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Multi-spectrum bands compatibility: New trends in stealth materials research

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The beginning of the history of stealth technology can trace back to the later period of the Second World War. The prototype of a jet-powered flying wing, Horten Ho 229, flew on March 1, 1944, designed by Walter Horten & Reimar Horten. Reimar Horten visualized the use of charcoal dust to absorb radar electromagnetic waves (EMWs). Despite the immature structure and the change in the war situation, Ho 229 was still a pioneering exploration of stealth materials. After the war, a significant quantity of scientists led by Americans devoted themselves to EMW absorbing theory research. Both of the fundamental theories in the stealth field, the Nicolson, Ross & Weir (NRW) method and the Naval Research Laboratory (NRL) arch method, were proposed in the 20th century [1–3]. With the expeditious development of EMW theories, stealth technologies have been expanded in the late 20th century [4].

The properties of the aircrafts' stealth ability are determined by two factors, EMW absorbing coatings and configuration designs. EMW absorbing coatings are composed of absorbing agents and basal materials [5]. Furthermore, the stealth abilities of EMW absorbing coatings are characterized through electromagnetic parameters, including relative complex permittivity ε_r ($\varepsilon_r = \varepsilon' - j\varepsilon''$) and relative complex permeability μ_r ($\mu_r = \mu' - j\mu''$) [6]. The vector network analyzer (VNA) can calculate both of the above parameters, which is a piece of testing equipment for EMW energy [7]. The original VNA prototype can be dated back to 200A Audio Oscillator produced by HP Development Company, L.P. in 1938. In 1999, Agilent Technologies was formed by HP as a new measurement company. Moreover, in 2014, Keysight Technologies was split from Agilent Technologies. Alongside optimizing EMW measurement equipment, material scientists have devoted themselves to novel EMW absorbing and shielding materials' development. Furthermore, in the last decade, EMW absorbing and shielding materials have gained widespread attention from practitioners specialized in chemistry, materials, and electronic information technology [8]. As-developed stealth materials have a vital application in military usage to ensure stealth aircraft's survival from enemy fire control radar systems. And for civilians, EMW absorbing and shielding materials should also not be neglected. For electronic equipment in the communication area, the clutter needs to be suppressed to fulfill communication stability. Thus, developing the EMW absorbing materials is a valuable research topic.

As mentioned above, the performance of EMW stealth coatings relies on electromagnetic parameters ε_r and μ_r [9]. From the views of materials scientists, microcosmic component regulation and morphology construction have become the most effective ways to optimize the absorption ability in the last decade [10,11]. EMW absorbing materials can be divided into three categories: resistant-type absorbent, dielectric-type absorbent, and magnetic-type absorbent. As for the chemical compositions, the as-mentioned absorbents consist of carbon-based derivatives, metals and alloys, ferrites, Si-C-N-based ceramics, etc. Several single-component absorbents have already been used for commercial productions, like carbonyl iron powder. Nevertheless, single-component absorbents have the features of weak absorption and narrow application wave band, owing to the poor magnetic loss or mismatching ε' with ε'' [12]. Therefore, composites binding the resistant-type to magnetic-type/dielectric-type have been brought into the focus of materials scientists. Carbonic materials and metal oxides, in particular, are the most studied systems [13]. It has been reported that core-shell hybrids of Fe_3O_4 nanocrystals/mesoporous carbon hollow spheres can achieve an ultrawide absorption band of 8.0 GHz with a coating thickness of 2.6 mm [14]. For stealth coating usage, mesoporous or porous carbon component is beneficial to abating the areal density, which has become one of the research hotspots [15,16].

Morphology construction design has been proved to be a practical approach for absorption peak and bandwidth manipulation, such as core-shell, york-shell, multi-dimensions combination, and metal-organic frameworks (MOFs) [20,21]. As shown in Fig. 1a, heterointerface engineering is an essential strategy to optimize electromagnetic parameters [17]. Contributing to the EMW absorption performance, heterointerface construction provides abundant attenuation loci and brings about high conduction loss, dipole polarization, or both [22,23]. Our group has found that by regulating point defects with K atoms in $LaCo_{0.9}Fe_{0.1}O_3$, the dielectric loss capability of perovskites can be enlarged and a wide effective bandwidth of 6.2 GHz can be obtained [24]. Furthermore, through the preparation of different elementary point defects, the radar cross-section (RCS) can be effectively reduced. Along with the lattice defects and other heterointerface engineering technics, constructing specific heterointerface structures is also assured of a vital place in EMW parameters manipulation [25–27]. Taking the core-shell structure as a typical example, our group has reported that the carbon shell is an excellent source of conductive paths [28]. Co@C nanotubes were prepared and verified to be a marvel EMW absorbent. The effective absorption bandwidth can reach over 5.0 GHz at 1.8 mm. While controlling the ratios of EMW absorbent/paraffin wax coatings, the strongest absorption crest value of reflection loss (RL)-thickness (d) curves is −48 dB with 30 wt% absorbent filler. In brief, both

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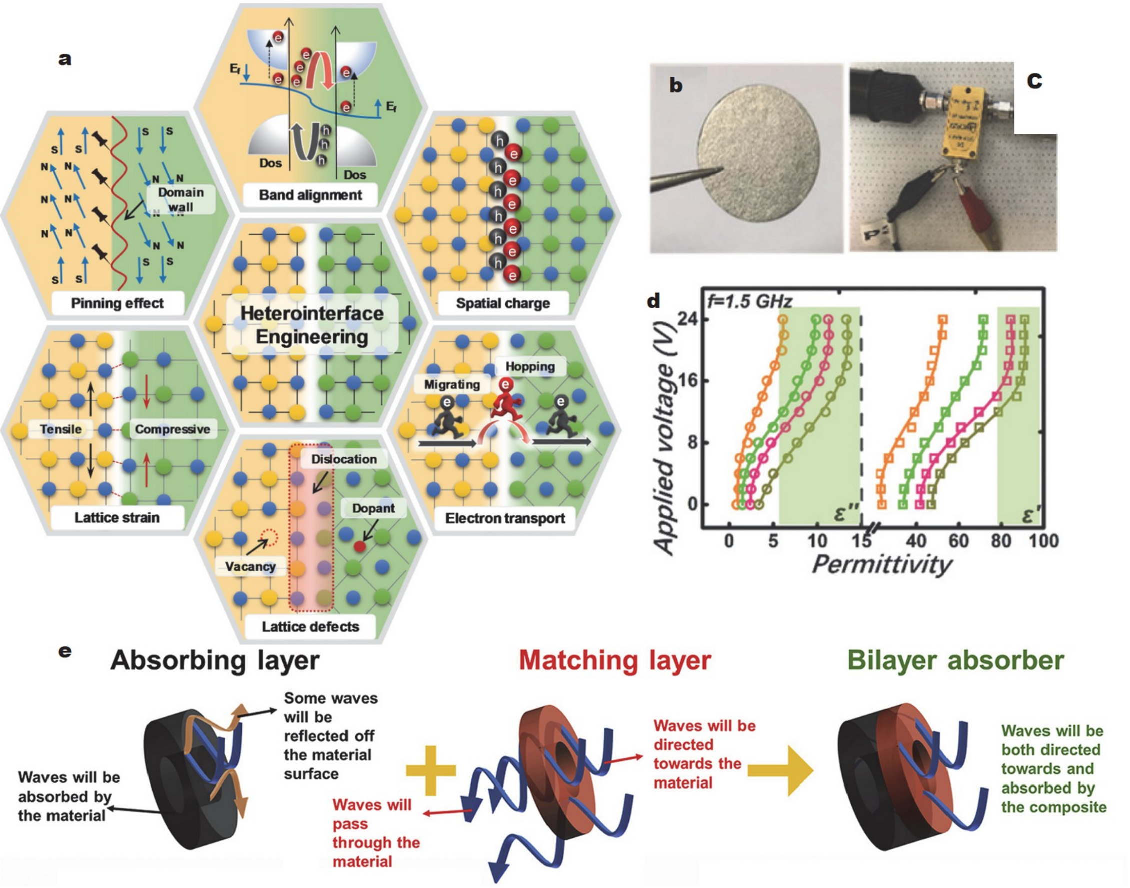

Figure 1 (a) Electromagnetic response mechanism of heterointerface engineering [17]. Copyright 2021, John Wiley and Sons. (b, c) Schematic illustration of the external voltage regulation method. (d) The dielectric parameters of the device with external voltage [18]. Copyright 2018, John Wiley and Sons. (e) Schematic diagram of 3D printing bilayer structure [19]. Copyright 2020, Elsevier.

component regulation and morphology construction design are commonly used productive methods to optimize the RL and normalized input impendence ($Z = Z_{in}/Z_0$), which is vital for incident EMW and crucial for stealth performance. Nevertheless, it cannot be ignored that the as-mentioned micro-regulation methods are limited by the shortcoming of poor low-frequency performance, uncontrollable EMW parameters, low molding and manufacturing efficiency [29].

To better solve the listed problems, researchers turn to external field regulation and additive manufacturing with tremendous enthusiasm [33,34]. In the low-frequency range, ε_r can hardly reach the baseline of the requirement of −10 dB RL value, owing to the restriction equation between the ε_r and testing EMW frequency [35]. Thus, our group combined two effective strategies together to adjust the ε' and ε'', component morphology and external electrical field. The external electric field was applied to the as-prepared device, as seen in Fig. 1b, c, which sensitively changed the ε' and ε'' values with the controllable voltage [18]. The ε' can be amplified up to nearly 100, as shown in Fig. 1d. Relying on the novel strategies, the low-frequency performance of the as-prepared devices can be optimized, with the absorption ratio of more than 85% incident EMW in the frequency range of 1.5–2.0 GHz.

As regards additive manufacturing, Zuo *et al.* [19] applied digital light processing (DLP) three-dimensional (3D)-printing equipment for EMW stealth materials synthesis. The commonly adopted RL and attenuation coefficient (α) basically determine the possibility of EMW penetrating the air-coatings interface and the absorbing ability of coatings. However, the need for a low reflection and a strong absorption raises a contradictive claim against EMW parameters for the optimization of stealth. RL should be as low as possible, leading to the fact that the real and imaginary parts of the above EMW parameters should not be too massive. Meanwhile, the α requests that μ_r should be as large as possible while ε_r is large enough, for the majority of materials' ε_r is larger than μ_r. For the sake of this contrary requirement, Zuo *et al.* [19] designed a multi-layered coating, which consists of a matching layer and an absorbing layer in Fig. 1e. The matching layer consists of graphene/$Li_{0.35}Zn_{0.3}Fe_{2.35}O_4$/polymethyl methacrylate; meantime, the absorbing layer is built with graphene/carbonyl iron powder/polymethyl methacrylate. The input impendence of the upper layer is greatly improved, and the lower layer has an electromagnetic loss ability 2–3 times larger than the upper one. The as-designed bilayer coatings reached the minimum RL value of −46.1 dB and a controllable absorption band range by optimizing the thicknesses of the bilayer. In addition, 3D-printing technology is a highly efficient approach to prepare coatings. Furthermore, the thicknesses can be accurately processed through the DLP mechanism.

The subsequent researchers have focused on external physical field manipulation and 3D-printing technics ever since. Still, even if the bottlenecks of low-frequency stealth performance and swift-assembled technic are both broken through, there are many problems unsolved, for example, how to realize the infrared frequency stealth compatibility and stabilize the integrity of materials at ultra-high temperatures, facing the aeronautic and astronautic demands. In the past five years, massive efforts have been devoted to relative domains.

Any object will transmit infrared radiation over 0 K, which can be a glitch for aircraft being detected by infrared thermal imaging and heat acquisition sensors. Infrared stealth plays another vital role in stealth technology, being distinct from EMW stealth [36], as the latter particularly estimates the wave frequency in 2–18 GHz. There are two basic ways to realize infrared stealth: tunable/low thermal emissivity in 3–5 and 8–14 μm and phase change materials (PCM). From the point of recyclability, tunable/low thermal emissivity material is the most commonly applied method to achieve infrared stealth performance. Besides, because of the background complexity of the target environment, the tunable infrared emissivity property is much more appropriate. In Fig. 2a, b, Sun *et al.* [30] demonstrated a scalable method to prepare a mid-infrared electrochromic film. The component system is based on the multi-walled carbon nanotube (MWCNT) and ionic conductive liquid layer. When applied to the external electrical field, the electrochromic film contrives a well-controlled mid-infrared thermal emissivity of 0.15–0.7. The as-reported film can be recharged cyclically over 3500 times. With a 4-V external voltage, the thermal image remains at 25°C appareled on the human body. Moreover, the application of electrochromic film is broadened for infrared thermal camouflage with 10 × 10 multi-pixel arrays. Other than merely infrared stealthy, with the rapid development of detective technology and multi-spectral scanner, the multi-band responsive stealth materials meet the urgent needs of the market. Scientists have made preliminary attempts in multi-spectrum bands. Yu *et al.* [31] designed a transparent wooden material with Cs_xWO_3 nanoparticles. Visible light transparency is considered other than near-infrared shielding performance. By controlling the content mass ratios of Cs_xWO_3 nanoparticles, a wooden film can be as transparent as up to 72% at 550 nm wavelength, as shown in Fig. 2c. The near-infrared shielding property is verified through a building architecture model and an infrared lamp, as can be seen in the inset of Fig. 2c. Due to the excellent shielding ability in the near-infrared wavelength range, the model house with the 0.05 wt% Cs_xWO_3/transparent wood cools the temperature nearly 15°C down compared with those normal glass-covered ones. But there is a fly in the ointment: the EMW absorption ability has not been achieved.

Analogously, former researchers have also synthesized many materials/coatings, which are stabilized at high temperatures and oxygen atmosphere. Luo *et al.* [32] demonstrated a promising anti-oxidation ceramic with a graphene base. Carbon-based materials are vulnerable under an oxygen atmosphere over 600°C, causing invalid performance. Silicon-boron carbonitride (SiBCN) applied herein plays the role of an inoxidizing protective layer for graphene@Fe_3O_4. Authenticated by the RL-frequency curve data in Fig. 2d, even after oxidation at 600°C, graphene@Fe_3O_4/SiBCN preserves a minimum RL value of −66.21 dB with an effective absorption bandwidth of 3.69 GHz. In addition, the precursor of SiBCN is ultraviolet-curable, which is a promising candidate for 3D printing and swift-assemble production. Having considered that, there is still a huge space to be permeated in environmental resistance and mechanical

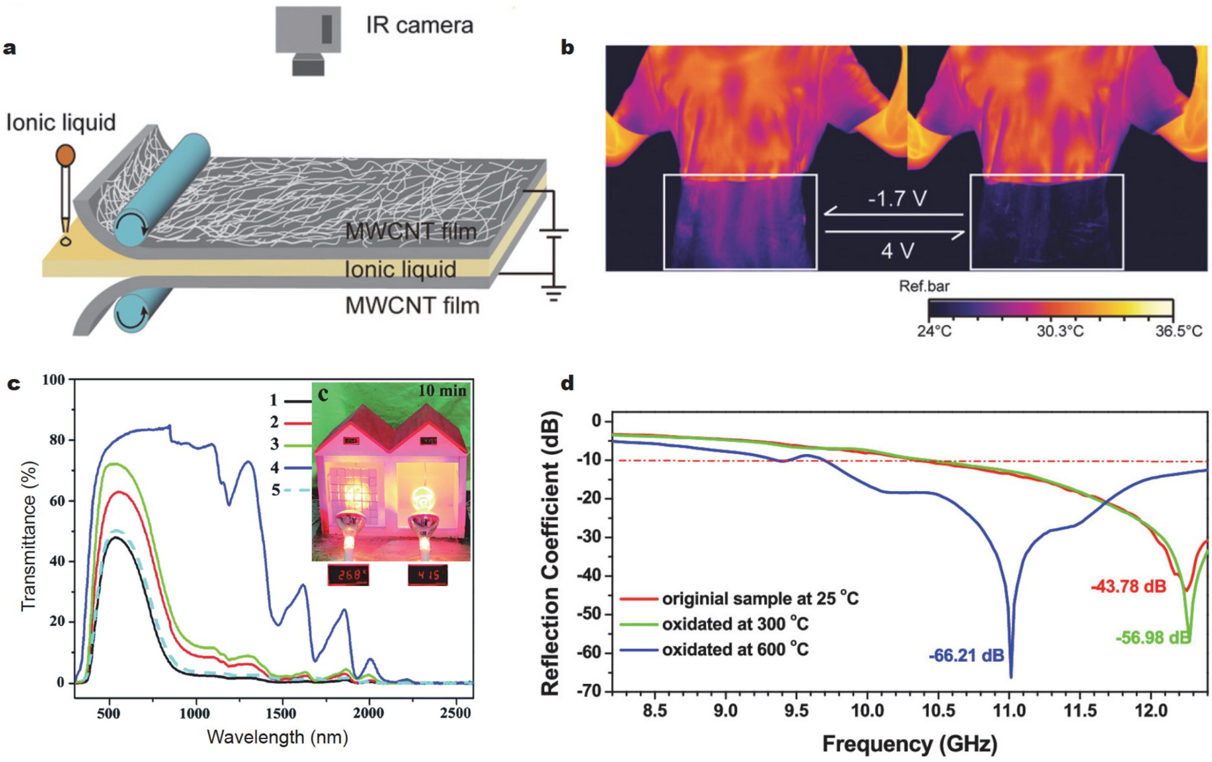

Figure 2 (a, b) Fabrication and thermal camouflage of the MWCNT-based device [30]. Copyright 2020, John Wiley and Sons. (c) Transmittances of the as-prepared samples with a house model for infrared shielding test [31]. Copyright 2017, Royal Society of Chemistry. (d) Reflection coefficients of different oxidation temperatures [32]. Copyright 2018, American Chemical Society.

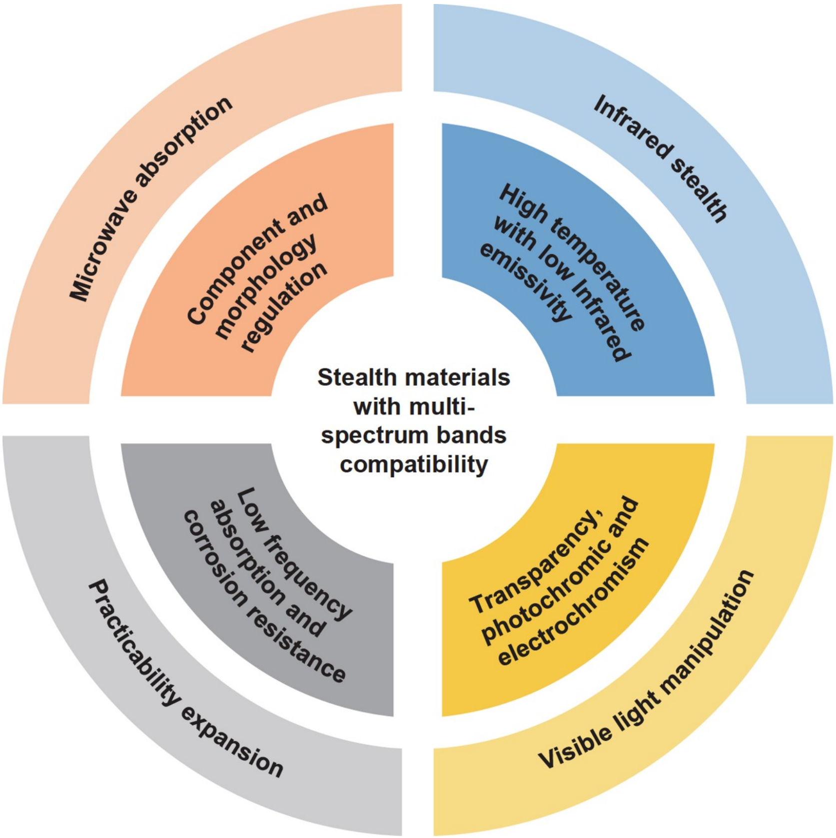

Figure 3 Summary of multi-spectrum bands compatibility of materials design.

property optimization.

In summary, the trends and vistas for stealth materials research are as follows, demonstrated in Fig. 3.

(1) The mature research domain of stealth materials is microwave absorption, especially focusing on the component and morphology regulation of coatings [37]. Carbon-based nano-/micro-particles and metal/metallic oxide comprise the most frequently developed ingredients [38,39]. Composite coatings can reach effective RL easily in the X band; EMW stealth coatings often demonstrate an effective bandwidth of approximately 5 GHz. How to actualize low-frequency absorbing property and ultra-wide bandwidth synchronously remains a challenge. A few feasible methods have been reported, including enlarging ε_r in a low-frequency region with an external electrical field, broadening the effective absorption band with thickness regulation, and improving normalized impedance with gradient components [40]. But there is still research space left for the following materials scientists because of the dissatisfactory thickness of coatings and poor low-frequency property.

(2) How to realize infrared and microwave stealth compatibility? Infrared stealth materials have also been studied for decades, yet the subject of infrared stealth materials possesses high conductivity, which leads to strong reflection in 2–18 GHz. Meantime, the bi-spectrum stealth compatible materials should bear high temperatures with low infrared emissivity. Differing from EMW stealth meant for aeronautics, infrared stealth/camouflage systems should not blindly seek low emissivity. Instead, tunable emissivity is much more practical, owing to the complex and changeable background. One of the as-described methods is external field control, including electrochromism. Besides, meta-structure or meta-material design is also proved as a feasible approach.

(3) How to realize visible light responsiveness for EMW stealth materials? Currently reported papers basically set their sights on transparent EMW shielding materials towards the absorbent application purpose due to the high conductivity of shielding materials [41]. Occasions, like windows in aircraft, put forward the urgent demand for transparent absorbing materials. Alongside transparency, photochromic and electrochromism aiming at visible light camouflage are also quite significant. Visible light manipulation will become a research hotspot in EMW absorbing materials particularly.

(4) From previous reports, EMW stealth materials are mainly related to coating formation and focus on the EMW stealth property merely. Thus, for coming industrial applications, the practicability expansion should also be taken into consideration [42–44], such as low-frequency property for radar frequency, anti-oxidation for astronautics, and corrosion resistance for ocean-going vessels. Ceramics and phase-changing materials are possible solutions. Yet, challenges remain for future researchers in the extension of service life.

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Conflict of interest The authors declare that they have no conflict of interest.

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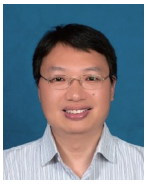

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多频谱兼容性: 隐身材料研究的新方向

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摘要 本文首先概括了上世纪隐身技术雏形、隐身参数计算机理、探测技术等的发展历史以及吸波材料的常见分类. 然后指出材料合成中多注重性能均衡的复合设计, 并着眼于反射损耗与阻抗匹配参数等性能调控方式, 如微观组分调节、形貌设计等. 近十年来, 随着应用需求的提高, 电磁波吸收为主的隐身材料趋向于低频、宽频的应用需求, 外场调控、阻抗渐变等设计思路引起关注. 而随着航空航天、地面装备等实际需求的增加, 电磁波吸收材料逐渐向红外/可见光等多频谱兼容、服役性能等方向发展. 如何实现红外发射率可调、可见光透明或伪装、耐苛刻环境成为了亟待解决的热点问题.