Development of a TG/DTA system with optical observation function and its advantages

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In conventional TG/DTA instruments, samples cannot be viewed directly because they are obscured by the ceramic furnace, heating elements, insulation, etc.

So, TG/DTA users are left to determine which phenomena occur by reviewing the TG and DTA curves obtained during the analysis.
Introduction – A Proposed Solution

- Almost all of these events could be confirmed visually.

- A new TG/DTA design featuring a furnace structure that makes sample observation possible.

- The new design should provide the same quality data output as the original instrument, while also providing additional useful information about the material flow, shape change, and even unexpected events.

- This presentation highlights some examples of the optical observation TG/DTA system in action.
1. Is it possible to view the inside of the furnace during measurement while maintaining:
   - uniform heating
   - high sensitivity
   - controlled environment (gas composition / flow rate)

2. Can images be recorded in a really useful manner?
   To view a sample, equipment has to be installed near the furnace, which can be challenging for a camera.
Furnace cover contains a view port and quartz furnace tube, which are visually transparent.

Camera support arm contains a cooling fan and rugged support mechanism.

Cut-away image of the sample observation furnace. Thermal modeling was used to match heat uniformity to standard furnace.
Modeling of Oven Tube Temperature Uniformity

STA7200

STA7200RV

Quartz window

Patent pending solution

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Sample Observation: TG/DTA of Graphite

*Graphite powder
  sample weight: 5mg
  heating rate: 10°C/min
  purge gas: Air 200mL/min

At high temperature, we can see volume of graphite decrease.
Sample Observation: TG/DTA of Graphite

This movie shows that particles of the graphite powder move during decomposition; likely due to the expansion of the gas forming; COx.
Overlaying Snapshots Linked to Data

- Characterize broad transitions – zoom in on PET melt

Sample: PET  
Rate: 10°C/min

Temp. / °C

- Simply add images at critical events
Sample Observation: TG/DTA of Coating Material

The color changes to a reflective gray.

The sheen changes from green to black and it shrinks.

*Coating material
sample weight: 5mg heating rate: 10°C/min purge gas: Air 200mL/min

We can follow changes in shape, color, and dimension from the images alone.

Great candidate for evolved gas analysis.
Sample Observation: TG/DTA of Coating Material

Color changes:
green → black → green → yellow-white → (uniform) white

Size changes.
Change in luster or sheen (reflectivity)
Sample appears to be a stack of thin films.
Sample Observation: TG/DTA of Solder
An oxidation start time of sample A is earlier than sample B. Shape of sample B changed from a square to a circle and became small.
Oxidation Induction Time measurement of PE

- The decomposition behavior of sample A is faster.
- The shape of A was stable, while B collapsed remarkably.
- This is the influence of additives such as antioxidant and the stabilizer.

*Polyethylene
- Sample weight: 5mg
- Heating rate: 20°C/min
- Purge gas: N₂ => O₂ 200mL/min
Sample Observation on Various Instruments

- Real-Time observation during measurement
- Thumbnail view and Slide show functions can be used during analysis
- Easy measurement and analysis by TA7000 software
You can see the dimension change during glass transition
Measuring physical changes

Useful for determining strain relaxation in polymeric materials
DMA with Sample Observation

Ability to Determine Sample “Necking”
The TG/DTA with sample observation capability features:
- A quartz furnace tube and a view port
- Image recording is accomplished with a camera unit designed with heat resistance in mind.
- Software allowing overlay of each image with the corresponding data

Testing of various materials with the new TG/DTA design has demonstrated a number of benefits, including:
- Data interpretation is easier when more information is available
- More exact understanding of transitions can be gained
- Changes in physical state, such as color, that may not have an associated mass change
- Unexpected or one-of-a-kind events can be explained
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