Hydrothermal Synthesis and Characterization of Gallium Oxide Micro and Nanocrystals

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Abstract: Currently, research of gallium oxide (GO) nano- and microcrystals is rapidly expanding with the demand for potential uses. GO has been shown to be a promising material for possible applications in many different fields including photocatalysis, biomedicine, and optoelectronic devices. In our lab (led by Dr. Strzhemechny) we examine both the fundamental (nature of crystal defects) and applied (antibacterial action) properties of GO. During the hydrothermal growth process of GO, we are producing different nano and microscopic morphologies of this material by controlling various growth parameters including varied pH and adding surfactants to the material. The synthesis procedure includes using the precursor material, gallium nitrate hydrate, ammonium hydroxide. We use a calcination furnace that can get to temperatures high enough to effectively synthesize GO. Now, with a thermocouple and pyrometer we can predict outcomes during the calcination step with high accuracy and precision.

Introduction

- Gallium Oxide (Ga_2O_3) is polymorphic material with several crystalline phases
- The Beta form (β-Ga_2O_3) has many potential electronic and biomedical applications due to physicochemical structure
- Many methods exist to synthesize equipment of biohazardous materials
- Hydrothermal Synthesis offers a safe and affordable method that allows for size and shape control

Methodology

- 1.27 g of Gallium Nitrate hydrate (chemical formula) mixed with 50 ml of DI water
- Ammonium hydroxide is added in various amounts to control pH
- Solution mixed and heated at 60 C for a variety of durations
- Solution heated at various temperatures and times
- Gallium oxyhydroxide (GaOOH) precursor crystals were washed in DI water, dried at 70 C for 6 hours
- GaOOH calcinated at 750 C for 5h, producing β-Ga_2O_3 crystals

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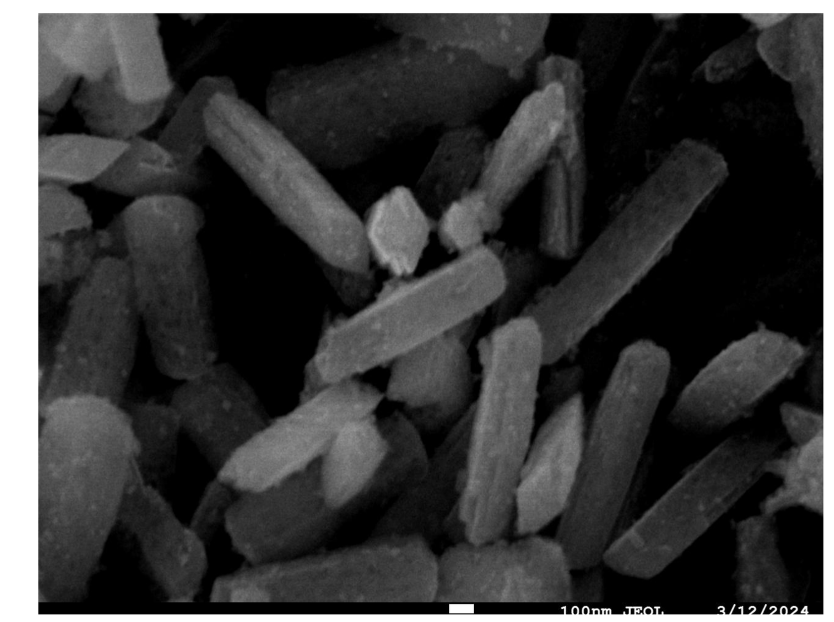
Materials with potential antibacterial properties are increasing in demand. One of them is gallium oxide (Ga_2O_3), which not only exhibits antibacterial properties but has more applications in various fields including photocatalysis, chemical sensors, and optoelectronic devices. Gallium hydroxide (GaOOH) micro- and nanocrystals can be synthesized from wet solutions at relatively low temperatures. However, much higher temperatures are required to convert GaOOH to Ga_2O_3. This conversion must occur with precise temperature control. In our lab we created a method of a controllable calcination of GaOOH and its successful conversion to Ga_2O_3. To confirm this, we performed comparative studies of our GaOOH samples before calcination and the resulting Ga_2O_3 after calcination.

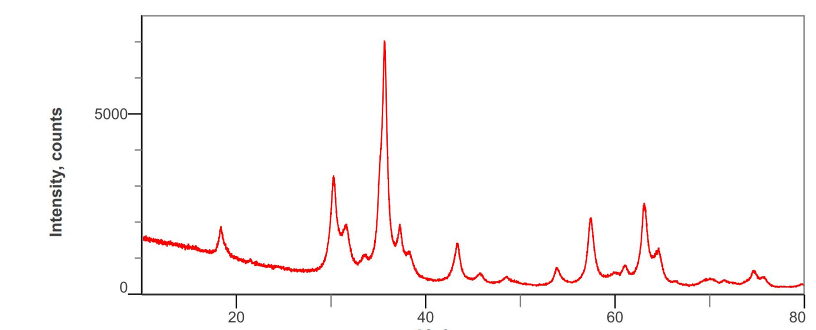
Synthesis

Pictured here is the calcination of GaOOH, synthesizing Gallium Oxide. We have a crucible carrying Gallium Oxyhydroxide powder that will bake for around five hours at a temperature of 750 degrees Celsius.

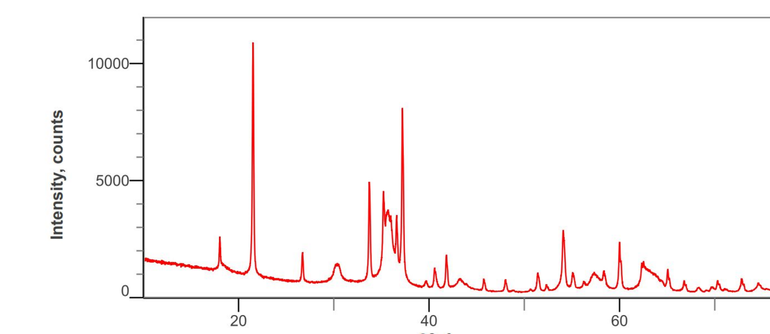
XRD Raman SEM Images



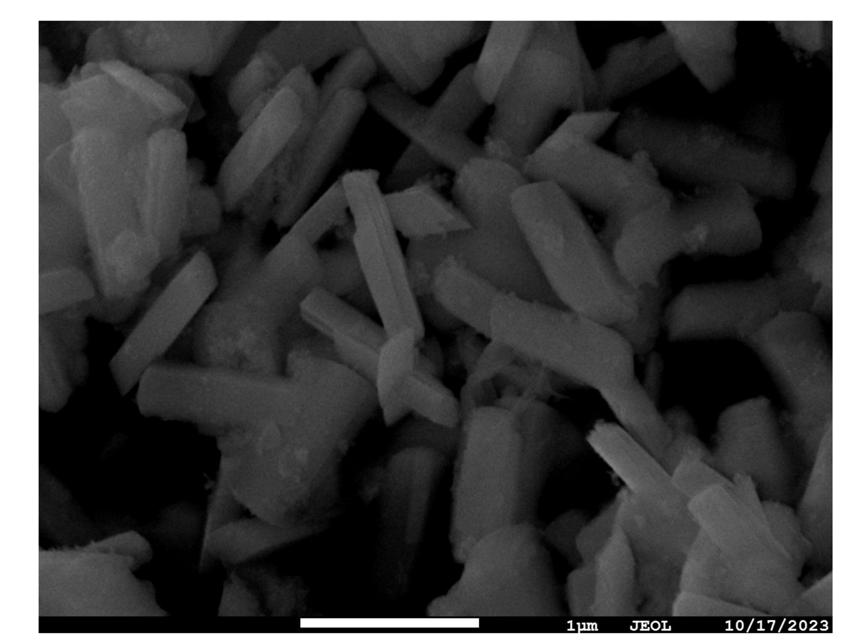
Ga_2O_3 SEM pH = 5



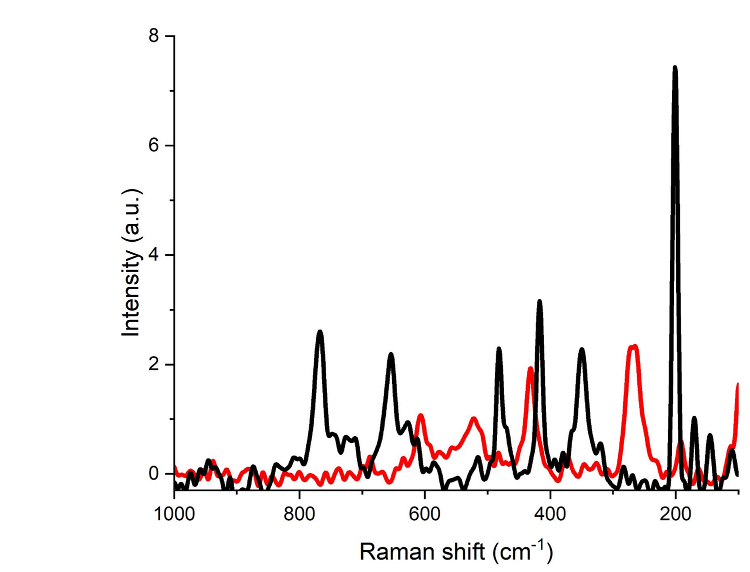
Ga_2O_3 XRD pH=5



GaOOH XRD pH=5



GaOOH SEM pH = 5



Further Studies

- We are planning to do a further analysis of the antibacterial activity in relevant bacterial environments
- Further investigation of the correlations between the relationship between logarithmic aging time control, pH control, alkali rate control, temperature control, and time control with their impact on the morphology of beta gallium hydroxide.
- Cathodoluminescence spectroscopy setup to study the optoelectronic characteristics of gallium hydroxide and gallium oxide that are irradiated by an electron beam which is then collected by an optical system.
- Finally use computational methods for easier quantification of varied morphologies of gallium hydroxide

Conclusion

- Now, with a thermocouple and pyrometer we can predict outcomes during the calcination step with high accuracy and precision
- Being able to control and monitor the temperature at a fixed value, has allowed our lab to create high quality materials which makes analysis and results to be much more clear.
- We can clearly see from the X Ray Diffraction that the precursor gallium oxyhydroxide and gallium oxide have different internal structures.
- New peaks in the raman shift show that we have totally different material despite looking similar because the peaks indicated different molecular bonds.
- Further, we can see a minimal to no change in the morphology of the material from the precursor gallium oxyhydroxide to gallium oxide