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

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1 Background

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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.
Hitachi 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 optical observation TG/DTA observed the exothermal peak, the weight loss and the ignition simultaneously [4].
In this presentation, the wood material was measured by optical observation TG/DTA at various heating rate. The result showed that there was relation the ignition and the heating rate.
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.

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Sample

- Nitrile rubber(NBR)
- Wodden piece
- Epoxy resin
- Graphite rod
- Graphite powder
- Carbon Nanotube Multi-walled, 3-20nm
- Carbon Nanotube Multi-walled, 40-60nm

Measurement

Instrument : STA7200RV with Optical observation unit
Gas flow : Air 200mL/min
Sample pan : Pt open pan

STA measurement were performed by various heating rate or various sample weight. And the relation between the results and the ignition were examined. We also got the activation energy by using Kinetic analysis software.

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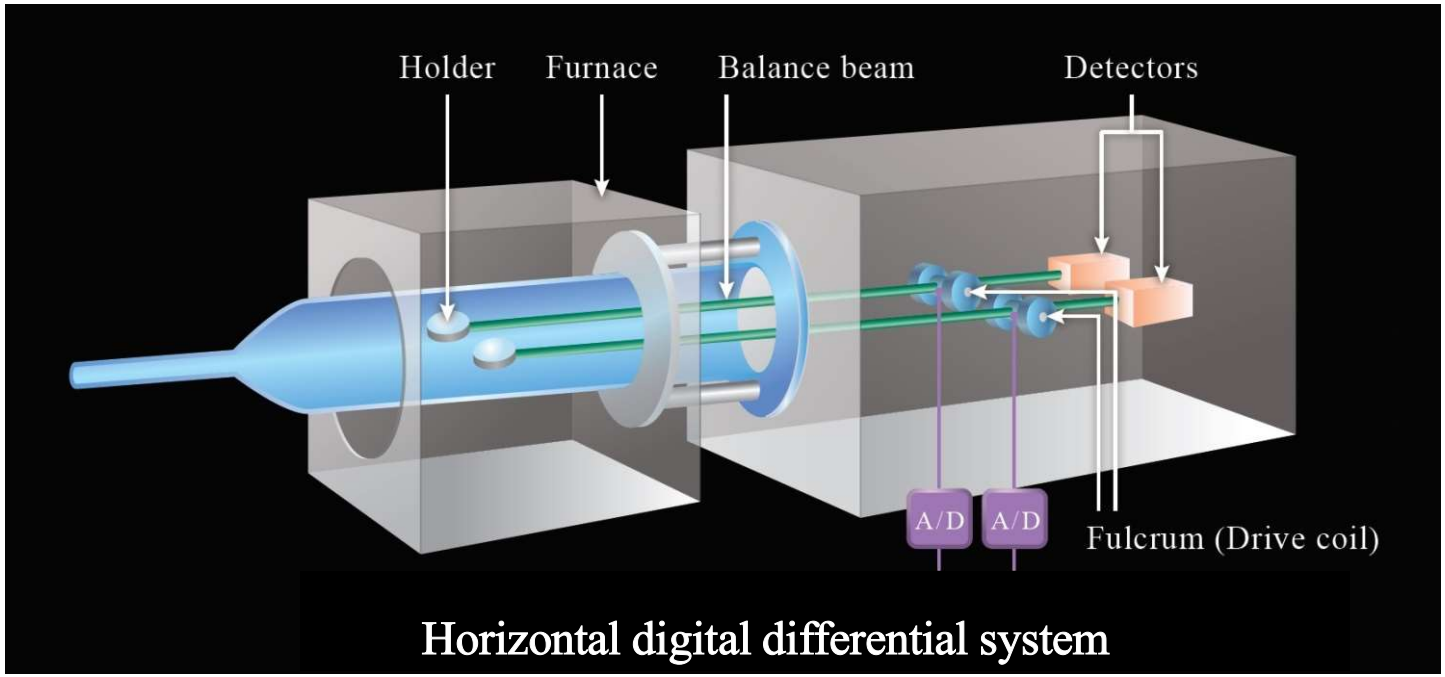
Instruments

Conventional STA



Horizontal digital differential system

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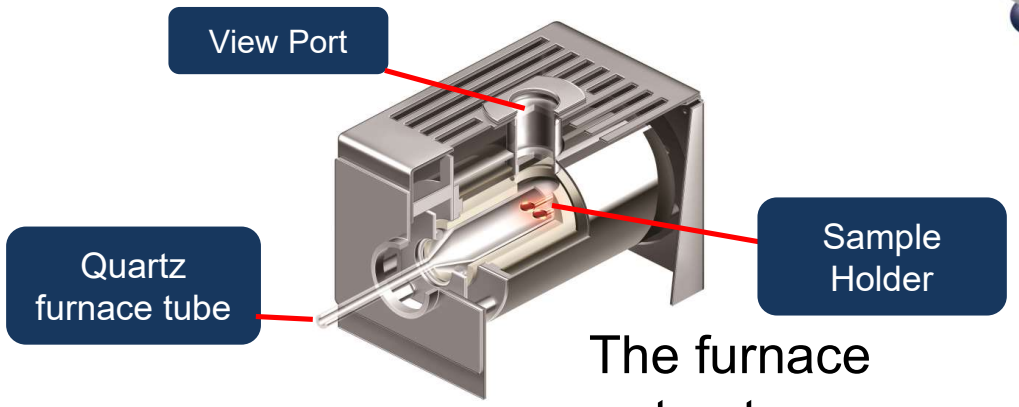
Instruments

STA system with Optical observation unit and Auto Sampler



The furnace structure

- ◆ 10M pixel camera (high-resolution photography)
- ◆ Digital zoom (ten phases, up to x5.7 magnification)
- ◆ The sample position adjustment free,
  the focal adjustment free,
  the illumination adjustment free.
- ◆Available for Auto Sampler

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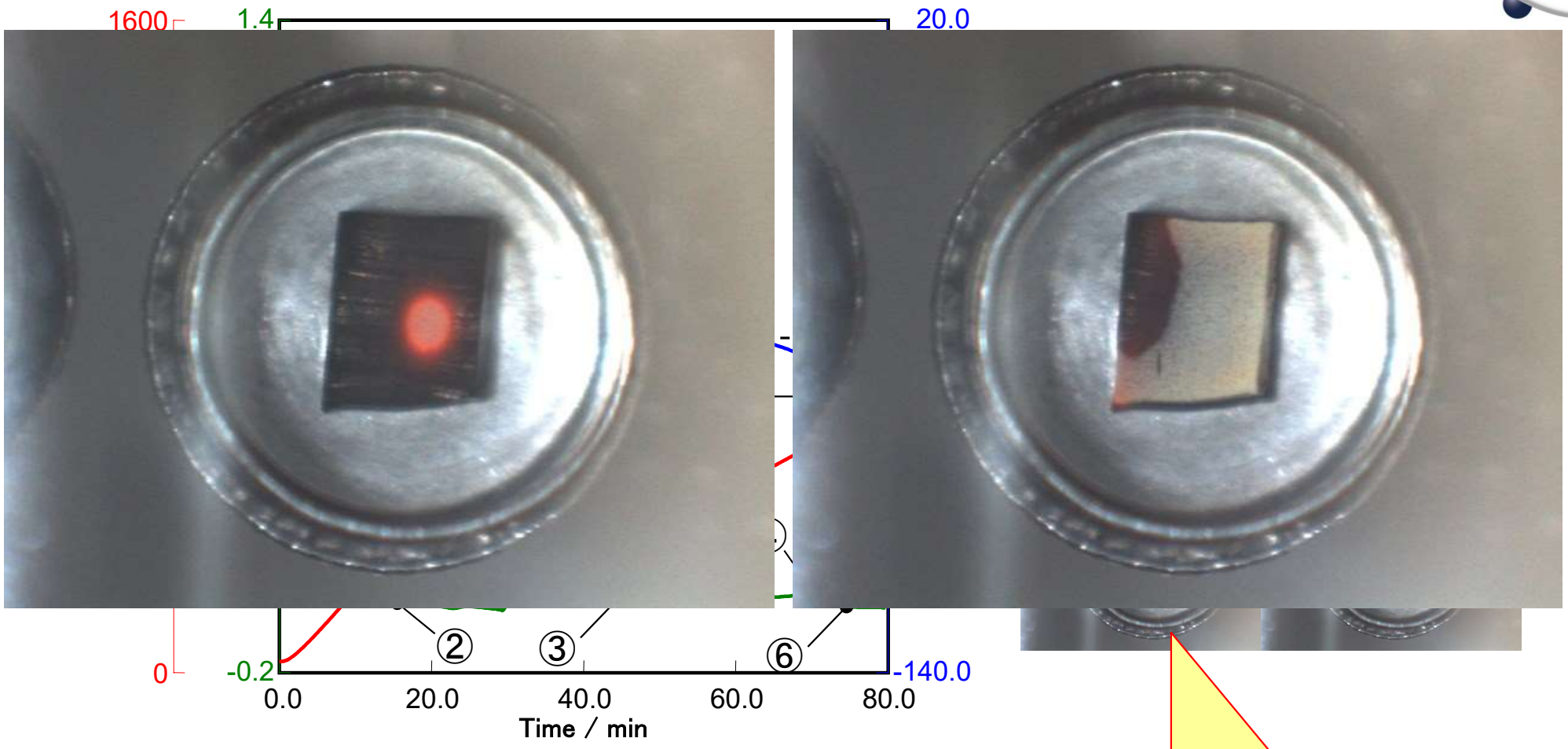
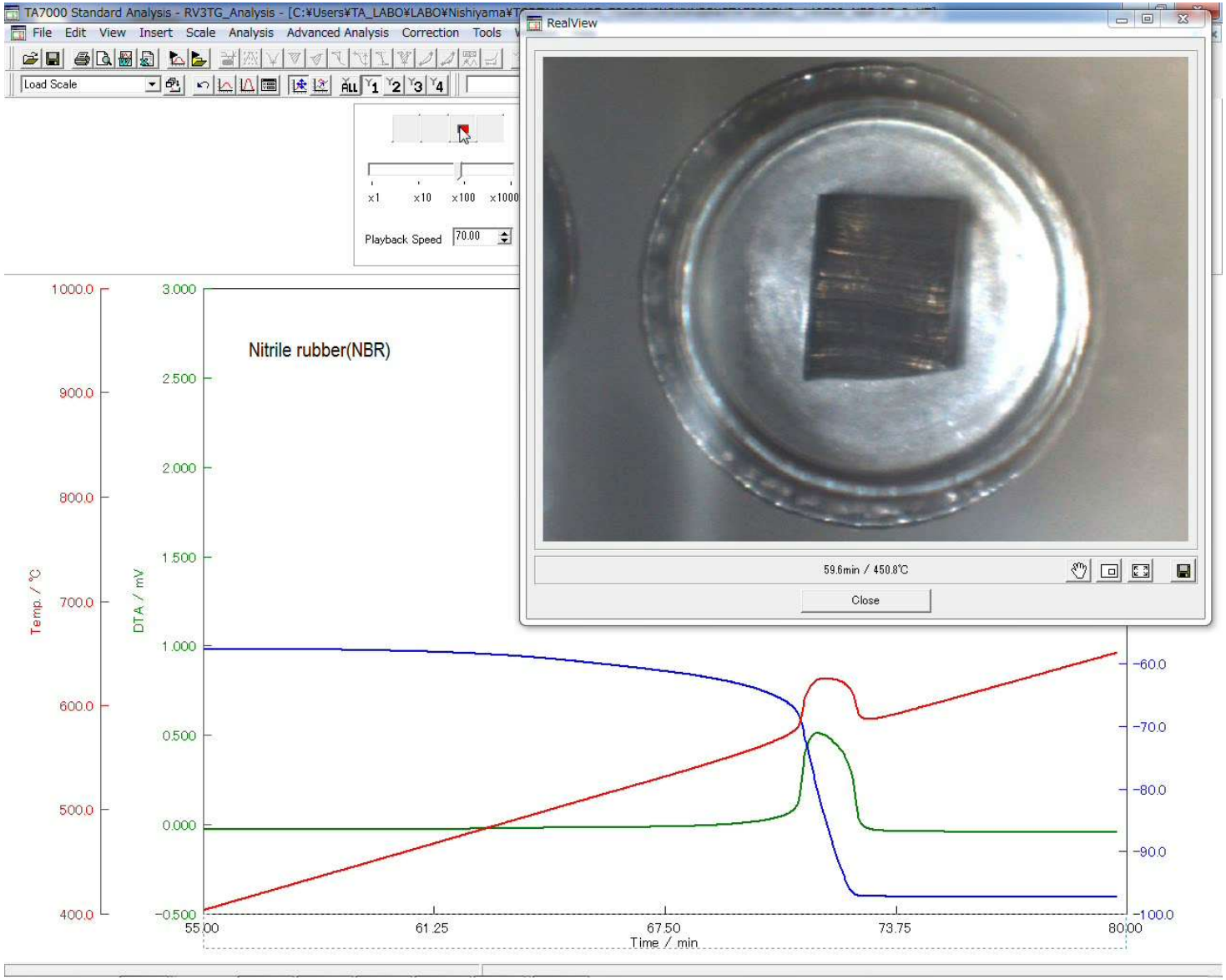
3 Results :Nitrile rubber(NBR)



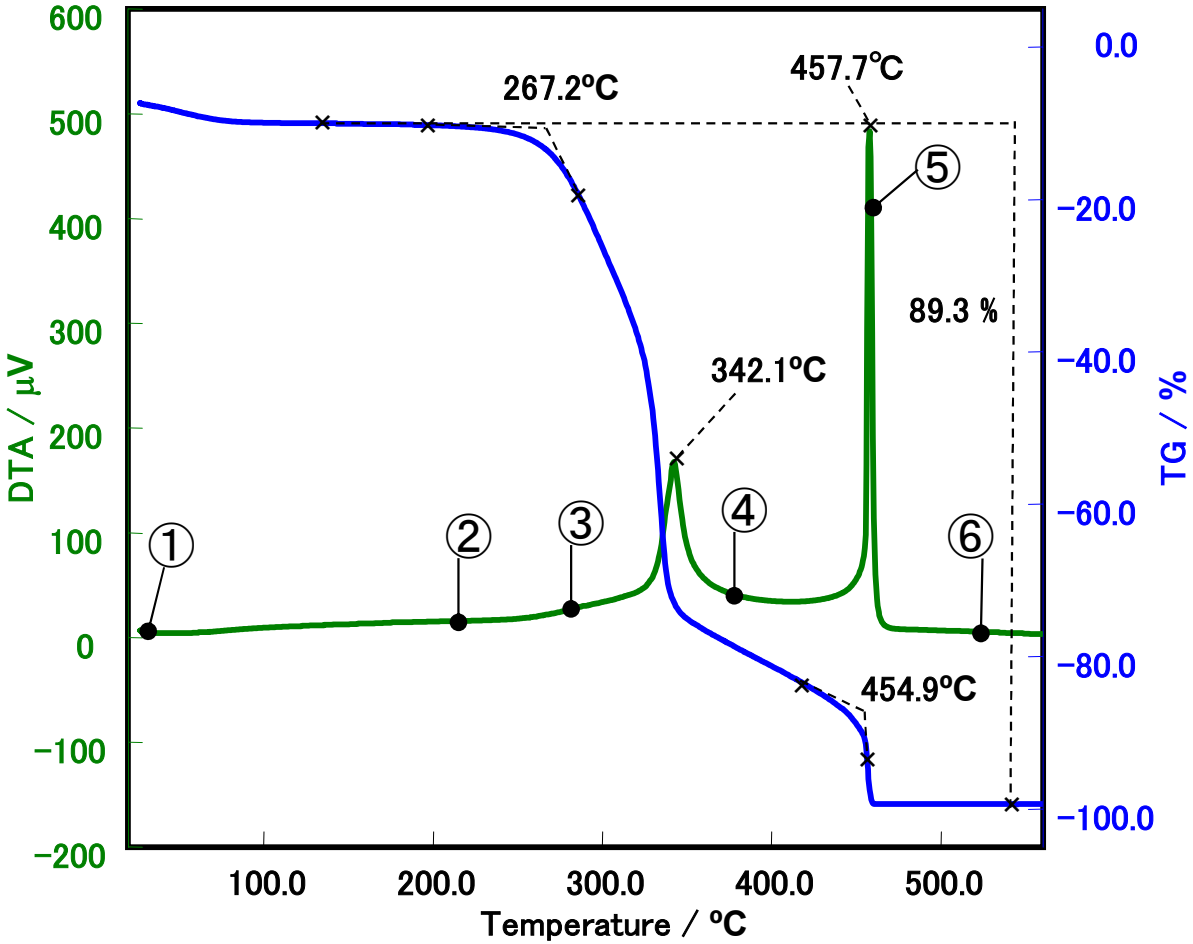
- Sample Weight : 5 mg
- Rate : 20 ºC/min(N_2), 10 ºC /min(Air)
- Gas flow : N_2 => Air 200mL/min

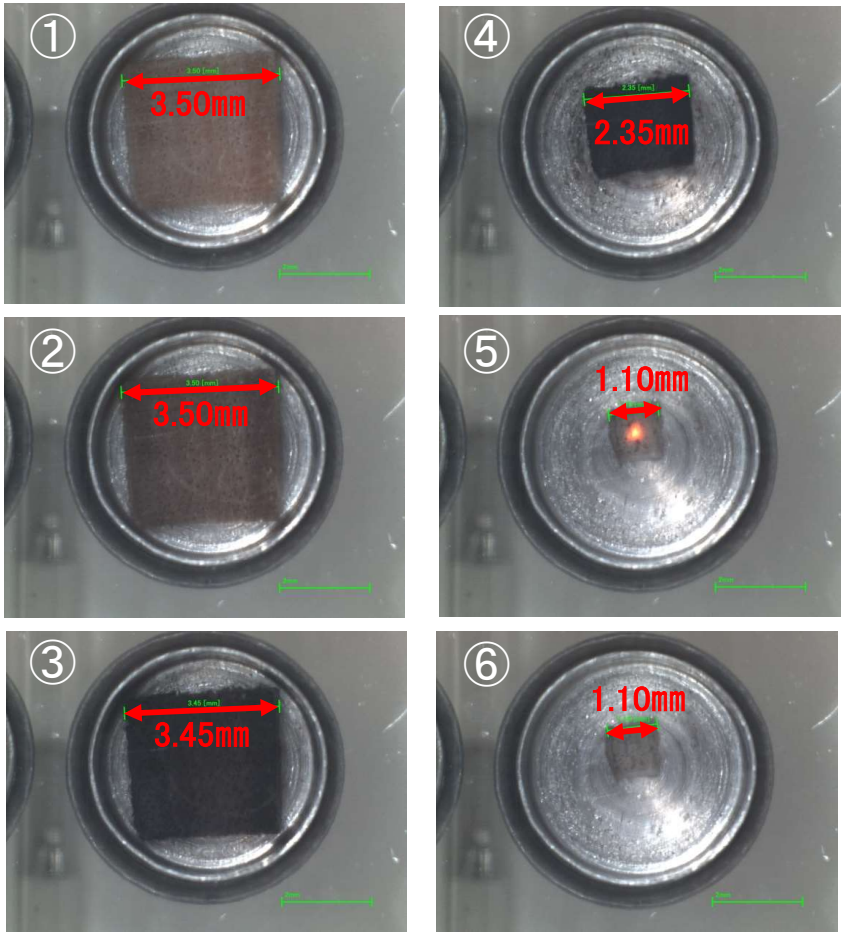
Ignition due to oxidative decomposition of the carbon.

3 Results :Nitrile rubber(NBR) movie



3 Results :Wooden piece





- Sample weight : 5 mg
- Heating rate : 10 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

3 Results :Wooden piece

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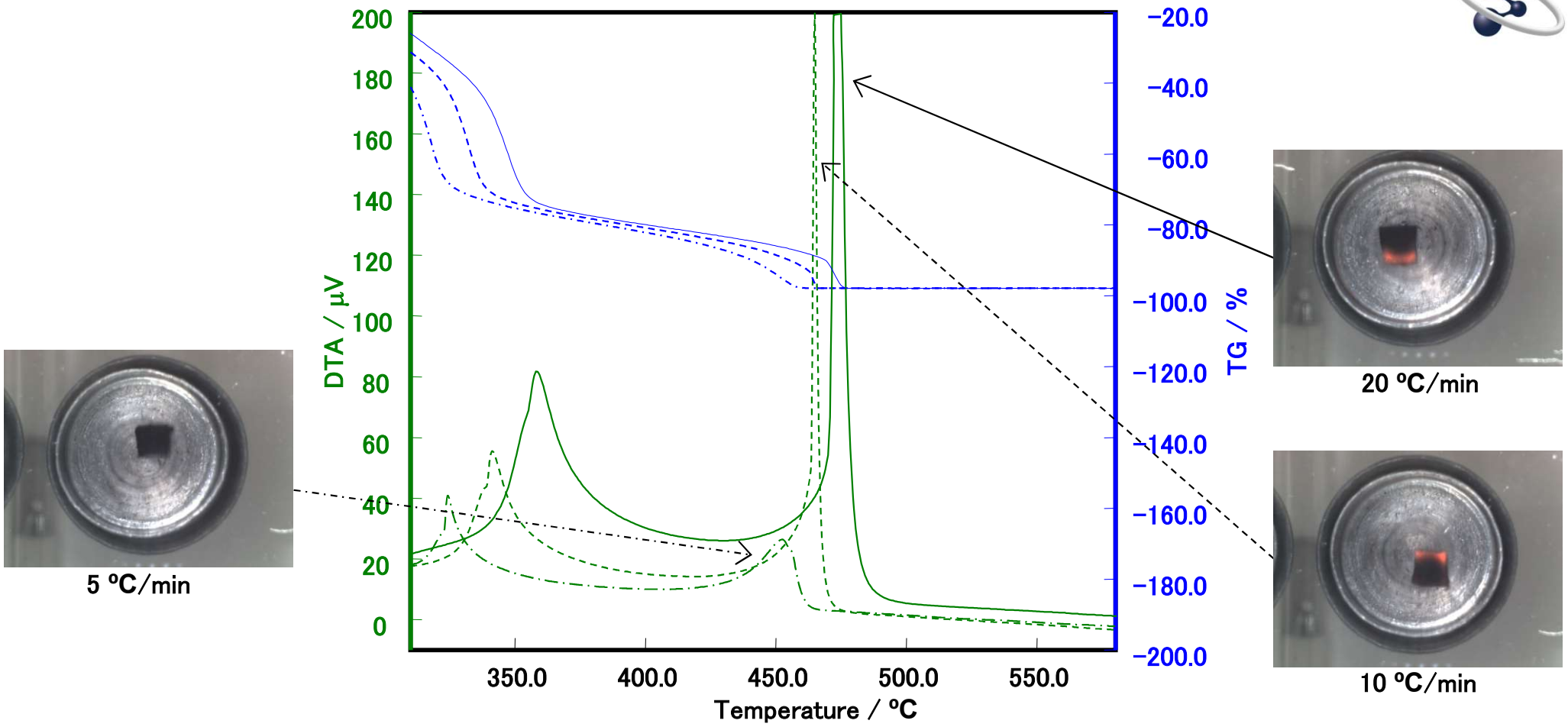
the relation between the ignition and the heating rate



5 °C/min
10 °C/min
20 °C/min

- Sample weight : 2mg
- Heating rate : 5, 10, 20 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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3 Results :Wooden piece -movie

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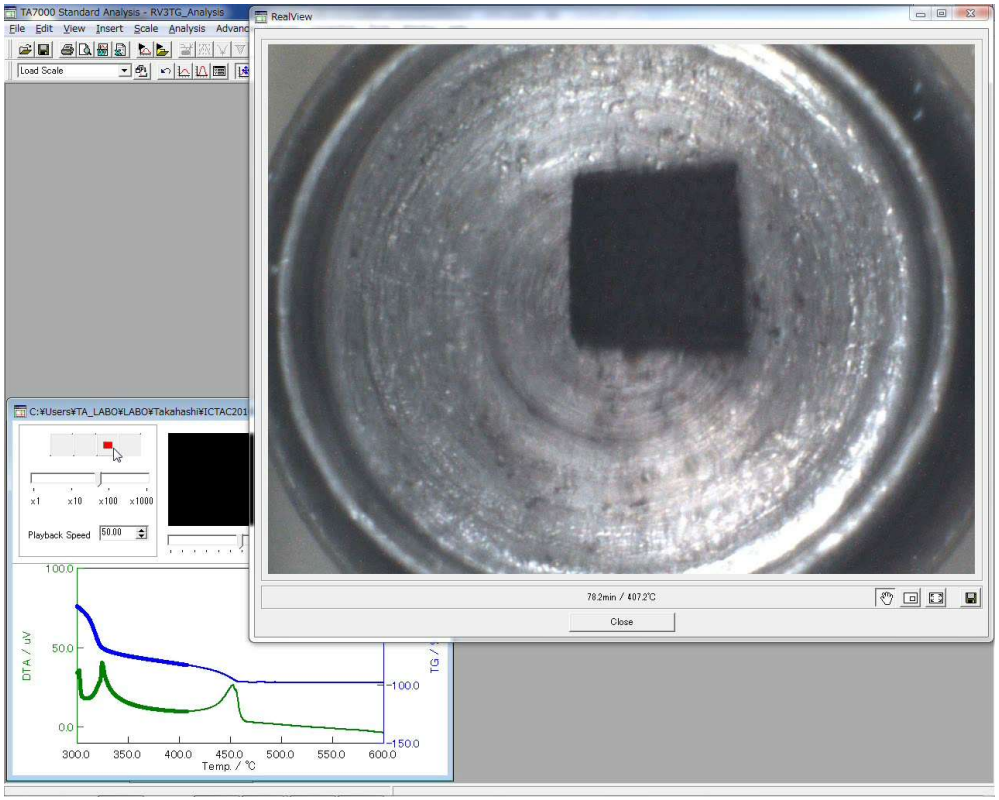
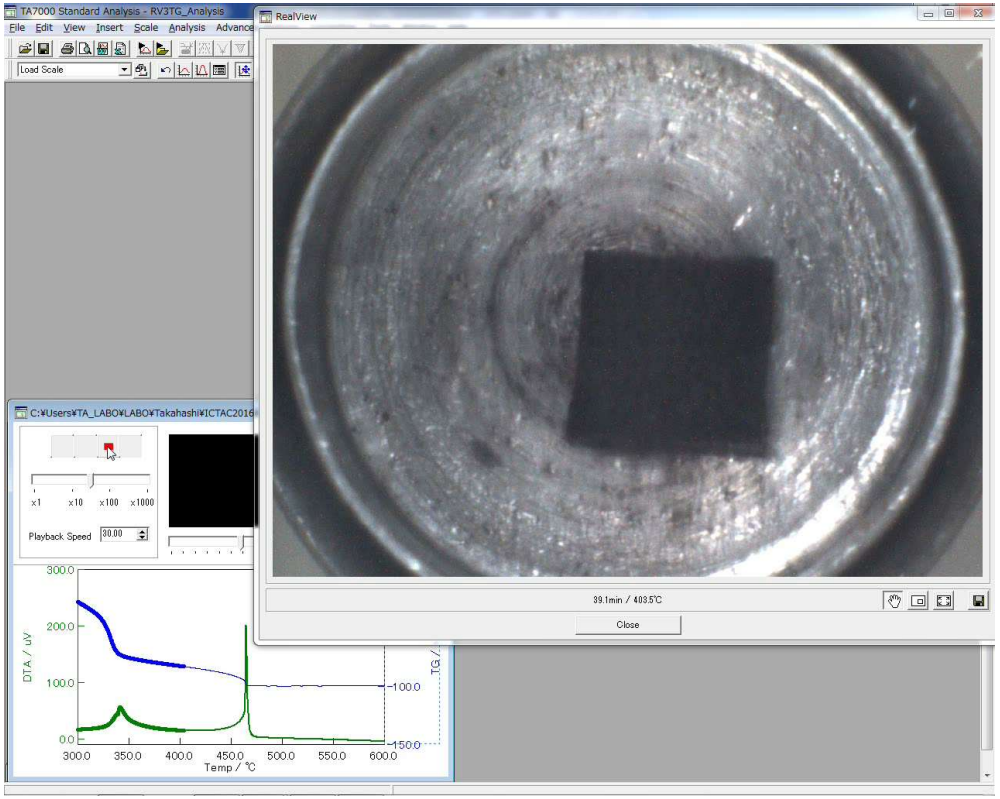
the relation between the ignition and the heating rate



5 ºC/min



10 ºC/min

- Sample weight : 2 mg
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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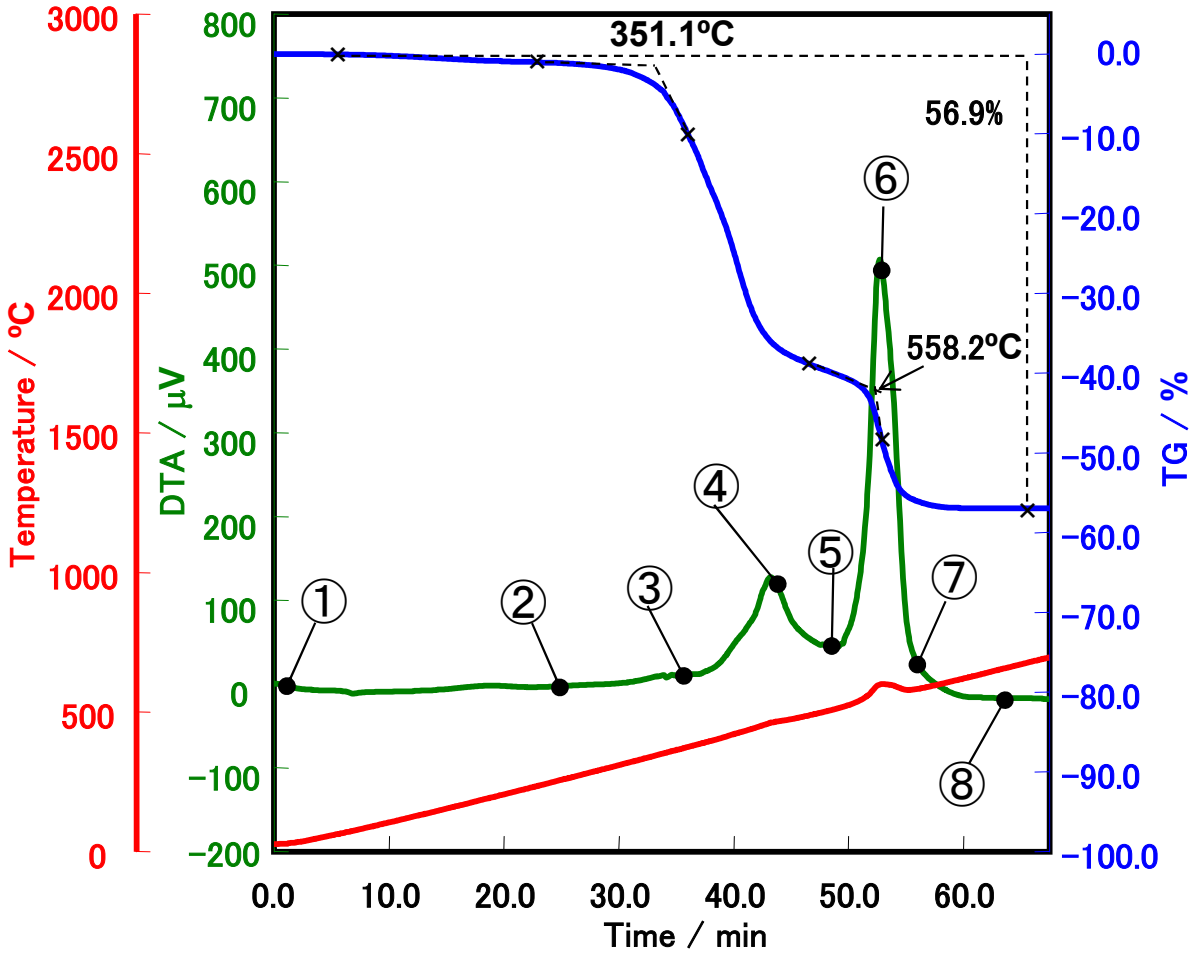
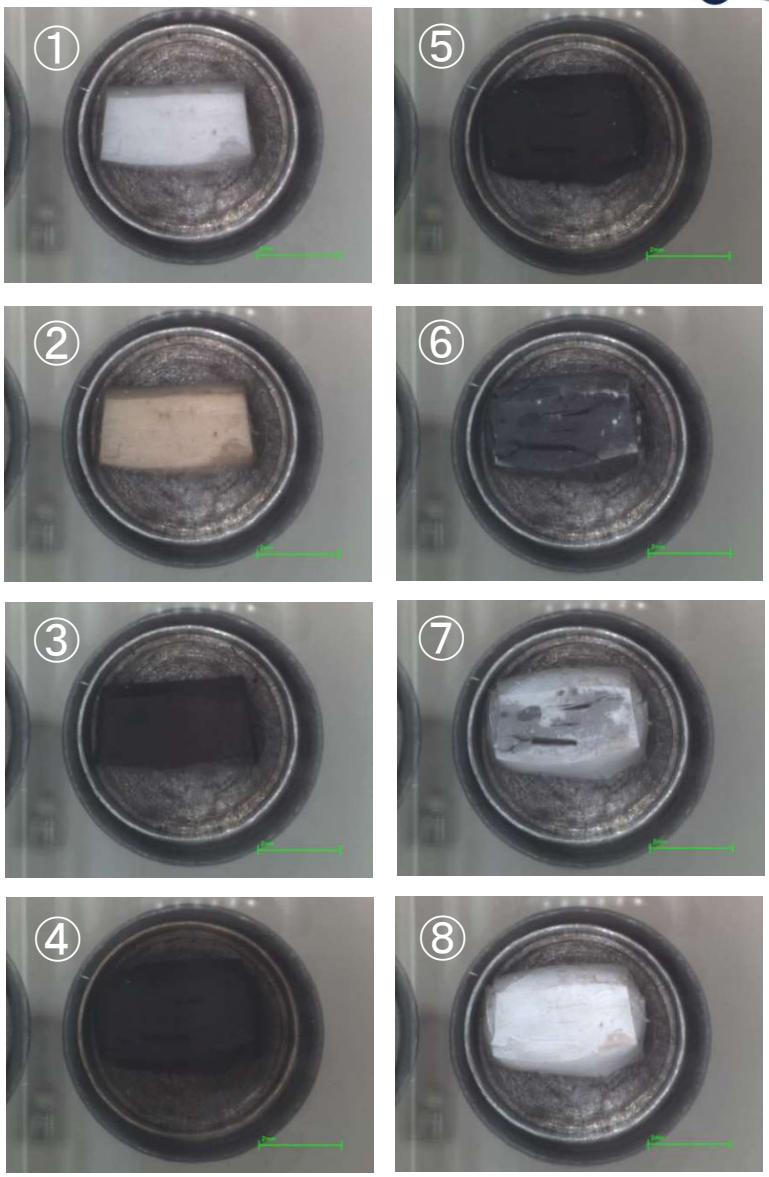
3 Results :Epoxy resin

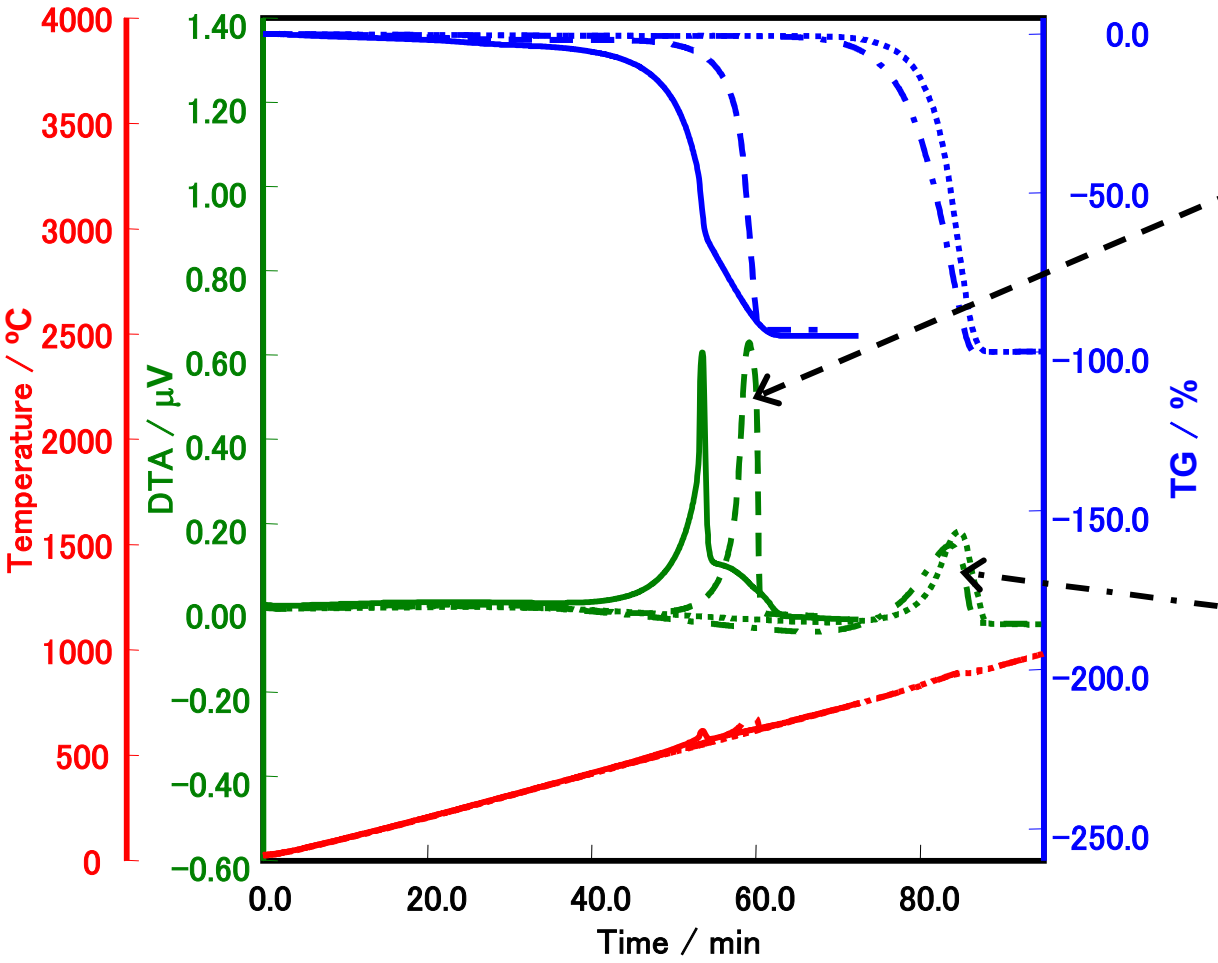


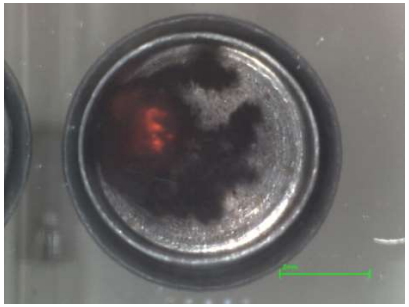


Epoxy resin with Al2O3

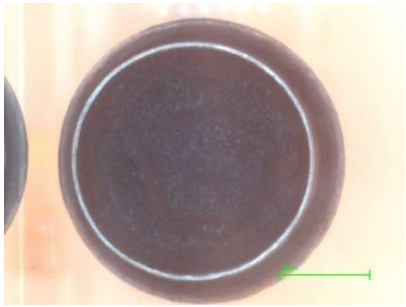
- Sample weight : 20 mg
- Heating rate : 10 ºC/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

3 Results :various Carbon





Carbon Nanotube
MW 3-20nm

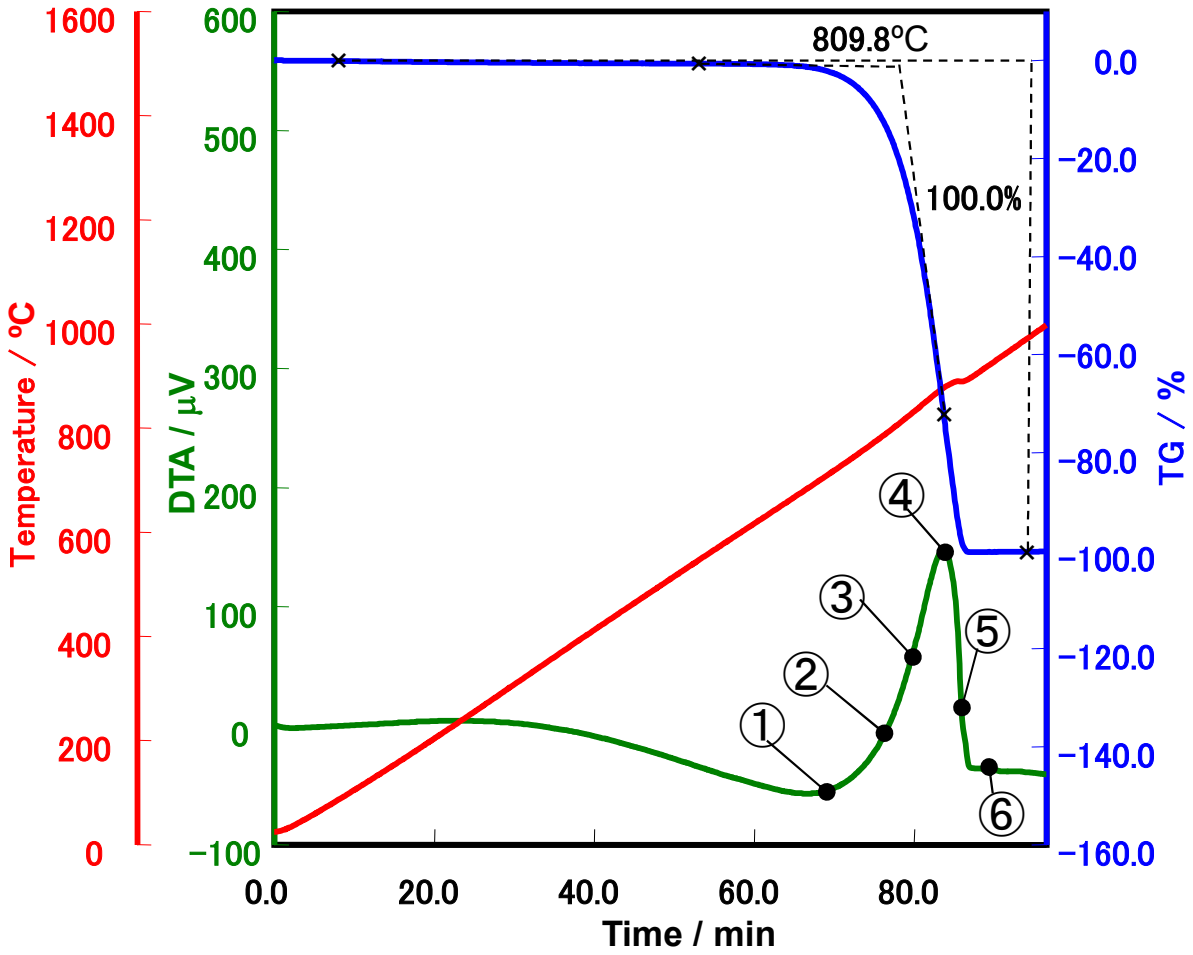


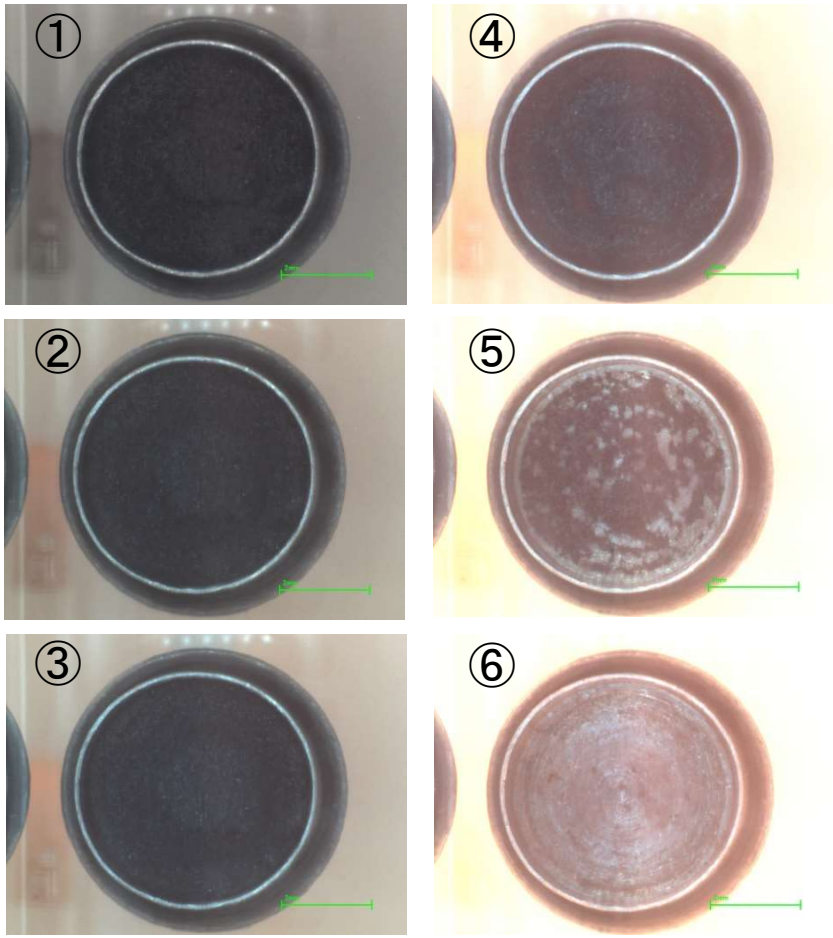
Graphite powder

Graphite rod
Graphite powder
Carbon Nanotube MW 3-20nm
Carbon Nanotube MW 40-60nm

Sample weight	: 5 mg
Heating rate	: 10 ºC/min
Sample pan	: Pt open pan
Gas flow	: Air 200mL/min

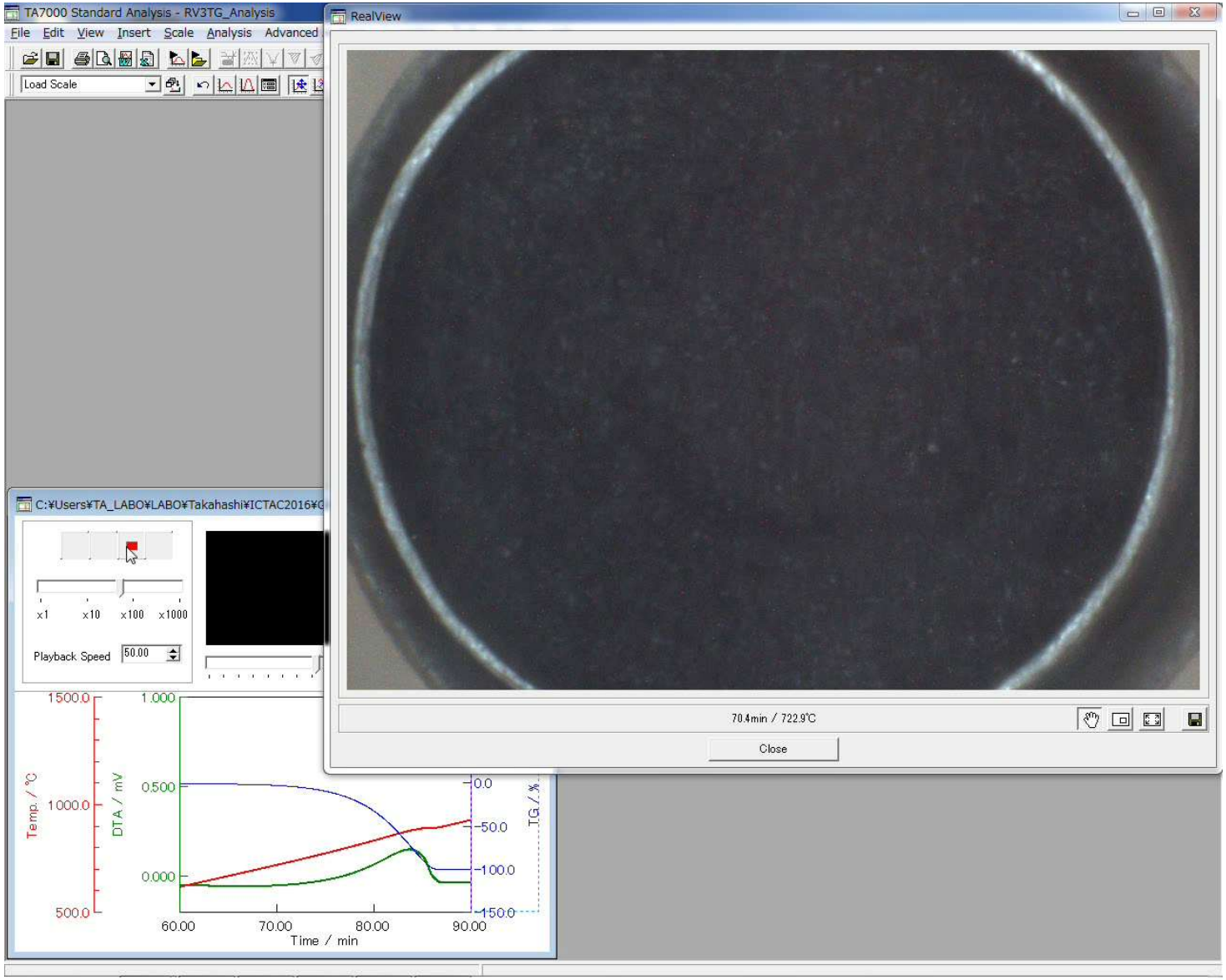
3 Results :Graphite powder





- Sample weight : 5 mg
- Heating rate : 10 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

3 Results :Graphite powder -movie



3 Results :Carbon Nanotube MW3-20nm

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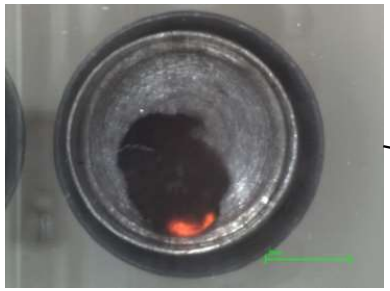
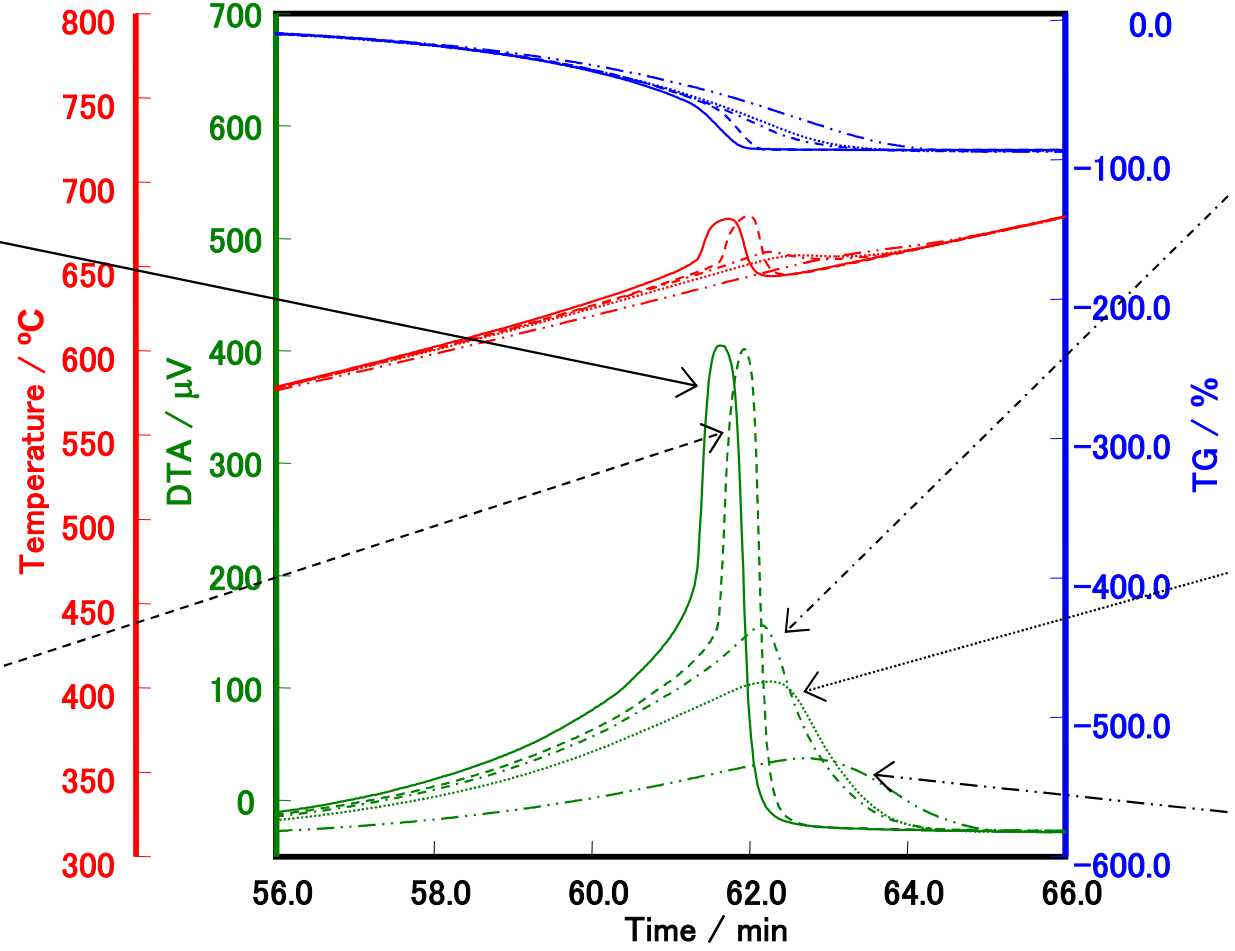
the relation between the ignition and the sample weight



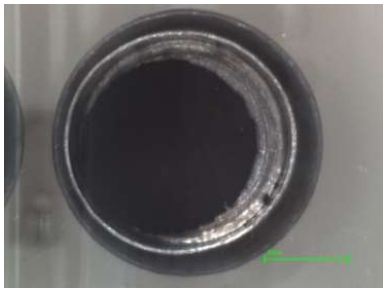
2.0mg

1.8mg



1.75mg

1.57mg



1.0mg

- Sample weight : 1.0 - 2.0 mg
- Heating rate : 10 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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3 Results :Carbon Nanotube MW3-20nm

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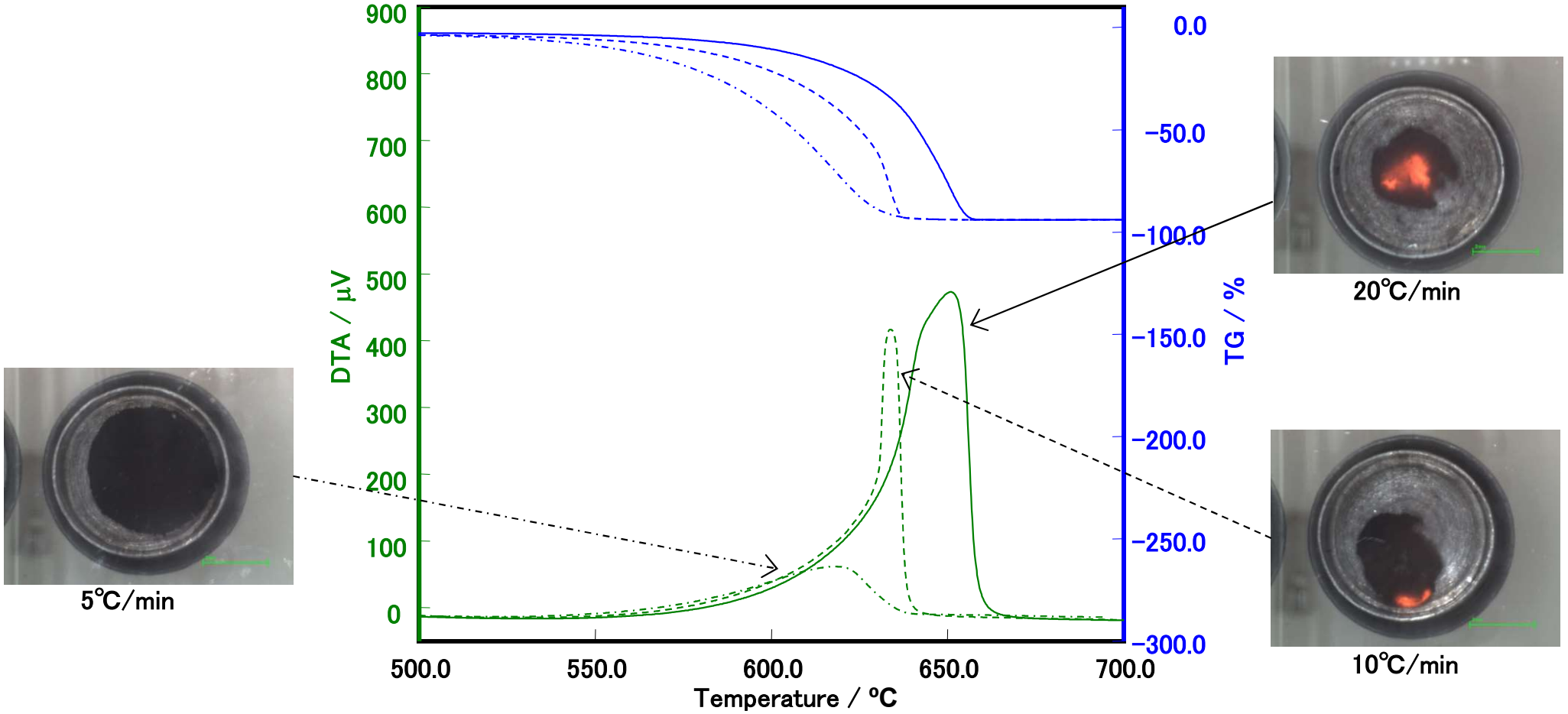
the relation between the ignition and the heating rate



- Sample weight : 2 mg
- Heating rate : 5, 10, 20 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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3 Results :Carbon Nanotube MW3-20nm -movie

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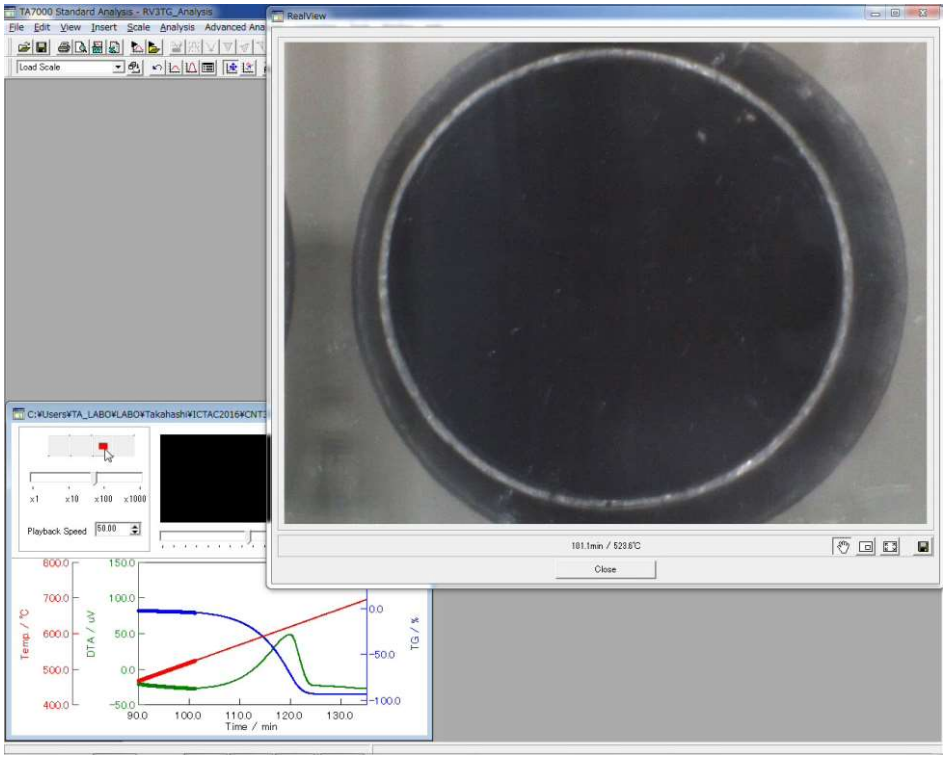
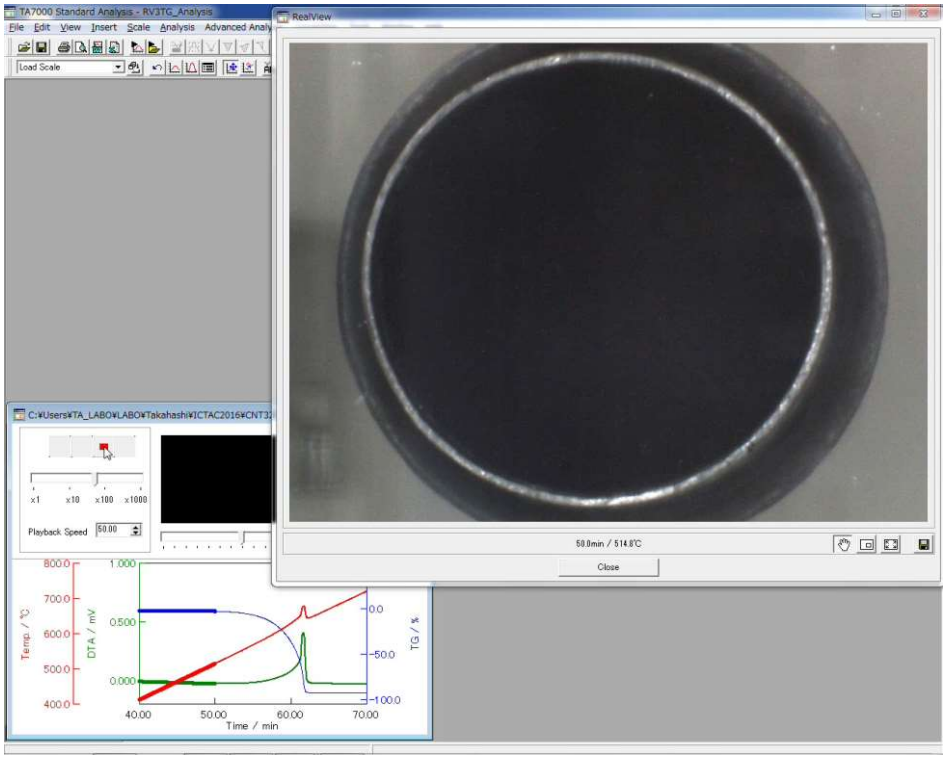
the relation between the ignition and the heating rate



5 ºC/min



10 ºC/min

Carbon Nanotube MW 3-20nm
- Sample weight : 2 mg
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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3 Results :Carbon Nanotube MW3-20nm

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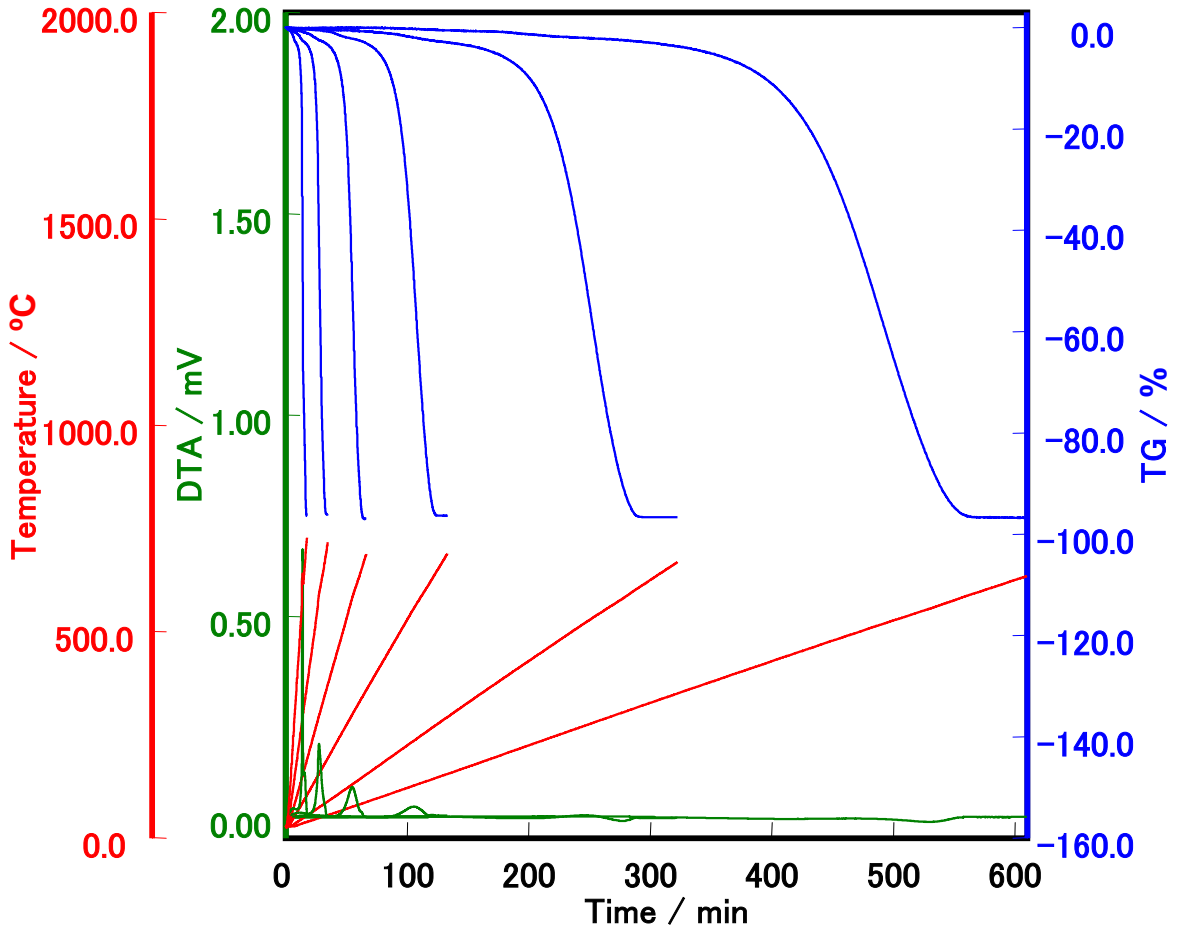
様々な昇温速度の測定結果(For Kinetic Analysis)



Carbon Nanotube MW 40-60nm

- Sample weight : 2 mg
- Heating rate : 1, 2, 5, 10, 20, 40 °C/min
- Sample pan : Pt open pan
- Gas flow : Air 200mL/min

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3 Results :Carbon Nanotube MW3-20nm

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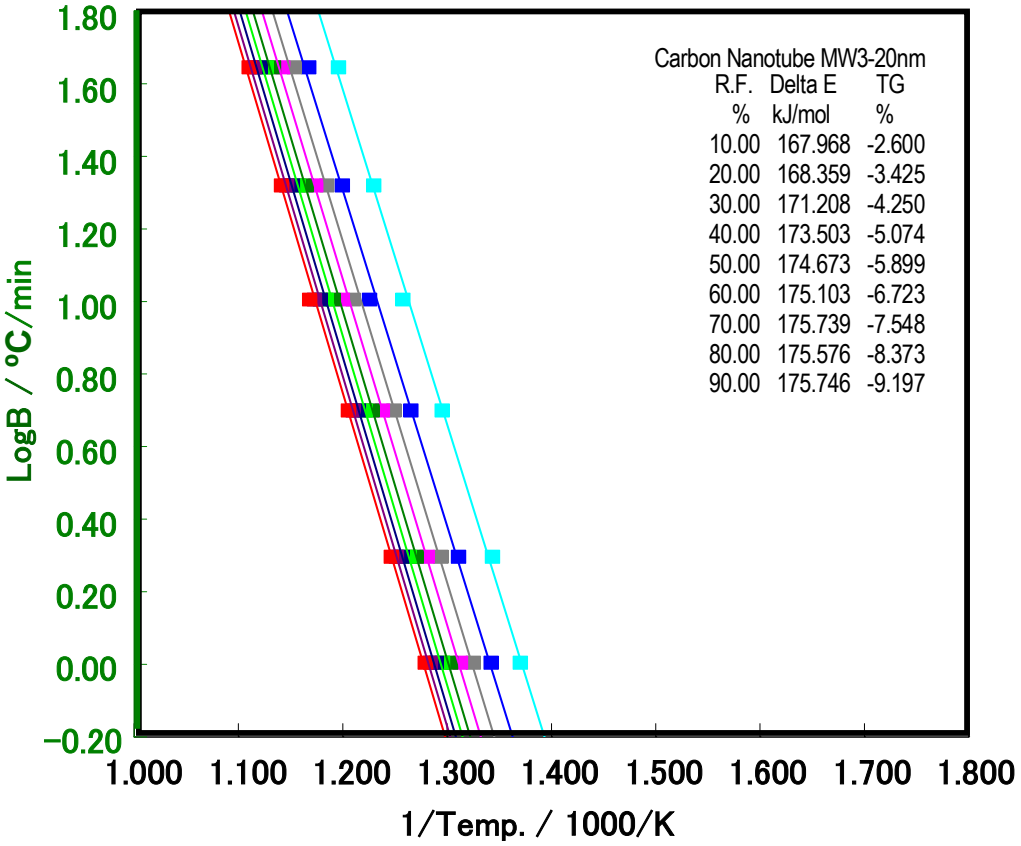
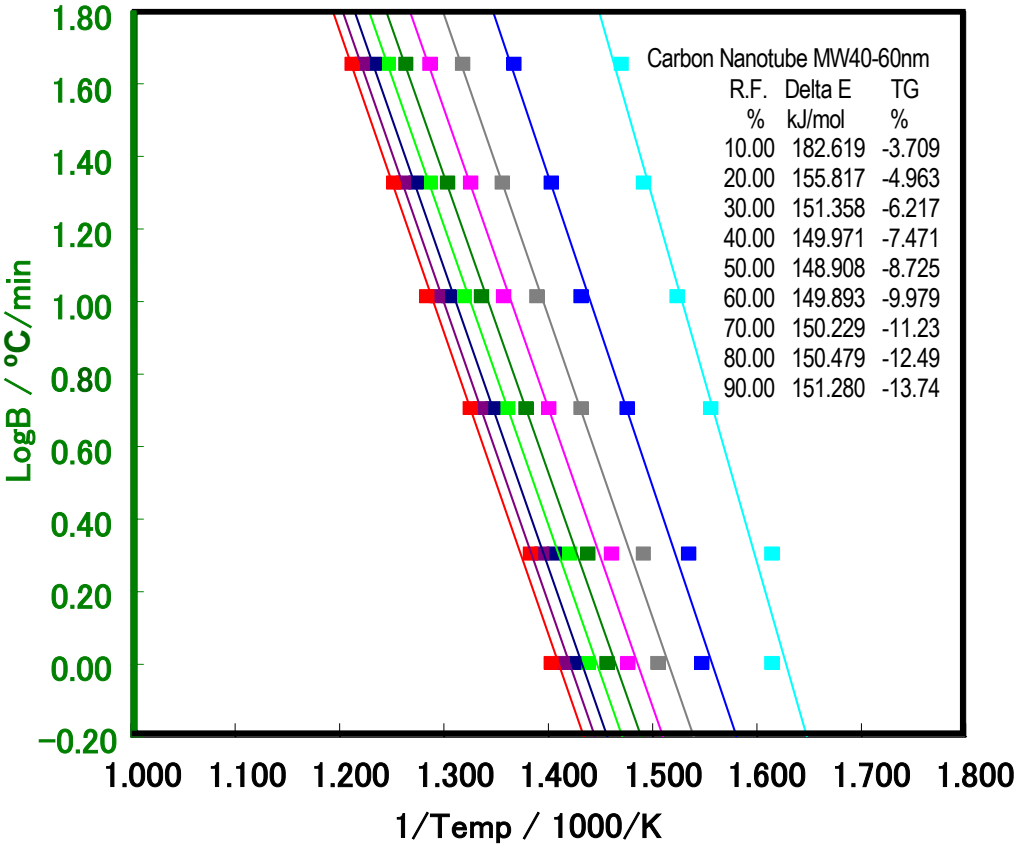
the activation energy by Kinetic Analysis



Result of Kinetic Analysis
Sample: Carbon Nanotube MW 3-20nm



Result of Kinetic Analysis
Sample: Carbon Nanotube MW 40-60nm

40-60nmは、3-20nmよりも活性化エネルギーが低い。40-60nmは低温から広い温度域で酸化分解を生じており、チューブの直径の分布が広域にわたっていることが予想される。

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4 Summery

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①We measured the wooden piece by STA with the optical observation unit.
When the wood was carbonized, we were able to observe the weight loss and the shrinkage of the material simultaneously. After that we observed the oxidation decomposition of the carbon. The decomposition showed the ignition especially at high heating rate.

② By the oxidation decomposition of carbon from epoxy resin, we could not observe the ignition.

The ignition of the carbon oxidation decomposition
③ is suggested that there was structure dependence of the carbon.

④ depends on the sample weight or the heating rate.

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END

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