Type of presentation: Poster

MS-1-P-3275 In-situ Electron beam induced transformation of boehmite to gamma alumina

Ozkaya D.[1]

[1]Johnson Matthey Technology Centre, Reading UK

Email of the presenting author: ozkayad@matthey.com

Gamma alumina is widely used as a support material for industrial catalysts such as refining petrochemicals or Fischer-Tropsch processes. Boehmite, Aluminum oxihydroxide (AlOOH), is the precursor to Gamma-alumina (γ-Al2O3) in the manufacturing process[1].The transformation from boehmite to gamma alumina is critical to the understanding of the structure of gamma alumina, thus has been widely studied [2]. The removal of water from the structure creates a metastable gamma phase with a structure that gets classified as tetragonal or cubic depending on the choice of unit cell [2]. The changes to the structure and surface area during this transformation are still not very well understood. One of the problems with studying boehmite using transmission electron microscopy (TEM) is the electron beam sensitivity of boehmite. Boehmite transforms to gamma alumina if observed at ambient temperatures in the TEM even under low beam dose conditions. Here, Cryo-TEM has been used to slow down the transformation under the beam in-situ as the sample de-hydrates and forms gamma alumina. The main aim of the study was to observe changes in the shape and form of the nanocrystalline material after it has transformed to gamma alumina. The properties of gamma alumina is what determines the diffusion rates and sintering behaviour of Platinum group metals (PGM's)in most of the current auto catalysts. The study was carried out using a Gatan Cryo holder at -184C in a FEI Tecnai F20 Twin lens configuration S/TEM. The boehmite sample was dispersed on a holey carbon coated Cu-grid and put into the cryo-holder and the microscope before cooling. Some ice formation was observed at some parts of the sample as boehmite itself contains some excess water. A sequence of images was acquired as function of time together with diffraction patterns to confirm the structural transformation. A sequence of diffraction patterns diffraction patterns of boehmite and gamma alumina are shown in Figure 1 where the rings formed have been identified using JEMS software. The difference is quite stark in the way the pattern goes as the boehmite is orthorhombic and gamma alumina is pseudo cubic or tetragonal structure. The characterisation of the diffractions patterns shown together with the calculated spacings on the diffraction patterns in figure 1.

Figure 2 shows the sequence of in-situ images from boehmite to gamma alumina, the only driving force for the transformation being the electron beam. Images were acquired in sequence together with the diffraction patterns at regular intervals. The images shows that the shape of the crystallites remain the same after the transformations has taken place. However there is a slight, in shrinkage in the size of the particles around 10-15% after the transformation.

Acknowledgement: I would like to thank Stephen Spratt from Johnson Matthey Technology Centre for help during cryo experiments

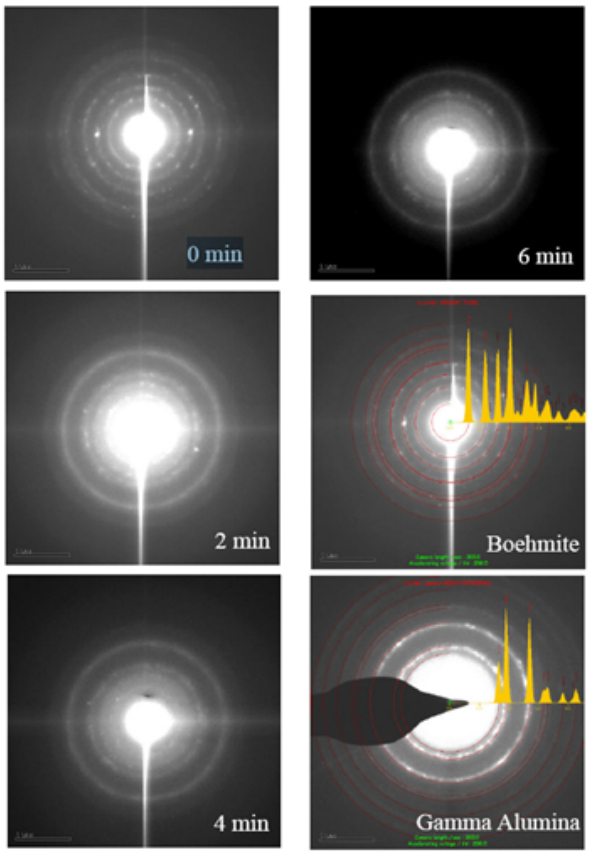

Fig. 1: Figure 1. Series of diffraction patterns at different time intervals showing the transformation from boehmite to gamma alumina under the electron beam. The calculated diffraction patterns from boehmite and gamma alumina are shown for comparison

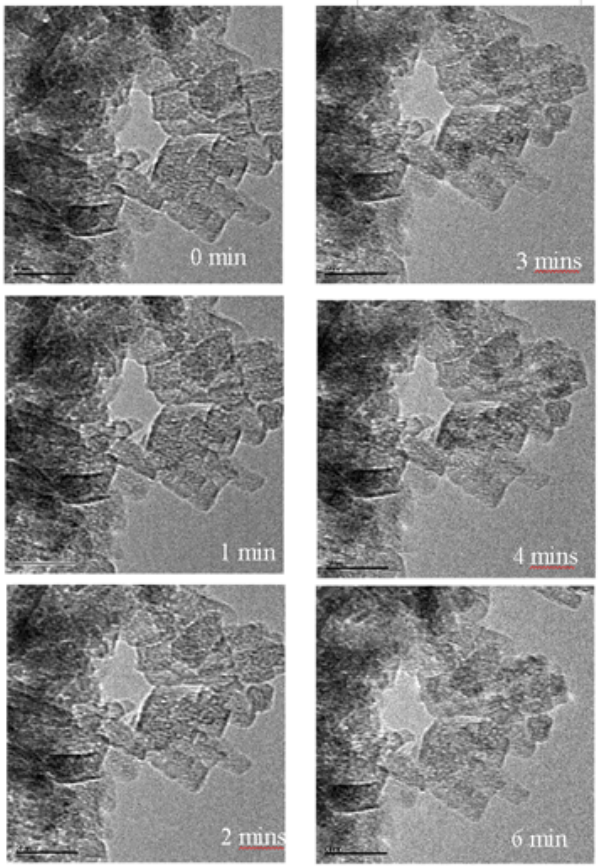

Fig. 2: Figure 2. Series of in-situ images taken at various time intervals from the same area that diffraction patterns were taken in figure. Transformation from boehmite to gamma alumina transformation does not cause drastic change in the crystal shape but a 10-15% reduction in size.