Research article

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Ultrathin, broadband, omnidirectional, and polarization-independent infrared absorber using all-dielectric refractory materials

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Abstract: Broadband long-wavelength infrared (LWIR) optical absorbers have important applications in thermal emission and imaging, infrared camouflaging, and waste heat and biothermal energy utilization. However, the practical application of broadband LWIR optical absorbers requires low-cost and facile fabrication of large-area structures with limited thickness. This paper reports the design and fabrication of an ultrathin, broadband, omnidirectional, and polarization-independent LWIR optical absorber composed of anodized aluminum oxide and highly doped Si using the gradient refractive index strategy. The average absorption of the broadband optical absorber is higher than 95% in the 8–15 μm wavelength range, and it has wide incident angle and polarization tolerances. More than 95% of the optical energy in the

wavelength range from 8 to 13 μm was absorbed within a depth of 8 μm , making this absorber the thinnest broadband LWIR dielectric absorber so far. The absorption remained above 90% after annealing at 800 $^{\circ}\text{C}$ in air. The infrared camouflage of the proposed absorber was successfully demonstrated with a human body background. With the advantages of facile fabrication, low-cost materials, restricted absorption thickness, and excellent thermal stability, the developed broadband LWIR optical absorber is very promising for the practical applications mentioned above.

Keywords: broadband optical absorber; infrared camouflage; long-wavelength infrared; refractory materials; ultra-thin absorber.

1 Introduction

The long-wavelength infrared (LWIR) thermal emission range from 8 to 14 μm lies within the transparent atmospheric window. As LWIR emission has attracted tremendous interest, the development of LWIR optical absorbers is of great significance for diverse applications such as infrared thermal imaging [1–4], thermal emission [5–7], chemical and biological sensing [8], infrared camouflaging [9, 10], waste heat utilization [11], and radiative cooling [12].

An ultrathin, low-cost broadband LWIR optical absorber that is easily fabricated is needed for efficient thermal radiation applications. Although numerous studies have been conducted on optical absorbers operating at visible and near-infrared wavelengths, much less research has been performed on broadband optical absorbers in the LWIR region. One strategy for producing broadband LWIR absorbers is multiresonator integration [13–18]. The absorber bandwidth can be broadened by integrating several resonators with different resonance wavelengths. However, ultrabroadband absorbers require more resonators and entail higher fabrication complexity

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and cost. Another strategy for designing broadband optical absorbers is the incident impedance matching strategy, which reduces the optical incident impedance and traps the light in absorbing media. This strategy can be applied either by using ultrasparse media such as carbon nanotubes [19, 20] and porous alumina [21–23], or by adopting a gradient refractive index profile [24–29]. But the absorption thickness needs to be further optimized to reduce material costs and improve energy efficiency. For example, vertically aligned single-walled carbon nanotubes have been demonstrated to improve absorption efficiencies in the LWIR region [19, 20]. Owing to impedance matching between the air and carbon nanotubes, reflection was suppressed and the free carriers in the carbon nanotubes gradually absorbed light. However, because of the low absorption coefficient of the impedance matching material in the LWIR region, the thickness of this ultrasparse-matrix broadband absorber that is required in order to achieve suitably strong absorption enhancement was reported to be up to hundreds of micrometers. Such a thickness increases the material cost and causes problems such as additional energy loss in seawater desalination applications [30]. The absorption thickness can be reduced by optimizing the gradient refractive index profile of the homogenous [25–28] or heterogeneous [24, 29] impedance-matching layer [31] on the absorbing media. Compared with a homogenous structure, using a heterostructure and tuning its refractive index profile is more flexible and reliable. Meanwhile, increasing the absorption coefficient of the absorbing media is also an effective way to reduce the absorption thickness. For the semiconductor absorbing medium, this can be achieved by increasing the doping concentration [32, 33].

In this study, we achieved low-cost fabrication of a large-area, omnidirectional, polarization-independent broadband LWIR optical absorber by combining a gradient refractive index dielectric layer with a highly doped Si substrate. The gradient refractive index layer was fabricated by coating with anodized aluminum oxide and subsequent low-cost dry/wet Si etching. The average absorption of our absorber is higher than 95% in the 8–15 μm wavelength range, and it has wide incident angle and polarization tolerances. More than 95% of the optical energy in the wavelength range from 8 to 13.2 μm was absorbed within a depth of 8 μm , making our absorber the thinnest broadband LWIR dielectric absorber so far. The absorption remained above 90% after annealing at 800 $^{\circ}\text{C}$ in air. Further, the infrared camouflage performance of our absorber under conditions of a human-body background was verified. Owing to its excellent broadband absorption performance, ultrathin absorption thickness, low cost, and facile large-

area fabrication, our broadband LWIR optical absorber is promising for practical applications in thermal emitters and imaging, infrared camouflage, and waste heat and bio-thermal energy utilization.

2 Results and discussion

Figure 1 depicts the structure design and fabrication process of our broadband LWIR optical absorber. The broadband optical absorber consists of three layers on a highly doped *p*-type Si substrate. The top layer is a 400-nm-thick anodic aluminum oxide (AAO) with a honeycomb structure, the middle layer is a ~ 1200 -nm-thick nanoporous Si honeycomb structure (NSH), and the bottom layer is 400-nm-thick nanoporous Si (NSi). There are three steps in the fabrication process (see the Experimental Section for details). First, the purchased 400-nm-thick mesoporous AAO film was transferred from the polymethyl methacrylate substrate to the highly doped Si substrate. Then, the AAO/Si was treated by reactive ion etching (RIE) to form a mesoporous Si honeycomb (SH) layer underneath the AAO, as shown in Figure 2(D). Finally, a nanoporous Si structure was formed in the mesoporous Si layer and the upper region of the highly doped Si substrate by isotropic electrochemical etching (EE). The porosity and thickness of the nanoporous Si were adjusted by the etching current and duration, and the RIE process modified the mesoporous/nanoporous Si layer thickness. By utilizing these processes to optimize the structure thickness, the thinnest broadband LWIR dielectric absorber fabricated thus far was achieved. As shown in Figure 1(D), we compared the absorption wavelength range and maximal absorption wavelength normalized by the absorption thickness for our absorber (black stars) and other reported absorbers. Our absorber provides superior broadband absorption and ultrathin absorption thickness. By controlling the porosity and thickness of the three layers on top of the Si substrate, we realized an impedance matching design with an ultrathin gradient refractive index structure that allows gradual absorption of LWIR light in the substrate.

We characterized the infrared broadband absorption of the device through the transmittance and reflectance measured by Fourier-transform infrared spectroscopy, that is, $A = 1 - T - R$. The measured transmittance is almost zero in the infrared range (2.5–25 μm). The measured absorption for both *s*- and *p*-polarized light is plotted in Figure 1(E) as solid lines. As shown in the inset of Figure 1(E), an average absorption of >95% was achieved from 5 to 18 μm at an incident angle $\leq 50^{\circ}$ for both *s*- and *p*-polarized light. Using effective medium theory and the transfer matrix method

Figure 1: (A–C) Schematic of the fabrication process. (A) Si substrate covered with mesoporous honeycomb AAO layer. (B) AAO/SH/Si structure formed after RIE. (C) AAO/NSH/NSi/Si structure formed after EE. The thicknesses of AAO, NSH, and NSi were 400 nm, 1.2 μm, and 400 nm, respectively. (D) Absorption wavelength range and maximal absorption wavelength normalized by absorption thickness for our absorber and other reported absorbers. (E) Measured (solid lines) and simulated (dashed lines) absorption spectra for the samples for s-polarized (black lines) and p-polarized (red lines) light at an incident angle of 15°. The inset shows the measured average absorption in the 8–15 μm wavelength range as a function of incident angles.

Figure 2: (A) Top view and (B) 45° view SEM images of the AAO/Si structure. (C) Top view and (D) 45° view SEM images of the AAO/NSH/NSi/Si structure. (E) Reflectance spectra of different structures: Planar Si (black line), planar Si with AAO (red line), AAO/SH/Si structure (green line), AAO/NSH/NSi structure (blue line), and NSH/NSi structure (yellow line).

[34], we calculated the reflectance and transmittance of our devices. Figure 1(E) depicts the calculated absorption spectra for s- and p-polarized light as dashed lines. The results agree well with the measured absorption spectra.

The difference between the simulated and measured absorptions at longer wavelengths and larger incident angles may have been caused by the fact that the refractive indices of the materials for the simulations and experiments

differed. The slight differences in absorption between the cases of *s*- and *p*-polarized incidence may be due to the small in-plane anisotropy of the honeycomb structure. The absorption valley near 3.4 μm is caused by a multilayer interference-induced reflectance peak.

We analyzed the function of each layer in the broadband LWIR optical absorber by measuring the reflectance spectra during fabrication and during the sequential removal of layers after fabrication. Figure 2 presents a scanning electron microscopy (SEM) image and related reflectance spectra for each step. First, we measured the reflectance of the *p*-type highly doped Si substrate. Compared to the average reflectance of 35% for conventional Si (resistivity 1–10 $\Omega\text{ cm}$), the average reflectance of the highly doped Si wafer in the infrared range is lower, being approximately 30% (Figure 2(E) black line). The reduced reflectance and improved absorption are due to the increased free carrier concentration in the highly doped Si. After the process of transferring the AAO mesoporous film onto the Si substrate, the reflectance decreases in the wavelength range of 3–8 μm , with a maximum amplitude of 10%, as shown by the red line in Figure 2(E). Considering that the refractive index of alumina is approximately 1.5 throughout the mid-infrared region and given the measured porosity of the AAO film, the effective refractive index of the AAO mesoporous film is calculated to be 1.2 using the effective medium theory of Bruggeman [35]. The reduction in reflectance is negligible in the 8–15 μm wavelength range. This is because the effective refractive index of the AAO mesoporous film is less than 1, in agreement with the reported abnormal dispersion curve of AAO in the multiphonon absorption region [36, 37]. Absorption in the AAO layer is limited because it is ultrathin. Next, we used the AAO template as a mask to etch the underlying Si by RIE. After dry etching, the reflectance of the device (green line in Figure 2(E)) is significantly lower than that before etching (red line) throughout the wavelength range from 3 to 15 μm , with an average decrease of more than 10%. At a wavelength of 5 μm , the reflectance decreases from 25 to 3%. The reduction in reflectance could be due to an improved gradient refractive index structure after dry etching. According to the effective medium theory of Bruggeman, the refractive index of the Si honeycomb structure is between those of the AAO mesoporous structure and Si substrate. However, there is still more than 10% reflectance at wavelengths longer than 8 μm . To further reduce the reflectance to less than 5% in the wavelength range of 5–15 μm , we employed an optimized electrochemical etching process. The formation of the nanoporous Si structure in the mesoporous Si and Si substrate by the electrochemical etching process improved the

multilayer gradient refractive index structures and reduced the reflectance. To demonstrate the importance of all the layers with gradient refractive indices for infrared broadband optical absorbers, we deliberately removed the AAO mesoporous layer by chemical etching and measured the reflectance. The average reflectance increases to more than 25% in the wavelength range of 5–18 μm , as shown by the brown line in Figure 2(E).

The key to realizing a broadband optical absorber based on gradient refractive index media is to optimize the refractive index profile and thickness for each layer. For materials with continuous refractive index changes, the curve of the refractive index change with depth can be described as [31]

$$n(l) = n_{\text{air}} + (n_{\text{Si}} - n_{\text{air}}) \left(\frac{l}{T} \right)^\kappa, \quad (1)$$

where n_{air} and n_{Si} are the refractive indices of the air and Si substrates, respectively; l is the depth; T is the total thickness of the gradient refractive index region; and κ is the parameter that controls the refractive index profile. T is fixed at 2 μm . The refractive index profiles vary with depth. Figure 3(A) presents the real part of the refractive index profile at a wavelength of 10 μm for $\kappa = 1.0$ (blue line), $\kappa = 1.4$ (red line), and $\kappa = 2.0$ (green line). The refractive indices of alumina and Si were measured using an ellipsometer. Figure 3(B) depicts the influence of κ on the absorption spectra. It is evident that an average absorption above 95% at wavelengths of 8–13.5 μm can be achieved when $\kappa = 1.4$. A larger value of κ reduces the absorption in the long-wavelength range, whereas a smaller value of κ reduces the absorption in the 6–8 μm wavelength range.

A broadband optical absorber with a continuous change in the refractive index is usually impractical and unnecessary. Instead, the gradient refractive index structure with a limited number of layers is often used in practical broadband optical absorbers. Our device has three gradient refractive index layers. We finely tuned the porosity and thickness of each layer to modify the overall refractive index profile of the layered structure such that it resembles as much as possible the optimized theoretical profile with $\kappa = 1.4$ (black line in Figure 3(A)). This careful gradient refractive index material design could be the reason for the experimental broadband, omnidirectional, and polarization-independent high absorption performance.

Optical absorption thickness analysis was conducted by performing finite-difference time-domain simulations for both highly doped Si wafers and our broadband LWIR optical absorber, as shown in Figure 4(A) and (B). The

Figure 3: (A) Refractive index as a function of depth for continuously changing materials with different κ (blue, red, and green lines) and the actual profile (black line) of the fabricated broadband absorber. (B) Contour plot of the absorption spectra as a function of κ for the device. The color scale indicates absorption from 75 (blue) to 100% (red).

common aspect of the two devices is that the optical absorption rate increases with depth. However, compared with the maximum absorption rate in highly doped Si of approximately 70%, the maximum absorption rate in the broadband optical absorber at the same depth is much greater, exceeding 97%. This finding demonstrates the beneficial antireflection effect of the incident impedance-matching layer. Figure 4(B) shows that the absorption in the mesoporous AAO layer (thickness 400 nm) is negligible in the LWIR region as the thickness of this layer is small compared with the incident wavelength. The abrupt change in the absorption contour at the wavelength of 11.63 μm , as seen in the measured refractive index of alumina (supplementary material Figure S1(B)), is due to the transition point of alumina, at which it is transformed from a dielectric to an epsilon-near-zero material. In the broadband optical absorber, more than 95% of the optical energy in the wavelength range from 8 to 13 μm is absorbed at a depth of 8 μm , which is smaller than the absorption wavelength. Therefore, the optical absorption performance of Si is improved with an absorption thickness smaller than the optical wavelength, which is very important for practical LWIR applications such as waste heat utilization, flexible photoelectric detection, and conversion processes.

High-temperature tolerance is usually required in practical applications of broadband optical absorbers, such as thermal emitters. As the materials in our broadband optical absorber are refractory, that is, the melting points of alumina and Si are 2054 and 1414 $^{\circ}\text{C}$, respectively, we tested the absorption behavior of our broadband optical

absorber in a high-temperature annealing process. Different annealing temperatures (600, 800, and 1000 $^{\circ}\text{C}$) and gas environments (air and Ar) were tested with an annealing time of half an hour. Figure 5(A) and (B) show the absorption spectra measured after annealing. Compared with the absorption of the sample without annealing (black line), for the sample annealed in air at 800 $^{\circ}\text{C}$, the absorption decreased slightly in the wavelength range of 7–11 μm , with a maximum decrease of 14% at a wavelength of 9 μm (red line in Figure 5(A)). When annealed in air at 1000 $^{\circ}\text{C}$ (yellow line in Figure 5(A)), the absorption further decreased with an average decrease of approximately 15% in the wavelength range of 4–9 μm compared with the absorption of the unannealed sample. The absorption changes for the sample annealed in Ar are much smaller than that for the sample annealed in air at temperatures higher than 800 $^{\circ}\text{C}$. The oblique SEM images of the sample annealed in air also exhibit a noticeable morphology change compared with the sample annealed in Ar, as shown in the insets of Figure 5. Thus, the reduced absorption of our broadband optical absorber could be due to the thermal oxidation of Si [38, 39] and the related thermal expansion effect [40]. As our broadband optical absorber achieves excellent absorption performance after annealing at 600 $^{\circ}\text{C}$ in air, it is promising for applications such as thermal emitters [5–7].

The fabrication process for our broadband optical absorber is facile, low cost, and scalable. The maximum dimensions of the prepared sample were 25 mm $\times$ 25 mm, as shown in the supplementary material. The strategy of using

Figure 4: Simulated absorption spectra of (A) planar Si and (B) the broadband absorber for different distances from the surface.

Figure 5: Room-temperature absorption of the as-prepared absorber (black line) and of the absorber after annealing at 600 (blue line), 800 (red line), and 1000 °C (yellow line) for 30 min under (A) air and (B) Ar atmospheres. The nearly unchanged absorption demonstrates the high-temperature tolerance of the absorber. The inset SEM images show the surface of the device after annealing at 1000 °C in air and Ar.

a gradient refractive index material for a broadband optical absorber is one that is highly adaptable for other substrates. For example, the porosity of the AAO mesoporous film can easily be tuned by choosing suitable pore size and pitch values in the two-step anodization process, and the porosity of the Si nanoporous film can be tuned by the EE process [41, 42]. Thus, the effective refractive index of each layer can be finely tuned to match the incident impedance from air to the absorbing media.

Our broadband LWIR absorber exhibited infrared camouflage under specific background conditions. Unlike metallic materials, the human body has high emissivity in the LWIR region. Therefore, metal products worn by people can easily be found using an LWIR camera. However, the surfaces of metal products can be disguised using our broadband LWIR absorber to achieve infrared camouflage under human-body background conditions. We chose an Ag mirror as the target object to be hidden with our absorber acting as the LWIR camouflage

material. Pictures of the Ag mirror on a human hand with and without being covered by our LWIR absorber were taken using visible and infrared cameras. Figure 6 shows the visual and infrared (wavelength range 8–14 μm) images at room temperature. It can be seen that as the absorption/emissivity of our absorber at 8–14 μm is similar to that of the human body, the absorber is not easily distinguished from the human body using the infrared camera. Hence, the portion of the Ag mirror behind the LWIR absorber is well hidden in the infrared image.

3 Conclusion

We reported a broadband far-infrared optical absorber fabricated by coating a gradient refractive index layer on a highly doped Si substrate. The gradient refractive index profile was optimized for a limited number of layers, and the effective refractive index in each layer was controlled

Figure 6: Infrared camouflage demonstration. (A, B) Photographs of the LWIR absorber on the human hand and Ag mirror, respectively. (C, D) Infrared images corresponding to (A) and (B), respectively, acquired using an infrared camera (detection wavelength: 8–14 μm).

by adjusting the porosity. The average optical absorption of the absorber was found to be more than 95% in the wavelength range of 8–15 μm , and large incident angle and polarization tolerances were observed as well as thin-film absorption and high-temperature tolerance. More than 95% of the optical energy in the wavelength range from 8 to 13 μm was absorbed within a depth of 8 μm , making our absorber the thinnest broadband LWIR dielectric absorber so far. Further, the infrared camouflage performance of our absorber under human-body background conditions was verified. The involved materials are low-cost, and the fabrication process is facile and scalable. The strategy of using a gradient refractive index material as a broadband optical absorber has excellent substrate adaptability potential. Thus, our far-infrared broadband optical absorber is promising for practical applications such as thermal emission and imaging, infrared camouflaging, and waste heat and biothermal energy utilization.

4 Experimental section

4.1 Sample fabrication process

First, the purchased mesoporous alumina film (Top Membranes Technology Co., Ltd., pitch 450 nm, pore diameter 340 nm, thickness 400 nm, area $1.5 \times 1.5 \text{ cm}$) was peeled off from the PMMA substrate and transferred to the silicon substrate by immersion in acetone. The silicon substrate was highly doped *p*-type silicon (Hefei Kejing - Materials Technology Co., Ltd., boron doped, crystal orientation [1 1 1], resistivity: 0.01–0.05 $\Omega \text{ cm}$, doping concentration: $1.3 \times 10^{18} \text{ cm}^{-3}$). Then reactive ion etching (Model: RIE-150A, Beijing Tailong Electronic Technology Co. Ltd.) was performed to etch the Si (Gas: CF_4 , Flow: 300 sccm, power: 200 W, duration: 250 s per cycle, seven cycles). The etching depth is about 1200 nm. Then, the sample was electrochemically etched with an electrolyte mixture of diluted HF (10 wt%) and ethanol (99.9 wt%) in a 1:1 volume ratio by the electrochemical workstation (Model: CS300, Wuhan Keente Instrument Co. Ltd.). The optimized etching condition was 35 mA current with 40 s etching time. The etched sample was washed with deionized water and blown dry with nitrogen. The annealing process was carried out by quartz tube furnace (Lindberg blue *m*) in either air or argon gas. The time of temperature ramping up/down was 1 h.

4.2 Characterization method

Scanning electron microscopy images were observed (Model: FEI Versa 3D, Thermo Fisher Scientific). The reflectance and transmittance spectra were measured by Fourier transform Infrared spectroscopy (Model: Nicolet continuum FT-IR microscope, Thermo Scientific). The wavelength range was from 2.5 to 25 μm . The incident angle was from 15° to 80°. The refractive index of highly doped silicon and alumina in the infrared wavelength was measured by an infrared ellipsometer.

The infrared imaging was taken by Camera Fotric 220s. The value of IR camera radiation was set to 0.95.

4.3 Simulation methods

The absorber was treated as multilayers, and the reflectance and transmittance were calculated by the transfer matrix method. The refractive index for Al_2O_3 and silicon was measured. The effective refractive index for each layer was calculated by Bruggeman's effective medium theory which was decided by their porosity. The porosity of AAO and NSi was calculated by analyzing the filling ratio of the high-resolution SEM image. The absorption thickness analysis was calculated by FDTD simulations (Lumerical Solutions).

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- The measured transmittance and a photograph of the sample; The real and imaginary part of the refractive index of aluminum oxide and silicon; The Backscattered electron imaging and energy-dispersive spectrum of the sample; The cross-section SEM image of the absorber and the SEM image of the absorber after removing the above AAO; The influence of the electrochemical etching current on the reflection of bulk silicon; The effect of the electrochemical etching duration on the reflection of bulk silicon; The effect of the RIE duration on the reflection of the AAO/Si structure; The electric field distributions ($|E|^2$) in the referenced silicon wafer and our silicon absorber; The Reflectance spectra of different regions on the absorber.