

## SUPPORTING INFORMATION

### **Synthesis of three-dimensional AlN-Si<sub>3</sub>N<sub>4</sub> branched heterostructures and their photoluminescence properties**

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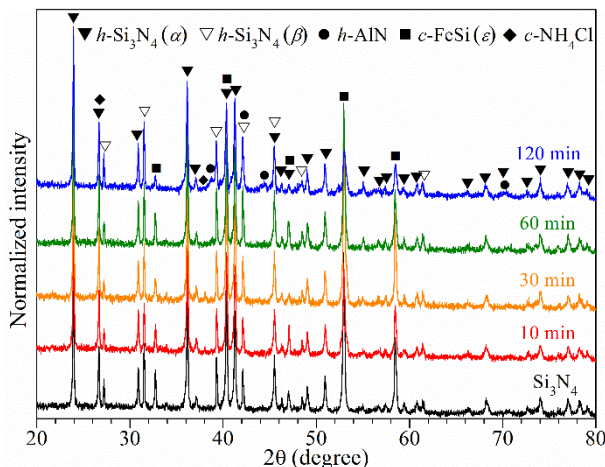
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### SI-1 XRD patterns of all the products in the range of 20-80°.

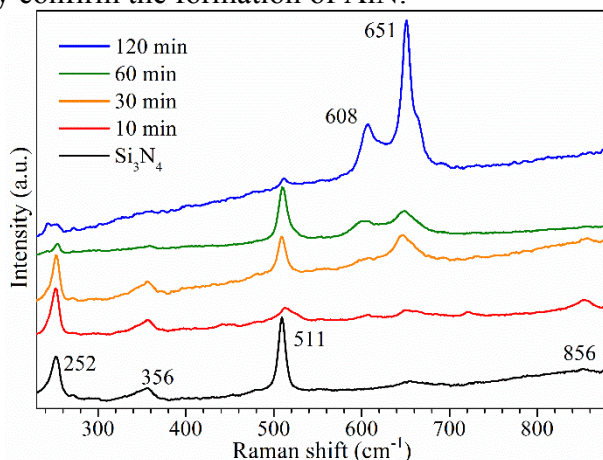
The XRD patterns of all the products in the range of 20-80° are shown in Fig. S1, the diffraction peaks of as-synthesized  $\text{Si}_3\text{N}_4$  product are indexed as  $h\text{-Si}_3\text{N}_4$  ( $\alpha$ ,  $\beta$ ) and  $c\text{-FeSi}$  ( $\epsilon$ ). After chemical vapor deposition of AlN, new weak diffraction peaks assigned to  $h\text{-AlN}$  appear. With elongating the deposition time, the intensity peaks of  $h\text{-AlN}$  become stronger, indicating the formation of more and more  $h\text{-AlN}$  phase.



**Fig. S1** XRD patterns of all the products in the range of 20-80°. The peaks marked with a triangle (▼), hollow triangle (▽) solid circle (●), and solid square (■), corresponding to  $h\text{-Si}_3\text{N}_4$  ( $\alpha$ , PDF#83-0700),  $h\text{-Si}_3\text{N}_4$  ( $\beta$ , PDF#76-0453),  $h\text{-AlN}$  (PDF#75-1620),  $c\text{-FeSi}$  ( $\epsilon$ , PDF#88-1298) and  $c\text{-NH}_4\text{Cl}$  (PDF#73-0365). Deposition times for AlN are 10, 30, 60, and 120 min.

### SI-2 Raman spectra of all the products.

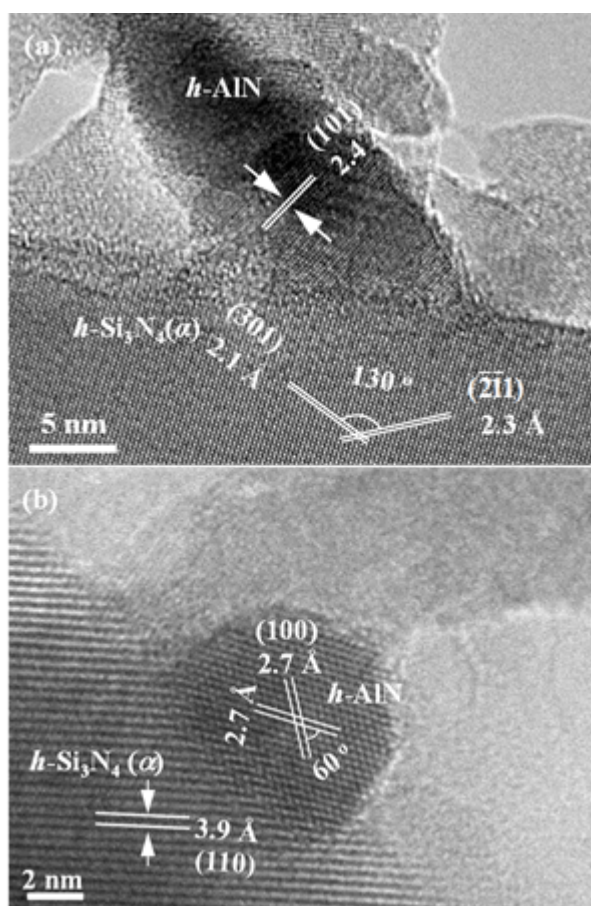
The Raman spectra of all the products are shown in Fig. S2. For as-synthesized  $\text{Si}_3\text{N}_4$  product, the five intensive Raman peaks are observed at ca. 252 (A1), 356 (E), 511 (A1), 651 (A1) and 856  $\text{cm}^{-1}$  (E), which are assigned to  $\alpha\text{-Si}_3\text{N}_4$  (space group  $P31c$ )<sup>S1</sup>. After depositing AlN, two new Raman peaks occur in ca. 608 and 651  $\text{cm}^{-1}$ , which are indexed to the A1 (TO) and E2 phonon modes of AlN (space group  $P63mc$ )<sup>S2</sup>. The results directly confirm the formation of AlN.



**Fig. S2** Raman spectra of all the products. Deposition time for AlN are 10, 30, 60, and 120 min.

### SI-3 HRTEM images of the 3D branched heterostructures.

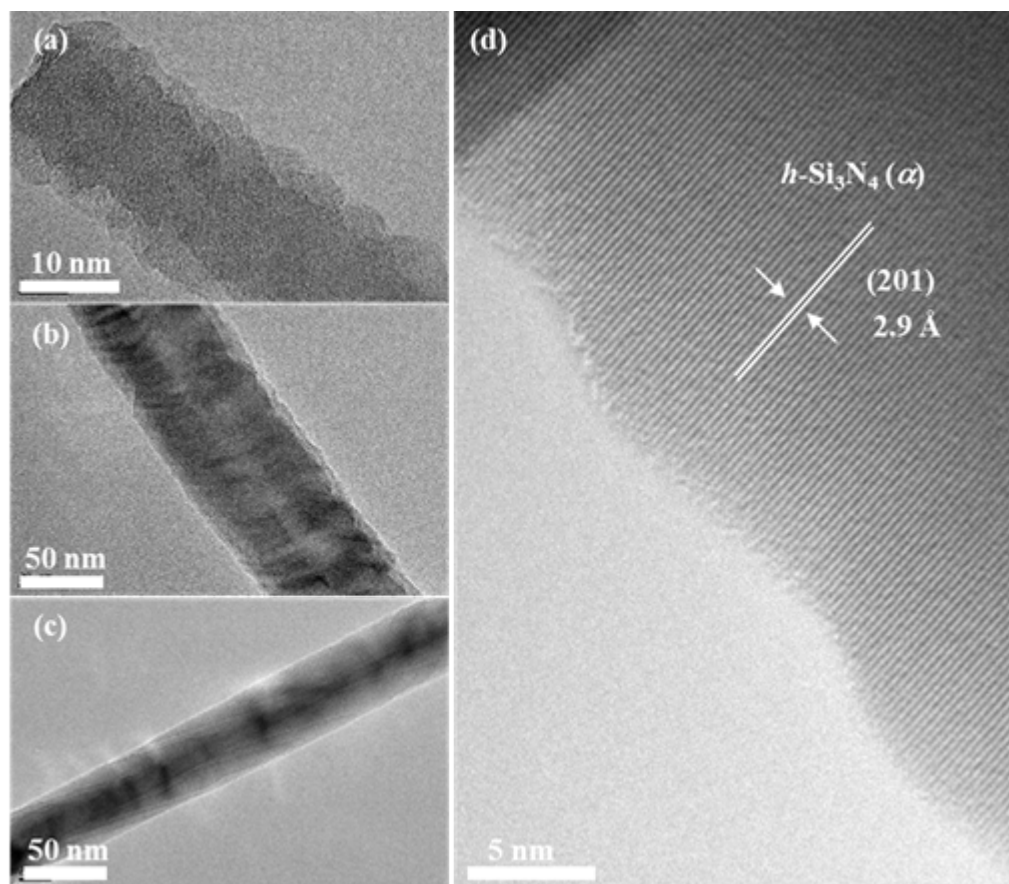
The HRTEM images of the 3D branched heterostructures are shown in Fig. S3. As shown in Fig. S3a (Fig. 6b is the part of Fig. S3a), the interplanar spacing of 2.4 Å corresponds to  $d_{101}$  of *h*-AlN and the interplanar spacings of 2.3 and 2.1 Å with dihedral angle of 130 ° are assigned to  $d_{\bar{2}11}$  and  $d_{301}$  of *h*-Si<sub>3</sub>N<sub>4</sub> ( $\alpha$ ). In Fig. S3b, the interplanar spacing of 2.7 Å is assigned to  $d_{100}$  of *h*-AlN while the interplanar spacings of 3.9 Å is assigned to  $d_{110}$  of *h*-Si<sub>3</sub>N<sub>4</sub> ( $\alpha$ ). The HRTEM results further confirm the formation of AlN on the surface of 1D Si<sub>3</sub>N<sub>4</sub> nanostructures.



**Fig. S3** HRTEM images of the 3D AlN-Si<sub>3</sub>N<sub>4</sub> branched heterostructures.

**SI-4 The surface of  $\text{Si}_3\text{N}_4$  nanostructures.**

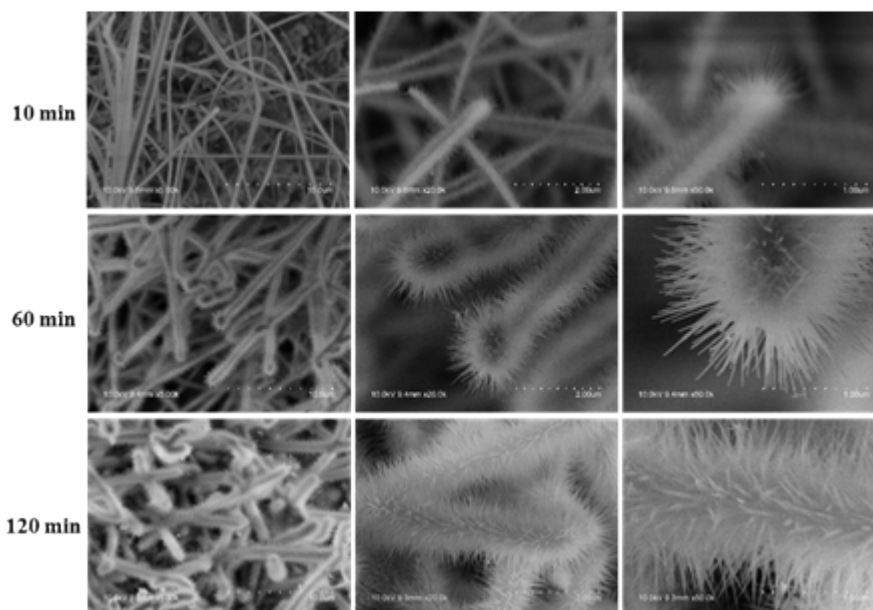
As seen in Fig. S4, the surface of  $\text{Si}_3\text{N}_4$  nanostructures is uneven, which indicates that there are a large amount of surface defects. These defects play an important role in the nucleation of AlN nanocones on the surface of 1D  $\text{Si}_3\text{N}_4$  nanostructures.



**Fig. S4** Microstructures of the surface of  $\text{Si}_3\text{N}_4$  nanostructures.

### SI-5 SEM images of the products from different deposition times for AlN.

The SEM images with different deposition time of AlN were shown in Fig. S5. It can be clearly seen that, with elongating the deposition time, the AlN nanocones on the surface of 1D  $\text{Si}_3\text{N}_4$  increased gradually in length and diameter.



**Fig. S5** Effect of deposition time on the diameter and length of AlN nanocones on the surface of 1D  $\text{Si}_3\text{N}_4$  nanostructures. The typical SEM magnifications are 5k, 20k and 50k. The longer the deposition time is, the thicker and longer AlN nanocones grow. Deposition times for AlN are 10, 60, and 120 min.

### References

- S1 J. A. Wendel and W. A. Goddard, *J. Chem. Phys.*, 1992, **97**, 5048-5062.  
S2 C. Liu, Z. Hu, Q. Wu, X. Wang, Y. Chen, H. Sang, J. Zhu, S. Deng and N. Xu, *J. Am. Chem. Soc.*, 2005, **127**, 1318-1322.