

Thermal Stresses and Strains

Objective

The objective of this laboratory is for you to explore the origin and magnitude of thermally generated stresses and strains.

Preparation

- Read chapter on dilatometry by Speyer
- Read introductory sections of the review on metal-glass seals
- Read sections 1-10 of the ASTM standard test method using resonant vibrations of a bar
- Read pages 111-121 of Introduction to Glass Science and Technology

Equipment

- Station for thermo-mechanical analysis (TMA)
- Station for measuring elastic constants using the resonant vibrations of a bar
- Samples of Kovar, Ti, borosilicate glass, soft glass
- Computer, data-acquisition board and software, plotting software, microphone, audio analysis software.

Introduction

Materials processing frequently involves elevated temperatures. An example is the joining of glass to a metal for forming air-tight seals for a wide variety of applications. At the temperature where the seal is formed, the glass flows and the stresses are small. But when the structure is cooled to room temperature, differences in the thermal expansions of the metal and glass generate thermal stresses at the interface that have to be managed. If this stress is too high, the seal will fracture or delaminate. The thermal stresses are a function of both the temperature integral of the thermal expansions coefficients and the elastic constants of the materials.

Session 1: Thermal stresses in sealing of glass

- Measure elongation of Kovar, Ti, borosilicate glass, and soda-lime glass by TMA.
- Use TMA to determine the processing temperature needed to reduce the viscosity of the glass into range needed to make a metal-glass seal.

- Measure elastic constants of Kovar, Ti, borosilicate glass and soft glass by resonant vibrations.
- Analyze your results to give the thermal stress of a simple geometry of Kovar-glass and Ti-glass bonds.

Session 2: Accuracy and precision of dilatometry and measurements of elastic constants

- How accurate are the measurements of elastic constants? What are the uncertainties? Does the measurement of the resonant frequency depend on how the bar is tapped? Does it depend on how the bar is suspended? Do the longitudinal and transverse vibrations give the same result? Why or why not?
- How accurate are the dilatometry measurements? Does changing the temperature ramp-rate change the results? Does this depend on the material? Consider how long it takes for the specimen to come to thermal equilibrium: the time-scale for thermal equilibration by conduction is the radius-squared divided by the thermal diffusivity. For glass, the thermal diffusivity is approximately $D \sim 0.01 \text{ cm}^2/\text{sec}$; for Kovar, $D \sim 0.1 \text{ cm}^2/\text{sec}$; for Mo, $D \sim 1 \text{ cm}^2/\text{sec}$.

Instrument procedures

Old TMS-2 TMAs

1. Turn on control units: The TMS-2 Thermomechanical Analyzer (done by TA's)
 - a. Set Range to x.01. Power to ON. Don't touch Zero Dial. (done by TA's)
 - b. Set temperature Controller Ramp Rate to $10^\circ\text{C}/\text{min}$ (done by TA's)
2. Turn off the Furnace and Controller.
3. Turn on controller. Set maximum temperature by using arrows on Eurotherm temperature controller
4. Measure your samples. There are a microbalance and calipers available.
5. To Load samples:
 - a. First cover the furnace with the metal plate so your sample does not fall in.
 - b. Lift probe assembly (on top, black) and latch it (clicks).
 - c. Load sample on platform
 - d. Carefully release latch
 - e. Add the appropriate weight to the pan on top. (Using nickel and slugs).
6. Very carefully raise the furnace around the quartz probe until it clicks and stays in place. Do not crack the probe.

Open Easy Sense 2250 (shortcut on desktop) and Enter the following configurations:

USB2250 Sensor Configuration - Card 1

File View Card Number Help

Item: 0

Sensor Type: TC Ref Junction, LM61

Data Logging:

Channel: Ref Junct Terminal

Range: +/-1.280 V

Column: C

Engineering Units: °F

Scale Factor: 180

Offset Factor: -76

Average: 0

Precision: 0.000

Tag ID: Item 0

Reading: 72.061

Upload Config to Card: **Activate**

Item: 1

Sensor Type: Vdc, Differential, Auto-Zero

Data Logging:

Channel: Ch 7

Range: +/-160 V

Column: D

Engineering Units: um

Scale Factor: 1000

Offset Factor: 0

Average: 100

Precision: 0.000

Tag ID: Item 1

Reading: 14.609

Upload Config to Card: **Activate**

Item: 2

Sensor Type: Thermocouples, Type K, SE

Data Logging:

Channel: Ch 5A

Range: -416 to 2498 F

Column: E

Engineering Units: °C

Scale Factor: .555

Offset Factor: -17.777

Average: 100

Precision: 0.0

Tag ID: Item 2

Reading: 450.8

Upload Config to Card: **Activate**

Data Logging Controls - Card 1

View

Scans Remaining: 65000 Off

Scan Interval: 6 seconds

Time Units: sec

Starting Row: 385 Row Increment

Reset Scans Remaining

Reset Elapsed Time

Single Shot

Stop

Remarks:

Insert Remark

Data Log: Excel Ascii

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7. Calibrate the LVDT output to zero with the FINE control. Do not use the big, inner COARSE control knob, using the coarse knob will waste a lot of time in calibrating it.
8. Start acquiring data on the computer.
9. Turn off the controller and furnace. Then turn on the furnace. Then turn on the controller to start ramping the temperature.
10. Let temperature run to your desired temperature. To stop increasing the temperature and cool the sample turn off the furnace. Leave the controller on to see the temperature. Remember do not melt the glass.
11. Stop acquiring data on the computer (click Stop).

New Pyris Diamond TMA

1. Open Pyris Manager by double clicking the icon on the desktop.
2. Click Diamond TMA HTI icon to start the data acquisition program.
 - a. Note: once open, do not close the program until you are completely finished.
3. If not already on, turn on Pyris Diamond TMA by pressing the big green power button on the lower left.
4. Press the link button. It will ask you to initialize the probe, click yes.
5. File -> New Method Editor
 - a. Under the **Sample Info** tab, fill out Sample ID and Operator ID and select where you want to save the data to. System geometry = Expansion/Disc. Height will be filled in automatically but diameter is not.
 - b. **Initial State** tab: Initial temp = 25°C, set desired Force.
 - c. **Program** tab, press add step. Select desired final temperature, and set ramp rate to 10°C/min.
6. Click on the **Sample Load** icon. For selected position, enter a value two millimeters greater than the measured sample height, and click **Move Tube**. Before loading the sample, cover the furnace opening with a metal plate to prevent the sample from falling in. Load the sample onto the stage with tweezers, and click **Close Gap**. When probe is set, click **Exit**.
7. Raise the furnace using the arrows on the machine. Close the gate.
8. Click the Reset Monitor button, then click the Start/Stop button to start.
9. To see the data being plot, Windows -> Instrument Viewer. Adjust the graph axis with the icons that appear.
10. The program should stop automatically when the set temperature is reached. However, to stop it manually for the glass sample, press the Start/Stop button again.
11. To unload the sample
 - a. Open the gate
 - b. Lower the furnace with the down arrow
 - c. Cover the opening with the metal place.
 - d. Click on the Sample Load icon and increase the value by 2 millimeters.
 - e. Remove the sample with tweezers.
12. Export the data by File → Export Data → ASCII format.
13. Check both boxes and click ok.
14. Use a USB drive to back up your data. You may also use netfiles or email the data to yourself.

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