Hydrothermal synthesis of single-crystal Cr-doped SrTiO₃ for efficient visible-light responsive photocatalytic hydrogen evolution

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Abstract

Visible-light-driven Cr-doped SrTiO₃ nanocubes were successfully synthesized by hydrothermal method in alkaline KOH conditions. X-ray diffraction spectroscopy (XRD), Raman spectra, x-ray photoelectron spectroscopy (XPS), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) were used to characterize the samples, and the Cr-doped SrTiO₃ possess cubic morphology with about 30–50 nm in size, and single-crystal feature. In addition, the Cr-doped SrTiO₃ extended light-harvesting properties to visible-light region which was testified by UV–vis absorption spectra, and excellent charge transfer and separation efficiency were approved by photo fluorescence spectra (PL), electrochemical impedance spectroscopy (EIS) and photocurrent response measurements. Among the synthesized photocatalysts, SrTiO₃ nanocubes doped with 2% Cr by molar ratio exhibits the highest photocatalytic activity, achieving 11.66 μmol of H₂ evolution during 5 h visible-light irradiation. This study provides a facile and effective way to enhance the performance of SrTiO₃-based photocatalysts.

1. Introduction

With the massive and continuous consumption of limited traditional fossil fuels resources, sustainable and clean energy sources are urgent to be exploited [1]. Hydrogen (H₂) is deemed as the most ideal fuel for releasing H₂O only, and possessing high calorific value of 142.4 kJ g⁻¹, which is about 2.75 and 5 times higher than hydrocarbon and alcohol-based fuels, respectively [2, 3]. Among the various methods for H₂ production, semiconductor photocatalysis technology is believed as a potential manner, because of the entire process involves environmentally friendly procedures, water as sources and solar energy as power for instance.

Strontium titanate (SrTiO₃), a typical perovskite oxide semiconductor with excellent chemical and optical stability, is considered as promising photocatalyst [4]. However, the wide band-gap energy (3.2 eV) restricts its photocatalytic performance, which could be responsive to the ultraviolet light range [5]. Therefore, modifications to SrTiO₃ are widely attempted to narrow the band-gap and thus enlarge its light-harvesting region. Chemical substitutional doping has been proposed as an effective method to modify SrTiO₃ [6], in order to obtain promoted photocatalytic activities, such as F-doped SrTiO₃ [7], Eu-doped SrTiO₃ [8], Al-doped SrTiO₃ [9], Cr-doped SrTiO₃ [10]. Among the abundant doped SrTiO₃ materials, Cr-doped SrTiO₃ exhibits variable properties caused by the suitable ionic radius of Cr³⁺ (0.615 Å), which lies between those of Sr²⁺ (1.18 Å) and Ti⁴⁺ (0.605 Å) ions [11, 12].

Another pivotal issue should be addressed that, both high crystallinity and specific surface area of semiconductor materials are essentially required for efficient photocatalysis, which the two demands are hardly achieved simultaneously [13]. Conventional solid-state reactions were commonly used to synthesize Cr-doped SrTiO₃, which lead to a high charge carriers mobility, while result in tiny specific surface area on the contrary [14, 15]. Wet-chemistry method were frequently employed to fabricate Cr-doped SrTiO₃ with small size, such as...
2. Experimental

2.1. Materials

AEROXIDE TiO2 P25 (80% anatase and 20% rutile) was obtained from Evonik industries. Potassium hydroxide (KOH, AR, 90%), chromium (III) nitrate nonahydrate (Cr(NO3)3·9H2O, AR, 99%) and chloroplatinic acid hexahydrate (H2PtCl6·6H2O, AR, Pt ≥ 37.5%) were purchased from Macklin Inc. Strontium nitrate (Sr(NO3)2, AR, 99.5%) and triethanolamine (C6H15NO3, TEOA, AR) were all analytical grade and purchased from Sinopharm Chemical Reagent Co., Ltd.

2.2. Synthesis of SrTiO3 nanocubes

In a typical procedure, 6.25 mmol Sr(NO3)2 (1.322 g) was dissolved in 10 ml deionized water with stirring until it transformed to a transparent solution. Meanwhile, x mol of KOH powders were dissolved in 20 ml of deionized water under continuous stirring. After the KOH aqueous solution was cooling down, 5 mmol P25 (400 mg) was slowly added in. Subsequently, the Sr(NO3)2 solution and P25/KOH suspension were mixed together under violently stirring for 1 h. Afterwards, the resulting precursor mixture was transferred into 50 mL stainless-steel Teflon-lined autoclave, and placed in an oven which was held at 180 °C for 12 h. After cooling down to room temperature naturally, the resultant was collected and washed with deionized water and ethanol for several times, and subsequently dried at 80 °C in the air. The SrTiO3 samples were labelled as STO-x according to the molar amount of KOH used in the experiments, and the value of x was set as 0.12, 0.24, 0.36 and 0.48, respectively.

2.3. Synthesis of Cr-doped SrTiO3 nanocubes

The procedure for the synthesis of Cr-doped SrTiO3 nanocubes was similar to that for pristine SrTiO3 nanocubes, while the amount of KOH was fixed at 0.36 mol. Meanwhile, the Cr3+ concentration in the SrTiO3 nanocubes was 0.1%, 0.5%, 1.0%, 2.0%, 5.0% and 10.0%, respectively, and the samples were labelled as CSTO-y (y = 0.1, 0.5, 1.0, 2.0, 5.0 and 10.0).

2.4. Characterization

X-ray diffraction (XRD) was performed on a D8 ADVANCE x-ray diffractometer (BRUKER, Germany) fitted with Cu Kα radiation over the 2θ ranges of 10–70° (4°·min⁻¹). Raman spectra were acquired by a LabRAM HR800 Raman spectroscopy (Horiba Jobin Yvon, USA), using Ar laser (532 nm) as excitation source. Scanning electron microscopy (SEM) images were obtained by JSM-7610F field-emission SEM (JEOL, Japan), while transmission electron microscopy (TEM) on a Tecnai G2 F20 S-TWIN microscope (FEI, USA). X-ray photoelectron spectroscopy (XPS) analysis was performed on an ESCALAB 250XI (Thermo Fisher, USA) with Al Kα (1486.6 eV) irradiation. Optical absorption was obtained using Cary 5000 ultraviolet-visible (UV–vis) spectrophotometer (Agilent, USA) with an integrating sphere, employing BaSO4 (SP, Maclin Co., Ltd) as a reference. The photoluminescence (PL) emission spectra of as-prepared samples were detected on a FLS920 fluorescence spectrometer (Edinburgh, UK), using the excitation wavelength of 365 nm.

2.5. Photocatalytic hydrogen evolution performance

Photocatalytic reactions for hydrogen (H2) evolution were carried out in a CEL-SPH2N water splitting system (China Education Aulight Co., LTD) with a 300 Xe-arc lamp (CEL-HXF 300) attached with a cut-off filter (λ ≥ 420 nm). Pt (1 wt%) was loaded on the surface of the samples as co-catalyst via the photodeposition method [20], using H2PtCl6 aqueous solution as precursors. Pt-modified photocatalyst (100 mg) was added to 100 mL of water containing 10 mL of triethanolamine as sacrificial agent. A GC-7920 gas...
chromatograph equipped with a TCD detector and a 5 Å molecular sieve packed column was used to detect evolution of hydrogen on line.

2.6. Photoelectrochemical measurements

The electrochemical and photoelectrochemical measurements were performed with CHI760E electrochemical workstation (Chenhua, Shanghai), which was equipped with a conventional three-electrode system consisted of a modified FTO working electrode, a Pt counter electrode, and a Ag/AgCl reference electrode. FTO was coated with 15 mg of photocatalysts in 0.5 wt% chitosan solution (300 μl acetic acid) and dried. The exposed geometry area of FTO was controlled to be 1.0 × 0.5 cm. A 300 W Xe-arc lamp with a 420 nm cut-off filter served as a light source and Na2SO4 aqueous solution (0.5 M) as electrolyte. The photocurrent response was recorded as switching the light source (on or off) every 30 s intervals at a bias of 0.2 V.

Results and discussion

The crystallographic structure and phase composition of the as-prepared samples were detected by XRD. As shown in figure 1(a), the characteristic diffraction peaks at 22.78°, 32.42°, 39.98°, 46.48°, 57.79° and 67.80° could be indexed to the (100), (110), (111), (200), (211) and (220) planes of cubic SrTiO3 (PDF #35-0734) [21]. Moreover, the XRD patterns shows quite a similar feature and the crystalline size was calculated via Scherrer formula. As listed in table 1, it indicates the crystalline size is slightly decreasing as the increasing addition of KOH, and then increasing when KOH is excessive, owing to a nonuniform crystal growth. The XRD patterns of as-prepared Cr-SrTiO3 maintained an almost identical feature as pure SrTiO3, as shown in figure 1(b), implying that Cr3+ doping did not change the structure of SrTiO3 even at 10% concentration of Cr dopants. Figure 1(c) shows a magnified patterns of the diffraction peaks for (110) planes, indicating a slight shift to low-angle after Cr3+ doping, and thus Cr3+ ions probably substituted Ti4+ ions in this case, owing to a ionic radius relations as Sr2+ (1.18 Å) > Cr3+ (0.615 Å) > Ti4+ (0.605 Å) [22].

Figure 1(d) shows the Raman spectra of pristine STO and CSTO, and it could be observed that the band at 792 cm⁻¹ becomes more evident, indicating Cr substituted for Ti sites which demonstrate deduction above [23]. Meanwhile, there are scarcely any bands at about 864 cm⁻¹, reflecting no Cr6+ ions could be found in the STO lattice [24], and Cr dopants are in the Cr3+ form existing in the STO matrix.

Figures 2(a)–(d) shows SEM images of pristine SrTiO3 synthesized via hydrothermal process with various KOH as mineralizer. All the undoped SrTiO3 exhibit a similar morphology of cubic shape with 90° corners, and
the average side-length about 40 nm, except that STO (0.12) possesses an analogous morphology of P25 with irregular shapes. Moreover, distinctions could also be identified that the edges seem to be explicit with the increasing addition of KOH, which accelerates the mineralization of SrTiO₃, while extra particles could be found on the surface of STO (0.48). Figures 2(e) and (h) shows the HRTEM images of STO (0.36) and CSTO-2, indicating vague edges of CSTO-2 compared to STO (0.36), which is attributed to the inhibited crystal growth affected by Cr³⁺ doping. Moreover, the selected-area electron diffraction (SAED) patterns of STO (0.36) and CSTO-2 (figures S1 and S2 is available online at stacks.iop.org/MRX/7/015047/mmedia) display bright spots, indicating the single-crystal nature. In addition, both the FFT patterns of STO (0.36) and CSTO-2 (figures 2(f) and (i)) reveal the identical feature of cubic structure, while the (110) lattice spacing of CSTO-2 (0.279 nm) (figures 2(g) and (j)) is slightly larger than that of STO (0.36) (0.276 nm). The results also prove Cr³⁺ is doped into the SrTiO₃ lattice by substitution Ti, which agrees with the XRD analysis above. In addition, the results reveal that the CSTO-2 has a surface area of 31.20 m²·g⁻¹, as shown in figure 2(k), which is slightly larger than that of STO (0.36) (28.15 m²·g⁻¹), owing to a smaller particle size of CSTO-2 by Cr³⁺ doping.

The chemical state of the pristine and Cr-doped SrTiO₃ was further examined by XPS technology. As shown in figure 3(a), the peaks centred at 134.23 and 132.48 eV are attributed to Sr 3d₃/₂ and Sr 3d₅/₂, respectively [25]. Splitting of Ti 2p (figure 3(b)) into to peaks at 463.93 and 458.18 eV denote the Ti 2p₃/₂ and 2p₁/₂, respectively [26]. The O 1 s spectra (figure 3(c)) could be deconvoluted into three peaks at 529.42, 531.84, and 532.95 eV, which is assigned to the lattice oxygen (Oₗ), surface hydroxyl groups (OₗOH) and adsorbed oxygen (Oₐds) [27]. Compared to that of STO, the high-resolution XPS spectra of Sr 3d, Ti 2p and O 1 s for CSTO-2 display hardly binding energy shifts, indicating no additional phases occur, which is consistent with the XRD results above. As shown in figure 3(d), the core level spectra of Cr 2p exhibit two peaks of 586.33 (Cr 2p₃/₂) and 576.93 eV (Cr 2p₁/₂), which are assigned to the Cr³⁺ state as previous reports, demonstrating Cr³⁺ doped SrTiO₃ was successfully obtained [28, 29].

The optical properties of the pristine SrTiO₃ and Cr³⁺-doped SrTiO₃ are investigated by UV–vis absorption spectra, as shown in figures 4(a), (b). It could be seen that almost no distinction among the pristine STO samples in light-absorption abilities, because of a similar crystallinity and size as demonstrated by XRD and SEM results.

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**Table 1.** Full width at half maximum (FWHM) and crystalline size of the as-prepared STO by various KOH calculated from Debye–Scherrer formulation.

| Samples  | FWHM | Crystalline size (nm) |
|----------|------|----------------------|
| STO(0.12) | 0.330 | 26.3 |
| STO(0.24) | 0.352 | 24.5 |
| STO(0.36) | 0.371 | 23.1 |
| STO(0.48) | 0.360 | 23.9 |

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**Figure 2.** SEM images of as-prepared undoped SrTiO₃ by various KOH addition (a) 0.12 mol (b) 0.24 mol (c) 0.36 mol (d) 0.48 mol. HRTEM images of (e) STO(0.36) and (h) CSTO-2 with corresponding (f, i) FFT patterns and (g, j) inverse FFT images for squared areas in the inset of (e) and (h), respectively. (k) N₂ adsorption–desorption plots of STO (0.36) and CSTO-2.
Figure 3. High-resolution XPS spectra for the STO and CSTO-2 (a) Sr 3d (b) Ti 2p (c) O 1s (d) Cr 2p.

Figure 4. UV–vis absorption spectra of (a) the pristine SrTiO$_3$ synthesized by various KOH and (b) Cr$^{3+}$-doped SrTiO$_3$ with different amount Cr dopants. (c) Valence band XPS of STO and CSTO-2. (d) Band alignment of pristine SrTiO$_3$ and Cr$^{3+}$-doped SrTiO$_3$. 
above. As shown in the inset of figure 4(a), the colour of the pristine SrTiO$_3$ is white, while those of Cr$^{3+}$-doped SrTiO$_3$ change from light to dark yellowish, and the UV–vis absorption spectra (figure 4(a)) exhibit a red-shift for the Cr$^{3+}$-doped SrTiO$_3$ compared to STO. Besides, a weak absorption tail (Urbach tail) in the visible-light region occurs for all the Cr$^{3+}$-doped SrTiO$_3$, which is attributed to the formation of mid-gap states caused by Cr dopants [30, 31]. In addition, Tauc plot of photon energy ($h\nu$) versus $(Ah\nu)^{1/2}$ for the indirect allowed transition SrTiO$_3$-based semiconductors [32–34], is used to calculate the band-gap energy of the samples [25]. As shown in figure 4(b), the calculated band-gap values for STO and CSTO-2 are 3.16 and 3.01 eV, respectively. The valence band (VB) position of STO and CSTO-2 were measured by VB-XPS. As presented in figure 4(c), the the VB potentials ($E_{VB}$) of STO and CSTO-2 are calculated to be 2.12 and 1.61 eV, by extrapolating the leading edge of the VB-XPS plots to its intercepts with the backgrounds near Fermi level [23]. Provided the obtained band-gap energy by Tauc’s results, the conduction band (CB) potential ($E_{CB}$) of the STO and CSTO-2 are calculated to be $−1.04$ and $−1.40$ eV. Therefore, the band alignments of the STO and CSTO-2 are illustrated in figure 4(d), and reduced band-gap of CSTO-2 is caused by occupied Cr$^{3+}$ level, which is about 1.0 eV higher than the valence band top formed by Ti 3d [35], while the elevated conduction band is also preferable to the reduction of H$^+$ ions to H$_2$ generation.

The H$_2$ evolution experiment was carried out to investigate the photocatalytic activities of the Cr-doped SrTiO$_3$ under visible-light irradiation ($\lambda \geq 420$ nm) with 1 wt% Pt as co-catalyst. Figure 5(a) shows the photocatalytic reactions of H$_2$ evolution up to 5 h, while no H$_2$ was detected when pristine STO(0.36) was used as photocatalyst because it could not response to visible-light. When Cr-doped SrTiO$_3$ was utilized, an apparent H$_2$ evolution was observed while the photocatalytic performance enhanced as the amount of Cr$^{3+}$ dopants increased. The CSTO-2 exhibits the highest photocatalytic activity with 11.66 $\mu$mol of H$_2$ evolution during 5 h visible-light irradiation. Additionally, further increasing the amount of Cr$^{3+}$ dopants lead to deteriorate the photocatalytic performance, which might be attributed to recombination centre of photo-induced charges caused by excessive dopants. Therefore, a suitable content of Cr$^{3+}$ dopants is crucial for the enhancement of the photocatalytic activity for Cr-doped SrTiO$_3$. Furthermore, CSTO-2 possess excellent stability during the photocatalytic reactions, which was testified by XRD exhibiting no structural or compositional change after H$_2$ evolution process, as shown in figure S3.

The fluorescence spectra with the excited wavelength of 365 nm was conducted to study the separation efficiency of photo-induced charge carriers, as shown in figure 5(b) [36]. It could be seen that the STO(0.36) can be excited, and the fluorescence intensity is the highest among the tested samples, which means the well-crystallized pristine STO is able to generate considerable amount of excitons, while cannot be utilized owe to the wide band-gap. The intensity of CSTO-2 is weaker compared to that of CSTO-10, suggesting a more efficient
charge separation due to the fact that excessive doping would be disadvantageous for the migration of photo-induced holes across the discrete impurity levels [29].

Figure 5(c) shows the photocurrent for the CSTO-2 during on/off cycles under full-spectrum illumination, which is about twice higher than that of CSTO-10, and the high response could be attributed to the enhanced charge transfer and separation efficiency [37]. Moreover, the STO also possesses photocurrent response to full-spectrum irradiation, which is even higher than that of CSTO-10, the phenomenon could be assigned to the contribution of the ultraviolet section, and suggest deteriorative migration of charge carriers owe to declined crystallinity on the other hand, agreed with the XRD result previously.

In addition, the electrochemical impedance spectra (figure 5(d)) was conducted for the purpose of further exploration about the migration and separation of photo-induced charges. The CSTO-2 possesses the smallest arc radius of Nyquist plots, indicating the most rapid charge migration and efficient separation [38, 39], which are in accordance with the above analysis. These results confirm that suitable Cr$^{3+}$ doping improve the photo-induced charge transfer and separation, and lead to the enhancement of photocatalytic H$_2$ evolution.

Based on the experimental and characterization results above, the possible mechanism for enhanced H$_2$ evolution by Cr-doped SrTiO$_3$ nanocubes could be described as figure 6. Synthesized via hydrothermal process and tailored the mineralizer KOH, single-crystal SrTiO$_3$ nanocubes with uniformed morphology and excellent charge mobility. However, the undoped SrTiO$_3$ crystals could not response to visible-light irradiation, and thus Cr dopants were induced in the hydrothermal procedure. The Cr doping causes lattice distortion by substituting the Ti sites on one hand, although the single-crystal feature is not changed, and introduces impurity energy levels in the band-gap of SrTiO$_3$ nanocubes. Therefore, the deduced band-gap energy and excellent charge carriers mobility lead to an enlarged light-harvesting properties, and enhanced charge conductivity and separation efficiency, as a result improved H$_2$ evolution under visible-light illumination is observed.

Conclusion

In summary, SrTiO$_3$ and Cr-doped SrTiO$_3$ nanocubes were synthesized via hydrothermal method, and the effects of Cr dopants were investigated in the range of 0.1%–10% range. Structural analysis indicates that the Cr dopants are most able to substitute Ti sites in the SrTiO$_3$ lattice, while do not alter the morphology and crystal features. Among all the samples, CSTO-2 exhibits the best photocatalytic performance in H$_2$ evolution, because of efficient charge migration and separation owing to a single-crystal feature, and enlarged light harvesting capability caused by impurity levels.

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