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Infrared stealth property based on semiconductor (M)-to-metallic (R) phase transition characteristics of W-doped VO_2 thin films coated on cotton fabrics

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ABSTRACT

The infrared stealth fabric was prepared using W-doped VO_2 (M) paints by the coating technology. The thermochromic W-doped VO_2 (M) was synthesized through hydrolysis method and two-step calcinations under N_2 atmosphere. The powders were evaluated by scanning electron microscopy, X-ray diffraction patterns and differential scanning calorimetry. The infrared emissivity of coated cotton fabric was measured by IR-2 Infrared Emissometer, which was as low as 0.752. The low infrared radiation intensity for coated cotton fabric was detected using the infrared imaging systems above the phase temperature of W-doped VO_2 (M). The results indicated that the W-doped VO_2 (M) thin films exhibited thermal infrared stealth performance, which could adapt to the surroundings spontaneously.

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1. Introduction

The development of infrared surveillance technology in modern world had a significant effect on military personnel and equipment with an effective concealment [1]. Generally, objects and human beings were exposed easily under the infrared detectors due to the high emissivity. Therefore, low-emissivity materials became the focus of IR stealth technology in many countries [2]. However, emissivity of outside environment was not constant due to the temperature, moisture, solar intensity and cloud cover [3,4]. The objects were invisible for infrared sensitive viewing devices under the protection of materials with low emissivity in certain times; but they could be re-exposed when conditions altered. Hence, it is necessary to obtain a sort of material whose emissivity was adaptable to the surroundings spontaneously.

Vanadium dioxide of monoclinic structure VO_2 (M) is regarded as the most important and promising material, since it exhibits phase transition at transition temperature $T_C = 68$ °C, with dramatic changes of optical properties in IR area [5]. Vanadium dioxide (VO_2) has many different allotropic phases including VO_2(R), VO_2(M), VO_2(B), VO_2(A), VO_2(C) and VO_2(D) [6–8]. VO_2 (B) is a metastable allotropic phase, which can transform to the more stable rutile structure VO_2 (R) by heating treatment, then became monoclinic VO_2 (M) after the cooling process [9]. The rest metastable polymorphs VO_2(A), VO_2(C) and VO_2(D) were less reported comparatively [10]. In particular, most of the studies focus on the monoclinic VO_2 (M) due to its thermochromic properties [5]. Below the transition temperature (Tc), monoclinic VO_2 (M) is a semiconductor, and above Tc, it becomes a rutile structure VO_2(R) which is metallic. This phase transition is fully reversible and associates with dramatic changes in electrical, magnetic and optical properties [11–13]. Moreover, Tc of VO_2 can be tuned to ambient temperature by doping with W, Nb, Mo and Ti for practical application [14–19]. Tungsten is one of the most effective dopant [14,15]. Therefore, with such properties, VO_2 material can be considered as a promising candidate for a variety of potential applications such as energy-efficient window coatings [20], thermal sensors [21], cathode materials for reversible lithium batteries [22], electrical and infrared light switching device [23,24].

However, there were few reports about the infrared stealth property of VO_2 in the literatures. Among them, Guinneton pointed out there was a fall of 60% in normal integrated emissivity of VO_2 thin film between 8 and 12 μm, which made it possible to be a sort of potential material for infrared stealth [11]. Moreover, the adaptive infrared camouflage property of VO_2 coated fabric was reported by Dongqing Liu [25]. Results showed that infrared emissivity in 8–14 μm of VO_2 coated fabric was 0.82, which decreased by only about 0.1 during the phase transition. Therefore, infrared stealth performance of VO_2 must be improved for practical application.

In this paper, W-doped VO_2 (M) paints were used to prepare coated cotton fabric. The VO_2 (M) samples with various W doping contents were prepared via hydrolysis of vanadyl sulfate mixed with different amounts of sodium tungstate dehydrate, followed by two-step thermal treatment under N_2 atmosphere. The infrared stealth property of cotton fabric with W-doped VO_2 films was measured by IR-2 Infrared

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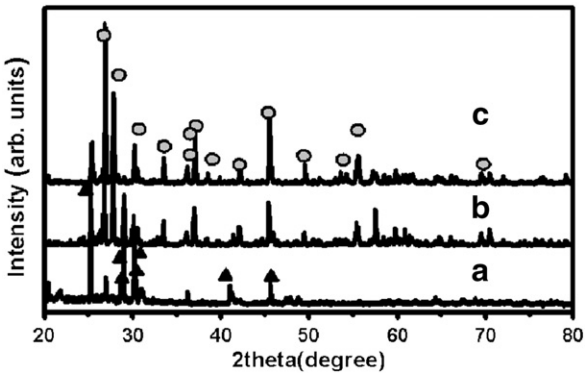

Fig. 1. XRD patterns of W-doped VO_2 samples: (a) W-doped VO_2 (B); (b) VO_2 (M); (c) W-doped VO_2 (M).

Emissometer and Infrared imaging systems. Results indicated that W-doped VO_2 (M) had low infrared emissivity and infrared radiation intensity, which exhibited an outstanding performance of thermal infrared stealth.

2. Material and methods

2.1. Materials

Vanadyl sulfate hydrate ($VOSO_4$, 98%) was purchased from Shanghai Huating Chemical Factory Limited Company. Sodium hydrogen carbonate ($NaHCO_3$), sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) and absolute ethyl alcohol (CH_3CH_2OH) were purchased from Sinopharm Chemical Regent Limited Company. Water borne polyurethane (PU-3645) was purchased from Sansheng Limited Company. The cotton fabric with a density of 300 g/m^2 was provided by Huafang Limited Company.

2.2. Preparation of W-doped VO_2 (M) powders

The synthesis method of W-doped VO_2 (B) with different amounts of tungsten was similar to hydrothermal process reported by Jianqiu Shi et al. [26]. In a typical synthesis, vanadyl sulfate hydrate (0.15 mol) and stoichiometric sodium tungstate dihydrate with different W/(W + V) molar percents (0%, 0.5%, 1.0%, 1.5%) were dispersed in 100 ml deionized water by vigorous stirring to form a green solution. Meanwhile, sodium hydrogen carbonate (0.3 mol) was dissolved in 100 ml deionized water and dropped into the above mixture. After stirring for an hour, the resulting brown powders were filtered and washed with deionized water till no sulfate was tested. Then, resulted samples were washed by absolute ethanol and dried at 40 °C under vacuum. The VO_2 (B) powders with different W-doped contents were prepared by subsequent calcination at 800 °C under N_2 atmosphere. Finally, VO_2 (M) powders with different W-doped contents were obtained through the secondary heat treatment at 500 °C under N_2 atmosphere in the atmosphere furnace. The products were named as x-W-doped VO_2, wherein x represented W/(W + V) molar percent.

Table 1
W molar percent of W-doped VO_2 in solutions and in the prepared solids.

W/(W + V)in solutions (%)	W/(W + V) in prepared solids (%)	Standard deviation
0	0	0
0.5	0.489	0.0017
1.0	0.971	0.0016
1.5	1.480	0.0019

2.3. Preparation of thermochromic coatings

The VO_2 solids with different molar ratios of W were mixed with zirconia beads, the dispersant and deionized water, at a weight ratio of 1:50:0.1:10 in a planetary ball mill, stirring for 60 min at a rotational speed 800 r/min. Then, W-doped VO_2 suspension was carefully embedded into the water borne polyurethane (PU-3645) with vigorous stirring for 2 h. The above solution was deposited on the cotton fabric by the automatic coater. The wet coating thickness controlled by thickness controller was 300 μm. Then the obtained W-doped VO_2 coated fabric was dried at 150 °C for 5 min with a shaping machine. As control experiment, pure water borne polyurethane coating on the fabric was also prepared.

2.4. Characterization

Wide angle powder X-ray diffraction measurements (WXRD) were carried out using a Rigaku diffractometer (Rigaku Ltd, 40 KV, 200 mA, Cu Kα, λ = 1.54056 Å). Energy Dispersion X-ray analysis (EDX) was determined on a IE 300X instrument (Oxford, United Kingdom) operating at 7.5 kV. The obtained data was quantified using Oxford Software, with pure metallic vanadium and tungsten used as standards. Scanning Electron Microscopy (SEM) was carried out on a JSM-5600LV scanning microscope (JEOL, Japan) to determine the morphology of samples at a microscale. Differential scanning calorimetry (DSC) was performed on a DSC204F1 system (NETZSCH, Germany) to measure the phase-transition temperature of W-doped VO_2 (M). Each sample was tested in air atmosphere from room temperature to 150 °C, with a heating rate of 10 °C/min. The infrared stealth durability of coated fabrics was

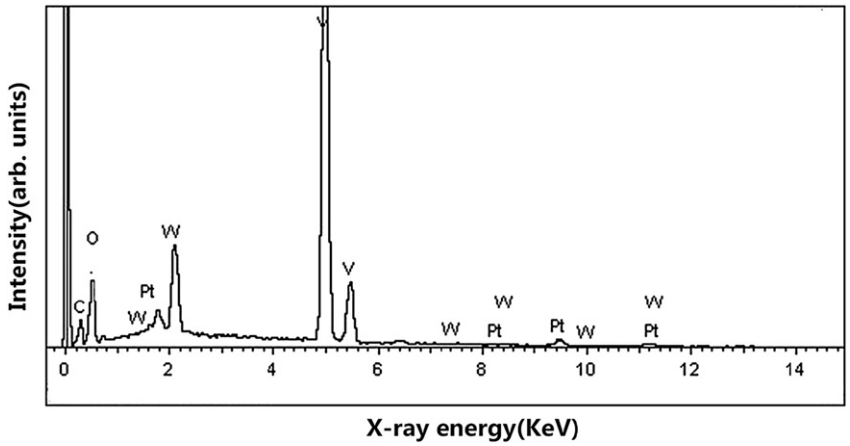

Fig. 2. EDX pattern of 1.0%-W-doped VO_2 (M) powders.

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