TA INSTRUMENTS 2940 THERMOMECHANICAL ANALYZER (TMA)

Insert Nickname Here

Operating Instructions

Table of Contents

1

Introduction

The TA Instruments 2940 Thermomechanical Analyzer (TMA) is a powerful and versatile analyzer that provides mechanical property measurements on a broad variety of materials from -150 to 1,000°C. The TMA is a useful tool for applications involving dimension change as a function of temperature, time, or force. TMA is usually used to precisely measure thermal expansion coefficients, but it can also be used to determine creep and stress relaxation behavior, heat deflection, melting temperatures, and glass transition temperatures. The TMA instrument works in conjunction with a controller and associated software to allow for easy data acquisition and analysis.

Safety

The following labels are displayed on the TMA instrument for your protection:

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch these areas or allow any material that may melt or burn to come in contact with these hot surfaces
	This symbol on the rear access panel indicates that you must unplug the instrument *before* doing any maintenance or repair work; voltages exceeding 120/240 Vac are present in this system. If you are not trained in electrical procedures, do not remove the cabinet covers unless specifically instructed to do so in the manual. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

Hot Stuff. The TMA is capable of heating samples to 1000°C. During testing, the furnace assembly becomes noticeably hot. After testing, the furnace assembly, sample, probe, and stage may remain hot for some time. **Do not touch the furnace assembly** or sample stage area during heating or while the sample is cooling from an elevated temperature.

Cold Stuff. The TMA is capable of cooling samples to -196°C through the use of liquid nitrogen. Because of its low temperature (-196°C), liquid nitrogen will cause tissue damage upon contact with your skin. When you work with liquid nitrogen, use the following precautions:

- Liquid nitrogen boils rapidly when exposed to room temperature. Only use liquid nitrogen in a well ventilated area to prevent displacement of oxygen in the air.
- Wear goggles or a face shield, cryogenic gloves, long pants, and closed-toed shoes.
- Transfer the liquid slowly to prevent thermal shock to the equipment. Use containers that have satisfactory low-temperature properties. Ensure that closed containers have vents to relieve pressure.

Sample Decomposition. The TMA is capable of heating samples to 1000°C. Many materials may decompose during the heating, which can generate hazardous byproducts. WARNING: If you are using samples that may emit harmful gases, vent the gases in an appropriate manner. In general, samples should not be heated above their decomposition temperatures to prevent the release of hazardous materials or contamination of the TMA.

Sample Melting. Since the TMA is capable of heating samples to 1000ºC, it is very possible that your sample will melt. WARNING: A melted sample can easily stick to the stage, probe, or furnace upon solidification. This will likely break or damage the instrument. Thus **DO NOT HEAT AN UNENCAPSULATED SAMPLE TO ANY HIGHER THAN 75% OF ITS MELTING TEMPERATURE**. For polymers, it can't hurt to encapsulate your sample even if you are reaching temperatures at or above 50% of the sample melting point.

Samples

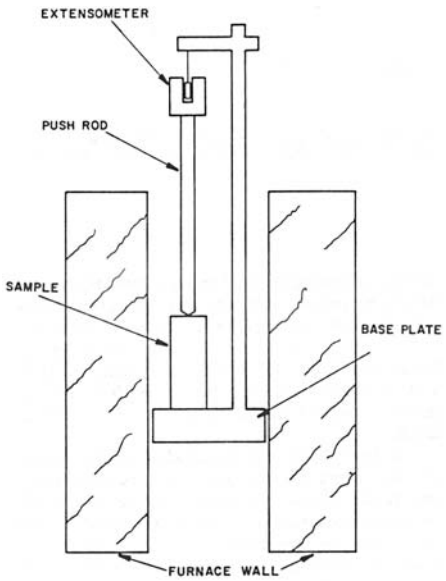
TMA samples should be smaller than a cylinder 10 mm in diameter and 25 mm tall.

- Samples for thermal expansion and glass transition measurements should have parallel and flat top and bottom surfaces.
- Samples for melting temperature measurements may be any shape. **NOTE:** To avoid making a mess with molten thermoplastics, you should encapsulate thermoplastic polymer samples in aluminum foil or a DSC aluminum pan (ask your instructor or TA, or see the Sample Preparation section of the DSC operating instructions, for information on specimen encapsulation). To avoid reactions between molten metals and the quartz probe and stage, you should sandwich your metallic sample between two plates of aluminum oxide.

2 Background Information

Thermomechanical analyzers (TMAs) are used to measure a variety of material properties, including coefficients of thermal expansion (CTE), melting temperatures, glass transition temperatures, heat deflection, and elevated temperature creep or stress relaxation behavior. One convenient aspect of the thermomechanical analyzer is that the same test can be used to find all of these material properties!

In the typical operation of a TMA, a small sample with parallel and flat top and bottom surfaces is placed on a quartz stage near a thermocouple. A quartz probe is lowered against the specimen with a constant applied force, and a heating/cooling unit is placed around the sample stage assembly. As the sample is heated or cooled, changes in dimension are measured by monitoring the motion of the quartz probe. The schematic figure below shows the typical setup for thermal expansion measurements.



While the specimen size does not need to fit any pre-set specification, there are some guidelines for its size and shape. Specimens should also be no larger than 10 mm diameter (or 8 mm square cross section) and 15 mm long, so that they will fit safely within the furnace and avoid overloading of the extension sensor. If you plan to analyze the specimens for thermal expansion or glass transition, they should have parallel top and bottom surfaces so that that TMA can better gauge changes in size.

The **coefficient of thermal expansion (CTE)** is a normalized number that describes the mechanical expansion or contraction of a material at different temperatures. It is an important property of a material, and failure to take into account the effect temperature has on the physical

size of materials has been known to cause many famous disasters, including the Challenger explosion. The mean coefficient of thermal expansion (CTE) is calculated as

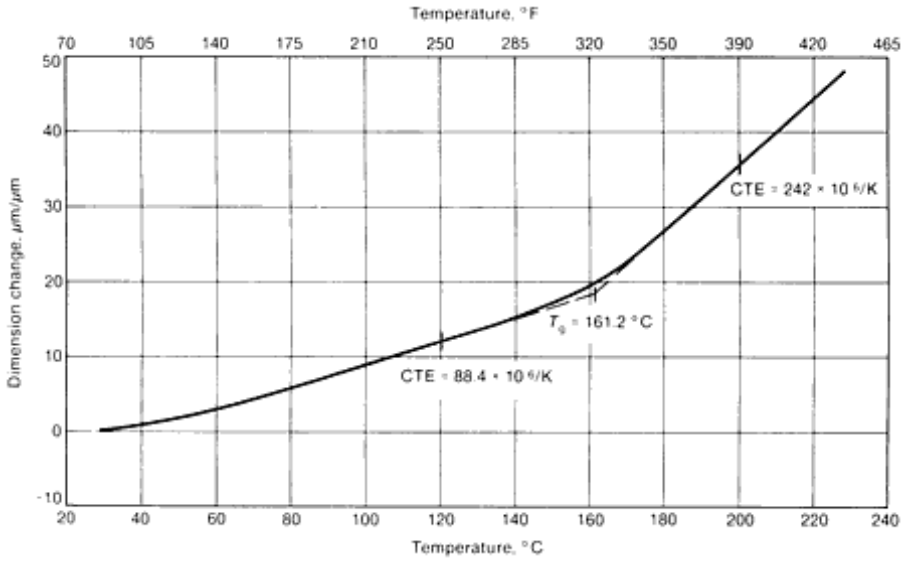
$$\alpha = \frac{\Delta L_s}{L_o \Delta T}$$

where α is the mean coefficient of thermal expansion in $^oC^{-1}$, ΔL_s is the expansion of the specimen in mm, L_o is the initial specimen length in mm, and ΔT is the temperature change in ^oC through the test. The CTE of a material is temperature dependent, and α is a reported mean for a particular temperature range. *More information on the CTE can be found in chapter 21 of your textbook.*

The **glass transition temperature (T_g)** is also an important characteristic of noncrystalline and semicrystalline materials, but T_g is a particularly significant property of many common polymers. At a temperature below T_g, amorphous and semicrystalline polymers tend to be hard and brittle because the polymer chains are locked in a tangled, coiled position. Above T_g, the polymeric chains are able to more easily rotate and slip past each other, and the polymer becomes softer and more ductile. Generally the glass transition point depends on the processing of the material, as well as that material's natural characteristics such as structure, bonding, and molecular weight. The T_g is accompanied by a change in the polymer's response to increasing temperature and is identified on a TMA plot as a change in the slope of the expansion-temperature curve. See the figure below for an example of the glass transition point.

While the TMA is not specifically designed to evaluate a material's **melting temperature**, it may be used to perform this task. In thermomechanical analysis, a material's melting temperature is identified as the point at which the sample stops expanding and starts flowing under the force applied by the TMA probe. Thus, the point at which an increase in temperature creates a decrease in size is that material's melting temperature.

Analysis of TMA curves is fairly straightforward, and all analyses may be performed within the TA Instruments Universal Analysis software. The figure below is a TMA curve for a fiberglass-polyester composite prepreg material. The curve shows glass transition temperature (T_g) and the difference between the CTE below and above the glass transition temperature of the polyester component of the composite.



References

- Callister: Sections 19.3, 15.11-15.14, 2.5-2.7
- Askeland: Chapter 21
- ASM Engineered Materials Handbook Desk Edition (Online), Thermal Analysis and Properties of Polymers

3

Instrument Operation

This section will walk you through the basic operation of the thermomechanical analyzer. You'll have to follow a series of tasks that includes

- Powering up the instrument,
- Loading your sample,
- Setting your testing conditions,
- Running a scan and collecting data, and
- Analyzing your results.

Startup

1. TMA: Turn on the main power switch followed by the heater power switch. Wait for the instrument to boot.
2. Computer: Turn on the computer. Log in using the following username and password:
   - User name:
   - Password:
3. Open the Thermal Advantage software by double-clicking on the **TMA-TA Instrument Control** icon on the desktop.

Sample Loading

The following figure highlights the sample stage area (in yellow), which is hidden behind the furnace assembly during operation of the TMA.

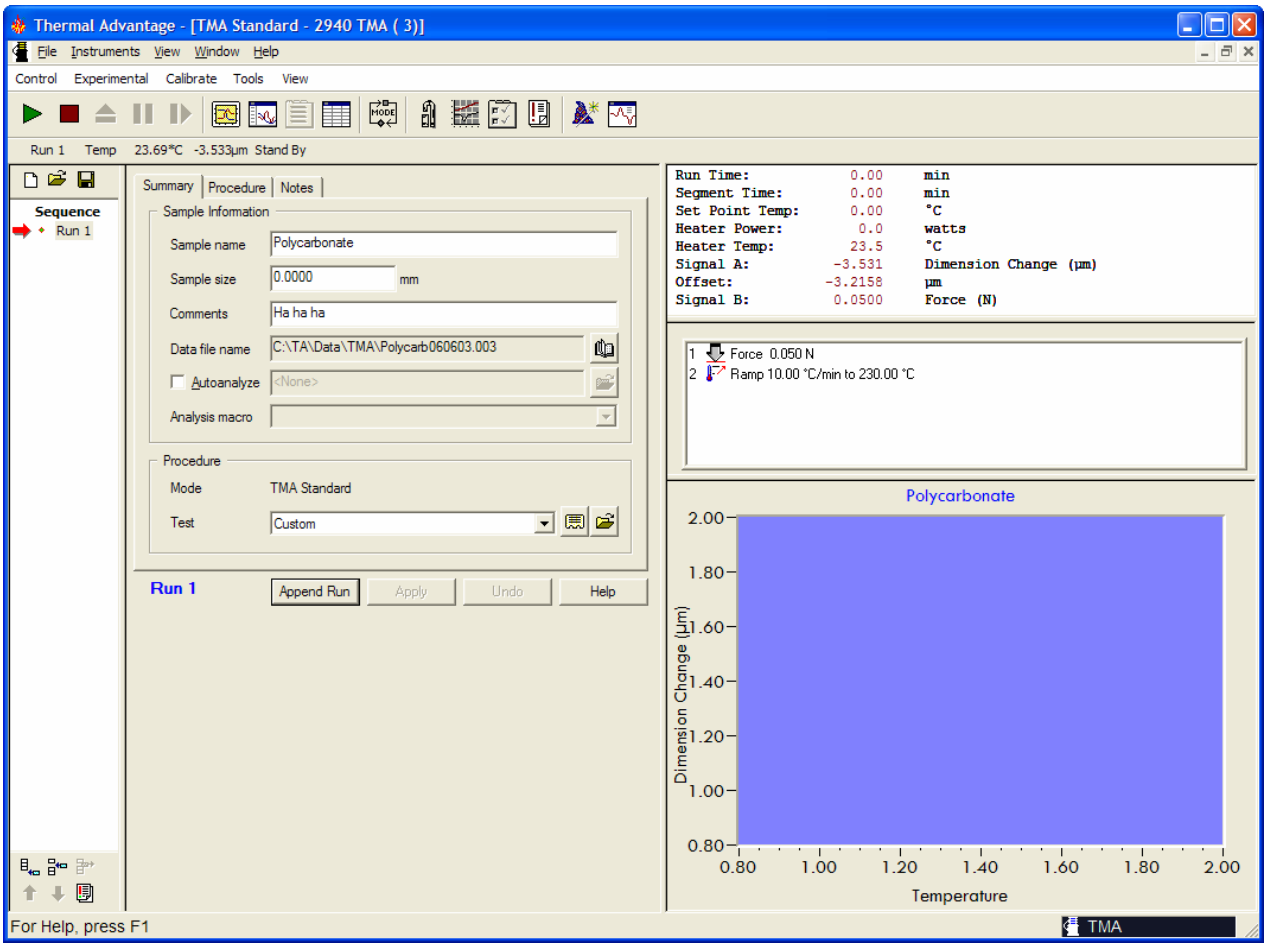
Load your sample, as follows:

1. Raise the furnace by pressing the **FURNACE** button on the instrument control panel.
2. Gently rotate the furnace to the left to move it out of the way of the sample stage. With the furnace in the raised and rotated left position, you should notice a cylindrical quartz stage (looks like a closed tube), a quartz probe (looks like a bent rod), and a thermocouple (metal wires within a white ceramic sheath).
3. Raise the probe with the **Probe up** button on the instrument control panel, then lower the probe with the **Probe down** button on the control panel. The probe should now be resting on the sample stage.
4. Zero the probe position by pressing the **Zero Length** button on the control panel. Wait…it takes a little while for the probe to zero.
5. You now need to raise the probe and position your sample on the stage. If you press the **Probe up** button one time, the probe will go up a little bit. If you need more room for your sample, press the **Probe up** button again, and the probe will rise to its maximum height.
6. Carefully position your sample on the stage.
7. Lower the probe by pressing the **Probe down** button on the control panel. You may need to press the button twice to lower the probe to your sample.
8. ***Gently*** rotate the furnace back to the center position (above the sample stage).
9. Lower the furnace by pressing the **Furnace** button on the control panel.

Test Conditions

The figure below shows the TMA instrument control software window.

- At the top of the window, you will notice a toolbar that includes buttons for starting (green "play" arrow) and stopping (red square) the run.
- At the left of the window, the run sequence is indicated. Usually a single run is shown here, unless you have set up a multiple run sequence for your samples.
- The middle of the window contains tabs that allow you to change the sample information and testing parameters.
- The top right of the screen shows the live signals from the hardware.
- The middle right shows the run sequence for your test.
- The bottom right shows a live graph of your data once the test has started.



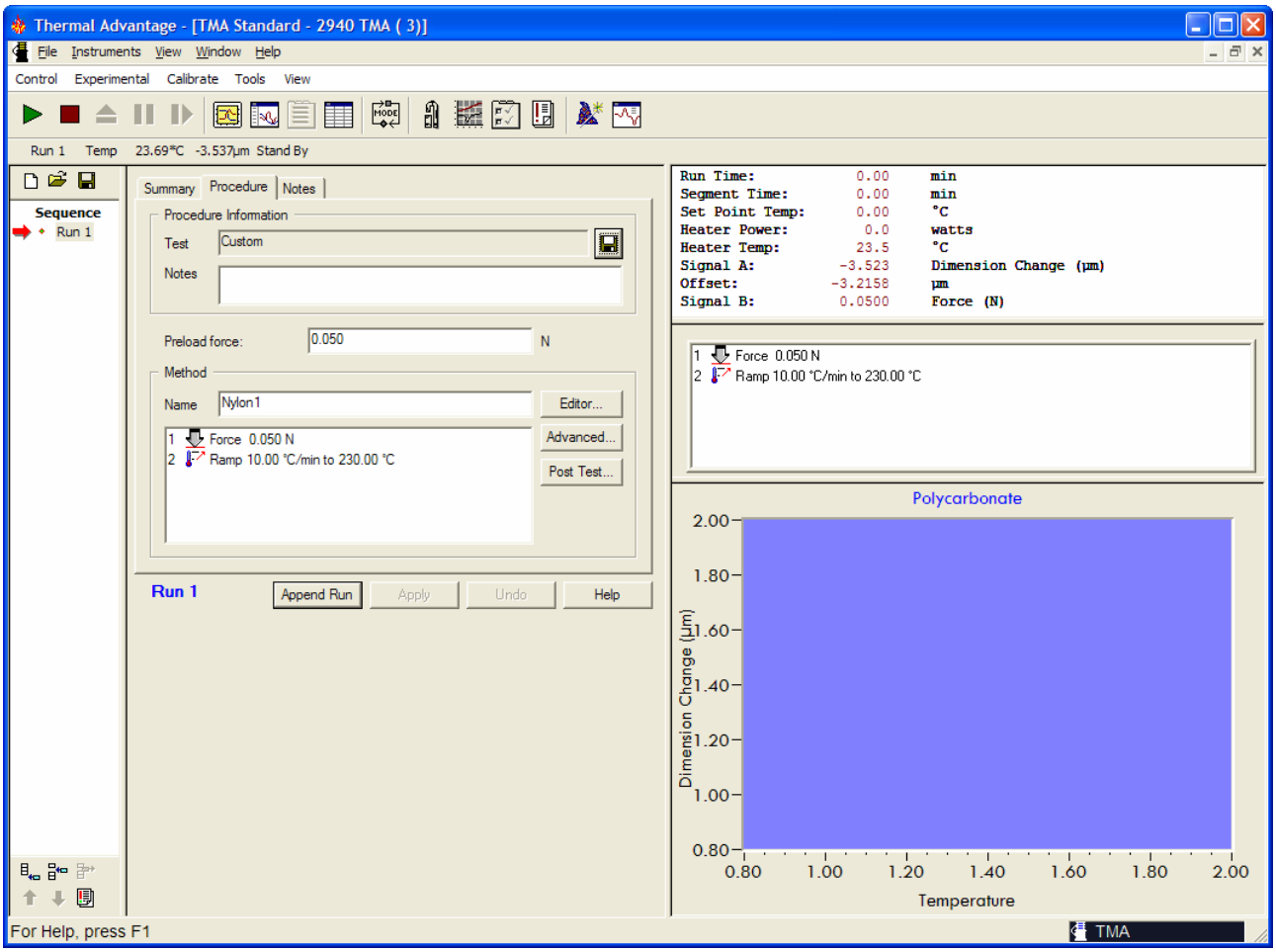
After you have loaded your sample and closed the furnace (see Sample Loading section), follow this sequence of steps to run a test:

Summary. This tab provides prompts for sample name, sample size, file name, etc.

1. Measure your sample height by pressing the **Measure Length** button on the instrument control panel (hardware). The measured sample height should be automatically displayed after the "Sample Size" prompt in the **Summary** tab.
2. Enter your sample name, comments, and data file name in the appropriate spaces.

Procedure. This tab allows you to customize the test parameters.

1. Click on the **Procedure** tab.
2. If you would like to make changes to the ramp rate, temperature, or applied force, click the **Editor…** button and make appropriate changes. The following settings are recommended for most specimens:
   - Force: 0.050 N
   - Ramp: 10.00 °C/min
   - Temperature: -150 °C to 1000 °C (depends on the sample)
3. Click the **Apply** button to apply your changes to the current run sequence.



Running the Test

To start the test run, simply click the green "play" arrow on the toolbar. Within moments, you should see data appearing on the graph.

Cleanup

After the furnace and stage assemblies have cooled sufficiently, remove your sample and **VERY CAREFULLY** clean off any debris on the sample stage. Cotton swabs are recommended for cleaning the stage.

The quartz probe and sample stage are quite fragile, and the probe position and balance calibrations may be affected if you apply too much force to the probe. If you have questions regarding cleaning of the probe or sample stage, ask your instructor or TA for assistance.

Hardware Shutdown

You may leave the instrument and computer turned on. If you would like to shut it down,

1. Turn off the orange **Heater** and **Power** buttons on the instrument control panel.
2. Shutdown the computer.
3. If the heater and/or sample stage areas are hot when you are ready to leave the lab, place a "WARNING: HOT" sign on the TMA.