## **Pyris Diamond DMA**

Dynamic Mechanical Analyzer

Measurement Procedure

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## Preface

Thank you for selecting the Perkin Elmer Instruments Viscoelasticity Measurement Module Pyris Diamond DMA.

The Pyris Diamond DMA Viscoelasticity Measurement Module (temperature range:  $-150^{\circ}$ C ~  $600^{\circ}$ C) measures the dynamic viscoelasticity and creep recovery and stress relaxation using bend, tension, shear and compression methods to measure things such as polymeric materials simply and with high precision, as a function of temperature, frequency, or time. This operation manual is intended to help the user make the most effective use of the instrument's functions by centering on the operation of the system hardware. Please read this manual thoroughly before using the instrument.

Never use this instrument for purposes other than those described above.

For more information about the system software, refer to the Pyris Station Operation Manual. For information concerning measurement and analysis, refer to the Measurement Procedure Operation Manual.

This operation manual is based on the following software version and hardware: Pyris Station Install Kit Ver. 7.0 ~

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## **Chapter 1 DMA Measurement & Analysis**

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This chapter provides a step by step overview of measuring and analyzing sample data taken from the DMA. If this is the first time you have operated a DMA module, perform all of the steps in this chapter using the provided standard sample (PMMA). For more information on the items listed in this chapter, refer to the Pyris Station Operation Manual.

## **1.1 Before Measuring**

#### System Setup

- 1. Connect the measurement module to the workstation.
- 2. When using various gases, attach the piping and adjust all gas pressures and gas flow amounts.
- 3. When using the automatic cooling system, make sure the liquid nitrogen tank is full and all tubing is properly connected.
- 4. If you intend to plot data after measurements are completed connect the plotter to the system.

|                                  | NOTE                                  |
|----------------------------------|---------------------------------------|
| For more information on connecti | ons refer to the following sections:  |
| Module/ Plotter connections      | Section 3.1 in the Pyris Thermal      |
|                                  | Analysis & Rheology System            |
|                                  | Operation Manual                      |
|                                  |                                       |
| Gas tubing                       | Chapter 2 in the Module Operation     |
|                                  | Manual                                |
| Auto cooling system Cha          | pter 2 in the Module Operation Manual |

#### **Booting Up the System Software**

- 1. Turn on the power to the work station and the module.
- 2. After several minutes the following login window will appear on the CRT screen.



Figure 1-1 Login Window

3. Type "PKI" as the Login name and click on the OK button or press the return key.



4. The Pyris software will boot up and the PKI Menu Window will appear on the screen.

| File     Selected     View       DSC_Measure     TODTA_Measure     TMASS_Measure       Robot_DSC_Measure     Robot_TGDTA_Measure       BND_Measure     Robot_TGDTA_Measure       DSC_Analysis     TGDTA_Analysis       TGDTA_Analysis     TMASS_Analysis       SSC_Sub     TGDTA_Sub       TGDTA_Sub     TMASS_Sub       DSC_Sub     TGDTA_Sub       TGDTA_Sub     TMASS_Sub       DSC_Sub     TGDTA_Sub       TMASS_Sub     DSC_Cp   | -                  | Ar                | pplication Manager – SE | IKO                    | · [      |
|---|--------------------|-------------------|-------------------------|------------------------|----------|
| Image: Semi sub of MASS_Measure     Image: Semi sub of MASS_Measure       ScMeasure     TODTA_Measure       Robot_DSC_Measure     Robot_TGDTA_Measure       BND_Measure     TEN_Measure       DSC_Analysis     TGDTA_Analysis       TGDTA_Analysis     TMASS_Analysis       Stice_Analysis     TSIce_Analysis       DSC_Sub     TGDTA_Sub       TMASS_Sub     DSC_Cp  | File Selected View |                   |                         |                        | Help     |
| Robot_DSC_Measure     Robot_TGDTA_Measure       BND_Measure     TEN_Measure       DSC_Analysis     TGDTA_Analysis       TGDTA_Analysis     TMASS_Analysis       SS_Curve_Analysis     TSIce_Analysis       DSC_Sub     TGDTA_Sub       TGDTA_Sub     TMASS_Sub       DSC_Sub     TGDTA_Sub  | DSC_Measure        | TGDTA_Measure     | TMASS_Measure           | Semi autoTMASS_Measure | 2        |
| Image: Signal system     Image: Signal system     Image: Signal system       BND_Measure     TEN_Measure     DES_Measure     DMS_SS_Measure       DSC_Analysis     TGDTA_Analysis     TMASS_Analysis     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Image: Signal system     Image: Signal system     SS_Curve_Analysis       Image: Signal system     Imag | Robot_DSC_Measure  | Robot_TGDTA_Measu | ire                     |                        |          |
| DSC_Analysis TGDTA_Analysis TMASS_Analysis SS_Curve_Analysis<br>f_Slice_Analysis T_Slice_Analysis<br>DSC_Sub TGDTA_Sub TMASS_Sub DSC_Cp   | BND_Measure        | TEN_Measure       | DES_Measure             | DMS_SS_Measure         |          |
| Image: Single constraints       f_Slice_Analysis       T_Slice_Analysis       Image: Single constraints   | DSC_Analysis       | TGDTA_Analysis    | TMASS_Analysis          | SS_Curve_Analysis      |          |
| DSC_Sub TGDTA_Sub TMASS_Sub DSC_Cp  | f_Slice_Analysis   | T_Slice_Analysis  |                         |                        | -        |
|   | DSC_Sub            | TGDTA_Sub         | TMASS_Sub               | DSC_Cp                 |          |
| DSC_Kinet TGDTA_Kinet DSC_Purity RheoDeltaE   | DSC_Kinet          | TGDTA_Kinet       | DSC_Purity              | RheoDeltaE             |          |
|   |                    | 5135353           | 00000                   | 50500                  | <u>k</u> |

Figure 1-2 PKI Menu Window

#### **Opening the DMS/SS Measurement Window**

- 1. Double click on the DMS\_SS\_Measure icon located on the main menu.
- 2. The DMS/SS measurement window will open.
- 3. Select Open from the DMS/SS measurement window File menu.
- 4. The following Device Selection window will appear.

| /dev<br>/dev<br>/dev<br>/dev<br>/dev<br>/dev | /ttyOpO<br>/ttyIpO<br>/ttyC10<br>/ttyC11<br>/ttyC12<br>/ttyC13<br>/ttyC14 | acted Nevid | °6 |  |
|--|---|-------------|----|--|
| /dev   | v/ttyOpŐ  |             |    |  |

Figure 1-3

5. Select the device you want from the Available Device List and click the OK button.

6. The following message, "Communicating. . .", will appear in the top left corner, and the selected module name and channel number will appear in the window bar. The module is now ready to measure. (If the communicating message does not appear after five or six minutes confirm that the selected channel and module are compatible.)



Figure 1-4

#### Initializing the Module

1. Press and hold down the Furnace Move switch on the front panel of the module until the furnace stops. The furnace first drops down and then moves to the back before stopping

## S NOTE

The furnace will only move when both the furnace move switch on the right and the furnace control switch on the left are pushed simultaneously.

- 2. If there is a sample placed in the measurement unit, remove the sample.
- 3. Click the Motor Move icon. The Position Settings/Tension Conditons window will be dispalyed.
- 4. Click the Initialize button to perform initialization.

- 1. Be sure to perform initialization after connecting the module. The origin point of the system is detected by performing initialization.
- 2. When performing initialization, make sure that the probe is in a free (samples etc. are not in a chucking state).

#### **Attaching Sample Unit Attachments**

| Measurement Mode              | Attachment                         |  |
|-------------------------------|------------------------------------|--|
| Bend Measurement              | Bend Measurement Attachment        |  |
| Single-Cantilever Measurement | Bend Weasurement Attachment        |  |
| 3point-Bending Measurement    | 3point-Bending Attachment          |  |
| Tension Measurement           | Tension Measurement Attachment     |  |
| F Control Measurement         | No specific attachment             |  |
| L Control Measurement         | No specific attachment             |  |
| Shear Mode                    | Shear Measurement Attachment       |  |
| Compression Mode              | Compression Measurement Attachment |  |

Exchange the sample unit attachments to best suit the needs of the measurement mode.



The 3point-bending, shear mode and compression mode attachments are options.

When the currently attached attachment is for a different measurement mode, perform the operations in the following sub-section entitled Preparing the Measurement Attachment. When the appropriate attachment is already attached continue onto Section 1.2 Placing the Sample.



It is necessary to perform calibrations in addition to the system calibrations in chapter 3 for single-cantilever and 3 Point-bending measurements.

For detailed information, refer to Appendix L Single-Cantilever Measurements and Appendix M 3 Point-Bending Measurements.

#### **Preparing the Measurement Attachment**

#### **Bend Measurement Attachment**

- 1. Remove the current attachment.
- Click the Motor Move icon. The Position Settings/Tension Conditions window will be displayed. Set the Target Movement Position to -16000µm and click the Move button. The probe will drop 16mm.
- 3. Attach the bend attachment to the gap setting plate. For detailed information on how to attach the attachment, refer to Section 5.1 in the Pyris Diamond DMA Module Operation Manual.
- 4. Once the bend attachment has been attached, remove the gap setting plate.
- 5. Click the Motor Movement icon. The Position Settings/Tension Conditions window will be displayed. Set the Offset Load to 700mN and click the OK button. The window close and you should observe the L (strain signal) value. If the L (strain signal) value is not within 0.5µm open the Position Settings/Tension Conditions window again and input the Offset Load again and continue to adjust the value until the L (strain signal) is within 0.5µm.

#### **NOTE**

- 1. The value entered in the Offset Load will be reflected in the initial system operation once the OK button is clicked.
- 2. When the Offset Load is changed by 10mN, the L (strain signal) changes to -3  $\sim$  -4  $\mu m.$
- Click the Motor Movement icon. The Position Settings/Tension Conditions window will be displayed. Set the Target Movement Position to -16000μm and click the Move button. The probe will drop 16mm.

#### **Tension Measurement Attachment**

1. Remove the current attachment.

- 2. Attach the tension attachment. For detailed information on how to attach the attachment, refer to Section 5.1 in the Pyris Diamond DMA Module Operation Manual.
- 3. Once the tension attachment has been attached, click the Motor Movement icon. The Position Settings/Tension Conditions window will be displayed. Click the Initialize button and perform initialization. The default chuck space (sample length) after initialization is 20mm.
- 4. Perform the following operations to align the origin point of the LVDT.
  - a. Click the Motor Movement icon. The Position Settings/Tension Conditions window will be displayed. Set the Offset Load to 0mN and click the OK button. The window will close.
  - b. Click the Motor Movement icon again. The Position Settings/Tension Conditions window will be displayed. Set the Offset Load to 700mN and click the OK button. The window will close and you should observe the L (strain signal) value. If the L (strain signal) value is not within 0.5µm open the Position Settings/Tension Conditions window again and input the Offset Load again and continue to adjust the value until the L (strain signal) is within 0.5µm.

- 1. The value entered in the Offset Load will be reflected in the initial system operation once the OK button is clicked.
- 2. When the Offset Load is changed by 10mN, the L (strain signal) changes to -3  $\sim$  -4 $\mu m.$

## **1.2 Placing the Sample**

#### Preparation

- 1. Make the sample into an appropriate shape beforehand. To determine the appropriate shape, refer to Appendix A and B in the Dynamic Mechanical Analyzer Measurement Procedure Pyris Diamond DMA Operation Manual.
- 2. Sample Fusion Protection Operations

When there is a possibility that the measurement sample may attach to the chuck or clamp during the measurement, apply an anti stick compound. A soapy water solution is a simple compound to use.

## S NOTE

If the sample is the sort that may stick to the measurement head or chuck, use an anti stick compound before setting the sample. A sillicon based spray works well.

#### **Placement Method**

#### **For Bend Measurements**

- When the chuck is at the origin point, click the Motor Movement icon. The Position Settings/Tension Conditions window will be displayed. Set the Target Movement Position to -16000µm and click the Move button. The chuck will drop to the sample set position.
- 2. Loosen the clamp knurled screws and the chuck hexagonal screws then slide the PMMA sample from the right through the space between the base and the clamp.
- 3. Adjust the position of the sample using a pair of tweezers, tighten both of the clamp knurled screws. Next, align the chuck hexagonal screw and tighten it.
- 4. Loosen both clamps temporarily, and refasten both clamps. Use the attached flathead screwdriver fasten.

## Information

- 1. When using soft samples such as rubber type samples, tighten the chuck side first and then both of the clamps so that the sample does not bend.
- 2. When loosening both clamps in step 4, the sample warp that appears when tightening the chuck screws in step 3 is elminated.

#### For Tension Measurements

1. Enter a value in μm units into the Movement Target Position and click the Move button. When Move is clicked, the stage motor position moves to the target position.

## Information

The standard length for the samples is 20mm. Therefore, when measuring samples with lengths of 20mm the Movement Target Position is  $0\mu m$ . When the sample length is 10mm enter -10000 for the Movement Target Position and enter 5000 for a sample length of 25mm then click the Move button.

2. Loosen the chuck and the clamp. Insert the sample from the bottom of the clamp as shown in the figure below, then tighten first the clamp and then the chuck.



3. Loosen the clamp screw temporarily and tighten the screws securely.

# NOTE 1. When tightening the chuck, secure the chuck base with one of your hands and be careful not to put excessive force on the probe. 2. By temporarily loosening the clamp screw in step 3, sample warp

created in tightening the clamps in step 2 can be eliminated.

4. Insert the radiation shield plate (for tension measurements) into the slit of the tension base so that the sample is maintained on the right side as shown in the figure.



Right Side View





## **1.3 Setting the Measurement Conditions** Setting the Dynamic Viscoelasticity Measurement Conditions Using Bend

Sinusoidal Oscillation Measurement

Click the Sample Condition Settings icon and open the window the enter data into each section.

| Viscoelasticity Sample Condition Setting  |   |
|---|---|
| Sample Name PHHH.<br>Length(ww) 20  | Ramp Temperature Control Hode 🗖<br>Temp. Program Settings |
| Rectangular Heas. Range   |   |
| Thickness(ww) 1.5   | Bending/Sinusoidal Oscillation                            |
| Operator .  | Frequency Setting   |
|   | Position and Tension Settings                             |
|   | Hanual Gas 1 Hanual Gas 2                                 |
|   | Fusion Protection [ 100000 (Pa)                           |
| Data File Name           [/C=/USERS/SEIX0/MHS100/PHHH-1              B Overwrite           B Auto Filename Update | Air Cooldown Temp. 1.2000.0<br>(C)                        |
| OK  | Cance I   |

Figure 1-6A Sample Conditions

|                         | Fr        | equency Setting | j - ch2.max100Hz 🛛 🖓 |
|-------------------------|-----------|-----------------|----------------------|
| <u>F</u> ile <u>E</u> d | it        |                 |                      |
|                         | 0.01 [Hz] | <u> </u>        | 1 [Hz] [ 1           |
|                         | 0.02 [Hz] | Ĭ 0.02          | 2 [Hz]               |
|                         | 0.05 [Hz] | 1 0.05          | 4 [Hz]               |
|                         | 0.1 [Hz]  | Ĭ 0.1           | 10 [Hz]              |
|                         | 0.2 [Hz]  | <u>ĭ</u> 0.2    | 20 [Hz]              |
|                         | 0.5 [Hz]  | 0.5             |                      |
|                         | 1 [Hz]    | 1 1             |                      |
| ×                       | 2 [Hz]    | <u>ĭ</u> 2      |                      |
|                         | 5 [Hz]    | 15              |                      |
|                         | 10 [Hz]   | 10              |                      |
|                         | 20 [Hz]   | <u>1</u> 20     |                      |
|                         | 50 [Hz]   | ž 50            |                      |
|                         | 100 [Hz]  | 100             |                      |
| <u> </u>                |           |                 |                      |
|                         | OK        |                 | Cancel               |

Figure 1-6C Frequency Settings



Figure 1-6B Temperature Program Settings

| Position and Tension Settings                  |            |
|--|------------|
|  |            |
|  |            |
|  | Initialize |
| Current Position(um) 0.0                       |            |
| Movement Target Position (um)                  | Hove       |
| Offset Load(wN) 🕺 710.00                       |            |
|  |            |
| Lamp. 10.0                                     |            |
| Hin. Tension/Compression 100.00                |            |
| Tension/Compression 1.500<br>Force Gain        |            |
| Force Amplitude<br>Initial Value(mN) [ 4000.00 |            |
| Annual Having Courses                          |            |
| Deformation Amount(um)                         |            |
| Position Movement 8.0                          |            |
| Creep Wait Time Constant . 0.0000              |            |
|  |            |
|  | Cancel     |
|  | Galice I   |



The following explanation uses the measurement of a PMMA sample as an example.

| Input the following information: |   |
|----------------------------------|---|
| Sample Name                      | PMMA  |
| Length                           | 20 (during standard clamp)                      |
| Rectangular Cross Section        | Select Rect. cross section with button.         |
| /Circle Cross Section            |   |
| Width                            | Sample Width Measure Value (Ex.10)              |
| Thickness                        | Sample Thickness Measure Value (Ex.1.5)         |
| Comments                         | Pyris Diamond DMA                               |
|                                  | PMMA Sample                                     |
| Operator                         | Users Name                                      |
| Dir. Contents                    | Click the button to confirm the filename        |
| Data File Name                   | /users/PKI/data_dms_bend.00                     |
| Overwrite                        | Off   |
| Auto Filename Update             | On  |
| Temperature Control Mode         | Select the Ramp Temperature Control mode        |
|                                  | using the button.                               |
| Temperature Program Settings     | Click this button with the mouse and set the    |
|                                  | following items.                                |
| Start Temperature                | 25°C  |
| Limit Temperature                | 200°C   |
| Heating Rate                     | 2°C/min   |
| Hold Time                        | 0 min   |
| Sampling                         | 1 sec   |
| End Step                         | 1   |
| Control Mode                     | Select Bending/Sinusoidal Oscillation using the |
|                                  | button.   |
| Frequency Setting                | Click the button to display the window and turn |
|                                  | ON the following switches: 0.5, 1, 2, 5, 10     |
| Position and Tension Settings    | Click the Position and Tension Settings button  |
|                                  | and input the following:                        |
| L. Amplitude                     | 10µm  |
| Force Amplitude Initial Value    | 4000mN  |
| Manual Gas 1,2                   | Off   |
| Fusion Protection (Pa)           | 0   |
| Air Cooldown Temperature         | 2000°C (Active only when using auto cooler)     |
| Shutdown After Measurement       | Off   |

- 1. Temperature program settings can also be modified through the Temperature Program icon.
- 2. Frequency settings can also be modified through the Frequency Setting icon.
- 3. To measure a sample using the same conditions you only need to enter the sample name and shape.
- 4. When all of the Frequency Settings are Off, the default setting of 2Hz will be used.
- 5. When measuring soft samples, set the Force Amplitude Initial Value to a low setting. (ex. 10mN)

Synthetic Oscillation Measurement

For the synthetic oscillation measurement, make the following changes to the previous settings for the sinusoidal oscillation measurement:

| nter |
|------|
| ı of |
|      |
|      |
|      |

## S NOTE

The L. Amplitude (target) for the Synthetic Oscillation measurement is relative to the standard frequency used. During the measurement, the actual L. Amplitude is comprised of each of the frequency components so the amplitude should be as close to 5 times the target value as possible. For Synthetic Oscillation measurements, the L. Amplitude should generally be set to  $5\mu$ m.

is – ch2.rhe

1.0

1

25.00

200.00

2.00

0.00

Time 1:27

1

4

50.00

50.00

0.10

0.00

End Step

2

200.00

50.00

2.00

0.00

Cancel

3

50.00

50.00

0.10

0.00

#### Setting the Dynamic Viscoelasticity Measurement Conditions Using Tension

| Viscoelasticity Sample Condition Setting   |  |
|--|--|
| Sample Hame PHHM<br>Length(mm) 1 20  | Ramp Temperature Control Mode 🗖<br>Temp. Program Settings      |
| Rectangular  |  |
| Hidth(mm) 10<br>Thickness(mm) 1.5  | Bending/Sinusoidal Oscillation                                 |
| Operator I   | Frequency Setting<br>SS Program Settings                       |
| Comments   | Position and Tension Settings                                  |
|  | 🗆 Hanual Gas 1 🗆 Hanual Gas 2                                  |
| Data File Name   | Fusion Protection [100000]                                     |
| [//c=/USERS/SEIK0/DHS6100/PHHA-1        ]           Image: Deerwrite         Image: Ruto Filename Update | Air Cooldown Temp. 2000.0<br>(C) Shutdown After<br>Heasurement |
| OK   | Cance I  |

Sinusoidal Oscillation Measurement

Click the Sample Condition Settings icon and open the window the enter data into each section.

File

Soak (sec)

Start Temp. (C)

Limit Temp. (C)

Figure 1-7B Temperature Program Settings

Sampling (sec)

Step

Start Temp. (C)

Limit Temp. (C)

Heating Rate (C∕∎in)

> lold Tim (min)

1.0

1 38,80

58,80

1

OK



Figure 1-7A Sample Conditions

Figure 1-7C Frequency Settings



Figure 1-7D Position Settings/ Tension Conditions

The following explanation uses the measurement of a PMMA sample as an example.

| Input the following information: |  |  |
|----------------------------------|--|--|
| Sample Name                      | PMMA   |  |
| Length                           | 20 (or auto read)  |  |
| Rect./Circ. Cross Section        | Select Rect. cross section with button                     |  |
| Width                            | Sample Width Measure Value (Ex.4.5)                        |  |
| Thickness                        | Sample Thickness Measure Value (Ex.0.2)                    |  |
| Comments                         | Pyris Diamond DMA  |  |
|                                  | PMMA Sample  |  |
| Operator                         | Users Name   |  |
| Dir. Contents                    | Click the button to confirm the filename                   |  |
| Data File Name                   | /users/PKI/data_dms_tens.00                                |  |
| Overwrite                        | Off  |  |
| Auto Filename Update             | On   |  |
| Temperature Control Mode         | Select the Ramp Temperature Control mode using the button. |  |
| Temperature Program Settings     | Click this button with the mouse and set the               |  |
|                                  | following items.   |  |
| Start Temperature                | 25°C   |  |
| Limit Temperature                | 200°C  |  |
| Heating Rate                     | 2°C/min  |  |
| Hold Time                        | 0 min  |  |
| Sampling                         | 1 sec  |  |
| End Step                         | 1  |  |
| Control Mode                     | Select Tension/Sinusoidal Oscillation using the            |  |
|                                  | button.  |  |
| Frequency Setting                | Click the button to display the window and turn            |  |
|                                  | ON the following switches: 0.5, 1, 2, 5, 10                |  |
| Position and Tension Settings    | Click the Position and Tension Settings button             |  |
|                                  | and input the following:                                   |  |
| L. Amp.                          | 10µm   |  |
| Min. Tension/Compression Force   | 200mN  |  |
| Tension/Compression Force gain   | 1.500  |  |
| Force Amplitude Initial Value    | 4000mN   |  |
| Approved Maximum Deformation     | 10000.0µm  |  |
| Position Movement Wait Time      | 8.0sec   |  |
| Creep Wait Time                  | 0.000sec/mN  |  |
| Manual Gas 1,2                   | Off  |  |
| Fusion Protection (Pa)           | 0  |  |
| Air Cooldown Temperature         | 2000°C (Active only when using auto cooler)                |  |
| Shutdown After Measurement       | Off  |  |

- 1. Temperature program settings can also be modified through the Temperature Program icon.
- 2. Frequency settings can also be modified through the Frequency Setting icon.
- 3. To measure a sample using the same conditions you only need to enter the sample name and shape.
- 4. Position Settings/Tension Conditions can also be modified using the Motor Movement icon.
- 5. When all of the Frequency Settings are Off, the default setting of 2Hz will be used.
- 6. When measuring soft samples, set the Force Amplitude Initial Value to a low setting. (ex. 10mN)

Synthetic Oscillation Measurement

For the Synthetic oscillation measurement, make the following changes to the previous settings for the sinusoidal oscillation measurement:

Comments Control Mode Frequency Setting

L. Amplitude

Sinusoidal Oscillation  $\rightarrow$  Synthetic Oscillation Tension/Synthetic oscillation mode.

Click the button to display the window and enter 0.5 (Hz) for the standard frequency.

 $10 \rightarrow 5$ 

## S NOTE

The L. Amplitude (target) for the synthetic oscillation measurement is relative to the standard frequency used. During the measurement, the actual L. Amplitude is comprised of each of the frequency components so the amplitude should be as close to 5 times the target value as possible. For synthetic oscillation measurements, the L. Amplitude should generally be set to  $5\mu$ m.

#### Setting the Dynamic Viscoelasticity Measurement Conditions for Creep Restoration (Using F Control Mode)



Click the Sample Condition Settings icon and open the window the enter data into each section.

Figure 1-8A Sample Conditions



Figure 1-8B Temperature Program Settings



Figure 1-8C SS Program Settings

The following explanation uses a tension measurement with a rubber sample (rubber band) at room temperature as an example.

| Input the following information: |  |
|----------------------------------|--|
| Sample Name                      | rubber band                                      |
| Length                           | 20   |
| Rect./Circ. Cross Section        | Select Rect. cross section with button           |
| Width                            | Sample Width Measure Value (Ex.1)                |
| Thickness                        | Sample Thickness Measure Value (Ex.1)            |
| Comments                         | Pyris Diamond DMA                                |
|                                  | Creep Restoration Measurement                    |
| Operator                         | Users Name                                       |
| Dir. Contents                    | Click the button to confirm the filename         |
| Data File Name                   | /users/PKI/data_dms_creep.00                     |
| Overwrite                        | Off  |
| Auto Filename Update             | On   |
| Temperature Control Mode         | Select the Ramp Temperature Control mode         |
|                                  | using the button.                                |
| Temperature Program Settings     | Click this button with the mouse and set the     |
|                                  | following items.                                 |
| Start Temperature                | 0°C  |
| Limit Temperature                | 0°C  |
| Heating Rate                     | 2°C/min  |
| Hold Time                        | 20 min   |
| Sampling                         | 1 sec  |
| End Step                         | 1  |
| Control Mode                     | F Control  |
| SS Program Settings              | Click the button to display the window and input |
|                                  | the following:                                   |
|                                  | Step 1 Step 2                                    |
| Start Load                       | 0 (mN) 0 (mN)                                    |
| Limit Load                       | 0 (mN) 100 (mN)                                  |
| Load Rate                        | 100 (mN/min) 100000 (mN/min)                     |
| Hold Time                        | 3 (min) 3 (min)                                  |
| End Step                         | 2  |
| Manual Gas 1,2                   | Off  |
| Air Cooldown Temperature         | 2000°C (Active only when using auto cooler)      |
| Shutdown After Measurement       | Off  |

- 1. Temperature program settings can also be modified through the Temperature Program icon.
- 2. SS Program settings can also be modified through the SS Program Settings icon.
- 3. To measure a sample using the same conditions you only need to enter the sample name and shape.
- 4. When performing creep measurements the object is to measure only the response of the strain (removing thermal expansion) in relation to stress. Therefore, the temperature is maintained at a constant (start temperature and limit temperature are identical).
- 5. For measurements at room temperature, turn off the power to the cooling controller. For the above settings, a dummy program is used to keep the start and limit temperatures below room temperature since the heater is not used when performing measurements.

## Setting the Dynamic Viscoelasticity Measurement Conditions for Strain Relaxation (Using L Control Mode)



Click the Sample Condition Settings icon and open the window the enter data into each section.

Figure 1-9A Sample Conditions

Figure 1-9B Temperature Program Settings



Figure 1-9C SS Program Settings

The following explanation uses a tension measurement with a rubber sample (rubber band) at room temperature as an example.

| Input the following information: |  |
|----------------------------------|--|
| Sample Name                      | rubber band                                      |
| Length                           | 20mm   |
| Rect./Circ. Cross Section        | Select Rect. cross section with button           |
| Width                            | Sample Width Measure Value (Ex.1) (mm)           |
| Thickness                        | Sample Thickness Measure Value (Ex.1) (mm)       |
| Comments                         | Pyris Diamond DMA                                |
|                                  | Rubber Sample                                    |
|                                  | Strain Relaxation Measurement                    |
| Operator                         | Users Name                                       |
| Dir. Contents                    | Click the button to confirm the filename         |
| Data File Name                   | /users/PKI/data_dms_relax.00                     |
| Overwrite                        | Off  |
| Auto Filename Update             | On   |
| Temperature Control Mode         | Select the Ramp Temperature Control mode         |
|                                  | using the button.                                |
| Temperature Program Settings     | Click this button with the mouse and set the     |
|                                  | following items.                                 |
| Start Temperature                | 0°C  |
| Limit Temperature                | 0°C  |
| Heating Rate                     | 2°C/min  |
| Hold Time                        | 20 min   |
| Sampling                         | 1 sec  |
| End Step                         | 1  |
| Control Mode                     | L Control  |
| SS Program Settings              | Click the button to display the window and input |
|                                  | the following:                                   |
|                                  | Step 1 Step 2                                    |
| Start Displacement               | 0 (μm) 0 (μm)                                    |
| Limit Displacement               | 0 (μm) 1000 (μm)                                 |
| Displacement Rate                | 10 (µm/min) 100000 (µm/min)                      |
| Hold Time                        | 3 (min) 3 (min)                                  |
| End Step                         | 2  |
| Manual Gas 1,2                   | Off  |
| Air Cooldown Temperature         | 2000°C (Active only when using auto cooler)      |
| Shutdown After Measurement       | Off  |

- 1. Temperature program settings can also be modified through the Temperature Program icon.
- 2. SS Program settings can also be modified through the SS Program Settings icon.
- 3. To measure a sample using the same conditions you only need to enter the sample name and shape.
- 4. When performing stress relaxation measurements the object is to measure only the stress changes by controlling the amplitude rectangularly. Therefore, the temperature is maintained at a constant (start temperature and limit temperature are identical).
- 5. For measurements at room temperature, turn off the power to the cooling controller. For the above settings, a dummy program is used to keep the start and limit temperatures below room temperature since the heater is not used when performing measurements.
# 1.4 Starting and Stopping Measurements

# For Sinusoidal and Synthetic Osciallation Modes

#### **Check Measurement Range**

Click the Sample Conditions icon and open the window. Click the Measurement Range button to display a graph of the measurement range. Check the measurement range and the shape factor.



Test Measurement

Click the TEST button in the upper left hand corner of the measurement window to perform a test measurement using the current parameters. If the measurement is completed correctly, the viscoelastic modulus (E',E'') and the tan $\delta$  measurement value are displayed in the message window.

Always perform a test measurement before performing the actual measurement.

| Message  |                                     |
|--|-------------------------------------|
| € Test measurements are finished.<br>↓ (freq.=2(Hz) H'=4.037957e+009(Pa) | H"=3.287705e+008(Pa) tanD=0.081632) |
| ОК   | Display Graph                       |

Figure 1-10

Click the range display button to display a circle mark in the measurement range graph of the test measurement data.



An error message is displayed when the test measurement results are incorrect or the measurement was not completed correctly. When this happens, follow the instructions in the error message which may include checking the sample chucking or modifying the sample shape.

# S NOTE

- 1. A message window is displayed after each test measurement is performed. The message windows are always displayed in the same position. After several test measurements are performed, many message windows are stacked on top of each other. To compare the results from these different tests, move each window to a different location on the screen. Click OK to close the message window.
- 2. Never display more than 5 message windows at one time when performing test measurements. Errors can occur in the measurements when more than 5 message windows are displayed at one time.

# S NOTE

Testing after clicking the TEST button is as shown in the diagram below. Tests can only be performed if the module is in the ready or isothermal status. Click the CANCEL button to cancel a test measurement in progress.



#### **Starting and Stopping Measurements**

The four module control buttons are located in the upper left hand side of the window. When one of these buttons is pushed the module will follow the temperature program settings and control the temperature.

- 1. Click the RUN button and measurement will begin. Shortly after wait 1 is displayed momentarily, the module communication display will read "Running 1...".
- 2. In the ramp modes, the measurement ends and the status returns to ready when the temperature program reaches the final step's temperature limit. For the step mode, the measurement is automatically concluded when the final measurement completes at the limit temperature and the status returns to ready. To stop the measurement during Running..., click on the STOP button.



### F, L Control Modes

- 1. Click the HOLD button then wait for the sample temperature to stabilize.
- 2. Once the sample temperature has stabalized, click the RUN button to begin the measurement.
- 3. In the ramp modes, the measurement ends and the status returns to ready when the temperature program reaches the final step's temperature limit. For the step mode, the measurement is automatically concluded when the final measurement completes at the limit temperature and the status returns to ready. To cancel the measurement click the STOP button.



### **Control Target During L Control**

L Control mode operates the step motor, moves the detector position up and down and introduces it into the stage.

Along with the movement of the stage, the probe moves up and down and the sample length changes in accordance with the L Program.

The stage is controlled by the following equation during the L control measurement:

Stage Position = Movement Target Position (the last place moved to using the Move button in the Motor Movement icon) + L Control Program Value (in the SS Program icon)

In other words, during the L Control Measurement, the stage moves only the output value of the L Program with the last used movement position set to  $0\mu m$ . The stage does not move in the Ready status.

# S NOTE

The stage movement origin point at the L Program control is the last used movement position and not the current position. Furthermore, if the space between the last used movement target position and the current position is too large, the stage moves large distance along with the starting of the L control setting.

To avoid this, use the Move button in the Motor Move icon before the measurement and move the stage to the desired position. By performing this operation, the current and target positions will be the same and the above problem will not occur.

### Parameter Precalibration and Initialization with Control Loop

There are several parameters to be set within the control loop. In order to measure a variety of samples using viscoelastic measurements, these parameters precalibrate the sample properties while continuing measurements. This is known as the precalibration feature.

This precalibration feature is extremely effective for continuous samples with viscoelastic changes but when the sample is exchanged and the sample materials change, measurement errors can occur causing an obstacle to measurements.

In this circumstance, use the method below to perform initialization of the updated control parameters for each control mode.

 All Sinusoidal and Synthetic Oscillation Modes
 Click the TEST button in the upper left corner of the measurement window and perform a

TEST measurement. The control parameters are initialized.

2. F Control Mode

To initialize the control parameters move to the sinoidal wave or synthetic wave, perform a test measurement, then return to the F control mode.

3. L control Mode

To initialize the control parameters move to the sinusoidal oscillation or synthetic oscillation, perform a test measurement, then return to the L control mode.

# S NOTE

To move to a different control mode, select the other control mode using the control mode selection button and click the OK button. It is necessary to confirm the selected control mode.

# Helpful hints . . .

#### Set measurement mode display

The following should normally be displayed below the buttons in the upper left of the module control window:

- Current module status (Isothermal Hold, Running, etc.)
- Deformation Mode (Bend, Shear, Tension, Compression, F, L)
- Temperature Mode (Ramp, Step)

#### Selecting the measure data display mode

Click the Select Display icon to open the display mode selection window. Select one of the following display modes:

# P Information

The Y Axis Display Signal changes depending on the deformation mode.

| Display Mode   | X Axis | Y Axis                    |
|----------------|--------|---------------------------|
| Type 1 (time)  | time   | L, F, Position, Temp.     |
| Type 1 (temp)  | temp   | L, F, Position, Temp.     |
| Type 2 (time)  | time   | M', M", tanð, L, F, Temp. |
| Type 2 (temp)  | temp   | M', M", tanð, L, F, Temp. |
| Type 3 (freq.) | freq.  | M', M", tanδ, L, F, Temp. |

Type 1 is displayed in real time for both the Ready and Running... status. Type 2 and 3, however, display the previous measurement data during the ready status, and the data is displayed in real time during the Running... status. Turn On the Y axis log display to to change the corresponding Y axis display to a log display.

|                      | Lugar I unit C Expression |
|----------------------|---------------------------|
| ⊖Type1 (Time)        | <b>☑ Y1(H')</b>           |
| ⊖Type1 (Temperature) | ☑ Y2(H")                  |
| ⊖Type2 (Time)        | ☑ Y3(tanD)                |
| ⊖Type2 (Temperature) | II ¥40                    |
| C Type3 (Frequency)  | D Y5()                    |
|                      | 🗆 Y6(Temp.)               |
|                      | 1                         |
|                      |                           |

Figure 1-11

#### Changing the number of Y-axis displayed

Click on either the Y1 axis or Y2 axis signal setting button to change the axis signal setting. Select All Y Axes to display all Y1 ~ Y4 set signals.

#### Enlarging or reducing the data display

Data can be enlarged or reduced using the scroll bars and sliders that accompany each axis. Data can also be modified using the Scale Value button located in the bottom left hand corner of the screen.

# **1.5 Analyzing Measurement Data** (Sinusoidal & Synthetic Oscillation Control Mode Data)

The data measured with the DMA module can be graphed with time, temperature, or frequency on the horizontal axis, and with E', E'',  $|E^*|$ , J', J'', tan $\delta$ ,  $\eta$  etc. on the vertical axis, then analyzed with the analysis job.

In general, the dynamic viscoelasticity quantities measured with the DMA module osciallation mode depends on both the measurement temperature and frequency, as expressed by the 3-dimensional coordinates in the figure below.



Figure 1-12 3-Dimensional Coordinates Expressing Dynamic Viscoelasticity

The Pyris Station uses the following 2 types of analysis jobs, and is designed so that threedimensional analysis data can easily be obtained from data displayed in 2-dimensional form.

1. f Slice Analysis Job

This job analyzes a frequency slice (vertical slice on the X2 axis in the figure above) of data at a fixed measurement frequency and is useful for analyzing temperature dispersion.

2. T Slice Analysis Job

This job analyzes slice data at a fixed temperature (vertical slice on the X1 axis in the figure above) and is useful for analyzing frequency dispersion.

### **Opening the DMA Analysis Window**

- 1. Double click on the f\_Slice\_Analysis or T\_Slice\_Analysis icon found in the PKI main menu window. (The following explanation will be based on the f slice analysis.)
- 2. The Slice Analysis window will open.
- 3. Analysis data has not been selected at this point so all items in the menu bar will be deactivated except for Open.
- 4. Select Open from the menu bar and an Open File window will open.
- 5. Select the target file you want to analyze from the file list (for example: /users/PKI/data\_dms.00) and click the OK button.

# S NOTE

When a file containing analyzed results is selected with the mouse, a data profile will be displayed in the panel located at the right of the file list.

6. The data is opened and displayed in the window and analysis can begin.

### **Assign Slice**

When opening data in f slice analysis, all frequency data used for the measurement is set for each slice and displayed on the screen for data from the original measurement.

In the PMMA measurement example, 5 slices (10Hz, 5Hz, 2Hz, 1Hz, 0.5Hz) are created. The individual frequencies corresponding to the data are displayed on the screen using different types of lines.



Figure 1-13

To display certain frequency slice data, use the procedures that follow to erase the unnecessary slices.

1. Click the Display Setting icon or select Display Settings from within settings on the command line. The Display Settings window and the assigned slices are displayed. In the following example, the 10Hz, 5Hz, 2Hz, 1Hz, 0.5Hz slices are assigned.



Figure 1-14

- 2. To display the 10Hz and 1Hz, for example, do the following:
  - a. Click on the 5Hz line with the mouse.
  - b. Click the Slice Delete command line from the Edit menu.
  - c. Use the same steps to delete the 2Hz and 0.5Hz slices.
  - d. Click the OK button. The following message is displayed:
    "The data will be redisplayed using the specified properties. Analysis results will be deleted. Please confirm."
    Click the OK button.

For detailed information on functions in the display setting window, refer to the Pyris Thermal Analysis & Rheology Station Operation Manual.

# Selecting Signals & Changing Scales

Select the type of signal to assign to the analysis data X-axis, and Y axis. For original analysis data, the signal settings for each axis are the same as from the previous analysis. For completely new analysis data, the initial settings are as follows:

|         | f Slice Analysis | T Slice Analysis |
|---------|------------------|------------------|
| X Axis  | Temp.            | f                |
| Y1 Axis | E'               | E'               |
| Y2 Axis | tanδ             | tanδ             |
| Y3 Axis | E"               | E''              |
| Y4 Axis | no display       | no display       |
| Y5 Axis | no display       | no display       |
| Y6 Axis | no display       | no display       |

For example, as the default settings, the axes are sliced and displayed as follows: X Axis:Temp, Y1 Axis:E', Y2 Axis:tanδ, Y3 Axis:E''.

To change the individual signal assignments, click signal selection found in the display settings window. For detailed information, refer to the Pyris Station Operation Manual.

Each axis scale can be adjusted as desired using the slider and scroll bar or by inputting values in a text field. You can also click the Scale button located in the lower left followed by the Yaxis Auto Set button to set the appropriate scale settings for each Y axis.

### Read

Reading  $tan\delta$  peak temperature

- 1. Click the Read icon.
- 2. Click the data select button in the Read window and select Y2(tanD).
- 3. While holding down mouse button 1 (left button), drag the mouse and move the cross cursor over the tan $\delta$  data. When the pointer cursor approaches the peak of the tan $\delta$  data, the cross cursor moves to the peak position of the tan $\delta$  data. Release mouse button 1 (left button) at this point. The cross cursor position determines the indicated point.
- 4. The temperature, frequency, and  $tan\delta$  value at the cross cursor point is displayed on the window and over the data.

# S NOTE

When you want to cancel the selected point for any reason, hold down mouse button 3 in the data area and drag down the edit menu to delete and release.

- 5. To read a different frequency peak (example: to select the 1Hz slice), display 1Hz by using the slider alongside the read window slice selection window.
- 6. Next, click 1Hz once. The cross cursor on the data moves over the 1Hz tan $\delta$  data. Then perform the operations in steps 3 and 4 again.
- 7. Click the OK button to close the window.

# **Reading E' onset temperature**

- 1. Click the Calculate Onset icon.
- Select Y1(E') and set the slice selection on the frequency data you want to read (for example, 1Hz).
- 3. The cross cursor is displayed over the 1Hz E' data. Indicate the following two points with the mouse:
  - Stability point before the E' data was shutdown.
  - The point where the E' angle drops.

(The indicated method is the same as that explained in step 3 for reading tan $\delta$  peak temperature.)

4. After the two points have been indicated, draw a line over the 2 points. The temperature, frequency and E' value at the cross point of the two lines (onset point) is displayed over the data and in the window.



Figure 1-15

### **Editing Analysis Data**

Occasionally you may find it necessary to move data numerical displays or edit a data point. For whatever reason, you can use the following methods to create the most suitable data points for your measurement needs:

#### **Changing Data Character String Positions**

- 1. Click on one of the four data analysis icons (these icons display a calculator and compass) to display an analysis window. To select a data point, click on one of the data points listed in the Analysis Result area.
- 2. Hold down mouse button #3 (right button) and the Edit menu will appear. Drag the mouse down to Move Character String and release the mouse button. A single item sub menu will appear. Click on the single item sub menu with the mouse.
- 3. A square frame will appear on the data point on the line graph. Click mouse button #1 (left button) on the frame and move the character string to the desired location.

#### Editing Data Points in the Calculate Onset Window

- 1. Click on one of the four data analysis icons (these icons display a calculator and compass) to display an analysis window. To select a data point, click on one of the data points listed in the Analysis Result area.
- 2. Hold down mouse button #3 (right button) and the Edit menu will appear. Drag the mouse down to Adjust Results and release the mouse button. A sub menu will appear. Align the mouse on the sub menu and click mouse button #1 (left button).
- 3. A cross cursor will appear on the data point on the line graph. Click mouse button #1 (left button) on the center of the cross cursor and move the data point to the desired location.

# **Correcting (Smoothing) Measurement Data**

Data can be corrected when there is a substantial amount of noise.

# S NOTE

When smoothing is performed all edited analysis results will be lost. Perform smoothing before starting analysis.

#### **Smoothing Correction**

- 1. Click on the Smoothing Correction icon.
- 2. Using the correction switch for the signal are where you want to perform smoothing, select whether to use a linear scale or a log scale.
- 3. Input the number of smoothing points for each signal and press return.
- 4. The smoothed data will then appear. Click OK to confirm the new data or Cancel to cancel the smoothed data.

| rest. | S        | moothi | ing    |                 | •   |
|-------|----------|--------|--------|-----------------|-----|
| Numbe | r of sma | othing | points | Correction      |     |
| Freq. | I        | 1      | units  | Linear Mode     | •   |
| Temp. | Ĭ        | 1      | units  | Linear Hode     |     |
| E'    | Ĭ.       | 1      | units  | Linear Hode     |     |
| E"    | 1        | 1      | units  | Linear Mode     |     |
| dL    | Ĭ        | 1      | units  | Linear Mode     | D   |
| tanD  | I        | 1      | units  | Linear Mode     |     |
| F     | Ĭ.       | 1      | units  | Linear Mode     |     |
| Time  | Ĭ        | 1      | units  | Logarithmic Hod | e 🗆 |
|       |          |        | _      |                 |     |
| ОК    |          |        |        | Cancel          |     |
| L     |          | ,      | L      |                 |     |

Figure 1-16

### **Making Data Printouts**

- 1. Click on the Plot icon and the following window will open.
- 2. Set all of the appropriate plot conditions.

| - | . Plot                                   | · [                           |  |  |
|---|--|-------------------------------|--|--|
|   | Plot Data Information 🛛 🔲 🗆              | Paper Size 🛛 🗛 📼              |  |  |
|   | Number of comments to be plotted         | Width 270 mm                  |  |  |
|   | 100 Characters 18 Lines                  | Height [185 💵                 |  |  |
|   | Axis Plot Yes 🗆                          | Plotter Start Position Offset |  |  |
|   | Y Axes to be Plotted Y1 to Y4 🗆          | x []0                         |  |  |
|   | Tick Value Point Size Standard 🗆         | Y []0 mm                      |  |  |
|   | Organization Name Seiko Instruments Inc. | Paper Feed Yes 🗆              |  |  |
|   | Plot Curve Yes 🗔                         |                               |  |  |
|   | Plot Analysis Results Yes 🗆              | Other Plotter Settings        |  |  |
|   |  |                               |  |  |
|   |  |                               |  |  |
|   | OK Plot                                  | Cancel                        |  |  |

Figure 1-17

3. When you have finished setting the conditions, click the OK button to output the data on the plotter. The following diagram shows an example of a data plot.



Figure 1-18

### **Saving Data**

Saving a File Under a Different Name

1. Click on the Save File As icon and the following window will open.

|  | · [   |
|--|---|
| Save Version As Latest Version(Version 1) 🗆            |   |
| Filter   |   |
| /home/seiko/DMSdemodata/%                              |   |
| Directory  | Files   |
| //home/seiko/DHSdemodata/.<br>/home/seiko/DHSdemodata/ | DHS-OHP Z<br>DHS-PHHA<br>DHS-PP<br>Nylon6<br>Nylon6-A<br>PHHA13f-t<br>PHHA13f-t<br>PHHA13f-tE<br>PHHA13fR<br>PHHA13fR<br>PHHA920128.1-1<br>PHHA920128.1-1 |
| Select   | 8   |
| /home/seiko/DMSdemodata/DMS-PMMA                       |   |
| OK   | Cancel  |

Figure 1-19

2. Enter a new file name and click the OK button.



#### **Updating a File**

1. Select Save from the File menu.



When save is selected from the file menu the file will be automatically saved. There will be **NO** messages displayed on the screen informing you the file has been saved.

### Helpful hints . . .

#### Changing the X-axis to temperature units

- 1. Click on the the Display Settings icon.
- 2. Select X(Time min) from the Select Signal menu.
- 3. Select Temp. C from the Select Signal area and click the OK button.
- 4. Click the Display Settings window OK button.
- 5. A message box will appear asking if it is OK to redisplay the data. Click the OK button.
- 6. The data will convert to X-axis temperature data.

#### Displaying the temperature in Kelvin units

- 1. Click on the Display Settings icon.
- 2. Select Set Signal Properties from the Select Signal menu.
- 3. The Set Signal Properties window will open.
- 4. Click on Temp in the Signal Selection area and make the following changes.



Figure 1-20

- 5. Click on the OK button to close the Set Signal Properties window.
- 6. The units for the temperature axis will be converted to Kelvin, however the data will not be displayed in an appropriate scale. Select Change to Maximum Value from the Scale pop up menu button located in the bottom left hand corner of the window. Finally, select Auto Y-Axis Adjustment from the same menu.

#### Cancelling the Display of Only Specific Slices and Signals

- 1. Cancelling the display of only specific slices
  - a. Open the Display Settings window.
  - b. Double click the slice you do not want to display.
  - c. The Change Slice Display Properties window appears. Turn OFF the switch of the particular Yaxis (for example, Y1 `Y3).
  - d. Click the OK button. Click the OK button on the display settings window.
  - e. A message window is displayed. Click the OK button.
- Cancelling the display of only specific signals
   a. Open the Display Settings window.
  - b. Click Display Properties from the command menu.
  - c. To erase the display of only the Y2axis (tanD), click Y2 Axis for All Slices and click Hide.
  - d. Click the OK button. Click the OK button on the display settings window.
  - e. A message window is displayed. Click the OK button.

# 1.6 Analyzing Measurement Data (F, L Control Modes)

Data measured using the DMA Module L Control or F Control modes are saved as TMA data. Use the TMA/SS Standard Analysis Software to perform analysis of the data.

### **Opening the TMASS Analysis Window**

- 1. Double click on the TMASS\_Analysis icon found in the PKI main menu window.
- 2. The TMASS Analysis window will open. Analysis data has not been selected at this point so all items in the menu bar will be deactivated except for Open.
- 3. Select Open from the menu bar and an Open File window will open.

4. Select the target file you want to analyze from the file list (for example: /users/PKI/data\_tma.00) and click the OK button.

# S NOTE

When a file containing analysis data is selected with the mouse a data profile will be displayed in the panel located at the right of the file list.

5. The data will be recalled from the file and displayed in the window. The data is now ready to analyze.

### **Read Point**

- 1. Click the Read Point icon.
- 2. Select the desired signal in Select Data.
- 3. Read the signal using one of the following methods:
  - a. Drag the cross-line cursor on the graph data to the desired position.
  - b. Use the Cursor pull-down menu in the Read Point window or the click mouse button #3 with the cursor in the graph data and select the cursor movement method. Move the cursor to the desired location and click Set Point(s) in the same menu.

For an explanation of other types of analysis, refer to the Pyris Thermal Analysis & Rheology Operation Manual.

### **Editing Analysis Data**

Occasionally you may find it necessary to move data numerical displays or edit a data point. For whatever reason, you can use the following methods to create the most suitable data points for your measurement needs:

#### **Changing Data Character String Positions**

- Click on one of the four data analysis icons (these icons display a calculator and compass) to display an analysis window. To select a data point, click on one of the data points listed in the Analysis Result area.
- 2. Hold down mouse button #3 (right button) and the Edit menu will appear. Drag the mouse down to Move Character String and release the mouse button. A single item sub menu will appear. Click on the single item sub menu with the mouse.
- 3. A square frame will appear on the data point on the line graph. Click mouse button #1 (left button) on the frame and move the character string to the desired location.

### **Other Corrections**

# S NOTE

Analysis results are erased once data correction is performed. Be sure to perform data correction before performing analysis.

#### **Smoothing Correction**

- 1. Click on the Smoothing icon.
- 2. Input the number of smoothing points for each signal.
- 3. Click on OK to smooth the data.

#### Length Normalization

- 1. Click on the Normalization icon. Their are two types of Normalization items: length and weight.
- 2. Enter the Normalize Weight or Normalize Length into the corresponding field.
- 3. The scale value will change and the peak size will change according to the normalization correction. Click on OK to confirm or Cancel to cancel this procedure.

### **Making Data Printouts**

- 1. Click on the Plot icon and the following window will open.
- 2. Set all of the appropriate plot conditions.



Figure 1-21



3. When you have finished setting the conditions, click the Plot button to output the data on the plotter or printer. The following diagram shows an example of a data plot.



### **Saving Data**

Saving a File Under a Different Name

1. Click on the Save File As icon and the following window will open.



Figure 1-23

2. Enter a new file name and click the OK button.



If you click the OK button without changing the file name a message window will appear informing you that the old file will be replaced with the current file. If OK is pressed again the file will be replaced. Be sure to change the file name if you do not want to lose the original data.

#### Saving a New File

1. Select Save from the File menu.



When save is selected from the file menu the file will be automatically saved. There will be **NO** messages displayed on the screen informing you the file has been saved.Setting the Sample & Measuring the Sample Length

### **Ending Measurements and Analysis**

#### **Closing the Measurement Window**

- 1. Click on File in the measurement window menu bar and select Close from the menu.
- 2. The following message will appear:



Figure 1-24

3. Click on the OK button to end.

#### **Closing the Analysis Window**

- 1. Click on File in the measurement window menu bar and select Close from the menu.
- 2. If you have saved your analysis data the window will close and analysis will end. If you have not saved your data the following message will appear.



Figure 1-25

3. Click on the OK button to end analysis. To save your analysis data click on the Cancel button, save the data, and return to step one above.

# Chapter 2 DMA Advanced Software Features

| • | Automatic Cooling Feature          | 2-2 |
|---|------------------------------------|-----|
| • | Protect Feature                    | 2-4 |
| • | Temperature Precalibration Feature | 2-5 |

# 2.1 Automatic Cooling Feature

Function: Reduce the temperature of the furnace to the desired level using compressed air (when measurements are completed).

Options: Auto cooling unit

#### **Operating Procedure**

1. Connect the auto cooling unit as shown in Figure 2-1 and start the air flow.



2. Open the Set Sample Conditions window and input the cooldown limit temperature into the Air Cooldown Temperature numerical field.



Figure 2-2

3. Perform normal measurement operations. When measurements are completed the module control will be put on READY and the auto cooling unit's magnetic valve will open, the air flow will begin, and the furnace will be cooled. The cooling unit will shut down when the sample temperature reaches the cooldown limit.

# 2.2 Protect Feature

| Function: | Prevent sample meltdown on the measuring head or probe. |
|-----------|---|
| Module:   | DMA module  |
| Options:  | None<br>Operating Procedure                             |

#### **Operating Procedure**

- 1. Click on the Set Sample Conditions icon.
- 2. Input a numerical (Pa) value for a E' lower limit value into the Fusion Protection numerical input field.

### **Activating the Protect Feature**

1. When the E' value is lower than the set value during measurement, the sample is determined to be softened and the measurement is forceably stopped.

# 2.3 Temperature Precalibration Feature

Function: Keep the difference between the program temperature and sample temperature at a minimum during measurement. This feature studies the relationship between both temperatures and corrects and controls the program temperature based on this relationship.

It is normally not necessary to use this feature when operating a DMA module.

- Module: All modules
- Options: None

#### **Operating Procedure**

Temperature calibration

- 1. Click on the Set Sample Conditions icon.
- 2. Click on the Temperature Control Mode pop-up button and select Precalibrated Temperature Control Mode.
- 3. Click on the Temperature Program Settings button to display the temperature program window.
- 4. Set the following temperature program.

(The following temperature program is an example of a temperature program with a temperature range from above room temperature to  $300^{\circ}$ C)

| End step $= 9$ |       |       |      |      |  |  |
|----------------|-------|-------|------|------|--|--|
| Step           | Start | Limit | Rate | Hold |  |  |
| 1              | 30    | 60    | 2    | 0    |  |  |
| 2              | 60    | 90    | 2    | 0    |  |  |
| 3              | 90    | 120   | 2    | 0    |  |  |
| 4              | 120   | 150   | 2    | 0    |  |  |
| 5              | 150   | 180   | 2    | 0    |  |  |
| 6              | 180   | 210   | 2    | 0    |  |  |
| 7              | 210   | 240   | 2    | 0    |  |  |
| 8              | 240   | 270   | 2    | 0    |  |  |
| 9              | 270   | 300   | 2    | 0    |  |  |
|                |       |       |      |      |  |  |

#### **Example of a Temperature Program**

# S NOTE

- 1. When the temperature precalibration feature is performed, the loss of temperature control may be immense for the temperature range outside of the the temperature precalibration.
- 2. When performing minus temperature range measurements, set the temperature program for the minus temperature.
- 3. An isothermal program with 7 steps or more is necessary. If the program does not have 7 steps, the precailbration results are not recorded.
- 4. A TEST cannot be performed during the Precalibration Temperature Control mode.
- 5. Perform measurements.
#### Measuring after calibration

1. Open the Sample Conditions setting window. Click on the Temperature Control Mode pop-up menu and select Normal Temperature Control mode. The temperature will now be controlled based on the precalibrated temperature.

### S NOTE

- 1. Make sure that the control mode is returned to Normal Temperature Control after the precalibration measurement is performed. If the temperature control mode is left on Precalibrated Control the calibration will not be activated and the results will be inaccurate. In other words, the precalibrated mode will remain "ON" when measurements are repeated.
- 2. If temperature precalibration is not performed correctly, the temperature control of the measurement following the precalibration is improper and the temperature program does not heat and cool as set. When this occurs, either perform precalibration again appropriately or use the following method clear the precalibration results using the following method and perform the measurement.
  - a. Click DMS ABC setting icon, and save the necessary ABC settings in a file.
  - b. Remove the back-up batteries and turn off the power to the module.
  - c. Insert the battery and connect the module to the station.
  - d. Enter the ABC settings from the file saved in step a.
- 3. When samples are to be measured under gas flows, set the precalibrated temperature conditions to match the actual sample measurement conditions in order to reduce errors. The temperature program data save feature can be set to either On or Off.

# **Chapter 3 System Calibration**

| PID Temperature Control Constants   | 3-4  |
|---|------|
| Sample Temperature Correction   | 3-5  |
| Dynamic Viscoelastic Measurement Calibration                                    | 3-9  |
| <ul> <li>Static Measurement Mode Load (F)<br/>Sensitivity Correction</li> </ul> | 3-20 |
| • Adjusting the Strain Signal Output<br>(Internal Micrometer Adjustment)        | 3-22 |

The DMS ABC Setting window is used to set the constants used for system calibration and control. We advise that you calibrate your system on a regular basis to maintain peak performance of your equipment.

The following calibrations are performed from the DMS ABC Setting window:

1. Temperature Control PID Constants Settings

The following three constants are settings for the furnace temperature control:

P=Proportional constant

I=Integral constant

D=Derivative constant

2. Sample Temperature Calibration

Compares the sample temperature with a known (literature) sample temperature and corrects the offset.

3. Viscoelastic Measurement System Calibration

Correction of the compliance, the inertia of probe mass, the viscoelasticity of the probe supporting system. Calibration of the elastic modulus from a measurement of a sample known elastic modulus.

## S NOTE

Above corrections 2 and 3 each utilize a calculation window that can be accessed from the Calibration window. Corrections are done automatically by the computer after necessary information has been input into the calculation window.

| DMS ABC Setting -                        | ch2.rheo_dms_ca   |     |
|--|-------------------|-----|
| ile                                      |                   |     |
|  | 1.                | 1   |
| lemperature Control<br>P Constant        | 1 2               |     |
| I Constant                               | 1 500             |     |
| D Constant                               | 1 1               | ]   |
|  |                   |     |
| Temperature Correction<br>Zero Order (C) | 1 -2              |     |
| First Order<br>(C/C)                     | 1 1               |     |
| Second Order<br>(C/C2)                   | I O               | ]   |
| Third Order<br>(C/C3)                    | I O               | 1   |
|  |                   |     |
| F/L gain                                 | 1.06322           |     |
| V cal<br>(N.sec/m)                       | 1.74369           |     |
| P mass<br>(g)                            | 81.4427           | ]   |
| E cal<br>(N/m)                           | 2870.7            | ][] |
| Compliance Correction<br>(um/g)          | i 0.001           | ]]  |
| t const<br>(sec)                         | 0.000132          | ] ] |
| TensionGain                              | 0.795802          | ]   |
| Load Span<br>(g)                         | 1200 <u>1</u> 200 | ] ] |
|  |                   |     |
| ОК                                       | Cancel            |     |

Figure 3-1



## **3.1 PID Temperature Control Constants**

The most suitable PID default values have been input for each analysis module. The PID default values are saved even when the module power and battery power is turned off. Normally, you will not need to change these values.

|              |   | Pyris Diamond DMA | Input Range  |
|--------------|---|-------------------|--------------|
| Temp Control | Р | 2                 | 0.001≤P≤1000 |
|              | Ι | 500               | 10≤I ≤1000   |
|              | D | 1                 | 0≤D≤1000     |

S NOTE

What effect does the PID have on measurement results?

- P The larger the P value the more effective the results.
- I The larger the I value the less effective the results.
- D The larger the D value the more effective the results.

### **3.2 Sample Temperature Correction**

#### **Sample Temperature Correction Purpose & Cautions**

The purpose of the sample temperature correction is to correct the temperature dispersion between the temperature signals output from the temperature of the sample and to make the temperature signal output close to the actual temperature of the sample.

With the DMA instrument, the output of the thermocouple installed near the sample is detected using the electrical circuitry and output as a sample temperature signal. The following 2 reasons can cause the dispersion between the output temperature signal and the actual sample temperature:

- 1. Fluctuations of the thermocouple (including measurement circuitry) output.
- 2. Temperature dispersion of the thermocouple tip area (physics type) and the sample.

Reason 1 can be corrected by output correction through the hardware of the thermocouple circutiry at the time the product is shipped.

This section describes the use of the correction feature for signal output using the software and how to correct the dispersion caused over time for reason 1 above and how to correct the dispersion of reason 2 as well. The correction constants determined from the results of the correction are recorded in the DMA module circuitry. The correction constants can also be saved as a file on the computer.

### S NOTE

The degree of the temperature dispersion caused by reason 2 above is affected by the following.

- Deformation Mode (Structure Around the Sample)
- Heating Rate

Furthermore, the value of the correction constant displaying the correction amount fluctuates based on the measurement conditions. Generally, the correction effectiveness of the drift caused by correction increases the closer the actual measurement conditions are to the corrected measurement conditions.

Because of this, it is recommended that the deformation mode and heating rate are compiled together, made into a file and saved as correction constants. When performing measurements, match the measurement conditions with the optimal correction constant file saved in the circuitry of the DMA module to lessen the fluctuations in the sample temperature signal. In the correction steps, first measure the transition temperature of standard sample, and set the transition temperature on the literature value. The following is an explanation of correction using  $2^{\circ}$ C/min conditions for a tension measurement and  $2^{\circ}$ C/min conditions for a bending measurement. When the measurement conditions vary from this, align with the measurement conditions and perform a correction measurement and create a correction constant file to use. When the instrument is shipped, sample temperature correction has been performed using a tension measurement with a  $2^{\circ}$ C/min condition.

#### **Correction Procedure**

Follow the procedure below (heating rate at  $2^{\circ}$ C/min.) to calibrate the sample temperature signal using the E" peak-top temperature (117°C at 1 Hz) which corresponds to the PMMA glass transition. Either bend or tension measurements can be used for the measurement. 1. Open the DMS ABC Setting window and record the temperature calibration 0 order value

2. Measure the PMMA sample using the following conditions:

#### For Bend Measurements

Sample : PMMA Approximately 1.5mm (thickness) x 10mm (width) x 50mm (length) Sample Conditions

| Length                   | 20mm                         |
|--------------------------|------------------------------|
| Cross-section            | Rectangular cross-section    |
| Width                    | Measured Value (about 10mm)  |
| Thickness                | Measured Value (about 1.5mm) |
| Temperature Control Mode | Ramp                         |
| Temperature Program      |                              |
| Sampling                 | 1sec                         |
| End Step                 | 1                            |
| Start                    | 30°C                         |
| Limit                    | 150°C                        |
| Heating Rate             | 2°C /min                     |
| Hold Time                | Omin                         |
| Frequency                | Only 1Hz ON                  |
| Control Mode             | Bend/Sinusoidal Oscillation  |

#### **For Tension Measurements**

Sample : PMMA Approximately 0.2mm (thickness) x 8mm (width) x 20mm (length) Sample Conditions

| Length                   | 20mm                         |
|--------------------------|------------------------------|
| Cross-section            | Rectangular cross-section    |
| Width                    | Measured Value (about 8mm)   |
| Thickness                | Measured Value (about 0.2mm) |
| Temperature Control Mode | Ramp                         |
| Temperature Program      |                              |
| Sampling                 | 1sec                         |
| End Step                 | 1                            |
| Start                    | 30°C                         |
|                          |                              |

| Limit                          | 150°C                          |
|--------------------------------|--------------------------------|
| Heating Rate                   | 2°C/min                        |
| Hold Time                      | Omin                           |
| Frequency                      | Only 1Hz ON                    |
| Control Mode                   | Tension/Sinusoidal Oscillation |
| Position and Tension Settings  |                                |
| L. Amp.                        | 10µm                           |
| Min. Tension/Compression Force | 300mN                          |
| Tension/Compression Force gain | 1.5                            |
| Force Amplitude Initial Value  | 4000mN                         |
| Approved Maximum Deformation   | 10000µm                        |
| Position Movement Wait Time    | 8.0sec                         |
| Creep Wait Time                | 0.0sec/mN                      |

- 3. After measuring, the peak temperature is read from E" data.
- 4. Calculate the new value to be entered as the temperature calibration 0 order using the following formula:

Value to be entered as 0 order value = a + 117 - b where,

- a = the 0 order value recorded in 1, and
- b = the E' peak temperature determined in 3.
- 5. Enter the value obtained in item 4 as the DMA ABC Setting window temperature calibration 0 order value.

### S NOTE

- 1. Be sure that values other than the temperature calibration do not change.
- 2. The results of calibration improve the more the calibration conditions resemble those under which actual measurement is performed. The conditions below are major factors when calibrating the sample temperature.
  - Heat conductivity of the calibration sample.
  - Heating rate.

When you need to measure samples with high conductivity, use the melting point for metal indium  $(156.6^{\circ}C)$  etc..

3. The temperature correction described above is performed using a heating process. On occasion, the displayed room temperature may be off somewhat. This is due to to the difference in heating rate (0°C/min. at room temperature) and does not indicate any abnormality in the function of the instrument.

## 3.3 Dynamic Viscoelastic Measurement Calibration

#### **Calibration Items**

The following five items are involved in viscoelastic measurement system calibration:

- 1. F/L Gain
   Stress and strain sensitivity ratio correction.

   2. With the Gamma is a strain sensitivity ratio correction.
- 2. Viscosity Correction Probe support system viscosity correction.
- 3. Probe Mass Inertia correction based on probe mass.
- 4. Elasticity Correction Probe support system elasticity correction.
- 5. Compliance Correction Measurement system compliance correction.
- 6. Time Constant Detection time constant correction.
- 7. Tension Gain Elastic sensitivity corrrection for tension and compression measurements.

#### **Calibration Method**

• To calibrate the system from the beginning, follow the series of instructions explained in this section from A to G.

The order of the operations is extremely important.

• Normally, periodic calibrations, it is sufficient to perform the steps F and G.

| Cal | ibration step  | Measurement<br>Attachment | Measurement Mode                   | Standard Sample                  |
|-----|--|---------------------------|------------------------------------|----------------------------------|
| А.  | Compliance Correction  | Bending                   | Shear<br>/Sinusoidal Oscillation   | Stainless plate<br>(w12,t3,l20)  |
| B.  | Time Constant  | Bending                   | Shear<br>/Sinusoidal Oscillation   | Steel plate<br>(w10,t0.4,l20)    |
| C.  | Viscosity Correction   | Bending                   | Shear<br>/Sinusoidal Oscillation   | None                             |
| D.  | Probe Mass   | Bending                   | Shear<br>/Sinusoidal Oscillation   | None                             |
| E.  | Elasticity Correction  | Bending                   | Shear<br>/Sinusoidal Oscillation   | None                             |
| F.  | F/L Gain,<br>Viscosity Correction,<br>Probe Mass,<br>Elasticity Correction | Bending                   | Bending<br>/Sinusoidal Oscillation | Steel plate<br>(w10,t0.4, 1 20)  |
| G.  | Tension Gain   | Tension                   | Tension<br>/Sinusoidal Oscillation | Aluminum plate<br>(w3,t0.1, 120) |

#### Preparation

- 1. Attach the Bend Measurement Attachment to the sample unit. For instruction refer to Section 5.1 Exchangin Sample Unit Attachments in the Pyris Diamond DMA Module Operation Manual.
- 2. Click the Motor Movement icon. Set the Target Pos. to -16000 ( $\mu m)$  and click the Move button.
- 3. Open the DMS ABC Setting window. Save the current individual values as a file using Save As.... in the File menu.
- 4. Manual input the following values and click OK.

| F/L Gain              | : | 1 |
|-----------------------|---|---|
| Viscosity Correction  | : | 0 |
| Probe Mass            | : | 0 |
| Elasticity Correction | : | 0 |
| Compliance Correction | : | 0 |
| Time Constant         | : | 0 |
| Tension Gain          | : | 1 |

5. If you are using the cooling controller, turn off the power.

#### **A.** Compliance Correction

- 1. Set a stainless plate sample with a 12mm (width) 3mm (thickness).
- Open the Condition setting window. Input the following settings and click OK. (Even though you are using the bend measurement attachment, select shear as the control mode)

| Control Mode              | Shear/Sinusoidal Oscillation                  |
|---------------------------|---|
| Sample                    |   |
| Length                    | 0.01mm  |
| Rectangular cross-section |   |
| Width                     | 1mm   |
| Thickness                 | 5.1mm   |
| Data Filename             | Enter an appropriate filename (ex. dmscal.00) |
| Temperature Control Mode  | Step  |
| Temperature Program       |   |
| Soak                      | 2 (sec)                                       |
| Start Temperature         | 0 (°C)  |
| Limit Temperature         | 10 (°C)                                       |
| Divisor                   | 20  |
| Frequency                 | Only 1Hz ON                                   |
| L. Amp                    | 20(µm)  |
|                           |   |

3. Click TEST to perform a test measurement. The measurement results are displayed in the message window on the screen. Check the information then click the OK button.

### S NOTE

The TEST mode measurement is used to to initialize the DMA control system.

- 4. Click the RUN button and perform a measurement.
- 5. Once the measurement is complete, open the measured data using f slice analysis (ex. dmscal.00). Set the X axis to time and calculate the average value of G'.

## S NOTE

After using the f slice analysis Display Settings, Signal Selection and Signal Properties Setting windows to set the signal selection to G'Pa, the A (gain) and B (offset) functions in the bottom left of the window should be 1, 0 respectively. If the values are different, enter A=1 and B=0 and click the OK. Next, click OK on the Display Settings window.

- 6. Open the DMS ABC Setting window. Click the Compliance correction calculation button. Input the elastic modulus value (M') from the message window into the Actual Measured value E' area in the automatic correction calculation window and click OK.
- 7. The compliance correction calculated value is input. If the value is correct it will be in the  $10^{-4} \sim 10^{-3}$  order. Click OK.

## **NOTE**

Be sure to click the OK button one time and close the window. If the window is left open, the correction value will not be activated.

#### **B.** Time Constant

- 1. Remove the stainless plate and chuck a 10mm width, 0.4mm thickness steel plate.
- 2. Open the Frquency Settings window, turn on the 100Hz button and click OK.
- 3. Select display type 2 (time), turn off the tanD Log Display and click the OK button.
- 4. Click TEST to perform a test measurement. The measurement results are displayed in the message window on the screen. Check the information then click the OK button.
- 5. Click Run and begin the measurement. Magnify the tan $\delta$  scale and read the tan $\delta$  measurement value.
- 6. Open the DMS ABC Setting window and click the time constant calculation. Input the following and click the OK button.

| Actual Measured tanD | : | tanδ read in step 5 |
|----------------------|---|---------------------|
| Offset               | : | 0.005               |
| Measurement Freq.    | : | 100 (Hz)            |

- 7. The calculation results are entered into the time constant. If the calculations is normal, the calculation value should be in the  $10^{-4}$  order. Click the OK button.
- 8. After clicking OK, make sure that the tan $\delta$  value of the measured data has dropped to around 0.005.
- 9. Click the STOP button and stop the measurement.

## S NOTE

If the measurement stops during steps  $5 \sim 8$ , click the RUN button again.

#### **C. Viscosity Correction**

- 1. Open the Frequency Settings window, click ON the 20Hz frequency only and click the OK button.
- 2. Remove the steel plate to create a blank status (leave chuck attached).
- 3. Click the Motor Move icon, enter 10mN for the Force Amplitude Initial Value. Enter values around 700mN for the Offset Load and adjust the value so that the L (strain signal) value is within 0.5μm.

### S NOTE

- 1. The Offset Load value is effective when the instrument is first operated after the value has been entered and the OK button clicked.
- 2. When the Offset Load is changed by 10mN, the L (strain signal) changes from -3 to -4 $\mu$ m.
- 4. Click Test and perform a test measurement. Make sure the elastic modulus value is displayed in the message panel and click the OK button
- 5. Click the Run button and enlarge the tan $\delta$  scale to 0 ~ 0.4 and read an approximate tan $\delta$  measurement value.
- 6. Open the DMS ABC Setting window. Enter a value of 0.5 ~ 2 in the V cal area and click the OK button.
- 7. Repeat step 6 until the tan $\delta$  data is in a range of about 0.002 ~ 0.02.

## S NOTE

With the sample absent, the signal is not stable due to  $\tan \delta = E''/E' = 0/0$ . There is no problem with the instrument in this case. Once the measurements in steps 6 through 7 are completed, click Run again.

#### **D.** Probe Mass

- 1. Click the STOP button and stop the measurement.
- 2. Click the Frequency Setting icon and turn ON frequency 1Hz, 10Hz, 20Hz. Click the OK button.
- 3. Open the Display Setting window, turn ON display type 3 (frequency) and click the OK button.

| S NOTE                    |        |   |  |
|---------------------------|--------|---|--|
| Check to make sure        | the f  | ollowing settings are entered in the Conditions |  |
| setting window. A         | fter c | hecking the settings, click the OK button.      |  |
| Length                    | :      | 0.01(mm)  |  |
| Rectangular Cross Section |        |   |  |
| Width                     | :      | 1 (mm)  |  |
| Thickness                 | :      | 5.1 (mm <sup>2</sup> )                          |  |

- 4. Click TEST to perform a test measurement. The measurement results are displayed in the message window on the screen. Check the information then click the OK button.
- 5. Click Run and begin the measurement. Magnify the G' scale.
- 6. Open the DMS ABC Setting window. Enter values of 50 ~ 100 in the P mass and click the OK button.
- 7. Change and adjust the probe mass values in step 6 until the E' signal is flat. If the operation works correctly, the appropriate value should be 50 ~ 100.

## S NOTE

If the measurement stops during steps  $6 \sim 7$ , click the RUN button again.

#### **E. Elasticity Correction**

- 1. Click the STOP button and stop the measurement.
- 2. Open the measured data using f Slice Analysis and read the 1Hz value of E' (1000 ~3000).
- 3. Open the DMS ABC Setting window. Enter the value from step 2 into elastic correction input field and click the OK button.

## S NOTE

The measurement value in step 2 is a blank measurement and is therefore relatively unstable. This is not an abnormality.

#### F. F/L Gain, Viscosity Correction, Probe Mass, Elasticity Correction

- 1. Perform chucking again on a chuck a 10mm width, 0.4mm thickness steel plate.
- 2. Click the Motor Movement icon, enter 2000mN for the Force Amplitude Initial Value then click the OK button.
- 3. Open the Condition setting window. Input the following settings and click OK. Sample

| Length                    | 20mm                        |
|---------------------------|-----------------------------|
| Rectangular cross-section |                             |
| Width                     | 10mm                        |
| Thickness                 | 0.4mm                       |
| Frequency                 | Only 1Hz ON                 |
| Control Mode              | Bend/Sinusoidal Oscillation |

## S NOTE

Measure the width and thickness of the stainless board and enter the actual measured values.

- 4. Click Test and perform a test measurement. Make sure the elastic modulus value is displayed in the message panel and click the OK button
- 5. Click the Run button and perform a measurement.
- 6. Once the measurement is complete, open the measured data using f slice analysis (ex. dmscal.00). Set the X axis to time and calculate the average value of E'.
- 7. Open the DMS ABC Setting window. Click the F/L Gain calculation button.

| 8. | Enter the following val | ues | s into the Auto Calculation window.                     |
|----|-------------------------|-----|---|
|    | Literature Value E'     | :   | 1.92E + 11  |
|    | Actual Measured E'      | :   | The average elasticity (E') value calculated in step 6. |
|    | Constant Changes        | :   | F/L Gain, Vcal, Pmass and Ecal All ON.                  |

 Click the OK button in the Auto Calculation window. The F/L Gain, Viscosity Correction, Probe Mass and Elasticity Correction values are chaned to the calculated results. Click the OK button.

#### **G. Tension Gain**

- 1. Attach the Tension Measurement Attachment to the sample unit. For instruction refer to Section 5.1 Exchangin Sample Unit Attachments in the Pyris Diamond DMA Module Operation Manual.
- 2. Open the Position Settings/Tension Conditions setting window and click the Initialize button. Once initialization is complete, click the OK button and close the window.
- 3. Perform chucking again on a chuck a 3mm width, 0.1mm thickness Alminum sample.
- 4. Open the Sample Setting window. Input the following settings and click the OK button. Sample Shape

| inple Shape                    |             |  |  |
|--------------------------------|-------------|--|--|
| Length                         | 20 (mm)     |  |  |
| Rectangular cross section      |             |  |  |
| Width                          | 3 (mm)      |  |  |
| Thickness                      | 0.1 (mm)    |  |  |
| Frequency Setting              | Only 1Hz ON |  |  |
| Control Mode                   | Tension     |  |  |
| Position and Tension Settings  |             |  |  |
| Min. Tension/Compression Force | 300mN       |  |  |
| Tension/Compression Force Gain | 1.5         |  |  |
| Force Amplitude Initial Value  | 4000mN      |  |  |

### S NOTE

Correctly observe the aluminum sample width and thickness and enter the actual measured value.

- 5. Click the TEST button to perform a test measurement. Leave the result message window open.
- 6. Click Run and begin the measurement.
- 7. Once the measurement is completed, open the measurement data using f Slice Analysis. Set the X axis to time and read the E' value. Calculate an average E' value.
- 8. Open the DMA ABC Setting window and click the Tension Gain calculation button.

9. Enter the following into the Auto Calculation window:

Literature E' Value : 6.91E + 10Actual Measured E' : The E' average value from step 7.

10.Click the OK button in the Auto Calculation window. The Tension Gain value is automatically updated (the calculation value becomes 0.8 ~ 1.2).

## **NOTE**

The Tension Gain value is recalculated and updated each time the OK button is clicked in the Auto Calculation window. To return to the previous calculation click the Cancel button.

11.Click the RUN button again and perform a measurement. Make sure that the elasticity of the aluminum is output correctly. This completes all calibrations.

## S NOTE

Normally, for periodic calibrations, it is sufficient to perform the steps in D. F/L Gain, Viscosity Correction, Probe Mass, Elasticity Correction with the Bend Measurement Attachment attached and then perform G. Tension Gain with the Tension Meausrement Attachment attached. These operations will complete system calibrations.

## 3.4 Static Measurement Mode Load (F) Sensitivity Correction

Perform the following operations to correct the sensitivity of the static measurement mode (F Control or L Control) load output.Weigh a known scale weight in the L Control Mode.

#### **Load Span Correction**

The load sensitivity will be corrected by inputting the constant "S". This constant is calculated using the following formula.

$$S = S' x \frac{M_0}{M} - - - (1)$$

S' : Load span value used when weighing

M<sub>0</sub>: True scale weight value [g]

M : Weighed value of scale weight [g]

Follow the steps below to automatically calculate the Load Span correction values.

- 1. In the Sample Settings window, select L Control Mode as the measurement mode and click the OK button to close the window.
- 2. Click the DMS ABC Setting icon and open the window. Enter 11768 for the Load Span.
- 3. Click the Motor Move icon and open the window. Enter 700mN for the Offset Load and adjust the value until the L (strain signal) is within  $0.5\mu$ m.

### S NOTE

- 1. The Offset Load value is effective when the instrument is first operated after the value has been entered and the OK button clicked.
- 2. When the Offset Load is changed to 10mN, the L (strain signal) changes from -3 to -4 $\mu$ m.
- 4. Use a piece of string or a fune clip and hang a 50g weight from the probe as shown below.

5. Create a program that holds the SS Program at  $0\mu$ m and holds the temperature program at  $0^{\circ}$ C.

| 55 Program Example | SS | Program | Example |
|--------------------|----|---------|---------|
|--------------------|----|---------|---------|

| End Step          | : | 1                                  |
|-------------------|---|------------------------------------|
| Start Position    | : | 0μm                                |
| Limit Position    | : | 0μm                                |
| Displacement Rate | : | 100µm/min (anything is acceptable) |
| Hold Time         | : | 10min                              |

- 6. Click the RUN button and begin the measurement.
- 7. After a short time, the L (strain signal) will stabilize at  $0\mu m$ . Read the F output value when this occurs.
- 8. Open the right Load Span Auto Calculation window in the DMS ABC Settings window.
- 9. Enter 490.333mN in the Literature Value and for the Actual Value enter the value from step 6 and click the OK button. The Load Span is automatically calculated and entered.

### ( Information

The gravitation applied to a 50g weight using the standard acceleration of free fall (9.80665ms-2) computes as follows: 0.05kg  $\times$  9.80665ms-2 = 490.333mN.

## **3.5 Adjusting the Strain Signal Output** (Internal Micrometer Adjustment)

If one of the following occurs, there is probably a problem in the adjustment of the micrometer within the system.

- a. With the sample removed, the strain signal is out of the  $\pm 30\mu$ m range even though the offset load value in the Motor Setting window has not been changed to a great extent.
- b. With the sample removed, the strain signal value is out of the  $\pm 50\mu$ m range when 700mN is entered for the offset load value in the Motor Setting window.

## S NOTE

In the following case, even if the symptoms above occur, there is not a need to adjust the micrometer. First check the items below before performing adjustments.

- The sample is attached. Remove the sample. With only the chuck attached, check the other items.
- The battery voltage is low or the batteries have just been replaced. In this case, check to see if case b above occurs when 700mN is entered for the Offset Load.
- 3. When the measurement mode is set to 3 point-bending (sinusoidal or synthetic oscillation), tension (sinusoidal or synthetic oscillation), compression (sinusoidal or synthetic oscillation) and a value other than 0 is entered for the Tension/Compression Minimum Value. In this case, check to see if case a or b above occurs when the mode is switched to the single-cantilever mode.
- 4. The measurement mode is set to the F Control Mode and a value other than 0 is entered for the 1 Step Start Load in the SS Program. In this case, check to see if case a or b above occurs when the mode is switched to the L Control Mode.

Follow the steps below to perform strain output adjustment using the micrometer.

- 1. Select the L Control Mode and make sure the system is in the Ready status.
- 2. Open the front panel.
- 3. Remove the front plate of the detection system purge box (made of stainless steel).
- 4. Click the Motor Move icon, enter 700mN for the Offset Load and click the OK button.
- 5. Adjust the micrometer so that the L (strain signal) is in the  $\pm 10\mu$ m range.

## NOTE

The micrometer is very sensitive to movements and only requires small movements for adjustments (1 turn = 1/10). Be careful not to turn the micrometer a large amount. When the signal value is over  $\pm 200 \mu$ m, contact Service.

6. After performing adjustments, tighten the screws on the front panel of the detection system purge box and close the front panel.

# Appendix A Dynamic Viscoelasticity Measurement Range

### **A-1. Introduction**

Whether the dynamic viscoelasticity of a certain sample can be measured depends on the elastic modulus of the sample, the sample shape and dimensions as well as the deformation mode (bend, tension, compression, shear) and measurement mode (sinusoidal osciallation mode, synthetic oscillation mode). A measurement range exists for each value and if the value is out of the range the measurement cannot be performed or the error of the measurement value is extremely large.

Furthermore, in order to obtain quality data it is extremely important to correctly confirm that the value is within the measurement range before performing the measurement. Before performing the measurement, adjust the sample to the optimum dimensions and select the appropriate deformation mode and measurement mode.

## **A-2. Sample Dimension Criterion**

For the elasticity modulus of the sample, refer to the literature values and measurement values when available. When the values are not known, place the sample in the instrument and perform a test measurement to determine the values.

For the sample dimensions, adjust (bend measurement) the thickness of the sample and make the cross section area smaller (tension measurement) for hard samples. The opposite should be performed for soft samples.

As a reference, a representative polymer sample is shown in Table A-1 below. Table A-2 shows the approximate elasticity range of other samples. The optimum sample shape can be determined from the elasticity modulus range but only the criterion for the sample dimensions for the bend measurement and tension measurement are shown here.

| Table | A-1 |
|-------|-----|
|-------|-----|

|                               |   |        | Sample   | Dimensior | Criterion *2 | 2                   |
|-------------------------------|---|--------|----------|-----------|--------------|---------------------|
| Representative Polymer Sample | Approximate Elastic Modulus Range <sup>*1</sup> | Bend M | leasurem | ent       | Tension      | Measurement         |
|                               |   | l(mm)  | w(mm)    | t(mm)     | l(mm)        | S(mm <sup>2</sup> ) |
| Polyethylene, Polypropylene   |   | 20     | 10       | 2~3       | 20           | 2~5                 |
| Polyvinyl chloride            |   | 20     | 10       | 3         | 20           | 4                   |
| Polyethylene Terephthalate    | ]   |        |          |           | 20           | 4                   |
| Nylon                         | ]   | 20     | 10       | 3         | 20           | 4                   |
| Polymethyl Methacrylate       |   | 20     | 10       | 1.5~3     | 20           | 4                   |
| Polyimide                     |   |        |          |           | 20           | 0.1~0.5             |
| Epoxy resin                   |   | 20     | 10       | 1~2       | 20           | 0.1~0.2             |
| Rubber                        | Log E' (Pa)                                     | 20     | 10       | 3~4       | 20           | 2~10                |

Table A-2

|  |                                     |                  | Sample | Dimension | n Criterion *2      |                     |
|--|-------------------------------------|------------------|--------|-----------|---------------------|---------------------|
| Other Samples                                | Approximate Elastic Modulus Range*1 | Bend Measurement |        |           | Tension Measurement |                     |
|  |                                     | l(mm)            | w(mm)  | t(mm)     | l(mm)               | S(mm <sup>2</sup> ) |
| Resin + Inorganic Fiber<br>Compound Material |                                     | 20               | 10     | 1~2       | (20)                | (1)                 |
| Inorganic Glass                              | ······                              | 20               | 10     | 1~2       |                     |                     |
| Aluminum Alloy                               |                                     | 20               | 10     | 0.5       | 20                  | 0.1~0.3             |
| Stainless Steel                              |                                     | 20               | 10     | 0.4       |                     |                     |
| Tungsten                                     |                                     | 20               | 5      | 0.4       |                     |                     |
|  | Log E' (Pa)                         |                  |        |           |                     |                     |

- \*1 The elasticity modulus shown here is an approximate range. Even the same polymer can change depending on molecular weight or crystallinity, and the presence or non-existance of crosslinking and mixing.
- \*2 The values shown here are reference values. When performing measurements there are cases when the values must be changed due to measurement conditions such as temperature range, frequency, deformation mode, measurement mode, etc..

## A-3. Finding the Optimum Sample Shape

### A-3.1 Shape Factor

It is necessary to calculate the shape factor  $\alpha[m]$  in order to qualitatively find the limit value of the sample shape. Use the equation below to find the shape factor  $\alpha[m]$ . The calculation equation for the shape factor changes depending on the shape of the sample (for bend measurements : circular and angular cross-section samples), the dimensions, and the deformation mode.

As value of the shape factor gets larger it is more difficult to perform deformation. As the value gets smaller deformation becomes easier. This point has the same qualities of the elasticity modulus but differs on the following points:

| Shape Factor       | Depends on the shape of the sample, dimensions and deformation |
|--------------------|--|
|                    | mode.  |
|                    | Does not depend on sample materials.                           |
| Elasticity Modulus | Does not depend on the shape of the sample, dimensions and     |
|                    | deformation mode.  |
|                    | Depends on sample materials.                                   |

As an example, the equation below shows the definition of the shape factor using a tension measurement. The shape factor  $\alpha$ , tension force F, and coefficient of the deformation amount x is as follows :



Where :  $\sigma$  : Stress

- ε : Strain
- E' : Elasticity Modulus
- $k_f$ ,  $k_x$ : Peculiar coefficient to sample shape, dimensions and deformation mode For tension measurements, inversely proportion each of the cross-section area S and sample length **l**.

A measurement cannot be performed when the amount of deformation is too small because the sample becomes more difficult to extend when the shape factor is large. In contrast, when the shape factor is too small, the amount of deformation of the sample becomes too large even with a amplitude of a small force resulting in the exceeding of the set strain amplitude. The instrument automatically adjusts so the amplitude of force is small but if the value is too small and the limit is exceeded, the measurement cannot be performed.

Even with other deformation modes, the shape factor has the same qualities. The following equations can be used to find the shape factor.

| Bending Measurement                  | $16wt^3$                                   |
|--------------------------------------|--|
| Rectangular Cross section : $\alpha$ | =1   |
|                                      | 100013                                     |
| Bending Measurement                  | $3\pi d^4$                                 |
| Circular Cross section : $\alpha$    | =2   |
|                                      | 1000l <sup>3</sup>                         |
|                                      | 2S   |
| Shear Measurement : $\alpha$         | =  |
|                                      | 10001                                      |
| Tension Measurement                  | S  |
| Compression Meas. : $\alpha$         | =4   |
|                                      | 10001                                      |
| $\alpha$ : shape factor (m)          | d : sample diameter (mm)                   |
| t : sample thickness (mm)            | S : sample cross sectional area ( $mm^2$ ) |
| w : sample width (mm)                | $\pi$ : pi                                 |
| l : sample length (mm)               |  |

#### A-3.2 Usable Measurement Range

The ranges in Figure A-1 can be measured using this instrument. The paralleogram area within the bold parallel lines in the elasticity modulus (X axis) and shape factor (Y axis) graph can be measured. In figure A-1, there are a maximum of 5 columns in the horizontal direction which allows the instrument to measure a sample with a shape factor  $\alpha$ [m] across 5 columns of the elastic modulus.

Furthermore, if the sample shape and dimensions change, the elastic modulus can be measured with a range of from  $1 \times 10^3$  [Pa] to  $1 \times 10^{13}$  [Pa]. Therefore, even when the elastic modulus of the sample changes due to time or temperature the changes can be estimated beforehand and the sample shape and dimensions adjusted so that the optimum shape factor can be used to cover the changing range of the elastic modulus of the sample.

For example, when using the standard sample PMMA (Poly Methacrylate) resin the elastic modulus changes from about  $4\times10^9$ [Pa] to about  $5\times10^6$ [Pa] using a temperature range from room temperature to  $180^{\circ}$ C. The upper and lower limits of these values is shown using the horizontal arrow in Figure A-2 with an appropriate position sufficiently enclosed within the range. The shape factor value of the Y axis is within the range of about  $7\times10^{-5}$  to about  $5\times10^{-4}$  and if it is a bend measurement, a length of 20mm, width of 10mm and thickness of 2mm approximately are ideal.



Figure A-1 Dynamic Viscoelasticity Measurement Range Using the Pyris Diamond DMA



Figure A-2 Finding the Sample Dimensions Using the PMMA Bend Measurement

#### A-3.3 Common Method of Finding the Sample Shape Using the Graph

The following is a common method for finding the appropriate sample size from this graph.

1. When the elasticity modulus of the sample is known

Plot the elasticity modulus of the sample (Pa) and the shape factor (m) on the graph. The sample can be measured if the plotted point is within the two parallel lines.

Also, if the elsticity modulus of the sample will change due to temperature fluctuations during the measurement, a curved line appears in the horizontal direction. Draw horizontal dotted lines from the plotted point for the entire changing range of the elastic modulus and make sure the entire range is within the parallelogram.

When it is outside or on the edge of the parallelogram, the sample shape must be changed. When the sample shape is changed, the shape factor (m) also changes and the plotted point moves in the up/down direction. Change the sample shape so the shape factor is in the measurement range.

2. When the elasticity modulus of the sample is unknown

Place a sample with an appropriate shape into the instrument and click the Test button to perform a test measurement. If the measurement is successfully completed, the results are displayed in the measurement data window and the approximate elastic modulus of the sample is known.

If the "A Low Zone Error occurred. (Is the sample too hard?)" message is displayed, change the shape of the sample to reduce the sample factor  $\alpha$  (m) and perform the measurement again.

By performing these changes, the approximate elastic modulus of the sample can be determined and the new value can be used in step 1 above to find the appropriate sample shape.

As shown in figure A-1, the Pyris Diamond DMA can continuously measure a sample with a fixed shape across 5 columns of elastic modulus change. However, in the synthetic oscillation mode, the upper limit of the AC power amplitude assigned to each frequency is 1/5 in comparison to the sinusoidal osciallation mode. Because of this, there are times when measurements cannot be peformed in the right 0.7 column (high elasticity Zone A). Furthermore, when compared to other frequencies, the accuracy of tan $\delta$ , E" and E' may drop in the higher frequency measurements of 20Hz for left column 1 (low elasticity Zone B).

### ( Information

- The drop in accuracy of the E" and tanδ signal during phase difference in high frequency measurements in zones B mentioned above is particularly severe. For this reason, check the conformity of a lower frequency measurement before using.
- 2. The measurement range shown in the parallelogram in Figure A-1 is the region where the accuracy of the E' measurement is within 30%, the center is of higher accuracy than either the left or right sides. For this reason, make sure the sample elastic modulus range is sufficiently near the center of the parallelogram when determining the sample shape.

The upper value (Emax) and lower value (Emin) of the elasticity measurement using the Pyris Diamond DMA can also be used to determine the equation numerically using the shape factor ( $\alpha$ ) obtained in equations 1 ~ 4.

Emax =  $10^{7}$  /  $\alpha$  (Pa), Emin =  $10^{2}$  /  $\alpha$  (Pa)
## Appendix B Static Viscoelasticity Measurement Range

The static viscoelasticity (F Control, L Control mode) measurement range varies from the dynamic viscoelasticity as shown in Figure B-1 below. The measurement range using F control is the area within the solid lines and the measurement range using the L control mode is the area to the left of the dotted line in the figure below. The method for determining the shape factor  $\alpha$  and elasticity is the same as the method used for dynamic elasticity.

When performing tension measurements, oscillation may occur even within the measurement range causing the measurement to be performed incorrectly. When this occurs, perform the following:

- 1. Reduce the change of the width of the input parameters for either the L or F control modes. (For example, set the start load and limit load to a range of  $0 \sim 500$ mN or the start position and limit position to a range of  $0 \sim 50$ µm.)
- 2. Change the sample shape and perform a measurement using the bend mode.



Figure B-1 Static Viscoelasticity (F Control, L Control) Measurement

# Appendix C Relationships Between the Various Quantities Used to Represent Dynamic Viscoelasticity

All materials have viscoelasticity, which is a combination of viscosity and elasticity in varying amounts. When this viscoelasticity is measured dynamically, there is a phase shift between the force applied as stimulus (stress) and the deformation measured as response (strain). Generally the measurement results are represented as a complex elasticity modulus to insure accurate expression. This relationship is shown below.

E''  $\tan \delta = ---.2$ E' **E**"  $2\pi f$ 1  $J^* = - = J' - iJ'' - .....4$ E\* E' J' = - $(E')^2 + (E'')^2$ **E**" J" = ------- .....6  $(E')^2 + (E'')^2$ 

If the relationship between equations 1 and 2 is plotted, we obtain a graph like that in the figure below.



# Appendix D Relationship Between Measurement Frequency and Transition Temperature

There is a general tendency in dynamic viscoelasticity measurements for the detected transition temperature to shift when the measurement frequency is changed.

This phenomenon is based on a principle called the time-temperature-superposition principle, where the transition temperature (for example, the top peak temperature of tan $\delta$ ) tends to rise when the measurement frequency is increased (see Figure D-1).



Figure D-1 Frequency and Temperature Relationship

The relationship between the top peak temperature T of  $tan\delta$  and the value of the frequency coordinate f is shown below. The transition activation energy can be obtained from data measured at multiple frequencies.

$$\Delta E = \frac{R}{0.4567} \frac{d (logf)}{d (1/T)}$$

f = Measurement frequency (Hz) R = Gas constant (= 8.31434 J/mol/deg) T = Absolute temperature (K) $\Delta E = Activation energy (J/mol)$ 

## Appendix E Units Used in Measuring Viscoelasticity

The default unit and analysis system for the measurement signals generated by this instrument is SI units. However, it is possible to change the units system and analyze according to the tables shown below. Unit system setting changes are performed on the Display Settings in the Analysis window. (For detailed information, refer to the Pyris Station operation manual.)

Table E-1 Elasticity Units and Conversion Factors (Gain Input Values)

| Units of E, G      | Pa | dyn/cm <sup>2</sup> | kg/cm <sup>2</sup>      | kg/mm <sup>2</sup>      | psi(lb/in <sup>2</sup> ) |
|--------------------|----|---------------------|-------------------------|-------------------------|--------------------------|
| Conversion Factors | 1  | 10                  | 1.02 x 10 <sup>-5</sup> | 1.02 X 10 <sup>-7</sup> | 1.45 X10 <sup>-4</sup>   |

Table E-2 Viscosity Units and Conversion Factors (Gain Input Values)

| Units of <b>η</b>  | Pa · sec | $kg/(m \cdot s)$ | poise | $kg/(m \cdot hr)$     | lb/(ft · s)            |
|--------------------|----------|------------------|-------|-----------------------|------------------------|
| Conversion Factors | 1        | 1                | 10    | 3.6 X 10 <sup>3</sup> | 6.72 X10 <sup>-1</sup> |

## Appendix F Sample Temperature Signal Polynomial Regression Correction

With DMA modules, the sample temperature is first measured using a thermocouple and is then output as a linearized temperature value based on an electromotive force table used for thermocouples. Generally, the correction of sample temperature is performed by following the steps in section 3.2 and using a PMMA sample. When there is systematic drift between the temperature in the designated location and the individual transition temperature measurement value of multiple samples or multiple transitions, the sample temperature output can be corrected to remove the drift.





The regression curve  $(y=a_0 + a_1x + a_2x^2 + a_3x^3)$  is determined using the method of least squares. If the regression order is n, and the correction data is represented by (xj, yj) (j=1, 2, --, m), then the coefficients ai(i=0, 1, --, n) are derived using the following matrix calculation.

$$\begin{bmatrix} a_{0} \\ a_{1} \\ a_{n} \end{bmatrix} = \begin{bmatrix} m & m & m & m \\ \sum 1 & \sum x_{j} & \cdots & \sum x_{j}^{n} \\ j=1 & j=1 & \cdots & j=1 \\ m & m & m & m \\ \sum x_{j} & \sum x_{j}^{2} & \cdots & \sum x_{j}^{n+1} \\ j=1 & j=1 & \cdots & j=1 \end{bmatrix} \begin{bmatrix} m \\ \sum y_{j} \\ m \\ \sum y_{j} \\ y_{j} \\ y_{j} \\ z_{j} \\ z_{j}$$

The following are the steps:

1. Input the following initial values for the Temperature Correction in the DMA ABC Setting window:

|                    | Default Values |
|--------------------|----------------|
| Temp. Corr 0 Order | 0              |
| 1 Order            | 1              |
| 2 Order            | 0              |
| 3 Order            | 0              |

2. Perform the necessary number of measurements of samples for calibration and analyze the data to detect the transition temperature.



3. Open the automatic calculation window for sample temperature correction

| Automatic Calibration Calculation       |                               |                                    |  |  |  |  |
|---|-------------------------------|------------------------------------|--|--|--|--|
| Regr                                    | Regression Order 1            |                                    |  |  |  |  |
|   | Literature Temp.<br>Value (C) | Actual Heasured<br>Temperature (C) |  |  |  |  |
| 1 لــــــــــــــــــــــــــــــــــــ | 156.600                       | 156.600                            |  |  |  |  |
| 2 لـ                                    | 232.000                       | <u>ĭ</u> 232.000                   |  |  |  |  |
| ГЗ                                      | 0.000                         | <u> </u>                           |  |  |  |  |
| Г4                                      | <u>ĭ</u> 0.000                | ž 0.000                            |  |  |  |  |
| Γ5                                      | <u> </u>                      | <u> </u>                           |  |  |  |  |
| F 6                                     | <u>i</u> 0.000                | <u> </u>                           |  |  |  |  |
| ז ד                                     | <u> </u>                      | 1 0.000                            |  |  |  |  |
| F 8                                     | <u> </u>                      | ž 0.000                            |  |  |  |  |
| Г 9                                     | 1 0.000                       | <u> </u>                           |  |  |  |  |
| F 10                                    | <u> </u>                      | <u>ĭ</u> 0.000                     |  |  |  |  |
| Г 11                                    | <u> </u>                      | 1 0.000                            |  |  |  |  |
| <u><b>F</b> 12</u>                      | <u>ĭ 0.000</u>                |                                    |  |  |  |  |
|   |                               | <u> </u>                           |  |  |  |  |
| OK Cancel                               |                               |                                    |  |  |  |  |
|   |                               |                                    |  |  |  |  |

Sample Temperature Correction Auto Calibration Window

4. Select either 1, 2, or 3 for the regression order.



- 5. Input the data to be used for correction (Literature Temperature, Actual Measured Temperature). The number of data used should be one greater than the regression order set in step 4. For example, if the regression order is "1" you should use two or more data values.
- 6. Click on the check boxes to the left of each data item to set the calculation data. As an example, use the above operations and set the regression degree to 2, sample A (Literature Value Temperature -20°C), sample B (Literature Value Temperature 80°C) sample C (Literature Value Temperature 160°C) sample D (Literature Value Temperature 250°C) sample E (Literature Value Temperature 320°C). For this example, when the measurement results are -21°C, 78°C, 160°C, 251°C, and 322°C, your window should look similar to the following:

|                     | Automatic Calibration Calculation                             |  |  |
|---------------------|---|--|--|
|                     | Regression Order <u>2</u>                                     |  |  |
|                     | Literature Temp. Actual Heasured<br>Value (C) Temperature (C) |  |  |
| ſ                   | ⊥ 1 Ĭ -20.000   Ĭ -21.000   Ĥ                                 |  |  |
| Press buttons       | _1 2 <u>1 80.000</u> <u>1 78.000</u>                          |  |  |
| 1 through 5 $\prec$ | ⊿ 3 ∐ 160.000 ∐ 160.000                                       |  |  |
| e                   | 4 <u>1</u> 250.000 <u>1</u> 251.000                           |  |  |
| Ĺ                   | _ 5 <u>I</u> 320.000 <u>I</u> 322.000                         |  |  |
|                     | <b>⊢</b> 6 ≚ 0.000  ≚ 0.000                                   |  |  |
|                     | Г 7 <u>I 0.000</u> <u>I 0.000</u>                             |  |  |
|                     | <b>〒</b> 8 ∐ 0.000 [∐ 0.000                                   |  |  |
|                     | <b>F</b> 9 <b>1</b> 0.000 <b>1</b> 0.000                      |  |  |
|                     | <b>□</b> 10 0.000 0.000                                       |  |  |
|                     | <b>□ 11 1 0.000</b> 1 0.000                                   |  |  |
|                     |   |  |  |
|                     |   |  |  |
|                     | Cance I   |  |  |

7. When the OK button is clicked, each of the temperature correction coefficients is automatically calculated and displayed.

## Appendix G The Relationship Between Young's Modulus and Rigidity

Young's modulus (stretch or vertical elasticity coefficient) and the rigidity coefficient (shear or horizontal elasticity coefficient) can be found with the DMA module, depending on the measurement mode.

Bend, Tension, Compression Mode  $\rightarrow$  Young's modulus measurement

Shear mode, Film Shear Mode  $\rightarrow$  Rigidity coefficient measurement The volume elasticity coefficient K, the Poisson ratio  $\sigma$ , Young's modulus E, and the rigidity coefficient G are all practical elasticity coefficients of isotropic materials. Only two of these elasticity coefficients are independent. The others can be derived from them. In other words,

$$K = \frac{EG}{9G-3E} \dots 1$$

$$\sigma = \frac{E - 2G}{2G} \dots 2$$

For many materials  $\sigma$ =0.5. If we apply this assumption to equation 2, we find that E=3G. This relationship is called the "3-times rule of elasticity coefficients" and is very helpful in data conversion

## Appendix H Using the Optional Shear Attachment

In order to measure in the shear mode, you must attach the sample to the shear attachment. Next, attach the sample affixed shear attachment to the measurement unit. Follow the steps below to perform the measurement.

#### **Attaching the Bend Chuck**

- 1. Follow the steps for Exchanging the Sample Unit Attachment in Section 5.1 of the Module Operation Manual and attach the bend measurement attachment.
- 2. Leave only the bend chuck base in place and remove the bend chuck and left and right bend bases.

#### Attaching the Sample

- 1. Prepare 2 samples of the same shape (for shape selection refer to Appendix A), the shear mode base with clamps, and jig tools used to position the chuck and sample (the base and chuck should be off the detector area).
- 2. Set the chuck and base on the jig and loosen the knurled bolts on both clamps.
- 3. Position the sample on both sides of the chuck with tweezers, tighten the knurled bolts, and then secure further by tightening the clamp set screws (see Figure H-1 below).



Figure H-1 Securing the Sample Using the Shear Attachment

#### Attachment to the Measurement Unit

- 1. Click the Motor Move icon, enter -1600µm for the Movement Target Position and click the Move button. Set the probe to the 16mm position as with the bend measurement.
- 2. Attach the sample affixed shear attachment to the measurement unit. To attach the attachment, secure the shear base to the brace using the 3mm hexagonal screws (4 screws) and attach the shear chuck to the chuck base using the 2.5mm hexagonal screws (2 screws) and tighten both.

#### Measurement

Select either Shear Sinusoidal or Shear Synthetic and set the measurement conditions paramaters to correspond to the bend measurement and click the RUN button to begin the measurement.

## Appendix I Using the Optional Compression Attachment

The Compression Accessory employs compression to perform dynamic viscoelasticity measurement using the DMA module.

- 1. Configuration
  - a. Probe compression plates
  - b. Clamp compression plates



Probe Side Compression Plate

- 2. Specifications
  - a. Plate Diameter :  $\phi 10mm$  (area=78.5mm<sup>2</sup>)
  - b. Sample Length : ~ 15mm
  - c. Elastic Modulus Range : Refer to Appendix A Dynamic Viscoelasticity Measurement
    - Range in this manual.
- 3. Measurement Procedure
  - a. Installing the Compression Plates

Click the Move button after setting the minimum tension/compression to 0 in the Position and Tension Settings window and the Movement Target Position to -7000. After matching the plate surfaces as shown in the figure below, firmly attach the plates to the chuck and clamp base, respectively.

**Clamp Side Compression Plate** 



Installing the Compression Measurement Accessory

#### b. LVDT Signal Origin Adjustment

Click the Motor Move icon to open the window. Enter a value of -5000 $\mu$ m for the Movement Target Position and click the Move button. The probe is moved to the -5mm position. Enter 0 for the minimum tension/compression and click the OK button. Input an offset load that will regulate the stress signal in the 0 ± 2 $\mu$ m range. The entered offset value is shown in the window, after the window OK button is clicked.

#### c. Setting the Compression Conditions

Input the following conditions in the Position and Tension Settings window:

| Min. Tension/Compression Force | : | 300mN |
|--------------------------------|---|-------|
| Tension/Compression Force Gain | : | 1.5   |
| Force Amplitude Initial Value  | : | 100mN |

For compression measurements, use the following ranges:

Tension/Compression Force Gain :  $\geq 0$ 

d. Setting the Sample

Because it is necessary to keep the plates parallel when setting the sample, use the movement target position setting and Move button.

- 1. Enter (sample length(mm) -7) x 1000 in the movement target position and click the Move button.
- 2. Pinch the sample between the plates and click the TEST button to confirm that measurement can be performed. Be sure that the sample is placed in the center region of the plates.

#### e. Starting Measurement

Open the Sample Condition setting window, enter the individual measurement conditions and click the OK button. Click the TEST button and confirm that the elastic modulus is an appropriate value for measurement. Next, close the furnace and press the RUN button to start measurement.

| S NOTE  |                     |    |  |  |  |
|---|---------------------|----|--|--|--|
| 1. Take note of the following information on the F force applied to the |                     |    |  |  |  |
| samp  | le.                 |    |  |  |  |
|   | F = Ft +            | FC | ) sin wt   |  |  |
|   | Ft=Fbase+F0*F0 gain |    |  |  |  |
|   | Ft                  | :  | oscillating force (the direction that pulls the sample |  |  |
|   | is positive)        |    |  |  |  |
|   | F0                  | :  | constant force   |  |  |
|   | Fbase               | :  | Min. Tension/Compression Force                         |  |  |
|   | F0 gain             | :  | Tension/Compression Gain                               |  |  |
|   |                     |    |  |  |  |
|   |                     |    |  |  |  |

- 2. Using the Hold button, when measuring with the Automatic Cooling Unit (optional accessory), may cause the sample to be pulled out and dropped due to thermal contraction. For this reason, we suggest using the RUN button instead. (When using RUN, a sample length compensation mechanism is activated and the compression is continuously applied to the sample according to the tension conditions set previously .)
- 3. Poor contact between the sample surface and the compression plate surface can cause large errors in the measurement values. When setting the sample, be sure that the sample and plate surfaces are in full contact with each other. If there is any uncertainty concerning the contact status, we suggest the use of instant contact cement, etc. to fasten the sample to the plate.

### Appendix J Using the Optional Film Shear Attachment

Remove the bend mode clamps and chuck, and place the film shear accessory as shown in the Figure J-1. Use the probe at the  $-16000\mu m$  (-16mm) position (same as the bend mode).



Figure J-1 Film Shear Mode

The selection of the deformation mode and the values to input for sample length are as listed below.

Deformation Mode : Shear length : 5mm



## Appendix K Using the Optional Bend Attachment

Besides the standard measurement head, (sample length 20mm) there are also two other varieties provided. See figure below. The 32mm measurement head is for thick samples, or samples that have a high elastic modulus. The 8mm head is for thin or soft samples.



32mm Head

8mm Head

Structure

| 32mm Head                      | 8mm Head                      |  |
|--------------------------------|-------------------------------|--|
| 32mm Bend Semicircle Plate (2) | 8mm Bend Semicircle Plate (2) |  |
| Steel Correction Plate (1)     | Aluminum Correction Plate (1) |  |
| Sample Fastening Chuck (2)     | Sample Fastening Chuck (2)    |  |
| M3Hesagonal Screws (4)         |                               |  |

#### **Elastic Modulus Correction**

When the optional bending measurement head is used, the elastic modulus must be recalibrated. Follow the procedure below to recalibrate the modulus.

1. Set the elastic correction standard sample using the settings for a 32mm and an 8mm head. After correctly entering the shape of the sample, click the TEST button and measure the standard sample at 1Hz to obtain the E' measurement value.

|           | Elastic Modulus Correction Plate       | E' Literature Value        |
|-----------|--|----------------------------|
| 32mm Head | Fe Plate : Width 10mm, Thickness 0.6mm | $1.92 \times 10^{11}$ (Pa) |
| 8mm Head  | Al Plate : Width 5mm, Thickness 0.2mm  | 6.91×10 <sup>10</sup> (Pa) |

- 2. Click the Calibration icon to open the Calibration setting window. Save the standard head calibration value as a file using the Save As command. Click the F/L Gain Calculation window switch to open the calculation window.
- 3. Enter the E' measurement value obtained in step 1 into the documented value in the Calculation window. Turn on the switches for F/L Gain, Viscosity Correction, Probe Mass, and Elasticity Correction. Click OK after checking the entered value and making sure the switches are turned on.
- 4. The individual calculation results for F/L Gain, Viscosity Correction, Probe Mass, and Elasticity Correction values are changed and displayed in the Calibration window.
- 5. Click the OK button to end calibration.

### S NOTE

- 1. When using the standard bend mode or a different measurement mode, open the file saved in step 2 and change the calibration settings.
- 2. It is best to save the 32mm and 8mm calibration setting values using the Save As command immediately after the changes are displayed after step 4 above.

# Appendix L Single-Cantilever Measurements

Single-cantilever measurements, as shown in the figure below, perform viscoelasticity measurements of only one side of the sample using a bend measurement attachment. This mode is extremely effective for samples with high elasticity or samples with a high elastic modulus that make it difficult to use the bending measurement.

When performing single-cantilever measurements, the calculation of the elastic modulus and the system calibrations are different in comparison to those of the bending measurement. Because of these factors, use the method below to perform the measurements.



Figure L-1



1. Measurement Attachment

Use the same bend measurement attachment as used with bending measurements.

2. Elastic Modulus of the Sample

The following equation is used for calculating the elastic modulus of the sample: Single-cantilever Measurements

| Rectangular Cross-section Samples | $\alpha =$ | wt <sup>3</sup>       |
|-----------------------------------|------------|-----------------------|
| Circular Cross section Samples    | or —       | $3\pi d^4$            |
| Circular Cross-section Samples    | α =        | 16*1000l <sup>3</sup> |

Sample diameter (mm)

 $\alpha$  : Elastic Modulus (m) d :

Sample Thickness (mm)

- $\pi$  : pi
- w : Sample Width (mm) 1 : Sample length (mm)

For the sample length l, as shown in the figure above enter a value of 1/2 in relation to the bending. For a 20mm bend attachment, enter 10mm. For a sample with the sample cross-section shape, the calculation of the elastic modulus for the single-cantilever and bending measurements, the single-cantilever will have a value of 1/2 that of the bending measurement. In other words, the single-cantilever is 0.3 columns smaller and the elasticity measurement range moves towards the high elasticity side by 0.3 columns. (Refer to Appendix A Dynamic Viscoelasticity Measurement Range).

3. System Calibrations

t :

- a. Perform dynamic viscoelasticity measurement calibrations as outlined in Chapter 3 of the Pyris Diamond DMA Operation Manual (Measurement Procedure) and save the constants in a different file. If calibrations have already been performed, save as a different file. If the file has already been saved, proceed to step b.
- b. Open the saved constants file saved in step a.
- c. Enter 0 for the compliance correction value. Do not change any other settings.
- d. Using the Motor Move icon, set the target movement to -16000(μm) and click the Move button. After movement is complete, click the OK button. Catching of a 12mm width, 3mm thickness stainless plate, as shown in the figure for single-cantilevers is performed.

### ( Information

Compliance calibrations can be performed from either the right or left position. However, be sure to have the measurement position of the sample the same as the calibration position. Likewise, the position must be the same for sample temperature calibration.

e. Open the Sample Conditions setting window, perform the settings shown below and click the OK button. (The bend attachment is being used but be aware that the measurement mode selection is on shear.)

| Shear · Sin | nusoi   | dal  |
|-------------|---|--|
| Length      | :   | 0.01mm   |
| Rectangula  | ar Cr   | oss-section  |
| Width       | :   | 1mm  |
| Thickness   | :   | 5.1mm  |
|             | Shear · Sin<br>Length<br>Rectangula<br>Width<br>Thickness | Shear · Sinusoi<br>Length :<br>Rectangular Cro<br>Width :<br>Thickness : |

| Data Filename<br>Temperature Control Mode | Enter the appropriate filename (ex. dmscal.00)<br>Step |   |          |  |
|---|--|---|----------|--|
| Temperature Program                       | I  |   |          |  |
|   | Soak   | : | 2 (sec.) |  |
|   | Start Temp.  | : | 0(°C)    |  |
|   | Limit Temp.  | : | 20       |  |
| Frequency Setting                         | 1Hz only ON  |   |          |  |
| Stress Amplitude                          | 20(µm)   |   |          |  |

f. Click the TEST button and perform a test measurement. After confirming the elasticity displayed in the measurement data message window, click the OK button.



- g Click the Measure button and perform a measurement.
- h. Once the measurement is complete, open the measured data using f slice analysis (ex. dmscal.00). Set the X axis to time and calculate the average value of the G' value.



Using the signal properties window, signal selection and display settings of f slice analysis, set the signal selection to G'Pa and make sure the A (gain) and B (offset) coefficients on the lower left are set to 1 and 0 respectively.

If the values are different, enter A=1, B=0 and click the OK button. Next, click the OK button on the Display Settings window.

i. Open the DMA ABC Setting window and click the Compliance Correction calculation button.

In the Actual Value G' of the Auto Correction Calculation window read in the average elasticity value (M') of G' from step e and click the OK button.

j. The value calculated in the compliance correction area is entered. If the value is correct, the calculation value is in the  $10^{-4} \sim 10^{-3}$  order. Click the OK button.

### (B) Information

Be sure to close the window once using the OK button. If the window is open, the correction will not be reflected in the steps that follow.

- k. The value entered using the DMA ABC Setting window is the correction value for the single-cantilever measurement. Save the file with a new name as a single-cantilever correct value file.
- 4. Single-cantilever Measurements

The maximum length of the sample for single-cantilever measurements is 35mm. Enter the measurement conditions using the Sample Condition Settings window. Enter a sample length 1/2 that of the bend measurement attachment. When using a 20mm bend measurement attachment, enter 10mm. For the measurement mode, select the Single-Cantilever Sinusoidal/ Synthetic control mode and click the OK button.

For sample chucking, secure the sample using the bend clamp on one side and the bend chuck as shown in the figure. Be careful so that the sample is not touching another bend clamp or the bend base.

### S NOTE

When securing the bend chuck screw of the probe, hold the bend chuck with your fingers so that the probe does not twist.

Click the Test button. After making sure the elasticity value is an appropriate value, close the furnace, click the Measure button and begin the measurement.

# Appendix M 3 Point Bending Measurements

3 Point Bending measurements, as shown in the figure below, measure viscoelasticity measurements using the optional 3 point bend measurement attachment. This mode is extremely effective for samples with high elasticity or samples with a high elastic modulus that are difficult to measure using the bending measurement.

When performing 3 point bending measurements, the calculation of the elastic modulus and the system calibrations are different in comparison to those of the bending measurement. Because of these factors, use the method below to perform the measurements.





1. Measurement Attachment

Use the 3 point bend measurement attachment (option).

Attaching the 3 Point Bend Measurement Attachment

- 1. Using the Motor Move icon, set the target movement to  $-16000(\mu m)$  and click the Move button. Once movement is complete, click the OK button.
- 2. Properly attach the 4 supports on the left and right sides of the 3 point bend base.
- 3. Make sure the 3 point bend sample base is attached to the 3 point bend sample holder (probe side) and slide it into the probe tip. While supporting the 3 point bend sample holder with your hand, place the gap setting plate on the 3 point bend sample holder (base side).
- 4. Align the 3 point sample base with the round indentation of the gap setting plate. Secure the 3 point bend sample holder (probe side) with the fastening screw.


Figure M-2

2. Elastic Modulus of the Sample

The following equation is used for calculating the elastic modulus of the sample: 3 Point Bending Measurements

|   |   | Destancylar Cross section  | n Samples |   | $4wt^3$                                 |
|---|---|----------------------------|-----------|---|---|
|   |   | Rectangular Cross-section  |           |   | $\alpha = \frac{10001^{3}}{10001^{3}}$  |
|   |   | Circular Cross-section Sam | ples      |   | $\alpha = \frac{3\pi d^4}{4^* 10001^3}$ |
| α | : | Elastic Modulus (m)        | d         | : | Sample diameter (mm)                    |
| t | : | Sample Thickness (mm)      | π         | : | pi                                      |
| W | : | Sample Width (mm)          | 1         | : | Sample length (mm)                      |

For the sample length l, enter 40mm. Using a sample with the same cross-section shape, if you calculate the elastic modulus (m) of the bend and 3 point bend, the 3 point bend is 1/32 of the bend measurement. In other words, the 3 point bend is 1.5 columns smaller and the viscoelasticity measurement range moves to higher elasticity 1.5 columns. Refer to Appendix A Dynamic Viscoelasticity Measurement Range.

- 3. System Calibrations
  - a. Perform dynamic viscoelasticity measurement calibrations as outlined in Chapter 3 of the Pyris Diamond DMA Operation Manual (Measurement Procedure) and save the constants in a different file. If calibrations have already been performed, save as a different file only. If the file has already been saved proceed to step b.
  - b. Open the saved constants file saved in step a.
  - c. Using the Motor Move icon, set the target movement to  $-15600(\mu m)$  and click the Move button. After movement is complete, change the Force Amplitude value to 100mN and click the OK button.
  - d. Place a steel plate as the sample with a width of 10mm and a thickness of 0.4mm.
  - e. Open the Sample Conditions setting window, perform the settings shown below and click the OK button.

| Sample Shape      | Length      | :       | 40mm       |  |  |
|-------------------|-------------|---------|------------|--|--|
|                   | Rectangula  | r Cros  | ss-section |  |  |
|                   | Width       | :       | 10mm       |  |  |
|                   | Thickness   | :       | 0.4mm      |  |  |
| Frequency Setting | 1Hz only ON |         |            |  |  |
| Control Mode      | 3 Point Ber | nding · | Sinusoidal |  |  |

## ( Information

Perform an actual measurement and enter the correct values for the width and thickness of the steel plate.

- f. Click the TEST button and perform a test measurement. After confirming the elasticity displayed in the measurement data message window, click the OK button.
- g. Click the Measure button and perform a measurement.
- h. Once the measurement is complete, open the measured data using f slice analysis. Set the X axis to time and read the E' value and calculate the average value.
- i. Open the ABC Setting window and click the F/L Gain "Calculate" button.
- j. Enter the following values into the Auto Calculate window: Literature Value E': 1.92 + 11 Actual Measured Value: Average value of E' calculated in step f. Constant Update : Turn on F/L Gain, Viscosity Correction, Probe Mass, and Elasticity Correction

- k. Click the OK button in the Auto Calculate window. The F/L Gain, Viscosity Correction, Probe Mass, and Elasticity Correction values in the DMS ABC Setting window are updated to the new values. Click the OK button.
- 1. The values entered into the DMS ABC Setting window are the 3 point bending corrected values. Save this as a new file for the 3 point bending corrected file.

## 4. 3 Point Bending Measurements

Enter (sample length (mm) - 16) × 1000 for the target movement using the Motor Move icon and click the Move button. Enter the other conditions and click the OK button. Open the Sample Conditions Setting window and enter each measurement condition. Enter 40mm for the sample length and select 3 point Bending Sinusoidal/Synthetic for the control mode. Click the the OK button.

Set the sample and click the Test button. After making sure the elasticity value is an appropriate value, close the furnace, click the Measure button to begin the measurement.

## Appendix-N Tension/Compression Force During Tension/ Compression Mode Measurements

When using the tension/compression mode, it is necessary to have appropriate control based on the status of the sample for the tension/compression force. The Pyris Diamond DMA applies the appropriate tension/compression force automatically even when the status of the sample changes. When the sample is soft, a small value is used so that the sample does not stretch or crush and when the sample is hard, AC amplitude is a sufficiently large number. The actual tension/compression force value is shown in the following equation:

Ft = (F base) + (F0 gain) \* F0

where,

| Ft      | : | Tension/compression force [N]         |
|---------|---|---------------------------------------|
| F base  | : | Minimum tension/compression force [N] |
| F0 gain | : | Tension/compression force gain        |
| F0      | : | AC Force Amplitude [N]                |

During Ready status, F0 = 0 [N] so Ft = F base and the tension/compression force Ft equals the minimum tension/compression force F base.

During the measurement, the AC force amplitude automatically changes in accordance with the size of the storage elasticity E' of the sample. F0 is proportionate to E' even with the shape factor  $\alpha$ . The tension compression force Ft also changes automatically in accordance with the size of the storage elasticity of the sample excluding when the F0 gain = 0.

The F0 gain value is normally entered between 1.2 and 1.5.

The relationship between the AC force amplitude and the tension/compression force Ft is as shown in the figure below. The thick line is the tension/compression force Ft applied to the sample. The wave shaped line displays the model of Ft when applying the AC force.





F0 gain = 1.5 Example









F0 gain = 0.0 Example



When the F0 gain = 0.0, the tension/compression force FT normally stays at a constant value even when the AC amplitude F0 fluctuates.

