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# From highly reactive amorphous tantalum oxide to tantalum nitride. Impact of the nitridation temperature on the optical and photocatalytic properties

Floriane Guyot, François Cheviré, Ronan Lebullenger, Nathalie Herbert

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# From highly reactive amorphous tantalum oxide to tantalum nitride.

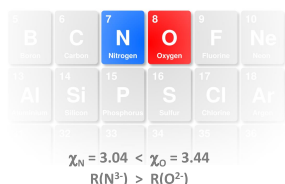
## Impact of the nitridation temperature on the optical and photocatalytic properties.

Floriane Guyot, François Cheviré, Ronan Lebullenger, Nathalie Herbert

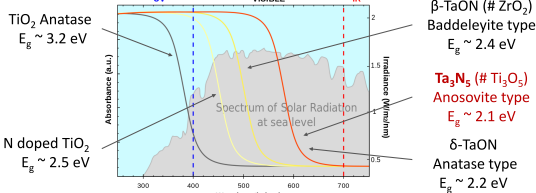
UMR CNRS 6226 Institut des Sciences Chimiques de Rennes (ISCR), Université de Rennes 1, F-35042 Rennes, France

Recently, much further attention has been paid to functional (oxy)nitrides as emerging materials for environmental and energy applications with a focus on the specific tuning properties offered by the nitrogen/oxygen substitution. Among them, **red tantalum nitride Ta<sub>3</sub>N<sub>5</sub>** is particularly regarded as a visible-light driven photocatalyst because its **narrow bandgap of 2.1 eV** allows for the harvesting of most of the visible range of the solar spectrum. In order to enhance the photocatalytic activity of the material, the morphology of the powder is an important parameter. Thus soft chemistry routes are usually required for the synthesis of the oxide precursor, i.e. Ta<sub>2</sub>O<sub>5</sub>, as conventional commercial products are usually not suitable due to their micrometric grain size. A better control of the particle size and specific surface area of the oxide precursor also allows for the lowering of the nitridation reaction time and temperature, and for limiting the generation of reduced defects detrimental to optical and photocatalytic properties of Ta<sub>3</sub>N<sub>5</sub>. However, there is only a few options for the starting materials for the synthesis of the Ta<sub>2</sub>O<sub>5</sub> using soft chemistry routes, such as oxalate (Ta(C<sub>2</sub>O<sub>4</sub>)<sub>3</sub>), chloride (TaCl<sub>5</sub>) or alkoxydes (Ta(OEt)<sub>5</sub>) which are costly and in some cases highly moisture sensitive. We propose here a **simple approach for the amorphization of commercial crystalline Ta<sub>2</sub>O<sub>5</sub>**, involving the use of potassium bisulfate (KHSO<sub>4</sub>), in order to improve its reactivity towards ammonia and thus lower the nitridation temperature.

### d<sup>0</sup>-metal based (oxy)nitrides as visible light absorbers

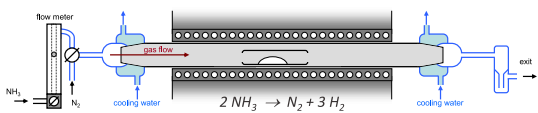
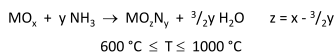


Effect nitrogen insertion on the optical gap (d<sup>0</sup> metal)

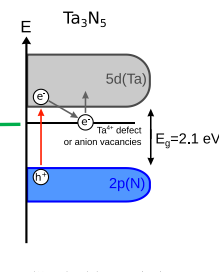
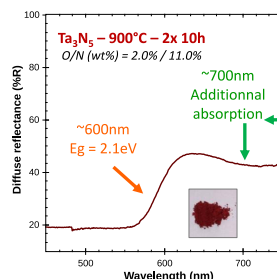
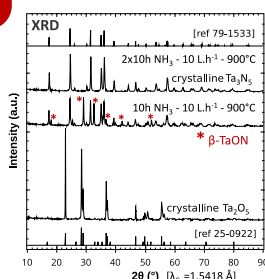


C. Burdo et al., Nano Lett. 3 (2008) 1049 - W.-J. Chun et al., J. Phys. Chem. B 107 (2003) 1798-1803 - T. Lüdtke et al., Inorg. Chem. 53 (2014) 11691

### Ta<sub>3</sub>N<sub>5</sub> from nitridation of crystalline tantalum oxide

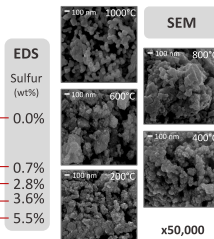
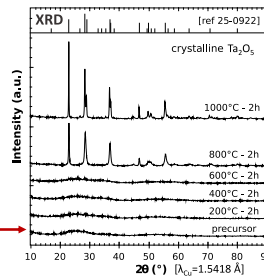
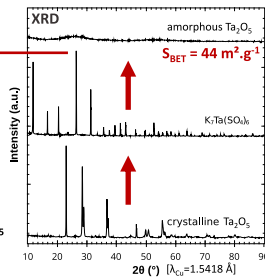
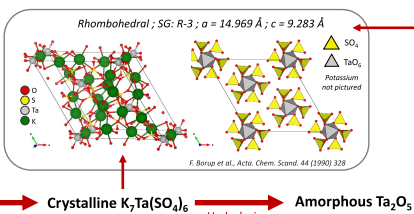
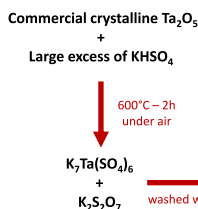


!!! Reductive atmosphere → formation of reduced type defect !!!  
!!! Charge balance 3 O<sup>2-</sup> = 2 N<sup>3-</sup> + Vac → anion vacancies !!!



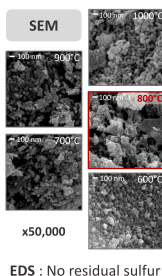
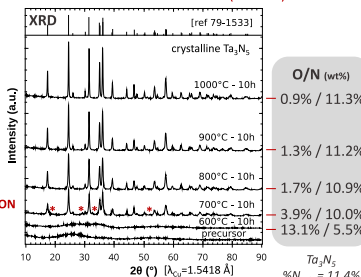
A. Dobrinin et al., Appl. Phys. Lett. 102 (2013) 033905

### Tantalum precursor from hydrolysis of K<sub>7</sub>Ta(SO<sub>4</sub>)<sub>6</sub>

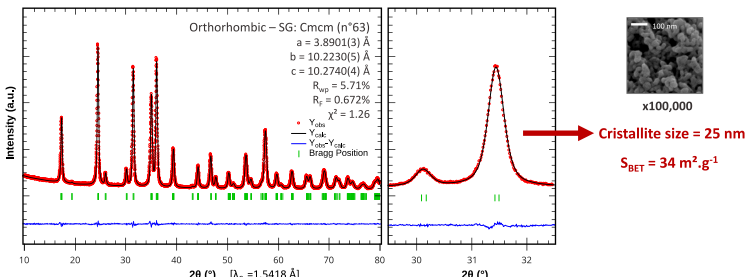


### Effect of nitridation temperature on the optical absorption and the photodegradation of Methylene Blue

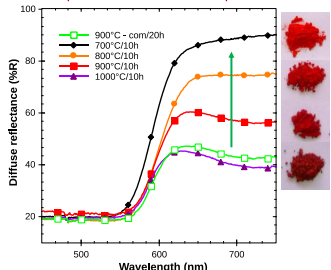
Nitridation under ammonia flow (10 L.h<sup>-1</sup>)



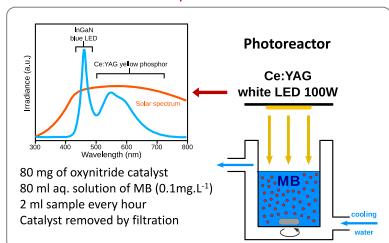
Rietveld refinement - Nitridation at 800°C for 10h



Absorption vs. Nitridation temperature



Photocatalytic tests conditions



Methylene Blue photodegradation monitored by UV/Vis at 660 nm

