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# Q-pod<sup>TM</sup>

## Quartz Crystal Monitor

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IPN 074-547-P1B



O P E R A T I N G M A N U A L

# Q-pod<sup>TM</sup>

## Quartz Crystal Monitor

IPN 074-547-P1B



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**DECLARATION  
OF  
CONFORMITY**

This is to certify that this equipment, designed and manufactured by:

**INFICON Inc.  
Two Technology Place  
East Syracuse, NY 13057  
USA**

meets the essential safety requirements of the European Union and is placed on the market accordingly. It has been constructed in accordance with good engineering practice in safety matters in force in the Community and does not endanger the safety of persons, domestic animals or property when properly installed and maintained and used in applications for which it was made.

**Equipment Description:** Q-Pod Quartz Monitor

**Applicable Directives:** 2006/95/EC (LVD)  
2004/108/EC (General EMC)  
2002/95/EC (RoHS)

**Applicable Standards:**

Safety: EN 61010-1:2001

Emissions: EN 61326-1:1997/A1: 1998/A2: 2001 (Radiated & Conducted Emissions)  
Class A: Emissions per Table 3  
(EMC – Measurement, Control & Laboratory Equipment)

Immunity: EN 61326-1:1997/A1: 1998/A2: 2001 (General EMC)  
Class A: Immunity per Table A1  
(EMC – Measurement, Control & Laboratory Equipment)

RoHS: Fully compliant

**CE Implementation Date:** April 2007 (Updated February 2011)

**Authorized Representative:** Steve Schill

A handwritten signature in black ink that reads "Steve Schill".

---

Thin Film Business Line Manager  
INFICON Inc.



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NOTE: These instructions do not provide for every contingency that may arise in connection with the installation, operation or maintenance of this equipment. Should you require further assistance, please contact INFICON.



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# Table Of Contents

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## Chapter 1

### Introduction

1.1	Introduction . . . . .	1-1
1.1.1	Related Manuals . . . . .	1-2
1.2	Instrument Safety . . . . .	1-2
1.2.1	Definition of Notes, Cautions and Warnings . . . . .	1-2
1.2.2	General Cautions and Warnings . . . . .	1-3
1.3	How to Contact INFICON . . . . .	1-4
1.3.1	Returning Your Q-pod . . . . .	1-4
1.4	Specifications . . . . .	1-4
1.5	Unpacking and Inspection . . . . .	1-5
1.6	Parts and Options Overview . . . . .	1-5
1.6.1	Base Configuration . . . . .	1-5
1.6.2	Sensors . . . . .	1-5

## Chapter 2

### Quick Start

2.1	Install Software . . . . .	2-1
2.2	Connect the Q-pod . . . . .	2-1
2.3	Using the Q-pod Software . . . . .	2-2
2.3.1	Main Display . . . . .	2-2
2.3.1.1	Reading Grid . . . . .	2-2
2.3.1.2	Materials Grid . . . . .	2-3
2.3.2	Setup Display . . . . .	2-3
2.3.2.1	DataLog . . . . .	2-3
2.3.2.2	Measurement . . . . .	2-3
2.3.2.3	Display . . . . .	2-3
2.3.2.4	Q-pods . . . . .	2-3
2.3.3	Graph Display . . . . .	2-4

## Chapter 3

### Communications

3.1	Introduction . . . . .	3-1
3.2	ASCII Command Set . . . . .	3-1
3.3	QPOD.DLL . . . . .	3-2
3.3.1	Registering QPOD.DLL . . . . .	3-2
3.3.2	Functions . . . . .	3-3
3.3.3	Events . . . . .	3-4
3.4	Calculating Rate and Thickness . . . . .	3-4
3.5	Sample Programs . . . . .	3-4

## Chapter 4

### Troubleshooting and Maintenance

4.1	Troubleshooting Guide . . . . .	4-1
4.1.1	Status LED . . . . .	4-1
4.1.1.1	LED illuminated steady . . . . .	4-1
4.1.1.2	LED flashes slowly (about 2 Hz) . . . . .	4-1
4.1.1.3	LED flashes rapidly (about 10 Hz) . . . . .	4-1
4.1.2	Troubleshooting the Q-pod . . . . .	4-2
4.1.3	Troubleshooting Sensors . . . . .	4-3
4.1.4	Troubleshooting Computer Communications . . . . .	4-8
4.2	Replacing the Crystal . . . . .	4-8
4.2.1	Front Load . . . . .	4-9
4.2.2	Cool Drawer . . . . .	4-10
4.2.3	Bakeable Sensor . . . . .	4-12
4.2.4	Sputtering Sensor . . . . .	4-13
4.2.5	Crystal Snatcher . . . . .	4-14
4.3	Crystal Sensor Emulator IPN 760-601-G2 . . . . .	4-15
4.3.1	Diagnostic Procedures . . . . .	4-16
4.3.1.1	Measurement System Diagnostic Procedure . . . . .	4-16
4.3.1.2	Feed-Through Or In-Vacuum Cable Diagnostic Procedure . . . . .	4-17
4.3.1.3	Sensor Head Or Monitor Crystal Diagnostic Procedure . . . . .	4-18
4.3.1.4	System Diagnostics Pass But Crystal Fail Message Remains . . . . .	4-19
4.3.2	Sensor Cover Connection . . . . .	4-20
4.3.2.1	Compatible Sensor Heads . . . . .	4-20
4.3.2.2	Incompatible Sensor Heads . . . . .	4-20
4.3.3	Emulator Specifications . . . . .	4-21

## Chapter 5

### Calibration Procedures

5.1	Importance of Density, Tooling and Z-Ratio . . . . .	5-1
5.2	Determining Density . . . . .	5-1
5.3	Determining Tooling . . . . .	5-2
5.4	Laboratory Determination of Z-Ratio . . . . .	5-2

## Chapter 6

### Measurement and Theory

6.1	Basics . . . . .	6-1
6.1.1	Monitor Crystals . . . . .	6-2
6.1.2	Period Measurement Technique . . . . .	6-4
6.1.3	Z-match Technique . . . . .	6-5
6.1.4	Active Oscillator . . . . .	6-6

## Appendix A

### Material Table

A.1	Introduction . . . . .	A-1
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# Chapter 1

## Introduction

### 1.1 Introduction

The INFICON Q-pod™ Transducer is a small, inexpensive and accurate way to measure thin film deposition rate and thickness. No external oscillator or power source is required.

The included Q-pod software displays rate, thickness, frequency, crystal life and a graph of rate versus time, for up to 8 Q-pods simultaneously. Q-pod readings can be logged to disk in spreadsheet friendly comma-delimited format.

There are also sample files included, written in Labview, Java and Visual Basic to help aid in the integration of the Q-pod into a system that is managed via software. See [section 3.5, Sample Programs, on page 3-4](#) for more details.

Figure 1-1 Q-pod



When reading this Q-pod manual, please pay particular attention to the NOTES, CAUTIONS, and WARNINGS found throughout the text. The Notes, Cautions, and Warnings are defined in [section 1.2.1 on page 1-2](#). Also, note the general warnings in [section 1.2.2, General Cautions and Warnings, on page 1-3](#).

## 1.1.1 Related Manuals

Sensors are covered in separate manuals. PDF files of these manuals are contained in the 074-5000-G1 CD, part of the Ship Kit.

- ♦ 074-154 - Bakeable Sensor
- ♦ 074-156 - Front Load Sensor, Single/Dual
- ♦ 074-157 - Sputtering Sensor
- ♦ 147-800 - Cool Drawer Sensor, Single/Dual

## 1.2 Instrument Safety

### 1.2.1 Definition of Notes, Cautions and Warnings

When using this manual, please pay attention to the NOTES, CAUTIONS and WARNINGS found throughout. For the purposes of this manual they are defined as follows:

**NOTE:** Pertinent information that is useful in achieving maximum instrument efficiency when followed.



#### CAUTION

Failure to heed these messages could result in damage to the instrument.



#### WARNING

Failure to heed these messages could result in personal injury.



#### WARNING - Risk Of Electric Shock

Dangerous voltages are present which could result in personal injury.

## 1.2.2 General Cautions and Warnings



### **CAUTION**

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Do not open the instrument case! There are no user-serviceable components within the instrument case.

Refer all maintenance to technically qualified personnel.

---



### **WARNING**

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Failure to operate the Q-pod in the manner intended by INFICON can circumvent the safety protection provided by the instrument and may result in personal injury.

---



### **CAUTION**

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Q-pod may not be suitable for use with RF sputtering systems or other electrically noisy environments.

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## 1.3 How to Contact INFICON

Worldwide customer support information is available under Support at [www.inficon.com](http://www.inficon.com) where you can contact:

- ♦ A Technical Support Engineer with questions regarding applications for and programming the Q-pod.
- ♦ A Service Engineer with questions regarding troubleshooting, diagnosing or repairing a defective Q-pod.
- ♦ Sales and Customer Service, to find the INFICON Sales office nearest to you.
- ♦ Repair Service, to find the INFICON Service Center nearest to you.

If you are experiencing a problem with your Q-pod, please have the following information readily available:

- ♦ The serial number and firmware version for your Q-pod.
- ♦ A description of your problem.
- ♦ An explanation of any corrective action that you may have already attempted.
- ♦ The exact wording of any error messages that you may have received.

### 1.3.1 Returning Your Q-pod

Do not return any component of your Q-pod to INFICON without first speaking with a Customer Support Representative. You must obtain a Return Material Authorization (RMA) number from the Customer Support Representative.

If you deliver a package to INFICON without an RMA number, your package will be held and you will be contacted. This will result in delays in servicing your Q-pod.

## 1.4 Specifications

Frequency Range . . . . .	1 to 6 MHz
Frequency Resolution . . . . .	0.05 Hz at 6 MHz @ 2 measurements per second)
Frequency Accuracy . . . . .	0.002%
Frequency Stability . . . . .	±2 ppm total, over 0° to 50°C
Thickness & Rate Resolution . . . . .	0.0613 Å (new crystal); 0.1091 Å (crystal @ 4.5 MHz); over 500 ms sample for material density = 1.0, Z-ratio = 1.0
Input . . . . .	BNC
Interface & Power . . . . .	USB, v2.0 or later, 15 ft. (457 cm) maximum allowable length
Size . . . . .	1 in. x 2 in. x 2.5 in. (25 mmx 50 mm x 64 mm)



Weight . . . . .	2 oz. (32 gm)
Computer Requirements . . . . .	Any PC running Windows® XP / 2000 / Windows 7 32-bit with one available USB port for each Q-pod

**1.5 Unpacking and Inspection**

- 1 If the Q-pod has not been removed from its packaging, do so now.
- 2 Carefully examine the card for damage that may have occurred during shipping. This is especially important if you notice obvious rough handling on the outside of the container. *Immediately report any damage to the carrier and to INFICON.*
- 3 Do not discard the packing materials until you have taken inventory and have at least performed successful installation.
- 4 Take an inventory of your order by referring to your order invoice and the information contained in [section 1.6.1](#).
- 5 To install the Q-pod, see [Chapter 2, Quick Start](#).
- 6 For additional information or technical assistance, contact INFICON, refer to [section 1.3 on page 1-4](#).

**1.6 Parts and Options Overview**

**1.6.1 Base Configuration**

Q-pod Transducer w/Software . . . . .	Q-POD
10 ft. USB Cable. . . . .	.068-0472
6 in. BNC Cable . . . . .	.755-257-G6
Technical Manual . . . . .	.074-547 on 074-5000-G1 CD
5.5 MHz Test Crystal . . . . .	.782-902-023

**1.6.2 Sensors**

Front Load Single Sensor. . . . .	SL-XXXXX
Cool Drawer Single Sensor . . . . .	CDS-XXFXX
Sputtering Sensor. . . . .	.750-618-G1
Front Load UHV Bakeable Sensor . . . . .	BK-AXF

**NOTE:** Multi-crystal (rotary) sensors & dual sensors should not be used with the Q-pod.

IPN 074-547-P1B

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## Chapter 2 Quick Start

### 2.1 Install Software

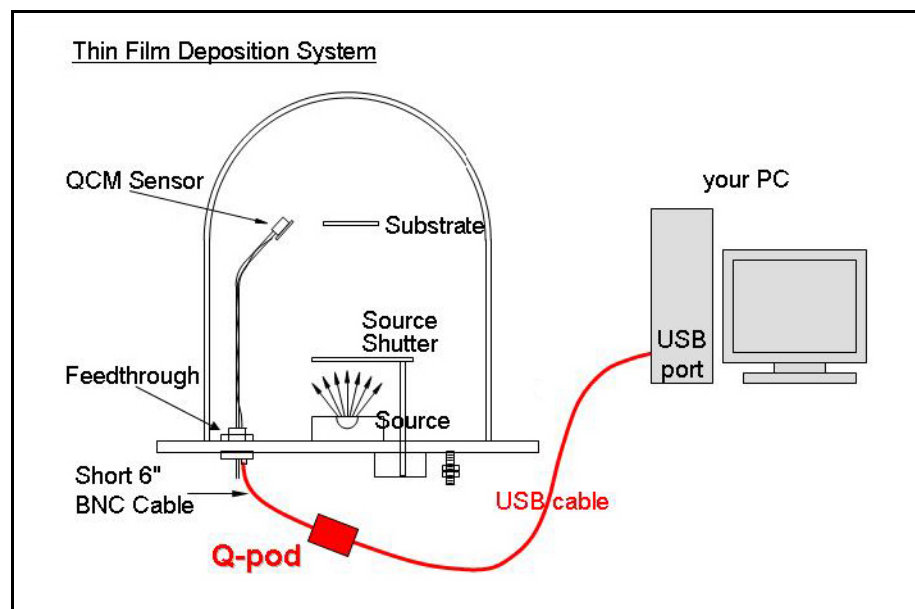
**NOTE:** If this is the initial Q-pod installation on this computer, you should install the Q-pod Software before connecting a Q-pod to the USB port.

- 1 Place the INFICON Program Disk in the computer's CD-ROM.
- 2 Ignore the prompt and double-click the **My Computer** icon.
- 3 Right-click the CD-ROM icon, then click **Open**.
- 4 Double-click the **Q-pod Transducer** file.
- 5 Double click **QPOD\_V#\_SETUP.EXE**.
- 6 Follow the on screen directions and select the default installation options.
- 7 When software installation is complete, exit the installation program.

### 2.2 Connect the Q-pod

- 1 Connect the USB cable to a computer USB port, then to the Q-pod. See [Figure 2-1](#).
- 1a If you are prompted to install the Q-pod drivers, select **Install the Software Automatically**.
- 1b If you are prompted concerning Windows Logo Testing, select **Continue Anyway**.

Figure 2-1 Q-pod Connections

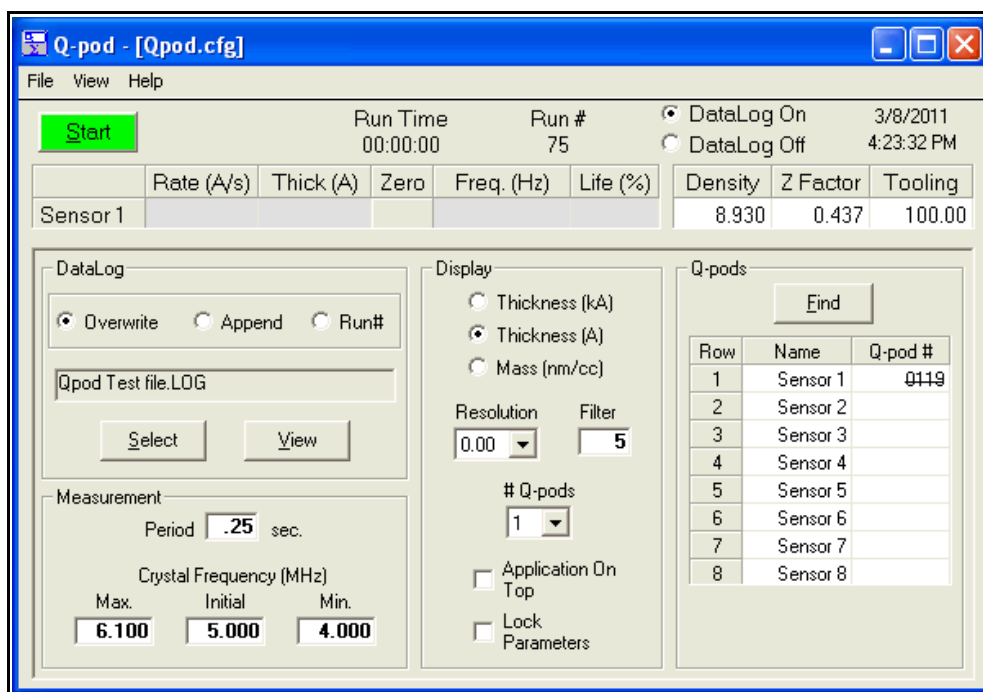


- 2 Use the 6 in. BNC cable provided to connect the Q-pod to the QCM sensor feedthrough.
- 3 The status light on the Q-pod illuminates when a quartz crystal is detected.

## 2.3 Using the Q-pod Software

Click the Q-pod desktop icon, or click **Start >> Programs >> Sigma Instruments >> Q-pod**. If you are prompted to assign Q-pod(s) to a channel, click **Yes**. Click **Help** to get more detailed information on materials and operation. Select **Setup** or **Graph** on the Main Screen menu to change the main screen display mode. See Figure 2-2.

Figure 2-2 Main Screen



### 2.3.1 Main Display

The top of the main screen is the main display. Here the Start button is used to start and record a run. Run time and run number are shown, along with the current time and date. The option to datalog during any given run is also given.

#### 2.3.1.1 Reading Grid

The left grid displays the measured values of each connected Q-pod. Click Start to display Rate and Thickness readings. Frequency and Life readings are always displayed. Click a channel's Zero button to zero the channel thickness reading. Shift-Click any channel Zero button to zero all thicknesses.

### **2.3.1.2 Materials Grid**

In the right grid, edit the Density, Z-Factor and Tooling values for the materials being deposited. Move your mouse over each setting to see a brief description.

## **2.3.2 Setup Display**

The lower section of the main screen is where run parameters can be changed and Q-pod communication information is presented.

### **2.3.2.1 DataLog**

Sets the file name, location and operation of disk data logging. Data log files can be set to overwrite the file name each time it is saved. It can append the data, by adding the newly recorded data to the end of the same file. It can also save each run in a new file designated by run number. Data is saved in comma delimited format for easy import into a spreadsheet.

### **2.3.2.2 Measurement**

Sets the basic measurement parameters for all Q-pods. The values shown above are correct for most applications.

### **2.3.2.3 Display**

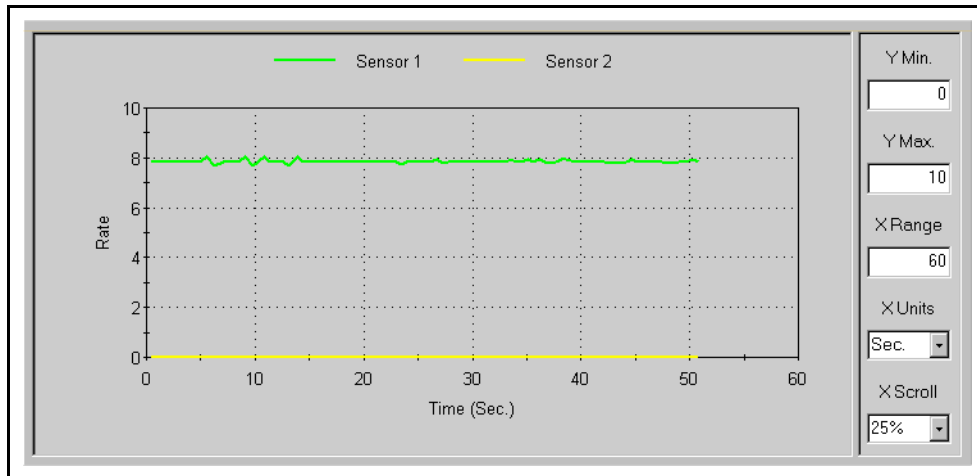
Sets the display parameters. Thickness can be displayed in  $\text{k}\text{\AA}$  or  $\text{\AA}$ . Mass can be displayed in  $\text{ug}/\text{cm}^2$ . Rate will be changed to  $\text{ng}/\text{cm}^2/\text{sec}$  if mass is the chosen option. Resolution of displayed readings may also be changed here (0.0 or 0.00). The rate reading filter can be set in this section. The Filter number represents how many measurements are averaged together to produce the displayed rate. The number of Q-pods being used is defined in this section as well.

### **2.3.2.4 Q-pods**

Sets the name and displays the serial number of the Q-pod assigned to each measurement channel. A strike-through serial number indicates the Q-pod is not connected. The software can monitor a maximum of 8 Q-pods simultaneously.

### 2.3.3 Graph Display

Figure 2-3 Graph Display



Graphs the deposition rate, thickness, or frequency of active measurement channels when Start is clicked. The graph viewed can be changed from the view option on the toolbar on the main screen. Settings to the right of the graph control basic graph functions. See the **Help** menu for advanced graph setup.

## Chapter 3

# Communications

### 3.1 Introduction

The Q-pod communicates via a USB (or USB2) port and a simple ASCII command set. An ActiveX DLL (Qpod.DLL) provides functions to set parameters, take readings, and handle USB communications.

### 3.2 ASCII Command Set

The commands below assume you can open the proper USB port for communications. The section on Qpod.DLL provides the tools needed to communicate with the Q-pod without knowledge of USB functions.

A Q-pod command consists of an exclamation character (&H21), then the command as detailed below, and terminated with a Carriage Return (&H13) and Line Feed (&H10). The response has the same format, that is !<response>CrLf.

#### @ Get version

Returns: Qpod VX.XX

#### A# Get Frequency and Set Status LED

Returns frequency counts. The B & C commands (see below) must be used to set the measurement period before using this command. The response is gate counts, which are converted crystal frequency using:

$$\text{Freq} = 200 \text{ MHz} * \text{GatePeriod} / \text{Response}$$

A response of -1 indicates that a new reading is not available.

The value sent with this command activates the Status LED. Send A1 to turn on the LED (crystal good), or A0 to cause the LED to flash at the measurement rate (crystal fail).

#### B# Set Gate Period

Sets the longest measurement period, determined by the lowest valid crystal frequency. If the desired measurement period is 0.25 seconds, and the minimum crystal frequency is 5.0MHz, then the command is: B1250000 (0.25 x 5MHz).

#### C# Set Measurement Period

The value sent is the period in seconds times the reference oscillator frequency, 50MHz. If the desired measurement period is 0.25 seconds, then the command is: C12500000 (0.25 x 50MHz)

### 3.3 QPOD.DLL

QPOD.DLL is an Active-X DLL that provides the functions necessary to get rate and thickness readings from up to 32 Q-pods.

In the function listings that follow, parameter and return value types are indicated by the first letter of the variable name:

i: Integer (16 bits)	f: Float (single)	c: Char (8 bits)	s: String
l: Long (32 bits)	d: Double	b: Byte (8 bits)	

Functions parameters are passed By Value unless specifically designated as ByReference (pointer).

Unless noted otherwise, the return values of functions are:

QPOD\_SUCCESS = &H0 . . . . . Command successful

QPOD\_BUSY = &H1 . . . . . Communications already in progress

QPOD\_TIMEOUT = &H2 . . . . . Did not respond

QPOD\_NOREADING = &H3 . . . . . No new reading ready

QPOD\_OVERFLOW = &H4 . . . . . Internal overflow error

QPOD\_BADPARAM = &H5 . . . . . Bad parameter value

QPOD\_BADCMD = &H6 . . . . . Bad command

QPOD\_BADSERNUM = &H7 . . . . . Serial number not connected

QPOD\_XTALFAIL = &H8 . . . . . Crystal fail

QPOD\_COMMERR = &H9 . . . . . Unknown communications error

QPOD\_UNKERR = &H99 . . . . . Unknown program error

#### 3.3.1 Registering QPOD.DLL

QPOD.DLL and ccrpTmr.DLL (used for timing functions) must be registered by Windows. The QpodDLL setup program installs and registers both files. To install and register them manually, copy both files to the **Windows\System32** folder. Click **Start**, **Run**, and type

```
regsvr32\windows\system32\qpod.dll
```

click **OK**, then type

```
regsvr32\windows\system32\ccrpTmr.dll
```

and click **OK**.



### 3.3.2 Functions

#### **FindQpods** (By Ref sSNArray(0 to 32)) [*!=IRtnVal*]

Searches for Q-pods connected to the computer. Populates sSNArray() with the serial numbers of up to 32 Q-pods. Return value is total number of Q-pods found.

Most QPOD.DLL functions require the serial number of a Q-pod as their first parameter. FindQpods should be the first function called. Additional calls to FindQpods can be made, but be sure to resend the parameters (period, density, etc.) for each Q-pod serial number you will use.

#### **InitFreq** (dFmax, dFmin, dFinit) [*!=IRtnVal*]

Initializes the maximum, minimum, and initial crystal frequency values for all attached Q-pods. The default values are Fmax=6.1 MHz, Fmin=5.0 MHz, Finit=6.0 MHz,. Crystal readings >Fmax or <Fmin will generate a crystal fail response to the GetReadings call. Call InitFreq immediately after FindQpods.

#### **GetReading** (sSerNum, By Ref dRdgArray(0 to 3)) [*!=IRtnVal*]

Populates dRdgArray with the Rate, Thickness, Frequency, and %Life for the Q- pod serial number. Thickness is returned in kA.

You can call get readings and test the return value to see if a new readings was ready (&H0) or not (&H3). Alternatively, you can set the Reading Timer and QPOD.DLL will raise a QpodReading event when a reading is ready. See SetReading Timer() and QpodReading() below.

#### **Material** (sSerNum, dDensity, dZFactor, dTooling) [*!=IRtnVal*]

Sets the Density, Z Factor, and Tooling used to convert frequency change into rate and thickness.

#### **Period** (sSerNum, dPeriod) [*!=IRtnVal*]

Sets the measurement period in seconds. Valid values are 0.1 to 2.

#### **SetReadingsTimer** (dInterval) [*!=IRtnVal*]

Sets the interval for polling Qpods for readings in milliseconds. A value of zero disables the QpodReadings event (see below).

#### **Zero** (sSerNum) [*!=IRtnVal*]

Sets the Q-pod thickness reading to zero.

#### **ZeroAll** () [*!=IRtnVal*]

Sets all attached Q-pod thickness readings to zero.

#### **Revision** (sSerNum, ByRef sRev) [*!=IRtnVal*]

Returns the firmware revision in sRev. A sRev value is: Qpod V1.00.

### 3.3.3 Events

**QpodReading** (sSerNum, ByRef dRdgArray(0 to 3), Time)

If SetReadingsTimer (see above) is passed a non-zero interval, the QPOD.DLL will poll every attached Q-pod for readings at the set interval.

For each Q-pod that has a new reading, a QpodReading event is raised. The sSerNum parameter is the serial number of the Q-pod with a new reading. The array dRdgArray holds Rate, Thickness, Frequency, and %Life for that Q-pod serial number. Thickness is returned in kA.

## 3.4 Calculating Rate and Thickness

In the Q-pod software, thickness and rate are calculated using the Z-match equation. The calculation can be divided into four parts:

#### 1 Calculate Thickness:

$$\text{Thickness} = (\text{AT\_CONST} * \text{DENS\_QUARTZ}) / (\text{PI} * \text{Density} * \text{Freq} * \text{ZRatio})$$

where AT\_CONST =  $16.68 \times 10^{12}$ , DENS\_QUARTZ = 2.648, PI = 3.1416, Freq is the frequency calculated from the A command.

#### 2 Correct for Z-Ratio:

$$\text{Thickness}_z = \text{Thickness} * \text{ArcTan}(\text{ZRatio} * \text{Tan}(\text{PI} * (\text{FreqInit} - \text{Freq}) / \text{FreqInit}))$$

where Thickness was calculated in step 1 and FreqInit is the initial (new) crystal frequency – typically 6MHz.

#### 3 Correct for Tooling:

$$\text{Thickness}_T = \text{Thickness}_z * \text{Tooling} * 0.01$$

#### 4 Calculate Rate:

$$\text{Rate} = (\text{Thickness}_T - \text{Thickness}_{T-1}) / \text{Period}$$

where Thickness<sub>T</sub> was calculated in step 3, Thickness<sub>T-1</sub> was from the last measurement, and Period is the elapsed time between measurements.

## 3.5 Sample Programs

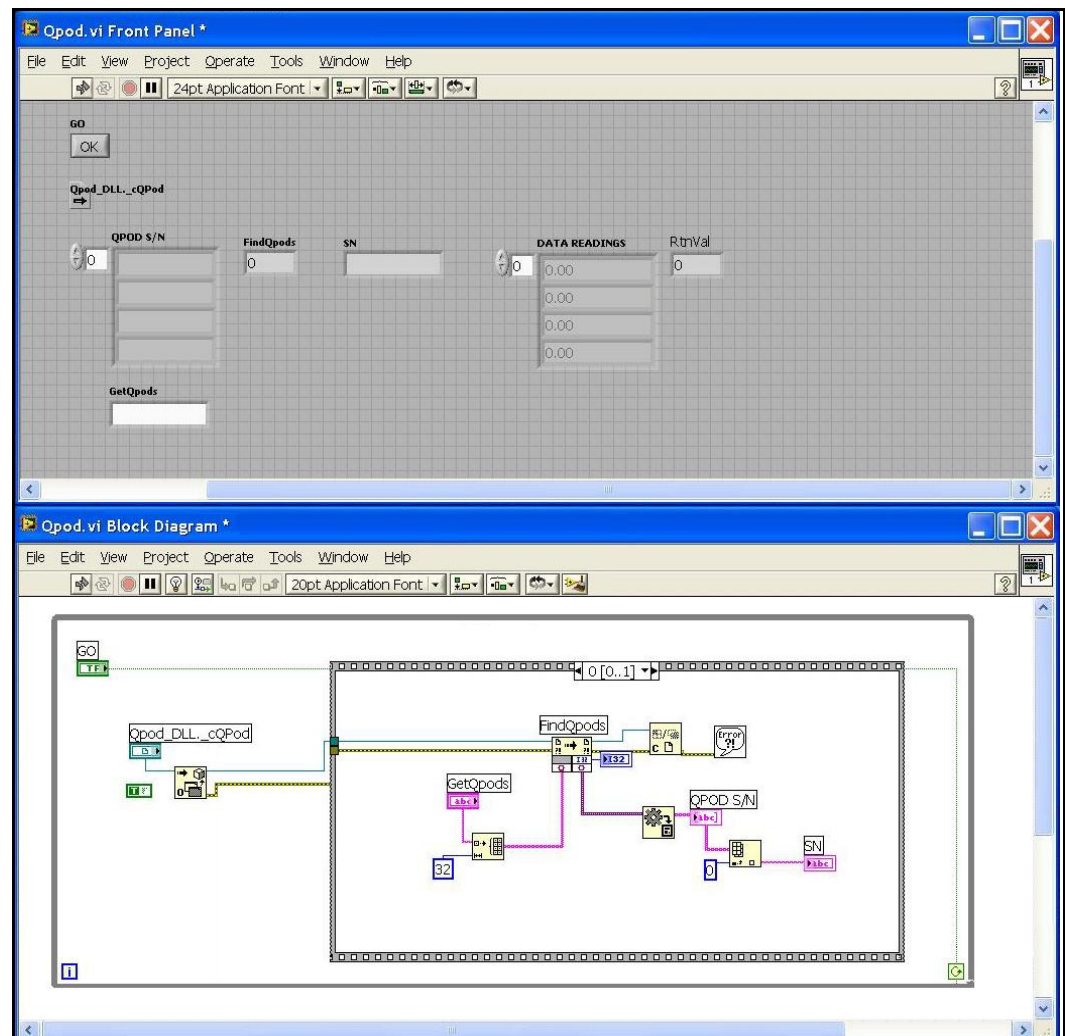
On the INFICON CDROM in D:\Qpod Transducer\_V100\_SAMPLES.exe, run this executable to set up the sample files for the Q-pod. When complete, they will be located here: C:\Program Files\Sigma Instruments\Q-pod\Sample Programs. These are sample interface files for use in Visual Basic and LabVIEW. These files are meant to act as means to aid the user in creating their own software and to allow the user to interface to the Q-pod with other devices in your operation.

**NOTE:** For the sample programs to work correctly, you should also install Sigma's Q-pod application.

Before using the LabVIEW demo, it may be best to familiarize oneself with operation of the Q-pod software. To run the LabVIEW demo(qpod.vi), click "Load DLL", "Use Card" and then set the Sensor parameters as desired. Click "Start" to display readings. Be sure to click "Unload DLL" before stopping the LabVIEW program. Otherwise, a Windows error may occur, and LabVIEW may shut down. LabVIEW 6 or higher is required.

**NOTE:** The sample LabView VI may require that you select the proper DLL for the two Automation RefNum controls. Right click each control, then click Select ActiveX Class. Check Show Creatable Objects Only, then select Sigma Instruments Qpod ActiveX DLL.

Figure 3-1 Labview Sample Program



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## Chapter 4

# Troubleshooting and Maintenance

### 4.1 Troubleshooting Guide

If the Q-pod fails to function, or appears to have diminished performance, the following Symptom/Cause/Remedy charts may be helpful.



#### CAUTION

**There are no user serviceable components within the Q-pod case.**

**Refer all maintenance to qualified personnel.**

#### 4.1.1 Status LED

If there is a problem with the Q-pod, the status LED will indicate as to the likely cause.

##### 4.1.1.1 LED illuminated steady

- ♦ Good crystal detected

##### 4.1.1.2 LED flashes slowly (about 2 Hz)

- ♦ No crystal connected
- ♦ Crystal frequency range not set correctly in software
- ♦ Crystal failure

##### 4.1.1.3 LED flashes rapidly (about 10 Hz)

- ♦ Software not installed
- ♦ Driver not installed
- ♦ Q-pod not detected by software
- ♦ Q-pod not assigned to a sensor channel (click Find to locate Q-Pod and then follow instructions to assign Q-Pod to a sensor channel)
- ♦ Q-pod hardware failure

## 4.1.2 Troubleshooting the Q-pod

Table 4-1 General Troubleshooting

SYMPTOM	CAUSE	REMEDY
1. Crystal fail message is always on.	a. Q-pod and/or sensor not connected.	a. Verify proper Q-pod/sensor connections.
	b. Q-pod malfunctioning.	b. If available, insert a known working Q-pod, or other monitor, in place of suspect one; if Q-Pod is confirmed bad, contact INFICON service department.
	c. Defective cable from feedthrough to Q-pod.	c. Use an Ohm meter or DMM to check electrical continuity or isolation as appropriate.
	d. Poor electrical contact in the sensor, feedthroughs, or in-vacuum cable.	d. Use an Ohm meter or DMM to check electrical continuity or isolation as appropriate.
	e. Failed crystal/no crystal.	e. Replace crystal with new INFICON crystal.
	f. Two crystals placed into the crystal holder.	f. Remove one of the crystals.
	g. Frequency of crystal out of range,	g. Verify that the crystal frequency is within the required range, use INFICON crystals. Set the desired range on the main screen on the software.

Table 4-1 General Troubleshooting

SYMPTOM	CAUSE	REMEDY
2. Noisy signal	a. Distance from sensor to Q-pod is longer than 40 in. (101.6 cm).	a. Replace cables to preserve the maximum allowable length.
	b. Mechanical/electric noise sources located near the Q-pod/cable .	b. Look for potential noise sources. Reposition the Q-pod/cables accordingly.
3. Frequency reading is unstable or drifting	a. Temperature (of the crystal) is changing. An AT-cut crystal frequency may drift as much as 10 Hz/°C.	a. Control the temperature of the chamber. Check watering cooling for flow and temperature. Check source to crystal distance (12 in. is ideal).
	b. Humidity (level on the crystal) is changing. Moisture being absorbed or exuded from the crystal surface,	b. Control the humidity of the chamber.

### 4.1.3 Troubleshooting Sensors

**NOTE:** Many sensor head problems may be diagnosed with a DMM (Digital Multi-Meter). Disconnect the short oscillator cable from the feedthrough and measure the resistance from the center pin to ground. If the reading is less than 10 megohms, the source of the leakage should be found and corrected. Likewise, with the vacuum system open check for center conductor continuity, a reading of more than 1 ohm from the feedthrough to the Q-pod indicates a problem. Cleaning contacts or replacing the in-vacuum cable may be required.

A somewhat more thorough diagnosis may be performed with the optional Crystal Sensor Emulator, 760-601-G1. See [section 4.3 on page 4-15](#) for a discussion of its use and diagnostic capabilities. A more detailed troubleshooting guide is shipped with the sensor. Refer to that manual for more detailed information in some cases.

Table 4-2 Troubleshooting Sensors

SYMPTOM	CAUSE	REMEDY
1. Large jumps of thickness reading during deposition.	a. Mode hopping.	a. Mode hopping is a byproduct of active oscillation with a heavily damped crystal. Temperature stabilization is key in diminishing this. Replace the crystal.
	b. Stress causes film to peel from crystal surface.	b. Replace crystal or use high performance RunSaver™ crystal; consult factory.
	c. Particulate or "spatter" from molten source striking crystal.	c. Thermally condition the source thoroughly before deposition, use a shutter to protect the crystal during source conditioning.
	d. Material build up, scratches or foreign particles on the crystal holder seating surface (improper crystal seating.)	d. Clean and polish the crystal seating surface on the crystal holder.
	e. Small pieces of material fell on crystal (for crystal facing up sputtering situation.)	e. Check the crystal surface and blow it off with clean air.
	f. Small pieces of magnetic material being attracted by the sensor magnet and contacting the crystal (sputtering sensor head.)	f. Check the sensor cover's aperture and remove any foreign material that may be restricting full crystal coverage.



Table 4-2 Troubleshooting Sensors

SYMPTOM	CAUSE	REMEDY
2. Crystal ceases to oscillate during deposition before it reaches the end of its normal life.	a. Crystal struck by particulate or spatter from molten source.	a. Thermally condition the source thoroughly before deposition, use a shutter to protect the crystal during source conditioning.
	b. Material on crystal holder partially masking crystal cover aperture.	b. Clean crystal holder.
	c. Existence of electrical short or open condition.	c. Using an ohm meter or DMM, check for electrical continuity in the sensor cable, connector, contact springs, connecting wire inside sensor, and feedthroughs.
	d. Thermally induced electrical short or open condition.	d. See 2c above.
<b>NOTE:</b> Crystal life is highly dependent on process conditions of rate, power radiated from source, location, material, and residual gas composition.		
3. Crystal does not oscillate or oscillates intermittently (both in vacuum and in air.)	a. Intermittent or poor electrical contact (contacts oxidized.)	a. Use an Ohm meter or DMM to check electrical continuity, clean contacts.
	b. Leaf springs have lost retentivity (ceramic retainer, center insulator.)	b. Carefully bend leaves to approx. 45° on ceramic retainer and 60° inside the sensor head.
	c. RF interference from sputtering power supply.	c. Verify earth ground, use ground strap adequate for RF ground, change location of Q-pod and cabling away from RF power lines.
	d. Cables not connected, or connected to wrong sensor input.	d. Verify proper connections, and inputs relative to programmed sensor parameter.

Table 4-2 Troubleshooting Sensors

SYMPTOM	CAUSE	REMEDY
4. Crystal oscillates in vacuum but stops oscillation after open to air.	a. Crystal was near the end of its life; opening to air causes film oxidation which increases film stress.	a. Replace crystal.
	b. Excessive moisture accumulates on the crystal.	b. Turn off cooling water to sensor prior to venting, flow warm water through sensor while chamber is open.
5. Thermal instability: large changes in thickness reading during source warm-up (usually causes thickness reading to decrease) and after the termination of deposition (usually causes thickness reading to increase.)	a. Inadequate cooling water/cooling water temperature too high.	a. Check cooling water flow rate, be certain that cooling water temperature is less than 30°C; refer to appropriate sensor manual.
	b. Excessive heat input to the crystal.	b. If heat is due to radiation from the evaporation source, move sensor further away from source and use silver crystals for better thermal stability; install radiation shield.
	c. Crystal not seated properly in holder.	c. Clean or polish the crystal seating surface on the crystal holder.
	d. Crystal heating caused by high energy electron flux (often found in RF sputtering.)	d. Use a sputtering sensor head.
	e. Poor thermal transfer (Bakeable.)	f. Use Al or Au foil washer between crystal holder and sensor body.

Table 4-2 Troubleshooting Sensors

SYMPTOM	CAUSE	REMEDY
6. Poor thickness reproducibility.	a. Variable source flux distribution.	a. Move sensor to a more central location to reliably sample evaporant, ensure constant relative pool height of melt, avoid tunneling into the melt.
	b. Sweep, dither, or position where the electron beam strikes the melt has been changed since the last deposition.	b. Maintain consistent source distribution by maintaining consistent sweep frequencies, sweep amplitude and electron beam position settings.
	c. Material does not adhere to the crystal.	c. Make certain the crystal surface is clean; avoid touching crystal with fingers, make use of an intermediate adhesion layer.
	d. Cyclic change in rate.	d. Make certain source's sweep frequency is not "beating" with the Q-pod's measurement frequency.
7. Large drift in thickness (greater than 200 Å for a density of 5.00 g/cc) after termination of sputtering.	a. Crystal heating due to poor thermal contact.	a. Clean or polish the crystal seating surface on the crystal holder.
	b. External magnetic field interfering with the sensor's magnetic field (sputtering sensor.)	b. Rotate sensor magnet to proper orientation with external magnetic field, refer to the sputtering sensor manual IPN 074-157.
	c. Sensor magnet cracked or demagnetized (sputtering sensor.)	c. Check sensor magnetic field strength, the maximum field at the center of the aperture should be 700 gauss or greater.

## 4.1.4 Troubleshooting Computer Communications

Table 4-3 Troubleshooting Computer Communications

SYMPTOM	CAUSE	REMEDY
1. Communications cannot be established between the host computer and the Q-pod.	a. Improper cable connection.	a. Verify for cable connections are seated properly.
	b. Driver not installed properly.	b. Reinstall drivers. Confirm the operating system is an accepted OS. If autorun driver install feature does not work, the driver can be installed from the device manager manually.

## 4.2 Replacing the Crystal



### CAUTION

Always use clean nylon lab gloves and plastic tweezers for handling the crystal (to avoid contamination which may lead to poor adhesion of the film to the electrode).

Do not rotate the ceramic retainer assembly after it is seated (as this will scratch the crystal electrode and cause poor contact).

Do not use excessive force when handling the ceramic retainer assembly since breakage may occur.

**NOTE:** Certain materials, especially dielectrics, may not adhere strongly to the crystal surface and may cause erratic readings.

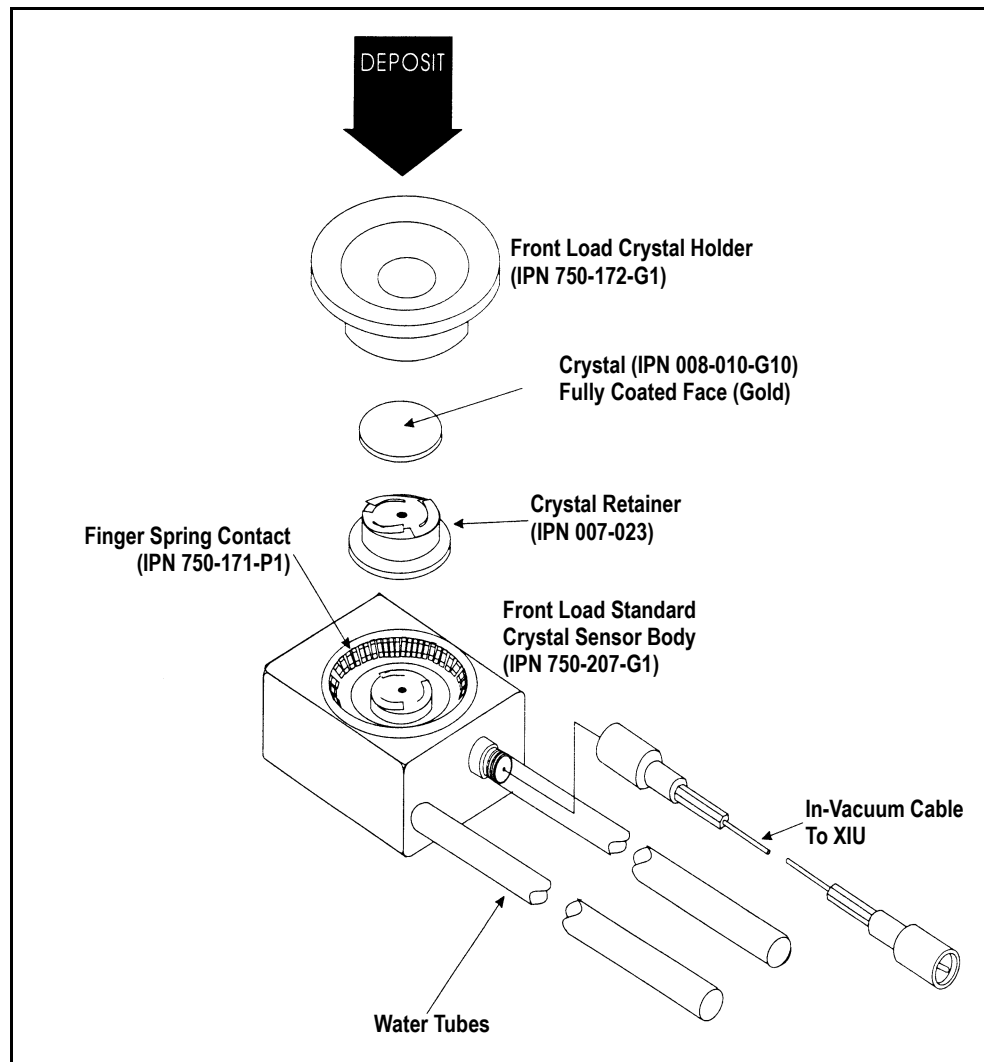
**NOTE:** Thick deposits of some materials, such as SiO<sub>2</sub>, Si, and Ni will normally peel off the crystal when it is exposed to air, as a result of changes in film stress caused by gas absorption. When you observe peeling, replace the crystals.

### 4.2.1 Front Load

Follow the procedure below to replace the crystal in the Front Load sensor: (see [Figure 4-1](#))

- 1 Gripping the crystal holder with your fingers, pull it straight out of the sensor body.
- 2 Gently pry the crystal retainer from the holder (or use the Crystal Snatcher; see [Figure 4-6 on page 4-14](#)).
- 3 Turn the retainer over and the crystal will drop out.
- 4 Install a new crystal, with the patterned electrode face up.
- 5 Push the retainer back into the holder and replace the holder in the sensor body.

Figure 4-1 Front Load Crystal Sensor (Exploded)



### 4.2.2 Cool Drawer

Follow the procedure below to replace the crystal in a Next Generation Cool Drawer™ sensor:

- 1 Using your thumb and index fingers, gently squeeze the sides of the retainer at the mid section then lift it up, away from the drawer, as shown in [Figure 4-2](#).
- 2 Hold the drawer by the handle and turn it upside down to remove the spent crystal.
- 3 Install a new crystal in the drawer. Observe its orientation. The pattern electrode should face upward as shown in [Figure 4-3](#).
- 4 Hold the retainer by its sides. Align its orientation notch with the drawer then gently and evenly push the retainer down until it snaps firmly into the drawer. see [Figure 4-3](#). Never push down (or pull up) on the contact spring, doing so may permanently damage it.
- 5 Inspect the whole assembly. The retainer should be even and engage the drawer at all four corners.

Figure 4-2 Cool Drawer - Removing The Crystal

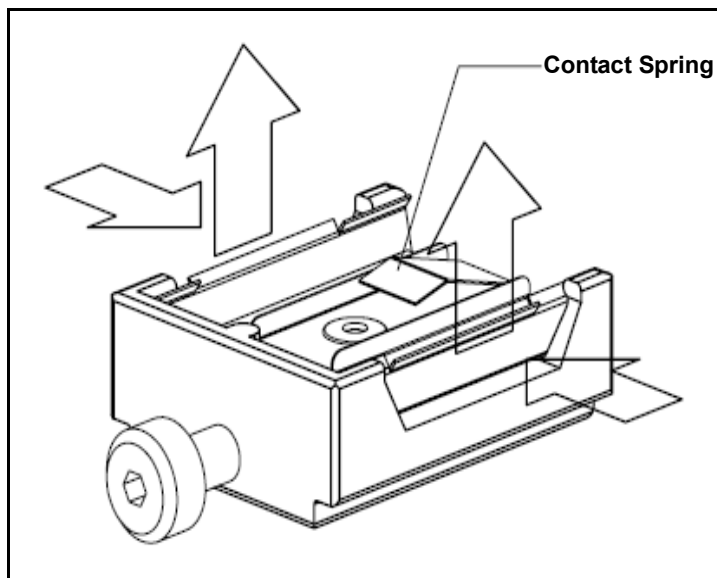
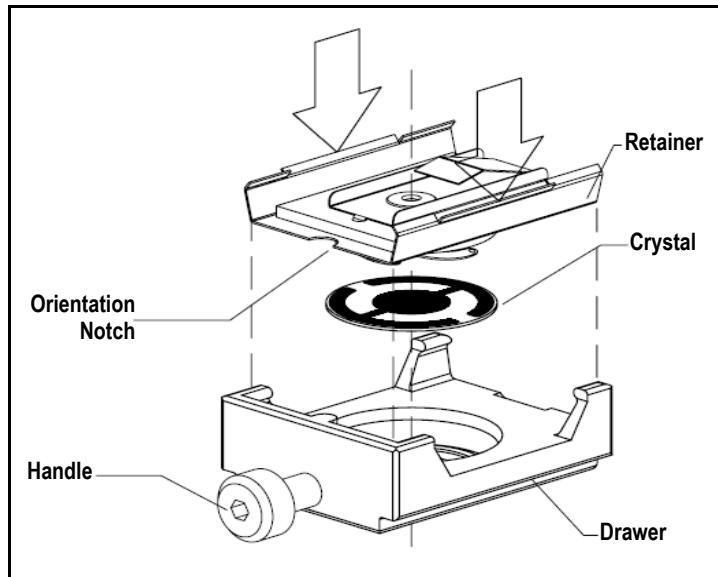


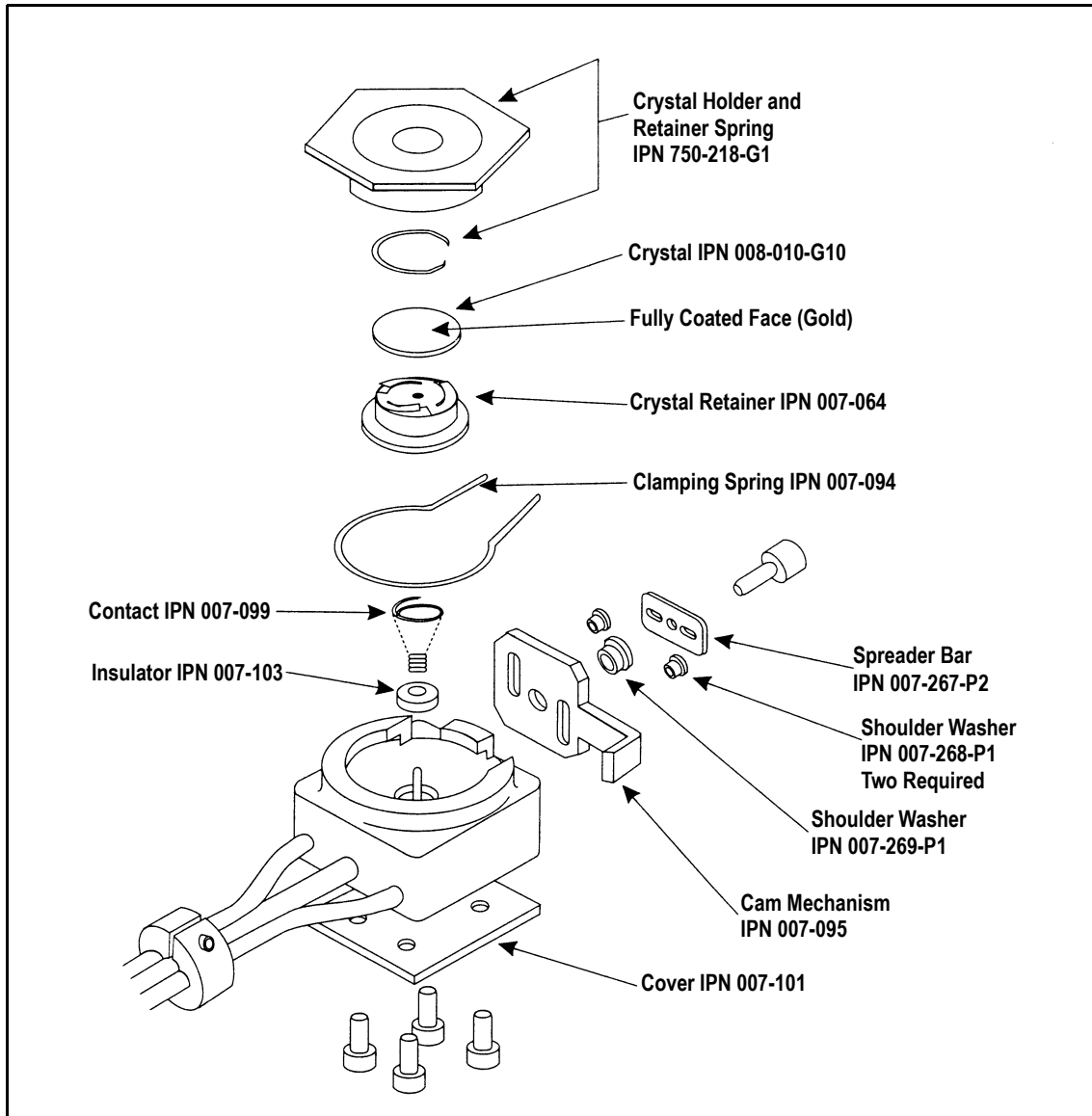
Figure 4-3 Cool Drawer - Replacing The Crystal



### 4.2.3 Bakeable Sensor

For the Bakeable sensor, the procedure is the same as the Front Load sensor except that you must first unlock the cam assembly by flipping it up. Once the crystal has been replaced, place a flat edge of the holder flush with the cam mechanism and lock it in place with the cam. See [Figure 4-4](#).

Figure 4-4 Bakeable Crystal Sensor



IPN 074-547-P1B

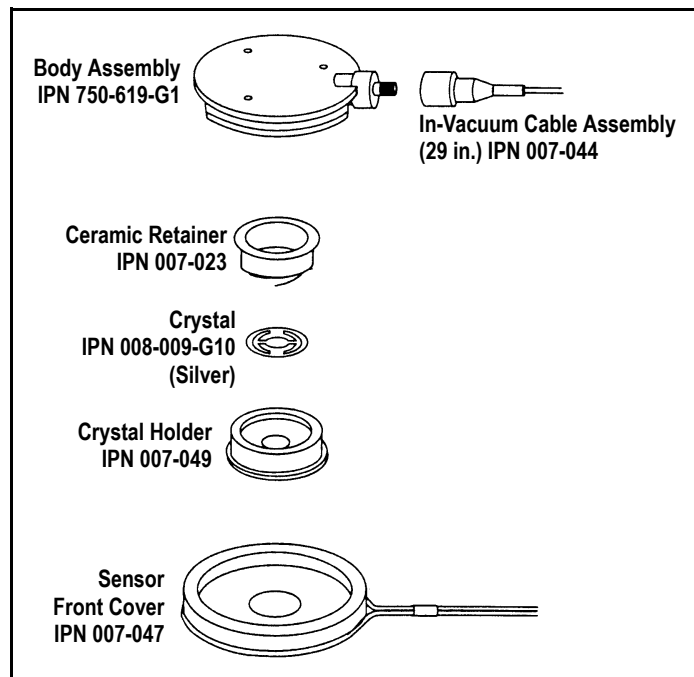


## 4.2.4 Sputtering Sensor

Observe the general precautions for replacing crystals and follow the instructions below to replace the crystal in a sputtering sensor.

- 1 Grip the body assembly with your fingers and pull it straight out to separate it from the water-cooled front cover. (You may have to disconnect the sensor cable in order to separate the parts.) See [Figure 4-5](#).
- 2 Pull the crystal holder straight out from the front of the body assembly.
- 3 Remove the ceramic retainer from the crystal holder by pulling it straight out with the crystal snatcher (see [section 4.2.5 on page 4-14](#)).
- 4 Turn the crystal holder over so that the crystal drops out.
- 5 Install a new crystal into the crystal holder with the patterned electrode facing the back and contacting the leaf springs on the ceramic retainer.
- 6 Put the ceramic retainer back into the crystal holder and put the holder into the body assembly of the sensor.
- 7 Align the position of the body assembly so that the connector matches with the notch on the front cover of the sensor. Snap the two parts together. Reconnect the sensor cable if it has been disconnected.

Figure 4-5 Sputtering Crystal Sensor

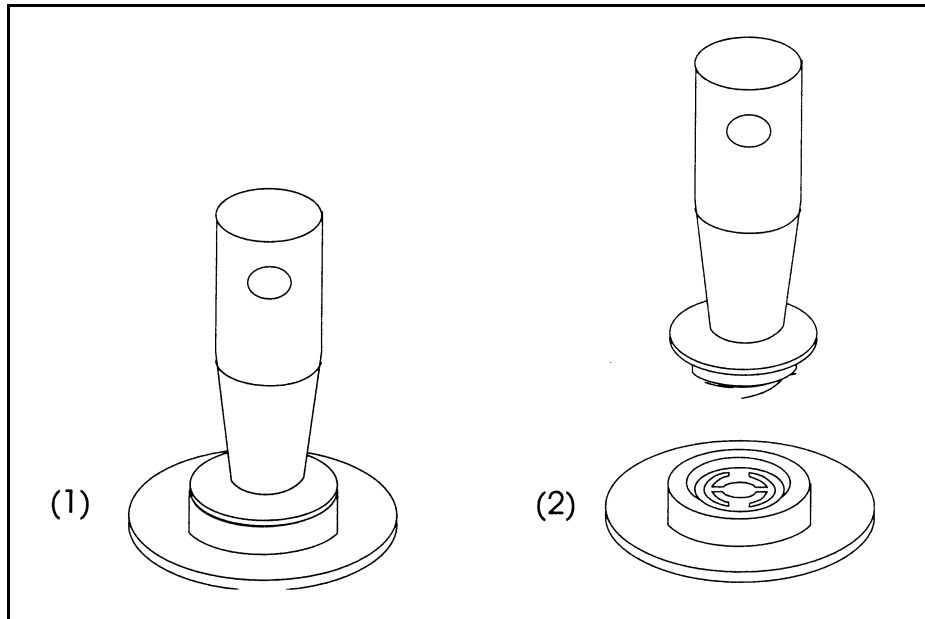


### 4.2.5 Crystal Snatcher

Use the crystal snatcher, supplied with the sensor, as follows:

- 1** Insert crystal snatcher into ceramic retainer (1) and apply a small amount of pressure. This locks the retainer to the snatcher and allows the retainer to be pulled straight out (2). See [Figure 4-6](#).
- 2** Reinsert the retainer into the holder after the crystal has been replaced.
- 3** Release the crystal snatcher with a slight side-to-side motion.

*Figure 4-6 Use of the Crystal Snatcher*



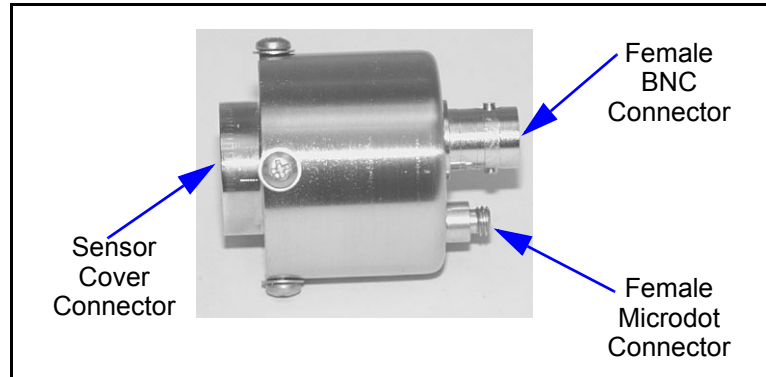
## 4.3 Crystal Sensor Emulator

### IPN 760-601-G2

**NOTE:** 760-601-G2 is fully compatible with all Thin Film Deposition Controllers.

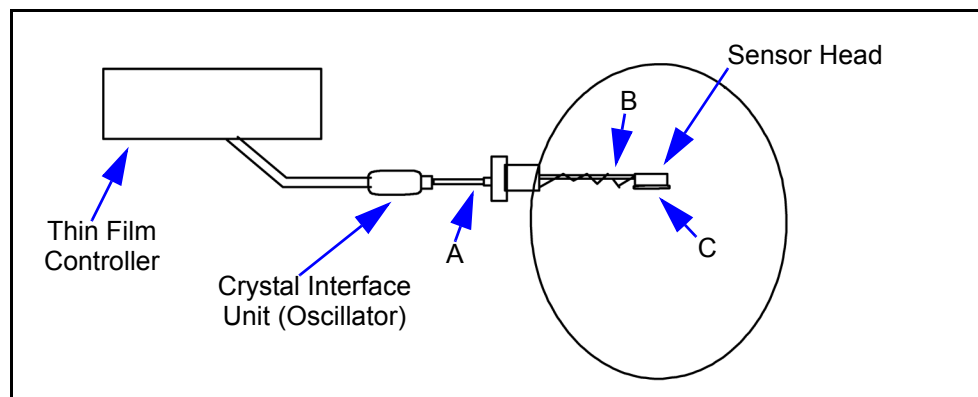
The Crystal Sensor Emulator option is used in conjunction with the Thin Film Deposition Controller to rapidly diagnose problems with the Deposition Controller's measurement system. See [Figure 4-7](#).

Figure 4-7 Crystal Sensor Emulator



The Crystal Sensor Emulator may be attached at various points in the measurement system, from the oscillator to the sensor head. It provides a known good monitor crystal with known good electrical connections. Using the emulator and the controller in a systematic manner provides a fast means of isolating measurement system, cable, or sensor problems. See [Figure 4-8](#).

Figure 4-8 Crystal Sensor Emulator Attachment Points



### CAUTION

This product is designed as a diagnostic tool, and is not intended for use in vacuum. Do not leave the Crystal Sensor Emulator installed in the vacuum system during processing.

### 4.3.1 Diagnostic Procedures

The following diagnostic procedures employ the Crystal Sensor Emulator to analyze a constant Crystal Fail message. The symptom is a Crystal Fail message that is displayed by the Q-pod software even after the monitor crystal has been replaced with a new good monitor crystal.

#### 4.3.1.1 Measurement System Diagnostic Procedure

- 1 Refer to [Figure 4-8 on page 4-15](#). Remove the six-inch BNC cable from the Feed-Through at point A.
- 2 Connect the Crystal Sensor Emulator to the 6 inch BNC cable at Point A.
  - ♦ If the Crystal Fail message disappears after approximately five seconds, the measurement system is working properly. Re-install the six-inch BNC cable to the Feed-Through. Go to [section 4.3.1.2](#).
  - ♦ If the Crystal Fail message remains, continue at step 3.
- 3 Disconnect the six-inch BNC cable from the Oscillator and from the Emulator.
- 4 Visually inspect the six-inch BNC cable to verify that the center pins are seated properly.
- 5 Use an Ohm meter to verify the electrical connections on the six-inch BNC cable.
  - ♦ There must be continuity ( $<0.2$  ohms, after accounting for resistance of ohmmeter leads) between the center pins.
  - ♦ There must be isolation ( $>10$  megohms) between the center pins and the connector shield.
  - ♦ There must be continuity between the connector shields.

Replace the six-inch BNC cable if it is found to be defective and repeat Step 2 of this procedure.
- 6 If the six-inch BNC cable is not defective, re-connect the six-inch cable to the oscillator and to the Crystal Sensor Emulator. If the Crystal Fail message remains, contact INFICON.

#### 4.3.1.2 Feed-Through Or In-Vacuum Cable Diagnostic Procedure

- 1 Refer to [Figure 4-8 on page 4-15](#). Remove the In-Vacuum cable from the Sensor Head at point B.
- 2 Connect the Crystal Sensor Emulator to the In-Vacuum cable.
  - ♦ If the Crystal Fail message disappears after approximately five seconds, the Feed-Through and In-Vacuum Cable are working properly. Re-install the In-Vacuum cable to the Sensor Head. Go to [section 4.3.1.3 on page 4-18](#).
  - ♦ If the Crystal Fail message remains, continue at step 3.
- 3 Disconnect the In-Vacuum cable from the Feed-Through and the Emulator. Disconnect the six-inch BNC cable from the Feed-Through.
- 4 Using an Ohm Meter, verify electrical continuity from the BNC center pin on the Feed-Through to the Microdot center pin on the Feed-Through. A typical value would be less than 0.2 ohms.
- 5 Verify electrical isolation of the center pin on the Feed-Through from the electrical ground (Feed-Through body). A typical value would be in excess of 10 megohms.

If the Feed-Through is found to be defective, replace the Feed-Through, re-attach the BNC and In-Vacuum cables, and repeat this procedure starting at Step 2, otherwise continue at step 6.

- 6 Verify electrical continuity from center pin to center pin on the In-Vacuum cable.
- 7 Verify that the center pin of the In-Vacuum cable is electrically isolated from the In-Vacuum cable shield.

If the In-Vacuum cable is found to be defective, replace the In-Vacuum cable. Re-attach the BNC and In-Vacuum cables, and repeat this procedure starting at Step 2, otherwise continue at step 8.

- 8 Connect the In-Vacuum Cable to the Feed-Through.
- 9 Verify electrical continuity from the center pin on the BNC connector of the Feed-Through to the center pin on the un-terminated end of the In-Vacuum cable.
- 10 Verify electrical isolation from the center pin to electrical ground (Feed-Through body).

If the Feed-Through/In-Vacuum cable system is found to be defective, look for defective electrical contacts at the Feed-Through to In-Vacuum cable connection. Repair or replace the Feed-Through as necessary. Re-attach the BNC and In-Vacuum cables and repeat this procedure starting at step 2. Otherwise, continue at step 11.

- 11** Connect the six-inch BNC cable to the Feed-Through and disconnect it from the Q-pod.
- 12** Verify electrical continuity from the center pin of the Microdot connector on the Feed-Through to the un-terminated end of the six-inch BNC cable.
- 13** Verify electrical isolation from the center pin to electrical ground (Feed-Through body).

If the Feed-Through/six-inch BNC cable system is found to be defective, look for defective contacts at the Feed-Through to BNC cable connection. Repair or replace the Feed-Through as necessary, re-attach the BNC cable to the XIU and In-Vacuum cable to the Crystal head and repeat this procedure starting at step 2.

#### **4.3.1.3 Sensor Head Or Monitor Crystal Diagnostic Procedure**

**NOTE:** The procedure is for use with front load style sensor heads.

- 1** Remove the Crystal Cover from the Sensor Head.
- 2** Refer to [Figure 4-7 on page 4-15](#). Connect the Crystal Sensor Emulator to the Sensor Head at Point C.
  - ♦ If the Crystal Fail message disappears after approximately 5 seconds the Sensor Head is operating properly. Remove the Crystal Sensor Emulator and re-insert the Crystal Cover into the Sensor Head.
  - ♦ If the Crystal Fail message remains, continue at step 3.
- 3** Disconnect the In-Vacuum cable from the Sensor Head and the Feed-Through. Remove the Crystal Sensor Emulator from the Sensor Head.
- 4** Using an Ohm meter, verify the electrical connections on the Sensor Head.
  - ♦ Verify there is electrical continuity from the center pin contact on the Microdot connector on the Sensor Head to the leaf spring contact in the Sensor Head. Take care not to apply too much pressure on the center pin of the microdot connector as it may become damaged.
  - ♦ There must be electrical isolation between the center pin of the Microdot connector and the Sensor Head body.

If the Sensor Head is found to be defective, contact INFICON to have the Sensor Head repaired.

**5** Connect the In-Vacuum Cable to the Sensor Head.

- ♦ Verify there is continuity ( $<0.2$  ohm) from the leaf spring contact in the Sensor Head to the center pin on the un-terminated end of the In-Vacuum cable.
- ♦ Verify there is isolation ( $>10$  megohm) between the leaf spring contact and the In-Vacuum cable shield.

If the Sensor Head or the In-Vacuum cable system is found to be defective, look for defective contacts at the In-Vacuum cable to Sensor Head connection, repair or replace the Sensor Head as necessary. Re-attach the In-Vacuum cable to the Feed-Through and repeat this procedure starting at step 2.

**6** Ensure that the leaf springs in the Sensor Head and those in the Ceramic Retainer are bent to an angle of approximately  $60^\circ$  and  $45^\circ$  from flat, respectively.**4.3.1.4 System Diagnostics Pass But  
Crystal Fail Message Remains**

If the system is operating properly yet the Crystal Fail message is still displayed, perform the following tasks.

- 1** On the Ceramic Retainer verify that the center rivet is secure. Repair or replace the Ceramic Retainer as necessary.
- 2** Inspect the inside of the Crystal Holder for build-up of material. Clean or replace the Crystal Holder as necessary.

After verifying the Sensor Head contacts, the Sensor Head/In-Vacuum cable connection and the ceramic retainer contacts, re-assemble the system. If the Crystal Fail message remains, replace the monitor crystal with a new monitor crystal. Verify that the monitor crystal works properly by inserting it into a known good measurement system. If you continue to experience problems, contact INFICON.

## 4.3.2 Sensor Cover Connection

The Crystal Sensor Emulator can be used to verify the measurement system for INFICON Thin Film Deposition Controllers and Monitors.

However, the Crystal Sensor Emulator's Sensor Cover Connector is compatible with some sensor heads, and is incompatible with others. This is discussed in the following sections.

### 4.3.2.1 Compatible Sensor Heads

The Sensor Cover Connection will fit the sensor heads shown in [Table 4-4](#).

Table 4-4 Compatible Sensor Heads

Sensor Head	Part Number
Front Load Single Sensor Head	SL-XXXXX
Front Load Dual Sensor Head	DL-AEXX

### 4.3.2.2 Incompatible Sensor Heads

The Sensor Heads for which the Crystal Sensor Emulator's Sensor Cover Connector will not fit are shown in [Table 4-5](#).

Table 4-5 Incompatible Sensor Heads

Sensor Head	Part Number
Front Load UHV Bakeable Sensor Head	BK-AXX
Cool Drawer Single Sensor Head	CDS-XXXXX
Sputtering Sensor Head	750-618-G1
CrystalSix Sensor Head	750-446-G1
Cool Drawer Dual Sensor Head	CDD-XXXX
Crystal12 Sensor Head	XL12-XXXXXX
RSH-600 Sensor Head	15320X-XX

**NOTE:** The Crystal Sensor Emulator's Sensor Cover will not fit the crystal holder opening of the older style INFICON transducers that have the soldered finger springs.



### **4.3.3 Emulator Specifications**

#### **Dimensions**

1.58 in. diameter x 1.79 in.  
(40.13 mm diameter x 45.47 mm)

#### **Temperature Range**

0 to 50°C

#### **Frequency**

760-601-G2: 5.5 MHz  $\pm$  1 ppm at room temperature

#### **Materials**

304 Stainless Steel, Nylon, Teflon®, brass. Some internal components contain zinc, tin, and lead.

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## Chapter 5

# Calibration Procedures

### 5.1 Importance of Density, Tooling and Z-Ratio

The quartz crystal microbalance is capable of precisely measuring the mass added to the face of the oscillating quartz crystal sensor. The Q-pod's knowledge of the density of this added material (specified in the density parameter in material grid) allows conversion of the mass information into thickness. In some instances, where highest accuracy is required, it is necessary to make a density calibration as outlined in [section 5.2](#).

Because the flow of material from a deposition is not uniform, it is necessary to account for the different amount of material flow onto the sensor compared to the substrates. This factor is accounted for in the tooling parameter in material grid. The tooling factor can be experimentally established by following the guidelines in [section 5.3 on page 5-2](#).

If the Z-Ratio is not known, it could be estimated from the procedures outlined in [section 5.4 on page 5-2](#).

### 5.2 Determining Density

**NOTE:** The bulk density values retrieved from [Table A-1](#) are sufficiently accurate for most applications.

Follow the steps below to determine density value.

- 1** Place a substrate (with proper masking for film thickness measurement) adjacent to the sensor, so that the same thickness will be accumulated on the crystal and substrate.
- 2** Set density to the bulk value of the film material or to an approximate value.
- 3** Set Z-Ratio to 1.000 and tooling to 100%.
- 4** Place a new crystal in the sensor and make a short deposition (1000-5000 Å).
- 5** After deposition, remove the test substrate and measure the film thickness with either a multiple beam interferometer or a stylus-type profilometer.
- 6** Determine the new density value with [equation \[1\]](#):

$$\text{Density(g/cm}^3\text{)} = D_1 \left( \frac{T_x}{T_m} \right) \quad [1]$$

where:

$D_1$  = Initial density setting

$T_x$  = Thickness reading on Q-pod

$T_m$  = Measured thickness

- 7 A quick check of the calculated density may be made by programming the Q-pod with the new density value and observing that the displayed thickness is equal to the measured thickness, provided that the Q-pod's thickness has not been zeroed between the test deposition and entering the calculated density.

**NOTE:** Slight adjustment of density may be necessary in order to achieve  $T_x = T_m$ .

### 5.3 Determining Tooling

- 1 Place a test substrate in the system's substrate holder.
- 2 Make a short deposition and determine actual thickness.
- 3 Calculate tooling from the relationship shown in [equation \[2\]](#):

$$\text{Tooling (\%)} = TF_i \left( \frac{T_m}{T_x} \right) \quad [2]$$

where

$T_m$  = Actual thickness at substrate holder

$T_x$  = Thickness reading in the Q-pod

$TF_i$  = Initial tooling factor

- 4 Round off percent tooling to the nearest 0.1%.
- 5 When entering this new value for tooling into the program,  $T_m$  will equal  $T_x$  if calculations are done properly.

**NOTE:** It is recommended that a minimum of three separate evaporations be made when calibrating tooling. Variations in source distribution and other system factors will contribute to slight thickness variations. An average value tooling factor should be used for final calibrations.

### 5.4 Laboratory Determination of Z-Ratio

A list of Z-values for materials commonly used are available in [Table A-1](#). For other materials, Z can be calculated from the following formula:

$$Z = \left( \frac{d_q \mu_q}{d_f \mu_f} \right)^{\frac{1}{2}} \quad [3]$$

$$Z = 9.378 \times 10^5 (d_f \mu_f)^{-\frac{1}{2}} \quad [4]$$

where:

$d_f$  = density (g/cm<sup>3</sup>) of deposited film

$\mu_f$  = shear modulus (dynes/cm<sup>2</sup>) of deposited film

$d_q$  = density of quartz (crystal) (2.649 gm/cm<sup>3</sup>)

$\mu_q$  = shear modulus of quartz (crystal) ( $3.32 \times 10^{11}$  dynes/cm<sup>2</sup>)

The densities and shear moduli of many materials can be found in a number of handbooks.

Laboratory results indicate that Z-values of materials in thin-film form are very close to the bulk values. However, for high stress producing materials, Z-values of thin films are slightly smaller than those of the bulk materials. For applications that require more precise calibration, the following direct method is suggested:

- 1** Establish the correct density value as described in [section 5.2 on page 5-1](#).
- 2** Install a new crystal and record its starting frequency,  $F_{co}$ . The starting frequency will be displayed on the main screen.
- 3** Make a deposition on a test substrate such that the percent crystal life display will read approximately 50%, or near the end of crystal life for the particular material, whichever is smaller.
- 4** Stop the deposition and record the ending crystal frequency  $F_c$ .
- 5** Remove the test substrate and measure the film thickness with either a multiple beam interferometer or a stylus-type profilometer.
- 6** Using the density value from step 1 and the recorded values for  $F_{co}$  and  $F_c$ , adjust the Z-ratio value in thickness [equation \[5\]](#) to bring the calculated thickness value into agreement with the actual thickness. If the calculated value of thickness is greater than the actual thickness, increase the Z-Ratio value. If the calculated value of thickness is less than the actual thickness, decrease the Z-Ratio value.

$$T_f = \frac{Z_q \times 10^4}{2\pi z p} \left\{ \left( \frac{1}{F_{co}} \right) A \tan \left( z \tan \left( \frac{\pi F_{co}}{F_q} \right) \right) - \left( \frac{1}{F_c} \right) A \tan \left( z \tan \left( \frac{\pi F_c}{F_q} \right) \right) \right\} \quad [5]$$

where:

$T_f$  = thickness of deposited film (kÅ)

$F_{co}$  = starting frequency of the sensor crystal (Hz)

$F_c$  = Final frequency of the sensor crystal (Hz)

$F_q$  = Nominal blank frequency = 6045000 (Hz)

$z$  = Z-ratio of deposited film material

$Z_q$  = Specific acoustic impedance of quartz = 8765000 (MKS units)

$p$  = density of deposited film (g/cc)

For multiple layer deposition (for example, two layers), the Z-value used for the second layer is determined by the relative thickness of the two layers. For most applications the following three rules will provide reasonable accuracies:

- ♦ If the thickness of layer 1 is large compared to layer 2, use material 1 Z-value for both layers.
- ♦ If the thickness of layer 1 is thin compared to layer 2, use material 2 Z-value for both layers.
- ♦ If the thickness of both layers is similar, use a value for Z-Ratio which is the weighted average of the two Z values for deposition of layer 2 and subsequent layers.

## Chapter 6

# Measurement and Theory

### 6.1 Basics

The Quartz Crystal deposition Monitor, or QCM, utilizes the piezoelectric sensitivity of a quartz monitor crystal to added mass. The QCM uses this mass sensitivity to control the deposition rate and final thickness of a vacuum deposition. When a voltage is applied across the faces of a properly shaped piezoelectric crystal, the crystal is distorted and changes shape in proportion to the applied voltage. At certain discrete frequencies of applied voltage, a condition of very sharp electro-mechanical resonance is encountered. When mass is added to the face of a resonating quartz crystal, the frequency of these resonances are reduced. This change in frequency is very repeatable and is precisely understood for specific oscillating modes of quartz. This heuristically easy to understand phenomenon is the basis of an indispensable measurement and process control tool that can easily detect the addition of less than an atomic layer of an adhered foreign material.

In the late 1950's it was noted by Sauerbrey<sup>1,2</sup> and Lostis<sup>3</sup> that the change in frequency,  $DF = F_q - F_c$ , of a quartz crystal with coated (or composite) and uncoated frequencies,  $F_c$  and  $F_q$  respectively, is related to the change in mass from the added material,  $M_f$ , as follows:

$$\frac{M_f}{M_q} = \frac{(\Delta F)}{F_q} \quad [1]$$

where  $M_q$  is the mass of the uncoated quartz crystal. Simple substitutions lead to the equation that was used with the first "frequency measurement" instruments:

$$T_f = \frac{K(\Delta F)}{d_f} \quad [2]$$

where the film thickness,  $T_f$ , is proportional (through  $K$ ) to the frequency change,  $DF$ , and inversely proportional to the density of the film,  $d_f$ . The constant,  $K = N_{at}d_q/F_q^2$ ; where  $d_q$  ( $= 2.649 \text{ gm/cm}^3$ ) is the density of single crystal quartz and  $N_{at}$  ( $= 166100 \text{ Hz cm}$ ) is the frequency constant of AT cut quartz. A crystal with a starting frequency of 6.0 MHz will display a reduction of its frequency by 2.27 Hz when 1 angstrom of Aluminum (density of  $2.77 \text{ gm/cm}^3$ ) is added to its surface. In this manner the thickness of a rigid adlayer is inferred from the precise measurement of the crystal's frequency shift. The quantitative knowledge of this

1.G. Z. Sauerbrey, Phys. Verhand .8, 193 (1957)

2.G. Z. Sauerbrey, Z. Phys. 155,206 (1959)

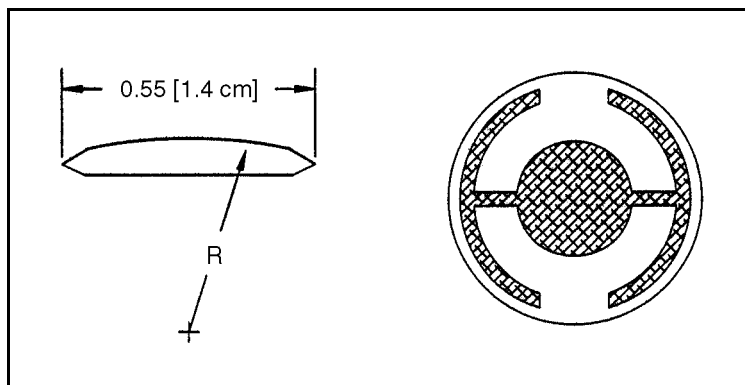
3.P. Lostis, Rev. Opt. 38,1 (1959)

effect provides a means of determining how much material is being deposited on a substrate in a vacuum system, a measurement that was not convenient or practical prior to this understanding.

### 6.1.1 Monitor Crystals

No matter how sophisticated the electronics surrounding it, the essential device of the deposition monitor is the quartz crystal. The quartz crystal shown in [Figure 6-1](#) has a frequency response spectrum that is schematically shown in [Figure 6-2](#). The ordinate represents the magnitude of response, or current flow of the crystal, at the specified frequency.

Figure 6-1 Quartz Resonator



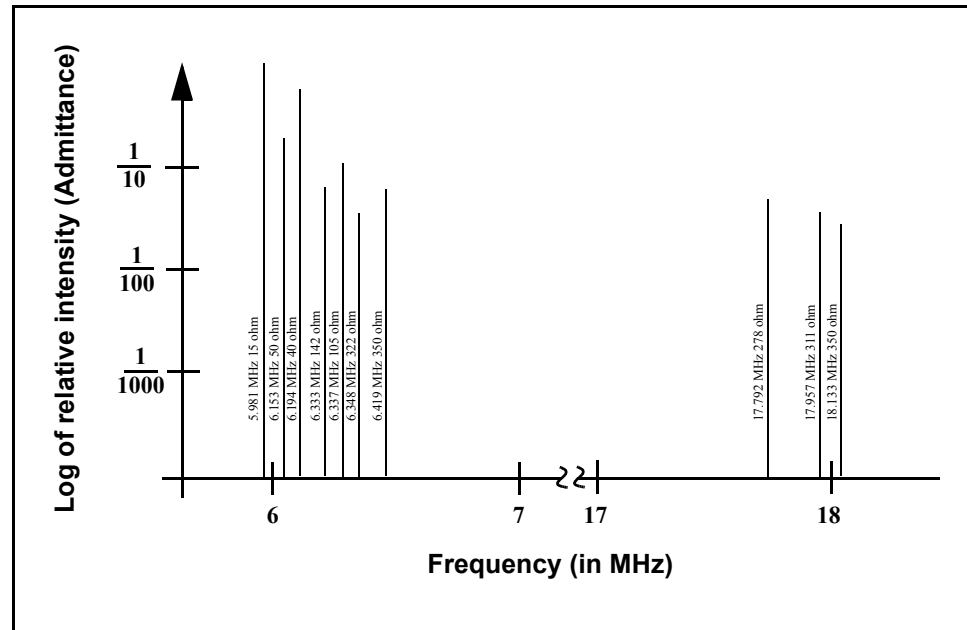
The lowest frequency response is primarily a “thickness shear” mode that is called the fundamental. The characteristic movement of the thickness shear mode is for displacement to take place parallel to the major monitor crystal faces. In other words, the faces are displacement antinodes as shown in [Figure 6-3](#). The responses located slightly higher in frequency are called anharmonics; they are a combination of the thickness shear and thickness twist modes. The response at about three times the frequency of the fundamental is called the third quasiharmonic. There are also a series of anharmonics slightly higher in frequency associated with the quasiharmonic.

The monitor crystal design depicted in [Figure 6-1](#) is the result of several significant improvements from the square crystals with fully electroded plane parallel faces that were first used. The first improvement was to use circular crystals. This increased symmetry greatly reduced the number of allowed vibrational modes. The second set of improvements was to contour one face of the crystal and to reduce the size of the exciting electrode. These improvements have the effect of trapping the acoustic energy. Reducing the electrode diameter limits the excitation to the central area. Contouring dissipates the energy of the traveling acoustic wave before it reaches the edge of the crystal. Energy is not reflected back to the center where it can interfere with other newly launched waves, essentially making a small crystal appear to behave as though it is infinite in extent. With the crystal’s vibrations restricted to the center, it is practical to clamp the outer edges of the crystal to a holder and not produce any undesirable effects. Contouring also



reduces the intensity of response of the generally unwanted anharmonic modes; hence, the potential for an oscillator to sustain an unwanted oscillation is substantially reduced.

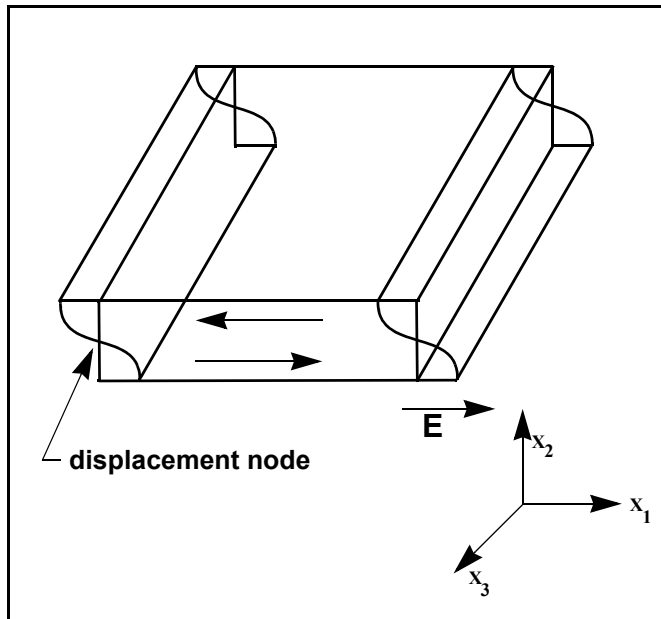
Figure 6-2 Frequency Response Spectrum



The use of an adhesion layer has improved the electrode-to-quartz bonding, reducing “rate spikes” caused by micro-tears between the electrode and the quartz as film stress rises. These micro-tears leave portions of the deposited film unattached and therefore unable to participate in the oscillation. These free portions are no longer detected and the wrong thickness consequently inferred.

The “AT” resonator is usually chosen for deposition monitoring because at room temperature it can be made to exhibit a very small frequency change due to temperature changes. Since there is presently no way to separate the frequency change caused by added mass (which is negative) or even the frequency changes caused by temperature gradients across the crystal or film induced stresses, it is essential to minimize these temperature-induced changes. It is only in this way that small changes in mass can be measured accurately.

Figure 6-3 Thickness Shear Displacement



### 6.1.2 Period Measurement Technique

Although instruments using [equation \[2\]](#) were very useful, it was soon noted they had a very limited range of accuracy, typically holding accuracy for DF less than  $0.02 F_q$ . In 1961 it was recognized by Behrndt<sup>4</sup> that:

$$\frac{M_f}{M_q} = \frac{(T_c - T_q)}{T_q} = \frac{(\Delta F)}{F_c} \quad [3]$$

where  $T_c$  and  $T_q$  are the periods of oscillation of the crystal with film (composite) and the bare crystal respectively. The period measurement technique was the outgrowth of two factors; first, the digital implementation of time measurement, and second, the recognition of the mathematically rigorous formulation of the proportionality between the crystal's thickness,  $l_q$ , and the period of oscillation,  $T_q = 1/F_q$ . Electronically the period measurement technique uses a second crystal oscillator, or reference oscillator, not affected by the deposition and usually much higher in frequency than the monitor crystal. This reference oscillator is used to generate small precision time intervals which are used to determine the oscillation period of the monitor crystal. This is done by using two pulse accumulators. The first is used to accumulate a fixed number of cycles,  $m$ , of the monitor crystal. The second is turned on at the same time and accumulates cycles from the reference oscillator until  $m$  counts are accumulated in the first. Since the frequency of the reference is stable and known, the time to accumulate the  $m$  counts is known to an accuracy equal to  $\pm 2/F_r$  where  $F_r$  is the reference oscillator's frequency. The

4.K. H. Behrndt, J. Vac. Sci. Technol. 8, 622 (1961)

monitor crystal's period is  $(n/F_r)/m$  where  $n$  is the number of counts in the second accumulator. The precision of the measurement is determined by the speed of the reference clock and the length of the gate time (which is set by the size of  $m$ ). Increasing one or both of these leads to improved measurement precision.

Having a high frequency reference oscillator is important for rapid measurements (which require short gating times), low deposition rates and low density materials. All of these require high time precision to resolve the small, mass induced frequency shifts between measurements. When the change of a monitor crystal's frequency between measurements is small, that is, on the same order of size as the measurement precision, it is not possible to establish quality rate control. The uncertainty of the measurement injects more noise into the control loop, which can be counteracted only by longer time constants. Long time constants cause the correction of rate errors to be very slow, resulting in relatively long term deviations from the desired rate. These deviations may not be important for some simple films, but can cause unacceptable errors in the production of critical films such as optical filters or very thin layered superlattices grown at low rates. In many cases the desired properties of these films can be lost if the layer to layer reproducibility exceeds one, or two, percent. Ultimately, the practical stability and frequency of the reference oscillator limits the precision of measurement for conventional instrumentation.

### 6.1.3 Z-match Technique

After learning of fundamental work by Miller and Bolef<sup>5</sup>, which rigorously treated the resonating quartz and deposited film system as a one-dimensional continuous acoustic resonator, Lu and Lewis<sup>6</sup> developed the simplifying Z-match™ equation in 1972. Advances in electronics taking place at the same time, namely the micro-processor, made it practical to solve the Z-match equation in “real-time”. Most deposition process controllers/monitors sold today use this sophisticated equation that takes into account the acoustic properties of the resonating quartz and film system as shown in [equation \[4\]](#).

$$T_f = \left( \frac{N_{at} d_q}{\pi d_f F_c Z} \right) \arctan \left( Z \tan \left[ \frac{\pi (F_q - F_c)}{F_q} \right] \right) \quad [4]$$

where  $Z = (d_q u_q / d_f u_f)^{1/2}$  is the acoustic impedance ratio and  $u_q$  and  $u_f$  are the shear moduli of the quartz and film, respectively. Finally, there was a fundamental understanding of the frequency-to-thickness conversion that could yield theoretically correct results in a time frame that was practical for process control. To achieve this new level of accuracy requires only that the user enter an additional material parameter,  $Z$ , for the film being deposited. This equation has been tested

5.J. G. Miller and D. I. Bolef, J. Appl. Phys. **39**, 5815, 4589 (1968)

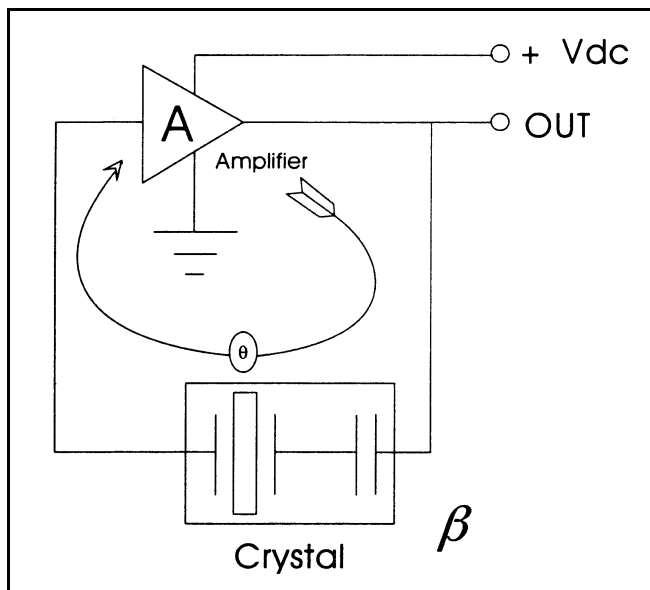
6.C. Lu and O. Lewis, J Appl. Phys. **43**, 4385 (1972)

for a number of materials, and has been found to be valid for frequency shifts equivalent to  $F_f = 0.4F_q$ . Keep in mind that [equation \[2\]](#) was valid to only  $0.02F_q$  and [equation \[3\]](#) was valid only to  $\sim 0.05F_q$ .

#### 6.1.4 Active Oscillator

The Q-pod relies on the use of an active oscillator circuit, Specifically the type schematically shown in [Figure 6-4](#). This circuit actively keeps the crystal in resonance, so that any type of period or frequency measurement may be made. In this type of circuit, oscillation is sustained as long as the gain provided by the amplifiers is sufficient to offset losses in the crystal and circuit and the crystal can provide the required phase shift. The basic crystal oscillator's stability is derived from the rapid change of phase for a small change in the crystal's frequency near the series resonance point, as shown in [Figure 6-6 on page 6-7](#).

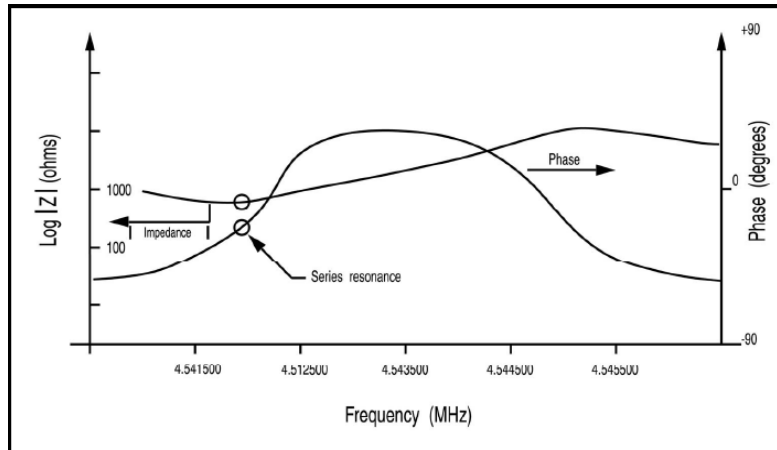
Figure 6-4 Active Oscillator Circuit



The active oscillator circuit is designed so the crystal is required to produce a phase shift of 0 degrees, which allows it to operate at the series resonance point. Long- and short-term frequency stabilities are a property of crystal oscillators because very small frequency changes are needed to sustain the phase shift required for oscillation. Frequency stability is provided by the quartz crystal even though there are long term changes in electrical component values caused by temperature or aging or short-term noise-induced phase jitter.

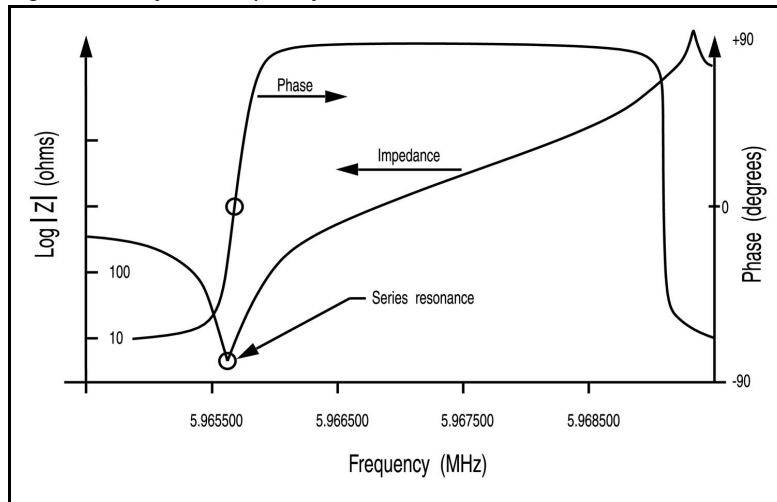
As mass is added to a crystal, its electrical characteristics change. [Figure 6-5 on page 6-7](#) is the same plot as [Figure 6-6](#) overlaid with the response of a heavily loaded crystal.

Figure 6-5 Heavily Loaded Crystal



The crystal has lost the steep slope displayed in Figure 6-6. Because the phase slope is less steep, any noise in the oscillator circuit translates into a greater frequency shift than that which would be produced with a new crystal. In the extreme, the basic phase/frequency shape is not preserved and the crystal is not able to provide a full 90 degrees of phase shift.

Figure 6-6 Crystal Frequency Near Series Resonance Point



The impedance,  $|Z|$ , is also noted to rise to an extremely high value. When this happens it is often more favorable for the oscillator to resonate at one of the anharmonic frequencies. This condition is sometimes short lived, with the oscillator switching between the fundamental and anharmonic modes, or it may continue to oscillate at the anharmonic. This condition is known as mode hopping and in addition to annoying rate noise can also lead to false termination of the film because of the apparent frequency change. It is important to note that the Q-pod will frequently continue to operate under these conditions; in fact there is no way to tell this has happened except that the film's thickness is suddenly apparently thinner by an amount equivalent to the frequency difference between the fundamental and the anharmonic that is sustaining the oscillation.

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## Appendix A

### Material Table

#### A.1 Introduction

The following [Table A-1](#) represents the density and Z-Ratio for various materials. The list is alphabetical by chemical formula.



#### CAUTION

**Some of these materials are toxic. Please consult the material safety data sheet and safety instructions before use.**

An \* is used to indicate that a Z-ratio has not been established for a certain material. A value of 1.000 is defaulted in these situations.

Table A-1 Material Table

Formula	Density	Z-Ratio	Material Name
Ag	10.500	0.529	silver
AgBr	6.470	1.180	silver bromide
AgCl	5.560	1.320	silver chloride
Al	2.700	1.080	aluminum
Al <sub>2</sub> O <sub>3</sub>	3.970	0.336	aluminum oxide
Al <sub>4</sub> C <sub>3</sub>	2.360	*1.000	aluminum carbide
AlF <sub>3</sub>	3.070	*1.000	aluminum fluoride
AlN	3.260	*1.000	aluminum nitride
AlSb	4.360	0.743	aluminum antimonide
As	5.730	0.966	arsenic
As <sub>2</sub> Se <sub>3</sub>	4.750	*1.000	arsenic selenide
Au	19.300	0.381	gold
B	2.370	0.389	boron
B <sub>2</sub> O <sub>3</sub>	1.820	*1.000	boron oxide
B <sub>4</sub> C	2.370	*1.000	boron carbide
BN	1.860	*1.000	boron nitride
Ba	3.500	2.100	barium
BaF <sub>2</sub>	4.886	0.793	barium fluoride

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
BaN <sub>2</sub> O <sub>6</sub>	3.244	1.261	barium nitrate
BaO	5.720	*1.000	barium oxide
BaTiO <sub>3</sub>	5.999	0.464	barium titanate (tetr)
BaTiO <sub>3</sub>	6.035	0.412	barium titanate (cubic)
Be	1.850	0.543	beryllium
BeF <sub>2</sub>	1.990	*1.000	beryllium fluoride
BeO	3.010	*1.000	beryllium oxide
Bi	9.800	0.790	bismuth
Bi <sub>2</sub> O <sub>3</sub>	8.900	*1.000	bismuth oxide
Bi <sub>2</sub> S <sub>3</sub>	7.390	*1.000	bismuth trisulphide
Bi <sub>2</sub> Se <sub>3</sub>	6.820	*1.000	bismuth selenide
Bi <sub>2</sub> Te <sub>3</sub>	7.700	*1.000	bismuth telluride
BiF <sub>3</sub>	5.320	*1.000	bismuth fluoride
C	2.250	3.260	carbon (graphite)
C	3.520	0.220	carbon (diamond)
C <sub>8</sub> H <sub>8</sub>	1.100	*1.000	parlyene (union carbide)
Ca	1.550	2.620	calcium
CaF <sub>2</sub>	3.180	0.775	calcium fluoride
CaO	3.350	*1.000	calcium oxide
CaO-SiO <sub>2</sub>	2.900	*1.000	calcium silicate (3)
CaSO <sub>4</sub>	2.962	0.955	calcium sulfate
CaTiO <sub>3</sub>	4.100	*1.000	calcium titanate
CaWO <sub>4</sub>	6.060	*1.000	calcium tungstate
Cd	8.640	0.682	cadmium
CdF <sub>2</sub>	6.640	*1.000	cadmium fluoride
CdO	8.150	*1.000	cadmium oxide
CdS	4.830	1.020	cadmium sulfide
CdSe	5.810	*1.000	cadmium selenide
CdTe	6.200	0.980	cadmium telluride
Ce	6.780	*1.000	cerium
CeF <sub>3</sub>	6.160	*1.000	cerium (III) fluoride
CeO <sub>2</sub>	7.130	*1.000	cerium (IV) dioxide
Co	8.900	0.343	cobalt



Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
CoO	6.440	0.412	cobalt oxide
Cr	7.200	0.305	chromium
Cr <sub>2</sub> O <sub>3</sub>	5.210	*1.000	chromium (III) oxide
Cr <sub>3</sub> C <sub>2</sub>	6.680	*1.000	chromium carbide
CrB	6.170	*1.000	chromium boride
Cs	1.870	*1.000	cesium
Cs <sub>2</sub> SO <sub>4</sub>	4.243	1.212	cesium sulfate
CsBr	4.456	1.410	cesium bromide
CsCl	3.988	1.399	cesium chloride
CsI	4.516	1.542	cesium iodide
Cu	8.930	0.437	copper
Cu <sub>2</sub> O	6.000	*1.000	copper oxide
Cu <sub>2</sub> S	5.600	0.690	copper (I) sulfide (alpha)
Cu <sub>2</sub> S	5.800	0.670	copper (I) sulfide (beta)
CuS	4.600	0.820	copper (II) sulfide
Dy	8.550	0.600	dysprosium
DY <sub>2</sub> O <sub>3</sub>	7.810	*1.000	dysprosium oxide
Er	9.050	0.740	erbium
Er <sub>2</sub> O <sub>3</sub>	8.640	*1.000	erbium oxide
Eu	5.260	*1.000	europium
EuF <sub>2</sub>	6.500	*1.000	europium fluoride
Fe	7.860	0.349	iron
Fe <sub>2</sub> O <sub>3</sub>	5.240	*1.000	iron oxide
FeO	5.700	*1.000	iron oxide
FeS	4.840	*1.000	iron sulphide
Ga	5.930	0.593	gallium
Ga <sub>2</sub> O <sub>3</sub>	5.880	*1.000	gallium oxide (b)
GaAs	5.310	1.590	gallium arsenide
GaN	6.100	*1.000	gallium nitride
GaP	4.100	*1.000	gallium phosphide
GaSb	5.600	*1.000	gallium antimonide
Gd	7.890	0.670	gadolinium
Gd <sub>2</sub> O <sub>3</sub>	7.410	*1.000	gadolinium oxide

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
Ge	5.350	0.516	germanium
Ge <sub>3</sub> N <sub>2</sub>	5.200	*1.000	germanium nitride
GeO <sub>2</sub>	6.240	*1.000	germanium oxide
GeTe	6.200	*1.000	germanium telluride
Hf	13.090	0.360	hafnium
HfB <sub>2</sub>	10.500	*1.000	hafnium boride
HfC	12.200	*1.000	hafnium carbide
HfN	13.800	*1.000	hafnium nitride
HfO <sub>2</sub>	9.680	*1.000	hafnium oxide
HfSi <sub>2</sub>	7.200	*1.000	hafnium silicide
Hg	13.460	0.740	mercury
Ho	8.800	0.580	holmium
Ho <sub>2</sub> O <sub>3</sub>	8.410	*1.000	holmium oxide
In	7.300	0.841	indium
In <sub>2</sub> O <sub>3</sub>	7.180	*1.000	indium sesquioxide
In <sub>2</sub> Se <sub>3</sub>	5.700	*1.000	indium selenide
In <sub>2</sub> Te <sub>3</sub>	5.800	*1.000	indium telluride
InAs	5.700	*1.000	indium arsenide
InP	4.800	*1.000	indium phosphide
InSb	5.760	0.769	indium antimonide
Ir	22.400	0.129	iridium
K	0.860	10.189	potassium
KBr	2.750	1.893	potassium bromide
KCl	1.980	2.050	potassium chloride
KF	2.480	*1.000	potassium fluoride
KI	3.128	2.077	potassium iodide
La	6.170	0.920	lanthanum
La <sub>2</sub> O <sub>3</sub>	6.510	*1.000	lanthanum oxide
LaB <sub>6</sub>	2.610	*1.000	lanthanum boride
LaF <sub>3</sub>	5.940	*1.000	lanthanum fluoride
Li	0.530	5.900	lithium
LiBr	3.470	1.230	lithium bromide
LiF	2.638	0.778	lithium fluoride

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
LiNbO <sub>3</sub>	4.700	0.463	lithium niobate
Lu	9.840	*1.000	lutetium
Mg	1.740	1.610	magnesium
MgAl <sub>2</sub> O <sub>4</sub>	3.600	*1.000	magnesium aluminate
MgAl <sub>2</sub> O <sub>6</sub>	8.000	*1.000	spinel
MgF <sub>2</sub>	3.180	0.637	magnesium fluoride
MgO	3.580	0.411	magnesium oxide
Mn	7.200	0.377	manganese
MnO	5.390	0.467	manganese oxide
MnS	3.990	0.940	manganese (II) sulfide
Mo	10.200	0.257	molybdenum
Mo <sub>2</sub> C	9.180	*1.000	molybdenum carbide
MoB <sub>2</sub>	7.120	*1.000	molybdenum boride
MoO <sub>3</sub>	4.700	*1.000	molybdenum trioxide
MoS <sub>2</sub>	4.800	*1.000	molybdenum disulfide
Na	0.970	4.800	sodium
Na <sub>3</sub> AlF <sub>6</sub>	2.900	*1.000	cryolite
Na <sub>5</sub> Al <sub>3</sub> F <sub>14</sub>	2.900	*1.000	chiolite
NaBr	3.200	*1.000	sodium bromide
NaCl	2.170	1.570	sodium chloride
NaClO <sub>3</sub>	2.164	1.565	sodium chlorate
NaF	2.558	1.645	sodium fluoride
NaNO <sub>3</sub>	2.270	1.194	sodium nitrate
Nb	8.578	0.492	niobium (columbium)
Nb <sub>2</sub> O <sub>3</sub>	7.500	*1.000	niobium trioxide
Nb <sub>2</sub> O <sub>5</sub>	4.470	*1.000	niobium (V) oxide
NbB <sub>2</sub>	6.970	*1.000	niobium boride
NbC	7.820	*1.000	niobium carbide
NbN	8.400	*1.000	niobium nitride
Nd	7.000	*1.000	neodymium
Nd <sub>2</sub> O <sub>3</sub>	7.240	*1.000	neodymium oxide
NdF <sub>3</sub>	6.506	*1.000	neodymium fluoride
Ni	8.910	0.331	nickel

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
NiCr	8.500	*1.000	nichrome
NiCrFe	8.500	*1.000	inconel
NiFe	8.700	*1.000	permalloy
NiFeMo	8.900	*1.000	supermalloy
NiO	7.450	*1.000	nickel oxide
P <sub>3</sub> N <sub>5</sub>	2.510	*1.000	phosphorus nitride
Pb	11.300	1.130	lead
PbCl <sub>2</sub>	5.850	*1.000	lead chloride
PbF <sub>2</sub>	8.240	0.661	lead fluoride
PbO	9.530	*1.000	lead oxide
PbS	7.500	0.566	lead sulfide
PbSe	8.100	*1.000	lead selenide
PbSnO <sub>3</sub>	8.100	*1.000	lead stannate
PbTe	8.160	0.651	lead telluride
Pd	12.038	0.357	palladium
PdO	8.310	*1.000	palladium oxide
Po	9.400	*1.000	polonium
Pr	6.780	*1.000	praseodymium
Pr <sub>2</sub> O <sub>3</sub>	6.880	*1.000	praseodymium oxide
Pt	21.400	0.245	platinum
PtO <sub>2</sub>	10.200	*1.000	platinum oxide
Ra	5.000	*1.000	radium
Rb	1.530	2.540	rubidium
RbI	3.550	*1.000	rubidium iodide
Re	21.040	0.150	rhenium
Rh	12.410	0.210	rhodium
Ru	12.362	0.182	ruthenium
S <sub>8</sub>	2.070	2.290	sulphur
Sb	6.620	0.768	antimony
Sb <sub>2</sub> O <sub>3</sub>	5.200	*1.000	antimony trioxide
Sb <sub>2</sub> S <sub>3</sub>	4.640	*1.000	antimony trisulfide
Sc	3.000	0.910	scandium
Sc <sub>2</sub> O <sub>3</sub>	3.860	*1.000	scandium oxide

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
Se	4.810	0.864	selenium
Si	2.320	0.712	silicon
Si <sub>3</sub> N <sub>4</sub>	3.440	*1.000	silicon nitride
SiC	3.220	*1.000	silicon carbide
SiO	2.130	0.870	silicon (ii) oxide
SiO <sub>2</sub>	2.648	1.000	silicon dioxide
Sm	7.540	0.890	samarium
Sm <sub>2</sub> O <sub>3</sub>	7.430	*1.000	samarium oxide
Sn	7.300	0.724	tin
SnO <sub>2</sub>	6.950	*1.000	tin oxide
SnS	5.080	*1.000	tin sulfide
SnSe	6.180	*1.000	tin selenide
SnTe	6.440	*1.000	tin telluride
Sr	2.600	*1.000	strontium
SrF <sub>2</sub>	4.277	0.727	strontium fluoroide
SrO	4.990	0.517	strontium oxide
Ta	16.600	0.262	tantalum
Ta <sub>2</sub> O <sub>5</sub>	8.200	0.300	tantalum (v) oxide
TaB <sub>2</sub>	11.150	*1.000	tantalum boride
TaC	13.900	*1.000	tantalum carbide
TaN	16.300	*1.000	tantalum nitride
Tb	8.270	0.660	terbium
Tc	11.500	*1.000	technetium
Te	6.250	0.900	tellurium
TeO <sub>2</sub>	5.990	0.862	tellurium oxide
Th	11.694	0.484	thorium
ThF <sub>4</sub>	6.320	*1.000	thorium.(IV) fluoride
ThO <sub>2</sub>	9.860	0.284	thorium dioxide
ThOF <sub>2</sub>	9.100	*1.000	thorium oxyfluoride
Ti	4.500	0.628	titanium
Ti <sub>2</sub> O <sub>3</sub>	4.600	*1.000	titanium sesquioxide
TiB <sub>2</sub>	4.500	*1.000	titanium boride
TiC	4.930	*1.000	titanium carbide

Table A-1 Material Table (continued)

Formula	Density	Z-Ratio	Material Name
TiN	5.430	*1.000	titanium nitride
TiO	4.900	*1.000	titanium oxide
TiO <sub>2</sub>	4.260	0.400	titanium (iv) oxide
Tl	11.850	1.550	thallium
TlBr	7.560	*1.000	thallium bromide
TlCl	7.000	*1.000	thallium chloride
TlI	7.090	*1.000	thallium iodide (b)
U	19.050	0.238	uranium
U <sub>3</sub> O <sub>8</sub>	8.300	*1.000	tri uranium octoxide
U <sub>4</sub> O <sub>9</sub>	10.969	0.348	uranium oxide
UO <sub>2</sub>	10.970	0.286	uranium dioxide
V	5.960	0.530	vanadium
V <sub>2</sub> O <sub>5</sub>	3.360	*1.000	vanadium pentoxide
VB <sub>2</sub>	5.100	*1.000	vanadium boride
VC	5.770	*1.000	vanadium carbide
VN	6.130	*1.000	vanadium nitride
VO <sub>2</sub>	4.340	*1.000	vanadium dioxide
W	19.300	0.163	tungsten
WB <sub>2</sub>	10.770	*1.000	tungsten boride
WC	15.600	0.151	tungsten carbide
WO <sub>3</sub>	7.160	*1.000	tungsten trioxide
WS <sub>2</sub>	7.500	*1.000	tungsten disulphide
WSi <sub>2</sub>	9.400	*1.000	tungsten silicide
Y	4.340	0.835	yttrium
Y <sub>2</sub> O <sub>3</sub>	5.010	*1.000	yttrium oxide
Yb	6.980	1.130	ytterbium
Yb <sub>2</sub> O <sub>3</sub>	9.170	*1.000	ytterbium oxide
Zn	7.040	0.514	zinc
Zn <sub>3</sub> Sb <sub>2</sub>	6.300	*1.000	zinc antimonide
ZnF <sub>2</sub>	4.950	*1.000	zinc fluoride
ZnO	5.610	0.556	zinc oxide
ZnS	4.090	0.775	zinc sulfide
ZnSe	5.260	0.722	zinc selenide

*Table A-1 Material Table (continued)*

<b>Formula</b>	<b>Density</b>	<b>Z-Ratio</b>	<b>Material Name</b>
ZnTe	6.340	0.770	zinc telluride
Zr	6.490	0.600	zirconium
ZrB <sub>2</sub>	6.080	*1.000	zirconium boride
ZrC	6.730	0.264	zirconium carbide
ZrN	7.090	*1.000	zirconium nitride
ZrO <sub>2</sub>	5.600	*1.000	zirconium oxide

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