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Effect of Precipitation Temperature on the Thermal Stability of Aluminum Hydroxide in the Bayer Process

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***Abstract*— A procedure to increase the thermal stability of aluminum hydroxide (ATH), KH-101LC, which is produced in the Bayer process, was developed. The effects of the amount of added seed and the precipitation temperature were investigated. The amount of Na_2O in the ATH was proved to be inversely proportional to the thermal stability. Increasing the precipitation temperature to higher than 100 °C well improved the thermal stability of ATH**

***Index Terms*— Aluminum hydroxide, thermal stability, precipitation, temperature**

I. INTRODUCTION

Aluminum hydroxide (ATH) is widely used in high polymers as flame retardants, inert fillers, and thermal insulators because of its unique properties, low price, and non-toxicity.[1], [2] However, some drawbacks such as relative low shaping temperature and low compatibility with high polymers limit the extensive application of ATH.[3], [4] The thermal stability of ATH must be well improved for it to be used as an effective ingredient of the thermal insulating layer in printed circuit board-copper clad laminate (PCB-CCL).

Much research has been pursued to improve the thermal stability of ATH, and various methods have been proposed, such as acid leaching, coating with silane, pre-heating, and partial dehydration.[5] Rigorous purification for reducing the amount of ion-insoluble substances (especially Na_2O) in ATH powder, generally by re-digestion, has been used to lower the conductivity of ATH.[6]

Commercial ATH (OL-104LEO, Albemarle Corporation, USA), which has good thermal stability, is used as a component of the thermal insulating layer in PCB-CCL in Korea, but it is expensive. In this study, a procedure was developed to increase the thermal stability of ATH (KH-101LC, KC corporation, Korea), which can be produced domestically without re-digestion, so that the cost is reduced but the thermal stability is the same as that of ATH (OL-104LEO). For this purpose, the precipitation parameters such as the added amount of seed and temperature were investigated. The optimum seed amount and precipitation temperature to achieve high thermal stability of ATH (KH-101LC) were determined.

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II. EXPERIMENTAL

A. Materials

The physicochemical properties of the raw material (KH-101LC, KC Corporation, Korea) and the target material (OL-104LEO, Albemarle Corporation, USA) are presented in Table 1.

Commercial ATH (HWF-100, Chalco, China), (Gibbsite phase, particle size: Dp50 = 130 μm) was used to prepare the seed with an Air Jet Mill. The Dp50, specific surface area (SSA), and Na2O content of the produced seed were 1.2-1.4 μm, 6.4 m^2/g, and 0.19%, respectively.

B. Precipitation Processing

Fifteen to 50 g of the produced seed was added to the Bayer liquor (KC Corporation, Korea) at the desired temperature (75-110 °C), and the temperature of the mixture was reduced to 65 °C linearly over 12 h. Then, the solid was filtered, washed with 70 °C distilled water, and dried before analysis.

Table 1. Comparison of the properties of KH-101LC and OL-104LEO

	KH-101LC	OL-104LEO
Moisture (%)	0.15	0.13
Na_2O(%)	0.24	0.12
Fe_2O_3(%)	0.008	0.010
Dp50 (μm)	1.5	2.1
Conductivity (μ/cm)	39.5	18.2
Whiteness	97.8	99.7
L-Na_2O (%)	0.016	0.014
Phase	Gibbsite	Gibbsite
Weight loss (150-288 °C)	22.858%	20.534%

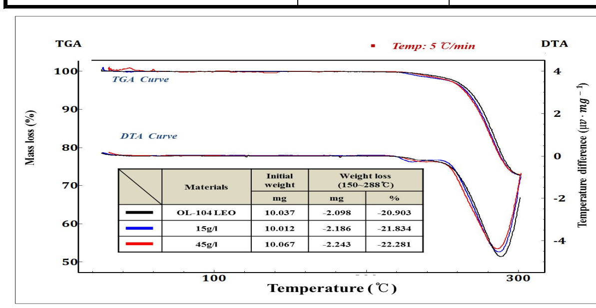

Fig. 1. Comparison of TGA-DTA curves of OL-104LEO and ATH powder produced at a precipitation temperature of 75 °C.

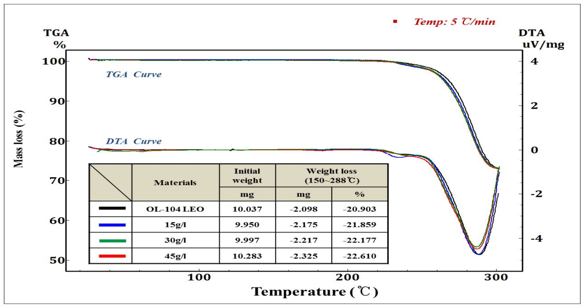

Fig. 2. Comparison of TGA-DTA curves of OL-104LEO and ATH powder produced at a precipitation temperature of 80 °C.

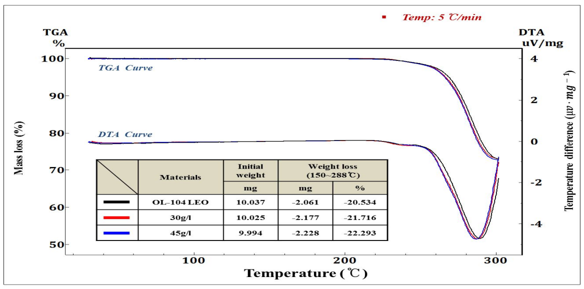

Fig. 3. Comparison of TGA-DTA curves of OL-104LEO and ATH powder produced at a precipitation temperature of 90 °C.

C. Testing Methods

The weight loss of the samples over a certain test temperature range, as determined in Thermo Gravimetric Analysis-Differential Thermal Analysis TGA-DTA measurements (Dtg60, Shimadzu), was used as an indication of thermal stability. The testing temperature interval was 25−320 °C, and the thermograms were obtained at a 5 °C/min heating rate and a 100 mL/min linear gas flow rate, under air atmosphere. In addition, scanning

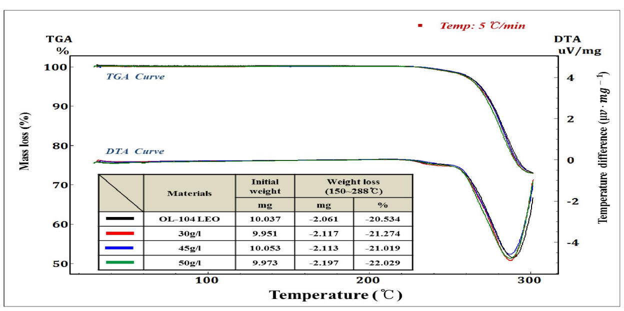

Fig. 4. Comparison of TGA-DTA curves of OL-104LEO and ATH powder produced at a precipitation temperature of 100 °C.

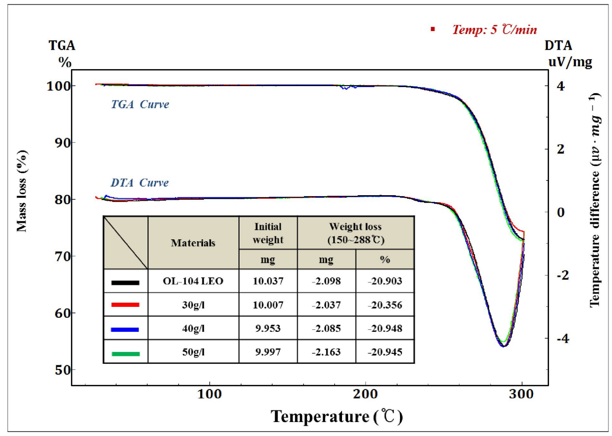

Fig. 5. Comparison of TGA-DTA curves of OL-104LEO and ATH powder produced at a precipitation temperature of 110 °C.

electron microscopy (SEM, JSM5600, JEOL) was employed to investigate the grain morphology of the samples. The structural characteristics of the samples were verified by X-ray diffractometry (XRD, ULTIMA IV, Rigaku) with Cu Kα radiation. The specific surface area (SSA) of the samples was analyzed by Brunauer-Emmett-Teller (BET; Belsoro-mini2, BEL JAPAN ING) analysis.

III. RESULTS AND DISCUSSION

a. Effect of Temperature and Amount of Added Seed on The Thermal Stability of ATH (KH-101LC)

In order to investigate the effect of temperature on the thermal stability of the product, the temperature of the Bayer liquor before seed addition was adjusted from 75 to 110 °C. The added amount of seed was varied from 15 to 50 g/L. The temperature of the Bayer liquor after seed addition was linearly decreased to 65 °C over 12 h. Analysis of the thermal resistance for each case studied is presented in Figs.1–5. The thermal resistance data of ATH (OL-104LEO) are given for comparison.

As shown in Figs. 1–3, in the temperature range 150–220 °C, the weight loss of samples produced by adding 15–45 g/L of seed was higher than that of OL-104LEO. In the temperature range 150–288 °C (temperature at which the

Table 2. Summary of the experimental conditions and results.

Material	Conditions			Results			
P/L	Seed (g/L)	Temp (℃)	Time (h)	Dp$_{50}$ (μm)	Na_2O (%)	DTG (5℃ /min)	
						220-250℃	250-288℃
Bayer Liquor	15	75-65	12	3.2	0.15	Bad	Bad
	45			2.6	0.17	Bad	Bad
	15	80-65	12	2.8	0.13	Bad	Bad
	30			1.9	0.15	Good	Bad
	45			1.9	0.16	Good	Bad
	30	90-65	12	2.5	0.13	Good	Bad
	45			2.2	0.14	Good	Bad
	30	100-65	12	2.8	0.12	Good	Good

	45			2.8	0.13	Good	Good
	50			2.5	0.13	Good	Good
	30	110-65	12	2.3	0.1	Good	Good
	40			2.4	0.12	Good	Good
	50			2.5	0.12	Good	Good

phase of ATH changes from gibbsite into boehmite is 285 °C), further weight loss of about 2% was observed as compared with that of OL-104LEO. In other words, when the temperature of the Bayer liquid was 75–90 °C, varying the amount of added seed from 15 to 45 g/L does not improve the thermal stability of ATH over the temperature range 150 to 288 °C. Notably, in the temperature range 220–250 °C, the thermal stability of the samples improved upon increasing the seed amount at the same precipitation temperature. Hence, in the following experiments, the amount of added seed was adjusted to 30–50 g/L.

The shapes of the DTG curves in Figs. 4 and 5 are similar to each other. As opposed to the curves in Figs. 1-3, over the tested temperature range, the DTG curves of the produced ATH were very close to that of OL-104LEO. When the temperature of the Bayer liquor was increased to 110 °C, the curves became consistent with those of OL-104LEO. The weight loss of ATH produced with 30 g/L seed addition was smaller than that of OL-104LEO over the tested temperature range (150–288 °C).

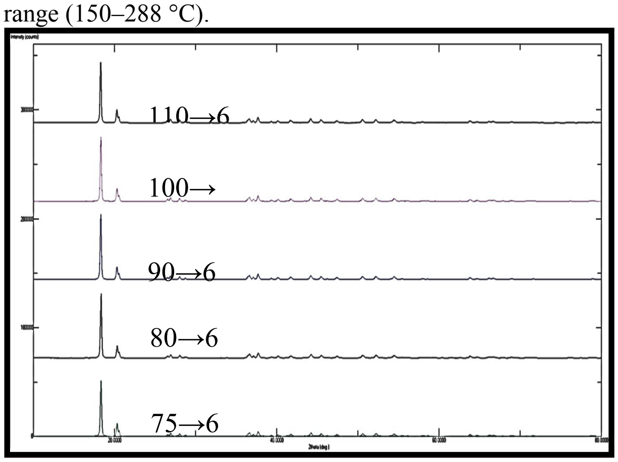

Fig. 6. XRD patterns of ATH powders produced at different precipitation temperatures.

In other words, increasing the Bayer liquor temperature to 110 °C before precipitation can markedly improve the thermal stability of ATH. The experimental conditions and results are summarized in Table 2.

b. Evaluation of other Physical Properties of the Sample Powder

The XRD patterns of samples obtained at different precipitation temperatures are shown in Fig. 6. All the curves are consistent with the standard Gibbsite structure (JCPDS card No: 01-0263). Thus, increasing the temperature did not change the crystal structure of the ATH.

The amount of Na2O in the samples was analyzed, and is presented in Fig. 7. The Na2O content in ATH decreased with an increase in the precipitation temperature from 80 to 110 °C. Accordingly, the thermal stability increased (the weight loss decreased) with a decrease in the Na2O content of ATH.

When the Bayer liquor temperature was 110 °C and the added seed amount was 30 g/L, the thermal stability of the powder produced from KH101-LC can be comparable to or better than that of OL-104LEO.

The SSAs of all the samples were analyzed, as presented in Fig. 8. The results suggest that KH101-LC produced by increasing the precipitation temperature to 110 °C has a much smaller SSA than that of OL-104LEO. This leads to more convenience in applications, especially when ATH is compacted with other materials, for example, in the thermal insulating layer of PCB-CCL.

Therefore, based on the obtained results, we conclude that by increasing the precipitation temperature (Bayer liquor temperature before seed addition) the thermal stability of ATH was increased, because of the decrease in Na_2O content. ATH with the same thermal stability as OL-104LEO was obtained in the Bayer process by increasing the precipitation temperature to 110 °C. This process is more economical and environmentally friendly than the re-digestion process used to prepare thermally stable ATH.

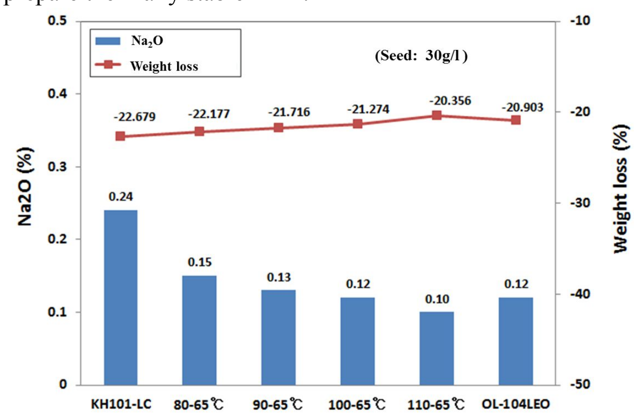

Fig. 7. Variation of the thermal stability and Na_2O content in ATH powders produced at different precipitation temperatures.

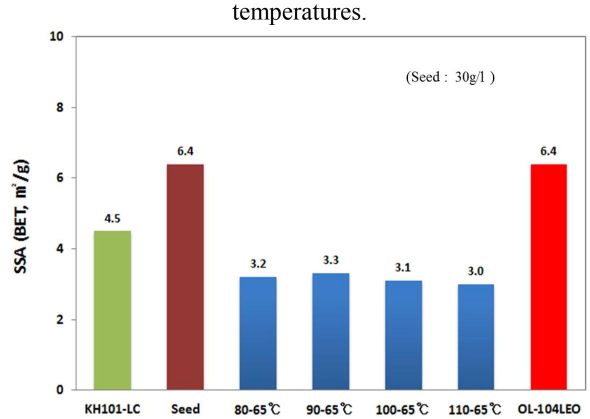

Fig. 8. Variation of the specific surface area (SSA) of ATH powders produced at different precipitation temperatures.

CONCLUSIONS

A procedure for producing ATH with thermal stability comparable to that of OL-104LEO, using KH101-LC, is established by increasing the precipitation temperature and varying the amount of added seed. Increasing the added amount of seed to 30–45 g/L at a precipitation temperature of 80 °C improves the thermal stability of ATH in the low-temperature range (220–250 °C). The thermal stability of ATH is inversely proportional to the Na2O content. ATH with the same thermal stability as OL-104LEO is produced in the Bayer process by increasing the precipitation temperature; the proposed method is economic and environmental important in practice.

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