2012 4th International Conference on Chemical, Biological and Environmental Engineering
IPCBEE vol.43 (2012) © (2012) IACSIT Press, Singapore
DOI: 10.7763/IPCBEE. 2012. V43. 9

Near Infrared Cut-off Characteristics of Various Perovskite-based Composite Films

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Abstract. Homogenous cesium tungsten tri-oxide (CsxWO3) and molybdenum tri-oxide (Cs0.33MoO3) powder was prepared by solvothermal reaction using (NH4)10H2(W2O7)6, H8MoN2O4 and Cs2CO3 aqueous solution followed by annealing. The cesium doped tungsten tri-oxide (CsxWO3) and the cesium doped molybdenum tri-oxide (CsxMoO3) nanocomposite films was deposited by the sol-gel bar-coating method onto PET-film (polyethylene terephthalate film, thickness 186 ㎛) substrate. The structure and sizes of ceramic particles was observed XRD and PSA spectrometer, the optical properties of their films were investigated by UV-VIS, NIR spectrometer. Synthesized particles typically formed cubic structure for good absorption of NIR, size observed as being 30-100 nm and <150 nm. CsxWO3, Cs0.33MoO3 nanoparticles showed a high transmittance in the visible wavelength region as well as excellent shielding capability of near-infrared (NIR) wavelength, indicating that CsxWO3, Cs0.33MoO3 nanoparticles have a appropriate characteristic as solar filter applications.

Keywords: Thermal insulating materials (TIM), Perovskite, Nanocomposite film, Nanodispersion, Near-infrared (NIR) shielding

1. Introduction

A strong demand for renewable energy sources to be developed result from that the modern energy consumption is continuously increasing. In view of this, many researchers are interested in transparent thermal insulating materials (TIM) for many potential desires in the various fields of industry. Particularly, solar control coatings for transparent TIM are transparent at $400 < \lambda < 700$ nm and effectively reflecting at $700 < \lambda < 3000$ nm. By controlling the near-infrared (NIR) regions between about 780 and 2500 nm, it would be help to reduce the energy for air conditioning and thereby decrease the emission of carbon oxides from buildings and automotives.

Meanwhile, tungsten trioxide(WO3) exhibits the transparent property in the visible and NIR lights and has a wide band gap of 2.62 eV5. Additionally, WO3-based electrochromic devices are mainly being researched for smart film applications with changeable modulations in optical spectra. A metallic conductivity and strong NIR absorption can be induced when free electrons are introduced into crystals by either decreasing the oxygen content or by adding ternary elements. The oxygen deficiency in tungsten oxides leads to a complex-ordered structure known as the Magneli structure, while the ternary addition of the positive ions leads to the tungsten bronze structure. It has been reported that the tungsten bronzes with the hexagonal phase are of particular interest in the application of electrochromic devices owing to the relatively high diffusion coefficients of hydrogen ions and metal ions compared with those of the orthorhombic phase and WO3. Until now, study on the synthesizing WO3 with different morphologies and properties has been widely reported. However, there is limited work reported on the synthesis and characterization of only

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CsxWO3 nanorod with controlled morphology by solvothermal reaction. Furthermore, heat-shielding properties of CsxWO3 nanoparticle have not been reported.

In this paper, we investigated the preparation and characterization, the two kinds of basic materials (CsxWO3, Cs0.33MoO3) properties and the optical properties of nanoceramic composite films for solar light control applications.

2. Experimental

2.1. Synthesis of Cesium Tungsten Trioxide Powders.

For synthesis of perovskite oxide, 99.99 % purity ammonium tungstate ((NH4)10H2(W2O7)6), 99.98 % purity ammonium molybdate (H8MoN2O4), 99.9 % purity trace metals basis Cesium carbonate (Cs2CO3) were starting materials. Those materials were dissolved in distilled water and stirring for 1 h at room temperature, and two feed solution were well mixed in the ceramic crucible. This mixed solution was dried at 180 ℃ for 8 hours with air in the heating chamber (model ON-O2GW, JEIO TECH, Korea). The prepared powder was heated at 450, and 500 ℃ for 1 h with an H2, N2 gas flow at H2/N2 cc/min = 90/10 and annealing at 500, 600, 700, 800, and 900 ℃ for 1 h with a N2 gas flow at N2 ccm = 100 in the vacuum furnace (model DVF-1600s, DAE HEUNG SCIENCE, Korea). As for cesium tungsten oxide powders, CsxWO3, cesium content at x = 0.33, 0.50, 0.75 and 1.00 mol were synthesized this study.

2.2. Preparation of Nanocomposite Films.

The composite particles were dispersed in a solvent ethanol with dispersed agent (BYK2001) in a strongly pulverization shake mill (turbo-mill, model SPEX 8000D, USA) with the iron ball (20 mm) and the zirconia bead (0.3 mm, ZrO2 94.5 %, Y2O3 5.1 %) for 3 hours. The dispersed particle size and distribution in dispersion was measured by a particle size analyzer (PSA, model ELSZ, OTSUKA ELECTRONICS, Japan) with quartz cell which is the dynamic light scattering methods.

Dispersed sol was coated with Urethane acrylic UV-curing binder onto the PET substrate by bar-coating methods. Dispersion and coating binder were mixed well in vial with rotating mixer (model MS 3basic, IKA, Japan). The prepared films were dried at 80 ℃ for 1min in heating chamber and illuminated with a mercury UV lamp UV-curing equipment (model LZ-U1O1DCH, LICHTZEN, Korea) at intensity 800 W/cm for 20 sec.

3. Results and Discussion

The hexagonal structure of the CsWO3 powder was confirmed both by Scanning electron microscope (SEM) and by X-ray diffraction (XRD). A synthesis of CsWO3 particle with annealing temperature of 800 ℃ was morphology as measured by SEM. The CsWO3 particles under the 100 nm can be observed in Fig. 1. Each CsWO3 nano-particle was aggregated with the mass of the few micron size. Those clusters shatter to pieces by pulverization dispersed methods.

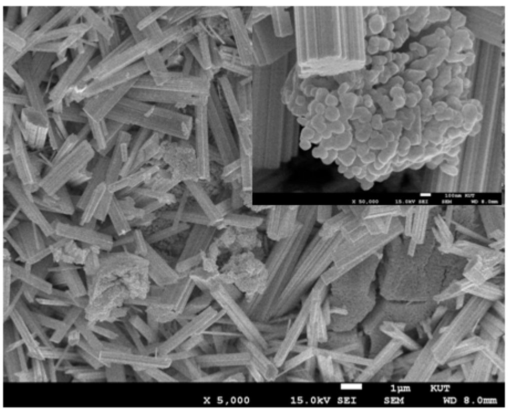

Fig. 1: Scanning electron microscope (SEM) image of Cs0.33WO3 nanoparticle.

The typical XRD pattern of synthetic Cs0.33WO3 nano-particle with the 800 ℃ annealing temperature sample was presented in Fig. 2. The particle of shows perovskite tungsten bronze of hexagonal symmetry as the major phase along with 2-3 weak lines WO3 of cubic symmetry.

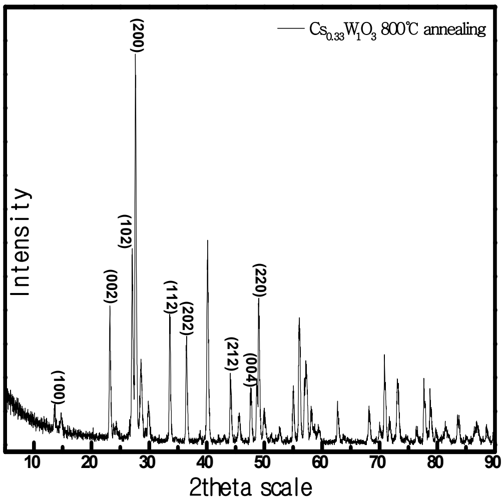

Fig. 2: Effect of annealing temperature to Cs0.33WO3; XRD (X-ray diffraction, 2 ∘/min ; 590 ∘) ; annealing at 800 ℃.

The optical transmittance of Cs0.33MoO3 nano-composite film was measured by using UV/VIS spectrometer and NIR spectrometer. Fig. 5 exhibits the properties of visible and NIR absorption with 600 ℃ annealed sample. These results shows dispersion of cesium molybdenum tri-oxide (Cs0.33MoO3) lead to the more strong NIR absorption on increasing content of Cs0.33MoO3, the NIR wavelength absorption increased. Namely, it means that the amounts of nano-sized Cs0.33MoO3 for efficient absorption of NIR increase with increasing contents of Cs0.33MoO3. The same optical trend has also been confirmed for the thin coating of those nanoparticles fabricated on polyethylene films and glass substrates.

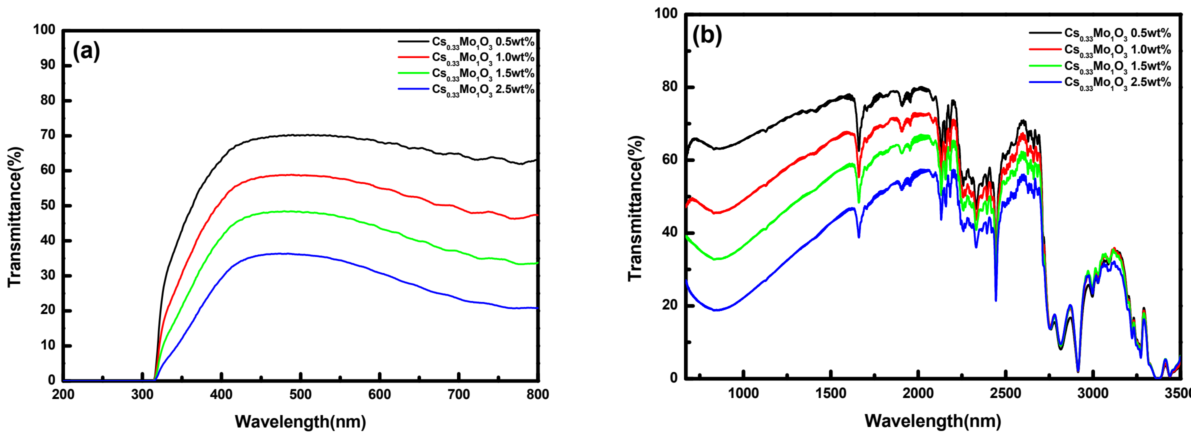

Fig. 3: Visible and NIR region transmittance properties of Cs0.33MoO3; (a) UV/VIS measured at 200~800 nm, and (b) NIR measured at 670~2500 nm.

4. Summary

In this work, the ceramic nano-composite films with homogeneous dispersions of tungsten and molybdenum bronze nanoparticles with with ternary additive cesium have been prepared by mixing urethane acrylic/UV-coating binder and dispersed sol depending on the dispersed time and the particle contents. Furthermore, we are synthesized the CsxWO3 and Cs0.33MoO3 with controlling contents and the using annealing at 800 and 600 ℃ in an N2 gas for 1h respectively. The structure and sizes of ceramic particles was observed XRD and PSA spectrometer, the optical properties of their films were investigated by UV/VIS, NIR spectrometer. Synthesized particles typically formed cubic structure for good absorption of NIR, size observed as being 30~100 nm and ≤ 150 nm. Especially, remarkable absorption of NIR wavelength ranges makes ceramic composite films goof candidate for use as a heat shielding window, it clearly shows useful for applications where heat shield is required.

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