Application Note TA6003

View on rigaku.com

TA6003 - Evolved gas analysis of PEFC

Introduction

In thermal analysis, the measurement condition influences the measurement results. Therefore, it is important to select the most desirable measurement condition in response to the objective of the measurement.

Conditions such as sample amount, heating rate as well as the measurement atmosphere are selected freely in thermal analysis measurement. Especially the measurement atmosphere, where it directly affects the sample's reaction, for example, measuring under atmospheric air may cause combustion or oxidation reaction and the suppression of these reactions can be confirmed by measuring under inert atmosphere. Therefore, it is essential to select the appropriate atmospheric gas suitable to the objective of the measurement.

The dehydration behavior of the sample is most affected by the effects of humidity and in some cases degradation reaction may also be affected. Hence, it is important to measure under a predetermined humidity to perform simulation on the sample's thermal behavior. Also, evaluating the material's hydroscopic behavior (percentage of water absorption) as a property is the most important factor. On the other hand, since dehydration behavior is determined from the heating process, accurately estimating the coefficient of water absorption is extremely difficult in the conventional thermal analysis.

Researchers have expended intense effort on fuel battery for energy resources of next generation. It is expected to applying the auto mobile, mobile phone and PC so on because polymer electrolyte fuel cell enables to make small size and operating temperature is lower than the other fuel battery.

We study the thermal analysis for electrolyte membrane in humidity atmosphere. Humidity control TG-MS is performed for evolved gas of electrolyte membrane, perfluorosulfonic acid, at thermal decomposition reaction. Since coupling TG-DTA and MS, it is available to measure not only for mass change but also qualitative of evolved gas at thermal decomposition reaction. In addition, coupling with the humidity generator, HUM-1, is available to perform the evolved gas analysis in both ordinary dry and humidity atmosphere.

Instrument: ThermoMass Photo

ThermoMass Photo is an evolved gas analytical system designed for real-time simultaneous measurements of thermogravimetry – differential thermal analysis (TG-DTA) coupled with electron impact ionization (EI) and the fragment-free photoionization (PI) mass spectrometry (MS) that performs measurements as a function of temperature or time. Humidity generator, HUM-1, is connected to ThermoMass Photo system. It enables to the user to perform heating under a constant relative humidity.

Measurement and analysis

TG measurement of Perfluorosulfonic acid, Nafion®, in dry and humidity, 12.0 kPa: 60℃,60%RH, atmosphere is shown Figure 1. The ion chromatograms of m/z 64 for SO_2, 47 for CFO, 50 for CF_2 and CF_3 for 69 are shown Figure 2.

In TG measurement result, in both of dry and humidity atmosphere are observed two stepwise of mass loss at around 300℃; however, a second mass loss is observed on the low temperature side in humidity atmosphere. In the MS ion thermogram, there is no difference in generation temperature of SO_2 (m/z 64) at around 300 ℃ in both atmospheres; however, in humidity atmosphere is observed like evolved gases of CF_2 due to decomposition on low temperature side at over 400℃ . This tendency is the same as the TG measurement result. Therefore, although decomposition of $-SO_3$ in end group of perfluorosulfonic acid is less affected by water vapor, decomposition of fluorine compound occurs on the lower temperature side, influenced more by water vapor than in dry atmosphere.

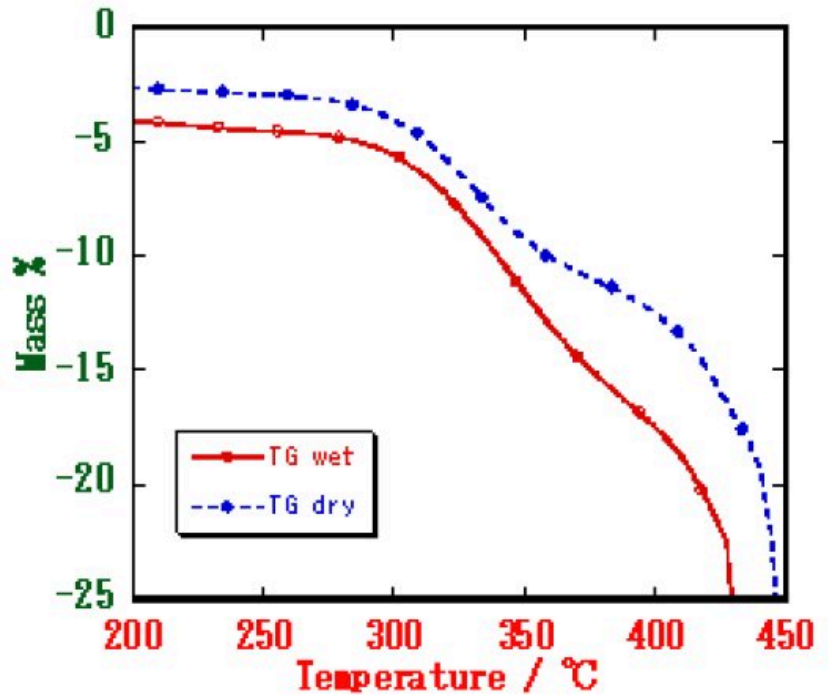

Figure 1: TG measurement result of Nafion
(Sample amount: 10 mg, heating rate: 10℃/min)

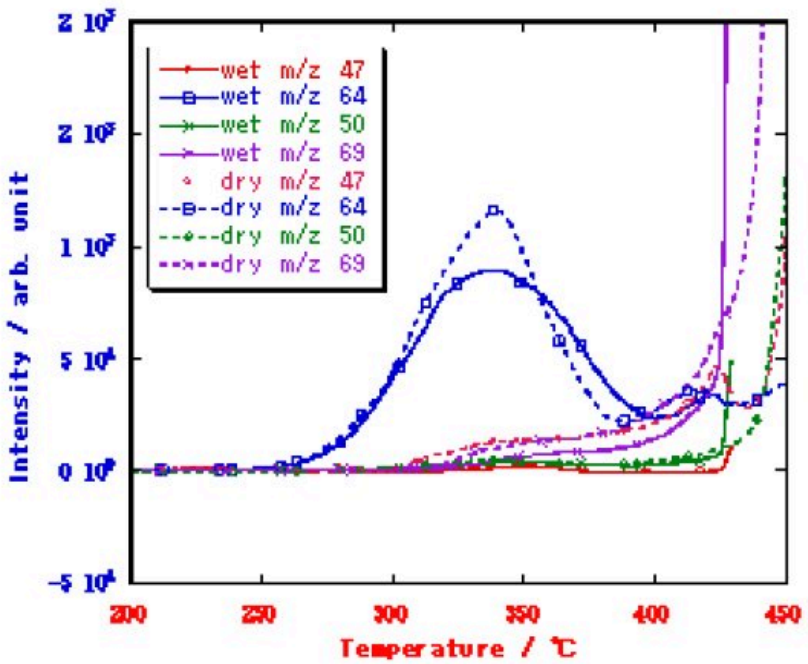

Figure 2: MS ion thermograms of Nafion

Related products

ThermoMass Photo

An integrated thermal analysis instrument capable of high-precision mass analysis of evolved gases without breaking the molecules, allowing direct measurement.