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## A Ta<sub>3</sub>N<sub>5</sub>/BN composite for enhanced photocatalytic water splitting

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**Narrow-bandgap Ta<sub>3</sub>N<sub>5</sub> nanorods were supported on a BN substrate to produce a composite photocatalyst using NH<sub>4</sub>Cl-based vacuum nitridation. The charge separation efficiency of Ta<sub>3</sub>N<sub>5</sub> was effectively enhanced due to electron transfer from Ta<sub>3</sub>N<sub>5</sub> to the BN substrate, boosting both the photocatalytic water oxidation and Z-scheme overall water splitting reactions.**

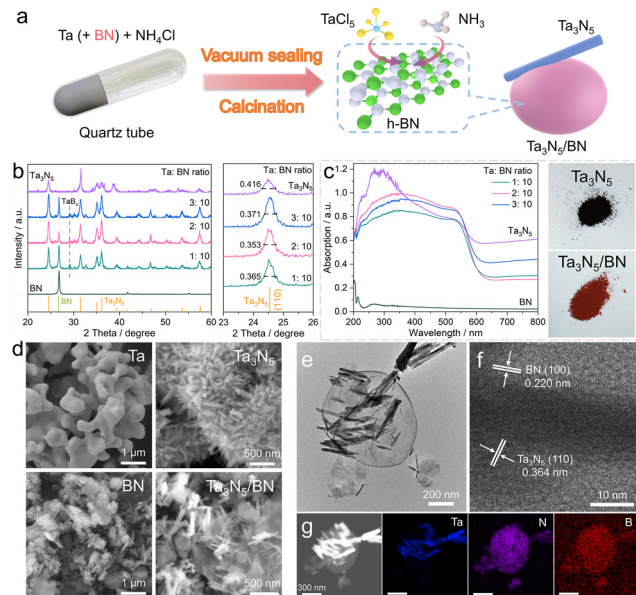
Solar energy conversion into clean hydrogen resources through a photocatalytic process is a promising strategy for sustainable energy systems.<sup>1,2</sup> However, the development of efficient photocatalysts is constrained by limitations in light harvesting, especially in the visible region that makes up 45% of the solar spectrum,<sup>3</sup> and photon conversion efficiency, restrained by serious bulk and surface charge recombinations.<sup>4–8</sup> Ta<sub>3</sub>N<sub>5</sub> has emerged as a particularly promising photocatalyst due to its bandgap of only 2.1 eV. The conduct band minimum (CBM) and valence band maximum (VBM) of Ta<sub>3</sub>N<sub>5</sub> are composed of Ta 5d and N 2p orbitals, respectively, thermodynamically allowing for both photocatalytic and photoelectrochemical water redox reactions.<sup>9,10</sup> In 2002, Hitoki *et al.* reported photocatalytic water oxidation on Ta<sub>3</sub>N<sub>5</sub> in the presence of an electron acceptor.<sup>11</sup> However, subsequent studies have demonstrated that the performance of Ta<sub>3</sub>N<sub>5</sub> is fundamentally limited by issues directly stemming from the synthesis method. Specifically, Ta<sub>3</sub>N<sub>5</sub> is typically prepared through an NH<sub>3</sub>-based thermal nitridation process, which meets challenges including the formation of low-valence metal ions and O<sub>N</sub> anti-site defects within the crystal structure. These intrinsic defects act as recombination centers that severely suppress the charge separation efficiency.<sup>12,13</sup> Furthermore, high temperature duration leads to particle agglomeration of the products, exacerbating charge recombination issues. As a consequence of these synthesis-related challenges, Ta<sub>3</sub>N<sub>5</sub> exhibits unsatisfactory photon conversion efficiencies in photocatalytic reactions.<sup>14–16</sup>

Nanostructural engineering, by precisely controlling the morphology and architecture of oxide precursors, has emerged as an effective strategy to improve the charge separation efficiency in Ta<sub>3</sub>N<sub>5</sub> photocatalysts by reducing the charge transport distances and extending the carrier lifetimes.<sup>17</sup> Ma *et al.* applied a Ta<sub>2</sub>O<sub>5</sub> and Na<sub>2</sub>CO<sub>3</sub> mixture to produce a NaTaO<sub>3</sub>/Ta<sub>2</sub>O<sub>5</sub> composite, which was then used as a precursor for thermal ammonolysis. This method suppressed Ta<sub>3</sub>N<sub>5</sub> agglomeration and enhanced the crystallinity.<sup>14</sup> Wang *et al.* prepared Ta<sub>2</sub>O<sub>5</sub>-coated Pt/SiO<sub>2</sub> nanoparticles for the fabrication of hollow Ta<sub>3</sub>N<sub>5</sub> nanoshells, and subsequently loaded CoO<sub>x</sub> onto the outer surface of Ta<sub>3</sub>N<sub>5</sub> with Pt left on the inner shell after removing SiO<sub>2</sub>.<sup>18</sup> The nanoshell structure not only provided a structural framework for efficient charge transport but also facilitated directional carrier extraction by spatially separating the reduction and oxidation cocatalysts, thus boosting charge separation. Wang *et al.* applied KTaO<sub>3</sub> as a vaporous Ta resource to produce Ta<sub>3</sub>N<sub>5</sub> nanorod single crystals and accelerate heating of the ammonium flow, which reduced the duration of the nitridation process. The highly crystallized Ta<sub>3</sub>N<sub>5</sub> nanorods with fewer defects realized one-step excitation in the overall water splitting reaction under visible light illumination.<sup>9</sup> Nanostructured precursors enable controlled Ta<sub>3</sub>N<sub>5</sub> particle morphology, effectively decreasing agglomeration and shortening the carrier transport distances, while structuring the catalyst/host interface promotes more efficient carrier transport, enhancing the charge separation efficiency and thereby unlocking Ta<sub>3</sub>N<sub>5</sub>'s potential for photocatalysis.

Our group has developed a NH<sub>4</sub>Cl-based vacuum nitridation method to synthesize transition metal (oxy)nitrides such as a Ta<sub>3</sub>N<sub>5</sub>/Ta thin film,<sup>19</sup> TaON powders<sup>20</sup> and ZnO:GaN nanoparticles.<sup>21,22</sup> For the preparation of the Ta<sub>3</sub>N<sub>5</sub>/Ta thin film, only a Ta wafer and NH<sub>4</sub>Cl were used as precursors. Upon heating, in the first step, NH<sub>3</sub> and HCl are produced from the decomposition of NH<sub>4</sub>Cl. The metallic Ta reacts with HCl to produce a gaseous TaCl<sub>5</sub> intermediate, which subsequently reacts with NH<sub>3</sub> to form Ta<sub>3</sub>N<sub>5</sub> particles on the surface of the Ta plate. The high permeability of the gaseous TaCl<sub>5</sub> and NH<sub>3</sub>

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**Fig. 1** (a) Illustration of the synthesis of the  $\text{Ta}_3\text{N}_5/\text{BN}$  composite. (b) XRD patterns of the BN precursor, bulk  $\text{Ta}_3\text{N}_5$ , and  $\text{Ta}_3\text{N}_5/\text{BN}$  samples prepared from different Ta:BN ratios. (c) UV-vis DRS of the BN,  $\text{Ta}_3\text{N}_5$ , and  $\text{Ta}_3\text{N}_5/\text{BN}$  powders, as well as digital images of the  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$  ( $\text{Ta}:\text{BN} = 2:10$ ) samples. (d) SEM images of the Ta and BN precursors and corresponding  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$  products. (e) TEM, (f) HRTEM, and (g) dark field image and EDS mapping images of the  $\text{Ta}_3\text{N}_5/\text{BN}$  ( $\text{Ta}:\text{BN} = 2:10$ ) sample. The referenced patterns of BN and  $\text{Ta}_3\text{N}_5$  in (b) are ICDD-00-045-1171 and ICDD-04-004-4564, respectively.

reactants not only leads to rapid crystallization of the solid product but also prevents  $\text{Ta}_3\text{N}_5$  from forming reduced Ta species by limiting exposure to an  $\text{NH}_3$  atmosphere.

Herein, we introduce two-dimensional BN as a substrate for the growth of  $\text{Ta}_3\text{N}_5$  nanoparticles in an  $\text{NH}_4\text{Cl}$ -based nitridation process. The involvement of BN in the precursor led to improved crystallinity and dispersion of the  $\text{Ta}_3\text{N}_5$  particles. Reductive photodeposition of Ag and Rh suggested that electrons were transferred from  $\text{Ta}_3\text{N}_5$  to BN under irradiation. Such directional charge migration greatly enhanced the photocatalytic activity of  $\text{Ta}_3\text{N}_5$  for water splitting, highlighting the benefits of photocatalyst-substrate interaction.

The  $\text{Ta}_3\text{N}_5$  bulk and  $\text{Ta}_3\text{N}_5/\text{BN}$  composite were synthesized by vacuum sealing a mixture of  $\text{NH}_4\text{Cl}$  and metallic Ta powder in a quartz tube with or without adding BN (Fig. 1a, with details shown in the SI). For  $\text{Ta}_3\text{N}_5/\text{BN}$ , the atomic ratios of Ta:BN were set as 1:10, 2:10 and 3:10, respectively. Fig. 1b displays the X-ray diffraction (XRD) patterns of the powders prepared with different Ta:BN ratios. All the BN-containing samples exhibit patterns of mixed BN and  $\text{Ta}_3\text{N}_5$  peaks, with a weak signal at  $\sim 29^\circ$  assigned to  $\text{TaB}_x$  impurities. The majority of the peaks of  $\text{Ta}_3\text{N}_5/\text{BN}$  are consistent with the standard  $\text{Ta}_3\text{N}_5$  patterns, indicating the high purity of  $\text{Ta}_3\text{N}_5$  in the obtained samples. No signals of metallic Ta are observed in the products. Additionally, the full-width half-maximum (FWHM) of the  $\text{Ta}_3\text{N}_5$  (110) diffraction peaks in the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples is lower than that of  $\text{Ta}_3\text{N}_5$ , indicating enhanced crystallinity of  $\text{Ta}_3\text{N}_5$

when BN is present during synthesis. Fig. 1c shows the UV-vis diffuse reflectance spectroscopy (DRS) of the BN,  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$  powders. The BN precursor exhibits negligible UV-visible light absorption due to its wide bandgap of 5.1 eV. The  $\text{Ta}_3\text{N}_5$  reference powder shows a broad absorption up to 600 nm corresponding to its bandgap (2.1 eV). The observed dark brown color, as opposed to dark red, can be attributed to elevated absorption in the near-infrared region. All of the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples exhibit smooth band-to-band absorption features typical of  $\text{Ta}_3\text{N}_5$ , revealing that the optical properties of  $\text{Ta}_3\text{N}_5$  did not change with the introduction of BN into the precursor, as corroborated by the Tauc plots in Fig. S1. Compared to  $\text{Ta}_3\text{N}_5$ , the relatively lower background levels above 600 nm for the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples suggest less absorption associated with intragap defect states. Notably, the Ta:BN = 2:10 sample achieves the strongest light absorption, showing that photon absorption does not linearly increase with a higher Ta ratio in the precursor.

The scanning electron microscopy (SEM) images (Fig. 1d) display the morphology of the precursors and the nitridation products. Without incorporating BN during nitridation, large Ta precursor particles are converted into aggregated  $\text{Ta}_3\text{N}_5$  nanorods. In the presence of BN, the nitridation products show more uniformly distributed and well-isolated  $\text{Ta}_3\text{N}_5$  particles dispersed on the BN substrate. The SEM images of the different  $\text{Ta}_3\text{N}_5/\text{BN}$  samples (Fig. S2) in back scattered electron (BSE) mode show that the dispersity of the  $\text{Ta}_3\text{N}_5$  nanorods in the Ta:BN = 2:10 sample is higher than that in the 1:10 and 3:10 samples. This demonstrates that the Ta content effectively impacts the growth of  $\text{Ta}_3\text{N}_5$  particles, highlighting  $\text{NH}_4\text{Cl}$ -based vacuum nitridation as a potential method for constructing regulated nanostructures. The transmission electron microscopy (TEM) image of the Ta:BN = 2:10 sample (Fig. 1e) shows  $\text{Ta}_3\text{N}_5$  nanorods with lengths around hundreds of nanometers lying on the BN substrate. The BN structure remains unchanged after the high-temperature nitridation. The TEM image clearly demonstrates that the  $\text{Ta}_3\text{N}_5$  particles in the obtained samples adopt a typical nanorod morphology, which is similar to the  $\text{Ta}_3\text{N}_5$  particles prepared directly from  $\text{TaCl}_5$  and  $\text{NH}_3$  precursors.<sup>23</sup> The high-resolution TEM (HRTEM) image in Fig. 1f exhibits the interface of the  $\text{Ta}_3\text{N}_5$  and BN particles. The close stacking of the 0.364 nm lattice fringes assigned to the (110) facet of  $\text{Ta}_3\text{N}_5$  and the 0.220 nm lattice fringes assigned to the BN (100) facet suggests the growth of  $\text{Ta}_3\text{N}_5$  on the BN substrate. Fig. 1g shows the TEM dark field image and energy dispersive X-ray spectroscopy (EDS) of the Ta:BN = 2:10 sample. The overlapped N and Ta signals further confirm the nanorod morphology of  $\text{Ta}_3\text{N}_5$  on the BN substrate.

XPS analysis was carried out on the  $\text{Ta}_3\text{N}_5$ , BN and  $\text{Ta}_3\text{N}_5/\text{BN}$  particles. The well-defined double peaks at 25.2 and 27.1 eV (Fig. 2a) assigned to Ta 4f<sub>7/2</sub> and 4f<sub>5/2</sub> in  $\text{Ta}_3\text{N}_5$  reveal that the  $\text{NH}_4\text{Cl}$ -based nitridation suppresses the formation of reduced Ta species. For the  $\text{Ta}_3\text{N}_5/\text{BN}$  particles, additional double peaks are observed at high field shift, which are supposed to be  $\text{TaO}_x$  impurities.<sup>24</sup> The O 1s spectrum (Fig. S3) reveals the existence of crystalline O species in  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$ , suggesting



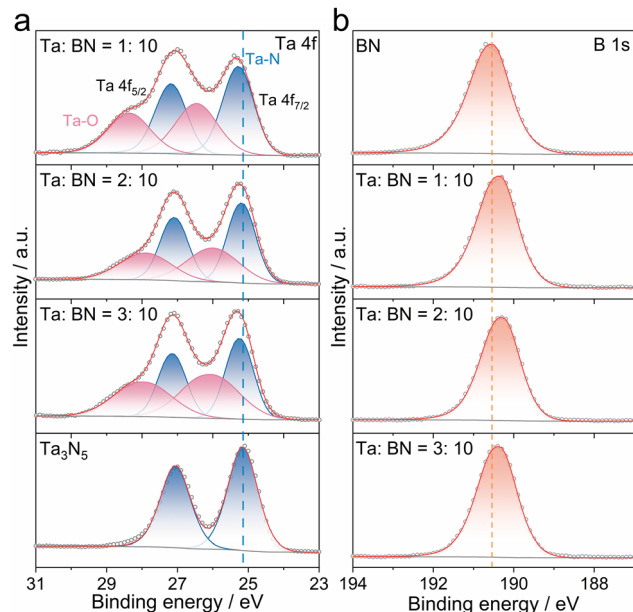


Fig. 2 XPS of the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples prepared from different precursor ratios, including  $\text{Ta}_3\text{N}_5$  and BN for reference. (a) Ta 4f and (b) B 1s spectra.

surface coverage of the  $\text{TaO}_x$  species. Besides, compared with  $\text{Ta}_3\text{N}_5$ , a slightly higher binding energy of the  $\text{Ta}^{5+}$  signals in Ta-N of the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples reflects a possible electron transfer from  $\text{Ta}_3\text{N}_5$  to BN. Relative to BN, the lower shift in B 1s binding energy for the  $\text{Ta}_3\text{N}_5/\text{BN}$  samples also suggests an interfacial electron transfer between the two components (Fig. 2b).

To verify electron transfer between  $\text{Ta}_3\text{N}_5$  and BN under illumination, visible light-driven photoreduction of  $\text{Ag}^+$  ions was performed, and Ag particles were loaded on the  $\text{Ta}_3\text{N}_5/\text{BN}$  composite photocatalyst. The 0.200 nm lattice fringes attributed to the Ag (103) facet are observed on both the BN and  $\text{Ta}_3\text{N}_5$  particles. The EDS mappings in Fig. 3c clearly distinguish the Ag nanoparticles loaded away from the  $\text{Ta}_3\text{N}_5$  nanorods. Considering that BN barely absorbs visible light, this indicates that photogenerated electrons are partially transferred from  $\text{Ta}_3\text{N}_5$  to BN, as illustrated in the structure diagram of Fig. 3c. As further evidence, by using  $\text{Rh}^{3+}$  as a precursor, Rh nanoparticles can also be reductively photodeposited on the BN substrate (Fig. S4). Mott-Schottky analysis and valence spectra (Fig. S5) reveal that the Fermi level of BN is closely aligned with that of  $\text{Ta}_3\text{N}_5$ . Such energetic alignment is beneficial for electron transfer from  $\text{Ta}_3\text{N}_5$  to BN.

Transient absorption spectroscopy was employed to investigate the electron transfer from  $\text{Ta}_3\text{N}_5$  to BN under light excitation and in solution. The pump and probe lights were set at 355 and 580 nm, respectively. For BN alone (Fig. S6), no meaningful transient absorption signal is observed as its band gap exceeds the pump light wavelength. In contrast, both  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$  show a weak negative absorption at 580 nm (Fig. 3d). To assign this signal, L-ascorbic acid is added as a hole scavenger. Here, the intensity of the negative absorption increases for both samples, confirming that this signal is associated with

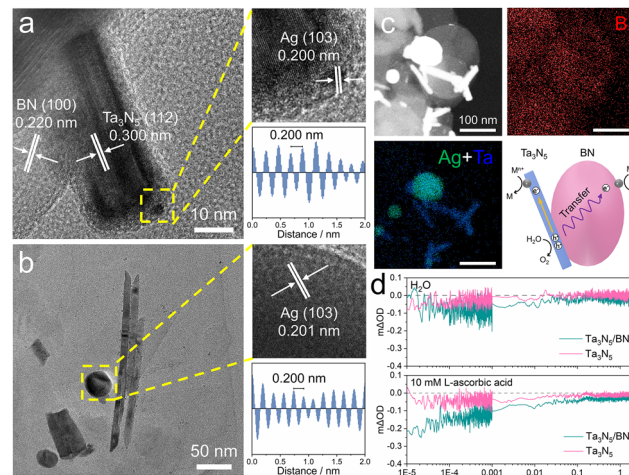


Fig. 3 HRTEM images and lattice fringe distances of the Ag particles loaded on (a)  $\text{Ta}_3\text{N}_5$  and (b) BN in the  $\text{Ta}_3\text{N}_5/\text{BN}$  particles. (c) Dark field image, EDS mappings and structure diagram of the  $\text{Ta}_3\text{N}_5/\text{BN}$  sample loaded with Ag. (d) Transient absorption spectrum for  $\text{Ta}_3\text{N}_5$  and  $\text{Ta}_3\text{N}_5/\text{BN}$  in water without (top panel) or with L-ascorbic acid (bottom panel). The pump and probe lights in (d) are 355 and 580 nm, respectively. The  $\text{Ta}_3\text{N}_5/\text{BN}$  sample is prepared from a Ta : BN = 2 : 10 ratio.

photogenerated electrons. It is worth noting that the negative absorption for  $\text{Ta}_3\text{N}_5/\text{BN}$  is significantly stronger than that for  $\text{Ta}_3\text{N}_5$ , indicating superior charge carrier separation in the former sample.

The catalyst-substrate electron transfer in  $\text{Ta}_3\text{N}_5/\text{BN}$  is supposed to promote its photocatalytic performance *via* improving electron-hole separation. Fig. 4a displays the activities of the photocatalytic oxygen evolution reaction (OER) on  $\text{Ta}_3\text{N}_5$  or

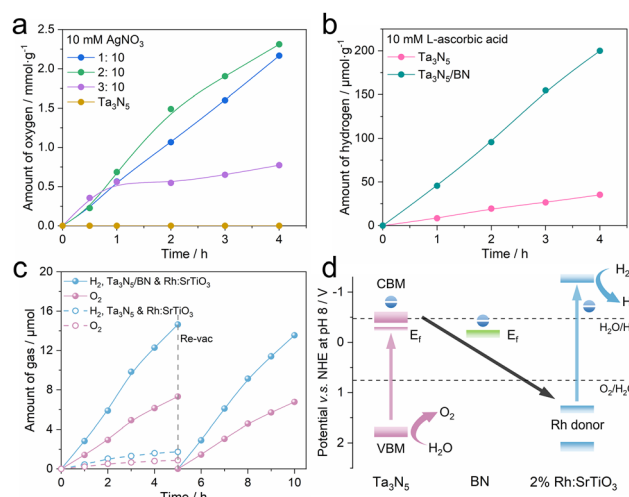
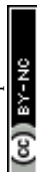


Fig. 4 (a) Time profiles of photocatalytic OER on  $\text{Ta}_3\text{N}_5/\text{BN}$  and bulk  $\text{Ta}_3\text{N}_5$  samples in  $\text{AgNO}_3$  reagent. (b) HER activities of  $\text{Ta}_3\text{N}_5/\text{BN}$  and  $\text{Ta}_3\text{N}_5$  in L-ascorbic acid reagent. (c)  $\text{H}_2$  and  $\text{O}_2$  production from physical oscillation between  $\text{Ta}_3\text{N}_5/\text{BN}$  and 2% Rh:SrTiO<sub>3</sub> at pH 5. (d) Diagram of Z-scheme electron transfer between  $\text{Ta}_3\text{N}_5/\text{BN}$  and Rh:SrTiO<sub>3</sub>. The  $\text{Ta}_3\text{N}_5/\text{BN}$  in (b) and (c) are prepared from a Ta : BN = 2 : 10 ratio and each assessment applied 30 mg of catalyst. 0.75 wt%  $\text{IrO}_2$  and 1 wt% Pt were loaded as cocatalysts for OER and HER, respectively.





Ta<sub>3</sub>N<sub>5</sub>/BN in the presence of AgNO<sub>3</sub> as an electron scavenger. An IrO<sub>2</sub> species is loaded on the catalysts to assist the OER process (Fig. S7). The Ta<sub>3</sub>N<sub>5</sub> nanorods barely produce oxygen due to rapid passivation by Ag coverage on their active sites.<sup>23</sup> However, Ta<sub>3</sub>N<sub>5</sub>/BN exhibits linear oxygen evolution in the early stages, revealing that electron interaction slows down the Ag passivation and preserves the photocatalytic activity. Among the Ta<sub>3</sub>N<sub>5</sub>/BN products, the Ta:BN = 2:10 sample has the highest water oxidation rate. This is likely due to better photon absorption (Fig. 1c) and the more uniform distribution of Ta<sub>3</sub>N<sub>5</sub> nanorods on the BN substrate (Fig. S2) for this sample.

Fig. 4b displays photocatalytic hydrogen evolution (HER) on Ta<sub>3</sub>N<sub>5</sub>/BN (Ta:BN = 2:10) and Ta<sub>3</sub>N<sub>5</sub> assisted with L-ascorbic acid as a hole sacrificial reagent. Pt (1 wt%) is optimized as the HER cocatalyst (Fig. S7). The Ta<sub>3</sub>N<sub>5</sub>/BN composite structure also shows a considerably enhanced hydrogen evolution rate compared with Ta<sub>3</sub>N<sub>5</sub> nanorods, although it remains much lower than the OER rate. This highlights the thermodynamic limitations of Ta<sub>3</sub>N<sub>5</sub>/BN for proton reduction (Fig. S8). To break such limitations, Fig. 4c displays a Z-scheme overall water splitting reaction by physical collision between Ta<sub>3</sub>N<sub>5</sub>/BN and Rh-doped SrTiO<sub>3</sub> (Rh:SrTiO<sub>3</sub>) particles. Under visible light illumination, stoichiometric hydrogen and oxygen evolution over 10 h is realized for Ta<sub>3</sub>N<sub>5</sub>/BN, while the Ta<sub>3</sub>N<sub>5</sub> exhibits significantly lower activity. Fig. 4d represents the band structure of Ta<sub>3</sub>N<sub>5</sub>/BN and Rh:SrTiO<sub>3</sub> determined from Mott-Schottky and bandgap assessment in Fig. S9. It is obvious that transfer of photogenerated electrons from Ta<sub>3</sub>N<sub>5</sub> to BN favors the Z-scheme electron transfer pathway, effectively overcoming the insufficient reduction driving force of Ta<sub>3</sub>N<sub>5</sub>/BN.

In summary, a Ta<sub>3</sub>N<sub>5</sub>/BN composite photocatalyst is synthesized *via* the NH<sub>4</sub>Cl-based vacuum nitridation process. The Ta<sub>3</sub>N<sub>5</sub> component exhibits highly crystallized nanorod morphology during growth on the BN substrate. Photodeposition of Ag metal, chemical state analysis and carrier dynamics assessment confirmed electron transfer between Ta<sub>3</sub>N<sub>5</sub> and BN. Enhanced photocatalytic performance in water splitting emphasizes the promotion of the charge separation efficiency by electron interaction at the catalyst/substrate interface. Our work not only provides a new method for synthesizing highly crystallized Ta<sub>3</sub>N<sub>5</sub> nanorods but also demonstrates an efficient composite structure for enhancing the photocatalytic performance of this promising photocatalyst.

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## Conflicts of interest

There are no conflicts to declare.

## Data availability

The data supporting this article have been included as part of the supplementary information (SI). Supplementary information is available. The detailed SEM images, XPS spectra, experiment conditions were displayed in supplementary information. See DOI: <https://doi.org/10.1039/d5cc03833b>.

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