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

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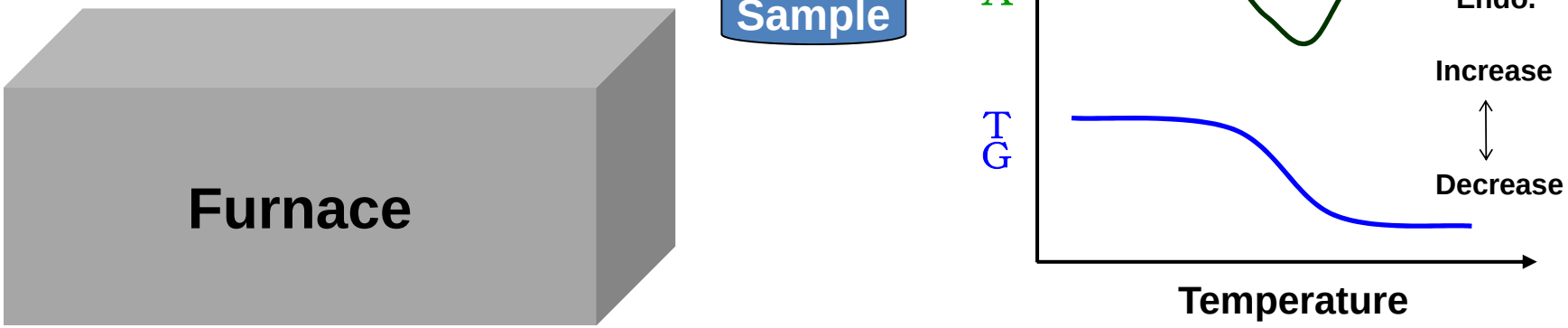
Hitachi High-Tech Science Corporation

Introduction - The Problem

- 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

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- 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

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Design Challenges

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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)

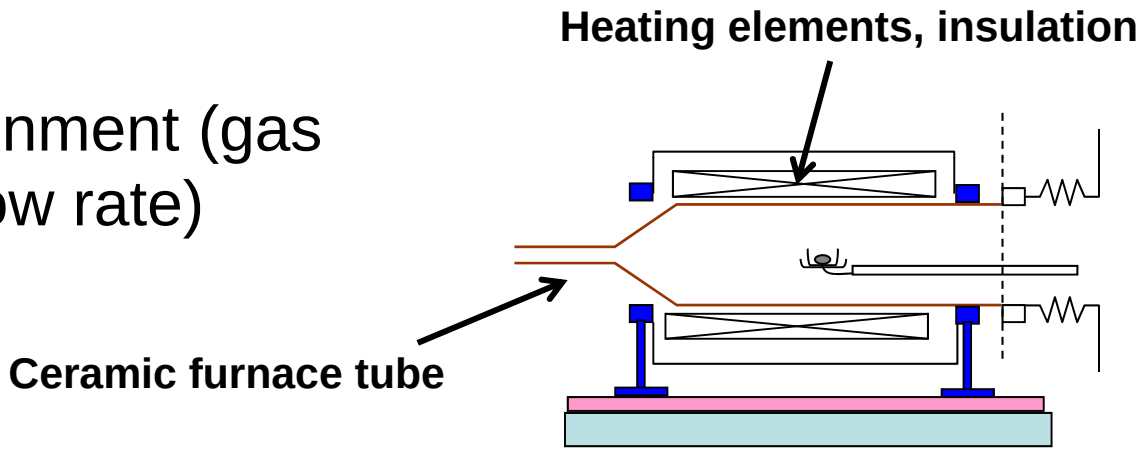

Fig.1 Structural features of a conventional horizontal TG/DTA

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.

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System Design Elements

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Top view of furnace

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

STA7200RV+RV-2TG

Cut-away image of the sample observation furnace.
Thermal modeling was used to match heat uniformity to standard furnace.

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Modeling of Oven Tube Temperature Uniformity

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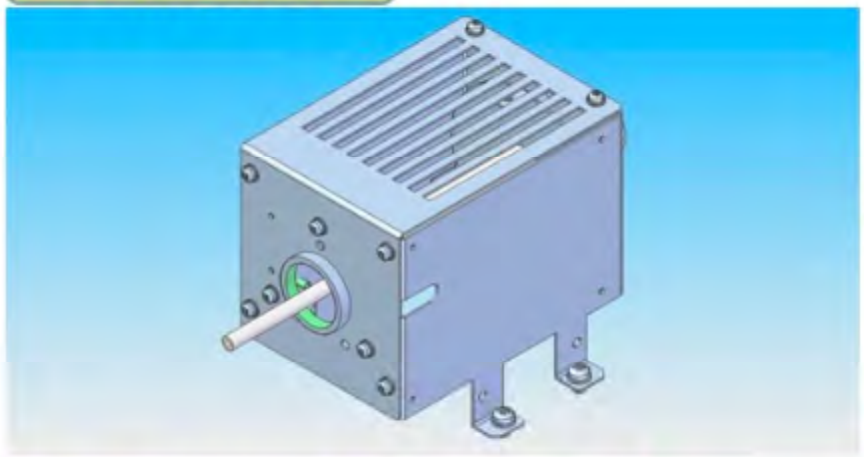
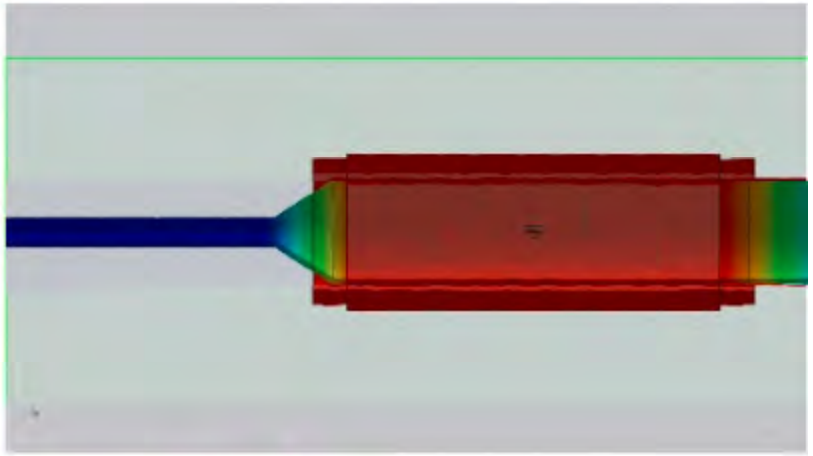
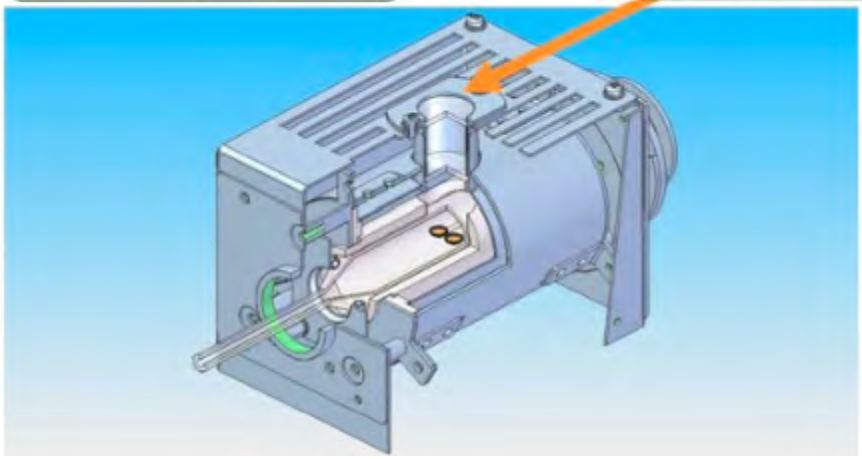
STA7200

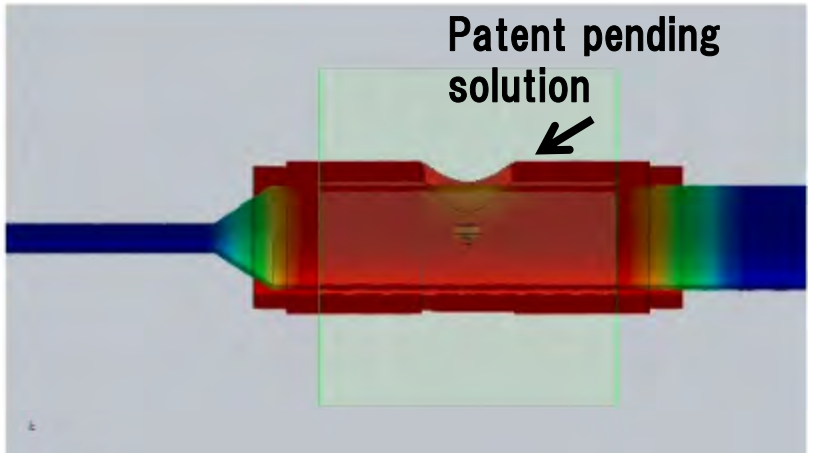




STA7200RV

Quartz window





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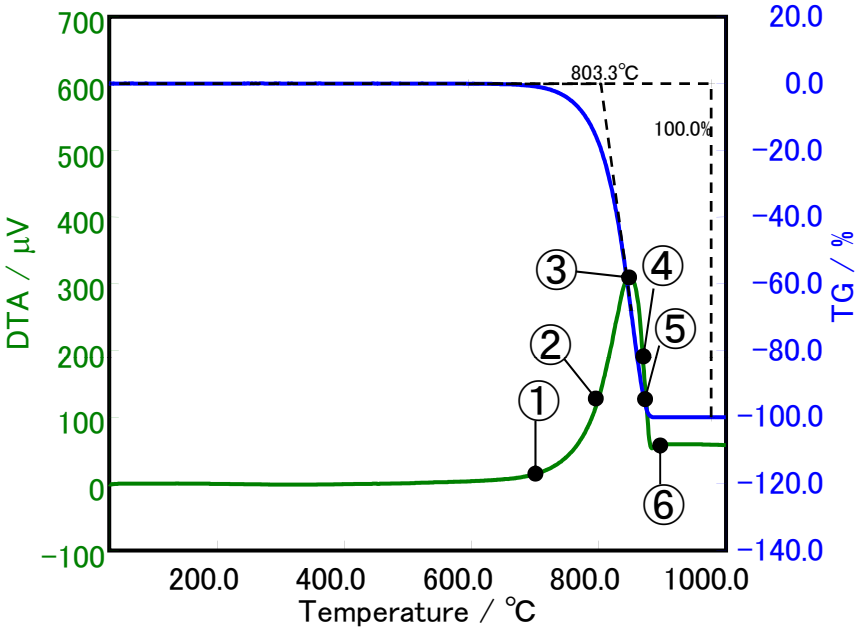
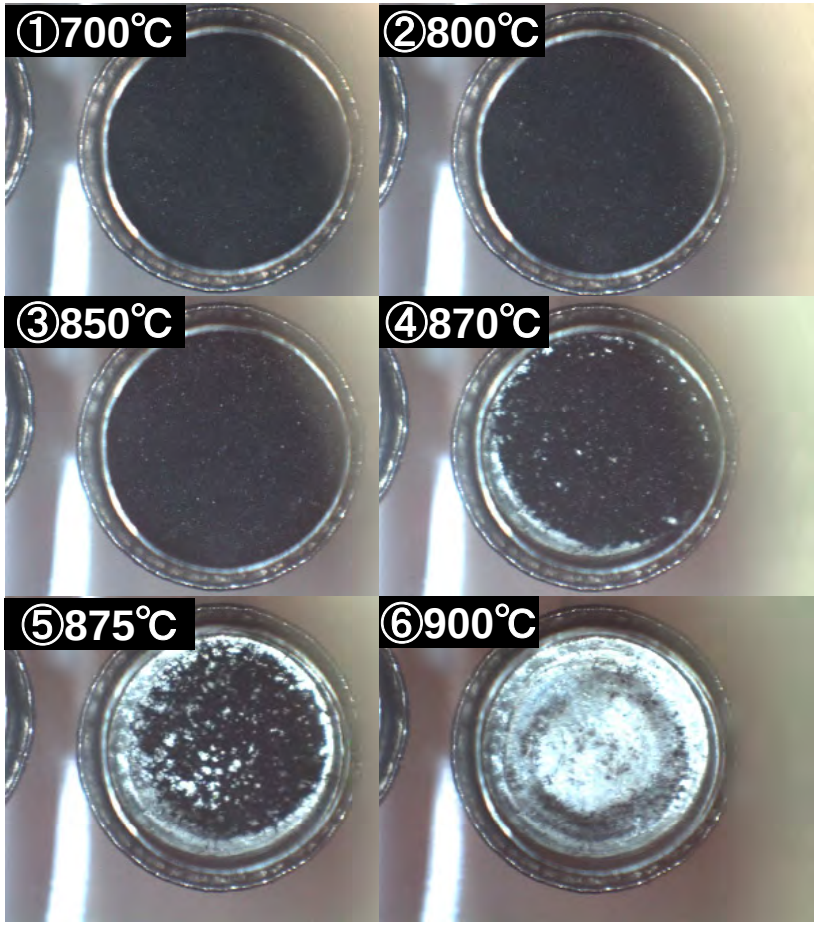
Sample Observation: TG/DTA of Graphite





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

At high temperature, we can see volume of graphite decrease.

Sample Observation: TG/DTA of Graphite

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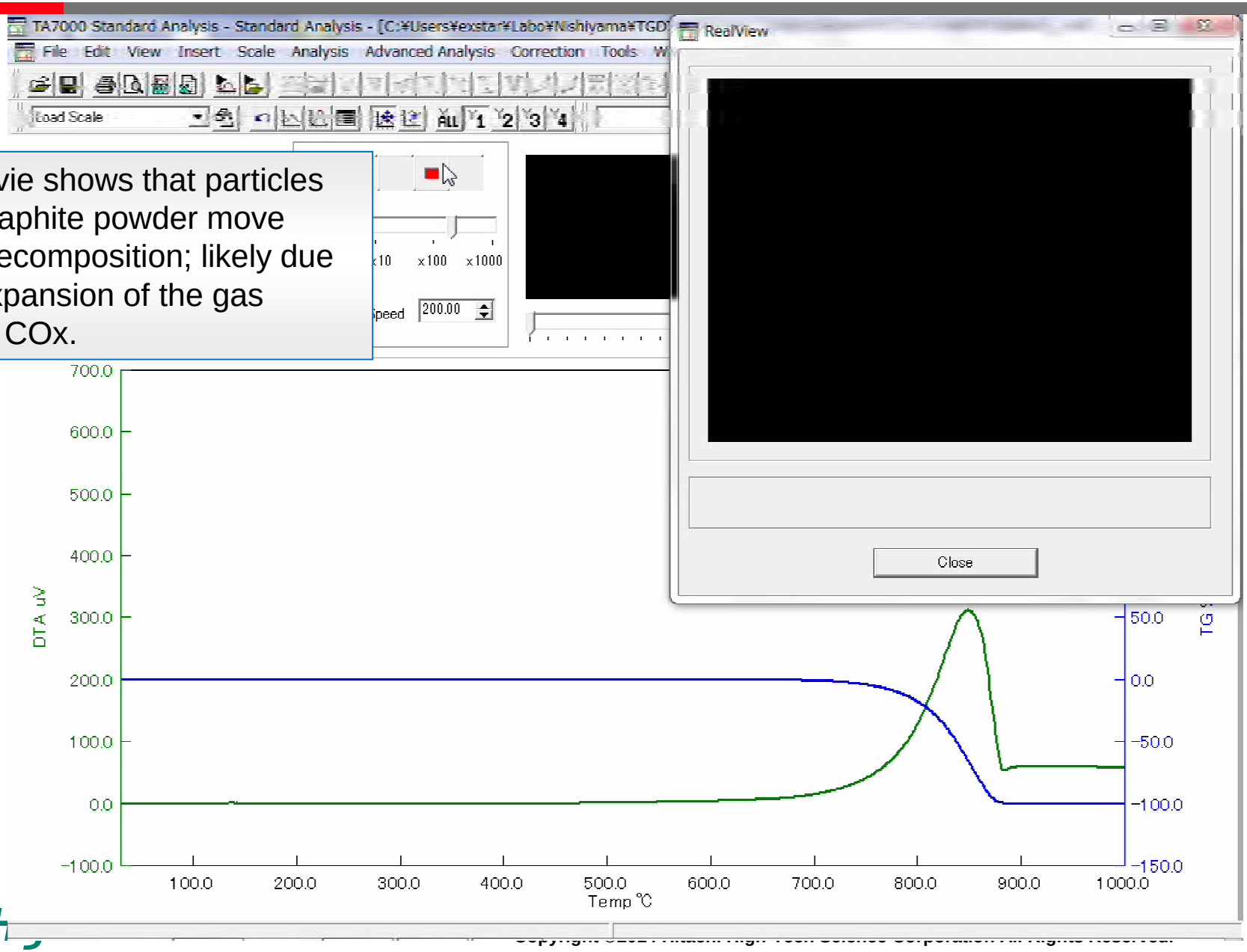
This movie shows that particles of the graphite powder move during decomposition; likely due to the expansion of the gas forming; COx.



Overlaying Snapshots Linked to Data

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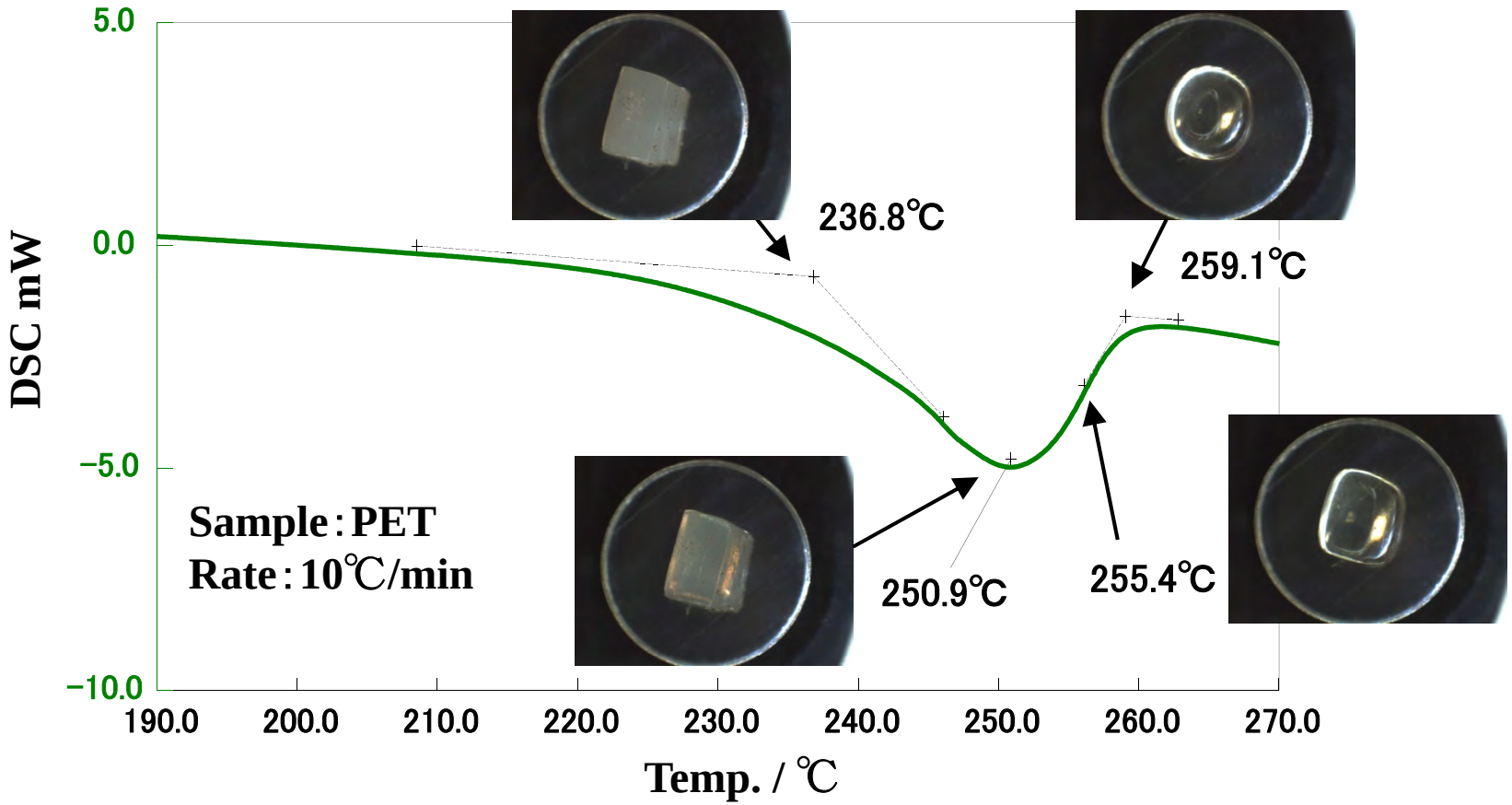
- **Characterize broad transitions – zoom in on PET melt**



- **Simply add images at critical events**

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Sample Observation: TG/DTA of Coating Material

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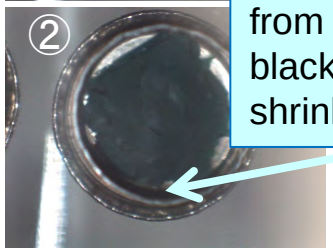
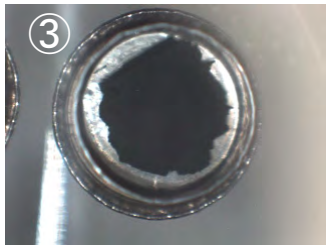
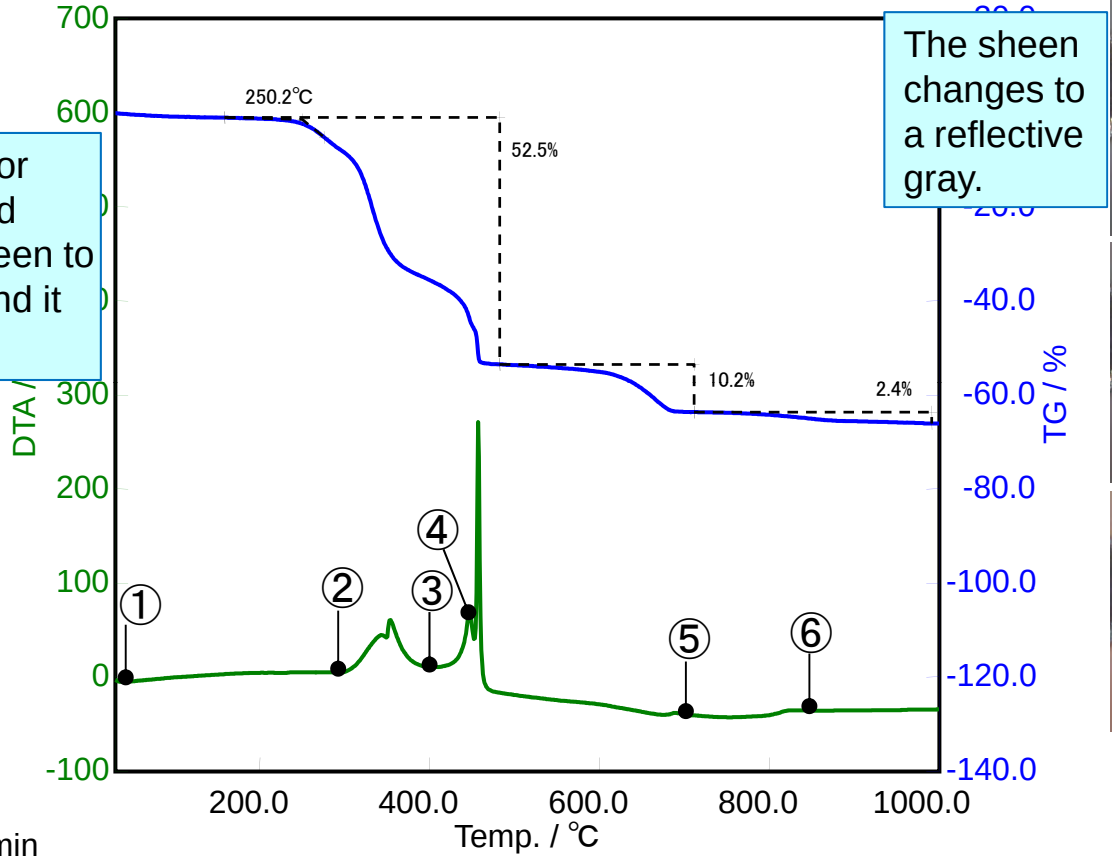
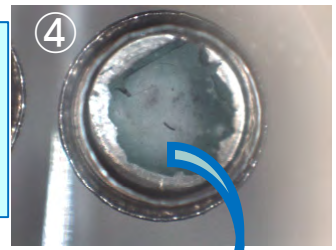


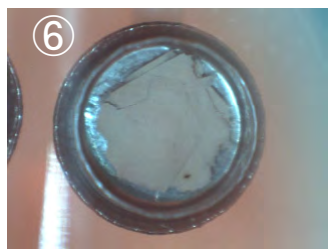


The color changed from green to black and it shrinks



The sheen changes to a reflective gray.





*Coating material
sample weight : 5mg
heating rate : 10℃/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.

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Sample Observation: TG/DTA of Coating Material

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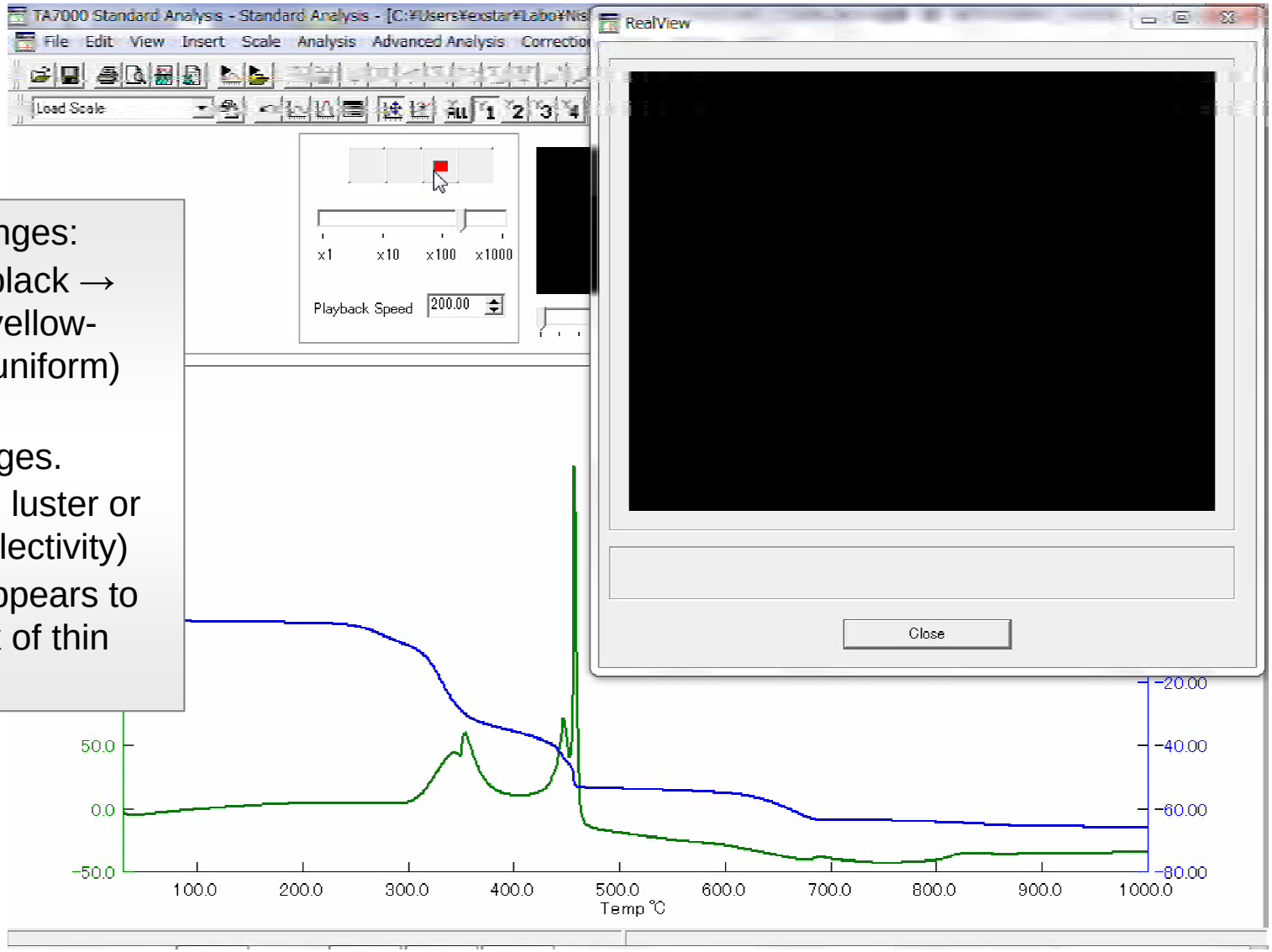
Color changes:
green → black → green → yellow-white → (uniform) white

Size changes.

Change in luster or sheen (reflectivity)

Sample appears to be a stack of thin films.



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Sample Observation: TG/DTA of Solder

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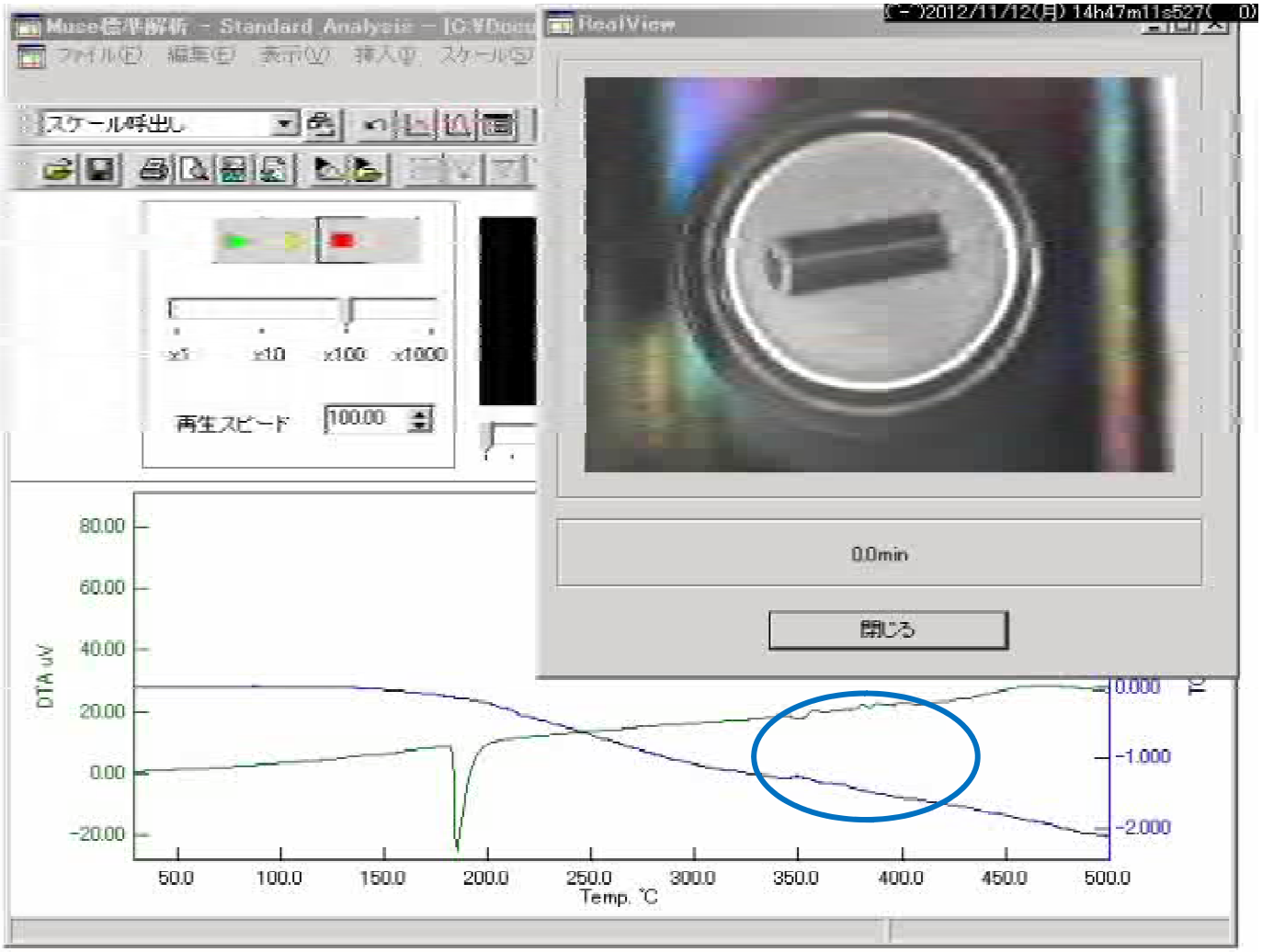

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Oxidation Induction Time Measurement of PE

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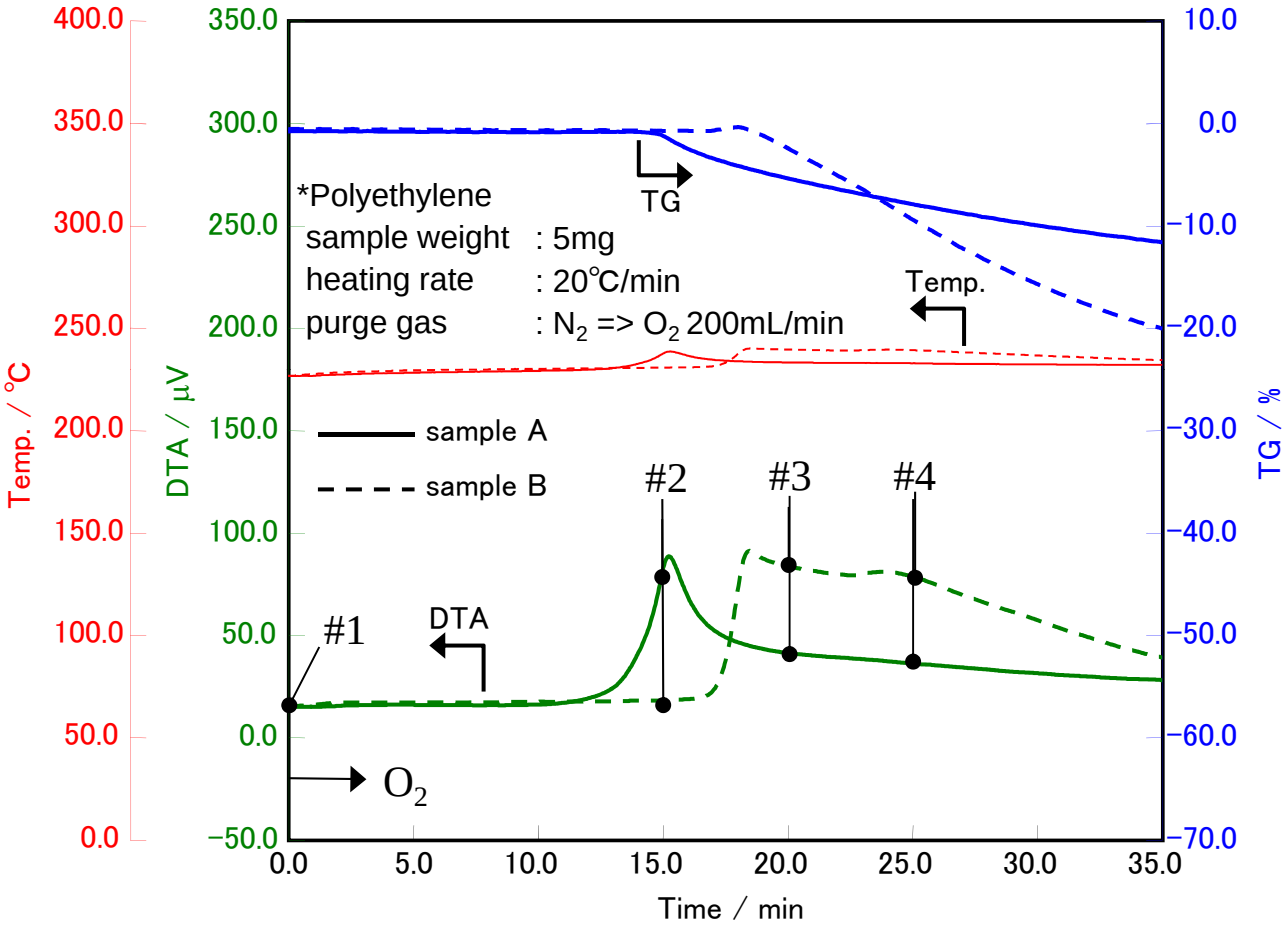

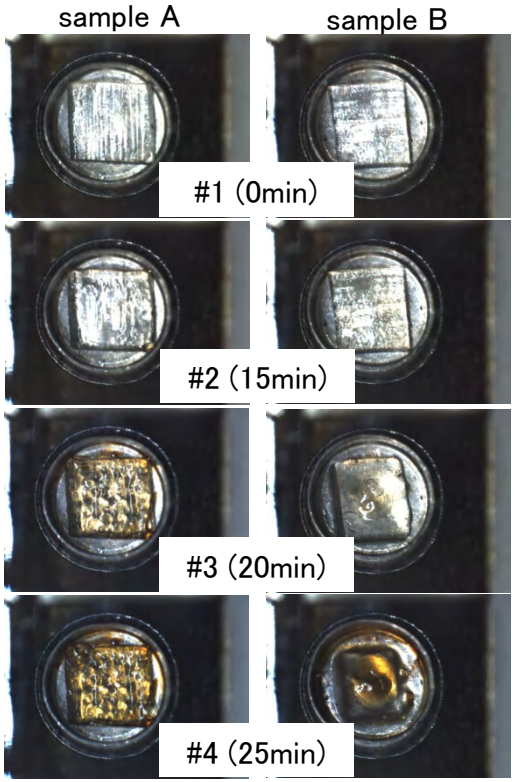

An oxidation start time of sample A is earlier than sample B. Shape of sample B changed from a square to a circle and became small.

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Oxidation Induction Time measurement of PE

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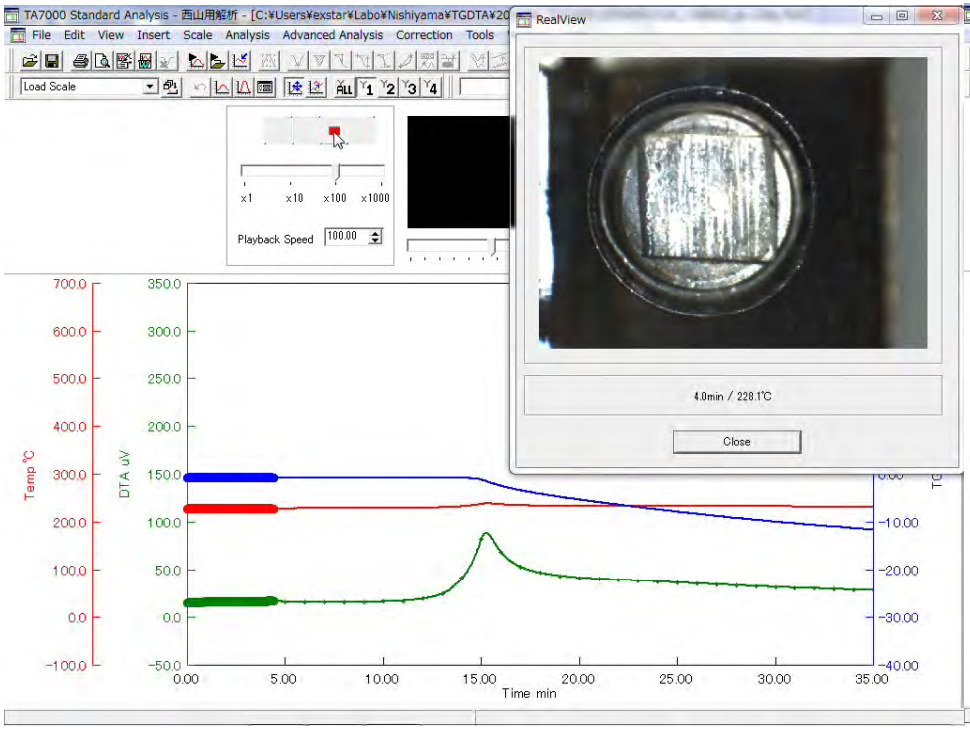
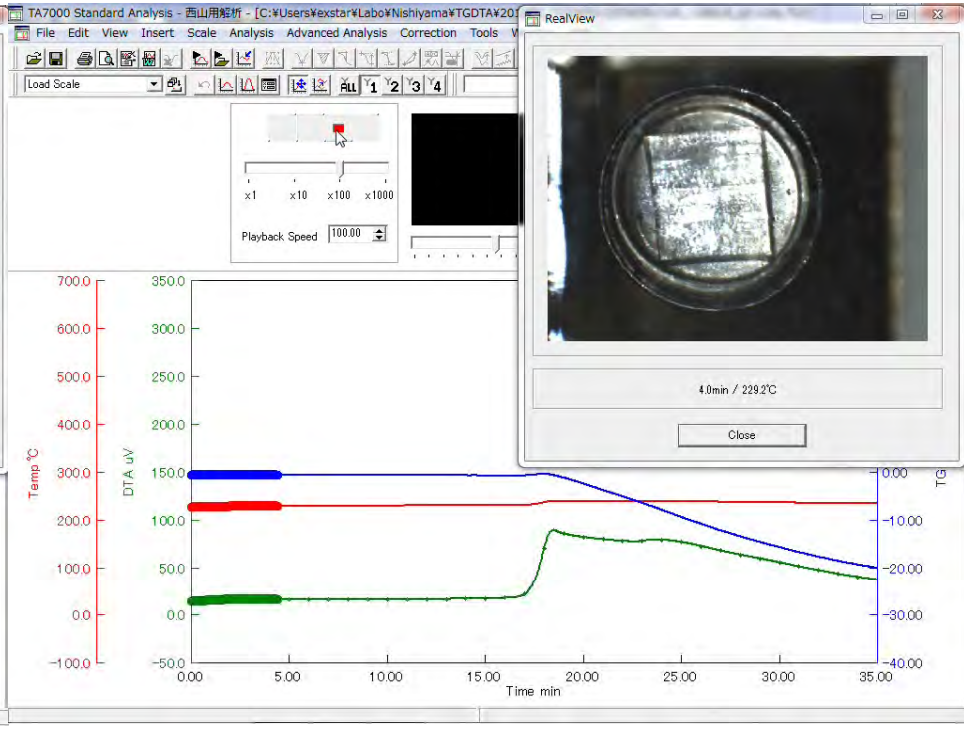
sample A



sample B



- **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℃/min
purge gas : N_2 => O_2 200mL/min

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Sample Observation on Various Instruments

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- Real-Time observation during measurement
- Thumbnail view and Slide show functions can be used during analysis
- Easy measurement and analysis by TA7000 software

DSC System

STA System

DMA System

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DSC of Optical Fiber Using Sample Observation

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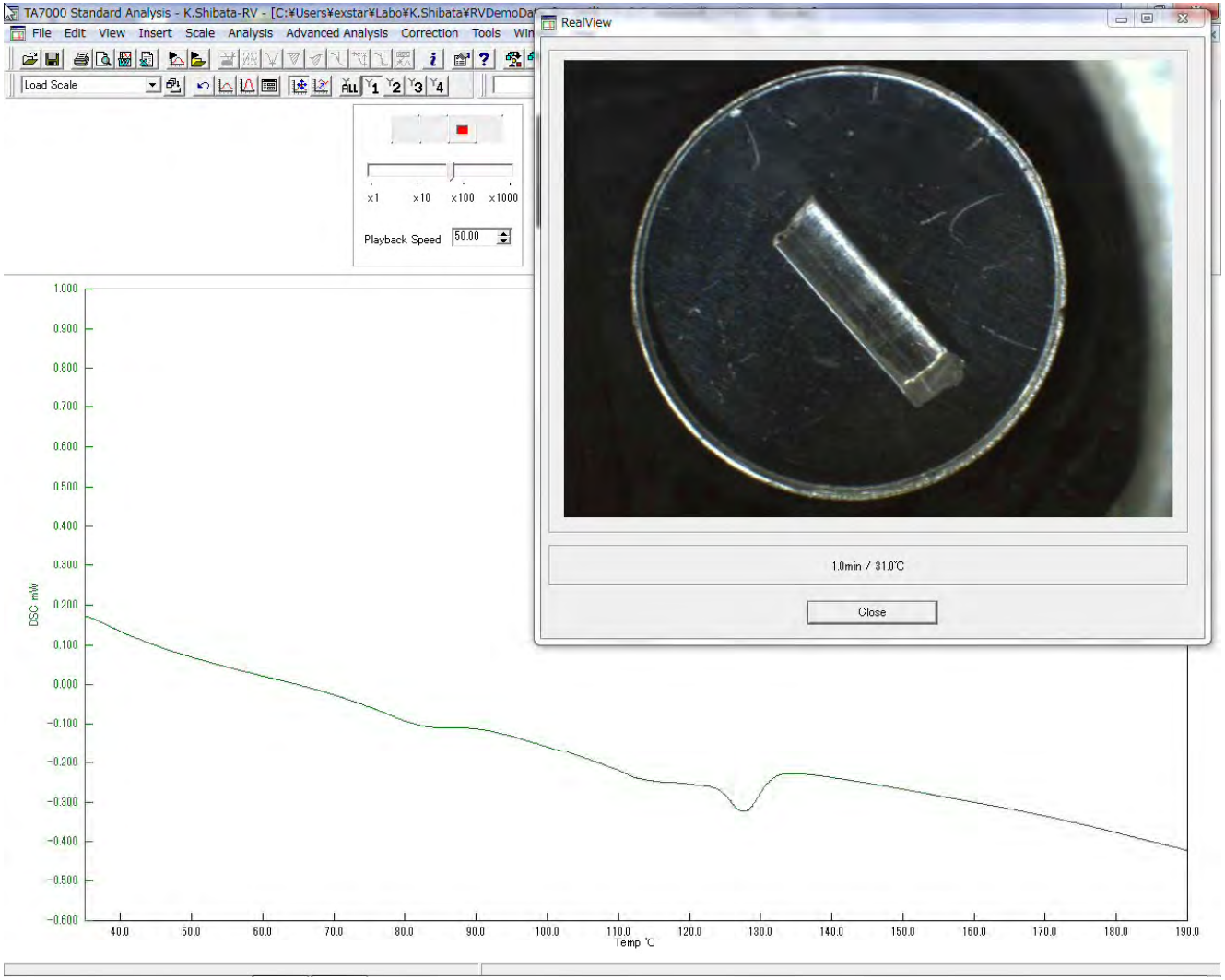

You can see the dimension change during glass transition

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Measuring physical changes

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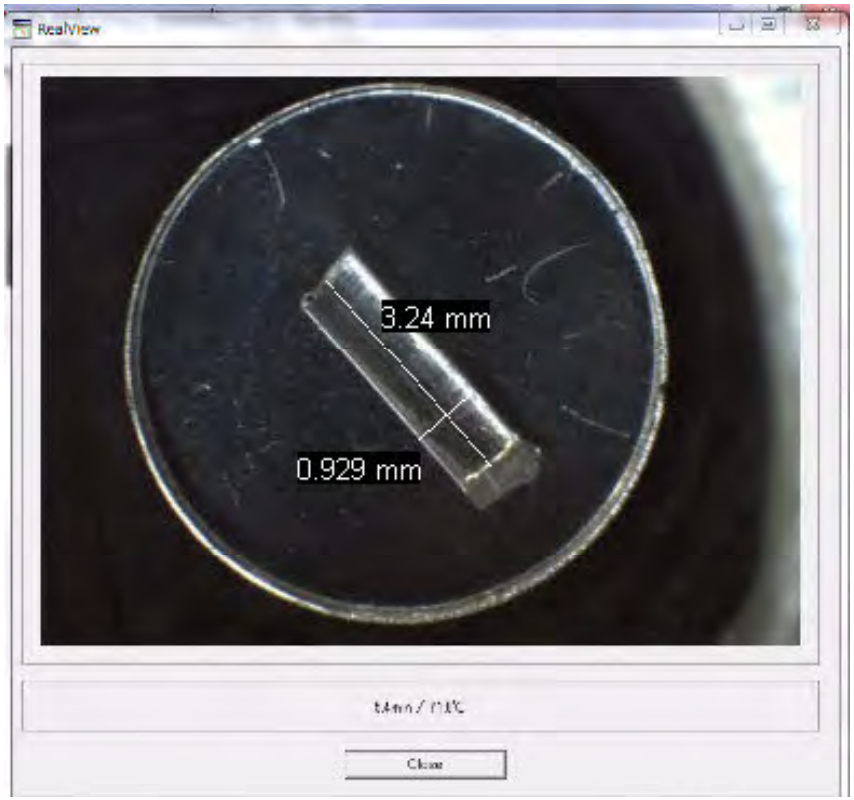
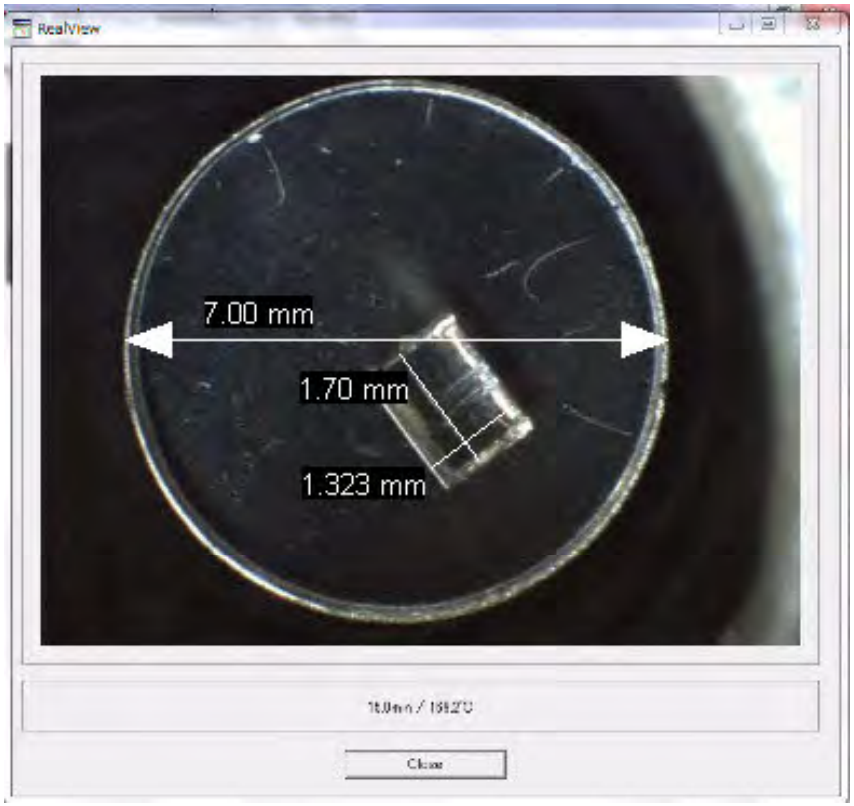
Useful for determining strain relaxation in polymeric materials





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DMA with Sample Observation

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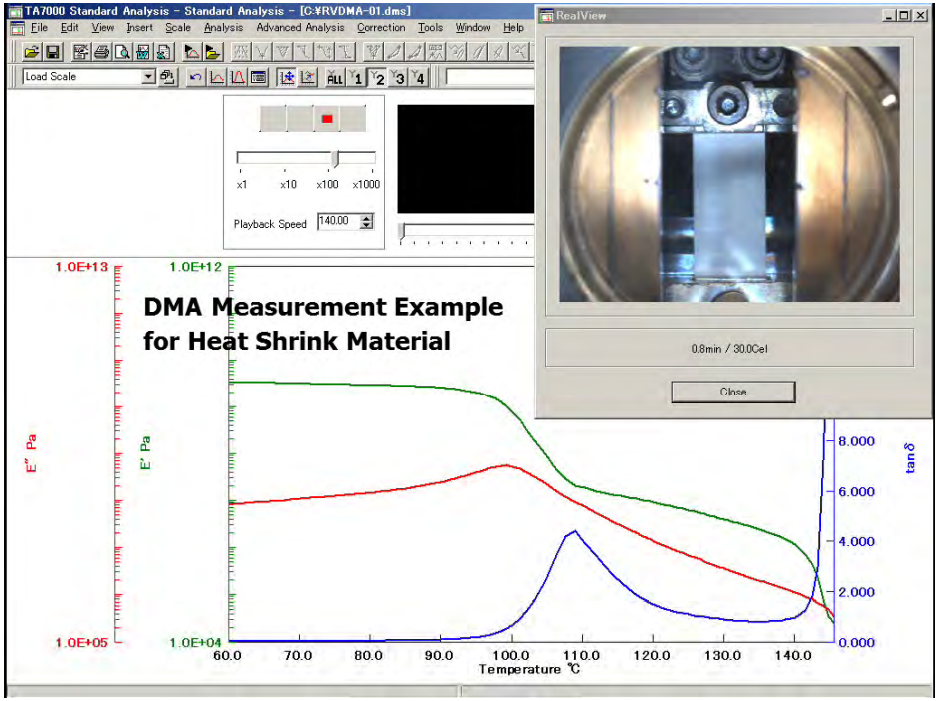
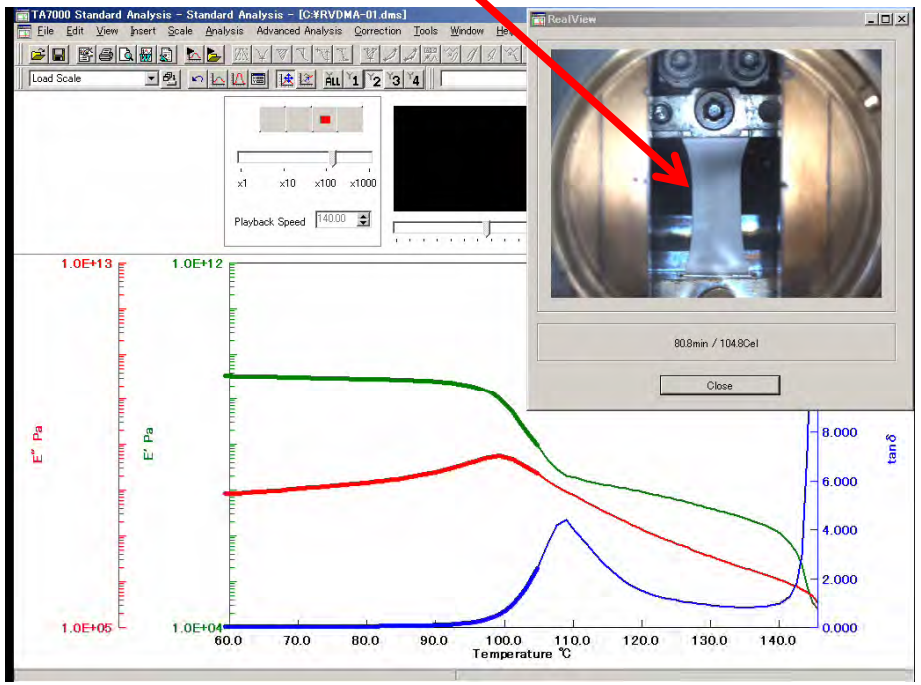
Ability to Determine Sample “Necking”





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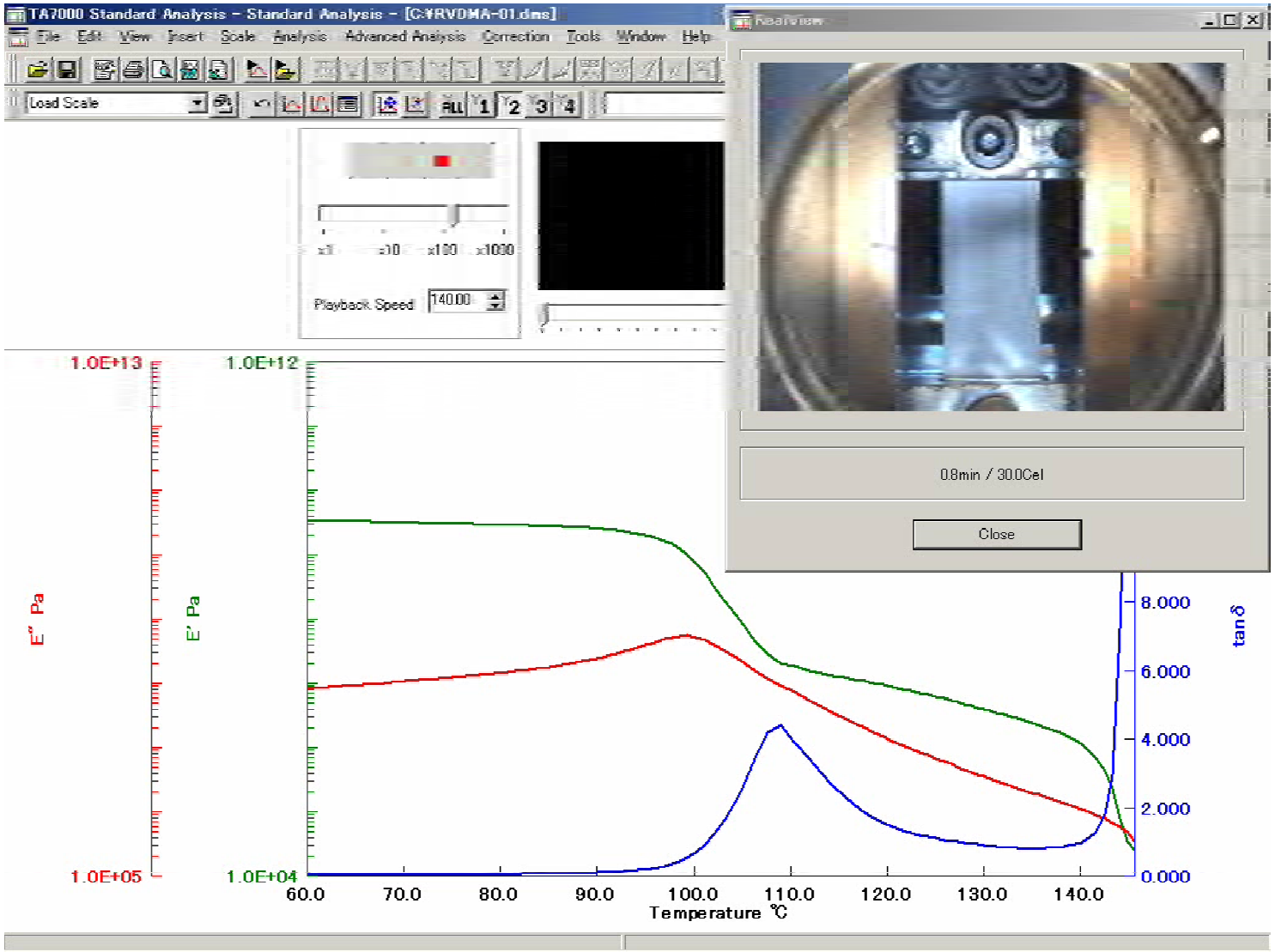
DMA with Sample Observation – Shrink Film



Summary

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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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END

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

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Creative Minds. Innovative Solutions.