Quantum Design



# Physical Property Measurement System®

# Dilatometer Option User's Manual

Part Number 1071-100 Rev. A0

#### **Quantum Design**

10307 Pacific Center Ct. San Diego, CA 92121-3733 USA Technical support (858) 481-4400 (800) 289-6996 Fax (858) 481-7410 Email ATLService@qdusa.com

First edition of manual completed February 2017.

#### Trademarks

All product and company names appearing in this manual are trademarks or registered trademarks of their respective holders.

# **Components of the Dilatometer Option**

Item	Description	Quantity
Cell Capsule (4071-120)	The capsule contains the cell, sample thermometer, rotator housing, and spherical housing. This rests inside the cryostat while a measurement is being performed.	1
Radiation Shield and Base Screw (4071- 200/210)	The radiation shield slides over the capsule. and the base screw secures the shield in place.	1
Dilatometer Probe (4071-130)	The probe provides electrical connection between the capsule and the module and puts the capsule in the cryostat sample chamber, in the center of the magnetic field.	1

Item	Description	Quantity
Image: Second state sta	The module contains electronics that supply the cell with a voltage, read the return signal and read the thermometer resistance values.	1
	The cable connects the module to	1
Cable Assembly (3071-100)	the probe.	
Balance Tester (4071-116)	The Balance Tester provides a way to measure the quality of cell mounting and test cell thermometry.	1
Dilatometer User Kit (4071-140)	The user kit contains all of the essentials for using the Dilatometer Option: Kapton sample spacers (3), CrystalBond <sup>TM</sup> 509, sample lapping tools (2), copper sample (2), fuses silica sample (2), thin-fused silica spacer (5), thick-fused silica spacer (5), spring (5), flat tweezer, pointed tweezer.	1 (See description for sub assembly quantities)

# C O N T E N T S

# **Table of Contents**

Con	Components of the Dilatometer Option		
PRF	EFACE	xi	
Con	itents and Conventions	xi	
P.1	Introduction	xi	
P.2	Scope of the Manual	xi	
P.3	Contents of the Manual	xii	
P.4	Conventions in the Manual	xii	
P.5	Safety Guidelines and Regulatory Information	xiv	
	P.5.1 Inspection for Damage	xiv	
	P.5.2 Electricity	xiv	
	P.5.3 Magnet Safety	XV	
	P.5.4 Cryogenic Safety	XV	
P.6	Disposal Information	xvi	
	P.6.1 RoHS Statement	xvi	
	P.6.2 WEEE Statement	xvi	
P.7	Contacting Quantum Design	xvii	
CH	APTER 1		
Ove	erview of the Dilatometer System	1-1	
1.1	Introduction	1-1	
1.2	What the System Measures	1-1	
1.3	Purpose of the Dilatometer Measurements	1-2	
1.4	Theory of Operation		
	1.4.1 Cell Circuit	1-3	
	1.4.2 AC Drive Voltage and Imbalance	1-4	
	1.4.3 Signal Processing	1-5	
	1.4.4 Thermometry	1-5	
1.5	Scope of the Dilatometer Option	1-6	
	1.5.1 Sample Thermal and Magnetic Characteristics	l-6	
	1.5.2 Sample Size	l-6	
	1.5.3 Temperature and Magnetic Field Range	1-6	
CH	APTER 2		
Inst	allation and Getting Started	2-1	
2.1	Introduction	2-1	
2.2	System Requirements	2-1	
2.3	Installing the Hardware and Software		
	2.3.1 Installing the Dilatometer Control Electronics and Cable		
	2.3.1.1 Install the Model UNI-K Dilatometer Module		
	2.3.1.2 Connect the Dilatometer Cable		
2 1	2.3.2 Installing the Software		
∠.4	2.4.1 Drangering the Somple		
	2.4.1 Freparing the Sample	2-4	

	2.4.2 Cleaning the Sample	2-6
	2.4.3 Mounting the Sample	2-6
2.5	Balance Meter	2-8
	2.5.1 Imbalance	2-8
	2.5.2 Operation	2-9
	2.5.2.1 Understanding the Imbalance Reading	2-9
	2.5.2.2 Check Thermometry	2-9
2.6	Preparing the System	2-10
	2.6.1 Radiation Shield	2-10
	2.6.2 Capsule to Probe Connection	2-10
	2.6.3 Navigating the Wizard	2-10
CHA	APTER 3	
Har	dware	
3.1	Introduction	
3.2	Dilatomer Hardware	
	3.2.1 Model CM-R Dilatometer Control Module	
	3.2.2 Dilatometer Cable	
	3.2.3 Probe	
	3.2.4 Capsule	
	3.2.4.1 Housing	
	3.2.4.2 Cell	
	3.2.4.3 Thermometry	3-5
	3.2.4.4 Serial Number	
	3.2.5 Balance Meter	
3.3	Quantum Design Part Numbers	
CHA	APTER 4	
Soft	ware	4-1
4.1	Introduction	4-1
4.2	Overview of the System Software	4-1
4.3	Dilatometer Option Controller	4-2
	4.3.1 Projects	4-3
	4.3.2 Project Navigation Pane	4-3
	4.3.2.1 Project Tab	4-4
	4.3.2.2 Calibrations Tab	4-5
	4.3.3 Wizard	4-6
	4.3.4 Measurements	4-8
	4.3.4.1 Thermal Expansion	4-8
	4.3.4.2 Magnetostriction	
	4.3.4.3 Continuous	4-10
	4.3.4.4 Sequence	
	4.3.5 Measurement Data	4-10
	4.3.5.1 Calculating New Cell Constant	
	4.3.6 Baselines	
	4.5./ Creating Views	
	4.5./.1 I nermal Expansion View	
	4.5./.2 Magnetostriction View	
	4.5.7.5 New Dilation view	
	T.J.O VIEWS	

4.4	Analysis I	Models	4-22
	4.4.1	Tool Chain	4-22
	4.4.1	.1 Capture	4-22
	4.4.1	.2 Slicer	4-23
	4.4.1	.3 Filter	4-23
	4.4.1	.4 Transform	4-23
	4.4.1	.5 Merge	4-23
СНА	PTER 5.		5-1
Calil	oration		5-1
5.1	Introducti	ion	5-1
5.2	Measuren	nent Correction	5-1
	5.2.1	Thermal Expansion Measurement Background	5-1
	5.2.2 H	Reference Tables	5-2
	5.2.3	The Cell Constant	5-3
CHA	PTER 6.		6-1

Thermal Expansion Measurements		6-1
6.1	Introduction	6-1
6.2	Overview of Thermal Expansion Measurements	6-1
6.3	Performing a Measurement	
	6.3.1 Mount the Sample and Prepare the System	
	6.3.2 Run the Thermal Expansion Measurement	6-3
	6.3.2.1 Aborting a Measurement	6-4
	6.3.3 Probe Removal Procedure by Wizard	6-4

CHA	PTER 7	7-1
Mag	netostriction Measurements	7-1
7.1	Introduction	7-1
7.2	Overview of Magnetostriction Measurements	7-1
7.3	Performing a Measurement	7-2
	7.3.1 Mount the Sample and Prepare the System	7-2
	7.3.1.1 Selecting and Changing the Cell Angle	7-3
	7.3.2 Run the Magnetostriction Measurement	7-4
	7.3.2.1 Aborting a Measurement	7-5
	7.3.3 Probe Removal Procedure by Wizard	7-5

СНА	PTER 8	3	8-1
Troul	bleshoo	ting and Maintenance	.8-1
8.1	Introduc	tion	.8-1
8.2	Contam	ination	.8-1
	8.2.1	Cleaning the Cell and Capsule	.8-2
	8.2.2	Maintaining the Sample Chamber	.8-2
8.3	Commo	n Mistakes and Interesting Results	.8-3
	8.3.1	Moisture in the Sample Chamber	.8-3
	8.3.2	Sample Falling Out	.8-4
8.4	Troubles	shooting Electrical Connections	.8-4
8.5	Broken	Cell	.8-5
	8.5.1	Quantum Design Repairs	.8-5

APF	PENDIX A	A-1
Widg	gets	A-1
A.1	Introduction	A-1
A.2	Dilatometer Capture Widget	A-1
	A.2.1 Overview	A-1
	A.2.2 Configuration File	A-1
	A.2.3 Command Line	A-2
	A.2.3.1 Starting The Widget	
	A.2.3.1.1 Parameters	
	A.2.3.2 Running The Widget	A-2
A.3	Dilatometer Slicer Widget.	A-3
	A 3 1 Overview	A-3
	A 3.2 Configuration File	A-3
	A 3 3 Command Line	A-4
	A 3 3 1 Starting The Widget	A-4
	A 3 3 1 1 Parameters	A-4
	A 3 3 2 Running The Widget	A-4
A 4	Generic Transform Widget	A-5
1 1. 1	A 4 1 Overview	A-5
	A 4.2 Configuration File	A-5
	A 4 2 1 Params Section	A-6
	A 4 2 2 The Register	A-6
	A 4 2 2 1 Row Register	A-6
	A 4 2 2 2 Parameters:	A-7
	A 4 2 2 3 Default Row Register (init for passthrough param)	A-7
	A 4 2 2 4 Global Register	A-7
	A 4 2 2 5 Parameters:	A-8
	A 4 2 2 6 Identifying the Comment Column	A-8
	A 4 2 3 The Transform Command Tree	A-8
	A 4.3 Command Line	Δ_9
	A 4 3 1 Starting The Widget	A-9
	$\Delta 4311$ Parameters	A-10
	A 4 3 2 Running The Widget	A-10
Δ 5	Generic Filter Widget	A-11
11.0	A 5.1 Overview	A-11
	A 5.2 Configuration File	A-11
	A 5 2 1 Global Parameters	A-11
	A 5.2.2 The Output Section Definitions	A-12
	A 5.3 Command Line	A-13
	A 5 3 1 Starting The Widget	A-13
	Δ 5 3 1 1 Parameters	A-13
	A 5 3 2 Running The Widget	Δ_13
A 6	General Merge Widget	Δ_14
11.0	A 6.1 Overview	A_14
	A 6.2 Configuration File	Δ_14
	A 6 2 1 Global Parameters	Λ_1/
	A 6.3 Command Line	Δ_15
	A 6 3 1 Starting The Widget	Λ_15
	A 6 3 1 1 Parameters	Δ_15
	A 6 3 2 Running The Widget	Δ_15

References
------------

# **Table of Figures**

Figure 1-1: Material Expansion	
Figure 1-2: Cell Circuit Diagram	
Figure 1-3: Drive Circuit Diagram	
Figure 2.1. Dilatometer CAN Madula Front Dista	2.2
Figure 2-1: Dilatometer CAN Module Front Plate	
Figure 2-2: Dilatometer Cable	
Figure 2-3: Sample Length Boundary	
Figure 2-4: Grinding Setup Diagram	
Figure 2-5: Cell with Kapton	
Figure 2-6: Mounted Sample with Kapton	
Figure 2-7: Mounted Sample	
Figure 2-8: Kapton Gap Cross-section View	
Figure 2-9: Probe Installation Wizard Open Chamber	
Figure 2-10: Probe Installation Wizard Purging the Chamber	
Figure 2-11: Probe Installation Wizard Project and Properties	
Figure 2-12: Probe Installation Wizard Probe Test	
Figure 3-1: Cable Pin-outs (as seen from exterior)	3_2
Figure 3-2: Probe Head Exploded View	3_3
Figure 3.3: Cell Cansule Radiation Shield Rase Screw	3_4
Figure 3-4: Cell Hinges	3_4
Figure 3.5: Cell Ablation and Wiring Diagram	3_5
Figure 3-6: Cell Serial Number Location	
Figure 3-9. Cell Serial Number Location	
Figure 5-7. Balance Meter	
Figure 4-1: Dilatometer Option Controller Main Dialog	
Figure 4-2: Changing the Current Project	
Figure 4-3: Project Tab	
Figure 4-4: Measurement Configuration	
Figure 4-5: Baseline Tables Configurations	
Figure 4-6: Views Configurations	
Figure 4-7: Calibrations Tab	
Figure 4-8: Cell Calibration	
Figure 4-9: Probe Calibration	
Figure 4-10: Measurement Status	
Figure 4-11: Entering Sample and Cell Properties Window	
Figure 4-12: Probe Test	
Figure 4-13: Measure	
Figure 4-14: Thermal Expansion Measurement	
Figure 4-15: Magnetostriction Measurement	
Figure 4-16: Continuous Measurement	
Figure 4-17: Sequence Measurement	
Figure 4-18: Measurement Regions	
Figure 4-19: Calculating a New Cell Constant	
Figure 4-20: Baselines	
G	

Figure 4-21:	Creating a Thermal Expansion Baseline Table	
Figure 4-22:	Creating a Magnetostriction Baseline Table	
Figure 4-23:	Baseline Configuration	4-14
Figure 4-24:	Create a View	
Figure 4-25:	Creating a Thermal Expansion Coefficient View	
Figure 4-26:	Creating a Magnetostriction Coefficient View	
Figure 4-27:	Creating a Dilation View	4-19
Figure 4-28:	View Header	
Figure 4-29:	View File Properties	
Figure 4-30:	Merged View	
Figure 6-1:	Thermal Expansion Measurement (vs T)	
Figure 7-1:	Selecting the Cell Angle	
Figure 7-2:	Accessing the Angle Set Screw	
Figure 7-3:	Magnetostriction Measurement (vs H)	
Figure 8-1:	Evidence of Contamination	
Figure 8-2:	Thermal Expansion Data with a Water Peak	
Figure 8-3	Measurement Data with Sample Falling Out	8-4
Figure 8-4:	Probe Connections Diagram from cable view and capsule view, respectively)	

# **Table of Tables**

Table 0-1: List of Components to be Removed Before Recycling or Disposal.	xvi
Table 2-1: Expected Imbalance Change from Cell Mounting Orientation	2-9
Table 3-1: Cable Pin-out DescriptionsTable 3-2: Dilatometer Part NumbersTable 3-3: Dilatometer User Kit Part Numbers	3-2 3-6 3-6

### P R E F A C E

# **Contents and Conventions**

# P.1 Introduction

This preface contains the following information.

- Section P.2 discusses the overall scope of the manual.
- Section P.3 briefly summarizes the contents of the manual.
- Section P.4 illustrates and describes conventions that appear in the manual.
- Section P.5 describes the safety guidelines and regulatory information in the manual
- Section P.6 contains disposal information
- Section P.7 contains information on how to contact your Quantum Design service representative.

## **P.2** Scope of the Manual

This manual discusses the Quantum Design Dilatometer Measurement System for the Physical Property Measurement System (PPMS) and DynaCool<sup>TM</sup> platforms. It contains information about basic functionality, describes the system hardware, and describes the control software.

For detailed information about the PPMS MultiVu application, which is the software running the PPMS family of instruments, refer to the *Physical Property Measurement System: PPMS MultiVu Application User's Manual* 

# **P.3** Contents of the Manual

- Chapter 1 provides an overview of the Dilatometer Measurement Option: its purpose, design, and capabilities.
- Chapter 2 explains the Dilatometer Option installation and describes how to prepare for a measurement.
- Chapter 3 provides details on the Dilatometer hardware
- Chapter 4 discusses the Dilatometer Software, explains how to use it, and briefly describes the theory behind it.
- Chapter 5 discusses the Dilatometer cell calibration and measurement correction factors.

- Chapter 5 discusses the Dilatometer cell calibration and measurement correction factors.
- Chapter 6 explains how to perform thermal expansion measurements
- Chapter 7 explains how to perform magnetostriction measurement.
- Chapter 8 discusses issues that may arise in the system hardware and how to keep the Dilatometer system in good condition.

# **P.4** Conventions in the Manual

File menu	<b>Bold</b> text distinguishes the names of menus, dialogs, options, buttons, and panels used in the software.
File>>Open	The >> symbol indicates that you select multiple, nested software options.
.dat	The Courier font indicates files, directory names and computer code.
Important	Text is set off in this manner to signal essential information that is directly related to the completion of a task.
Note	Text is set off in this manner to signal supplementary information about the current task; the information may primarily apply in special circumstances.

### CAUTION!

Text is set off in this manner to signal conditions that could result in loss of information or damage to equipment.



### WARNING!

This symbol signals specific caution or conditions that could result in system damage, bodily harm or loss of life.



#### ELECTRIC SHOCK!

This symbol signals electrical hazards that could result in bodily harm or loss of life. Used at all accessible 200-230 V and 380-408 V power outlets.



#### WARNING!

This symbol signals **cryogenic hazards** that could result in bodily harm and loss of life. Used wherever accessible parts could reach temperatures below 0°C (32°F).



#### PROTECTIVE CONDUCTOR TERMINAL

The protective conductor terminal symbol in the left figure identifies the location of the bonding terminal, which is bonded to conductive accessible parts of the enclosure for safety purposes.



#### EUROPEAN UNION CE MARK

The presence of the CE Mark on the equipment signifies that it has been designed, tested and certified as complying with all applicable European Union (CE) regulations and recommendations.



#### ALTERNATING VOLTAGE SYMBOL

This international symbol indicates an alternating voltage or current.



### STANDBY SYMBOL

The power standby symbol indicates a sleep mode or low power state. The switch does not fully disconnect the device from its power supply, depressing the button switches between on and standby.

# **P.5** Safety Guidelines and Regulatory Information

Before using this product, please read the entire content of this User's Manual and observe all instructions, warnings and cautions. These are provided to help you understand how to safely and properly use the Dilatometer Measurement Option and reach its best performance.

Quantum Design Inc. disclaims any liability for damage to the system or injury resulting from misuse or improper operation of the system.



WARNING!

If the equipment is used in a manner not specified by the manufacturer, the protection provided by the equipment may be impaired.

Do not position the equipment so that it is difficult to operate or disconnect the device.

### P.5.1 Inspection for Damage

The Dilatometer Measurement option is carefully packaged at the factory to minimize the possibility of damage during shipping. Inspect the box for external signs of damage or mishandling. Inspect the contents for damage. If there is visible damage to the instrument upon receipt, inform the shipping company and Quantum Design immediately.



### WARNING!

Do not attempt to operate this equipment if there is evidence of shipping damage or you suspect the unit is damaged. Damaged equipment may present additional hazards. Contact Quantum Design technical support for advice before attempting to power on and operate damaged equipment.

### P.5.2 Electricity

In case of emergency, switch off the breaker on the back of the pump cabinet labeled "MAIN PWR" and the breaker on the compressor. If your system is a cryocooled instrument, switch off the main breaker on the compressor.

Observe the following safety guidelines when you use your system:

- To prevent electrical shock, unplug the system before you install it, adjust it, or service it. Permit only qualified electricians or Quantum Design personnel to open electrical enclosures and perform electrical servicing and checks. In cryocooled instruments, to prevent electrical shock, disconnect the compressor from the power before you install it, adjust it or service it.
- Keep electrical cords in good working condition, and replace frayed and damaged cords.
- For continued protection against fire hazard, electric shock and irreversible system damage, replace fuses only with same type and rating of fuses for selected line voltage.
- In general, keep liquids away from the PPMS and Dilatometer equipment.
- Keep the PPMS and Dilatometer hardware away from radiators and heat sources. Be sure to follow circuit breaker specifications outlined in the PPMS manual.

## P.5.3 Magnet Safety

Observe the following precautions for magnet safety. Also make certain you review the material in Chapter 1 in the *Physical Property Measurement System: Hardware Manual*.

### WARNING!

Large magnetic fields are dangerous to anyone wearing a pacemaker or other electrical medical device. Make certain any person wearing a pacemaker or similar device stays at least 3.0–4.5 m (10–15 ft.) from the PPMS Dewar whenever the PPMS superconducting magnet is charged.



#### WARNING!

Large magnets, such as the PPMS superconducting magnets, can attract iron and other ferromagnetic materials with great force. Keep all iron, nickel, and other ferromagnetic objects at least 5.0 m (16.5 ft.) from the PPMS Dewar.

The observable effects of magnetic fields are listed in chapter 1 in the *Physical Property Measurement System: Hardware Manual.* 

### P.5.4 Cryogenic Safety

When you work with cryogenic materials such as liquid helium and liquid nitrogen, keep in mind that (1) they can expand when subjected to room temperature and (2) they can burn.



### WARNING!

The extreme cold of liquid and gaseous cryogens can cause serious burns. Always wear protective clothing, including thermal gloves, eye protection and covered shoes when you work with liquid helium, liquid nitrogen or any other cryogen.



#### WARNING!

In a poorly ventilated room, helium can displace air and lead to asphyxiation. Always perform cryogen transfers in a well-ventilated room. In the event of a rupture or spill, vent the room immediately and evacuate all personnel.

Cryogens stored in confined spaces are subject to extremely high pressure buildup, resulting in dangerous explosions. Pressure relief valves are installed on the PPMS Dewar and cooling annulus, and rupture disks are installed on the vacuum sleeve and Dewar to eliminate the possibility of explosion. Do not remove, disable, or otherwise tamper with these safety devices.

# P.6 Disposal Information

The Dilatometer Measurement Option is excluded from the requirements of:

• DIRECTIVE 2011/65/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 8 June 2011 on the restriction of the use of certain hazardous substances in electrical and electronic equipment (RoHS 2)

The Dilatometer Measurement Option complies with the requirements of:

 DIRECTIVE 2012/19/EU OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL of 4 July 2012 on waste electrical and electronic equipment (WEEE)

### P.6.1 RoHS Statement

The Dilatometer Measurement Option is explicitly excluded from the Recast RoHS Directive in Article 2(4)(f) which covers: *"equipment specifically designed solely for the purpose of research and development, only made available on a business-to-business basis"*.

### P.6.2 WEEE Statement

WEEE Statement: Dilatometer Measurement Option is WEEE compliant, as Quantum Design uses recyclable materials in the fabrication of the equipment. Several components require special handling and processing, and these components must be removed at time of decommissioning for proper handling before recycling/disposal. Contact Quantum Design for updated procedure/recommendations before disposal.

<b>Component Description</b>	Identifying Highlighted Image	
Dilatometer Module		

<i>Table 0-1:</i>	List of C	Components	to be	Removed	Before	Recycling	or	Disposal.
	····· · · · · · · · · · · · · · · · ·	- · · · · · · · · · · ·			· · · ·			r

<b>Component Description</b>	Identifying Highlighted Image
Pre-amp Board and Cell interface boards	
Dilatometer Cell connectivity boards	

# P.7 Contacting Quantum Design

If you have trouble with your Dilatometer Measurement Option or your system, please contact your local Quantum Design service representative for assistance. See www.qdusa.com for the information about your local representative. You will be asked to describe the problem, the circumstances involved and the recent history of your system.

# **Overview of the Dilatometer System**

## **1.1** Introduction

This chapter contains the following information.

- Section 1.2 describes what the Dilatometer Option measures.
- Section 1.3 discusses the purpose of measuring thermal expansion and magnetostriction.
- Section 1.4 explains the theory of operation for the Dilatometer Option.
- Section 1.5 discusses the range of samples that can be measured and special features of the option.

# **1.2** What the System Measures

The Dilatometer Option is used to measure the change in length, or dilation, of a sample, and the temperature derivative of dilation, called the linear thermal expansion coefficient. Dilation is expressed as fractional total length change referenced to a fixed temperature, typically 300 K.



Figure 1-1: Material Expansion

$$\frac{\Delta L(T)}{L(300 \text{ K})} = \frac{L(T) - L(300 \text{ K})}{L(300 \text{ K})}$$
(1.1)

The quantity of interest for most samples is the linear thermal expansion coefficient  $\alpha$ , which is the fractional length change for an infinitesimally small temperature change.

$$\alpha = \frac{1}{L(300 K)} \frac{dL(T)}{dT}$$
(1.2)

Strictly speaking, L(300 K) in equation 1.2 should be replaced with L(T), but typical dilation values are low enough for this distinction to be quite small.

Dilatometer Option User's Manual Rev. A0	Quantum Design
February 2017	

In addition, the dilatometer measures magnetostriction, the field derivative of the magnetostrain of a sample. Similar to dilations, the magnetostrain is typically expressed as fractional total length change referenced to a zero applied magnetic field.

$$\frac{\Delta L(H)}{L(0\ 0e)} = \frac{L(H) - L(0\ 0e)}{L(0\ 0e)}$$
(1.3)

Another quantity of interest is the magnetostriction coefficient  $\lambda$ , which is the fractional length change for an infinitesimally small applied magnetic field change.

$$\lambda = \frac{1}{L(0\ 0e)} \frac{dL(H)}{dH} \tag{1.4}$$

# **1.3** Purpose of the Dilatometer Measurements

Understanding the thermal expansion and magnetostriction of materials has applications in a number of scientific fields.

In engineering, the most dramatic example is known as railroad buckling. Year after year, the iron rails undergo the heat waves of summer and freezes of winter, resulting in deformations in the track and ultimately derailed cars. Very similar problems arise in pipelines and interstate roadways, making precisely sized and placed joints essential. The aerospace industry studies materials like Invar for optimal performance over a wide range of temperatures. Materials with well characterized magnetostriction on the other hand are widely used in sensors, actuators, and other electromechanical devices.

Material science and crystallography will benefit significantly from the Dilatometer measurements. Detailed below are just a few of its uses in these fields.

- The thermal expansion coefficient can help locate and characterize phase transitions. In some cases, these are phase transitions that cannot be seen by other methods such as heat capacity, magnetism or resistance measurements. With this information one can begin to isolate various properties and their dependence on electrical properties and density.
- The thermal expansion coefficient is a dominant factor in some representations of the Grüneisen parameter. This parameter provides better insight into the vibrational properties of materials.
- Characterizing the thermal expansion and magnetostriction of complex materials such as heavy fermions will help give insight into their unique physical and electrical properties.
- Characterizing the volumetric expansion of a material can be used in the study of the Ehrenfest Theorem in quantum mechanics.

# **1.4** Theory of Operation

### 1.4.1 Cell Circuit

The cell circuit is composed of two capacitors in series with an in-phase voltage detector, VD, between them and driven by a series of transformers (Figure 1-2.). The system is based on a comparative measurement between the two capacitors, specifically a ratio of their elastances. This ratio removes many measurement dependencies such as temperature and frequency. The circuit is designed such that dilation is directly proportionate to a displacement value, x, and the following relationships hold.



Figure 1-2: Cell Circuit Diagram

$$C_A = \frac{\varepsilon A}{d+x} \text{ and } C_B = \frac{\varepsilon A}{d-x}$$
 (1.5)

Furthermore, if V\_A=U+ $\Delta$ U and V\_B=-U+ $\Delta$ U, it can be shown that,

$$V_{Detector} = U\left(\frac{\Delta U}{U} - \frac{x}{d}\right) \tag{1.6}$$

By controlling the input AC voltage, it is possible to balance the capacitance bridge such that  $V_{Detector} = 0$  and solve for x using equation 1.7. This equation can also be expressed in terms of the balanced capacitances,  $C_A$  and  $C_B$ . Note that due to cell geometry, the actual expansion is given by equation 1.8.

$$\frac{\Delta U}{U} = \frac{x}{d} \quad and \quad \frac{\Delta U}{U} = \frac{C_B - C_A}{C_B + C_A} \tag{1.7}$$

$$\Delta L = \frac{x}{\cos\left(\frac{\pi}{6}\right)} \tag{1.8}$$

As the system temperature changes and the sample dilates, a PID control loop maintains  $V_{Detector} = 0$ , as will be discussed in Section 1.4.3. This ratio, presented in equation 1.7, is known as imbalance, a dimensionless parameter. It is an indication of the efforts going towards balancing the circuit.

Dilatometer Option U	ser's Manual Rev. A0
Februar	ry 2017

Changing the imbalance is discussed in the following section, Section 1.4.2, and using the imbalance is discussed in Section 5.2.3.

The importance of maintaining  $V_{Detector} = 0$  can be seen when stray capacitance is accounted for in the system. Stay capacitance arises in the coaxial cables while carrying the signal from the voltage detector to the preamplifier in the probe. This can be seen in Figure 1-2. labeled C<sub>s</sub>.

$$V_{balance} = U\left(\frac{\Delta U}{U} - \frac{x}{d}\right)\gamma : \gamma = \frac{1}{1 + \frac{1}{2}\left[1 - \left(\frac{x}{d}\right)^2\right]\frac{C_s}{C_{1/2}}} and C_{1/2} = \frac{\varepsilon A}{d}$$
(1.9)

For small displacements,  $\left(\frac{x}{d} \ll 1\right)$ , this correction results in the signal being reduced by about a factor of 4 due to  $C_{S..}$  The dependence on  $\frac{x}{d}$  in any correction factor means that it would need to be calculated with every measurement. Operating at  $V_{Detector} = 0$  eliminates  $\gamma$  from the imbalance and significantly simplifies the measurement.

Understanding  $C_S$  and  $C_{1/2}$  further shows just how important it is to eliminate  $\gamma$  from the measurement. First,  $C_S$  is temperature dependent and the coaxial cable has a complex and always changed temperature profile due to gradients in the probe. Second,  $C_{1/2}$  depends on the electric permeability of gas between the cell gaps. To calculate this would require temperature, pressure and impurities information at minimum. In addition,  $C_S$  and  $C_{1/2}$  are related in a non-linear equation. Thus, the Dilatometer Option is designed to operate at  $V_{Detector} = 0$ .

### **1.4.2** AC Drive Voltage and Imbalance

As previously mentioned the cell circuit voltages  $V_A = U + \Delta U$  and  $V_B = -U + \Delta U$  are adjusted in order to maintain a balance point in the circuit's voltage divider. The voltage is adjusted by both tap changes in the main transformer and changes to a digital-to-analog converter (DAC).



Figure 1-3: Drive Circuit Diagram

The main transformer has variable turns and adjusts  $\Delta U$  through tap changes. These tap changes are controlled by a relay device in the Dilatometer module. By design, each tap change on MA is equal to the tap change needed for MB in order to maintain equation 1.7 (Figure 1-3.).

The drive signal has a frequency of 2.34 kHz and operates at a constant voltage. The module maintains  $V_A - V_B = 2U$ , whatever U may be, also in order to maintain the necessary conditions for equation 1.7.

In order to measure to within a high degree of accuracy, the trim transformer is designed with a bifilar coil. This technique ensures that the windings trap the same amount of flux, and it greatly reduces sensitivity to effects like flux leakage and drift due to temperature.

The following equations represent the relationships between tap/DAC counts and imbalance. The drive imbalance represented by one tap and one DAC count are as follows.

$$I_{DAC} \approx 7.947 \times 10^{-9} \tag{1.10}$$

$$I_{Tap} \approx 0.781 \times 10^{-2} \tag{1.11}$$

Note:

In order to begin the measurement process in balance, the Dilatometer firmware runs a binary search algorithm to find the correct initial imbalance setting.

### 1.4.3 Signal Processing

Accurately reading the signal coming from the capacitance bridge in the cell circuit is essential for adjusting the drive circuit to remain in balance and calculate sample expansion. In order to do this a chain of electronics first prepares the signal. This all works within a PID control loop that is not tuned or controlled by the user.

**Note:** There is a small amount of following error in the PID loop.

The signal is amplified as close to its source as possible. This occurs in the probe head. The signal is then amplified and prepared to be sent across the cable to the module. In the module, the signal is demodulated and read by the firmware in order to calculate the necessary adjustments to the drive signal.

### **1.4.4** Thermometry

To accurately measure the sample temperature, a thermometer is mounted in the capsule. The resistance is measured relative to a series of calibration resistors. Unlike other Quantum Design products, the resistance calibration is performed for each individual measurement. This both avoids the step function effects of a time interval calibration and completely eliminates system drift from the temperature data. The measured resistance value is then compared to a Quantum Design calibration baseline table and converted to a temperature value. Interpolation is used to provide the software with a continuous reference for calculating temperature values.

# **1.5** Scope of the Dilatometer Option

### **1.5.1** Sample Thermal and Magnetic Characteristics

The Dilatometer Option has the ability to measure any sample that is solid at room temperature; independent of its electrical and magnetic properties.

### 1.5.2 Sample Size

The sample size has three dimensions, each with different constraints (Figure 1-4.). For reference, X marks the side that will rest against the sides of the cell grooves. The measurement axis, A, must be  $2 \pm 0.05$ mm and is based on cell geometry. The axis perpendicular to the measurement axis, B, must be less than 3 mm. Reasons for this distance and how to create both A and B are detailed in Section 2.4.1. The third axis, C, should be less than 3 mm and is also based on cell geometry.

### **1.5.3** Temperature and Magnetic Field Range

The Dilatometer Option has no specific temperature or magnetic field range of its own. The range is limited by the PPMS system which is designed to operate from system base temperature to 400 K and 0 Oe – 160,000 Oe, depending on the model. This product is not designed to be compatible with the Quantum Design He-3 or DR inserts and therefore cannot go lower than 1.8 K.

Special Features

- This is the first commercially available fused silica dilatometer.
- Changes in sample length are measured directly as opposed to inferring change from a capacitance measurement.
- The user may manually rotate the cell outside of the cryostat to vary the angle between the sample axis and the magnetic field.
- Samples can be mounted in different orientations to handle sample anisotropy.
- The fused silica cell eliminates eddy currents and minimizes measurement background.
- A grounded sample configuration and fully shielded cell remove any effect the sample would have on the measurement.
- All raw data is saved and can be easily accessed. This allows for experimenting with the processing parameters many times on the same data set.
- The Dilatometer has the capacity to perform both thermal expansion and magnetostriction measurements. In fact, both measurements can be performed back-to-back without changing the sample.

# **Installation and Getting Started**

## 2.1 Introduction

This chapter contains the following information.

- Section 2.2 lists system requirements for the Dilatometer option.
- Section 2.3 explains how to install the dilatometer hardware and software.
- Section 2.4 explains the process of creating, cleaning, and mounting a sample for measurement.
- Section 2.5 describes the purpose of the Balance Meter and explains how it is operated.
- Section 2.6 explains how to use the software wizard to finish preparing for a measurement.

## 2.2 System Requirements

- Computer running a current version of the MultiVu software. Current versions can be found at www.qdusa.com.
- The Dilatometer Option is compatible with the Physical Property Measurement System (PPMS), EverCool<sup>TM</sup>, EverCool II<sup>TM</sup> and DynaCool<sup>TM</sup>.

# 2.3 Installing the Hardware and Software

### 2.3.1 Installing the Dilatometer Control Electronics and Cable

To begin installing the Dilatometer Option, the module and computer need to be in communication. For further instruction on installing the module and opening communication with the computer, see the appropriate user's manual for the specific model of module tower.



Figure 2-1: Dilatometer CAN Module Front Plate

#### 2.3.1.1 INSTALL THE MODEL CM-R DILATOMETER MODULE

The control electronics for the Dilatometer Option are contained in the Model CM-R Dilatometer Module. On the PPMS system, find room for the Dilatometer module in the CAN tower. The Dilatometer module does not have any specific cooling or power requirements and can operate in any available module bay. The following instructions ensure safe installation of the module for both the user and the electronics.

- 1. Turn off the power to the module tower.
- 2. Remove the cover plate from one of the unused module bays and insert the Model CM-R Dilatometer Module into the empty bay.
- 3. Once the module is inserted and the mounting thumbscrews screwed in, turn on the power to the tower. Once the self-test is complete, the COP LED will be solid or blinking green. The PWR LED will blink alternating between red and green until the Dilatometer Option Controller is opened in MultiVu. This indicates a normal startup.



#### WARNING!

Hot plugging the Model CM-R Dilatometer Module will destroy the internal circuit. Please follow the instructions above every time you install or remove a module from the module tower.

#### 2.3.1.2 CONNECT THE DILATOMETER CABLE



Figure 2-2: Dilatometer Cable

Connect the DB-25 cable end to the Dilatometer module, JR-1, as denoted on the front panel. Once the probe is installed as detailed in Section 2.6, the other end of the cable can be attached to the probe head. Both the probe head and the cable have a red dot that line up to indicate the proper orientation of the plug.

**Note:** Note the controller cable may be plugged and unplugged at any time (hot plugging) to both the probe and the module.

When unplugging the cable, pull by the collar and not by the cord. This releases a locking mechanism and allows the cable to unplug.

### 2.3.2 Installing the Software

The software may already be installed on your PC. If it is not, or if it is an older version, you should obtain installation CDs or download the appropriate installers from the Quantum Design website (www.qdusa.com).

- 1. Install MultiVu version 1.5.11 or later on the target PC by running Setup.exe from the provided installation CDs or download package.
- 2. Install Dilatometer version 0.9.9.19 or later on the target PC.

If there are any previous versions of either MultiVu or Dilatometer software, these will be upgraded by the installer.

# 2.4 Sample Mounting

**Important:** Read this section carefully and in its entirety before using the Dilatometer Option. Understanding each step as well as the next step will be crucial in successfully preparing for a measurement.

### 2.4.1 Preparing the Sample

The dimensions for the sample are defined by (a) the ideal width of the sample groove and (b) the distance between gaps on the sides of the groove.

a. The ideal width of the groove is that which a sample will cause the cell imbalance to be zero. This gives maximum range of measurement for both positive and negative expansion. This value is roughly 2 mm. In order to achieve this, the user must make the side of the sample that is to be measured either 2 mm or sum to 2 mm when used in combination with a fused silica spacer. The thick and thin provided spacers are roughly 1.52 mm and .33 mm along their



Figure 2-3: Sample Length Boundary

measuring axis, respectively. Although the 2 mm goal is important, as long as the cell passes the Balance Meter check it is acceptable for measurement. Refer to Section 2.5.2.1 for more information on acceptable imbalance values.

- b. To avoid sample shift during measurement, it is important that the sample only touches two sides of the cell; one on each half. Keeping it between the lines indicated on Figure 2-3 will ensure this. The distance is roughly 3 mm and creates a physical upper bound on sample length.
  - **Note:** The sample will not touch the bottom of the sample groove. This will be shown in the mounting process in Section 2.4.3.

The following steps describe the process of creating a sample.

- 1. Use a wire saw or the preferred cutting method for your sample to give the cell its rough dimensions; slightly larger than 2 mm width, 3 mm maximum length, and 3 mm maximum height.
- 2. If the sample cannot achieve the width of greater than 2 mm, see instruction later on in the section concerning spacers.
- 3. Now that the sample has been cut, a grinding process will be used to create flat and squared surfaces for measurement. Provided in the user kit are two lapping tools, .081-inches and .079-inches. First use the .081-inch tool to create a flat side on one end of the measurement axis. Remove the sample and use the .079-inch tool to grind the *opposite side*. This leaves two parallel surfaces with the optimal 2 mm distance between them. These are the two surfaces that will contact the cell. See instruction later on in the section for using the lapping tools.
- 4. Once the sample has been ground to the exact length, it is ready to be cleaned and then mounted.

The grinding process requires lapping tools, sandpaper, CrystalBond<sup>TM</sup> 509, and a hot plate. The following steps describe the process of grinding one side of a sample.

- 1. Using a hot plate, heat the lapping tool to 123°C.
- 2. Place the sample inside the groove of the lapping tool with the side to be ground facing upwards. The side in contact with the lapping tool must be already relatively flat.
- 3. Apply pressure to the top of the sample with the bare end of a Q-tip. The purpose is to keep CrystalBond out from between the lapping surface and the sample. See Figure 2-4. for a similar example, but with a spacer included.
- 4. Apply CrystalBond to the sample by melting it around the base of the sample. This will secure it to the lapping tool.
- 5. Remove the lapping tool from the hot plate, and wait for the tool to cool and the wax to harden.
- 6. Once at room temperature, turn the lapping tool upside down and rub it against sandpaper, grinding the sample to the desired length. Be sure to grind gently, so as to not stress the adhesive seal, and always grind in a figure-eight motion.
- 7. Once the sample is no longer in contact with the sandpaper, tu, and then remove the sample.
- 8. In order to remove all of the CrystalBond, place the sample in a container and submerge the sample in acetone. Place the container in an ultrasonic bath for 5 minutes. Visually inspect the sample under a microscope and repeat this step if wax remains.
  - **Note:** If the sample reacts with acetone, contact Quantum Design for assistance. Another means will need to be found to secure the sample to the lapping tool.

It is important to notice that using the lapping tool on sandpaper will also slowly wear away at the tool itself. On the side of a tool is its initial height marked in inches. Eventually the tool will need to be replaced when the actual height strays too far from this number.

In the case that the sample is less than 2 mm a spacer will need to be used. The spacers are made from the same fused silica as the cell and thus have negligible effects on the calibration values. The following steps describe the process of creating a sample with a spacer included.

1. The first grinding, using the .081-inch lapping tool, will actually grind the spacer. For this process the sample must be flat on both sides. Place the sample at the base of the groove in

the lapping tool and place the spacer on top of it. The spacer must be large enough that its height on top of the sample is greater than the tool surrounding it (indicated on Figure 2-4.).

2. Follow the same steps as without a spacer to finish the process, however, when using the .079-inch lapping tool the spacer will be on bottom and the sample surface will be ground.



Figure 2-4: Grinding Setup Diagram

### 2.4.2 Cleaning the Sample

Contaminants on the sample and cell are very detrimental to the quality of the measurement. For this reason, cleaning the cell, sample, Kapton, tweezers, spacer and spring prior to mounting the sample is extremely important. Once the cleaning process has begun, only handle the cell and materials while wearing gloves. The following steps will ensure all of the necessary tools are ready to proceed on to the mounting process. If at any point the tools touch a surface that is unclean, start this process over.

Cleaning the Cell:

- 1. Remove all measurement tools from the cell. This includes the spring, Kapton, spacer and sample.
- 2. Fully submerge the cell in isopropanol and remove after 20-40 minutes.
- 3. Fully submerge the cell in methanol and remove after 20-40 minutes.
- 4. Gently spray N2 gas to dry the cell. Be sure to spray in the capacitance gaps as well as the gaps around the hinges.
- 5. Using a microscope, visually inspect the cell to make sure it is dry and contaminant free. If the cell is dry, it is ready to be used for a measurement.



#### WARNING!

Spraying  $N_2$  gas between the cell gaps is dangerous. If the gas is sprayed with too much pressure the hinges will break and destroy the cell.

Cleaning the Measurement Tools:

- 1. Using a clean container, submerge the spacer, Kapton, sample, spring and tweezer tips in acetone.
- 2. Place the container in an ultrasonic bath for 5 minutes. Be sure that the container is properly weighted and will not tip over during the bath. Once finished, remove the materials from the container.
- 3. Using a container, submerge the spacer, Kapton, sample, spring and tweezer tips in isopropanol.
- 4. Repeat step 2.
- 5. Using a container, submerge the spacer, Kapton, sample, spring and tweezer tips in methanol.
- 6. Repeat step 2.
- 7. Use N<sub>2</sub> gas to dry the materials. When drying the tweezers, be careful to blow from below the tips, upwards, as to not blow contaminants back towards the sterile portion. When drying the other materials, grip them firmly with the tweezers inside a metal tray to avoid them blowing away.

### 2.4.3 Mounting the Sample

Once the sample, cell, Kapton, spacer and spring have been cleaned, the sample is ready to be mounted. The Balance Meter will serve as a mounting station. Begin by connecting the capsule to the Balance Meter and tightening the securing clamp. In development, some have preferred mounting the sample on a well-lit table and others under a microscope. Before proceeding the user will need to know

Quantum Design

which they prefer. The following steps will ensure proper sample mounting.

One groove will hold the sample and the other the spring. Choose the groove you would like to hold the sample and lay the Kapton along its base. Note that changing the groove of the sample will result in a negative or positive sign change in the measurement, thus consistency is most convenient to the user. (See Table 2-1 in Section 2.5.2.1 for details on this sign change).

- 1. Note the sample groove the sample is mounted in. The user will be prompted to enter this into the option software. The grooves are denoted right and left, relative to the fixed half of the cell being on the bottom. For example, in Figure 2-5 the right (R) and left (L) grooves are in their proper orientation.
- 2. Place the sample in the groove, above the Kapton. The provided fused silica and copper samples have been cut so that the optimal side to measure is obvious. It is neither the longest nor the shortest side. It can be difficult to fit the sample into the groove. If this is the case, you may use the bare end of a Q-tip to gently open the cell and widen the gap.
- 3. The best method for inserting the spring is to pick it up by its closed end with the flat duck bill tweezers. Once it is held above the groove, opposite of the sample, use the pointed tweezers to compress the spring around its middle. Lower its open end into the groove without letting the pointed tweezers into the groove as well. Release tension from the pointed tweezers first, followed by the flat tweezers.
- 4. Slide the Kapton strip out from under the sample by pulling towards the bottom of the capsule. It is important that the gap created by the absence of Kapton remains as is. The best way to keep the sample from moving is to pull the Kapton in a sweeping, concave, up motion.



Figure 2-5: Cell with Kapton



Figure 2-6: Mounted Sample with Kapton



Figure 2-7: Mounted Sample

If the sample does move, increasing or decreasing the gap in any way, a correction needs to occur. It may be necessary

to return to step 1. If the sample is still fit inside the groove, it may be possible to slide the Kapton back underneath it. Gently press the sample down with the bare end of a Q-tip, and try removing the Kapton again.

With practice, sample mounting becomes routine. In the meantime, there are a few important mistakes to avoid.

• The surface of the cell is very fragile, especially at its corners. The tweezers will easily chip away the material and degrade the health of the cell. Be sure not to insert the tweezers into the grooves. Be sure to pinch the spring whenever moving it, as opposed to dragging it along the groove walls while compressed. Be sure to not drop the tweezers or anything else onto the cell.

- Using the wooden end of a Q-tip to open the cell is a very effective technique, however it must be done with caution. Dust particles and fibers, if present, have a large effect on the data's quality, and so using the bare end is essential. Dust and fibers can be removed through the cleaning process discussed in Section 2.4.2.
- The gap created beneath the sample by removing the Kapton should be consistent. A visual check that the top of the sample is parallel with the cell surface and there is a gap underneath the sample is sufficient. Figure 2-8 shows a cross-section of the sample mounted with this gap.



Figure 2-8: Kapton Gap Cross-section View

# 2.5 Balance Meter

The Balance Meter is designed to ensure the sample is mounted properly, the sample's expansion will not leave the bounds of the Dilatometer's ability to measure, and as a means to check the health of the capsule's thermometry. As discussed in Section 2.4.3, the Balance Meter also serves as the sample mounting station.

If the user is preparing for a magnetostriction measurement, the angle will be selected and changed while the capsule is in the Balance Meter. Refer to Section 7.3.1.1. for instructions on preparing the cell angle.

### 2.5.1 Imbalance

Imbalance is a unit-less measure of the steps taken to balance the capacitance bridge. Imbalance is defined as the ratio of the change in capacitance gap to the capacitance gap of the balanced system. It can also be defined as the ratio of the amplitude of the AC drive voltage to the average of that same drive voltage. Imbalance and the importance of operating at the balance point are thoroughly discussed in Section 1.4.

If the magnitude of the imbalance at room temperature is too high, the sample may expand beyond the system's ability to measure. The Balance Meter is used in order to avoid this very issue.

#### Quantum Design

### 2.5.2 Operation

In order for the Balance Meter to function properly, the capsule must be firmly connected at the base, and a sample must be mounted. To turn the Balance Meter on, press the black button to the right of the display screen. A reading will appear after a few seconds. The Balance Meter will turn off on its own after a few minutes. The Balance Meter uses one AA battery as a power source.

#### 2.5.2.1 UNDERSTANDING THE IMBALANCE READING

The Balance Meter operates in a similar way to the Dilatometer module concerning imbalance, however, the module is limited to  $\pm 0.5$  while the Balance Meter will read  $\pm 1$ . For this reason, any reading in the range of  $\pm .5$  is acceptable. It is convenient to the user that the Balance Meter reading ranges from  $\pm 1$ . In the case that the acceptable conditions are not met, there is some indication of how much the sample length needs to be adjusted. Although any initial imbalance in the range of  $\pm .5$  will be recognized by the module, the optimal value depends on the amount of expansion of the sample.

- If the sample expansion is unknown, the optimal initial imbalance is 0. This gives maximum range for reading the expansion in the case of either positive or negative movement.
- If the sample expansion is small, the user has a lot of freedom and need only stay clear of the boundaries of measurement for the initial imbalance reading.
- If the sample dilation is known to be large, the user will want to mount the sample in such a way that as the sample expands, it approaches and passes the zero imbalance point. This will give maximum range for dilation readings. To do this effectively, the difference between the cell grooves and the way in which the sample will expand must both be well understood. Refer to Table 2-1. For example, if a sample is known to have high positive dilation across a thermal range, it would be acceptable to mount said sample in the right cell groove with an initial imbalance of .3. As the sample expands, the imbalance will decrease and the user has a range of .8 to complete the measurement. Note that right and left are from the perspective of looking at the cell with the fixed half on bottom.

Cell Groove (fixed cell half on bottom)	Sample Change	Imbalance Change
Right (R)	Expand	Decrease (-)
Right (R)	Contract	Increase (+)
Left (L)	Expand	Increase (+)
Left (L)	Contract	Decrease (-)

Table 2-1: Expected Imbalance Change from Cell Mounting Orientation

#### 2.5.2.2 CHECK THERMOMETRY

Using an ohmmeter, you may check the thermometer resistance and confirm that it matches the room temperature value. This is a four-point measurement as to precisely measure the thermometer resistance and ignore contact and lead resistances. For more information on the thermometry, see

Section 1.4.4. Information on healthy thermometer readings and troubleshooting the thermometry can be found in Section 8.4.

# 2.6 **Preparing the System**

### 2.6.1 Radiation Shield

The radiation shield serves to create a more uniform and controllable temperature surrounding the cell. It also acts as a faraday cage for the measurement signal. Once a sample is mounted, the radiation shield slides onto the capsule, covering the cell. The base screw is then added to the bottom to secure the radiation shield and seal the capsule. Firmly tighten the base screw.

### 2.6.2 Capsule to Probe Connection

The capsule firmly connects at the end of the probe. Once the user is sure of a good electrical connection between the two, four screws are used to secure the connection throughout the measurement. Using the 5/64-inch Allen key, insert the four screws through the probe end and fasten them into the capsule. Be sure that the capsule is secure, but do not tighten the screws too firmly, due to the range in temperatures they will undergo during a measurement.

### 2.6.3 Navigating the Wizard

Once the capsule has been secured to the probe, the system will be almost ready to begin measurements. An installation wizard on the Dilatometer Option Controller will finish preparing the system.

Launch the MultiVu software on the computer and then activate the Dilatometer Option from the **Utilities >> Activate Option >> Dilatometer >> Activate** menu command.

In the upper left had corner of the main dialog of the Option Controller, select **Wizard**. The following instructions will direct the user through preparing the system through the wizard.

1. Select **Open Chamber**, shown in Figure 2-9. Once the chamber status is at 300 K and open, select **Next** >>.

Probe Installation	Wizard	X
	Chamber Status:	
	199.99K, stable, closed	
	Instructions:	
	Press "Open Chamber" to bring the chamber to room temperature and vent. Open Chamber	
	<< Back Next >> Cance	

Figure 2-9: Probe Installation Wizard Open Chamber

- 2. Remove the sample chamber baffle cap.
- 3. Gently slide the probe into the PPMS-system. Once fully inserted, lock the two black clamps on the probe head.



#### WARNING!

Many PPMS options use grease. Do **NOT** insert the probe into a chamber with grease. The sample chamber must be cleaned before using the Dilatometer Option.

- 4. Plug the Dilatometer cable into the probe head. There is a red dot on the cable connector and a matching red dot on the probe head. Make sure these two dots line up. Also, check that the DB-25 end of the cable has already been connected to the module at JR-1.
- 5. Select **Purge**, shown in Figure 2-10. Select **Next** >>.

robe Installa	ation Wizard	
	Chamber Status:	
	300K, stable, open	
	Instructions:	
	Chamber is now flooding. Insert the calorimeter puck into the sample chamber. Make sure the charcoal holder is installed on the baffles. Press "Purge" to purge the chamber before continuing. Purge	
	<< Back Next >>	Cancel

Figure 2-10: Probe Installation Wizard Purging the Chamber

6. Select a current project for the measurement using the **Project...** button.

- 7. All of the fields visible in Figure 2-11. are not editable. To edit them, select New. This will open a different window that is discussed at the beginning of Section 4.3. Fill out all of the necessary parameters. Use a sample name and comment that will make identifying the data for post
  - processing convenient. Enter the sample length in millimeters. The rotator angle should be entered in degrees. For more information on the rotator angle, see Section 7.3.1.1. Sample location is referring to the right or left sample grooves. Refer to Chapter 3 for finding both the cell and probe serial numbers. Once finished, select **Save New** to return to Figure 2-11. Make sure all of the information entered is correct, and select **Next** >>. If the information is incorrect, select New to reenter the parameters.

Probe Installation Wizard		
Current Project:	My First Project	Project
A Project is a folder locat Normally you would keep	ion that contains measurement data, views, and sample prope o the same project for one sample or a study of related sample	erties information. 15.
Sample and Cell Proper	ties:	New
Provide your sample and	cell properties here.	
Sample Name:		
Comments:	Comments for sample and cell prop	
Sample Length (mm):	5.4321	
Rotator Angle (deg):	0	
Sample Location:	R	
Cell Serial Number:		
Dilatometer Probe Serial Number:		
	<< Back Next >	Cancel

Figure 2-11: Probe Installation Wizard Project and

- 8. The final window in the wizard process, shown in Figure 2-12, displays system settings. This ensures that the probe and module electronics are working properly.
- 9. Select Finish.

Probe Installation Wizard			E
Probe Test			Testing Retest
This test makes sure the hardw selected thermometer calibrati	are of the Dilatometer pr on match expectations.	obe and electrics is working co	prrectly and that the
	Measured	Expected	
Cell Thermometer:			
Cell Imbalance:			
Dilatometer Bridge Status:			
Test Results:			* *
		< < Back Finish	Cancel

Figure 2-12: Probe Installation Wizard Probe Test
### CHAPTER 3

# Hardware

## 3.1 Introduction

This chapter contains the following information.

- Section 3.2 discusses and illustrates the Dilatometer hardware components.
- Section 3.3 provides Quantum Design part numbers for the individual components.

## **3.2** Dilatomer Hardware

### **3.2.1** Model CM-R Dilatometer Control Module

The Model CM-R Dilatometer Module contains electronics that simultaneously control the voltage applied to the capacitors, measure the imbalance signal and measure the cell temperature. Below is a brief description of import module features.

- The module houses the AC drive that supplies voltage to the cell parallel plate capacitors. This signal operates at 2.34 kHz.
- The read back signal comes from a balanced capacitance bridge in the cell.
- The module performs a tap search for this balance point before starting the measurement. It uses a PID loop to narrow control drive voltages and component gain for optimal measurement conditions.
- Imbalance and thermometry resistances are read by the firmware with a single time stamp.

The front panel of the module has two LED indicating lights labeled "PWR" and "COP." A green and red flashing PWR light indicates the module is plugged in properly to the power supply. The red light will stop once the Option Controller is opened. A green COP light indicates the module is properly connected to and talking with the computer. There are also two BNC-cable jacks on the front panel labeled "SIG" and "OSC," labeled JR-2 and JR-3 respectively. This feature enables the user to check system and measurement health with an oscilloscope. OSC is the drive signal. SIG is the unmodulated

signal coming directly from the Dilatometer cable. Under normal operation the user will not need to use these connectors. However, they are extremely helpful with troubleshooting issues. This is discussed in detail in Chapter 8.

### **3.2.2** Dilatometer Cable

The Dilatometer controller cable assembly carries the thermometer resistance values, imbalance signals and the AC drive voltage between the Dilatometer module and the cell. See Section 2.3.2.3 for specific instructions on connecting and disconnecting this cable. Figure 3-1 and Table 3-1 provide a description of the signals carried in the cable.



Figure 3-1: Cable Pin-outs (as seen from exterior)

Description	Circular Connector	DB- 25P Connector
SIG A	1	4
SIG B	2	17
HTR A	3	13
HTR B	4	25
+15V	5	21
-15V	6	9
Analog Ground	7	18
Thermometer I+	8	1
Thermometer I-	9	14
Thermometer V+	10	2
Thermometer V-	11	15
Gain 0	12	11
Gain 1	13	23
VA	14	19
Drive Ground	15	6
VB	16	7

Table 3-1: Cable Pin-out Descriptions

HTR A and HTR B are heater wires. The module has the capacity to control a heating device, however these connections end with the probe head. There is currently no heater on the Dilatometer capsule. This may be a feature to be considered in the future. Gain 0 and Gain 1 control the gain levels in the signal preamplification. This feature is mainly for test purposes, as the user will most likely operate at max gain all of the time to remain well above the noise floor. SIG A and SIG B are the two wires that handle the measurement signal after it has been converted to differential. The 15V supplies power to the preamplifier. Analog ground is a part of the grounding schematic associated with the module and preamplifier, separate from the signal and drive ground, which are connected.

## **3.2.3 Probe**

The Dilatometer probe consists of a head and a shaft. The head houses the probe pre-amp board which handles signal amplification. This enables the signal to travel to the CAN module with minimum attenuation. The probe serial number is located on the back of the head in the form "DPR XXX,", and DPR stands for 'Dilatometer probe'. The probe head also has two black clamps that secure it firmly to the PPMS system.

The shaft houses wire that connect the capsule to the probe head. It is made of four separate stainless steel tubes. Two of the shaft tubes also have a copper-nickel alloy innertube for electrically shielding the signal and drive wires. Along the tubes are sets of baffles. The purpose of the baffles is to block thermal radiation from above and create a more uniform temperature gradient from the bottom of the sample chamber to the top. Because they are made of an insulating material, they also separate the probe ground



Figure 3-2: Probe Head Exploded View

and the dewar ground. A 'more uniform temperature gradient' is a very relative statement, as the temperature of the shaft is not measured. The user will notice this most when the cryostat has gone from 1.8 K to 300 K in a relatively short amount of time. When the user removes the probe, the ends will be room temperature, but the middle of the shaft will still be very cold. Carefully handle the probe to avoid touching this middle region. At the bottom of the probe shaft, four screws secure the probe to the capsule.

Another important feature of the probe is its length. At roughly 34 inches, it places the sample in the center of the magnetic field. This is essential for interpreting magnetostriction measurements.



#### WARNING!

Do **NOT** open the probe head. There are no user serviceable parts inside. Any issues that can be traced to the probe will have to be addressed at Quantum Design.

## 3.2.4 Capsule

The Dilatometer capsule is the most intricate and important piece of hardware in the system. The housing holds two boards that help carry signals from the cell and thermometer into the probe leads. For the safety of the product, the user should never attempt to remove any components from the capsule.

#### 3.2.4.1 HOUSING

The Dilatometer capsule is made up of a rotator housing and a sphere housing. Both are made of copper and gold plated to prevent oxidation. The sphere is secured in the rotator housing by the angle set screw. A 5/16-inch Allen key is provided in the user kit for adjusting the set screw. The Dilatometer housing has approximately a one-inch diameter to fit the sample chamber of the PPMS systems.

Once a sample is ready for measurement, the thermal radiation shield is fit over the capsule. The base screw then secures the shield to the capsule. The orientation is made clear in Figure 3-3.



Figure 3-3: Cell Capsule, Radiation Shield, Base Screw

#### 3.2.4.2 CELL

The Dilatometer cell is machined from fused silica. It has the same molecular composition as fused quartz, but its amorphous structure adds the necessary strength. Fused silica was chosen because of its extremely low thermal expansion below 300 K, hence, minimizing background in the measurement.

Once the cell is machined, it is metalized. This protects the sample grooves, but is mainly intended to create the conductive surfaces for the capacitors. After metalizing, the surface is laser ablated to create electrically isolated regions for the cell circuit.

The two cell halves are connected by hinges on either side. The hinges, also metalized, are soldered to the sides as indicated in Figure 3-4. The hinges allow the teeth to move in relation to each other, changing the capacitance gaps.



Figure 3-4: Cell Hinges

The capacitors and cell circuit are formed by a laser ablation process that removes the metal surface. The different regions are shown in Figure 3-5. Each capacitor has two halves of a bottom tooth, and the entire upper surface forms the divided portion of the circuit. This upper region has been previously represented as V\_Balance in Chapter 1. The signal ground (SHLD) and drive ground (CT) are electrically connected by the hinges.

The cell geometry was chosen for a few different benefits. First, along with the grounding scheme, fringe field effects are controlled. Second, as the sample expands, the capacitance gaps change

complementary to one another. Third, the laser ablation can be done in such a way that the sample is completely grounded and the conductance of the sample has no effect on the capacitors. Fourth, the capacitors are able to maintain parallelism to within an acceptable degree of error as the sample dilates.



Figure 3-5: Cell Ablation and Wiring Diagram

#### 3.2.4.3 THERMOMETRY

The thermometer used was chosen for the Dilatometer Option because of its high sensitivity at low temperatures and low magnetoresistance.

The thermometer is mounted on the back of the cell, behind the upper circuit board on the spherical housing. With the help of the thermal radiation shield, this provides the most accurate sample temperature. The thermometry signals are carried through four separate wires to the probe and eventually to a bridge thermometry circuit in the Dilatometer module.

#### 3.2.4.4 SERIAL NUMBER

The serial number is very important for identifying the correct capsule for Option Controller input. It is located on the bottom of the capsule frame housing (Figure 3-6). The number is engraved in the form, 'DCP XXX,' where DCP stands for 'Dilatometer capsule'.



See Section 5.6 for more information on managing calibration files.

Figure 3-6: Cell Serial Number Location

## **3.2.5** Balance Meter

The Balance Meter enables the user to check the sample mount before proceeding with a measurement. The hardware is composed of an aluminum enclosure and a plastic capsule support and securing clamp. The enclosure houses the circuit board and a battery (AA).

The front panel consists of four ports for checking the capsule thermometry, a screen to read the imbalance, and a power button. This device is designed to turn off by itself after a few minutes.

The theory of operation for the Balance Meter is discussed in Section 2.5.



Figure 3-7:Balance Meter

# 3.3 Quantum Design Part Numbers

Component	Description	Part Number
Module	See Section 3.2.1	4101-750
Cable	See Section 3.2.2	3071-100
Probe	See Section 3.2.3	4071-130
Capsule	See Section 3.2.4	4071-120
Balance Meter	See Section 3.2.5	4071-116

Table 3-3: Dilatometer User Kit Part Numbers

User Kit Components	Description	Part Number
Copper Standard	A prepared copper sample	4071-406
Fused Silica Standard	A prepared fused silica sample 4071-	
Sample Spacer, thick	A thick fused silica spacer for mounting samples	4071-408
Sample Spacer, thin	A thin fused silica spacer for mounting samples	4071-409
Spring	Adds tension to hold the sample in place throughout the measurement	4071-404
CrystalBond <sup>TM</sup> 509	Wax used in sample grinding	4071-401
Lapping Tool	0.077-inch sample grinding	4071-403
	0.079-inch sample grinding	4071-402
	0.081-inch sample grinding	4071-414

### C H A P T E R 4

# Software

## 4.1 Introduction

This chapter contains the following information.

- Section 4.2 presents an overview of the purpose and scope of the Dilatometer software.
- Section 4.3 presents the Dilatometer Option Controller.
- Section 4.4 describes the data processing model used by the Option Controller.

## 4.2 **Overview of the System Software**

The Dilatometer software handles writing data files, controlling various system parameters, and processing the data. The Dilatometer software also manages all calibration data. The Dilatometer software is activated from within MultiVu by selecting the Utilities >> Activate Option >> Dilatometer >> Activate. In order to deactivate the software, follow the same path and select >> Deactivate as the last option.

Once the Dilatometer Option Controller is activated, the user will begin by using the wizard to configure the system, install the probe and input new information concerning the cell and sample. All measurement data is recorded to a .sjson data file associated with the project. Because of the value of raw data, this file will never be altered or deleted by the user. In order to create a view of the data, the software will access this raw file each time, but write to a new view file. All the data files can be accessed through the project navigation pane in the Option Controller. It is recommended that the user not modify the file and folder structure directly. Doing so may corrupt the project.

For installation instructions see Section 2.3.

## 4.3 Dilatometer Option Controller

The Dilatometer Option Controller is the user interface that allows you to navigate and use the Dilatometer Option. This is the first window that will appear when the user activates the Dilatometer Option (Figure 4-1). If the window is closed, it can be reopened in the MultiVu tabs under **Measure** >> **Dilatometer**. Notice that closing the window does not deactivate the option or stop the measurement.

🚺 Dilatometer Option - In	nstall					83
Wizard	Measure	New Sample/Cell Properties				
Current Project: In:	stall				Project.	
Project Calibrations						
▷ Measurement Data ▷ Baseline Tables ▷ Views						
Measurement Status:		Running Views:				
Abort	New View	ViewName			Status	
Cell Imbalance (ppm):	0.000	Black Temperature (K):	300.00	Field (Oe):		0.00
PID Feedback:	Closed, Standard	Cell Temperature (K): Delta (K):	1 519.55 -1 219.55			

Figure 4-1: Dilatometer Option Controller Main Dialog

The main dialog has a number of important features. In the upper left hand corner are the measurement command buttons. Wizard... will be used for installing the probe and preparing the system. Measure... will direct the user to choose and initiate a specific measurement. New Sample/Cell **Properties...** will configure the sample and cell properties. This is normally done in the wizard, however the option is provided here in the case that the information needs to be updated. Every time the probe is installed through the wizard, new configurations will be added. Below this is the current projects bar. This feature loads all the data files and views from a particular project into the Option Controller. The majority of the main dialog is taken up by the project navigator. The Project tab shows project specific data and views, while the **Calibrations** tab shows data that can be used by all of the projects. By left clicking once on items in the project navigator, information on the data will appear on the right hand side in the configurations pane. This is convenient for data identification as well as editing the specific configurations for view production. Note that the fields are not directly editable, but **Edit** buttons can be found in the configurations pane and by right clicking the items. At the bottom of the main dialog are the measurement status and running view status sections. These show the live system parameters, allow the user to abort the measurement, stop a running view and enable the user to display a running view as a graph in MultiVu.

## 4.3.1 Projects

Projects are collections of data, baseline tables, configurations and views. The convenience of a project is that it allows the user to group measurements as they see fit. Below are some examples of various project organizations.

- Project by sample: a collection of all the data taken on a single type of material
- Project by experiment: a collection of data from a range of samples intended for comparison
- Project by operator: a collection of data taken by a specific user
- Project by time: a collection of data taken during a specific period of time



Figure 4-2: Changing the Current Project

In order to manage the current project on the Option Controller, select the option on the far right of the main dialog: **Project...** Figure 4-2 shows the options for changing the current project. The project can be changed in the wizard when installing a probe. A project cannot be opened or closed during a measurement or if there are any running views.

When creating a new project, you will be prompted to select the new project's parent folder. After selecting a parent folder, the user will be prompted to enter the project name. A project folder consists of the following folders:

- Baseline Files found in this folder will contain baseline data for thermal expansion coefficient or compare data results to a previous good run of data.
- Raw raw collected data will be contained in the data.sjson file.
- Regions A collection of .cfg files associated with thermal, magneto, or continuous measurements. These files specify start and end time stamps of measurements.
- Sample cell properties A collection of . cfg files with information about a specific sample setup.
- Views A collection of folders associated with specific views that will have all the .cfg files and resultant data files from widgets used to create the view.dat file.

To open a previous project, the user has two options. 1) The user can select **Open Project** and navigate to the project folder name. 2) The user can also mouse-over **Open Recent Project**; a list of the last ten projects open will be displayed for the user to select which project to open.

## 4.3.2 **Project Navigation Pane**

The project navigation pane in the main dialog allows the user to easily explore all of the project and calibration data. The project pane is divided into two portions. On the left are the project and calibrations tabs. On the right, configurations and further options will be displayed in the

configurations pane. Once an item is selected in the project or calibrations tab by a single left click, configurations and action buttons will appear specific to that item in the configurations pane.

#### 4.3.2.1 PROJECT TAB

Within each project there will exist measurement data, baseline tables and views as seen in Figure 4-3.

Project	Calibrations			
D Meas D Basel D View:	surement Data line Tables s			

Figure 4-3: Project Tab

By selecting the **Measurement Data** header, the dialog in Figure 4-4 will appear in the configurations pane. This shows the current sample and cell properties and provides three buttons for running a measurement, and editing the current sample property information.

Measure vs T	Measur	re vs H	Measure Continuous
New Sample	e Cell Property	Det	fine New Range
	Current Sample	e Cell Properties	
Sample Name:	FuSi		
Sample Comment:			
Sample Length (mm):	2.006		
Rotator Angle (deg):	0		
Sample Slot:	L		
Cell Serial Number:	DCP016		
Probe Serial Number:	DPR015		

Figure 4-4: Measurement Configuration

By selecting the **Baseline Tables** header, Figure 4-5 will appear in the configurations pane. This presents three buttons for adding a baseline table to the current project. Creation of new baselines is discussed later in this chapter. It is likely that the user will use the same baseline table across multiple projects. A user can copy a baseline table from another project, but must be aware that the raw data used to create the base line is with the original project.

New Baseline Table	vs. Temperature	New Baseline	Table vs. Field
	Copy Baseline Table	From Another Project	

Figure 4-5: Baseline Tables Configurations

By selecting the **View** header, section Figure 4-6 will appear in the configuration pane. The option will display buttons to create four different views. The user can create a view to calculate the thermal expansion coefficient, magnetostriction coefficient, a dilation view from sweeping temperature or a sweeping field. The fourth option will allow the user to create a combined view which will merge multiple data file measurements into one data file.

New Baseline Table vs. Temperature	New Baseline Table vs. Field
Copy Baseline Table	From Another Project

Figure 4-6: Views Configurations

As with most controls in this software all of these options are also accessible by right clicking the header in the left hand pane.

#### 4.3.2.2 CALIBRATIONS TAB

The calibrations tab contains cell calibrations, probe calibrations, and published data tables for copper and fused silica samples. These calibrations are independent of the projects.

Project Calibration	s	
Calibrations		
✓ Cells		
▷ C123		
DCP010		
DCP011		
DCP012		
DCP014		
DCP015		
DCP016		
✓ Probes		
DPR011		
DPR012		
DPR014		
DPR015		
P DPR016		
P Probeiuz	<b>D</b> .	
Standard Keferer	ice Data	
Copper Std		
Fusista		
[L		

Figure 4-7: Calibrations Tab

By expanding the calibration node to display cell and probe subtrees, the user can select a specific capsule/cell serial number or probe serial numbers. Figure 4-7 shows what will appear for either cell or probe calibrations in the configurations pane. This indicates all of the important properties of the cell or probe and gives the user the ability to change the properties. By editing the cell or probe property, the old version will be archived and the new, updated version will be listed under current calibration. When the user creates a new sample cell property under measurement header, they will indicate which cell and probe they used. A timestamp is used to indicate which calibration version of the capsule or cell was active at the time of creation. By updating the calibration versions and archiving older versions, information is not lost when creating views. Creating views with current and archived versions will be discussed later.

The cell calibrations are stored in ...\Dilatometer\Calibrations\Cells\DCPXXX (XXX indicates the serial number of the cell), where the thermometer calibration file is stored in the cache folder. There is a folder for raw data, for the user to save more information for the cell, and a third folder that houses a

collection of archived and current version of the calibrations. Selecting a cell calibration in the navigation pane, the option will display Figure 4-8 in the configuration pane.

	Edit
Serial Number:	DCP016
Cell Constant:	0.157848794324765
Cell Capacitance:	15
Creation Time:	12/5/2016 9:33:45 AM
Thermometer Table:	DCP016_TTM244DDF73_Smoothed.tab

Figure 4-8: Cell Calibration

The probe calibrations are stored in ...\**Dilatometer**\**calibrations**\**Probe**\**DPRXXX** (where XXX indicates the serial number of the probe). The capacitance of the probe is provided. Selecting a probe calibration in the navigation pane, the option will display Figure 4-9 in the configuration pane

	Edit
Serial Number:	DPR016
Probe Capacitance:	36.4
Creation Time:	12/21/2016 7:46:30 AM

Figure 4-9: Probe Calibration

At the bottom of the option control window is a live update of the dilatometer option (Figure 4-10). The option displays the cell imbalance in real time and the PID Feedback, delta between the system temperature and the sample temperature and magnetic field. This helps the user to determine if the module is in the correct state and the balance point of the cell is a valid reading and has not railed. The delta temperature is useful, since the option controls the system temperature, but records the sample temperature.

Cell Imbalance (ppm):	0.000 Block Temperature (K):	300.00	Field (Oe):	0.00
PID Feedback: Closed, S	andard Cell Temperature (K):	1 519.55		
	Delta (K):	-1 219.55		

Figure 4-10: Measurement Status

## 4.3.3 Wizard

The wizard is used for installing and removing the Dilatometer probe. It handles chamber operations, bringing the system to room temperature, entering new sample and cell properties, changing the project and confirming the system preparation was successful.

When activating the wizard, the user will be prompted to open the sample chamber. If the temperature is below 300K, it will warm the system to a safe temperature before venting. Once the system is vented, it is safe to install the probe with capsule mounted. Once the probe is inserted, the user will then click purge. This will perform a purge and seal operation on the chamber. As the system is cycling through the pump operations, the user may continue through the wizard.

On the next page of the wizard the user can create a new project or open an existing one. Once a project is open, they can enter in the sample information for their current setup by clicking **New**. Another window (Figure 4-11) will appear for the user to enter the sample name and any comments that pertain to the sample.

Sample and Cell Propert	ies	
Sample Name: Comments:		
Enter your sample desc as the title of your grap	iption (chemical formulas, lab n hs and in measurement listings.	otebook reference, and more). This will appea This is for information purposes only.
Sample Length (mm):	0	
The sample length shou blank and enter it later	ld be between 1.95 mm and 3.0. Fyou like. This will be used when	5 mm. Enter its exact length here. Leave it n calculating Thermal Expansion.
Rotator Angle (deg): Record the angle of the	0 rotator in degreees.	
Sample Slot: Which slot is the sample	Left • loaded into (see drawing).	
Cell Serial Number: Probe Serial Number:		

Figure 4-11: Entering Sample and Cell Properties Window

Note: Comments that pertain to the measurement can be added later when running a measurement.

At this point the user does not have to enter in the sample length, but in order to create a view, the user must update this field. The user can note the rotation angle  $(0-90^{\circ})$  of the sphere in the capsule, which slot they entered the sample (left or right slot), and then select which probe and capsule is used. Note that you are not selecting the calibration version of the probe or capsule, but instead indicating your current setup for the measurements.

The last page of the wizard is the probe test (Figure 4-12). This test makes sure the hardware of the dilatometer probe and electronics are working correctly and the selected thermometer calibration matches expectations. The user will press the **Test** button to perform the test. Once the test has finished a measurement of the cell thermometer, cell imbalance, the module state and the test results will appear. If one of these fails, it will show up in the results. If the probe is unplugged when this test is performed, it will fail and indicate to the user that the probe is unplugged.

robe Test			Test
This test makes sure t selected thermometer	the hardware of the D r calibration match ex	ilatometer probe and electronics i pectations. Press "Test" button to p	s working correctly and that the perform test.
	Measured	Expected	
Cell Thermometer:			
Cell Imbalance:			
PID Feedback:			
Fest Results:			* *

Figure 4-12: Probe Test

The wizard does not need to be invoked in order to run a measurement, but ensures that everything is hooked up and reading correctly before starting a measurement. If the user wants to update the sample cell property without using the wizard, they can use the button on the option controller.

### 4.3.4 Measurements

To begin a measurement, select **Measure...** in the upper left hand corner of the Dilatometer Option Controller main dialog. From here there are three options for measurement (Figure 4-13): Measure Thermal Expansion (vs T), Measure Magnetostriction (vs H) and Continuous Measurement.

Measure	Measure Thermal Expansion (vs T)
Install	Measure Magnetostriction (vs H)
	Continuous Measurement

Figure 4-13: Measure

On the option controller window is measurement status. This will tell the user when the measurement is starting up and initializing the module. If the user inputs a wait time for the measurement, the status will change to a countdown. Once the system stabilizes at the start of the measurement, the status will change to running. While a measurement is running, if the user wants to quit the measurement, they can press the **Abort** button.

#### 4.3.4.1 THERMAL EXPANSION

When starting a thermal expansion measurement (Figure 4-14), the user can enter in the scan name for the measurement. Any duplicate name will have a number appended to the end. The user can enter in comments that pertain to the measurement. The user will enter a start and end point for the sample temperature along with how fast they want to ramp temperature. The user may select which mode they want to acquire data in, either coarse or standard. Coarse is meant for quick diagnostics and does not

have the resolution of standard. If the user is not at the start temperature, the system will ramp to the start temperature at maximum rate, at which point the user can indicate a wait time before starting a measurement.

Scan Name: Te	mperature	: Scan			
Comments:					
This will perform a the	ermal expa	ansion measurem	ent by auto	matically rampin	g the temperature.
		200	1		
Start Temperature (I	к):	300			
End Temperature (K	):	2			
Rate (K/min):		0.1	]		
	ſ	Fast Settle			1
Approach Mode:	l	Turtue			J
Approach Mode: Acquisition Mode:	(	Standard Resolu	tion	•	]
Approach Mode: Acquisition Mode: Settle Time (secs) at	Start?:	Standard Resolu	tion	•	

Figure 4-14: Thermal Expansion Measurement

#### 4.3.4.2 MAGNETOSTRICTION

Starting a Magnetostriction measurement is similar to starting a thermal expansion measurement. (Figure 4-15). Fill each field with the necessary information and then select **OK** to begin the field ramp and data collection.

📡 Magnetostriction Measuremer	t (vs H)	
Scan Name: Field Scan		
Comments:		
This will perform a magnetos	riction measurement by recording ex	pansion data while sweeping feld.
Start Field (Oe):	-90000	
End Field (Oe):	90000	
Rate (Oe/sec):	10	
Approach Mode:	Linear	•
Magnet Drive Mode:	Persistent	•
Acquisition Mode:	Standard Resolution	•
Settle Time (secs) at Start?:	0	
		OK Cancel
		Cancel

Figure 4-15: Magnetostriction Measurement

#### 4.3.4.3 CONTINUOUS

Running a continuous measurement (Figure 4-16) starts data collection and continues until instructed to stop. Only dilation views can be created on continuous measurement.

	ontinuous	Measurement				
Comments:						
This will start (or sto Start Stop	p) a continu	uously running n	neasuremen	t.		
This will start (or sto Start Stop Acquisition Mode:	p) a continu	uously running n	neasurement ution	5	•	

Figure 4-16: Continuous Measurement

#### 4.3.4.4 SEQUENCE

Sequence commands are useful for automating multiple measurement runs. The Dilatometer Option provides three commands, as seen in Figure 4-17, one for each type of measurement: thermal expansion, magnetostriction and continuous measurement.



Figure 4-17: Sequence Measurement

### 4.3.5 Measurement Data

The measurement data is presented as a navigation tree of sample/cell properties with the associated measurement regions. When a user performs a measurement, it will always append the measurement region to the latest sample cell property. The user can perform multiple types of measurements for any given sample cell property. This is shown in Figure 4-18.



Figure 4-18: Measurement Regions

When a thermal measurement is completed, the option will display the scan name, the start and end temperatures and the magnetic field. After selecting the thermal measurement region, the option will display buttons to create a new thermal expansion view, a new dilation view, edit the measurement properties or calculate a new cell constant. The user can also see the current properties and comments for the measurement region.

When a magnetic field measurement is completed, the option will display the scan name, the start and end fields and the sample temperature. Similar to thermal measurements, the user can create a new magnetostriction coefficient view, a new dilation view or edit the measurement region properties.

When a continuous measurement is completed, it shows how long the data was recorded and only allows the user to edit the measurement properties or create a dilation view from the data.

#### 4.3.5.1 CALCULATING NEW CELL CONSTANT

After performing a thermal measurement, the user has the option to calculate a cell constant. The user must perform a fused silica thermal measurement and a copper thermal measurement using the standards provided. This window is shown in Figure 4-19. The user will need to select the standard published data for each associated run from the drop-down menus.

When the user has entered the information needed, they can click calculate. When a new cell constant is calculated, a window will appear with the updated cell constant asking if the user wants to update the current calibration of the cell.

🚺 Calculate New Cell Consta	int		
This procedure is used to ca update the current cell calib necessarily calibrated) therm to the same length. For exan specify the standard referen	lculate the cell coi ration. In order to val expansion mea nple, Fused Silica ce data for each si	nstant (mm/imbalance u perform this calculation surements on standard for sample #1 and Copp can and the Comparison	nit) for the dilatometer cell and , you must perform two (not reference samples that are very close er for sample #2. You also need to Temperature to be used.
Reference Sample #1:			
Measurement Scan for Sample #1:	Cu (399.64 K to	o 3.03 K, at 0.23 Oe)	•
Reference Data for Sample #1:	Copper Std		•
Reference Sample #2: Measurement Scan for Sample #2:	FuSi (400.01 K t	to 3.03 K, at 0.37 Oe)	
Reference Data for Sample #2:	FuSi Std		•
Comparison Temperature: Current Cell Constant (DCF	Low(K): 016):	79.9 0.157848794324765	Hi(K): 300
			Calculate Close

Figure 4-19: Calculating a New Cell Constant

### 4.3.6 **Baselines**

Baselines are a smoothed tabular representation of measured data. In order to calculate the thermal expansion coefficient, a Fused silica baseline (FuSi measurement) is needed in order to subtract out part of the background of the sample measurement. Baselines can also be used for comparisons of relative data.



Figure 4-20: Baselines

When the baseline header is selected, the option displays three buttons in the configuration pane: New Baseline Table vs Temperature, New Baseline Table vs Field and Copy Baseline From Another Project, as seen in Figure 4-20. Selecting the new baseline table vs temperature (Figure 4-21), gives the user the option to create 2 types of baselines: a standard reference baseline table or a baseline table for relative expansion studies.

Create Thermal Expansion Baseline Table	
Create Standard Reference Baseline Table Create a Baseline Table for Relative Exp	ole bansion Studies
This analysis creates a Standard Reference b thermal expansion curves. You must specify reference curve.	laseline Table for a known reference sample that can be used for generating absolute both your raw scan data and standard reference data which provides the absolute
Allow only scans with the same cell/probe c	alibration versions [Recommended]
Measurement Region(s) □ Cu (399.64 ✓ FuSi (400.01	K to 3.03 K, at 0.23 Oe) [Date: 12/1/2016 4:57:55 PM] [Cell: DCP016, Probe: DPR015] K to 3.03 K, at 0.37 Oe) [Date: 12/4/2016 12:03:21 AM] [Cell: DCP016, Probe: DPR015]
Number of Temperatures	100
Standard Reference Data:	FuSi Std 🔹
Select the literature reference data that prov	vides the know absolute thermal expansion values for this sample.
Temperature Slope Averaging:	2 min (~1.2 K resolution)
Dilation Averaging and Output Interval:	10 sec 🔹
Output Table:	
New (enter title)	Default Table Name
Overwrite Table	<none> ~</none>
Cell Calibration Version:	DCP016, 11/30/2016 2:50:41 PM *
Probe Calibration Version:	DPR015, 11/17/2016 8:28:01 AM, (Current) *
	Create Table Cancel

Figure 4-21: Creating a Thermal Expansion Baseline Table

A standard reference baseline table is used for generating absolute thermal expansion curves. You must specify both your raw scan data and standard reference data. When selecting a specific measurement region for a baseline, the user can enter how many points they want in the curve. The user will then have to select the standard sample they are measuring. Typically, this will be a FuSi sample. Set the averaging values for dilation and temperature, specify the baseline name and choose which probe and cell calibrations were used.

When creating a baseline table for relative expansion studies, the analysis is performed without selecting a known reference sample. This would be used when creating a background table for subtraction in a thermal expansion curve. The user will select a measurement region, number of points, averaging, name and cell and probe calibrations.

i nis creates a baseline table that is a function of magnetic field. A Wagnetostriction baseline table can be subtracted from any other Magnetostriction measurement and is how you calculate relative magnetostriction.				
· · · · · · · · · · · · · · · · · · ·				
Allow only scans with the same cell/probe c	alibration versions [Recommended]			
weasurement region(s) Field Scan D	p (03/ 04 to 9553522 Oe, at 336 K) (Date: 12/3/2016 11:27:39 AM) [Cell: DCP016, nown (9999952 Oe to 0.34 Oe, at 3.05 K) [Date: 12/3/2016 11:27:39 AM] [Cell: DCP016,			
*				
۲ مراجع المراجع	m ) 100			
Number of Fields Field Slope Averaging:	100 30 sec (~8.8E+02 Oe resolution)			
Vumber of Fields Field Slope Averaging: Dilation Averaging and Output Interval:	100 30 sec (~8.8E+02 Oe resolution) 10 sec			
Number of Fields Field Slope Averaging: Dilation Averaging and Output Interval: Output Table:	100 30 sec (~8.8E+02 Oe resolution) 10 sec ·			
Number of Fields         Field Slope Averaging:         Dilation Averaging and Output Interval:         Output Table:	III III IIII IIII IIIIIIIIIIIIIIIIIIII			
Number of Fields         Field Slope Averaging:         Dilation Averaging and Output Interval:         Output Table:             New (enter title)          Overwrite Table	100           30 sec (~8.8E+02 Oe resolution)           10 sec           Default Table Name <none></none>			
Vumber of Fields Field Slope Averaging: Dilation Averaging and Output Interval: Output Table:	100           30 sec (~8.8E+02 Oe resolution)           10 sec           Default Table Name <none>           DCP016, 11/30/2016 2:50:41 PM</none>			

Figure 4-22: Creating a Magnetostriction Baseline Table

A baseline table vs field (Figure 4-22) is a table that can be subtracted from any other magnetostriction coefficient data and is used to calculate relative magnetostriction. Similar to thermal tables, the user will select the region and number of points for the data file.

The user also has the ability to move a baseline from one project to another. The raw data will stay with the original project in which the baseline was created.

When selecting a baseline, information on the baseline is populated in the configuration pane, as seen in Figure 4-23. The user has the ability to edit the baseline data file by either changing the name, selecting a different standard reference or deleting points from the table. The user can also view the baseline in MultiVu, either as created or as an interpolated graph. The user also has the ability to save the baseline in another location with the **Save As...** button.

Edit		View in MultiVu	
View Interpolate	d in MultiVu	Save As	
Table Name:	Fusi baseline		
Sample Length (mm):	2.006		
Field (Oe):	0.367324322		
Rotation Angle (deg):	0		
Std. Reference Data:	FuSi Std		
Temperature (K)	Dilation (ppm)	Expansion Coefficient (ppm/K	
3.16702032596448	4033.4567615	5 0.021271171140331	
4.16154311966872	4033.4374980	8144 0.0204040602832904	
5.15606591337295	4033.4206712	8071 0.0195002395429152	
6.15058870707719	4033.4062811	5783 0.0185597089192054	
7.14511150078143	4033.3943277	1279 0.017582468412161	
0 12062420440566	1000 00100	1550 0.0165695190017910	

Figure 4-23: Baseline Configuration

## 4.3.7 Creating Views

There are four types of views that the Dilatometer software prepares as shown in Figure 4-24: Thermal Expansion, Magnetostriction, Dilation and a Combined View.

In order to create a new view, select **New View** on the main menu of the Option Controller. The user may also select the measurement region they would like to produce a view from. The various view options are shown in Figure 4-19, and each is discussed in detail below.



Figure 4-24: Create a View

#### 4.3.7.1 THERMAL EXPANSION VIEW

When creating a new thermal expansion coefficient view, the window shown in Figure 4-25 will appear. The user can create 4 types of thermal expansion coefficient views: 1) Absolute Thermal Expansion (with Standard Reference Baseline), 2) Absolute Thermal Expansion (Assuming Ideal FuSi Cell Expansion), 3) Relative Thermal Expansion and 4) Other.

🚺 Thermal Expansion Coefficient View				
View Name: Thermal Expans	1 Coeff View			
<ul> <li>Absolute Thermal Expansion Coe</li> <li>Absolute Thermal Expansion Coef</li> <li>Relative Thermal Expansion Coef</li> <li>Other</li> </ul>	:ient (with Standard Reference Baseline) :ient (With Ideal FuSi Cell Expansion) ent			
Analyze data to create Absolute Thermal Expansion Coefficient. This analysis requires a previous measurement of a known standard reference sample that has been converted to a Standard Reference Baseline Table (a Baseline Table with known reference data specified). The resulting Absolute Thermal Expansion is calculated by subtracting the Baseline and adding the Standard Reference Data.				
Select Measurement Scans for Ar	ysis:			
Allow only scans with the same cell	robe calibration versions [Recommended]			
FuSi (400.01 K to 3.03 K, at 0.37 Oe) [Date: 12/4/2016 12:03:21 AM] [Cell: DCP016, Probe: DPR015]      Stream Any Data After:				
Baseline Table:	Fusi baseline (3.17 K to 399.98 K, at 0.37 Oe)			
Standard Reference Data:	FuSi Std 🔹			
Compare Result To:	FuSi Std 🔹			
Temperature Slope Averaging:	2 min 🔹			
Dilation Averaging and Output In	erval: 10 sec 🔹			
Cell Calibration Version:	٣			
Probe Calibration Version:	Ţ			
	Apply Save Only Cancel			

Figure 4-25: Creating a Thermal Expansion Coefficient View

When creating an Absolute Thermal Expansion (with Standard Reference Baseline), the user will select a baseline that has the associated standard reference data. The absolute thermal expansion is calculated by subtracting the baseline and adding the standard reference data to the raw measurement.

When creating an Absolute Thermal Expansion (assuming Ideal FuSi Cell Expansion), this option does not perform a baseline subtraction but does add the FuSi standard reference data.

Relative Thermal Expansion is used when one is not interested in absolute thermal expansion, but rather the thermal expansion compared to another measurement (as stored in a baseline). If the user selects a baseline it will be included in the data file for comparison.

When selecting Other, one can use both a thermal expansion baseline table and standard reference data from any project baseline table or global reference data.

The user is allowed to select more than one measurement region for a view, however the option will only use one probe and cell calibration in the analysis. The user is presented with three options when processing data. 1) Allow only scans with the same cell/probe calibration [Recommended], 2) Allow scans with different calibration version for the same cell/probe serial number and 3) Allow scans with any cell/probe calibration [Be careful].

When using the first option, only measurement regions associated with the same cell and probe calibration versions will be displayed. The user will not have the option to select which calibration to use. It will be based on the sample cell properties chosen when the measurements were taken.

The second option allows scans with different calibration versions for the same cell/probe serial numbers. The user will have the ability to select which calibration version to use in processing the data. Be careful about processing a measurement with a wrong calibration version.

The third option displays all the measurements and all the calibration files available. Be careful when processing multiple files; make sure the calibrations selected are correct for all measurements.

The user can process live data (a measurement that is still running) or process the data after the measurement is complete. As a measurement is running it is always appending new information to the .sjson file. By clicking **Stream Any Data After** and selecting the current run, the user can create a live view that will update as data is created. If the user does not click **Stream Any Data After**, the option will stop appending to the view once it reaches the end of the .sjson file.

The user can measure the supplied copper and fused silica standards in the user kit and compare the results to published data by selecting **Compare Results To**. This will look at the percent difference and absolute difference between published data and measured data.

There are temperature slope and dilation rate drop-down menus to select the averaging windows in units of time. Selecting a longer time for **Temperature Slope Averaging** will average out spikes in the sample temperature rate data. Selecting a shorter averaging time for dilation rate will prevent accidentally averaging out a feature of the sample. The time chosen for dilation rate averaging is how often data is written to the file. If the user wants to control the number of points per kelvin, they will have to convert the averaging time window to temperature window based on the ramp speed of the measurement.

#### For example:

If the user has a temperature ramp rate of 0.6K/min(.01K/sec), with 10s averaging, they will have a data point every 0.1K

(Dilation Rate Averaging) \* (Sample Temperature Rate) = (Data point per Kelvin) (4.1)

By clicking apply the user will process the measurement regions.

#### 4.3.7.2 MAGNETOSTRICTION VIEW

When creating a new Magnetostriction Coefficient view, the window shown in Figure 4-26 will appear. The user has the option to create an Absolute Magnetostriction Coefficient or Relative Magnetostriction Coefficient (subtracting a Baseline Table).

View Name:	Magnetostriction Coeff V	/iew	
Absolute Mag Relative Magr	netostriction Coefficient letostriction Coefficient (sub	stracting a Baseline Table)	
Calculate Magn Relative Magne Baseline Table.	etostriction Coefficient (expa tosriction will subtract an ex	ansion versus field). Absolute Magnetostr isting curve and requires that you specify	riction is simply the sample expansion vs field. • a previously created Magnetostriction
Select Measure	ment Scans for Analysis:		
Allow only scan	with the same cell/probe c	alibration versions [Recommended]	•
Stream Any	Data After:		
Stream Any I	Data After:	<pre></pre>	
Stream Any I Baseline Table: Field Slope Ave	Data After:	( <none>)</none>	
Stream Any I Baseline Table: Field Slope Ave Dilation Averag	Data After: graging: ging and Output Interval:	<none> 2 min 10 sec</none>	۰ ۰ ۰ ۰
Stream Any I Baseline Table: Field Slope Ave Dilation Averag Cell Calibration	Data After: rraging: jing and Output Interval: vVersion:	(None>) 2 min 10 sec	• • •
Stream Any Baseline Table: Field Slope Ave Dilation Averag Cell Calibration Probe Calibrati	Data After: rraging: jing and Output Interval: Version: on Version:	( <none>) (2 min) (10 sec)</none>	• • • • • •

*Figure 4-26: Creating a Magnetostriction Coefficient View* 

Absolute Magnetostriction Coefficient is simply the sample expansion vs field. The ability to select baselines is disabled.

By selecting, **Relative Magnetostriction Coefficient (subtracting a Baseline Table)**, the user may select a magnetostriction baseline to be subtracted out of the measurement.

The user will have the same three cell/probe calibration options as the Thermal Expansion Coefficient View. The user can stream any live measurement or post process a completed run.

To calculate Magnetostriction Coefficient the user must select averaging parameters for field slope and dilation/output interval.

Selecting cell and probe calibrations is similar to creating Thermal Expansion Coefficient Views.

#### 4.3.7.3 NEW DILATION VIEW

When creating a new dilation view, the window shown in Figure 4-27 will appear. All measurement regions are available for selection. Below are specifics on the entry fields and the raw expansion view creation process.

- The user can select multiple measurements to be included in the view.
- An offset can be chosen. This will perform a translation on the data by either a fixed offset input by the user or by setting the first measurement to the zero point.
- The user has the same three options for selecting the cell and probe calibrations as for other views.

🚺 Dilation View					<b>-</b>
View Name:	Dilation View				
This simple analysi for raw logging of	s does not attempt to p mixed field and tempera	erform any expansion o ature data.	lerivatives and is me	ant to show only r	aw dilation. Use this view
Select Measureme	ent Scans for Analysis:				
Allow only scans w	ith the same cell/probe	calibration versions [Re	commended]		•
Cu (399.64 K tư Field Scan Up (t Field Scan Dow FuSi (400.01 K t	3.03 K, at 0.23 Oe) (Dat ).37 Oe to 89999.52 Oe, n (89999.52 Oe to 0.34 ( o 3.03 K, at 0.37 Oe) (Da	e: 12/1/2016 4:57:55 P/ at 3.04 K) [Date: 12/3/2 De, at 3.05 K) [Date: 12/ te: 12/4/2016 12:03:21	4] [Cell: DCP016, Pr 016 10:36:08 AM] [4 3/2016 11:27:39 AM AM] [Cell: DCP016,	obe: DPR015] Cell: DCP016, Prob I] [Cell: DCP016, Pr Probe: DPR015]	e: DPR015] obe: DPR015]
Stream Any Dat	a After:				Ŧ
Add Fixed Dilatio	n Offset				
<ul> <li>No Offset</li> <li>Fixed Offset (ppr</li> <li>Make First Dilation</li> </ul>	n) Measurement Zero	-3923			
Cell Calibration V	ersion:				Ŧ
Probe Calibration	Version:				
			Apply	Save On	ly Cancel

Figure 4-27: Creating a Dilation View

## 4.3.8 Views

Under the View header in the left navigation pane is a collection of views created shown in Figure 4-28. These are listed alphabetically. Here the user can view the Thermal Expansion Coefficient data, Magnetostriction Coefficient data, Dilation data and Merged data.

🗘 Dilatometer Option - Install			# - • <b>•</b>
Wizard Measure	New Sample/Cell P	roperties	
Current Project: Install			Project
Project Calibrations		New Thermal Expansion Coefficient View	New Magnetostriction Coefficient View
<ul> <li>Messurement Data</li> <li>Cu (201/2016 0940)</li> <li>Cu (399.64 K to 3.03 K, at 0.23 Ce)</li> <li>FuSi (12/02/2016 15:16)</li> <li>Field Scan D (0.37 Ce to 89999.52 Oe, at 3.04 K)</li> <li>Field Scan Down (89995.22 Ce to 0.34 Oe, at 3.05 K)</li> <li>FuSi (1400.01 K to 3.03 K, at 0.37 Ce)</li> <li>Baseline Tables         <ul> <li>Fusi baseline (3.17 K to 399.98 K, at 0.37 Ce)</li> <li>Viewsi</li> <li>Cu Thermal Expansion Coeff View</li> <li>FuSi Thermal Expansion Coeff View</li> <li>Magnetostriction View Down</li> <li>Magnetostriction View up</li> <li>Merged Magnetostriction View</li> </ul> </li> </ul>		New Dilation View	New Combined View
Measurement Status:	Running Views:		
Abort New View	ViewName		Status
Cell imbalance (ppm): 0.00	Block Temperature (K):	300.00 Field (Oe):	0.00
PID Feedback: Closed, Standar	Cell Temperature (K): Delta (K):	1 519.55 -1 219.55	

Figure 4-28: View Header

By clicking on any of the views, the right configuration pane will be updated with options for the user to Open the Graph, (Re)generate, Edit, Rename View or Export View. The user will also see information about how the view was created. The properties of the view shown are seen in the Figure 4-29.

🗘 Dilatometer Optior	ı - Install				- • <b>·</b>
Wizard	Measure	New Sample/Cell F	Properties		
Current Project:	Install				Project
Project Calibration	5		Open Graph	(Re)Generate View	Edit
<ul> <li>Measurement Da</li> <li>Cu (12/01/20)</li> <li>Cu (399.6</li> <li>FuSi (12/02/20)</li> </ul>	ta .6 09:40) 4 K to 3.03 K, at 0.23 Oe) 016 15:16)		Rename View		Export View
Field Scan Field Scan	Up (0.37 Oe to 89999.52 Oe, at 3.04 K) Down (89999.52 Oe to 0.34 Oe, at 3.05 K)		View Name:	Cu Thermal Expansion Coeff View	
FuSi (400.0 Baseline Tables	J1 K to 3.03 K, at 0.37 Oe)		Analysis Type:	Absolute Thermal Expansion with Standar	d Reference Baseline
Fusi baseline Views Cu Thermal B EuSi Thermal	3.17 K to 399.98 K, at 0.37 Oe) xpansion Coeff View		Selected Measurement Scans:	Cu (399.64 K to 3.03 K, at 0.23 Oe)	
Magnetostrict	ion View Down		Stream Data After:		E
Merged Magr	etostriction View		Baseline Table:	Fusi baseline (3.17 K to 399.98 K, at 0.37 C	De)
			Std. Ref. Table:	FuSi Std	
			Comparison Table:	Copper Std	
			Temp. Slope Average:	120	
			Dilation Averaging:	10	
			Cell Cal. Version:	DCP016, 12/5/2016 9:33:45 AM, (Current)	
			Probe Cal. Version:	DPR015, 11/17/2016 8:28:01 AM, (Current	)
		· · · · · · · · · · · · · · · · · · ·			•
Measurement Status:		Running Views:			
Abort	New View	ViewName		S	Status
Cell Imbalance (ppm):	0.000	Block Temperature (K):	3	300.00 Field (Oe):	0.00
PID Feedback:	Closed, Standard	Cell Temperature (K): Delta (K):	-12	519.55 219.55	
				I	

Figure 4-29: View File Properties

When a series of views have been created, the user can combine or merge multiple files into one. By clicking New **View** >> **New Combined View**, the user will see Figure 4-30. The user can select which views to merge and can append a prefix to each view to help identify the data columns.

View Name:	Merged View		
This combines t multiple analyze allowed to be n	the ouput from two or more v ed curves into a single graph herged.	iews into a single view. This is con view. Only generated views (views	venient for combining that have .dat files) are
Select Current	Views to Combine:	Output File Column Prefi	xes (optional)
Magnetostric	tion View Down	Down	
Magnetostrio	tion View up	Up	
FuSi Thermal	Expansion Coeff View		
Cu Thermal E	xpansion Coeff View		

Figure 4-30: Merged View

## 4.4 Analysis Models

### 4.4.1 Tool Chain

The tool chain is the primary means for data processing used by the Dilatometer software. The tool chain works by piping data through a series of widgets that are each designed to perform a specific type of manipulation on the data. Typically, the tool chain will be handled by the Option Controller in view production. However, the benefit of the tool chain is that advanced users may access the widgets individually outside of the Option Controller and perform custom post-processing. (Refer to the Appendices for more information on accessing and customizing the widgets.) Below is a brief description of each widget. All of the widgets can be found in \Dilatometer\system.

#### 4.4.1.1 CAPTURE

The Dilatometer Capture Widget, dilcapture.exe, reads directly from the CAN module and writes data in JSON format. The output is saved in a .sjson file and serves as the source for all the data analysis carried out by the Dilatometer software. The Dilatometer Capture Widget is the only

Quantum Design

widget that communicates with the system hardware. This widget is not intended for use outside of the Option Controller.

#### 4.4.1.2 SLICER

The primary function of the Dilatometer Slicer Widget, dilslicer.exe, is to read .sjson files from the Capture Widget and outputs for a .dat file. Thermal resistance rate, cell imbalance rate and field rate are all calculated. Linear interpolation is used on system temperature, chamber pressure and field data. This widget is also not intended for use outside of the Option Controller.

#### 4.4.1.3 FILTER

The Generic Filter Widget, MVFilter.exe, reads and writes MultiVu-style .dat files and performs averaging operations on the data. Mean, midpoint, median, range, standard deviation and slope can be applied to various data subsets. The user may wish to use this widget as a standalone executable.

#### 4.4.1.4 TRANSFORM

The Transform Widget, MVTransform.exe, reads and writes MultiVu-style .dat files and can perform a number of operations on elements of a single data row. The Transform Widget performs operations on elements in a single data time stamp. The user may wish to use this widget as a standalone executable.

#### 4.4.1.5 MERGE

The Merge Widget, MVMerge.exe, takes two or more .dat files and collates them into a single file. The user may wish to use this widget as a standalone executable.

# Calibration

## 5.1 Introduction

This chapter contains the following information.

• Section 5.2 addresses the important factors necessary in measurement correction.

## 5.2 Measurement Correction

### 5.2.1 Thermal Expansion Measurement Background

The Dilatometer thermal expansion measurement is composed of two distinct factors: sample thermal expansion and cell thermal expansion. The cell thermal expansion will be defined as *the measurement background* ( $\Delta L$  background). If the cell were ideal, the measurement background could be taken directly from literature. In practice, a number of small imperfections create an error that must be accounted for. In this case, the cell is constructed from fused silica to take advantage of the small thermal expansion. This is referred to as the *relative effect* (equation 5.1).

$$\Delta L^{Sample} - \Delta L_{Background} = \Delta L^{Sample}_{Measured}$$
(5.1)

Equation 5.1 is rearranged to represent the corrected thermal expansion measurement in equation 5.2.

$$\Delta L^{Sample} = \Delta L^{Sample}_{Measured} + \Delta L_{Background}$$
(5.2)

Quantum Design

The relative effect shows how the  $\Delta L^{sample}_{measured}$ ,  $\Delta L_{background}$ , and  $\Delta L^{sample}$  are all related. As previously mentioned, if the fused silica cell was perfect,  $\Delta L_{background}$  would be equal to the thermal expansion of published fused silica. Therefore, the small imperfections can be solved by measuring a fused silica sample to get  $\Delta L_{background}$  in equation 5.3. A fit can be created *for*  $\Delta L_{background}$  using the Dilatometer software to represent the cell effect.

$$\Delta L_{Background} = \Delta L^{FuSi}_{Literature} - \Delta L^{FuSi}_{Cell \ Effect}$$
(5.3)

Combining equations 5.2 and 5.3 results in the complete representation of thermal expansion measurement corrections.

$$\Delta L^{Sample} = \Delta L^{Sample}_{Measured} + \Delta L^{FuSi}_{Literature} - \Delta L^{FuSi}_{Cell \ Effect}$$
(5.4)

Equation 5.4 represents the total correction in terms of non-normalized dilation. However, the more significant measurement is the thermal expansion coefficient,  $\alpha$ . This is represented by equations 5.5 and 5.6.

$$\alpha^{Sample} = \alpha^{Sample}_{Measured} + \alpha_{Background}$$
(5.5)

$$\alpha_{Background} = \alpha^{FuSi}_{Literature} - \alpha^{FuSi}_{Cell \, Effect} \tag{5.6}$$

Based on equation 5.5, any sample measured can be corrected using the measurement background in equation 5.6. The two terms used to determine the background will be addressed in more detail in the following section on reference tables.

It is important to note that the composition and manufacturing process of fused silica can change its thermal expansion coefficient significantly. To calculate the most accurate background from measured cell effect, it is best to only use the fused silica sample which is provided by Quantum Design and matches the cell material.

### 5.2.2 Reference Tables

The Dilatometer Option uses two reference tables to obtain  $\alpha$ : copper standard and fused silica standard. The Dilatometer Option software uses interpolation methods to create continuous references from all of these tables.

Two Standard reference tables are provided for the Dilatometer software.  $\alpha^{Copper}_{Literature}$ , thermal expansion coefficient [ppm/K] vs. sample temperature [K], can be used to verify a copper measurement.  $\alpha^{FuSi}_{Literature}$ , thermal expansion coefficient [ppm/K] vs. sample temperature [K], is the other reference table used in calculating the measurement background.

The fused silica standard reference table is taken from two publications, Okaji 1995 and White 1999. The copper standard reference is taken from Kroeger and Swenson 1977 and White 1999.

## 5.2.3 The Cell Constant

The cell constant,  $\theta$ , is essential to all thermal expansion and magnetostriction measurements. Each capsule will be shipped along with a cell constant value; however, the user may wish to verify or change the cell constant. For instruction on calculating and saving the cell constant see Section 5.3.3.

The Dilatometer Option directly measures change in imbalance. In order to convert imbalance to dilation, the cell constant is used, defined in equation 5.7. This number varies from cell to cell and is expressed in millimeters over imbalance.

$$\Delta L \ [mm] = \theta \Delta I \qquad where \ \theta = \frac{Thermal \ Expansion \ [mm]}{Imbalance} \tag{5.7}$$

When calculating the cell constant, a sample with known thermal expansion is needed, typically copper. From equation 5.4, a similar expression can be derived for a copper sample.

$$\Delta L^{Copper}_{Literature} = \Delta L^{Copper}_{Measured} + \Delta L^{FuSi}_{Literature} - \Delta L^{FuSi}_{Cell \, Effect}$$
(5.8)

Combining equation 5.7 and equation 5.8, we get an expression for the cell constant.

$$\frac{\Delta L^{Copper}_{Literature} - \Delta L^{FuSi}_{Literature}}{\Delta I^{Copper}_{Measured} - \Delta I^{FuSi}_{Cell Effect}}$$
(5.9)

To solve for  $\Delta I^{Copper}_{Measured}$  and  $\Delta I^{FuSi}_{Cell Effect}$ , thermal measurements of Copper and fused silica must be taken. Fused silica has equal sample length at two temperatures, 79.9 K and 300 K. By comparing the values at the two temperatures an accurate cell constant is obtained. For convenience let  $\Delta L^{Copper}_{Literature} = \Delta L_{Cu}$ ,  $\Delta L^{FuSi}_{Literature} = \Delta L_{FuSi}$ ,  $\Delta I^{Copper}_{Measured} = \Delta I_{Cu}$ and  $\Delta I^{FuSi}_{Cell Effect} = \Delta I_{FuSi}$ .

$$\theta = \frac{\left(\Delta L_{Cu}(79.9) - \Delta L_{Cu}(300)\right) - \left(\Delta L_{FuSi}(79.9) - \Delta L_{FuSi}(300)\right)}{\left(\Delta I_{Cu}(79.9) - \Delta I_{Cu}(300)\right) - \left(\Delta I_{FuSi}(79.9) - \Delta I_{FuSi}(300)\right)}$$
(5.10)

$$\Delta L_{FuSi}(79.9) = \Delta L_{FuSi}(300) \tag{5.11}$$

$$\theta = \frac{\left(\Delta L_{Cu}(79.9) - \Delta L_{Cu}(300)\right)}{\left(\Delta I_{Cu}(79.9) - \Delta I_{Cu}(300)\right) - \left(\Delta I_{FuSi}(79.9) - \Delta I_{FuSi}(300)\right)}$$
(5.12)

Once this number is calculated and saved to the capsule's calibration data file, it may be used for all future samples that are measured in the same capsule.

# **Thermal Expansion Measurements**

## 6.1 Introduction

This chapter contains the following information.

- Section 6.2 presents an overview of the thermal expansion measurement.
- Section 6. 3 explains how to perform a thermal expansion measurement.

## 6.2 **Overview of Thermal Expansion Measurements**

The Dilatometer Option performs measurements of the sample's thermal expansion coefficient and dilation. The Dilatometer software measures imbalance while ramping the system temperature. The software then uses the cell constant to solve for dilation, calculates the thermal expansion coefficient, and adds the cell background for correction. This calibration technique is discussed thoroughly in Section 5.2.1. Having both the cell constant and an accurate fused silica thermal expansion baseline table is essential to the thermal expansion measurement of a sample.

The measurement begins with preparing and mounting the sample, opening the Option Controller in MultiVu, and loading the probe into the PPMS system through the wizard. The wizard will prompt the user to enter in specific information concerning the setup. After selecting the thermal expansion measurement option, parameters will be entered for the specifics of the measurement. Once initiated, data will be available to the user to start creating views in real time and watch the progression of the measurement.

When a thermal expansion measurement is running, the sample chamber is purged. It is important to purge and seal the chamber rather than set the system to high vacuum mode. Having an exchange gas in the chamber is essential for an even heat distribution.

Once the measurement is initiated, the system will begin to search for tap setting closest to balancing the system. After the tap has been set, the PID control loop will be closed, and the system will converge to the balance point by adjusting the drive voltages. Upon initiating the measurement, the system will begin to take measurements at a rate of 2 samples per second. Because this rate is set as a constant, changing the thermal ramp rate will change the number of data points collected.

Once the measurement has ended the system will remain stable at temperature until the user commands a change. The probe must not be removed unless the system is at room temperature, vented and at zero field. All of this is done by the wizard apart from returning the system to zero field.

## 6.3 **Performing a Measurement**

### 6.3.1 Mount the Sample and Prepare the System

The following is a checklist to ensure all the necessary steps have been taken before starting a measurement. This list is included as a reminder and is not meant to be comprehensive. Refer back to Chapter 2 for instructions on cleaning, installation and getting started.

- □ Make sure that the Dilatometer module is installed and communicating with the computer.
- □ Clean the Kapton, mounting tools and sample. It is up to the user whether or not the capsule needs cleaning.
- $\Box$  Mount the sample.
- □ Using the Balance Meter, find the initial imbalance of the cell. If the imbalance reading is within ±0.5, the sample is ready for measurement. If the sample has an unknown thermal expansion, the imbalance should be as close to zero as possible. At this point it is optional to test the capsule thermometry.
- $\Box$  Slide on the radiation shield and screw on the capsule base.
- □ Install the capsule to the probe. Remember that due to the range in conditions the capsule will undergo, the screws should be fully in, but not overly tightened.
- □ Open the appropriate version of MultiVu and activate the Dilatometer Option Controller with the command Utilities >> Activate Option >> Dilatometer >> Activate.
- □ Select the **Wizard...** tab and follow the instructions for the final steps on preparing the system for measurement (Section 2.6.3).
## 6.3.2 Run the Thermal Expansion Measurement

Once the capsule, probe and cable have all been installed through the wizard, the measurement can begin. To initiate a thermal expansion measurement, select **Measure... >> Measure Thermal Expansion (vs T)** on the main dialog of the Dilatometer Option Controller. Figure 6-1 will open. The following are important to remember when setting the measurement parameters.

• Once the measurement begins, the system will ramp to the Start Temperature at its fastest temperature rate. This rate varies by system.

📜 Thermal Expansio	n Measuren	ent (vs T)	<u>_   ×</u>
Scan Name:	Temperature	Scan	
Comments:			
	I		
This will perform a	thermal expan	sion measurement by automatically ramping the temp	erature.
Start Temperatur	re (K)	300	
End Temperature	(К)	2	
Rate (K/min)		0.1	
Acquisition Mode	:	Standard Resolution	
Settle Time (secs	) at Start?:	0	
Start Streaming Ne	w View of Thi	Measurement	
		OK	Cancel
		- OK	

Figure 6-1: Thermal Expansion Measurement (vs T)

- Cancel returns the user back to the Dilatometer Option Controller main dialog.
- Be sure to use a scan name and add comments that will clearly distinguish the measurement for post-processing views.
- It is important to set the settle time at start to a high enough value that the system will find an equilibrium at the particular temperature.
- Remember that the sample collection rate is constant. Therefore, the lower the temperature ramp rate, the more samples that will be collected, and the more accurate the measurement will be.
- Once the temperature ramp ends, the system will remain at the End Temperature.

Notice in the bottom left hand corner there is an option for opening a streaming view of the measurement. If selected, a new view will be created with default configuration values. These default values are described in Section 4.3.5.1. For custom values, initiate the measurement without this box checked and create a new view from the main dialog measurement status pane.

Complete each field entry and select **OK** to initiate the measurement.

The alternative to running a thermal expansion measurement is to start a continuous measurement. Once the continuous measurement begins, the user has the option of manually changing the settings of the system in MultiVu or using sequence commands to automate a measurement process. For more information on sequence commands see Section 4.3.3.4.

#### 6.3.2.1 ABORTING A MEASUREMENT

You may abort a measurement at any time. To abort a measurement, select **Abort** in the main dialog measurement status pane. Unlike other PPMS options, you may not pause a Dilatometer measurement through the Option Controller. Aborting a measurement will stop data collection and the temperature ramp. Once aborted, the temperature will remain steady until prompted to do otherwise.

If the measurement is running in continuous mode, aborting will only stop the collection of data by the Option Controller. MultiVu sequences can be paused or aborted separately. See Section 4.3.3.4 for more on sequences.

### 6.3.3 Probe Removal Procedure by Wizard

The wizard is used to remove the probe when the system has reached room temperature, zero field and no measurement is in process. Once the probe is removed, the capsule and sample can be removed. The capsule should immediately be stored back in the Pelican<sup>TM</sup> box provided by Quantum Design.

Start by selecting the **Wizard...** tab again. The process is very similar to installing the probe. All of the figures for this set of instructions can be found in Section 2.6.3.

- Select **Open Chamber**. This will bring the system to room temperature, seal off any helium flow and bring the chamber pressure to atmosphere.
- Unplug the cable that connects the probe to the module from the probe head. Remember to pull on the collared portion of the cable that has the red dot. This releases a locking mechanism in the cable.
- Unclip the two black clamps that secure the probe to the PPMS.
- Gently slide the probe out of the PPMS and lay it on a table.
- Place the PPMS sample chamber baffle back on (including the O-ring). This will seal the sample chamber.
- Select **Purge** in the wizard. It is good practice to leave the system purged with the baffle on when not in use. Because there is no probe, select Cancel in the wizard, and do not enter new sample or cell properties.



#### WARNING!

Do **NOT** remove the probe from the system when the sample chamber is below room temperature. Room air will flood the chamber and water molecules will freeze onto its sides, creating issues for future measurements. It is safe to always use the wizard for removing the probe, as it will warn against this.

The following steps should be followed in order to remove the sample from the capsule.

• Only the pointed and flat tweezers are needed to remove the sample and the spring. Using clean tweezers is recommended. If the user wishes to run consecutive measurements using the same capsule, this will avoid having to clean the cell and capsule a second time.

- Begin by removing the spring. The best practice is to grip the top of the spring with the flat tweezers. Using the pointed tweezers, compress the middle of the spring until it is not in contact with the cell. Be careful not to place the ends of the tweezers in the sample groove, as this can damage the cell edges. Slowly remove the spring and set it on a clean surface or back in the bag provided with the user kit. Be careful that the spring is compressed before removing it. Failure to do so will drag the spring edges along the surface of the cell and quickly chip away at delicate areas such as corners.
- Using the pointed tweezers, gently remove the sample, gripping it by either end of its longest axis. Again note that touching the sample on either end of its measurement axis is not advised, as it could compromise the parallel surfaces necessary for future measurement.



### WARNING!

Do **NOT** place the ends of the tweezers inside the cell grooves. This will damage the grooves and potentially the capacitance gap.

# **Magnetostriction Measurements**

## 7.1 Introduction

This chapter contains the following information.

- Section 7.2 presents an overview of the magnetostriction measurement.
- Section 7. 3 explains how to perform a magnetostriction measurement.

## 7.2 **Overview of Magnetostriction Measurements**

The Dilatometer Option performs measurements of the sample's magnetostriction coefficient and magnetic strain.

The Dilatometer software measures imbalance while ramping the system field. The software then uses the cell constant to solve for magnetic strain and calculates the magnetostriction coefficient. Having an accurate cell constant is important for this measurement.

The measurement begins with preparing and mounting the sample, opening the Option Controller in MultiVu and loading the probe into the PPMS system through the wizard. The wizard will prompt the user to enter in specific information concerning the setup. After selecting the magnetostriction measurement, parameters will be entered for the specifics of the measurement. Once initiated, data will be available to the user to start creating views in real time and watch the progression of the measurement.

When a magnetostriction measurement is running, the sample chamber is purged. It is important to purge and seal the chamber rather than set the system to high vacuum mode. Having an exchange gas in the chamber is essential for an even heat distribution. Magnetostriction measurements are commonly taken at temperatures other than 300 K.

Once the measurement is initiated, the system will begin to search for a tap setting closest to balancing the system. After the tap has been set the PID control loop will be closed, and the system will converge to the balance point by adjusting the drive imbalance. Upon initiating the measurement, the system will

begin to take measurements at a rate of 2 samples per second. Because this rate is set as a constant, changing the field ramp rate will change the number of data points collected.

Once the measurement has ended, the system will remain stable at temperature until the user commands a change. The probe must not be removed unless the system is at room temperature, vented and at zero field. All of this is done by the wizard, except for returning the system to zero field.

## 7.3 **Performing a Measurement**

### 7.3.1 Mount the Sample and Prepare the System

The following is a checklist to ensure all the necessary steps have been taken before starting a measurement. This list is included as a reminder and is not meant to be comprehensive. Refer back to Chapter 2 for instructions on cleaning, installation, and getting started.

- □ Make sure that the Dilatometer module is installed and communicating with the computer.
- □ Clean the Kapton, mounting tools and sample. It is up to the user whether or not the capsule needs cleaning.
- $\Box$  Mount the sample.
- Using the Balance Meter, find the initial imbalance of the cell. If the imbalance reading is within  $\pm 0.5$ , the sample is ready for measurement. If the sample has an unknown thermal expansion, the imbalance should be as close to zero as possible. At this point it is optional to test the capsule thermometry.
- □ Choose the cell angle and lock it into place with the set screw using the 3/32-inch Allen key found in the Dilatometer user kit. Instructions for this are found in this chapter in Section 7.3.1.1.
- $\Box$  Slide on the radiation shield and screw on the capsule base.
- □ Install the capsule to the probe. Remember that due to the range in conditions the capsule will undergo, the screws should be fully in, but not overly tightened.
- □ Open the appropriate version of MultiVu and activate the Dilatometer Option Controller with the command Utilities >> Activate Option >> Dilatometer >> Activate.
- □ Select the **Wizard...** tab and follow the instructions for preparing the system for measurement (Section 2.6).

#### 7.3.1.1 SELECTING AND CHANGING THE CELL ANGLE

An important feature of magnetostriction measurements with the Dilatometer Option is that the cell angle, and consequently the sample angle, can be changed with respect to the orientation of the field.

The angle options are marked on the capsule, surrounding the spherical cell housing around the top of the cell. There is a single mark on the spherical cell housing to indicate the angle that is chosen. The marks each indicate a change of 10 degrees. The options provided are -20° ranging to 110°. The following instructions are given for changing the angle. The angle should always be changed while the capsule is mounted in the Balance Meter.

- Place and secure the capsule in the Balance Meter.
- Through the capsule securing screw on the Balance Meter, there is a hole for accessing the set screw for the cell angle (Figure 7-2). Turning the screw requires a 3/32-inch Allen key. This is provided in the user kit. Unscrew the angle set screw <sup>1</sup>/<sub>4</sub> turn. It is extremely important that the screw is only loosened. If unscrewed too far, the spherical cell housing is likely to fall out and the cell could be damaged.
- Once the spherical housing is loose, gently rotate to the preferred angle. Refer to Figure 7-1 for angle reference.
- When the mark on the sphere housing matches the desired angle mark, firmly tighten the angle set screw.

Be sure to record the angle you are measuring, as you will be prompted to input the parameter to the Dilatometer Option Controller. Refer to Section 7.3.4 for this step.



Figure 7-1: Selecting the Cell Angle



Figure 7-2: Accessing the Angle Set Screw

Note:

Figure 7-1 may be misleading. The numerical angle indicators are **NOT** engraved on the capsule.

## 7.3.2 Run the Magnetostriction Measurement

Once the capsule, probe and cable have all been installed through the wizard, the measurement can begin. To initiate a magnetostriction measurement, select **Measure...** >> **Measure Magnetostriction (vs H)** on the main dialog of the Dilatometer Option Controller. Figure 7-1 will open. The following are important to remember when setting the measurement parameters.

Once the measurement begins, the system will ramp to the Start
 Field at its fastest field rate. This rate varies by system.

🚺 Magnetostriction I	Measurement (vs H)	- 🗆 🗡
Scan Name:	Field Scan	
Comments:		
This		
This will perform a n	nagnetostriction measurement by recording expansion data while sweeping reid.	
Start Field (Oe)	-90000	
End Field (Oe)	90000	
Rate (Oe/sec)	10	
Acquisition Mode:	Standard Resolution	
Settle Time (secs)	at Start?: 0	
Start Streaming Nev	v View of This Measurement	
	OK Can	icel

Figure 7-3: Magnetostriction Measurement (vs H)

- **Cancel** returns the user back to the Dilatometer Option Controller main dialog.
- Be sure to use a scan name and add comments that will clearly distinguish the measurement for post-processing views.
- It is important to set the settle time at start to a high enough value that the system will find an equilibrium at the particular field.
- Remember that the sample collection rate is constant. Therefore, the lower the field ramp rate, the more samples that will be collected, and the more accurate the measurement will be.
- Once the field ramp ends, the system will remain at the End Field.

Notice in the bottom left hand corner there is an option for opening a streaming view of the measurement. If selected, a new view will be created with default configuration values. These default values are described in Section 4.3.5.2. For custom values, initiate the measurement without this box checked and create a new view from the main dialog measurement status pane.

Complete each field and select **OK** to initiate the measurement.

The alternative to running a magnetostriction measurement is to start a continuous measurement. Once the continuous measurement begins, the user has the option of manually changing the settings of the system in MultiVu or using sequence commands to automate a measurement process. For more information on sequence commands see Section 4.3.3.4.

#### 7.3.2.1 ABORTING A MEASUREMENT

You may abort a measurement at any time. To abort a measurement, select Abort in the main dialog, measurement status pane. Unlike other PPMS options, you may not pause a Dilatometer measurement through the option controller. Aborting a measurement will stop data collection and the magnetostriction measurement field ramp. Once aborted, the field and temperature will remain steady until prompted to do otherwise.

If the measurement is running in continuous mode, aborting will only stop the collection of data by the Option Controller. MultiVu sequences can be paused or aborted separately. See Section 4.3.3.4 for more on sequences

## 7.3.3 Probe Removal Procedure by Wizard

The wizard is used to remove the probe when the system has reached room temperature, zero field and no measurement is in process. Once the probe is removed, the capsule and sample can be removed. The capsule should immediately be stored back in the Pelican<sup>TM</sup> box provided by Quantum Design.

Start by selecting the **Wizard...** tab again. The process is very similar to installing the probe. All of the figures for this set of instructions can be found in Section 2.6.3.

- 1. Select **Open Chamber**. This will bring the system to room temperature, seal off any helium flow and bring the chamber pressure to atmosphere.
- 2. Unplug the cable that connects the probe to the module from the probe head. Remember to pull on the collared portion of the cable that has the red dot. This releases a locking mechanism in the cable.
- 3. Unclip the two black clamps that secure the probe to the PPMS.
- 4. Gently slide the probe out of the PPMS and lay it on a table.
- 5. Place the PPMS sample chamber baffle back on (including the O-ring). This will seal the sample chamber.
- 6. Select **Purge** in the wizard. It is good practice to leave the system purged with the baffle on when not in use. Because there is no probe, select **Cancel** in the wizard and do not enter new sample or cell properties.



#### WARNING!

Do **NOT** remove the probe from the system when the sample chamber is below room temperature. Room air will flood the chamber and water molecules will freeze onto its sides, creating issues for future measurements. It is safe to always use the wizard for removing the probe, as it will warn against this.

The following steps should be followed in order to remove the sample from the capsule.

1. Unlike the many materials involved in the mounting process, only the pointed and flat tweezers are needed to remove the sample and the spring. Using clean tweezers is recommended. If the user wishes to run consecutive measurements using the same capsule, this will avoid having to clean the cell and capsule a second time.

- 2. Begin by removing the spring. The best practice is to grip the top of the spring with the flat tweezers. Using the pointed tweezers, compress the middle of the spring until it is not in contact with the cell. Be careful not to place the ends of the tweezers in the sample groove, as this can damage the cell edges. Slowly remove the spring and set it on a clean surface or back in the bag provided with the user kit. Be careful that the spring is compressed before removing it. Failure to do so will drag the spring edges along the surface of the cell and quickly chip away at delicate areas such as corners.
- 3. Using the pointed tweezers, gently remove the sample, gripping it by either end of its longest axis. Again note that touching the sample on either end of its measurement axis is not advised, as it could compromise the parallel surfaces necessary for future measurement.



### WARNING!

Do **NOT** place the ends of the tweezers inside the cell grooves. This will damage the grooves and potentially the capacitance gap.

# **Troubleshooting and Maintenance**

## 8.1 Introduction

This chapter contains the following information.

- Section 8.2 discusses the importance and procedure for keeping the hardware clean.
- Section 8.3 provides example data and explains common issues in Dilatometer measurements.
- Section 8.4 discusses problems that indicate bad electrical connections and how to locate said connection.
- Section 8.5 discusses indications of a broken cell and the procedure for repairs through Quantum Design

## 8.2 Contamination

Small amounts of contamination will lead to poor measurement quality. This can come from dirty tools or samples, left over wax on the sample or residue from previous samples. To prevent this, it is important to keep the cell and capsule clean and keep the PPMS system maintained. Figure 8-1 shows three data sets to demonstrate the effects of contamination: red is standard copper data from literature, green is measured copper data before cleaning and blue is measured copper data after cleaning.



Figure 8-1: Evidence of Contamination

## 8.2.1 Cleaning the Cell and Capsule

The procedure for cleaning a cell is outlined in Section 2.4.2. When the capsule has been in an environment in which it is likely to have attracted dust particles or other contaminants, it is advised to clean the capsule before taking measurements. If, however, the cell is kept in its case, handled with care and only moved from the PPMS system to the Balance Meter, cleaning may be less frequent. The user can always wait until bad data begins to appear before cleaning the cell again. When the radiation shield and capsule exterior become dirty, gently clean the surface with an alcohol wipe.



#### WARNING!

Do **NOT** use the ultrasound bath technique to clean the Dilatometer capsule. This will damage to the cell

## 8.2.2 Maintaining the Sample Chamber

Keeping the sample chamber on the PPMS system clean can be just as important. Contact the Quantum Design Service Department for more information on cleaning the system. The following are basic guidelines for maintaining the sample chamber.

- Always keep the sample chamber closed. If there is no probe in the system, always replace it with the baffle that came with your PPMS system.
- Keep the O-ring near the probe clamps in good condition. Failure to do so will allow contaminants into the sample chamber. To find this O-ring, turn the probe upside down with the head resting on a table and the shaft in the air. The O-ring is positioned surrounding the shaft, on the surface of the probe head.

• Keep the sample chamber free from grease. Some PPMS options use grease as a thermal conductor in the sample chamber. If one of these options has been recently used, it is advised to clean the sides of the chamber with alcohol before using the Dilatometer Option. Take care not to damage the electrical pins at the base of the sample chamber.

## 8.3 Common Mistakes and Interesting Results

This section addresses a few common issues with the Dilatometer Option: the causes, the effects shown in plotted data and possible solutions. The information provided in this section by no means represents a comprehensive list of things that could go wrong with a measurement.

The best test for measurement accuracy is to measure a copper sample and compare the results with the standard copper reference table. This method is shown in Figure 8-1. Copper has a relatively large thermal expansion, and thus issues in the measurement will be very noticeable.

Another good test is to run the sample through multiple temperature scans before looking at the data. Taking the sample from 2 K to 400 K a few times can help settle the sample into its mounted position and produce higher quality data. This is not necessary for all samples.

### 8.3.1 Moisture in the Sample Chamber

Moisture in the sample chamber is an issue. When this occurs, a spike, referred to as a water peak, is present in the data around 300 K. Figure 8-2 shows thermal expansion coefficient data from a fused silica sample during a temperature ramp from 1.8 K to 400 K.

As the system cools, the water vapor freezes. When the system temperature begins to rise, the ice liquefies very briefly, and at roughly 270 K it vaporizes due to the low pressure of the chamber. This is precisely where the data begins to go askew, degrading the quality of the measurement.



Figure 8-2: Thermal Expansion Data with a Water Peak

If there appears to be moisture in the sample chamber, the best method for drying is to heat the system as much as possible and start a continuous vacuum. Heat the chamber to as close to 400 K as the sample can handle. To do this, select **Instrument >> Temperature... >> Set Point >> Set** in MultiVu. Once the chamber has reached the chosen temperature, start the continuous vacuum by selecting **Instrument >> Chamber...>> Pump Cont.** in MultiVu.

## 8.3.2 Sample Falling Out

There is a chance that the sample could fall out of the cell during a measurement, due to improper sample mounting. If the spring falls out, the sample will most likely fall out, leaving no tension on the cell grooves. By observing the measurement feedback, it will be very evident there is an issue.

Cell imbalance is the user's first check for noticing issues in the measurement. Figure 8-3 displays the effects of a sample falling out of the cell groove. This measurement was done on a fused silica sample which should have extremely low thermal travel. Around



Figure 8-3: Measurement Data with Sample Falling Out

minute 204 the data jumps drastically, passing the zero imbalance point and settling at a much higher tap value. By connecting an oscilloscope to the BNC ports, JR-2 and JR-3, on the module front panel, the user may observe increased signal noise as well.

If the user suspects the sample has fallen out, simply abort the measurement, bring the system back to 300 K, remove the probe, and remount the sample. If each step of the sample preparation and mounting is handled with care, this should not be a frequent issue.

## 8.4 Troubleshooting Electrical Connections

Another issue that can arise in the Dilatometer Option is broken electrical connections. There are three possible places that the connections can go bad: the capsule, the probe and the cable. This section will address both how to recognize when an electrical connection is broken and how to find the source of the issue. If the problem is found to be in the capsule, it must be sent back to Quantum Design for repairs. If the problem is found in the probe or cable, a local Quantum Design service employee can come to fix the issue.

One way that an issue in the wiring can be noticed is through the Dilatometer Option software parameters. The most likely candidates will be total imbalance, sample temperature and system temperature. If the sample temperature and the system temperature are not closely following one another in a temperature ramp this indicates issues with the thermometry wiring. If the total imbalance is resting at  $\pm 0.5$ , the system has railed and there is an issue in either the signal or the drive wiring. A second way to notice issues in the electrical connections is by using the Balance Meter. When the sample has been mounted and the capsule is resting in the Balance Meter, check the imbalance and the thermometry. If the imbalance reading is  $\pm 0.998$ , one of two things could be occurring. Either your sample is significantly too large, significantly too small or there is a problem with the signal or drive wiring.

Once the issue is noticed, continuity checks can be run with a multi-meter on the probe and cable. First make sure all the connections are not grounded. Next begin individually checking the continuity from both sides of the device. For information on the cable connections, refer to Section 3.2.2. Using Table 3-. and Figure 8-4, the probe can be checked.



Figure 8-4: Probe Connections Diagram from cable view and capsule view, respectively)

If the probe and cable thermometry seems to be functioning properly, rest the capsule in the Balance Meter, and with the help of a multi-meter, check the resistance between the terminals. If there is no issue with the thermometer, the readings should be high (around 70 Ohms) for terminals of opposite sign and low (around 2 Ohms) for terminals of the same sign.

If the issue is found with the Balance Meter and the sample size has been determined to be acceptable, refer to Section 8.5.1 for Quantum Design Repairs.

## 8.5 Broken Cell

There are two indications that the cell is broken. The first has been covered in the previous section on electrical connections. There must be a bad connection inside of the capsule. The second is a fracture in the cell hinges. The cell is unable to perform measurements with a broken hinge. The best way to tell if a hinge is broken is by very gently moving the upper half of the cell back and forth and examining the hinges under a microscope. If they do not move together, one is broken. This issue is easy to identify once it is looked for.

## 8.5.1 Quantum Design Repairs

For help with repairs, please contact your local Quantum Design service representative. See www.qdusa.com for information concerning your local representative. You will be asked to describe the problem, the circumstances involved and the recent history of your system.

### A P P E N D I X A

# Widgets

## A.1 Introduction

This appendix contains the following information.

- Section A.2 presents an overview of the Dilatometer Capture Widget.
- Section A.3 describes Dilatometer Slicer Widget.
- Section A.4 provides an overview of the Generic Transform Widget.
- Section A.5 describes the Generic Filter Widget.
- Section A.6 offers an overview of the General Merge Widget.

## A.2 Dilatometer Capture Widget

### A.2.1 Overview

The Dilatometer Capture Widget (DCW) reads from hardware buses and does no data conditioning at all. It saves packets in streaming JSON (.sjson). It is the only widget that talks to hardware. The output data is the raw original data and should never be overwritten.

In addition to dilatometer data, the DCW also captures a set of base-system data like System Temperature, Chamber Pressure, Field, Temperature Status and some others.

To aid in downstream reading, we terminate reads with a comma and carriage return. A single "JSON object" (aka dictionary) is normally meant to correspond to a single timestamp.

## A.2.2 Configuration File

There is no configuration file for the Capture Widget.

## A.2.3 Command Line

#### A.2.3.1 STARTING THE WIDGET

DilCapture.exe <options>

All options are traditional Unix style long-form parameters with double dashes and dashed names, like so: --my-param=myvalue.

A.2.3.1.1 PARAMETERS	
help	Gives the help text.
port=n	Specifies the optional startup port for communicating with this widget via the TCP socket.
output="filename"	Specifies the output file path and name. This is the main .sjson output file. By default, this file is located in the same folder as the executable but with a .sjson extension. This file is NEVER overwritten.
command-dump="filename" Saves all commands (with a timestamp) received on the command port to a file along with any response strings. This file is NOT overwritten.	

#### A.2.3.2 RUNNING THE WIDGET

Typing the single letter, lower case or capital, commands A (Abort), P (Pause), R (Resume), S (GetStatus) are valid commands at the command prompt.

## A.3 Dilatometer Slicer Widget

### A.3.1 Overview

The Dilatometer Slicer Widget (DSW) reads the streaming JSON (.sjson) output from the DCW (DilCapture.exe) and streams out a MultiVu-type output file from the input.

The .sjon file contains data from disparate sources that are collected at different frequencies. The DSW combines and interpolates them to create output rows that each contain all the necessary output fields at a single time-stamp value.

## A.3.2 Configuration File

The configuration file consists of a single JSON dictionary that defines all the transformations that will be applied to the raw .sjon file to produce the output file. Here is an example illustrating the structure:

```
"view name": "Thermal Expansion 3",
  "scans": [
       "scan_start": 3675617088.201,
       "scan_end": 3675655393.0,
       "scan header": {
         "cell constant": 0.168,
         "sample length": 1.998,
         "dilation offset": 0.0,
         "offset mode": "set",
         "rotator angle": 0.0,
         "sample_slot": "R",
         "therm cal file":
"C:\\Qddynacool\\Dilatometer\\Calibrations\\Cells\\C123\\cache\\THD3C2543C1 smoothed.tab"
       }
     },
     ł
       "scan start": 3675665549.0,
       "scan end": 3675749947.0,
       "scan header": {
         "cell constant": 0.168,
         "sample_length": 1.998,
         "dilation_offset": 0.0,
         "offset mode": "set",
         "rotator angle": 0.0,
         "sample slot": "R",
         "therm cal file":
"C:\\Qddynacool\\Dilatometer\\Calibrations\\Cells\\C123\\cache\\THD3C2543C1_smoothed.tab"
    3
  ],
  "run_config": {
   "time_pad": 10
}
```

This sample input file generates outputs only for the timestamps bracketed by the scan\_start and scan\_end timestamps. At the beginning of each scan, insert a header delimited by BEGIN:PARAMS/END:PARAMS containing the set of key value pairs indicated in the scan\_header entry. These key value pairs will be parsed by downstream widgets.

If the scan\_start entry is missing, it is assumed to be zero. If the scan\_end parameter is zero, it is assumed to be the equivalent of infinity. You can leave both out, too. That just means to process everything in the input.

The view\_name parameter is used as the output MultiVu data file title.

## A.3.3 Command Line

#### A.3.3.1 STARTING THE WIDGET

DilSlicer.exe <options>

All options are traditional Unix style long-form parameters with double dashes and dashed names, like so: --my-param=myvalue.

#### A.3.3.1.1 PARAMETERS

help	Gives the help text.
port=n	Specifies the optional startup port for communicating with this widget via the TCP socket.
config-file="full fil	<b>ename</b> " Specifies the location of .cfg file that is read at startup. By default, this file is located in the same folder with the executable and has a .cfg extension.
input="filename"	Specifies the input file path and name. This is the main .sjson file. By default, this file is located in the same folder as the executable but with a .sjson extension.
output="filename"	Specifies the output file path and name. By default, this file is located in the same folder as the executable but with a .out.dat extension.
command-dump='	<b>'filename''</b> Save all commands (with a timestamp) received on the command port to a file along with any response strings. This file is NOT overwritten.
mode=continuous	<b>one-shot</b> Run in continuous or one-shot mode. Continuous runs forever looking for new data in the input file. One-shot will terminate when the end of the input file occurs. The default is one-shot.

#### A.3.3.2 RUNNING THE WIDGET

Typing the single, lower case or capital, letter commands A (Abort), P (Pause), R (Resume), H (Refresh), S (GetStatus) are valid at the command prompt.

## A.4 Generic Transform Widget

## A.4.1 Overview

The Generic Transform Widget (GTW) performs a number of mathematical transformations on a MultiVu data file to produce an output MultiVu data file.

Examples of operations supported:

- Set column two's value to the value of a table-lookup of column one's value, where the table is read from an external file.
- Remove, rename and add data columns.
- Set a column value to a specific constant value.
- Set a column value to another column value multiplied by a constant.
- Set a column value to another column offset by a value.
- Set column three's value to the value of column one's offset (+ or -) by the table-lookup of column two's value, where the table is read from an external file (or a dictionary).
- Set column three's values to the fractional (or percent) difference between column one's value and column two's value.

## A.4.2 Configuration File

The configuration file consists of a single JSON dictionary that defines all the transformations that will be applied to the input data file to produce the output file. Here is an example illustrating the structure:

```
"params": {"init for passthrough":true},
"register":
 "row":
            (initialized and updated for every input row)
  ſ
    {"name":"sys_temp", "in":"System Temperature (K)",
       "out":"System Temperature (K)", "init":""},
     {"name":"th res", "in":"Therm Resistance (ohms)" },
    {"name":"sample temp", "out":"Sample Temperature (K)" },
    {"name":"cell imbal", "in":"Cell Imbalance (ppm)",
       "out":"Cell Imbalance (ppm)"},
    {"name":"dilation", "out":"Dilation (ppm)"},
    {"name":"rotator_angle", "out":"Rotator Angle (deg)"}
  "global": (init at start, updated every read of a BEGIN: PARAMS block)
    {"name":"sample length", "in":"SampleLength"},
    {"name": "cell_contant", "in": "CellConstant"},
{"name": "rotator_angle", "in": "RotatorAngle", "init": "0"},
{"name": "myrowcount", "init": "0"}
  ]
},
"transform":
["sequence",
 ["setrowitem", "sample_temp",
```

```
["interpolate",
    ["getrowitemfloat","th res"],
    "c:\qdppms\...\ThermometerCal00123.tab"
   1
  ["setrowitem", "dilation",
   ["div".
    ["mult",
     ["getrowitemfloat", "cell_imbal"],
     ["getglobalfloat","cell constant"]
    ["getglobalfloat"," sample_length"]
   1
   'setrowitem", "rotator angle",
   ["getglobalfloat", "rotator angle"]
  ]
]
Ş
```

There is a params section, register section, and a transform section. The params section is for general option setting. The register section declares the variables and inputs and outputs. The transform section defines the operations that are performed on the data as each row and parameter block is processed.

#### A.4.2.1 PARAMS SECTION

This section in the configuration is used for specifying execution parameters for the widget.

- **init\_for\_passthrough:** This key attribute is true if you want all the input rows passed-through to the output file. (See the section on the Default Row Register.) The default is true.
- **timestamp\_name:** By default, the name of the timestamp column on input is derived from the "DATATYPE, TIME,<col#>" metadata in the [Header] section. Specify a different name here.
- **timestamp\_output\_name:** By default, the output timestamp name is the same as timestamp name but can be overridden to be something else here.
- **comment\_name:** By default, the name of the comment column on input is derived from the "DATATYPE, COMMENT,<col#>" metadata in the [Header] section. Override with something different here.
- **comment\_output\_name:** By default, the output comment name is the same as comment\_name but can be overridden to be something else here.

#### A.4.2.2 THE REGISTER

The Register is the declaration section for all the variables used during transform operations. It defines both input and output columns, assigns symbolic names to them and allows purely internal names. You can only manipulate data by register variable name. Row and Global registers (described below) are separate namespaces, so you can have the same name in both places.

#### A.4.2.2.1 ROW REGISTER

The row register entries define the columns that are read from the input buffer and written to the output buffer. If the init\_for\_passthrough parameter is true (the default), all the columns defined in the input file are written to the output unless explicitly named in the register and dispositioned otherwise. The row register can also define temporary intermediate values (if the "in" and "out" are both omitted).

The "values" in the register are maintained as string values, so as to accommodate any data type. For math operations, conversions are performed by the transform operations.

During processing of each input row, the row register contains values initialized and read from the input row. As the transform sequence is evaluated, the values in the row register reflect all updates from setrowitem commands. As a result, the order of the getrowitemfloat and setrowitem commands in the transform sequence are important.

#### A.4.2.2.2 PARAMETERS:

name:	Name used for manipulating the values in the register. Must be unique only within the row register.
in:	This is the full MultiVu column name, including the units, from the input file. The presence of this parameter means that the value of this register entry is updated with each read of the input file with the corresponding input column. Leaving this parameter blank means that this is not an input field.
out:	This is the full MultiVu column name on the output file. The presence of this parameter explicitly defines this column in the output MultiVu file and causes the value to be written out as each output row is written. Leaving this parameter blank means this it is not an output field.
init:	By default, the register entries are initialized to a null string after an input row is read and before the row values are copied into the register. You can specify a different initialized value using this parameter. The argument is always a string, since register values are always strings.

#### A.4.2.2.3 DEFAULT ROW REGISTER (INIT\_FOR\_PASSTHROUGH PARAM)

If the parameter init\_for\_passthrough is true, then the row register is initialized using all the columns defined in the input file. This is so that if you provide an empty config["register"]["row"] section in the config file (with init\_for\_passthrough=true), the result will be that all the columns in the input file are passed to the output file unchanged. This is handy if you want to use the transform widget for adding or modifying only a few of the columns.

Now, if you want to override these register entries, you need to create an entry with the exact same "in" parameter. This new entry will completely replace the corresponding entry in the initialized register.

#### A.4.2.2.4 GLOBAL REGISTER

The global register contains values that are initialized at program start and remain persistent for the duration of execution, with one important exception. The values are automatically updated when a BEGIN:PARAMS/END:PARAMS section is encountered in the comment column on the input rows and there is a parameter key name in that section that matches one of the "in" parameters in the global register. The global register values can also be updated with the "setglobal" transform command.

During processing, the BEGIN:PARAMS/END:PARAMS sections in the input rows are passed through to the output without any changes. While global register entries are updated when matching keys are encountered in BEGIN:PARAMS/END:PARAMS sections, there is no direct effect on the output BEGIN:PARAMS/END:PARAMS sections when a "setglobal" command is processed. Another way of saying this is that the widget only modifies row values and so there is no "out" parameter available for the global register entries.

A.4.2.2.5 PARAME	TERS:
name	Must be unique only within the global register.
in	This is the key value name in the BEGIN:PARAMS/END:PARAMS section of the input MultiVu file. The absence of this parameter means it is a simple global that is not automatically updated.
init:	By default, the register entries are initialized to a null string at program start.

#### A.4.2.2.6 IDENTIFYING THE COMMENT COLUMN

Comment column is identified on the input file by the "DATATYPE, COMMENT,<col#>" meta data in the [Header] section of the input file.

#### A.4.2.3 THE TRANSFORM COMMAND TREE

The transform section defines the transformation operations that are performed on each input row to generate an output row. The rules are presented as a nested array that is evaluated from left to right, depth first. It is "compiled" for efficiency at the start and recompiled whenever a BEGIN:PARAMS/END:PARAMS section is processed on the input file. The first element in each array is a command, followed by a parameter list.

Example:

[ "div", 2.0, 10.0 $] \rightarrow 0.2$ 

Each element in an array can be a string, float, or another array that when compiled and evaluated returns a string or float (never another array, though).

If you do not specify a "transform" entry in the configuration file (you leave it blank), it is assumed to be ["sequence"], which is an empty sequence. This can be useful if the only operations you want to perform are pruning or renaming columns from the input file, in which case the register section contains all the needed information.

["ADD", <VALUE1>, <VALUE2>] COMMAND

This adds value1 and value2.

"SUB", <VALUE1>, <VALUE2>] COMMAND

This subtracts value2 from value1.

"MULT", <VALUE1>, <VALUE2>] COMMAND

This multiplies value1 and value2.

["DIV", <VALUE1>, <VALUE2>] COMMAND

This divides value1 by value2.

"IF", <OPERATION>, <ARG1>, <ARG2>, <RESULT1>, <RESULT2>] COMMAND

This is a conditional operation. For example, the transform expression: ["if","eq",["getglobalstring", "sample\_slot"], "R", 1.0, -1.0] will return 1.0 if the value in the global register is "R". Otherwise it returns -1.0.

Quantum Design

Available Operations are: "eq" (equals) - Tests for equality. "neq" (not equal) – Tests for inequality "gt" (greater than) – Tests if arg1 > arg2 "gte" (greater than or equal) – Tests if arg1 >= arg2"lt" (less than) – Tests if arg1 < arg2 "lte" (less than or equal) – Tests if arg1 <= arg2

Note that "eq" and "neq" are type aware and works with result parameters that are floats or strings.

["SEQUENCE", <PROC1>, <PROC2>, ..., <PROCN>] COMMAND

This function evaluates each of the argument objects from first to last but does not return any results.

["SETROWITEM", <REGISTER NAME>, <VALUE>, <FLOAT FORMAT>] COMMAND

Set an entry given by the string register\_name in the register to the value. The float\_format parameter is optional. If present, float\_format is the format specifier (see toString() C# documentation or equivalent).

["SETGLOBAL", <REGISTER\_NAME>, <VALUE>] COMMAND

This is nearly identical to setrowitem, except it sets the values in the global register.

["GETGLOBALFLOAT", <REGISTER\_NAME>] COMMAND

Get the current value of the named global and return it as a float.

["GETGLOBALSTRING", <REGISTER\_NAME>] COMMAND

Get the current value of the named global and return it as a string.

["INTERPOLATE", <VALUE1>, <FILE\_NAME>, ...] COMMAND

This returns the table interpolated value for value1 by using the table located at file\_name.

["INTERPOLATESLOPE", <VALUE1>, <FILE\_NAME>, ...] COMMAND

This is almost identical to interpolate except it returns .dYdXofX() instead of .YofX().

["GETROWITEMFLOAT", <ROW\_REGISTER\_NAME>] COMMAND

Get the current value of the named row register and return it as a float.

### A.4.3 Command Line

#### A.4.3.1 STARTING THE WIDGET

MVTransform.exe <options>

All options are traditional Unix style long-form parameters with double dashes and dashed names, like so: --my-param=myvalue.

A.4.3.1.1 PARAME	TERS
help	Gives the help text.
port=n	Specifies the optional startup port for communicating with this widget via the TCP socket.
config-file="full fil	ename" Specifies the location of .cfg file that is read at startup. By default, this file is located in the same folder with the executable and is called MVTransform.cfg.
input="filename"	Specifies the input file path and name. This is the input MultiVu data file. By default, this file is located in the same folder as the executable, but called MVTransform.in.dat.
output="filename"	Specifies the output file path and name. By default, this file is located in the same folder as the executable and called MVTransform.out.dat.
command-dump="	<b>'filename''</b> Save all commands (with a timestamp) received on the command port to a file along with any response strings. This file is NOT overwritten.
mode=continuous	<b>one-shot</b> Run in continuous or one-shot mode. Continuous runs, forever looking for new data in the input file. One-shot will terminate when the end of the input file occurs. The default is one-shot.

#### A.4.3.2 RUNNING THE WIDGET

Typing the single, lower case or capital, letter commands A (Abort), P (Pause), R (Resume), H (Refresh), S (GetStatus) are valid at the command prompt.

## A.5 Generic Filter Widget

### A.5.1 Overview

The Generic Filter Widget (GFW) performs smoothing and averaging operations like mean, midpoint, median, range, standard deviation and slope. It reads and writes a MultiVu data file. In the output file, each output row represents an operation on a range of input rows.

## A.5.2 Configuration File

The configuration file consists of a single JSON dictionary that defines all the transformations that will be applied to the input data file to produce the output file. Here is an example illustrating the structure:

```
'params": {"row buffer size": 1000, "output interval sec": 5.0,
      "time pad":30.0, "param section_passthrough":true,
      "init for passthrough":false, "max input gap":5.0 }
"outputs":
 ſ
   {"out":"System Temperature (K)", "in":"System Temperature (K)",
      "filter":"mean", "time range":5.0},
   {"out":"Sample Temperature (K)", "in":"Sample Temperature (K)",
      "filter":"mean" },
   {"out":"Sample Temperature Rate (K/sec)".
      "in":"Sample Temperature Rate (K/sec)",
     "filter":"mean", "time range":60.0},
   {"out":"Sample Temperature Range (K)", "in":"Sample Temperature (K)",
      "filter":"range" },
   {"out":"Temperature Status (code)", "in":"Temperature Status (code)",
      "filter": "midpoint" }
 ],
}
```

#### A.5.2.1 GLOBAL PARAMETERS

- **row\_buffer\_size:** This is the maximum number of rows of the input file that are held in memory and available for filter operations. This should be larger than is needed to support the global time\_pad setting or any of the time\_range settings for the outputs.
- **output\_interval\_sec:** This is the desired output interval for rows.
- **time\_pad:** This determines how much data (in seconds) both before and after the output setpoint (outTime) is required before an output row at outTime can be generated. If time\_pad=30.0, then at least 30 seconds' worth of input rows both before and after a given outTime is needed in order to generate an output point. Note that time\_pad\*2 should be greater or equal to the largest time\_range in the outputs section.
- **param\_section\_passthrough:** When set to true (the default), BEGIN:PARAMS/END:PARAMS blocks in the comment column are passed-through line-for-line with no averaging or concatenation. If false, these lines are treated like any others.

- **init\_for\_passthrough:** When set to true (default is false), the outputs configuration section is preconfigured to pass-through all the input columns with the mean filter applied (except the comments column, which uses the "stringconcat" filter).
- **max\_input\_gap:** This is the maximum input time interval in seconds allowed before the averaging is restarted. This controls how gaps (in time) are handled on the input.

#### A.5.2.2 THE OUTPUT SECTION DEFINITIONS

The outputs section normally has one entry per output column. Each entry identifies the type of filter to be used, the parameters for the filter and the name of the input column used for the source data.

The time and comments columns do not need to be specified explicitly if the [Header] section of the input file identifies these two special columns properly. For the time column, the default behavior is a "mean" filter with time\_range=output\_interval\_sec. For the comments column (with the exception of BEGIN:PARAMS/END:PARAMS blocks when param\_section\_passthrough=true), the default is a "stringconcat" filter also with time\_range=output\_interval\_sec.

If the init\_for\_passthrough parameter is set to true, then all the other columns are configured with a "mean" filter. Any entries entered into the "outputs" configuration that have a matching "out" parameter will override the default output definition. Similarly, if there are entries in the outputs section that have the same "out" parameter, the final entry will be used.

**Outputs Section Parameters:** 

- **out:** This is the full MultiVu column name that is created on the output file. This parameter is normally unique across all the output definitions; otherwise the later one will be used.
- in: This parameter identifies the input data column that is processed by the filter. If it is omitted, it is assumed to be the same as the "out" parameter. Since more than one output column may involve operations on the same input column, there is no requirement that the "in" parameter be unique. If this column name is not found on the input file, a null string will be written out.
- **filter:** This parameter defines the operation that is performed to create the output value. The input data for the operations is the set of values that is formed by including all the points from the "in" column having timestamps that are between outTime time\_range/2 and outTime + time\_range/2. The following operations are defined:
  - *mean:* returns the average of the column values.
  - o *median*: returns the value at the midpoint after sorting the column values in the range.
  - o *minimum*: for integer or float formatted strings, will return the minimum value.
  - o *maximum:* for integer or float formatted strings, will return the maximum value.
  - o *midpoint*: returns the value at or before the midpoint of the set of column values.
  - o *first*: returns the first value of the set of column values.
  - *last:* returns the last value of the set of column values.
  - *range:* returns the range of the set of column values.
  - o *stddev:* returns the standard deviation of the set of column values.
  - o *sum:* returns the sum of the set of column values.
  - o *sumofsquares*: returns the sum of the squares of the set of column values.
  - o *count:* returns the count of the set of column values.
  - *stringconcat:* outputs a quoted string corresponding to the concatenation, separated by the separator parameter, of all the values in the input.
  - o *rate:* returns the time rate of change (slope in time) of the column by fitting a line to the points.
- **time\_range:** Defines the extent of the set analyzed. By default, this is initialized to output\_interval\_sec.

- **separator:** For the "stringconcat" filter, this specifies the string used between the concatenated values in the output. The default is a comma.
- **timestamp\_name:** By default, the name of the timestamp column on input is derived from the "DATATYPE, TIME,<col#>" metadata in the [Header] section. Specify a different name here.
- **timestamp\_output\_name:** By default, the output timestamp name is the same as timestamp name but can be overridden to be something else here.
- **comment\_name:** By default, the name of the comment column on input is derived from the "DATATYPE, COMMENT,<col#>" metadata in the [Header] section. Override with something different here.
- **comment\_output\_name:** By default, the output comment name is the same as comment\_name but can be overridden to be something else here.

### A.5.3 Command Line

#### A.5.3.1 STARTING THE WIDGET

MVFilter.exe <options>

All options are traditional Unix style long-form parameters with double dashes and dashed names, like so: --my-param=myvalue.

#### A.5.3.1.1 PARAMETERS

help	Gives the help text.
port=n	Specifies the optional startup port for communicating with this widget via the TCP socket.
config-file="full file	ename" Specifies the location of .cfg file that is read at startup. By default, this file is located in the same folder with the executable and is called MVFilter.cfg.
input="filename"	Specifies the input file path and name. This is the input MultiVu data file. By default, this file is located in the same folder as the executable but called MVFilter.in.dat.
output="filename"	Specifies the output file path and name. By default, this file is located in the same folder as the executable and called MVFilter.out.dat.
command-dump="	<b>filename</b> " Save all commands (with a timestamp) received on the command port to a file along with any response strings. This file is NOT overwritten.
mode=continuous a	<b>one-shot</b> Run in continuous or one-shot mode. Continuous runs forever, looking for new data in the input file. One-shot will terminate when the end of the input file occurs. The default is one-shot.

#### A.5.3.2 RUNNING THE WIDGET

Typing the single, lower case or capital, letter commands A (Abort), P (Pause), R (Resume), H (Refresh), S (GetStatus) are valid at the command prompt.

## A.6 General Merge Widget

### A.6.1 Overview

The Generic Merge Widget (GMW) is able to read two or more MultiVu data files and collates them into a single output file.

## A.6.2 Configuration File

The configuration file consists of a single JSON dictionary that contains the mapping to output columns from the input columns. Here is an example illustrating the structure:

#### A.6.2.1 GLOBAL PARAMETERS

- **init\_for\_passthrough:** When set to true (default is true), the outputs configuration section is preconfigured to pass through all the input columns from the two input files and apply prefixes to output columns as defined in the parameters prefix1 and prefix2.
- **prefix1:** When init\_for\_passthrough is set to true, this is the prefix (default is "A.") that is applied to the input column names on input1 to give the corresponding output column name.
- **prefix2:** When init\_for\_passthrough is set to true, this is the prefix (default is "B.") that is applied to the input column names on input2 to give the corresponding output column name.
- **timestamp1\_name:** By default, the name of the timestamp column on input1 is derived from the "DATATYPE, TIME,<col#>" metadata in the [Header] section of input1. Specify a different name here.
- **timestamp2\_name:** By default the name of the timestamp column on input2 is derived from the "DATATYPE, TIME,<col#>" metadata in the [Header] section of input2. Specify a different name here.
- **timestamp\_output\_name:** By default, the output timestamp name is the same as timestamp1\_name but can be overridden to be something else here.
- **comment1\_name:** By default, the name of the comment column on input1 is derived from the "DATATYPE, COMMENT,<col#>" metadata in the [Header] section of input1. Override with something different here.

- **comment2\_name:** By default, the name of the comment column on input2 is derived from the "DATATYPE, COMMENT,<col#>" metadata in the [Header] section of input2. Override with something different here.
- **comment\_output\_name:** By default, the output comment name is the same as comment1\_name (or comment2\_name if no comment on input1) but can be overridden to be something else here.
- **output\_title:** By default, the TITLE defined in the [Header] section of the output file is the concatenation of the TITLEs from the two inputs. You can override it here.
- **maxbuffertime:** An input processing tuning parameter. The default is 60 seconds. This is the maximum delay time that can be experienced between the two input files without causing a regen/refresh event (deleting output and reprocessing the inputs to give the output).

### A.6.3 Command Line

#### A.6.3.1 STARTING THE WIDGET

MVMerge.exe <options>

All options are traditional Unix style long-form parameters with double dashes and dashed names, like so: --my-param=myvalue.

#### A.6.3.1.1 PARAMETERS

help	Gives the help text.
port=n	Specifies the optional startup port for communicating with this widget via the TCP socket.
config-file="full fil	<b>ename</b> " Specifies the location of .cfg file that is read at startup. By default, this file is located in the same folder with the executable and is called MVMerge.cfg.
input1="filename1	" <b>and -input2="filename2</b> " Specifies the input file path2 and name2. These are the input MultiVu data files. By default, these files are located in the same folder as the executable but called MVMerge.in1.dat and MVMerge.in2.dat.
output="filename"	Specifies the output file path and name. By default, this file is located in the same folder as the executable and called MVMerge.out.dat.
command-dump="	<b>filename</b> " Save all commands (with a timestamp) received on the command port to a file along with any response strings. This file is NOT overwritten.
mode=continuous 6	<b>one-shot</b> Run in continuous or one-shot mode. Continuous runs forever looking for new data in the input file. One-shot will terminate when the end of the input file occurs. The default is one-shot.

#### A.6.3.2 RUNNING THE WIDGET

Typing the single, lower case or capital, letter commands A (Abort), P (Pause), R (Resume), H (Refresh), S (GetStatus) valid at the command prompt.

- Ashcroft, N. W., Mermin, N. D., 1976. Solid State Physics. *Thomson Learning, Inc.* The United States. Cornell University. Chapters 23 and 25.
- Kroeger, F.R. Swenson, C. A. 1977. Absolute linear thermal expansion measurements on copper and aluminum. *Journal of Applied Physics* 48: 853-864.
- Okaji, M. et al 1995. Laser interferometric dilatometer at low temperatures. Cryogenics 35: 887-891.
- Quantum Design. 2000. Physical Property Measurement System: PPMS MultiVu Application User's Manual.
- ———. 2011. Physical Property Measurement System: Hardware Manual.
- White, G.K. Ho, C. Y and R. E Taylor. 1999. Thermal Expansion of Solids: Thermal Expansion of Reference Materials. Materials Park, OH: *ASM International*. 269-285. Print.