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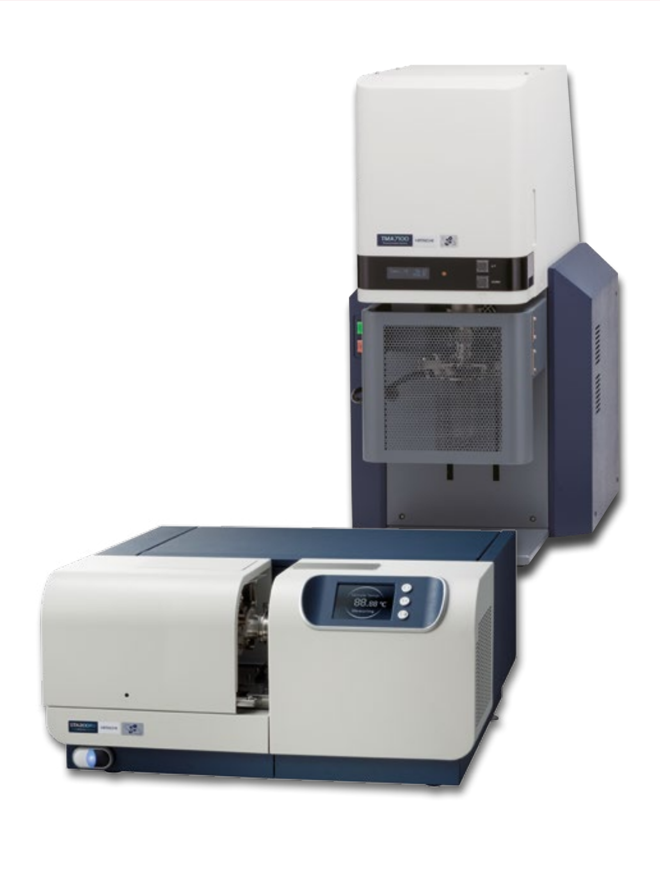
NEXTA STA300 & TMA7300



Thermal analysis of kaolin for ceramic production

INTRODUCTION

Kaolin, or china clay, is a soft white clay that has been used for centuries in porcelain production. Today, kaolin is used in the manufacture of many products, from rubber to medicines, but it's still an important raw material in the ceramics industry thanks to its light color and high firing temperatures.

The natural white color of kaolin and its high fusion temperature makes it ideal for white ceramic products. It is also used as a filler and a fire-resistant material. As a mined product, kaolin is made up of the mineral kaolinite and varying amounts of other minerals, such as halloysite. Additional minerals are added to the raw material during manufacture, as well as other processing, such as bleaching, that is necessary if the raw material contains yellow iron oxide pigments.

For ceramic production, kaolin is mixed with silica, feldspar and ball clay to ensure the final product has the right characteristics. Plasticity and shrinkage need to be at the right level so that the kaolin behaves as expected during the molding and firing process during ceramics manufacture. To ensure the kaolin performs as expected during manufacture, the thermal-physical properties of the material, such as shrinking ratio and weight decrease, are measured as part of the pre-production quality control process.

The thermal analysis techniques simultaneous thermo gravimetric analysis (STA) and thermal mechanical analysis (TMA) are the methods used for evaluating the processing characteristics of kaolin prior to molding and firing. Using these two methods together allows you to fully evaluate the properties of the clay including water evaporation and dehydration, crystallization of alumina and silicic acid and verify whether mullite and cristobalite have formed. Hitachi's NEXTA STA300 and TMA7300 are ideal for kaolin analysis due to their wide temperature range, low noise and high sensitivity.

Hitachi High-Tech Analytical Science's family of thermal analyzers have been employed in the field for more than 45 years, delivering world-class performance for precise materials and process characterization measurements, including thermal analysis of kaolin in ceramic manufacturing.

HITACHI INSTRUMENTS FOR THERMAL ANALYSIS OF KAOLIN

NEXTA STA300

Hitachi's NEXTA STA300 makes it easy to ensure that raw materials meet specification and behave as expected during production. With an unsurpassed level of baseline stability, world-class sensitivity, and advanced, high temperature TGA and DSC capability, the NEXTA STA slots easily into ceramic production quality control and development programs. Automated features, such as a 50-slot auto-sampler, help to speed up analysis to aid high-volume throughput.

TMA7300

The Hitachi TMA7300 is a versatile thermo mechanical analyzer that can evaluate expansion, shrinkage, creep, stress relaxation and even DMA within a single, solid and extremely reliable and sensitive instrument. Invaluable for incoming quality assurance or outgoing inspection, the TMA7300 is versatile enough to support new product development projects where a wide variety of thermo mechanical analyses are needed.

For thermal analysis of kaolin for ceramic applications, these analyzers work together to give a complete picture of thermal behavior. Both are designed to meet Hitachi's high reliability standards to minimize downtime of the instruments. Within these analyzers, high-sensitivity technology and extremely low levels of noise ensure you get accurate results. And both instruments can reach temperatures up to 1500ºC, essential when analyzing kaolin.

ALL-INCLUSIVE SOFTWARE WITH CAPACITY FOR NEW APPLICATIONS

Both analyzers come with Hitachi's intuitive and advanced NEXTA TA software, which gives you options for how you need to operate the instrument. New users can get reliable and accurate results and experienced operators can use these instruments for more advanced analysis. All modules are included with the instruments, so if you decide to expand your use into new applications, you won't have to purchase additional modules. The software includes three modes of operation:

- Guidance mode for step by step measurement and analysis including a calibration wizard.
- Simple mode for more experienced users carrying out routine analysis that requires a simple interface. All important features are available on the main screen.
- Standard mode where all modules are included, and more complex analysis can be set up.

PERFORMANCE AND RESULTS

A Fisher Chemical sample of kaolin powder was used for these measurements. Prior to analysis, the sample was mixed with water and then dried and left to solidify at room temperature. The test conditions were as follows:

TG / DSC measurements with the NEXTA STA300
Sample size: 40mg of crushed powder
Heating rate: 20ºC / minute
Temperature range: 30ºC to 1500ºC
Atmosphere: air
Sample pan: Platinum open pan

TMA measurements with the TMA7300
Sample size: 7mm rectangle of dried sample
Heating rate: 5ºC / minute
Temperature range: 30ºC to 1500ºC
Atmosphere: air
Sample pan: alumina sample cylinder
Load setting: 100mN

TG/DSC and TMA measurement results for kaolin over entire temperature range

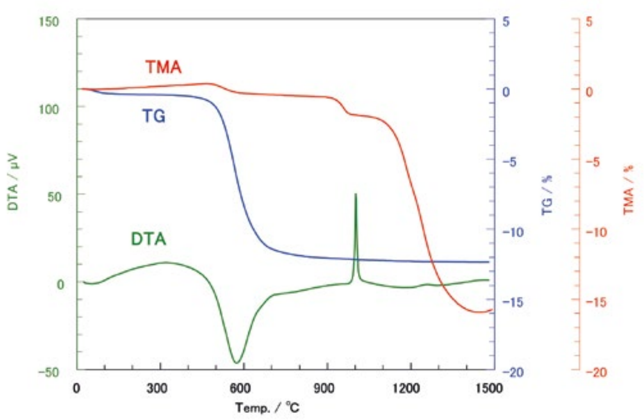


Figure 1 TG/DSC and TMA measurement results for kaolin

Figure 1 presents the results from 30ºC to 1500ºC. Table 1 below summarizes the results seen for each trace and the changes within the kaolin that are likely to beoccurring at each stage

Temperature	DSC	TG	TMA	Phenomena
30ºC — 450ºC	—	Weight loss	Expansion	Evaporation of adhesion water and thermal expansion
450ºC — 700ºC	Endothermic peak	Weight loss	Shrinkage	Dehydration of structured water
900ºC — 1050ºC	Exothermic peak	—	Shrinkage	Crystallisation of alumina and silicic acid
1100ºC — 1400ºC	Exothermic peak	—	Shrinkage	Formation of mullite and cristobalite

We'll now look at four temperature ranges more closely to discuss how the DSC, TG and TMA results can be used together to characterize kaolin.

Results for room temperature to 200ºC

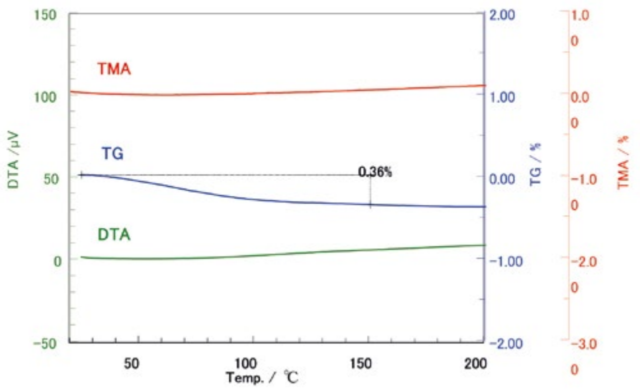


Figure 2 Detailed view of the measurement result temperature range: R.T to 200ºC

In this lower temperature range, the DSC curve is flat, but the TG curve shows a slight weight increase of 0.36% up to 150ºC. This is likely due to water evaporating from the sample and this value is useful when determining the moisture absorption characteristics of the powder. The TMA curve indicates a 0.1% expansion which is thermal expansion caused by the increase in temperature.

Results for 300ºC to 900ºC

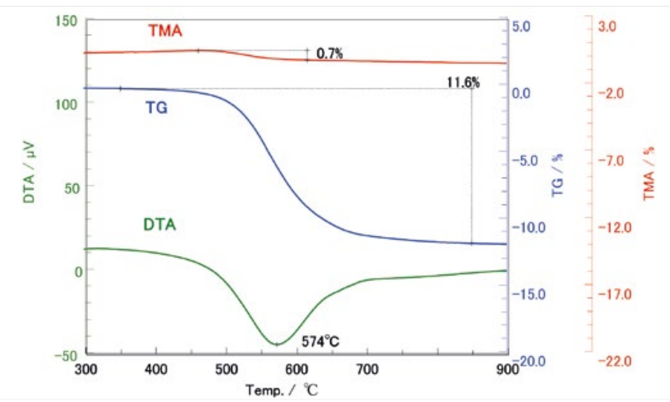


Figure 3 Detailed view of the measurement result temperature range: 300ºC to 900ºC

Within this temperature range, we can see a broad endothermic peak at 574ºC within the DSC curve, a weight decrease of 11.6% from the TG curve and a shrinkage of 0.7% from the TMA measurements. This is the temperature range where kaolin dehydrates, losing the water contained within the structure, and the endothermic peak, reduction in weight and shrinkage can all be attributed to this dehydration. The amount of water loss varies with the kaolin composition, and these results can give information on weight decrease ratio and compounding ratio.

Results for 900ºC to 1050ºC

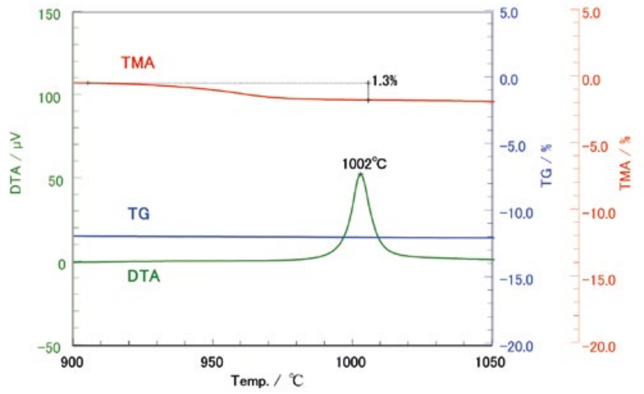


Figure 4 Detailed view of the measurement result temperature range: 900ºC to 1050ºC

The DSC curve for this temperature range shows an exothermic peak beginning at 1002ºC and the TMA curve shows a small shrinkage of 1.5%. Within this temperature range, the most likely cause of these results is the crystallization of alumina and silicic acid. As the crystallization process does not change the mass of the sample, the TG curve remains flat. The amount of alumina and silicic acid within a kaolin sample can vary enormously, and the temperature, size and shape of the exothermic peak at this temperature range will vary according to composition.

NB: The DSC and TMA curves on figure 4 imply the shrinkage and exothermic peaks occur at different temperatures. In reality, this is due to the different heating rates of the STA and TMA. As the sample size is larger in the TMA, the temperature distribution within the sample would be quite large if the heating rate was at 20ºC / min. Thus, the heating rate is reduced to 5ºC / min, which appears to reduce the shrinkage temperature.

Results for 1050ºC to 1500ºC

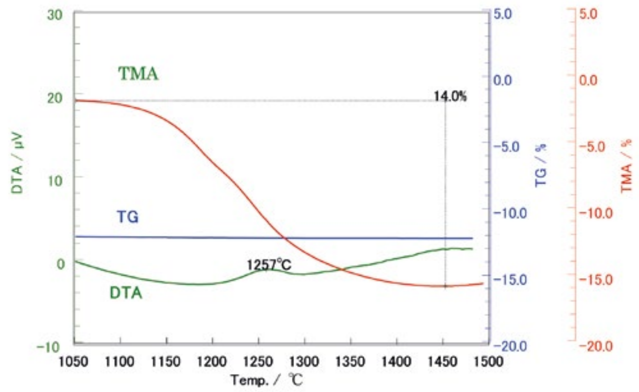


Figure 5 Detailed view of the measurement result temperature range: 1050ºC to 1500ºC

For this highest temperature range, the DSC curve shows a small exothermic peak at 1257ºC and the TMA curve shows a 14% shrinkage. These can be attributed to the formation of mullite and cristobalite within the sample. There is no change of mass, so the TG curve remains flat. The relatively large shrinkage at this temperature range is an important factor to consider during ceramic fabrication, and measurement of the shrinkage ratio by TMA gives valuable information on what to expect.

SUMMARY

The **NEXTA STA300** and the **TMA7300** can be used in tandem predict the thermal behavior of kaolin during molding and firing. A large temperature range, low noise and high sensitivity ensure reliable results for shrinkage and dehydration ratio during production, and also give information about the composition of the raw kaolin and formation of cristobalite and mullite at high temperatures.

Many applications have been optimized for Hitachi High-Tech Analytical Science's thermal analyzers. For more information on other applications, please contact our experts at **contact@hitachi-hightech.com**.

NEXTA STA: COMPLETE QUANTITATIVE THERMAL ANALYSIS

Designed for complete thermal analysis of materials, including thermal resistance, decomposition temperature, melting point and specific heat testing, the NEXTA STA combines DSC and TGA to deliver TGA applications and more within a single analyzer.

The NEXTA STA range offers:

- Ultimate accuracy and precision even when measuring trace amounts of material
- Superior heating technology that meets the most advanced applications of TGA
- Cp measurement in a wide temperature range
- Unique, Real View camera system for viewing material behaviour on screen
- Easy to use with automated features, intuitive software and simple report creation

TMA7000: VERSATILE THERMAL ANALYSIS OF MECHANICAL CHARACTERISTICS

The TMA7000 range can carry out a range of measurements within a single instrument – from precise TMA analysis, including thermal expansion, glass transition and softening, to DMA testing and creep measurement.

The TMA7000 range offers:

- High accuracy with a high sensitivity, low noise TMA signal over a wide measurement range of up to +/- 5 mm
- Ability to carry out TMA, DMA, stress-strain, creep and stress-relaxation measurements
- Capability to measure minor changes of low-expansion material and thin films
- Versatility for a range of applications due to superior control technology and a complete range of measurement probes
- Improved thermal efficiency with auto cooling systems that reduce operating costs and increase throughput

Visit **www.hitachi-hightech.com/hha** for more information.

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