

In-Situ Characterization of the Synthesis of $\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$ Under Non-Ambient Atmospheres

Claudia J. Rawn^{1,2}, John Robert Salasin^{1,2}, Michael R. Koehler³, Sabrina E.A. Schwerzler^{1,2}, and David J. Keffer¹

¹ Materials Science and Engineering Department, University of Tennessee, Knoxville

² Center for Materials Processing, University of Tennessee, Knoxville

³ Joint Institute for Advanced Materials Diffraction Facility, University of Tennessee, Knoxville, TN

$\text{Ca}_{12}\text{Al}_{14}\text{O}_{33}$, the mineral mayenite, is a highly functional material that crystallizes into a clathrate caged structure. The positively charged cage has anions occluded within the cages for charge balancing purposes. The occluded anion composition space is diverse with a multitude of monoatomic species (O, N, S, F, Au, etc.), poly-atomic species (CN, N_2H_4 , NH_2 , NO_2 , O_2 , C_2 , etc.), and non-atomic electrons that can occupy the center of the cage. The formation of the clathrate C12A7 and speciation of the occluded species are sensitive to atmospheric conditions such as process atmosphere and moisture content, and formation pathways have been theorized through thermodynamic, “cook and look” experimentation. We characterize the kinetic behavior of C12A7 crystallization from amorphous sol-gel and solid-state synthesis precursors utilizing an Anton Paar HTK-1200N furnace mounted on a Malvern PANalytical Empyrean diffractometer. Utilization of the PIXcel3D area detector allows for rapid data collection (<60 s) during ramping and the characterization of formation temperatures, reaction pathways, and derived stabilizing anions as a function of process atmosphere. These studies complement thermodynamic studies and reveal the kinetic behavior and formation pathways.