Proton Magnetic Resonance Studies on the Thermally Dehydrated Products of Alumina Trihydrate

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(Received September 21, 1960)

In our previous communication[1], the thermal decomposition of alumina trihydrate was studied from the change of line shapes of proton magnetic resonance absorption in the thermally dehydrated products. In the present communication, the phase transformations accompanied with the thermal dehydration process of alumina trihydrate have been studied from the variation of second moments of the resonance lines in dehydrated products.

The starting material, bayerite, and the detecting apparatus here employed were the same as those used in the previous experiments[1]. The specimens used for the NMR measurements were prepared from this bayerite by the thermal dehydration at 100~800°C which was carried out for 3 hr. in the air. Each specimen was sealed into an evacuated glass tube which was pumped out to a high vacuum at 110°C for 6 hr.

The variation of the second moments calculated from the experimental derivative curves are shown in Fig. 1 as the function of the temperature. Below 220°C, the second moment was 13~14 gauss^2 showing no significant change with the temperature. However, in the range from 220 to 400°C the second moment

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1) T. Hagiwara and T. Saito, This Bulletin, 33, 1463 (1960).

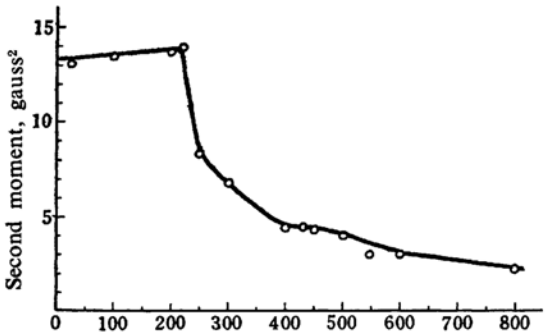

Fig. 1. Variation of the second moments with the temperature in dehydrated products of the starting material.

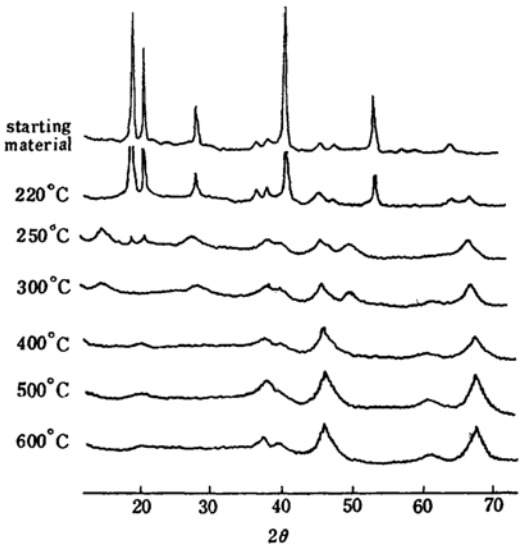

Fig. 2. X-ray diffraction diagrams of the samples dehydrated at various temperatures.

decreases gradually with the rise of temperature, and then at about 400°C the second moment becomes roughly stationary value of 4~5 gauss². Above 550°C the second moment gradually decreases again.

The above variation of second moments is interpreted as follows:

1) The sudden decrease of second moment at about 220°C seems to show the phase transition from the starting material to the hydrous alumina which has a low value of second moment.

2) The gradual decrease of second moments with the rise of temperature in the range from about 250 to 400°C could be regarded as a result due to the change of mixing ratio of the aluminum hydroxide-hydrous alumina phase.

3) The roughly stationary values of 4~5 gauss² at about 400~500°C seem to show the phase of hydrous alumina.

The X-ray diffraction study of the compounds was also done. The diffraction diagrams of several specimens which were obtained from this study are shown in Fig. 2. From these diagrams, it is ascertained that the specimens below 220°C having the value of 13~14 gauss² are crystallographically the same as the starting material, bayerite, and that each specimen at 250° and 300°C shows the diagram of mixed phase of böhmite and η-alumina, but that the specimens at about 400~500°C having the value of 4~5 gauss²—which are explained to be hydrous alumina—are η-alumina[2].

It is interesting to note that the transition temperatures of 220 and 400°C are obtained from the variation curve of second moment showing in Fig. 1, while each transition temperature coincides with that of phase transformations obtained from X-ray diffraction studies, respectively.

The author wishes to express his hearty thanks to Professor Tokutaro Hagiwara for his kind direction and also wishes to express his sincere thanks to Professor Tunahiko Shidei and Dr. Syukuro Yano in Kyoto University for their continual guidance and encouragement throughout this study.

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2) A. S. T. M., X-ray powder data file.