Discovery of an excellent IR absorbent with broad working waveband: Cs₅WO₃ nanorods

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Experimental

Analytical grade chemicals, tungsten hexachloride (WCl₆), cesium hydroxide monohydrate (CsOH·H₂O), dehydrated ethanol and acetic acid, purchased from Kanto Kagaku Co.Inc. were used without further purification as starting materials. In a typical experiment, a certain amount of WCl₆ was dissolved into the dehydrate ethanol with violently stirring, then the desired amount of CsOH was introduced to the above yellowish solution of WCl₆. After becoming the mixture homogeneous, 10ml acetic acid was introduced. The final concentration was adjusted to 0.015M of WCl₆ with 50 at.% of CsOH. The mixed solution was introduced into a Teflon-lined autoclave of 100 ml internal volume, followed by solvothermal treatment at 240°C for 20h.

This method, which is named as water controlled-release solvothermal process (WCRSP), is a novel and facile method to synthesize nanorods of tungsten bronze type CsxWO₃. Water molecules were released slowly by the reaction between acetic acid and dehydrate ethanol during the solvothermal process. More details about the WCRSP were shown in Ref.1 and Ref.2. Dark blue colored products were centrifuged and washed with water and ethanol 4 times, respectively, followed by vacuum drying at 60°C over night. It was found that the calcinations under atmosphere of N₂ at 500°C further enhanced the absorption ability of CsxWO₃.

The coating slurry was formed by mixing the CsxWO₃ powder with collodion and ethanol at a mass ratio of ethanol: collodion: CsxWO₃=1:0.93:0.15. Then, the coating slurry was painted on a quartz glass by an applicator with concave in depth of 12.5μm. The phase compositions of the samples were characterized by X-ray diffraction analysis (XRD, Shimadzu XD-1) using graphite-monochromized CuKα radiation. The size and shape of the nanoparticles were observed by transmission electron microscopy (TEM, JEOLJEM-2010). The optical response of the thin film was measured by using a spectrophotometer (JASCO V-670), giving output of transmittance in the UV, visible, and infrared ranges (200-2700nm). Energy-dispersive X-ray spectrometer (EDS) was employed for approximate elemental analyses. FT-IR measurements were conducted by using the FTS7000 series (DIGILIB). Thermographic measurements were recorded by thermographimeter (FLIR System i7).


Fig. S1 EDS profile of Cs₆WO₃ nanorods
Fig.S2  Transmittance spectra of (a) Cs$_{0.32}$WO$_3$ nanorods synthesized by WCRSP process and (b) Cs$_{0.32}$WO$_3$ synthesized by solid state reaction.

Fig.S3  FT-IR spectra of (a) Cs$_{0.32}$WO$_3$ nanorods synthesized by WCRSP process and (b) Cs$_{0.32}$WO$_3$ synthesized by solid state reaction.
Fig. S4  Transmittance spectra of (a) the thin film of Cs$_{0.32}$WO$_3$ nanorods synthesized by WCRSP process and (b) ITO glass (10 Ω·□$^{-1}$)

Fig. S5  XRD pattern of Cs$_x$WO$_3$ nanorods
Fig. S6 Thermographic image of Cs$_{0.32}$WO$_3$ powder before irradiation.
Fig.S6 Thermographic image of WO$_3$ powder after irradiation for 10 seconds.
Fig. S7 The transmittance spectra of Cs₆WO₃ coated on common glass.