INVESTIGATION OF DEHYDROXYLATION OF GIBBSITE INTO BOEHMITE BY DSC ANALYSIS

J. Perić, R. Krstulović and M. Vučak

Faculty of Technology, University of Split, Teslina 10/V, 21000 Split, Croatia

Abstract

The dehydroxylation of gibbsite into boehmite was investigated by means of DSC analysis under non-isothermal conditions in the temperature range 453–673 K at heating rates from 2.5 to 20.0 K min⁻¹. Mathematical analysis of the experimental DSC curves for the mechanism and kinetics of the gibbsite dehydroxylation process. The kinetic curves $\alpha=f(t)$ and $\alpha=f(T)$ are sigmoidal in shape; their inflection points and the $v_m$ point of the curves $v=f(t)$ and $v=f(T)$ are interrelated and are defined by the concept of a stationary point. The activation energy for the first stage of gibbsite dehydroxylation in the temperature range 453–673 K is $132.92\pm 8.33-142.26\pm8.33$ kJ mol⁻¹.

Keywords: boehmite, DSC, gibbsite

Introduction

A large number of methods have been suggested for the determination of kinetic parameters from dynamic thermogravimetric measurements and differential thermal analysis [1–5]. In previous work [5], the process of dehydroxylation of aluminium minerals was examined by means of non-isothermal thermogravimetric analysis. The present paper describes the treatment of experimental data obtained by means of differential scanning calorimetry (DSC) under non-isothermal conditions in order to establish the kinetic parameters of the dehydroxylation of $\gamma$-Al(OH)₃.

The reaction kinetics was examined only for the first stage of gibbsite dehydroxylation, i.e. the dehydroxylation of gibbsite to secondary boehmite in the temperature range 453–673 K. The choice of upper temperature limit was influenced by the characteristics of the apparatus.

Experimental

Sample

This was a gibbsite $\gamma$-Al(OH)₃ sample produced industrially in 1983 according to the Bayer process.

0368-4466/96/ $5.00

© 1996 Akadémiai Kiadó, Budapest

John Wiley & Sons, Limited

Chichester
X-ray diffractometry

A Philips X-ray diffractometer with CuKα radiation was used to check the purity of the sample (Fig. 1). The gibbsite was observed to have been partially converted to boehmite by ageing under the normal pressure and temperature conditions.

![Comparison of X-ray diffraction patterns of gibbsite sample. Diffractogram: a) recorded in 1983; b) recorded in 1992](image)

DSC analysis

The DSC analysis was carried out under dynamic non-isothermal conditions with a flow of extra-pure nitrogen in a Perkin-Elmer DSC-4 differential scanning calorimeter [6]. The operating conditions were: temperature range: 453–673 K, heating rate: 2.5, 5.0, 10.0, 15.0 or 20.0 K min⁻¹, cooling rate: 320 K min⁻¹, flow rate of extra-pure nitrogen: 30 ml min⁻¹.

The dehydroxylation of gibbsite into boehmite is an endothermic process:

\[ \text{Gibbsite} \rightarrow \text{Boehmite} \]