \quad (7)$$

In the formula, ε_i and f_i represent the dielectric function and volume percentage of the material, respectively, ε and ε_h represent the effective permittivity of the composite system and the dielectric function of the dielectric matrix, and m represents the number of materials used for composite in the EMA model. Y is related to the depolarization factor.

There are two practical approaches commonly used in the EMA model: (1) Maxwell-Garnett theory; and (2) Bruggeman theory. Maxwell-Garnett theory is applicable to the situation in which the host material is completely surrounded by other materials, such as cermet materials or coated by the spherical microstructures, described by the formula as:

$$\frac{\varepsilon - \varepsilon_b}{\varepsilon + Y\varepsilon_b} = f_a \frac{\varepsilon_a - \varepsilon_b}{\varepsilon_a + Y\varepsilon_b} \quad . \quad (8)$$

The Bruggeman theoretical model is suitable for the conditions of arbitrary polymerization of material, presented by the most composite film materials with the formula described as:

$$f_a \frac{\varepsilon_a - \varepsilon}{\varepsilon_a + Y\varepsilon} + f_b \frac{\varepsilon_b - \varepsilon}{\varepsilon_b + Y\varepsilon} = 0 \quad . \quad (9)$$

In the two formulas shown above, a and b present the embedded and matrix material, respectively.

The Maxwell-Garnett and Bruggeman EMA models can be used to deal with the diffuse and aggregate microstructure as for the real and ideal situations as shown in Figs. 3 and 4, respectively.

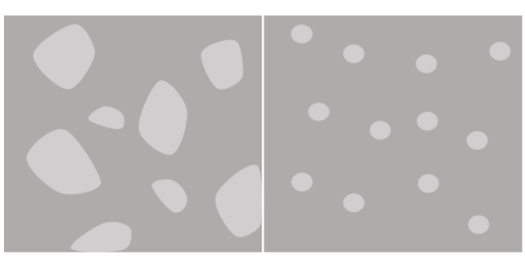

Fig. 3 Maxwell-Garnett's diffuse microstructures (a) real microstructure, (b) ideal situation
图3 麦克斯韦-加纳特色散模型的弥散微结构 (a)真实的微结构,(b)理想情况

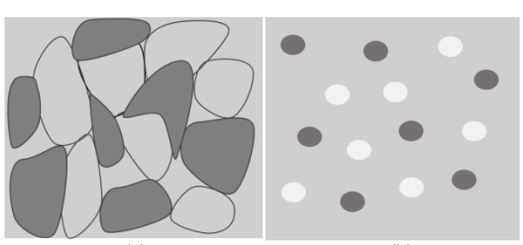

Fig. 4 Bruggeman's aggregate microstructures (a) real microstructure, (b) ideal situation
图4 布鲁格曼色散模型的聚集微观结构 (a)真实的微观结构,(b)理想情况

The Bruggeman approach usually is more applicable for the study of the optical constants of composite materials. The metal materials like Ti, W and Pt will have better absorption performance to be composited with the dielectric materials like SiO_2 and MgF_2 in the study. The Film Wizard software was used to optimize the thickness of each film layer and the ratio of the two materials. After optimization, the absorption spectra of the multilayer films in the wavelength range of 300~1 600 nm are obtained as shown in Fig. 5.

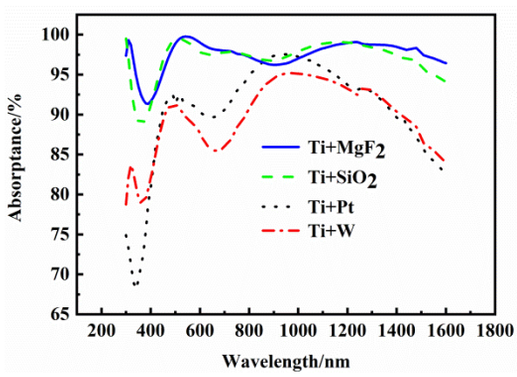

Fig. 5 Absorption spectra of the four-layer structure with the absorption layer consisting of metal-metal alloy and metal-dielectric cermet, respectively
图5 吸收层材料分别是由金属-金属合金和金属-介电金属陶瓷组成的四层膜系结构的光热转换薄膜的吸收光谱

According to the data simulation, the average absorptance of the four-layer film structures with the absorption layers composed by Ti-W, Ti-Pt, Ti-SiO_2 and Ti-MgF_2 are 89.85%, 90.54%, 97.08% and 97.49%, respectively, in the wavelength region of 300-1 600 nm. By comprehensively analyzing the absorption spectra including the average value of absorptance of the film structure, it can be seen clearly that the metal-dielectric cermet is more suitable as the material of the absorbing layer in the thin film structure than the metal-metal alloy. Especially, the Ti-MgF_2 cermet performs a high absorptance as an absorption layer of the multilayer films in the 300~1 600 nm wavelength range. Therefore, a new type of four-layer film structure using Ti-MgF_2 cermet as the absorption layer will be prepared and analyzed in the work.

3 Experimental details and result analysis

Both of Ti and MgF_2 were fabricated on a Si (100) substrate at 4.5×10^{-6} Torr in an electron beam assisted sputtering system (INFOVION, Seoul, South Korea). Ti and MgF_2 were deposited by the direct current (DC) sputtering and radio frequency (RF) sputtering methods, respectively. The growth pressure was fixed at 2×10^{-3} Torr by a throttle valve with the argon (Ar) gas flow rate of 20 sccm (standard cubic centimeter per minute) [14]. The sputtering power for MgF_2 was fixed at 150 W, while the sputtering power for Ti was set at 40 W, 60 W, 80 W

and 100 W, respectively, to vary the concentration of Ti in the composite thin films.

The relative proportion of the element in the composite materials were measured by a Shimadzu / KRATOS X-ray photoelectron spectrometer. The proportion of each element in the Ti-MgF_2 nanocomposite under different deposition power for Ti is estimated by Energy Dispersive Spectrometer (EDS) analysis. The results are listed in Table 1. In terms of the atomic ratio, it can be seen that the metal Ti is partial- chemically activated in the cermet, instead of the pure metal embedded in the medium MgF_2.

Table 1 Proportion of individual elements in Ti–MgF_2 composites prepared under different sputtering power conditions

表1 在不同溅射功率条件下制备的Ti–MgF2复合材料中各个元素所占的比例。

Sample	Deposition power of Ti (W)	Atomic ratio		
		Ti	Mg	F
1	40	0. 13	0. 24	0. 94
2	60	0. 18	0. 18	0. 94
3	80	0. 22	0. 14	0. 94
4	100	0. 29	0. 12	0. 94

An angle-variable spectroscopic ellipsometer (J. A. Woollam VASE) was used to characterize the optical constants of the composite thin films. The ellipsometric parameters (Ψ, Δ) were acquired over a wavelength range of 300~1 100 nm at three different incident angles: 65°, 70° and 75°.

In data fitting, a proper theoretical dispersion model should be used to obtain the optical constants of the nanocomposite films. As mentioned above, the EMA model was suitable to depict the dispersive function of the materials composed by two or more media in different phases with the assumption that the optical properties of each individual medium are not influenced by the other[15]. In this work, however, Ti may be partial-chemically activated in the Ti-MgF_2 nanocomposites, resulting indifferent optical properties and depending on the film fabrication conditions in the experiment. To simplify the data fitting, a modified Lorentz-Drude dispersion model was chosen to fit the experimental data.

A three-phase model, consisting of air/Ti-MgF_2 composite layer/Si substrate, was applied in the data-fitting procedure. For the Ti-MgF_2 layer, the modified Lorentz-Drude model with three oscillators was chosen. The quality of fitting was verified by minimizing the difference between the measured and fitted data, defined by the root mean square error (RMSE):

$$RMSE = \sqrt{\frac{1}{2N-M}\sum_{i=1}^{N}\left[\left(\frac{\Psi_i^{mod}-\Psi_i^{exp}}{\sigma_{\Psi,i}^{exp}}\right)^2+\left(\frac{\Delta_i^{mod}-\Delta_i^{exp}}{\sigma_{\Delta,i}^{exp}}\right)^2\right]}, \quad (10)$$

where N is the number of the experimental data points, M is the number of parameters, σ is the standard deviation. The superscripts *mod* and *exp* refer to the modeled and measured data, respectively.

The optical constants of the composites were characterized by ellipsometry. Since the EDS measurement confirmed the interaction between Ti and MgF_2, the optical constants was obtained by modifying the ellipsometric parameters associated with the Lorentz-Drude model. The measured and fitted ellipsometric parameters at the incident angle of 65° are presented in Fig. 6. The fitting results of Ti-MgF_2 composites with different components are in good agreement with the experimentally measured data showing the RMSE factor of about 0. 14, which proves the reliability of the data fitting.

The optical constants by fitting of the ellipsometric parameters based on the Lorentz-Drude model is shown in Fig. 7. The refractive index n and the extinction coefficient k can be appropriately adjusted by the ratio of Ti in the MgF_2 matrix. With the increase of Ti concentration in the composite, the values of n and k of the composite increase in the 300~1 600 nm wavelength range, which is mainly due to the increase of the metal Ti content in the nanocomposite.

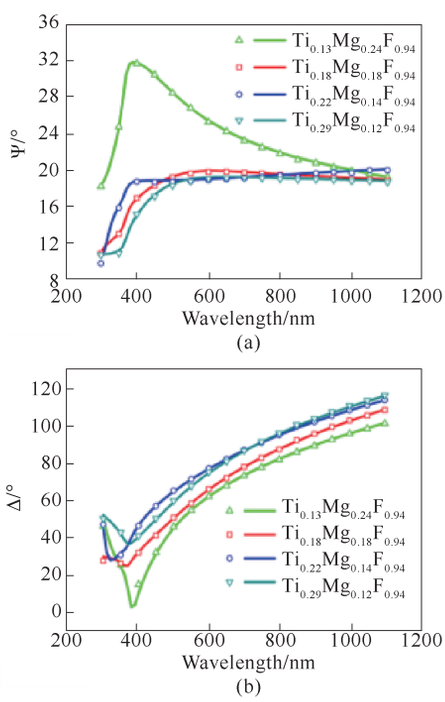

Fig. 6 Measured (symbol) and fitted (line) ellipsometric parameter Ψ and Δ, (a) and (b), of the composite films at the incident angle of 65°

图6 在入射角为65°时测量(符号)和拟合(线)的不同比例的Ti-MgF2复合材料的椭偏参数Ψ和Δ,(a)和(b)

4 Multilayer structures

After data fitting, the optical constants of Ti-MgF_2 cermet with different components were obtained and compared with the ideal optical constants. It can be seen that the optical constants of the nanocomposite thin film with Ti sputtering power of 80 W is close more to that of the

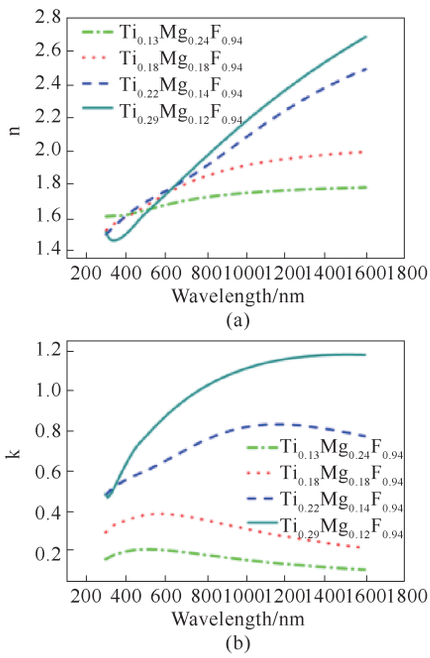

Fig. 7 The refractive index n and extinction coefficient k, (a) and (b), of the Ti-MgF_2 nanocomposite thin films at different concentrations of Ti
图7 不同掺杂浓度的Ti-MgF2纳米复合薄膜的折射率n和消光系数k(a)和(b)

ideal ones, so that the nanocomposite thin film of Ti-MgF_2 cermet was used as the absorption layer in the new four-layer film structure. In addition, The optical constants of SiO_2 and Cu was obtained from Handbook of Optical Constants of Solids.

The design and simulation of the new multilayer film structure are performed by using the Film Wizard software based on the transmission matrix method. Exception of the silicon substrate on the bottom, the structure of the new four-layer film from the bottom to top is: (1) infrared emissivity reduction of the thick Cu layer; (2) optical interference of the dielectric and transparent SiO_2 layer; (3) the absorption of the Ti-MgF_2 cermet layer; (4) protection and antireflection of the SiO_2 layer.

In this article, the Film wizard software based on the transfer matrix method is used to simulate the design of the multilayer film, and the thickness of each layer determines the absorptance of the photon-to-heat conversion film. In the data simulation process, the thickness of the layers is set as the optimal variable. The optimized thickness of the layers obtained for the new four-layer film structure is that: SiO_2 (57.9 nm)/Ti-MgF_2 (113.7 nm)/SiO_2 (12.1 nm)/Cu (>100 nm). By comparing with the film structure using pure Ti as the absorption layer, the simulated values of absorptance are shown in Fig. 8. The average absorptance of the new four-layer structure by using the Ti-MgF_2 cermet as the absorption layer reaches to about 97.1% that is higher than 89.9% of the four-layer structure with the pure Ti metal layer and is close to 98.2% of the six-layer structure.

After a new four-layer film structure with high photon-to-heat conversion efficiency was designed by simulation, the new film device was fabricated by a magnetron sputtering coating instrument. The reflectivity R of the new four-layer film is measured by Shimadzu UV-Vis NIR Spectrophotometer UV-3600. Since the transmittance T of the new four-layer films with a very thick metal layer in the structure is nearly zero, the absorbance A of the new four-layer film can be calculated by A=1-R. The measured and simulated absorptance of the new film device is shown in Fig. 9 with an average absorptance of 95.1%. The SEM image of the multilayer films cross section is shown in Fig. 10. The thickness of each layer is about SiO_2 (68.4 nm)/Ti-MgF_2 (123.6 nm)/SiO_2 (16.3 nm)/Cu (294.1 nm).

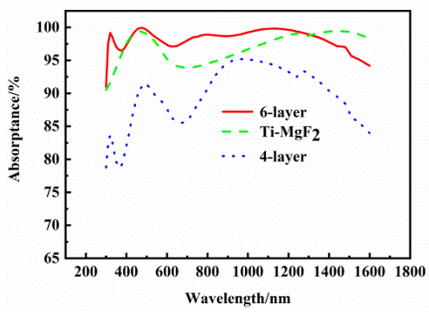

Fig. 8 Absorptance of new four-layer photon-to-heat conversion film compared to that of previous four-layer and six-layer film structures using Ti as the absorption layer
图8 新设计的四层膜系结构光热转换膜与以往使用Ti作为吸收层的四层和六层膜结构吸收率曲线图

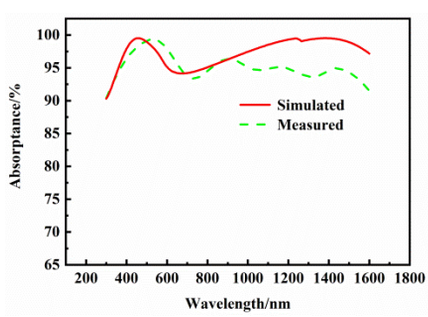

Fig. 9 Measured and simulated absorptance of the new four-layer film structure with high photon-to-heat conversion efficiency
图9 具有较高光热转换效率的新型四层薄膜结构的光热转换薄膜吸收率测量和模拟曲线

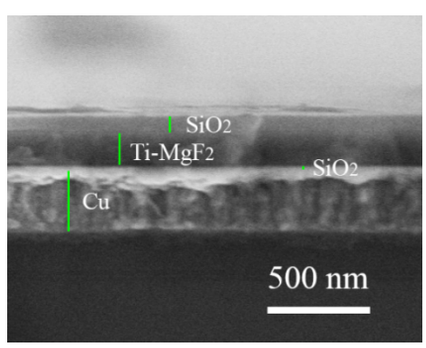

Fig. 10 The SEM image of the new four-layer film structure
图10 新型四层薄膜结构的光热转换薄膜的SEM图像。

5 Conclusion

In this work, the ideal optical constants of the absorbing layer material of the four-layer film structure with higher photon-to-heat conversion efficiency were studied and obtained. By exploring the optical constants of composite materials based on the EMA model, the optical constants of the Ti-MgF_2 cermet can have the spectral dispersion feature close to that of the ideal ones. Therefore, the Ti-MgF_2 cermet was chosen to act as the absorption layer material prepared under the optimal experimental conditions, typically the Ti-MgF_2 cermet was deposited with Ti sputtered at the power of 80 W. The optimal structure of the new film device from top to bottom deposited on the Si substrate is: SiO_2 (57.9 nm)/Ti-MgF_2 (113.7 nm)/SiO_2 (12.1 nm)/Cu (>100 nm). The measured experimental data show that the average absorptance of 95.1% in the wavelength range of 300~1 600 nm is achieved for the of the new multilayer film device, which has higher photon-to-heat conversion efficiency with the simpler film structure to be put into practical fabrication and application in the future.

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