Hi, my name is zsl, a phd candidate of Zhejiang uni. my research interest focus on energy storages materials, especially newly two-dimensional materials of MXenes.

Herein, I will introduce my work of Vaporing Deposition Red Phosphorus to Prepare Nitrogen-Doped Ti 3 C 2 T x MXenes Composites for Lithium-Ion Batteries

Before introducing the experimental method, let we take a look at the basic properties of MXenes and phosphorus. What is the meaning of this work carried out?

Phosphorus (P) was promising anode materials in LIBs. It is cost and theoretical capacity could reach up to about **[two thousand six hundred micro apei hour per gram]**. So, red phosphorus has deep potential in future application. However, two problems of volume expansion and low conductivity limit its further application.

MXenes were firstly discovered by Yury group in 2011. It is well-known that MXenes are particularly prominent in high conductivity, but the theoretical capacity of the MXenes is only about [**three hundred twenty micro anpei hour per gram**]

Therefore, A work to prepared composites of red P and MXenes is necessary. Herein, we design a chemical vapor deposition method to prepare **N-doped MXenes and P composites** for the first time. The detailed preparation method is shown in **Figure a** of this page.

Step 1 after etching of MAX phases, multilayered MXenes were obtained.
Step 2 with the assistance of TMAOH and subsequent ultrasonic treatment, few-layered MXenes were prepared.
Step 3 after spray drying process, the walnut-like structure matrix of **N-doped MXenes** were obtained.
Finally, the final product of **N-doped MXenes and phosphorus composites** were obtained after vaporization and condensation of red P.

In order to investigate the delaminated condition of the Few-layered MXenes, SEM and AFM test were measured. After annealing treatment, the few-layered MXenes nanosheets were obtained. According to SEM images of the MXenes nanosheets, there was no visible restacking phenomenon of nanosheets, combined with AFM results, which show that the number of layers is about 2−3, and the length of the ab plane is 5 μm.

As clearly can be seen from figure g-d. After vaporization and condensation of red P into three-dimensional porous architecture of the N-doped MXenes matrix, **N-doped MXenes and P composites were synthesized**, which become rougher, and caves nearly disappeared compared with the pure matrix because of condensation of red P particles.

TEM results show that the particle size of red P is below 100 nm in Figure j and k, which was

adsorbed at the surfaces of the N doped MXenes matrix and beneficial for releasing capacity of Phosphorus anodes

STEM results in **Figure l** show that the distribution shapes of phosphorus and key elements of Ti, [taɪ'tenɪəm; tɪ-], C, and N are extremely overlapped with each other, indicating that the red phosphorus particles were distributed **uniformly** at the N doped MXenes matrix.

The electrochemical performance of the composites was carried out, including CV measurements, rate performance and long cycling performances.

During the scanning process, there were mainly one sharp peak and two smaller peaks, which could be assigned to stepwise lithiation or de-lithiation process.

The discharging and charging plateau can also be kept after 200 cycles, demonstrating good reversibility of the composites.

XPS measurements show the Ti-O-P bond was formed in the composites, granting the robust structure of the composites during cycling, leading to obvious improvement of electrochemical performance in the MXenes field yet.

The electrochemical kinetics of the composites was investigated by CV measurements at different scanning rates. The results indicate that the **capacitive** contribution was increased by increasing the scanning rate

Because of increased interlayer spacing of MXenes caused by volume expansion of P and continuous ions intercalation/deintercalation process. The charge transfer resistance was reduced during cycling process.

TEM images of the composite electrodes after 1000 cycles clearly indicates that the interlayer spacing of the MXenes increased to 1.18 nm, providing direct evidence to verify our above-mentioned demonstration.

In summary, **N-doped MXenes and phosphorus composites** are derived through spray drying and vapor deposition of red phosphorus
XPS confirms unique Ti-O-P chemical bond existed in the composites, endowing the stable structure of the composites even after long cycles at higher current density.

When used as anodes materials in LIBs, the composites show one of the best electrochemical performances in MXenes-based materials.

More importantly, this method is universal and **reproducible**, via different matrix design and preparation, a systematic study on MXenes and red P composites work can

be put forward and optimized.

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