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Meeting-report

Thermal Evolution of Alumina through Dehydration of Aluminum Hydroxide

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Recent works suggest unexplained infrared features in some low-mass stars, known as Asymptotic Giant Branch (AGB) stars, are from two different polymorphs of aluminum oxide (Al_2O_3 or alumina), either amorphous or corundum (α-aluminum oxide) [1]. Aluminum oxides have been well studied in the field of material sciences. Alumina's crystal structure can vary based on thermal conditions of its environment making it metastable [2]. This work focuses on creating different metastable forms of alumina from a variety of aluminum hydroxide starting material, including gibbsite, bayerite ($Al(OH)_3$) , boehmite (AlOOH) and an amorphous $Al(OH)_3$, for astronomical applications to assist in our understanding of dust around AGB stars. Our process of material synthesis follows [1], where each sample of aluminum hydroxide is annealed at 300, 500, 700, 800, 900, 1000, & 1100 -°C for 2 hours. The resulting materials are characterized using X-ray diffraction (XRD), differential scanning calorimetry (DSC), Transmission Electron Microscopy (TEM), and Selected Area Electron Diffraction (SAED). The current findings being presented are related to the gibbsite-derived alumina. We compared the thermal evolution of these samples to results from [1, 3].

The TEM and SAED images were collected using a JOEL ARM200F microscope at 200kV and adapted with a 2kx2k CCD camera and using doubled tilt sample holder. We have observed the crystal structures of our samples using SAED patterns to study the structural evolution of the phases. Figure 1 shows micrographs of the region of analysis (a)-(d) and the inset the crystalline structures used to index the patterns. Figures 1 (e) to (h) show the SAED patterns and the zone axes indexed using Java Electron Microscopy Simulations (JEMS) software package. The XRD diffractograms were collected using a Panalytical Empyrean X-ray Diffractometer. The collected XRD diffractogram analysis was accomplished using Profex and structure reference files were collected from the Crystalline Open Database (COD).

At low temps ~300°C, the $Al(OH)_3$ undergoes a dehydration reaction to form boehmite (AlOOH). Upon further heating, to ~500°C, the OH groups are broken, and the AlOOH samples are converted into a metastable structure of Al_2O_3, depending on the thermal environment. The confirmed structures from XRD analysis are η-Al_2O_3 (FCC), κ-Al_2O_3 (orthorhombic), α-Al_2O_3 (rhombic). These samples also lose mass as they are heated to higher temperatures above 500°C. The mass loss is in the magnitude of ~30% of its initial mass; this is in the form of volatiles.

In conclusion, we have synthesized a sequence of polymorphs of alumina, from a gibbsite precursor via annealing. Our characterization shows how the annealing temperatures give rise to specific phase changes. Our future work includes starting with other aluminum hydroxide precursors. The bayerite and boehmite derived alumina XRD characterization has been completed, yet the TEM and SAED information still needs to be collected. Further future work will include studying and characterizing other aluminum oxide bearing minerals, and applying what we learn to astronomical modeling of AGB stars.

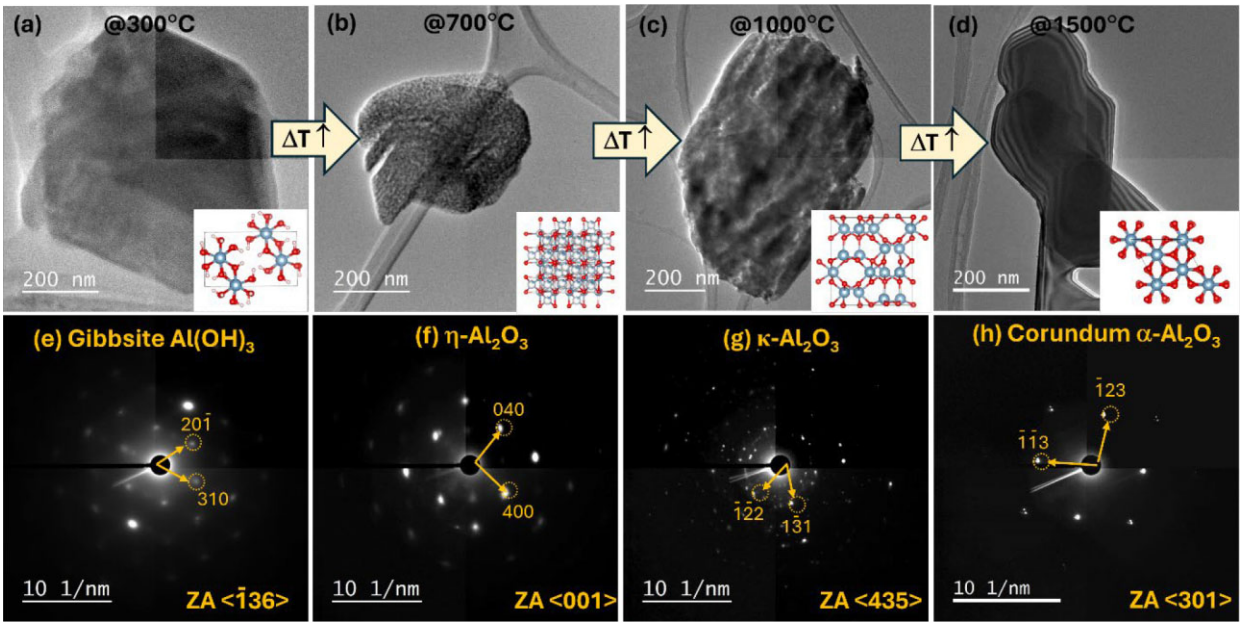

Figure 1. TEM images of alumina sample evolution derived from Gibbsite (top) & indexed SAED of alumina samples derived from different annealing temperatures (bottom).

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