Application Brief

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Example of Thermal Analysis which is necessary for Ceramics Products Design

—Thermal Analysis of Kaolin—

1. Introduction

Kaolin is clay which contains kaolinite and halloysite etc. It is generated from alteration of granite by weathering and hydrothermal action. Chemical formula is $Al_2Si_2O_5(OH)_4 \cdot nH_2O$ is added for halloysite component. It is necessary for the material of ceramics. It is used for fillers and fire-resistant material. In case kaolin is used for the material of ceramic ware, it is important to understand the thermo physical properties such as shrinking ratio and rate of weight decrease in process control and quality control.

Thermogravimetry / Differential Thermal Analyzer (TG/DTA) and Thermal Mechanical Analyzer (TMA) of thermal analysis instrument are used for evaluating the material of ceramics and the working process as it has the capability to measure endothermic, exothermic phenomena, weight change characteristics, thermal expansion, and thermal shrinkage characteristics.

In this brief, kaolin is measured and evaluated with TG/DTA and TMA.

2. Measurement

The sample is Fisher Chemical Kaolin poweder. The sample is dried and solidified at room temperature after the water is added and mixed.

Measurements are performed using TG/DTA6300 Thermogravimetry / Differential Thermal Analyzers and TMA/SS6300 Thermomechanical Analyzer.

For TG/DTA measurement, the sample is heated at 20℃/min from 30 to 1500℃ in air atmosphere in a platinum open pan. 40mg of powder sample is prepared on measurement sample pan after the dried sample is crushed into powder.

For TMA measurement, the sample is heated from 30 to 1500℃ at 5℃/min in an air atmosphere. Load is 20mN. Alumina ceramics probe and sample cylinder are used. Dried sample is cut off 7mm rectangular and stayed vertically. Load setting for TMA measurement is usually 100mN. However as it is really week against the load, the load value is set small.

3. Results

Figure 1 shows TG/DTA and TMA measurement results of kaolin.

DTA curve shows endothermic peak in the around of 600℃ and exothermic peak in the around of 1000℃.

TG curve shows weight loss in the around of 600℃. TMA curve shows the expansion up to around 450℃ and shrinkage until 1400℃.

From the result, the change which may happen in each temperature range and phenomenon which may occur are summarized in Table 1. It explains regarding 4 ranges of temperature from low to high.

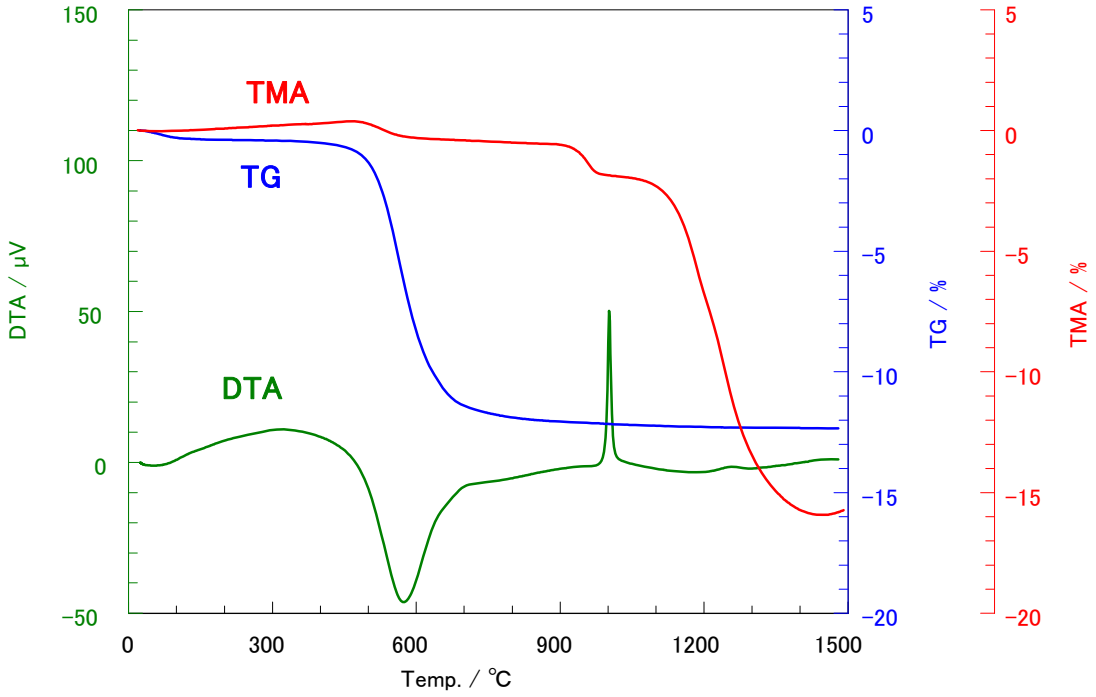

Figure1 TG/DTA and TMA measurement results of kaolin

Table1 Changes in kaolin and the phenomena which likely to occur

Temperature	DTA	TG	TMA	Phenomena
30℃-450℃	—	Weight loss	Expansion	Evaporation of adhesion water and thermal expansion
450℃-700℃	Endothermic peak	Weight loss	Shrinkage	Dehydration of structured water
900℃-1050℃	Exothermic peak	—	Shrinkage	Crystallization of Alumina and silicic acid
1100℃-1400℃	Exothermic peak	—	Shrinkage	Formation of mullite and cristobalite

Figure 2 shows the extended figure for the range from room temperature to 200℃. DTA curve does not show any changes but TG curve shows slight 0.36% weight decrease until 150℃. TMA curve shows only 0.1% expansion. The reason of 0.36% is likely by the water evaporation. Adhesion water is the residue when dried, or the one which vapor is adsorbed on kaolin powder after dried. Moisture-absorption characteristic of kaolin powder can be identified by the amount of adhesion water.

Slight expansion is the thermal expansion caused by temperature increase.

Figure 3 shows the extended figure for the range of 300 to 900℃. DTA curve shows broad endothermic peak at 574℃. TG curve shows 11.6% weight decrease. TMA curve shows 0.7% shrinkage. In this range of temperature, structured water of kaolin dehydrates which causes endothermic, weight decrease, and shrinkage and makes various unique curves. The amount of the dehydration varies up to the composition of kaolin. The information such as weight decrease ratio and compounding ratio can be achieved.

Figure 4 shows the extended figure for the range of 900 to 1050℃. DTA curve shows sharp exothermic peak at 1002℃. TG curve does not show any changes. TMA curve shows slight 1.3% shrinkage. In this range of temperature, exothermic heat and shrinkage likely occur because of the crystallization of alumina and silicic acid. It does not cause weight change as the crystallization keeps mass.

This measurement shows sharp exothermic peak. However, the temperature, size, and shape of the exothermic peak varies greatly depending on where kaolin is extracted. In some cases there would be no exothermic peaks.

Here, there is a difference in temperature in exothermic peak in DTA curve and shrinkage in TMA curve. It is caused by the different in heating rate between that of TG/DTA and TMA. For TMA measurement, as the sample size is big, the temperature distribution in the sample becomes wider in the same heating rate as TG/DTA. Thus the heating rate is reduced to make temperature distribution in the sample smaller, the shrinkage temperature shifted to the low temperature.

Figure 5 shows the extended figure for the range of 1050 to 1500℃. DTA curve shows the small exothermic peak at 1257℃. TG curve does not show any changes. TMA curve shows 14% shrinkage. In this temperature range, mullite and cristobalite are formed which causes exothermic heat and shrinkage. It does not cause weight change as the mass is kept. The shrinkage ratio in this temperature range is biggest. In case it is used as the material of ceramics, shrinkage ratio has to be considered before it is fabricated. Measurement of the shrinkage ratio by TMA can be a valuable part of this fabrication process.

4. Summary

As a result of the TG/DTA and TMA measurement of kaolin, in the order from low temperature, the phenomena such as evaporation of adhesion water, dehydration of structured water, crystallization of alumina and silicic acid, and the formation of mullite and cristobalite have occurred. It shows the unique peaks and shifts in DTA, TG ,TMA curves. By evaluating some data of thermal analysis, we can understand each unique phenomenon and characteristics. Also, the presence of endothermic heat and exothermic heat, the quantitative determination of weight decrease ratio and shrinkage ratio can be understood from each curve. It enables the evaluation of ceramic material production process.

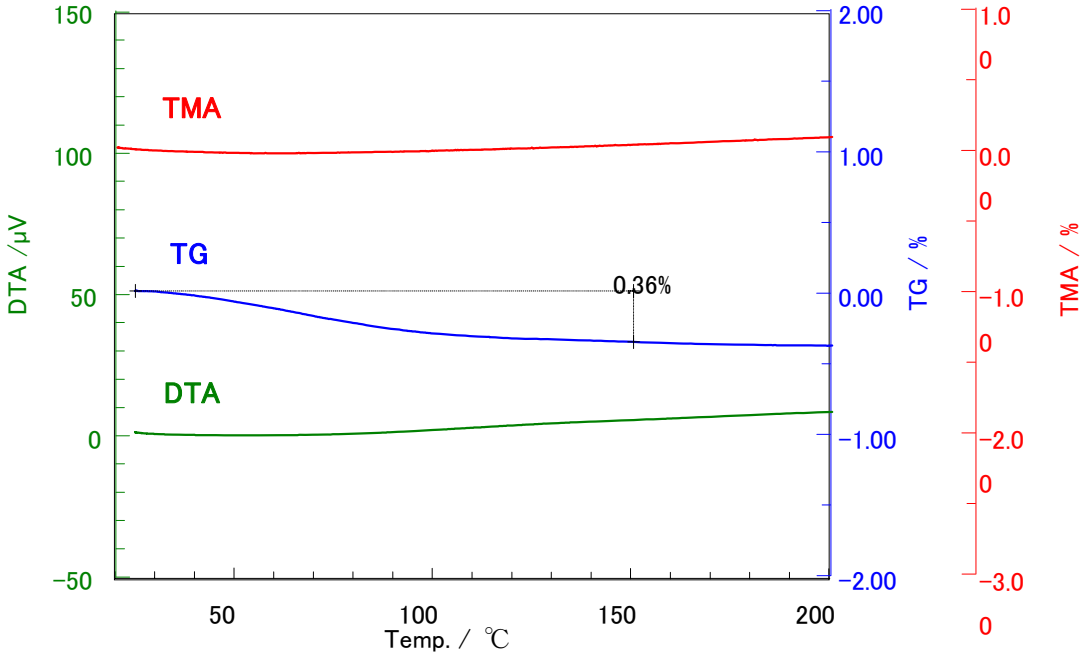

Figure 2 Close Up of the measurement result temperature range: R.T. to 200℃

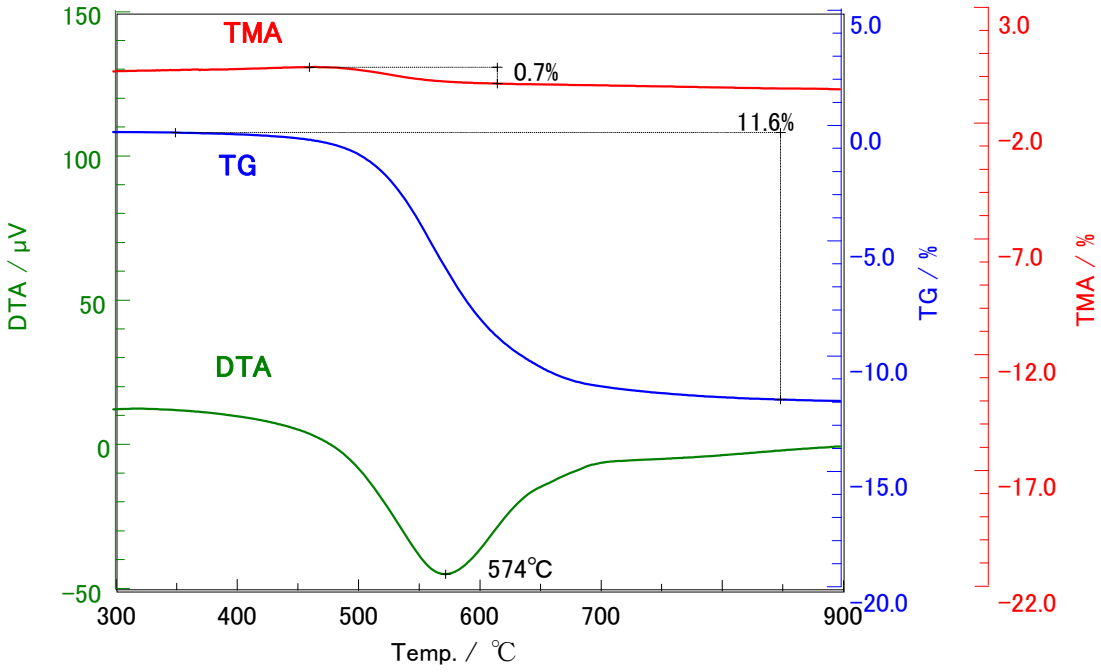

Figure 3 Close Up of the measurement result temperature range: 300℃ to 900℃

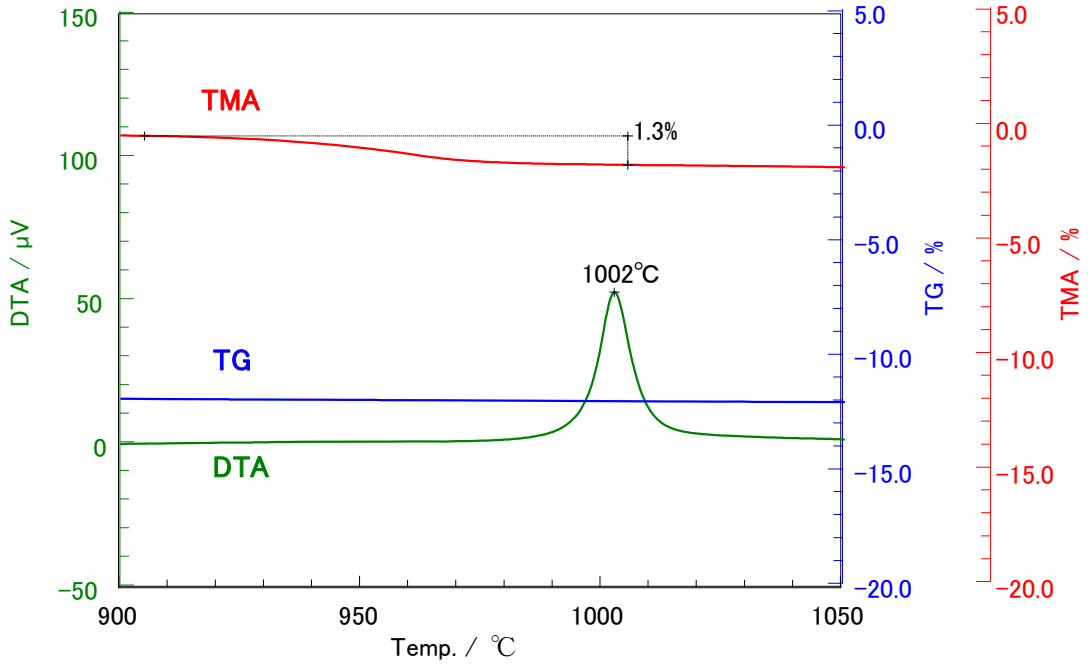

Figure 4 Close Up of the Measurement Result temperature range: 900 to 1050℃

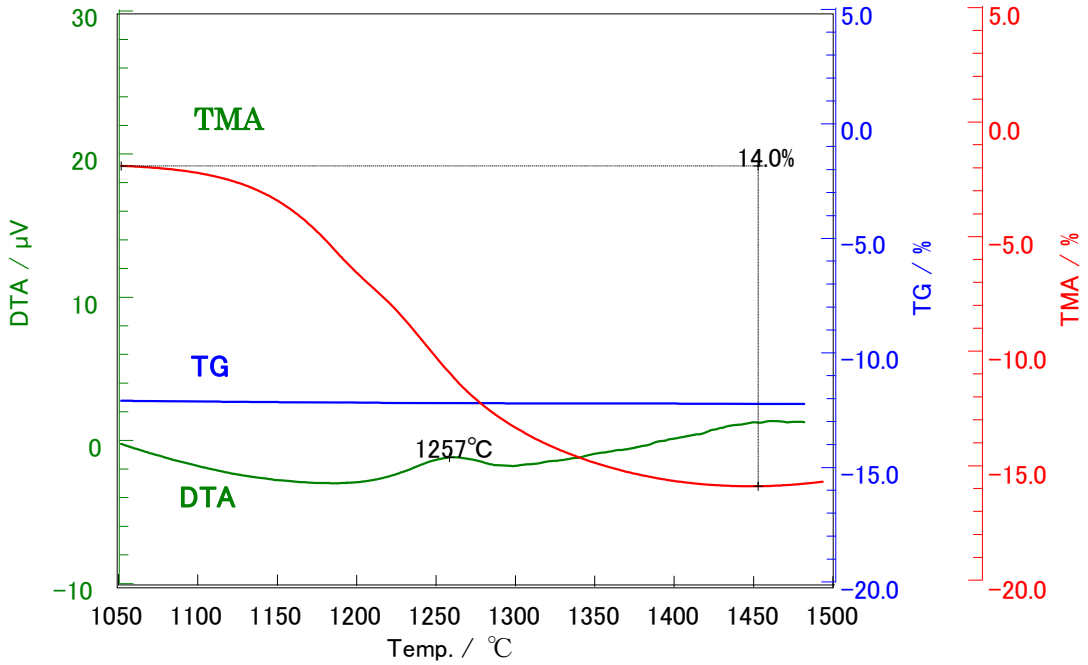

Figure 5 Close Up of the measurement result temperature range: 1050 to 1500℃