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TiO_2/disordered mesoporous carbon photothermal composites with gradient refractive index structure

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Overcoming low utilization of solar energy, enhancing light absorption, and exploring efficient photothermal materials are current research priorities. In this paper, Developing efficient photothermal materials with silicon lithium glass matrix, introducing gradient refractive index structure into carbon materials, and doping TiO_2 to enhances light absorption. The resulting silicon lithium gradient refractive index TiO_2/disordered mesoporous carbon exhibits superior photothermal properties, particularly under 980 nm laser, its temperature is significantly increased by 6.5 ^{o}C compared to undoped lithium silicon mesoporous carbon. The composite material has good photothermal properties, and has great development prospects in the photothermal field.

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Keywords: Light absorption intensity, Photothermal conversion material, Gradient refractive index structure, Photothermal performance

1. Introduction

Photothermal conversion is the most direct and simple way to use solar energy technology, which has attracted close attention of many researchers and has become one of the research focuses of solar energy technology [1,2]. Solar thermal conversion material was an significant material, which directly converted solar energy into heat in the process of using solar energy [3-5]. At present, the research on photothermal conversion materials had been relatively mature. Common photothermal conversion materials contained metal based materials [6-8], carbon based materials [9-11], semiconductor materials [12-14], organic polymer materials [15-17] and composite photothermal materials [18-20]. However, the bottleneck of the development of photothermal conversion technology has also been exposed. Among them, the biggest problem is that photothermal materials can not effectively use solar energy to achieve the ideal requirements in the process of photothermal conversion. Therefore, it is very important to develop low cost and high efficiency photothermal materials.

Some researchers used the method of preparing specific structure photothermal conversion materials to improve the efficiency of photothermal conversion. Zhu prepared a black TiO_2 photothermal material, which had a special nano cage structure to improve the water evaporation rate of the material. The experimental results showed that the water evaporation efficiency of the black TiO_2 photothermal material can reach 70.9% [21]. Zhang prepared many Cu_7S_4 photothermal films with different shapes, which improved the energy conversion efficiency to 77.1% [22]. The former was that the material prepared had a special internal structure, and the latter had a special external shape structure, both of which improved the photothermal conversion efficiency of the material to a certain extent. Some researchers also used the method of preparing specific photothermal composites to improve the efficiency of photothermal conversion. Yi reported a hollow black TiAlOx composite with a solar light absorption of up to 90.2% [23]. Lan prepared an amorphous oxidation (MnO_x) nanocomposite based on anatase TiO_2 nano sheets, which could accelerate the photothermal catalytic reaction [24]. Although they improved the light heat conversion efficiency to a certain extent, there was still much room to improve the light heat conversion efficiency.

In this paper, TiO_2/disordered mesoporous carbon photothermal composites with gradient refractive index structure were successfully prepared by screen printing and low-temperature sintering. The introduction of gradient refractive index structure to reduce the reflection of the inner surface of the material and improve the transmittance of photons, thus greatly enhancing the light absorption performance of the photothermal material and effectively improving the photothermal performance of the composite [25,26]. The disordered mesoporous carbon is a kind of carbon based photothermal materials. The introduction of TiO_2 can effectively improve the light absorption performance and the photothermal conversion effect of the photothermal composites [27-30].

2. Experimental details

The chemicals to be prepared in the preparation of silicon lithium base glass mainly include silicon dioxide SiO_2 (analytical pure, 99%), lithium trioxide Li_2CO_3 (analytical pure, 99%), zinc oxide ZnO (analytical pure), titanium dioxide TiO_2 (chemical pure, 98%), tungsten trioxide WO_3 (analytical pure, 99%), anhydrous ethanol, terpineol $C_{10}H_{18}O$ (analytical pure, 99%), diethylene glycol butyl ether $C_8H_{18}O_3$ (99%) and ethyl cellulose (analytical pure). These reagents are purchased from official websites such as the Aladdin Reagent Network, the Sinopharm and the McLean Reagent Network. All drugs can be directly used without further purification. The deionized water prepared is also purified by the Milli-Q water purification system.

2.1. Preparation of silicon lithium matrix glass

In this paper, (66-x)SiO_2-10Li_2O-24ZnO-2TiO_2-xWO_3 is used as the matrix glass, and x represents the mole fraction of WO_3. The refractive indexes of x can be adjusted to 0, 3 and 6 to correspond to 1.572, 1.596 and 1.62 respectively, and they can be labeled as SLW1, SLW2 and SLW3 respectively. Calculate according to the composition design of the silicon lithium base glass, then use an electronic balance to weigh the amount required for each component, mix them together, and grind them until all the powders are completely mixed evenly. Then use the melting technology to pour the evenly mixed powder into the crucible and place it in a high-temperature lifting furnace. First, raise the temperature to 300 °C and keep it for half a time, so as to prevent the raw materials from expanding and overflowing due to the gas generated during the decomposition of Li_2CO_3 when heating the powder. Then it is raised to 850 °C and kept warm. Then it is calcined at about 1500 °C for two hours, and the temperature is lowered to 1400 °C to continue the reaction. Finally, the glass melt is poured into the preheated mold by pouring and pressing, and then it is annealed to obtain silicon lithium matrix glass. The x value is changed. The silicon lithium matrix glass with different refractive index can be prepared by repeating the above process.

2.2. Preparation of silicon lithium graded index glass

The preparation of gradient index glass needs to be divided into three steps: first, the configuration of gradient index silicon lithium glass slurry needs to be completed by dissolving the ground glass powder in the organic adhesive at a solid to liquid ratio of 5:4 and fully stirring, and the gradient index glass powder is obtained in the upper layer. Weigh the terpineol ($C_{10}H_{18}O$), diethylene glycol butyl ether ($C_8H_{18}O_3$) and ethyl cellulose according to the mass ratio of 48:48:4, and mix them in a heat collecting constant temperature heating magnetic stirrer and stir them at 80 °C for more than 1 hour, so that they can be fully dissolved to obtain the required organic adhesive. Secondly, gradient index glass films were prepared on high-precision quartz plates by screen printing technology. After printing according to the refractive index from small to large, place the glass substrate on a hot plate with a temperature of 150 °C and dry it for 20 minutes. Finally, silicon lithium gradient index glass was prepared by low temperature sintering technology. Use the special high temperature box furnace for air medium to test the sintered glass at 500 °C, 545 °C, 590 °C and 635 °C respectively. Finally, the best sintering temperature for preparing lithium silicate glass in this experiment is determined according to the contrast results of the transmissivity of these glasses.

The difficulty in the final low-temperature sintering is how to determine the optimal sintering temperature of the silicon lithium glass. In order to solve this problem, the first step is to sinter at 500 °C. It is found that the sintered glass is slightly turbid. After the permeability of the glass is measured in the spectrometer, it is determined that the silicon lithium glass is difficult to sinter into an ideal glass state at 500 °C. So the sintering temperature is increased, and the sintered glass is tested at 545 °C, 590 °C and 635 °C respectively. Finally, the best sintering temperature for preparing silicon lithium glass in this experiment is determined according to the contrast results of the transmissivity of these glasses. Fig. 1 intuitively shows the transmissivity of the glass measured after sintering at these four different temperatures. It can be found that the sintering temperature of 500 °C can't reach a good transparent state at all, and the transparency is far lower than other sintering temperatures. The glass sintered at 590 °C has the best transparency, and its transmissivity is more than 60%. It might be because at this temperature, compared to other temperatures, glass exhibits the best crystallinity. Light encounters fewer interfaces within the glass, thereby reducing light scattering losses and improving transparency. Therefore, the best sintering temperature for low-temperature sintering in the process of preparing silicon lithium gradient index structure glass can be determined as 590 °C.

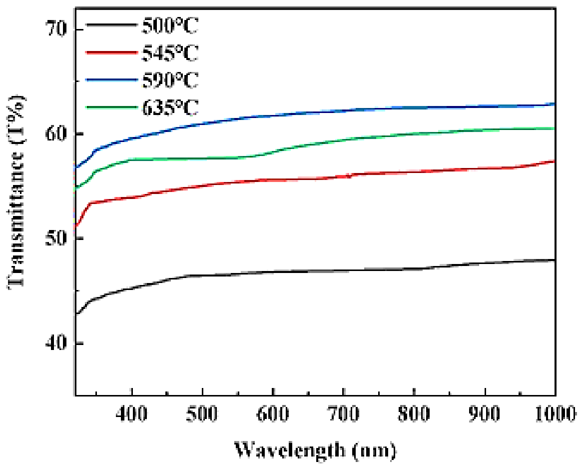


Fig. 1. Change of transmissivity of silicon lithium glass at different sintering temperatures (color online)

2.3. Preparation of gradient refractive index TiO_2/disordered mesoporous carbon glass films in silicon lithium system

The preparation process of lithium silicon gradient index TiO_2/disordered mesoporous carbon glass film can be mainly divided into two parts: preparation of lithium silicon gradient index glass and TiO_2/disordered mesoporous carbon materials. The preparation method of the former has been mentioned in the previous section. It only needs to coat the TiO_2/disordered mesoporous carbon material on the silicon lithium gradient refractive index glass to complete the preparation. Therefore, the following is how to prepare TiO_2/disordered mesoporous carbon photothermal composites.

First, prepare the chemicals needed in the preparation process. The disordered mesoporous carbon provided by the ChemWise and the titanium dioxide TiO_2 deionized water provided by Sinopharm have been purified by Milli-Q water purification system. Weigh an appropriate amount of disordered mesoporous carbon and TiO_2 on the electronic balance in a ratio of three to two. Pour the doped powder into the prepared organic binder for ultrasonic treatment for more than half an hour so that they can be mixed and dissolved. Then place the beaker containing TiO_2/disordered mesoporous carbon slurry in a collector type constant temperature heating magnetic stirrer and heat it in a water bath at 80 ℃ until the slurry is uniformly mixed. Finally, the prepared TiO_2/disordered mesoporous carbon paste is coated on the other side of the lithium silicon gradient index glass, that is, the glass substrate with the lowest refractive index, and dried on the hot plate until it is solidified on the glass to form a film, which indicates that the lithium silicon gradient index TiO_2/disordered mesoporous carbon glass film is successfully prepared.

3. Results

3.1. Absorption characteristics

The absorption spectrum of the prepared silicon lithium system gradient refractive index TiO_2/disordered mesoporous carbon glass film was tested with the spectrometer, and the absorption test results are shown in Fig. 2. From the figure, it can be found that the gradient refractive index TiO_2/disordered mesoporous carbon material of the silicon lithium system has excellent absorption performance, and the overall trend of its absorption curve is to decrease, but at last there is a temporary small increase. Meanwhile, the strange peaks and kinks originate from the absorption peak of the glass material itself. According to the absorption curve of the sample in the 370 nm - 1000 nm wave band, it can be found that the absorption performance of the sample is the best in the visible light section, especially there are two absorption peaks, and the overall absorption performance is significantly higher than that in the near-infrared wavelength. Compared with the absorption at 808 nm and 980 nm, it can be found that the absorption at the latter wavelength is better, which means that when the laser with 980 nm and 808 nm is used to irradiate the sample, the former will absorb more photons than the latter, and the prepared lithium silicon system gradient refractive index TiO_2/disordered mesoporous carbon glass film, as a photothermal material, can convert the absorbed light energy into heat energy, This shows that the heat released by the sample after photothermal conversion under 980 nm laser is more than that under 808 nm laser.

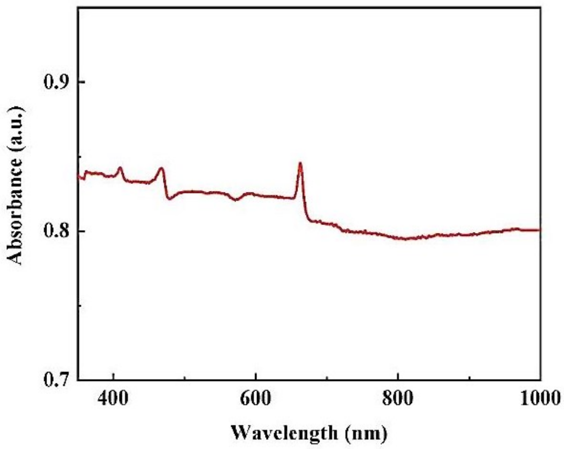


Fig. 2. Absorption spectrum of gradient refractive index TiO_2/disordered mesoporous carbon glass film of silicon lithium system

3.2. Photothermal characteristics

In order to test the photothermal properties of the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the silicon lithium system under white light, the temperature change of the sample with time under white light irradiation was measured experimentally. The experimental results show that the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the silicon lithium system can raise the initial temperature of water at 24.50 °C to 48.43 °C after 900 seconds of white light irradiation, which is a sufficient increase of 23.93 °C, indicating that the sample has certain photothermal properties. However, this is lower than the ideal temperature. With the introduction of TiO_2 and the combination of silicon lithium matrix glass, the absorption performance of the sample is significantly enhanced. The photo thermal performance of the sample should be increased to above 50 °C according to the ideal state. In order to distinguish the factors affecting the photothermal performance of the sample, the temperature of pure water was tested under white light. As shown in Fig. 3, the temperature changes of the sample and pure water under white light irradiation can be compared intuitively. It is found in the figure that the pure water irradiated by white light only increases by 4.95 °C. Removing the factor that white light power makes the water temperature rise, it can be seen that the sample actually increases by 18.98 °C, which conforms to the ideal temperature rise state. Based on the influence of white light irradiation on the photothermal performance of the sample, combined with the analysis of the absorption performance of the sample at 808 nm and 980nm wavelengths, it is concluded that the photothermal performance of the sample is closely related to the wavelength and laser efficiency. Therefore, the influence of laser wavelength and laser power on the photothermal performance of the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the lithium silicon system will be studied in detail.

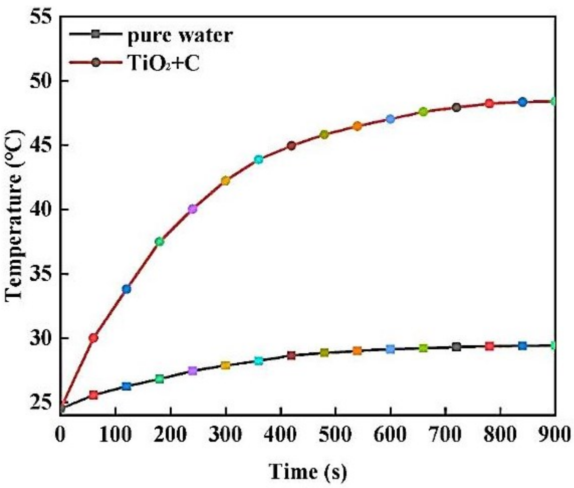
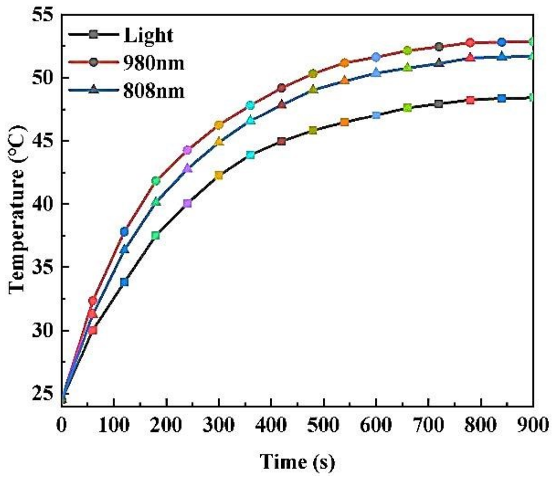


Fig. 3. Temperature change of pure water and silicon lithium gradient index TiO_2/disordered mesoporous carbon glass film under white light (color online)

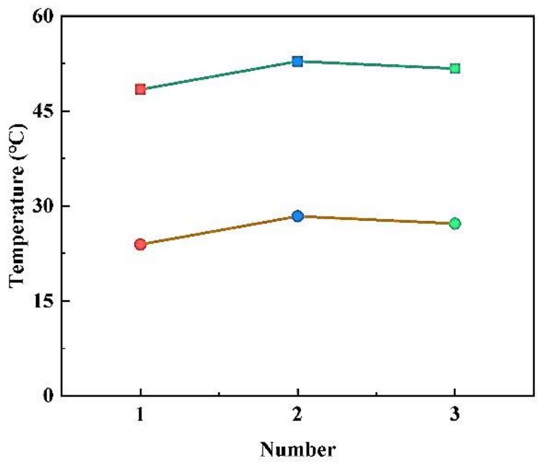
4. Disscussions

4.1. Effect of laser wavelength on photothermal properties of samples

According to the absorption spectra of the samples, it can be found that the absorption properties of the samples are different at different wavelengths. In order to further study the relationship between the laser wavelength and the photothermal performance of the sample, under the same laser power, different wavelength lasers are used as laser sources to irradiate the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the silicon lithium system, test the temperature change of the sample, and compare and analyze the photothermal performance of the sample under white light, as shown in Fig. 4. Fig. 4 (a) shows the temperature rise of the sample after 900 s of exothermic irradiation under white light, 980 nm laser and 808 nm laser respectively. It is obviously found that the temperature rises fastest when the laser with the wavelength of 980 nm is used for irradiation, and the water temperature can rise to 52.87 °C at most when the heat is released. The temperature rise of the sample under 808 nm irradiation followed closely, and the difference between the two temperatures was only 1.16 °C. The temperature rise of the sample under white light irradiation is obviously not as high as that of other wavelength lasers, but the difference is slightly larger because there are more influencing factors that the white light intensity is lower than that of other nano wavelength lasers and the loss of light in the transmission process. Their specific temperature difference changes are shown in Fig. 4 (b), which can visually observe the change trend of the photothermal properties of the samples under three different light sources, first increasing and then decreasing. This shows that the photothermal properties of the gradient refractive index TiO_2/disordered mesoporous carbon glass films of the silicon lithium system are also different under different wavelengths and different light sources, and the laser wavelength can affect the photothermal effect of the photothermal conversion materials to a certain extent.



(a)

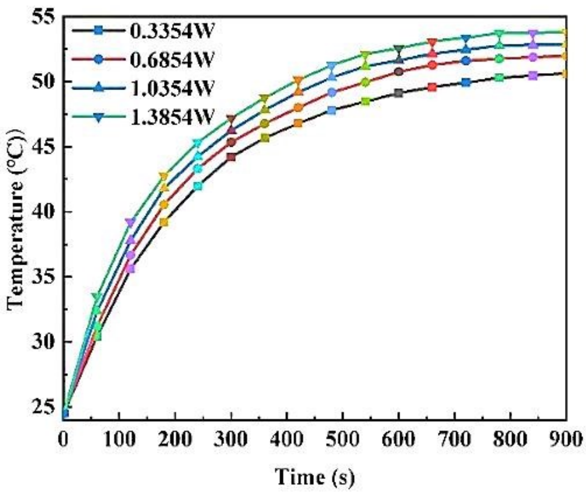


(b)

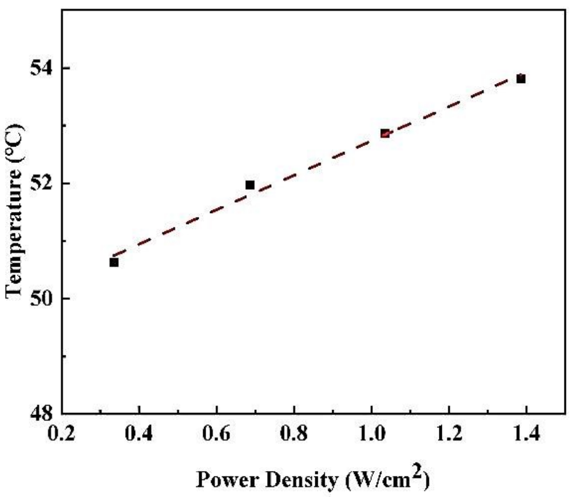
Fig. 4. (a) Temperature rise curve of sample under white light, 980nm laser and 808nm laser irradiation, (b) Temperature difference change under three different light sources (color online)

4.2. Effect of laser power on photothermal properties of samples

In addition to the light source with different laser wavelength will affect the photothermal performance of the sample, the laser power will also affect the temperature change of the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the silicon lithium system. The higher the laser power is, the more heat will be released from the sample under this power laser. In order to discuss the influence of laser power on the photothermal performance of the sample, the photothermal performance of the sample is tested by changing the laser power of 980 nm laser to 0.3354 W, 0.6854 W, 1.0354 W and 1.3854 W respectively.



(a)



(b)

Fig. 5. (a) Temperature change of sample irradiated for 900s under different laser power, (b) Linear fitting of laser power density and temperature (color online)

Fig. 5 (a) shows the photothermal test results of the samples under 980nm lasers with four different laser powers. According to the comparison of the temperature change of the results, it can be found that each time the laser power is increased, the temperature of the water temperature changes will increase. When the laser power is increased to 1.3854 W, the temperature of the sample will increase to the highest, which can rise to nearly 55 ^{o}C. This phenomenon proves that the photothermal performance of the sample is proportional to the laser power. With the increase of the laser power, the photothermal effect of the sample will also increase. Fig. 5 (b) can be obtained by fitting the temperature rise changes of samples under different laser powers. According to the fitting results, the laser power is not only proportional to the temperature, but also the linear relationship between them is Y=49.75X+3.18, indicating that the temperature of samples under this laser power can be increased by about 3.18 ^{o}C with each unit of power density (W/cm^2).

4.3. Effect of changing TiO_2 doping mode on the photothermal properties of samples

TiO_2 has the ability to improve the light absorption capacity in the ultraviolet region, and can promote the photothermal conversion. Doping TiO_2 can improve the photothermal performance of the silicon lithium system gradient index disorder mesoporous carbon glass film. Therefore, this paper studies and prepares the silicon lithium system gradient index TiO_2/disorder mesoporous carbon glass film, and compares the photothermal performance of the silicon lithium system gradient index disorder mesoporous carbon glass film with and without TiO_2 doping through experiments. Figure 6 compares the temperature rise of the two samples under white light. The results show that the temperature rise of the sample doped with TiO_2 is higher than that of the sample without TiO_2, but the improvement effect is not very ideal, only 2.55 ^{o}C. This is because TiO_2 possesses the characteristic of high refractive index ($\geq$2.55). Combining it with a lithium silicate substrate glass creates a composite photothermal absorbing material with a gradient refractive index interlayer structure, which is advantageous for photothermal absorption performance. Therefore, it indicates that the introduction of TiO_2 can indeed improve the photothermal performance of the disordered silica lithium gradient index mesoporous carbon glass film, but the improvement of the photothermal effect of the sample is still lacking. In order to effectively improve the photothermal properties of the gradient refractive index TiO_2/disordered mesoporous carbon glass film of the silicon lithium system, the following experiments were carried out from the perspective of changing the TiO_2 doping mode.

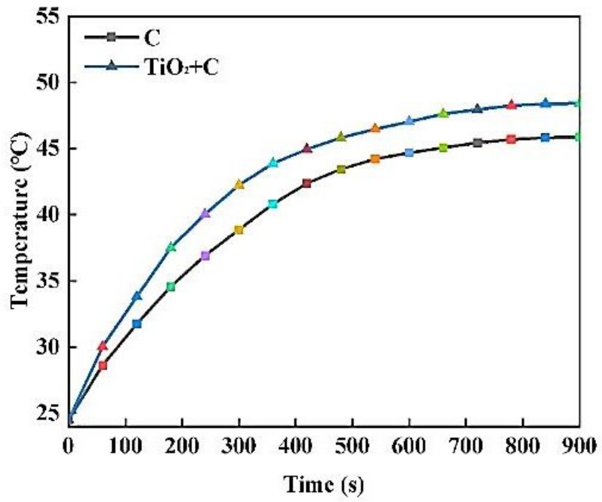


Fig. 6. Temperature change of TiO_2 samples with and without doping under white light irradiation (color online)

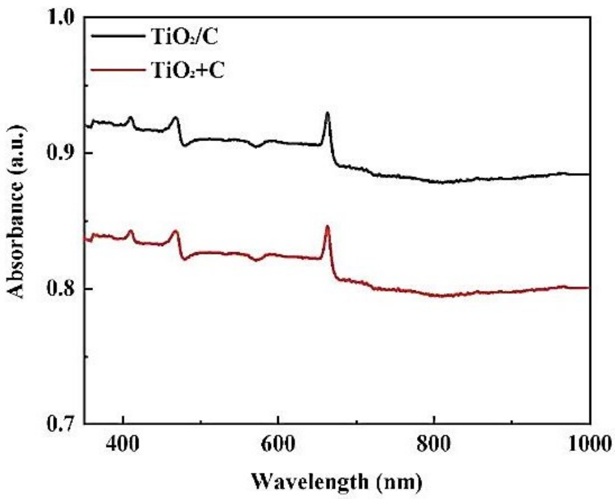
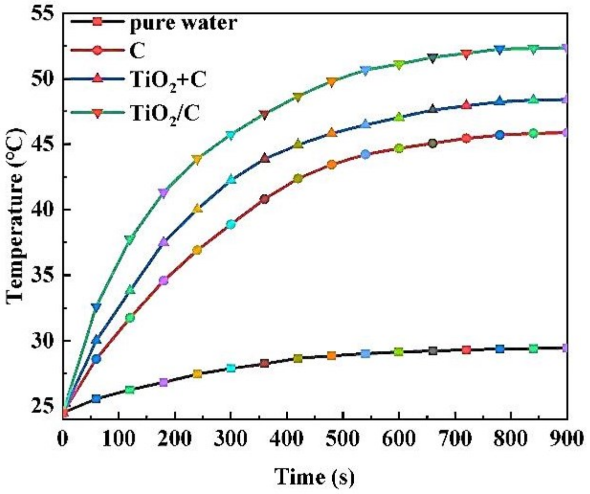
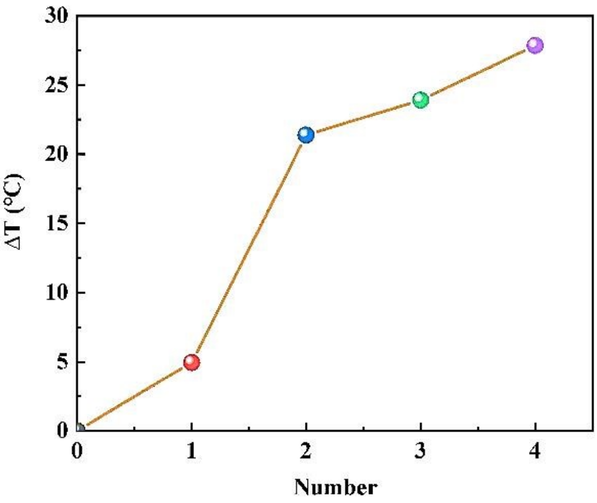


Fig. 7. Comparison of absorbance of samples after optimizing doping methods (color online)

The silicon lithium system gradient refractive index TiO_2/disordered mesoporous carbon glass film prepared in this paper is a layer of silicon lithium system gradient refractive index glass after TiO_2 and disordered mesoporous carbon are proportionally doped together. In order to study and test whether TiO_2 doping mode will affect the photothermal performance of the sample, and if so, how it will affect, the preparation method of the photothermal composite material of TiO_2 and disordered mesoporous carbon has been improved. In this experiment, TiO_2 free gradient index disordered mesoporous carbon glass film of silicon lithium system was prepared first, and then the same amount of TiO_2 was weighed according to the previous proportion and mixed evenly with the organic binder before being coated on the silicon lithium system gradient index disordered mesoporous carbon glass film, so that two layers of glass films of different photothermal materials were formed on the silicon lithium system gradient index glass. In order to distinguish the two TiO_2 doping methods, the former is doped together in TiO_2+C, and the latter is overlapped in TiO_2/C. Fig. 7 compares the absorption effect of the samples under the two doping methods. The second TiO_2/C doping method makes the absorption effect of the samples more prominent. The absorbance of the samples doped with TiO_2 under this method is nearly 0.0836 higher than the original, so this confirms that the second doping method is improved on the basis of the first method and can effectively improve the absorption capacity of the samples. It is possible that the composite material formed by TiO_2/C exhibits a higher refractive index compared to TiO_2+C. This enables the formation of a gradient refractive index composite structure more conducive to light absorption. The improvement of absorption characteristics means that the photothermal performance should also be improved. In order to verify this conjecture, the two samples were irradiated under white light for 900s in order to test their photothermal performance. Finally, according to the experimental results, the comparison of their temperature changes was shown in Fig. 8. From the temperature change curves of samples under two different doping methods in Fig. 8 (a), it seems that the second two layer film TiO_2/C doping method is more excellent. When the white light irradiation lasts for 900s, the temperature of the sample can rise to about 52.50 ^{o}C, reflecting the change in the preparation method of lithium silicon gradient refractive index TiO_2/disordered mesoporous carbon glass film, which can greatly improve the temperature rise of the sample, and the heating rate can also be effectively increased. In order to compare the change trend of temperature difference of samples in various ways, Fig. 8 (b) is drawn according to the temperature difference range. It can be seen from the figure that the whole curve shows a trend of continuous growth. Compared with the temperature rise of the original sample, its temperature difference range has increased by nearly 3.95 ^{o}C, and compared with the sample without TiO_2 doping, its temperature has effectively increased by nearly 6.50 ^{o}C. From this, it can be concluded that changing the TiO_2 doping mode has an impact on the photothermal performance of the sample, and the gradient index TiO_2/disordered mesoporous carbon glass film of silicon lithium system prepared by the second optimized two layers of different photothermal material film superposition mode (TiO_2/C) can have better photothermal effect.



(a)



(b)

Fig. 8. Temperature change of (a) sample and (b) temperature difference under different doping conditions after TiO_2 is doped in different ways under white light (color online)

5. Conclusion

In this paper, according to the best gradient refractive index structure (including glass substrate) with refractive index distribution of 1.52, 1.572, 1.596 and 1.62, the silicon lithium system gradient refractive index TiO_2/disordered mesoporous carbon glass films were successfully sintered at the best sintering temperature of 590 ℃. The temperature of the sample irradiated by white light for 900s can reach 23.93 ^oC higher than the initial temperature of water. The effects of various factors on the photothermal properties of the sample, such as laser wavelength, laser power and TiO_2 doping mode, are discussed and analyzed. Among them, light sources with different laser wavelengths have influence on the photothermal properties of the samples. The results show that the temperature of the samples irradiated by 980 nm laser changes the most, and the temperature can rise to 52.87 ^oC. The photothermal performance of the sample is directly proportional to the laser power. Under 980nm laser irradiation, the sample temperature can rise to nearly 55 ^oC at the highest power. According to the fitting curve of power density and temperature, the relationship between the two is Y=49.75X+3.18. Finally, the TiO_2 doping method was optimized. The absorbance of the sample prepared under the second two-layer TiO_2/C superposition method was nearly 0.0836 higher than that of the original sample. The temperature change energy measured under white light was about 52.50 ^oC, which was significantly increased by 6.50 ^oC compared with the disordered mesoporous carbon composites with gradient refractive index of silicon lithium system without TiO_2 doping. However, the sample prepared by the first TiO_2+C doping method only made the sample without TiO_2 3.95 ^oC higher. Therefore, the optimized TiO_2 doping method has a better effect, which can effectively improve the photothermal performance of the samples, and prepare gradient index disordered mesoporous carbon photothermal composites with better photothermal effect.

Acknowledgements

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