Investigation of ignition due to oxidative decomposition by using TG/DTA with sample observation

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Hidehiro Takahashi[1], Eita Shimoda[1], Yoshikazu Nishiyama[1], Masaaki Muraoka[1], David Peter Semiono[2]
([1]Hitachi High-Tech Science Corporation, [2]Hitachi High-Technologies Europe GmbH)

Introduction

In conventional TG/DTA, the furnace opacity precludes direct sample observation during measurement. Thus, the physical changes of the sample relative to the changes in TG and DTA signal were never understood as well as they could be. The phenomena experienced by the obscured sample could only be estimated by DTA and TG curves.

We developed a newly-designed TG/DTA furnace that allows sample observation during the measurement, and showed application data for this instrument [1], [2], [3]. When wood material decomposed by oxidation, the sample observation TG/DTA observed the exothermal peak, the weight loss and the ignition simultaneously [4].

In this presentation, 6 materials were measured by sample observation TG/DTA at various conditions. Some result showed the ignition on the oxidative decomposition.

Various kinds of carbon were also measured by this system and compared the results. Especially the relations between the ignition and the measurement conditions of carbon nanotube were examined.

Samples and Conditions

Materials

- Nitrile butadiene rubber (NBR)
- Wood chip
- Graphite rod
- Graphite powder
- Carbon Nanotube Multi-walled, 3-20nm（CNT MW3-20）
- Carbon Nanotube Multi-walled, 40-60nm（CNT MW40-60）

Measurement:

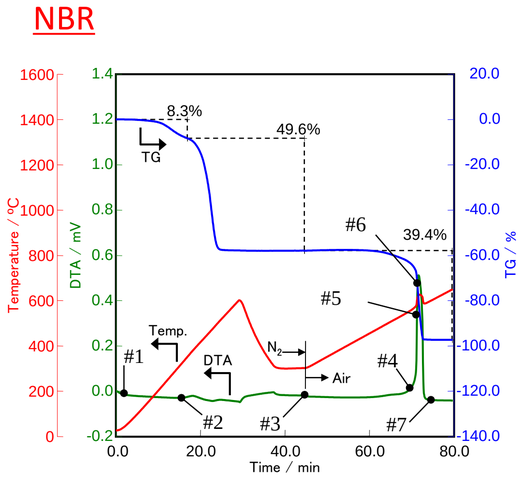
Sample Pan : Platinum open pan
Gas flow : Air 200 mL/min

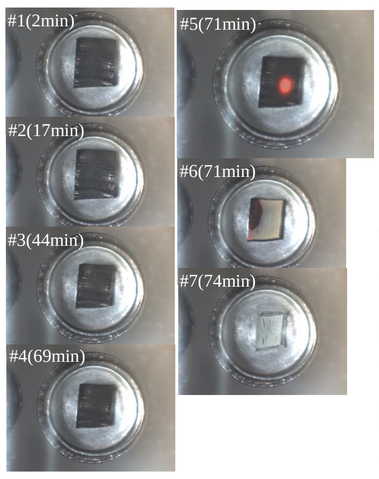
STA system
with sample observation unit

Horizontal digital differential system

Results

NBR

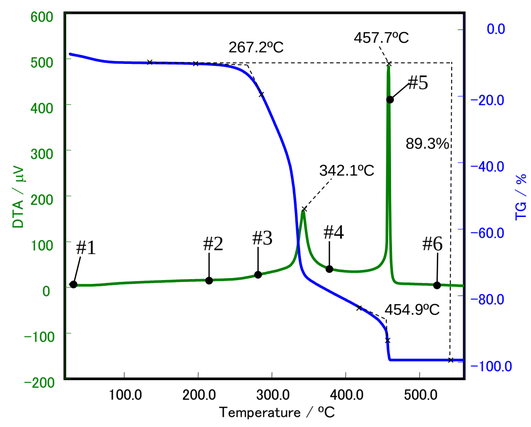


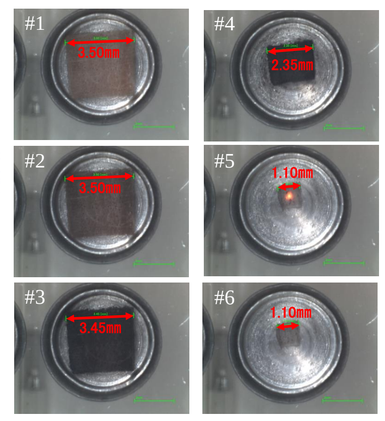


The shrinkage and the weight loss occurred simultaneously. And the ignition of the material were able to observe.

・Sample Weight : 5 mg
・Rate : 20 ºC/min(N_2), 10 ºC /min(Air)
・Gas flow : N_2 => Air 200mL/min

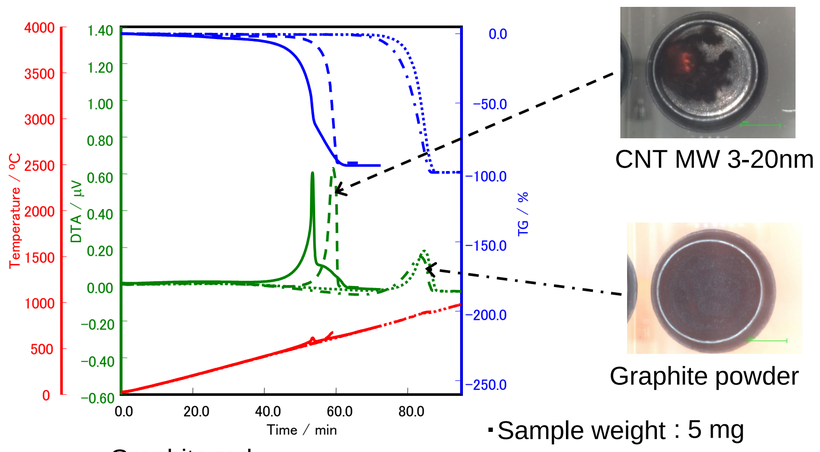
Wood Chip





・Sample weight : 5 mg
・Heating rate : 10 ºC/min

Various Carbon Forms



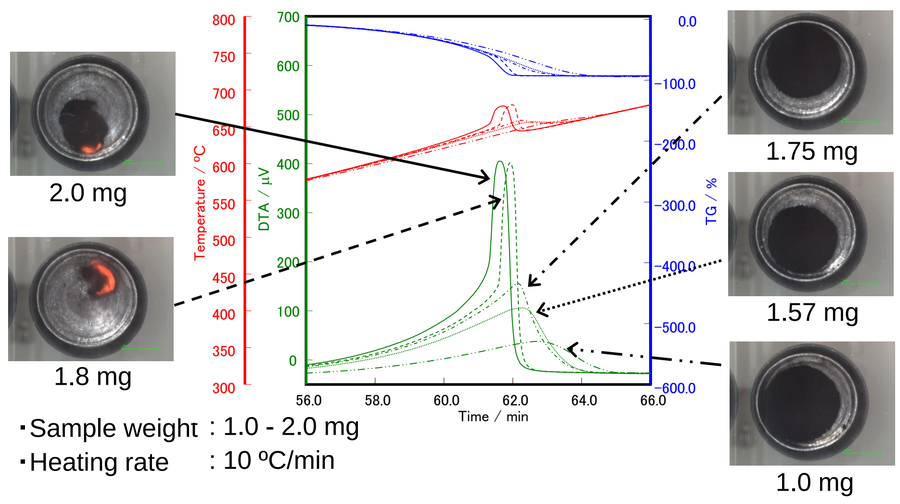
CNT MW 3-20nm

Graphite powder

・Sample weight : 5 mg
・Heating rate : 10 ºC/min

Graphite rod
Graphite powder
CNT MW 3-20nm
CNT MW 40-60nm

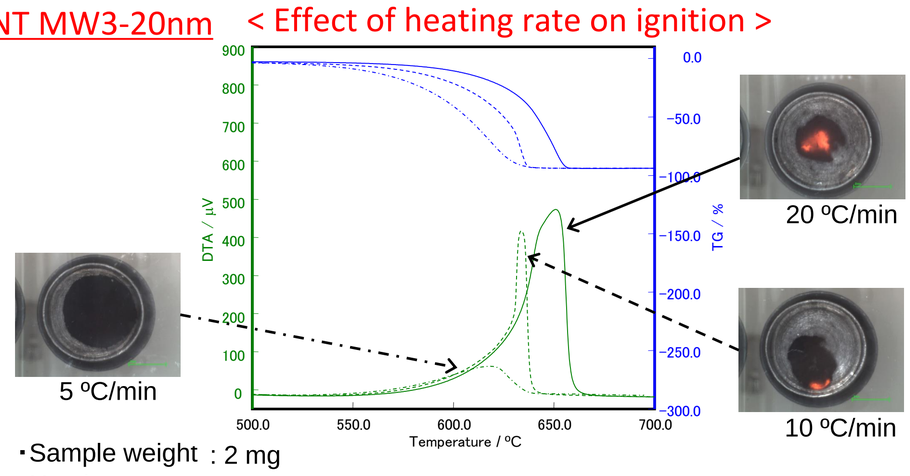
CNT MW3-20nm < Effect of sample weight on ignition >



・Sample weight : 1.0 - 2.0 mg
・Heating rate : 10 ºC/min

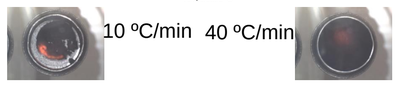
The ignition depended on the sample weight or the heating rate.
And when the ignition occurred, the DTA peak had a characteristic shape.

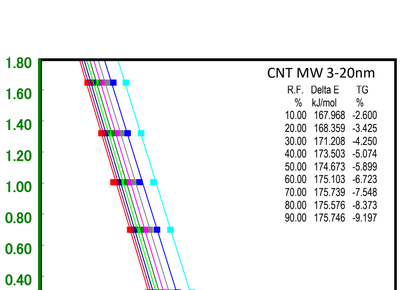
CNT MW3-20nm < Effect of heating rate on ignition >

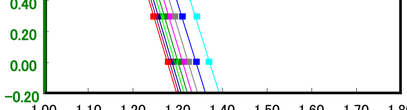


・Sample weight : 2 mg
・Heating rate : 5, 10, 20 ºC/min

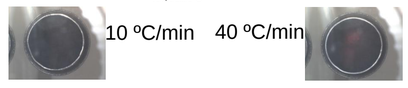
CNT MW 3-20nm

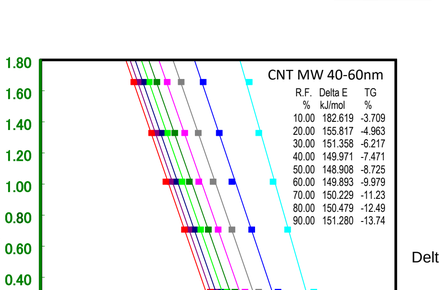


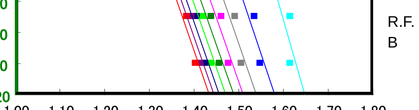




CNT MW 40-60nm







The ignition of CNT WM3-20 was clearer than that of CNT MW40-60.
The kinetic analysis were performed by the Ozawa method to each TG signal of CNT.

DeltaE : Activation Energy (kJ/mol)
R.F. : Reaction Ratio (%)
B : Heating Rate (ºC/min)

Summary

#1 NBR and pieces of wood were measured by STA with the sample observation unit. When the specimens were decomposed or carbonized, we were able to observe the weight loss and the shrinkage of the material simultaneously. Next, the oxidation decomposition of the carbon was observed. The decomposition revealed ignition.

#2 Study of the ignition during carbon oxidation decomposition suggests that there is a structure dependence.

#3 The ignition depends on the sample weight or the heating rate.

#4 Future direction: additional study of sample configuration, activation energy and ignition.

[1] Y. Nishiyama, K. Shibata, K. Yamada, “Development of TG/DTA with optical observation and its advantage”, 49th Japanese Conference on Calorimetry and Thermal Analysis, Nov. 1st 2013, Narashino (Japan)
[2] Y. Nishiyama, H. Takahashi, S. Nishimura, “The design for high temperature measurement of TG/DTA that enabled optical observation”, 50th Japanese Conference on Calorimetry and Thermal Analysis, Sep. 30th 2014, Osaka (Japan)
[3] H. Takahashi, E. Shimoda, Y. Nishiyama, “The evaluation of oxidation decomposition using optical conservation TG/DTA”, 51st Japanese Conference on Calorimetry and Thermal Analysis, Oct. 8th 2015, Saitama (Japan)
[4] Brian Goolsby, K. Shibata, M. Iwasa, Y. Nishiyama, “Development of a TG/DTA Instrument with High-Resolution Sample Observation Capability and Some Applications”, 43rd Annual Conference of the North American Thermal Analysis Society, Aug, 12nd 2015, Montreal (Canada)