Electronic Supplementary Information

Synthesis of mesoporous single crystal Ga_2O_3 nanoplate with improved photoluminescence and high sensitivity in detecting CO†

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Experimental sections and characterizations

Experimental sections

Sample Preparation

$KGaO_2$ solid powders were prepared by heating stoichiometric mixture of K_2CO_3 and Ga_2O_3 at 950 ^oC for 12 h. For the preparation of GaOOH, in a typical procedure, 10 mL of $KGaO_2$ aquatic solution (0.2 $molL^{-1}$) was added into 10 mL of CH_3COOH (0.2 $molL^{-1}$) aquatic solution and stirred for 3 h at room temperature to form GaOOH single crystal nanoplate, then the sedimentation was separated by centrifugation, washed with deionized water and dried at 60 ^oC for 2h.

α-Ga_2O_3 and β-Ga_2O_3 were obtained by heating the as-prepared GaOOH at 550 ^oC for 2 h and 800 ^oC for 3 h with a heating rate of 5 $^oC\ min^{-1}$, respectively.

Characterizations

The as-prepared $HGaO_2$, α-Ga_2O_3 and β-Ga_2O_3 were characterized by x-ray diffractions (XRD) for phase identification on the Rigaku Ultima Ⅲdiffractometer and by transmission electron microscope (TEM; FEI Tecnai G2 F30 S-Twin) and field emission scanning electron microscop (FE-SEM; NOVA230, FEI Ltd.)for microstructural observations. The specific surface area was determined by an adsorption apparatus (Micromeritics TriStar 3000, USA) based on the BET method. The photoluminescence (PL) spectroscopy was obtained by using the Cary eclipse fluorescence spectrophotometer (USA).The thermal analysis was carried out by using the thermogravimetric-differential scanning calorimetry (TG-DSC) on NETZSCH STA 449F3. The detected range of temperature is from room temperature to 1000 ^oC with a heating rate of 10 $^oC\ min^{-1}$.

Photocatalytic activity test

Photocatalytic activities of β-Ga_2O_3 nanoplate for pure water splitting were evaluated in a gas-closed circulation system. The NiO cocatalyst was loaded by impregnation method using the aqueous solution of $Ni(NO_3)_2 \cdot 6H_2O$. 0.3 g of β-Ga_2O_3 nanoplates and ca.1 mL of water containing the appropriate amount (0.003 gram of Ni) of $Ni(NO_3)_2 \cdot 6H_2O$ were well dispersed in a Al_2O_3 crucible. The solution was stirred using a glass rod and simultaneous evaporated on a water bath. The dried powder was heat treated at 270 ^oC for 1 h in air using a muffle furnace. For the photocatalytic reactions, the NiO modified β-Ga_2O_3 nanoplates were dispersed in 390 mL of reactant solution by a magnetic stirrer in an inner irradiation cell made of quartz. The light source was a 400 W high-pressure mercury lamp (SEN; HL400EH-5). Similarly, photocatalytic water splitting of the commercial β-Ga_2O_3 was evaluated as a reference. The gas amounts of H_2 and O_2 from H_2O splitting were determined using gas chromatography (Shimadzu; GC-8A, MS-5A column, TCD, Ar carrier).

Electrochemical Sensor

Electrochemical response was measured at room temperature with a conventional three-electrode system and an electrochemical workstation (CHI 600B, China). All the potentials were referred to a saturated calomel electrode (SCE). A platinum plate was used as the counter electrode. Bare and modified glassy carbon (GC) electrodes (surface area is 0.0707 cm^2) were used as the working electrode. The GC electrodes were polished with aqueous slurries of successfully finer alumina powder (down to 0.05 μm), and then carefully rinsed with Milli-Q water in an ultrasonic bath for 5 min. After ultrasonicating the β-Ga_2O_3 suspension (powder of commercial β-Ga_2O_3 or β-Ga_2O_3 nanoplates in Milli-Q water) for 1 h, the as-prepared suspension containing 2 mg samples was uniformly cast onto the surface of the GC electrode. The as-modified electrode was dried under ambient conditions overnight before use. The electrolyte (0.4 $molL^{-1}$ $NaClO_4$) was in thoroughly anaerobic conditions by bubbling with high-purity nitrogen. The target gas, 50 ppm of CO, was flowed into the detecting system.

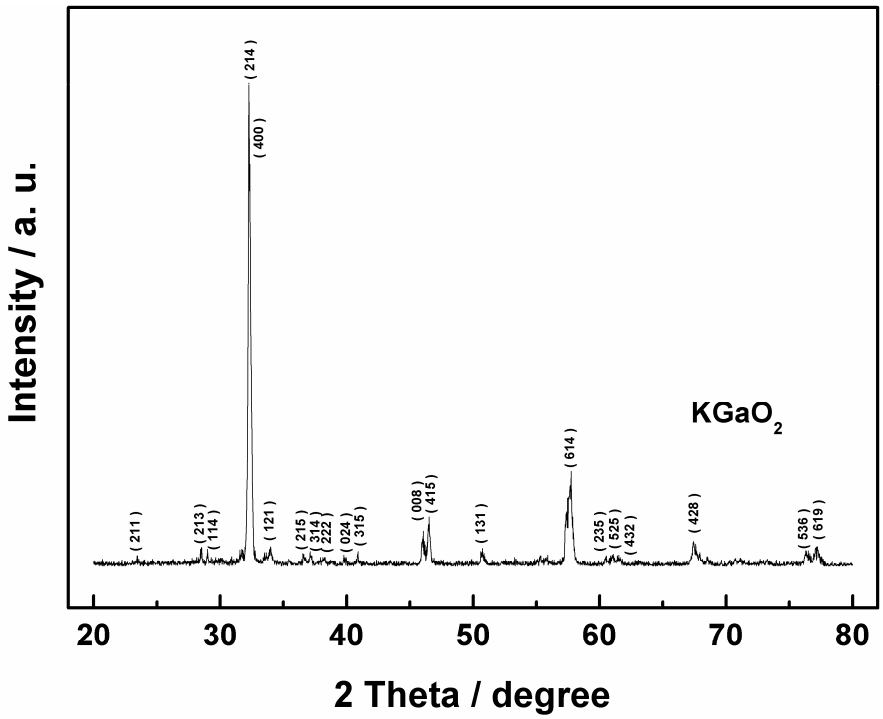

Figure S1. XRD pattern of the $KGaO_2$ powder obtained by solid state reaction of K_2CO_3 and Ga_2O_3 at 950 ^oC for 12 h. The XRD pattern is in good agreement with JCPDS 80-1194.

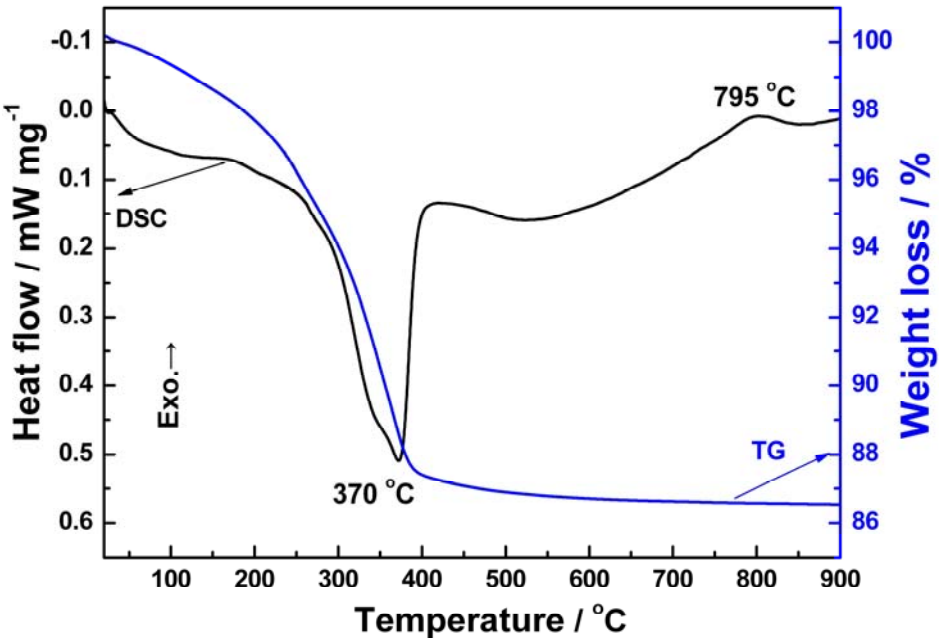

Figure S2. TG-DSC curves of the as-prepared single crystal GaOOH nanoplate.

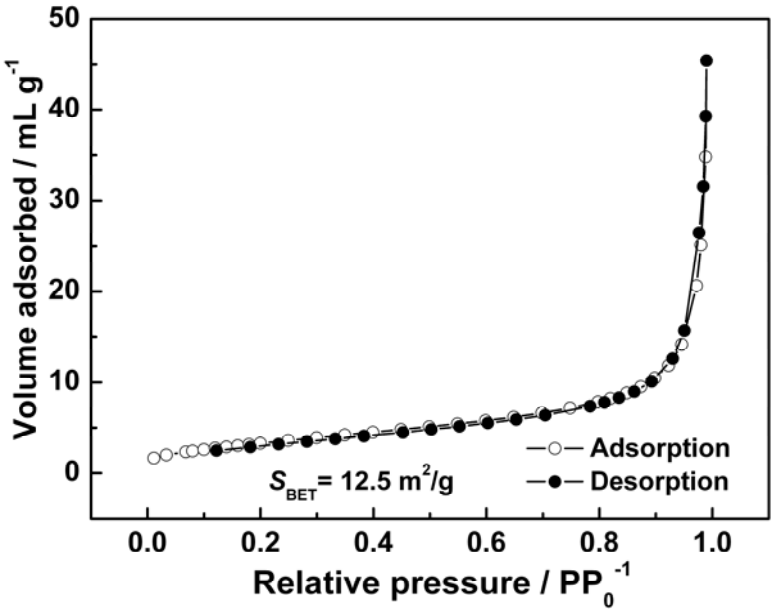

Figure S3. Nitrogen absorption-desorption isotherms of single crystal GaOOH nanoplate.

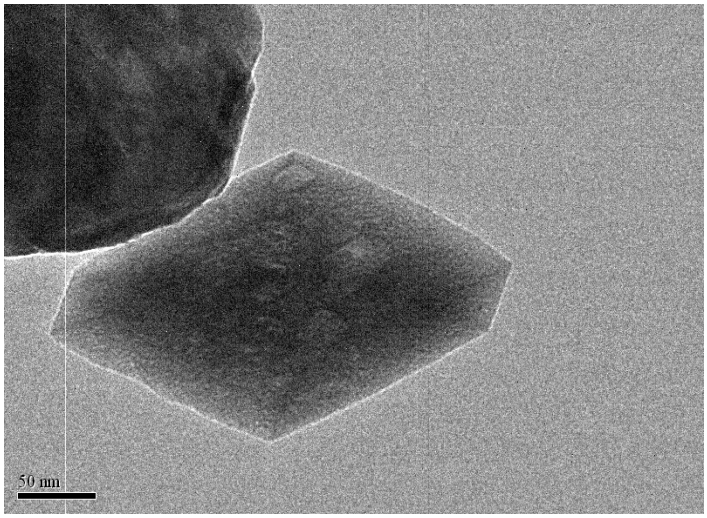

Figure S4. Typical TEM image of single crystal GaOOH nanoplate.

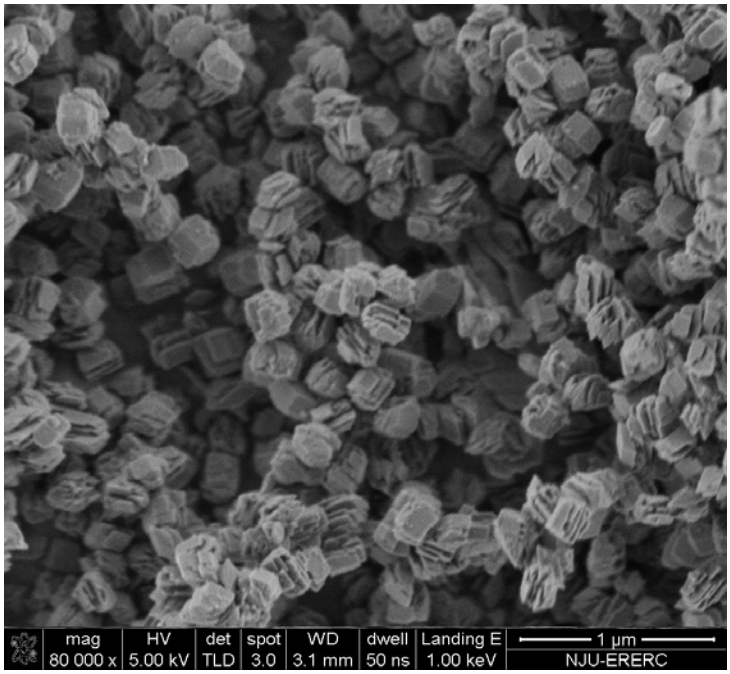

Figure S5. SEM image of the single crystal GaOOH nanoplate prepared by room-temperature ion exchange.

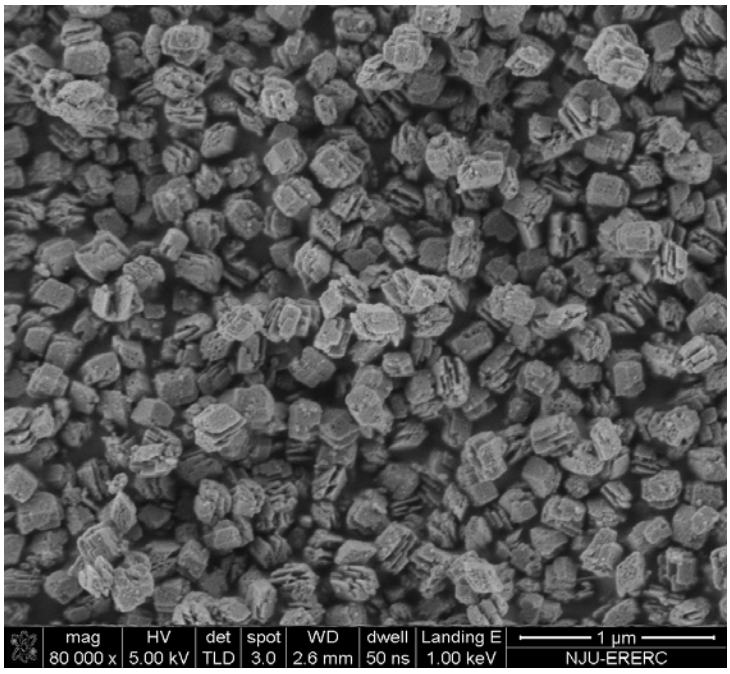

Figure S6. SEM image of the single crystal α-Ga_2O_3 nanoplate prepared by heating GaOOH at 550 ^{o}C for 2 h.

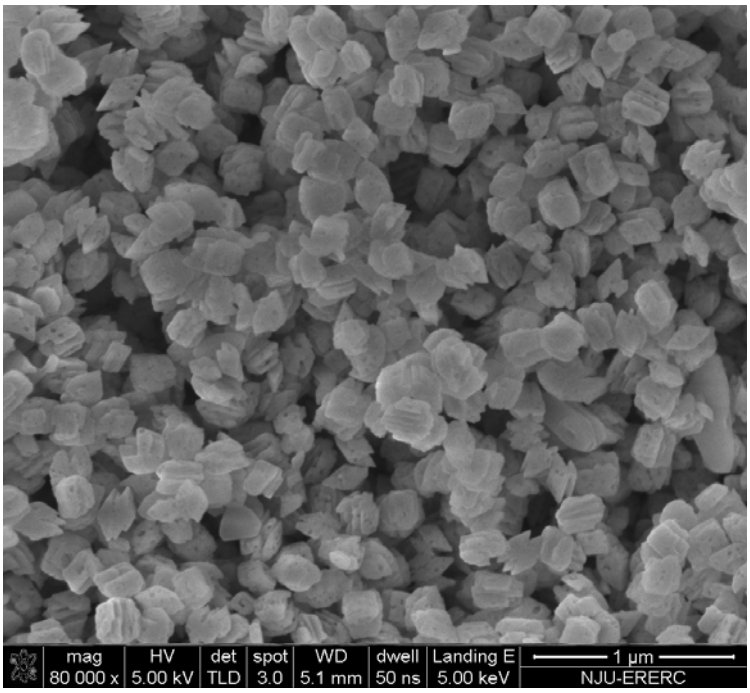

Figure S7. SEM image of the single crystal β-Ga_2O_3 nanoplate prepared by heating GaOOH at 800 ^oC for 3 h.

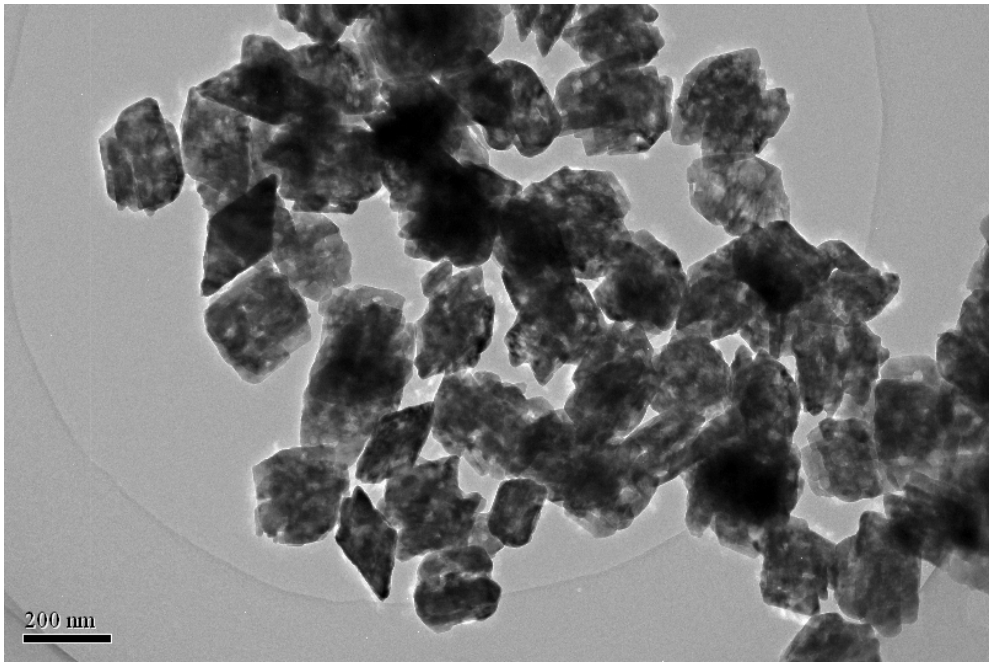

Figure S8. TEM image of the single crystal β-Ga_2O_3 nanoplate prepared by heating GaOOH at 800 ^oC for 3 h. Apparently, the mesopores form in the single crystal nanoplate.

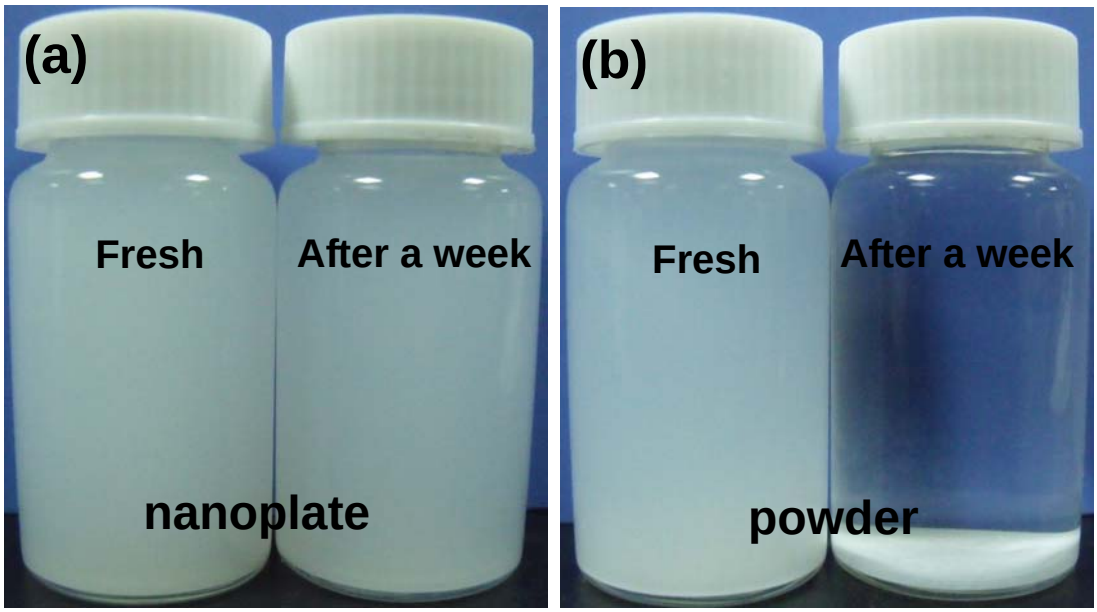

Figure S9. Comparison of stability for (a) colloidal β-Ga_2O_3 nanoplate suspension and (b) commercial β-Ga_2O_3 powder suspension before and after a week.

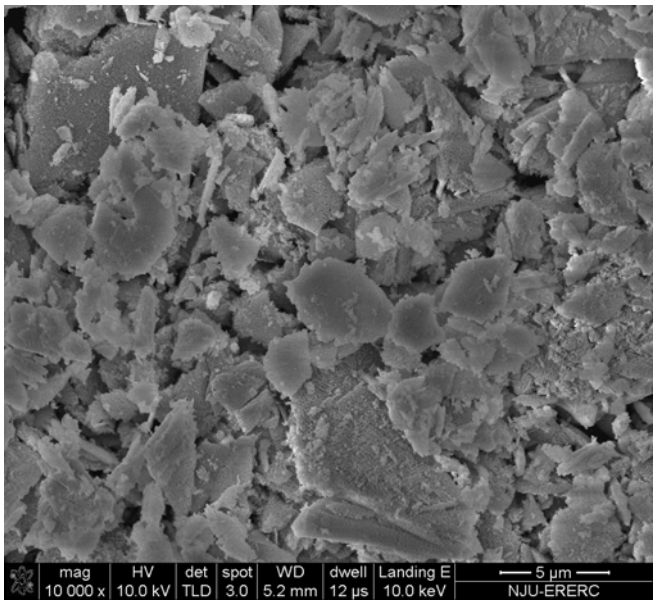

Figure S10. SEM image of the commercial β-Ga_2O_3 powder.

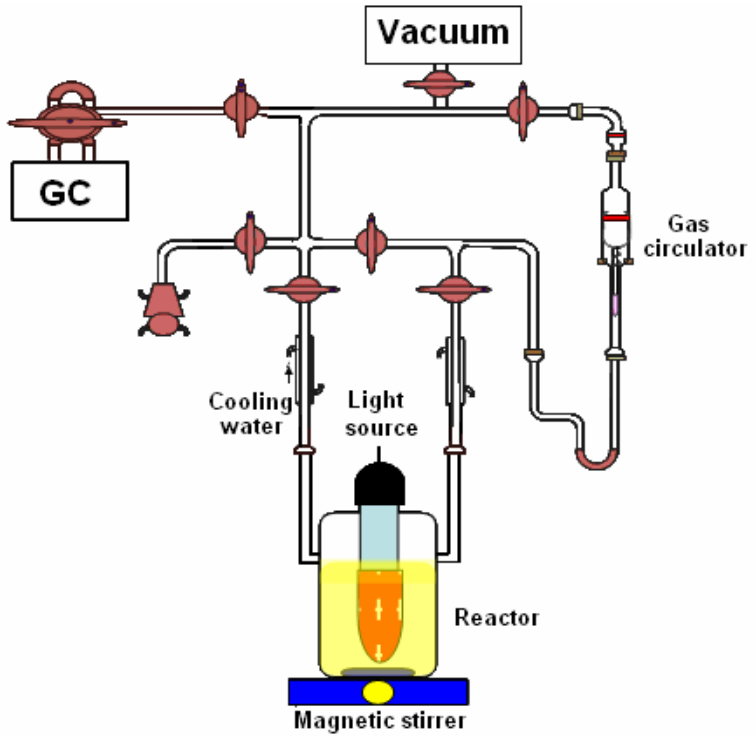

Figure S11. The reaction setup for evaluating the H_2 and O_2 from water splitting over the β-Ga_2O_3.

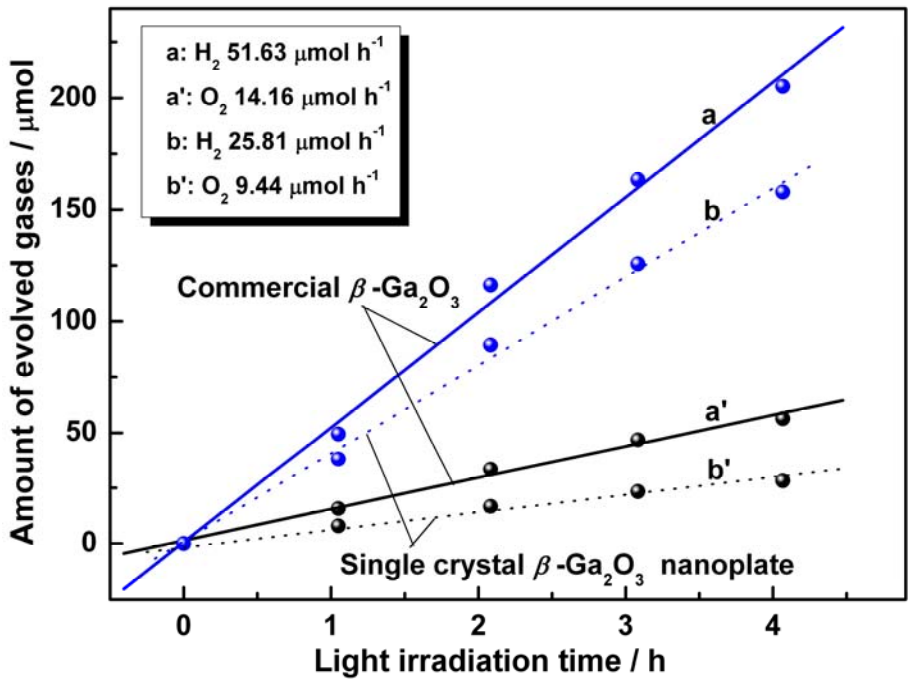

Figure S12. Amount of evolved H_2 and O_2 gassess over the commercial and single crystal β-Ga_2O_3 as a function of light irradiation time.

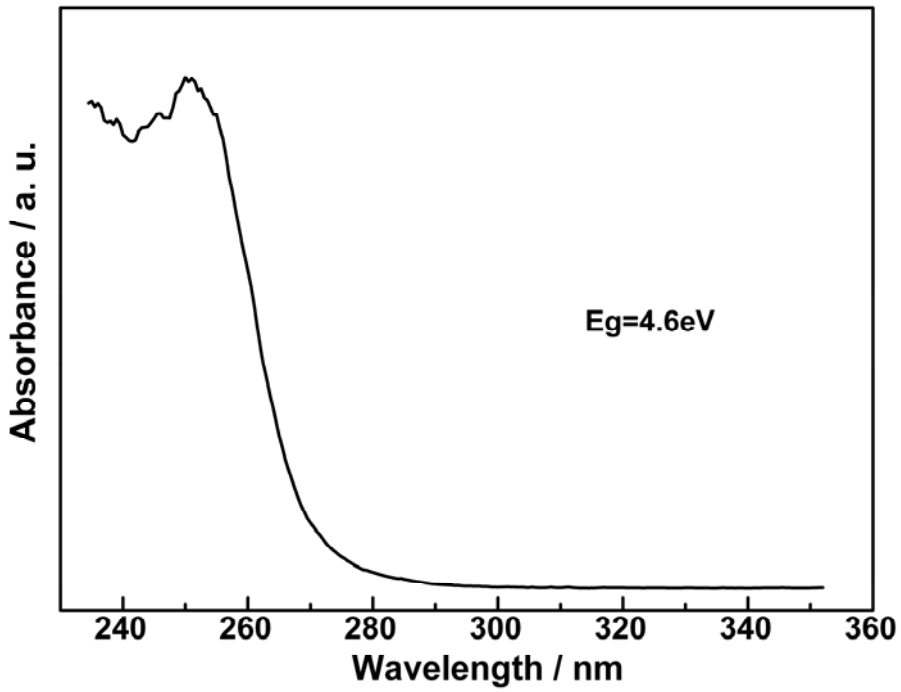

Figure S13. UV-Vis absorption spectrum of the single crystal β-Ga_2O_3 nanoplate .