

综合物性测量系统 (PPMS) Dynacool -14T

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PPMS综合介绍--发展



第一代 PPMS



第二代 Ever cool



第三代 Dynacool



❁ 美国 Quantum Design 公司的综合物性测量系统—Physical Property Measurement System (PPMS-14T-DynaCool)是集电、磁、热等多种物性测量能力于一身的现代测量仪器，可在1.9-400 K的温度范围（温控精确度 $\pm 1\%$ ）、磁场从零-14T特斯拉的范围对多种材料的磁学、热学、电学等物性进行高精度的测量，例如，在磁学性质方面可测量磁化强度随温度的变化 $M(T)$ 、磁化强度随磁场的变化 $M(H)$ （磁滞回线）、交直流磁化率、磁各向异性等；在电输运性质方面，可测量交直流电阻率 R_T 、伏安特性 I_V 、霍尔系数 $R(H)$ 等；热学方面进行比热测量等。





- ❁ **温度范围：1.9K-400K 温度精度：1% ， 温度变化率：0.01-12K/ min, 300K -1.9K 降温时间：小于40分钟。**
- ❁ **磁铁：14T纵向磁场 场均匀度：0.01% ， 扫描速度：0.2-100 Oe/sec （14 T 范围内） ， 漏场30cm内小于5Oe**
- ❁ **真空：普通真空， 1 torr, 高真空 10^{-4} torr （比热和热导） 。**

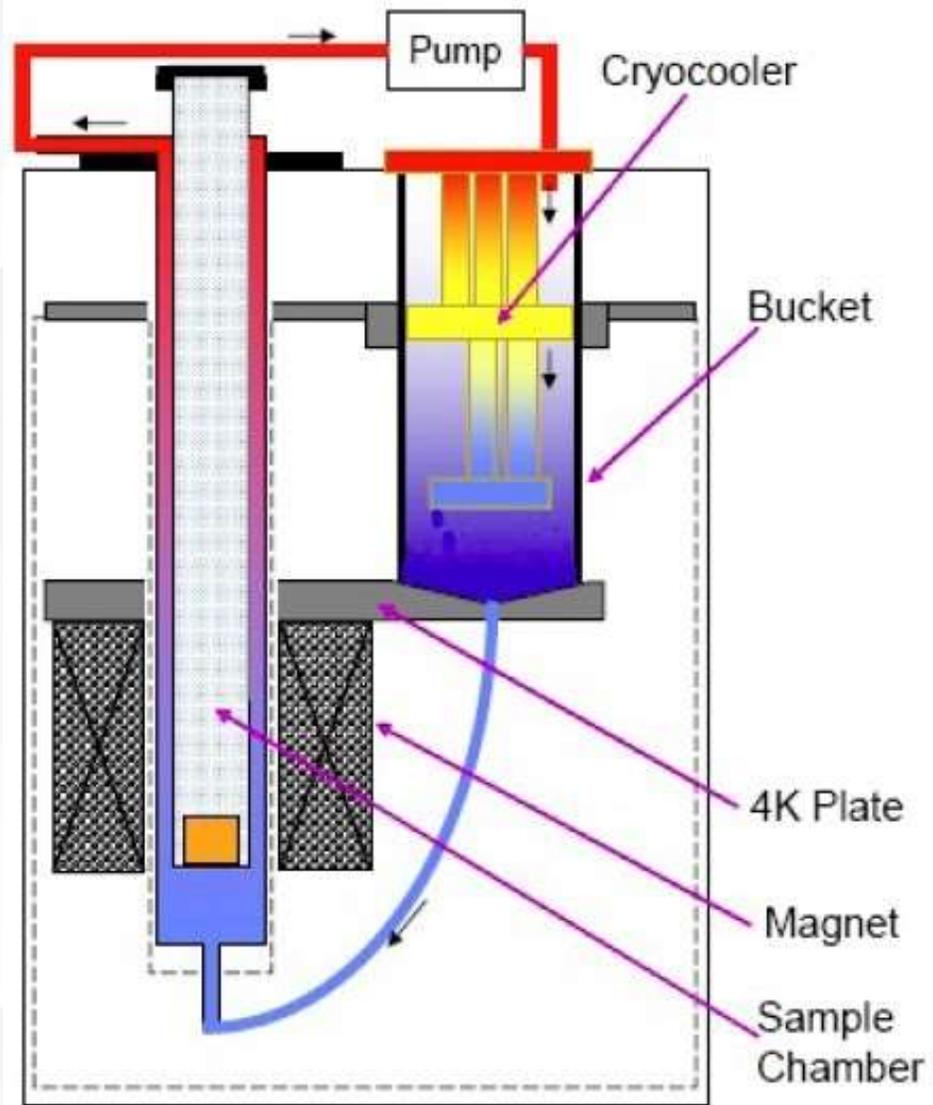


完全无液氦综合物性测量系统

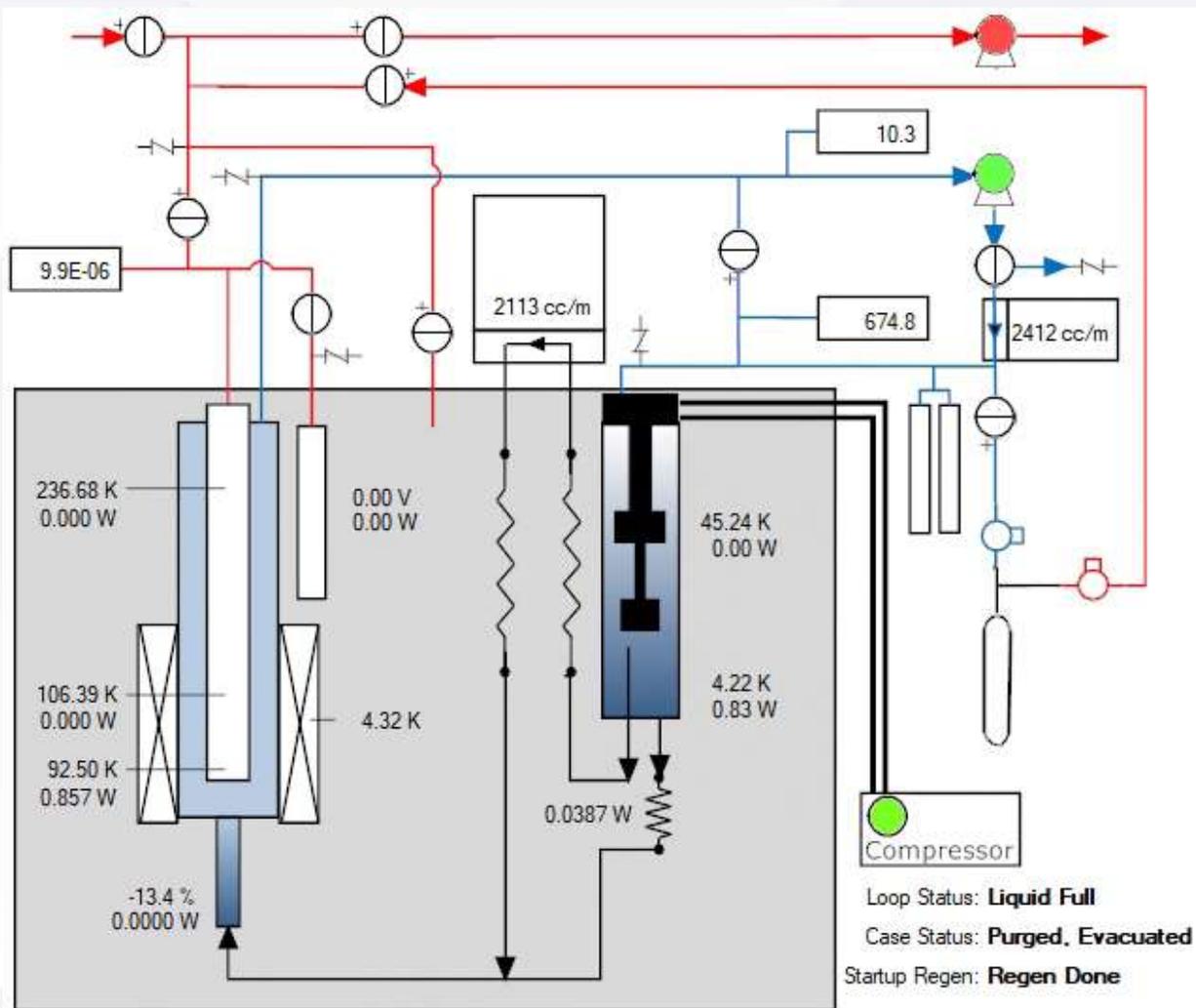
PPMS® DynaCool™



所谓完全无液氦，是说我们不需要准备液氦，但是它的运行还是需要液氦的。开机时，它自带压缩机把氦气压缩成液氦，自己存在一个氦气闭循环系统，内部只有少量的液氦就可以维持低温和实现降温。



DynaCool 将不再需要任何液态制冷剂来维持运行，而是通过一个二级脉冲管式制冷机，同时用于冷却超导磁体和进行低温控制，并且基于脉冲管式制冷机极低振动的特点，使得测量处于一个非常低的振动环境中。

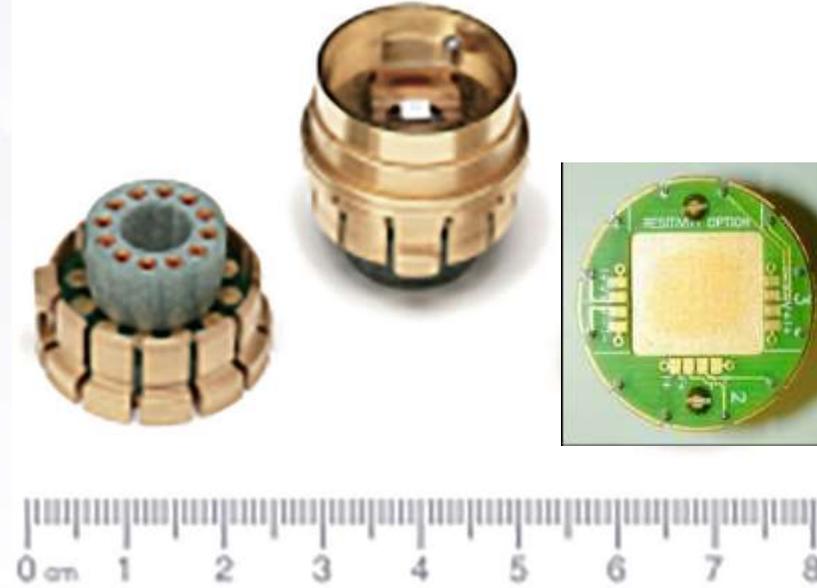
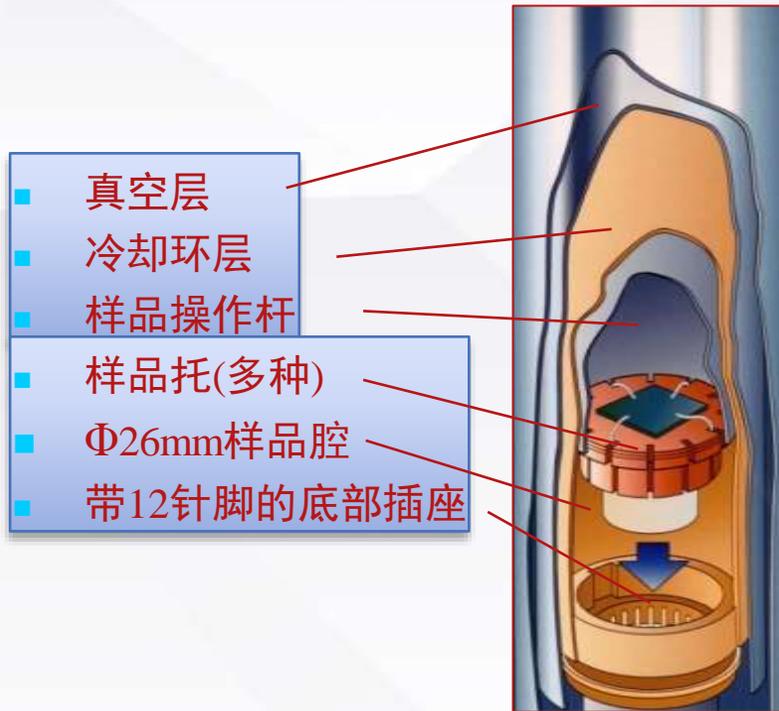


三路气体循环系统：

- 1，红色线为样品腔气体循环，主要是换样的时候对样品腔进行充气，抽气。
- 2，蓝色线为氦气的气液之间的循环，为闭循环，氦气经压缩机压缩为液氦，降温过程中吸热，气化，再压缩。
- 3，黑色为杜瓦内部液氦之间的循环，一部分维持冷头温度，一部分用来降温和维持磁体温度。



PPMS硬件—样品腔



PPMS样品室为内径26mm的圆柱形，底部带有12针脚，用来读取样品上的信号以及系统温度等信息。样品采用专用的样品托来按放样品，样品托底部的12针脚孔余样品室底部的12针脚契合，进行无干扰的数据传输，从而达到高精度测量的要求。不同测量采用不同的样品托，既方便安装样品，也减少了外界环境对样品的影响，让样品的温度更加稳定。

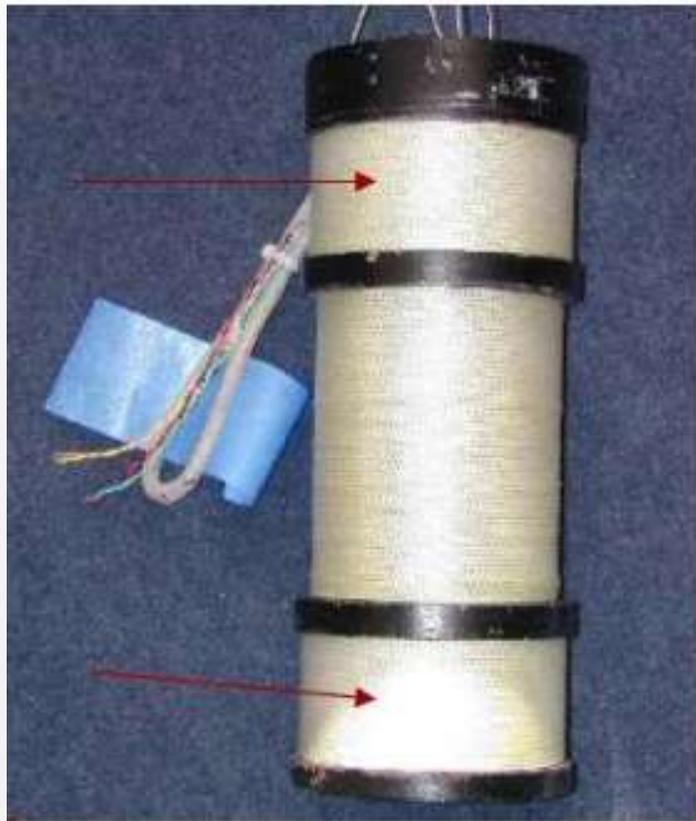
还在样品腔室中上段加装了电热丝，从而解决了以往的低温测量系统所遗留的样品室从低温升温后，样品室颈部仍然处于较低温度的问题。



PPMS附件——取样工具



比热、电阻、磁性测量等，放样取样，专门的送样杆。
一圈钢丝通过上面头上的一个开关控制松紧，样品托上有一圈凹槽，通过搬动开关，来卡住或者松开样品托。



- ❁ 采用超导材料绕成超导线圈，通电流之后产生磁场，利用液氮维持在超导温度下。
- ❁ 磁场方向为上下
- ❁ 磁场变化速度， 100Oe/s



PPMS附件—测试模块

TEMP CTRL,
温度控制模块

ETO Module
高级电输运

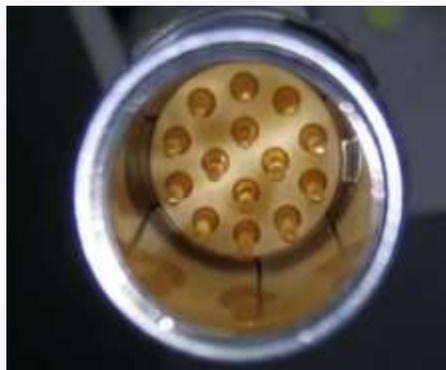
BRT Module
桥式电阻

HC Module
比热模块

Motor Module
振动马达

ACMS Module
交直流磁性测试

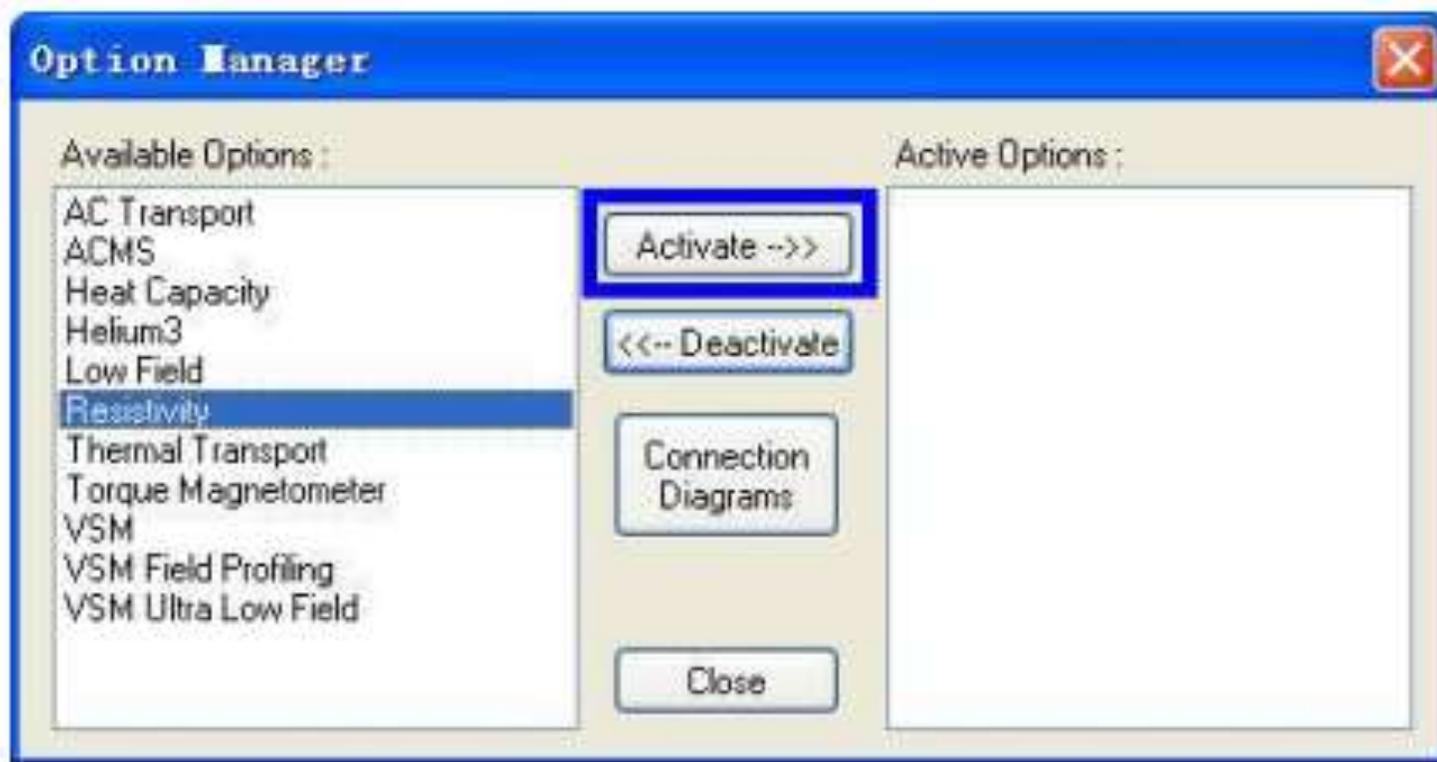




- ❁ 主机与测试模块采用统一的接口，每一种测试都由不同的模块来完成，需要哪个模块就连接这个模块的接头。
- ❁ 软件上进行激活。



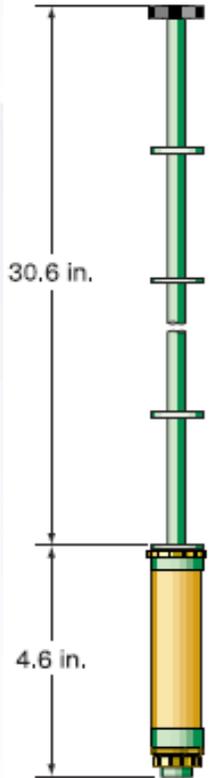
工具栏中点击 Utilities >> Activate Option



注意：插拔接头时 Active option 区域必须为空。



PPMS附件——交/直流磁性测量选件(ACMS)



灵敏度

AC: 2×10^{-8} emu

DC: 2.5×10^{-5} emu

AC驱动频率: 10 Hz – 10 kHz

AC驱动幅值: 0.002 – 17 Oe

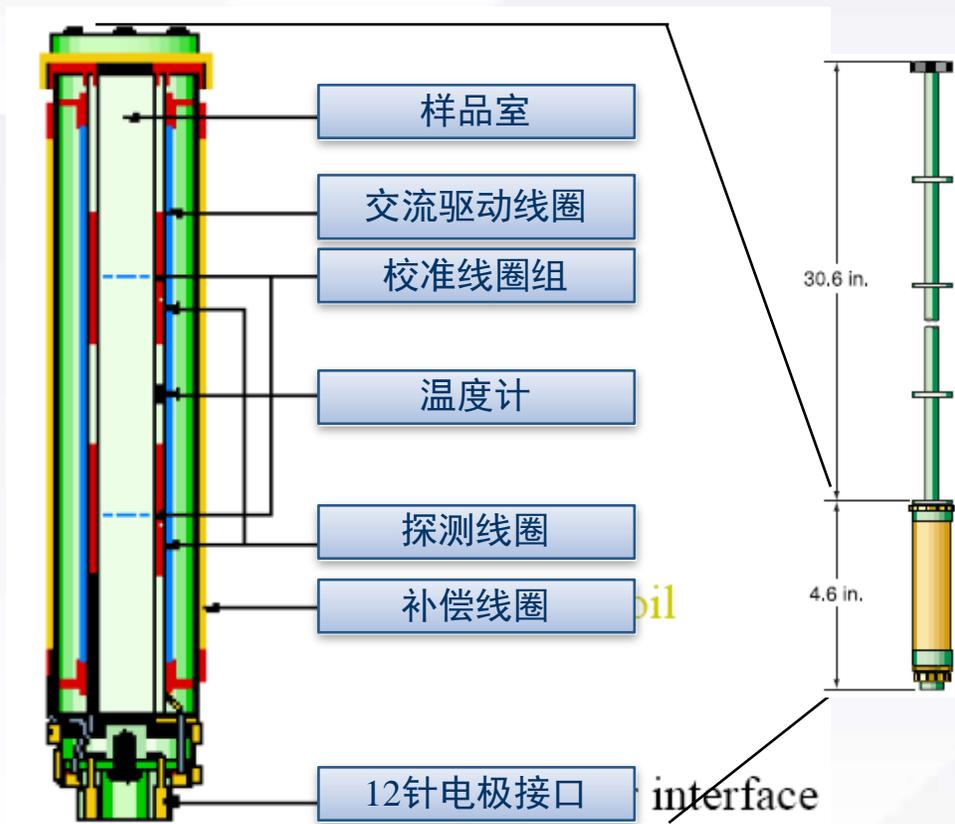
DC提拉速度: 100 cm/s

样品尺寸: $\Phi 3$ mm \times 6 mm

谐波分析: 1 – 10 f



PPMS附件——交/直流磁性测量选件(ACMS)



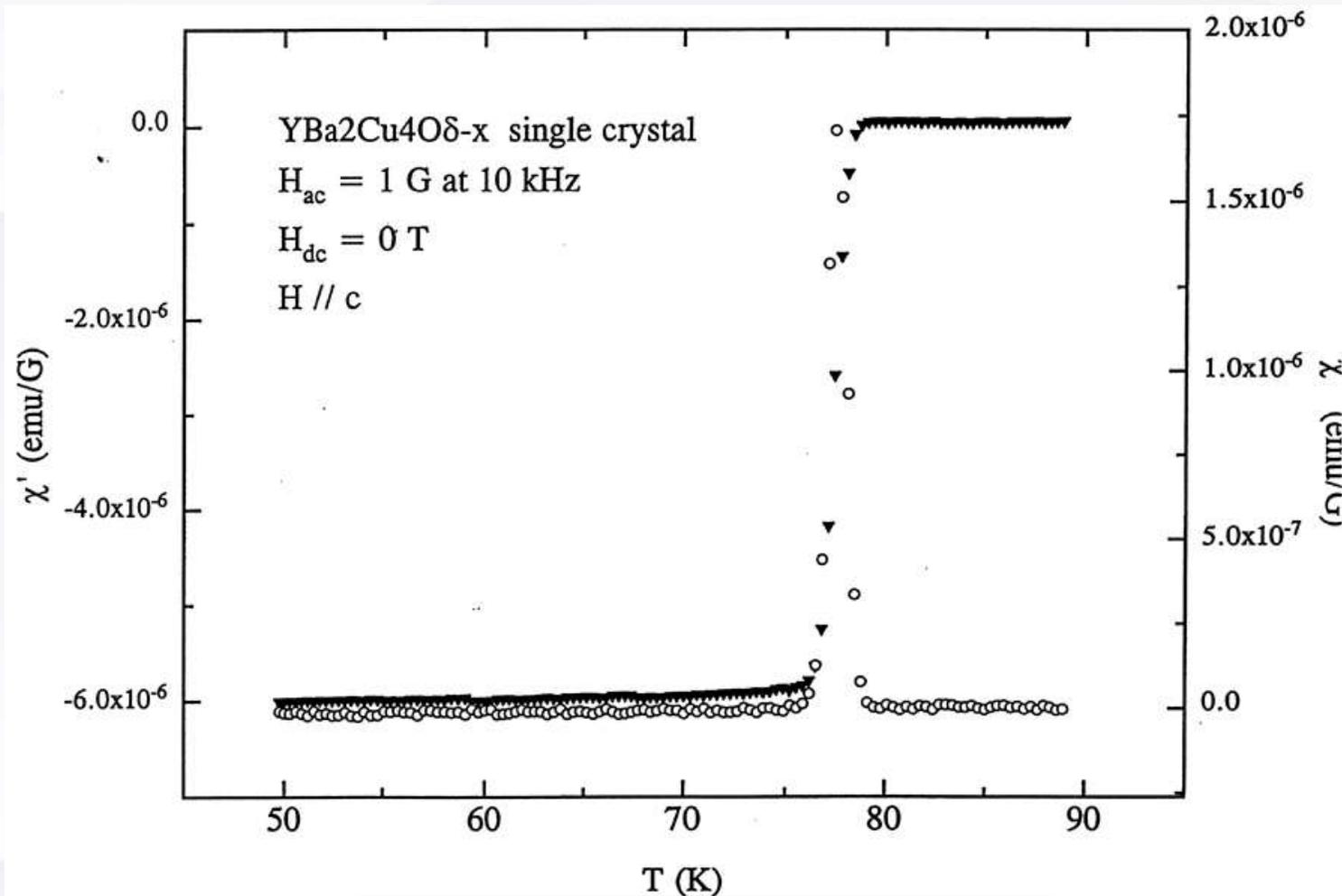
ACMS 探测线圈组



- 可以测量薄膜、块材、单晶
- 使用专用样品安装平台，方便、快捷、易定位



AC磁化率的测量数据



YBCO单晶的AC磁化率测量数据

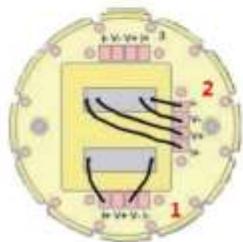




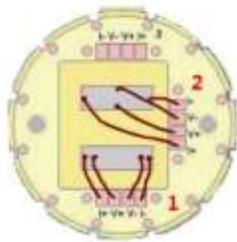
DC Resistivity (BRT)和Electrical Transport Option (ETO)

专用样品托设计，方便快捷，可同时测量多个样品

(1) 样品上做好电极再粘在或焊在 PUCK 的测量通道上（仅 Ch1 和 Ch2 可用），记住通道编号。



Ch1: 两线法电阻(>10MΩ)
Ch2: 四线法电阻(<10MΩ)



Ch1: 四线法电阻(<10MΩ)
Ch2: 四线法 Hall (<10MΩ)

DC的技术参数

- 电流范围：5 nA-5 mA
- 最高电压：95 mV
- 最大测量电阻：4μΩ - 4 MΩ
- 精确测量范围：1Ω - 1 MΩ
- 测量精度：0.01% (典型值)

可测量电阻值为10 nΩ – 5GΩ，可对各种导体、半导体和绝缘体等进行全电阻段的测量，并可进行伏安特性、霍尔效应和微分电阻等测量。

技术参数：

电阻测量范围：四线法 10⁻⁸Ω – 10⁶Ω

二线法 10⁶Ω – 5 × 10⁹Ω

电压范围：± 4.5 V

电流范围：10nA-100mA 持续操作

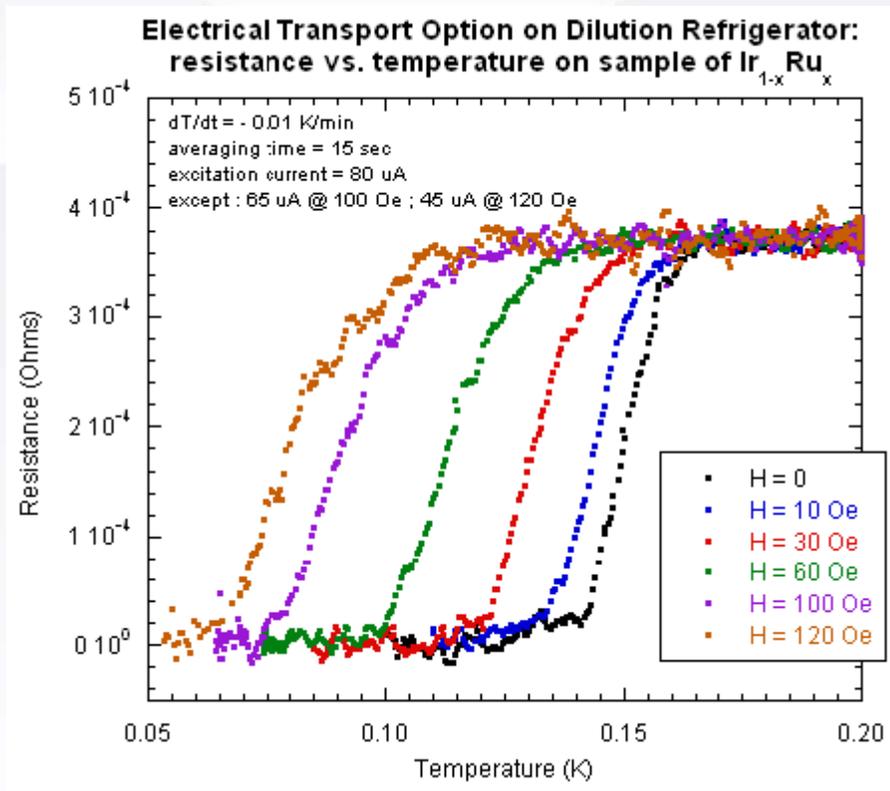
频率范围：直流，交流(0.1Hz-200Hz)

电阻测量精度：0.1% (R < 200 kΩ)

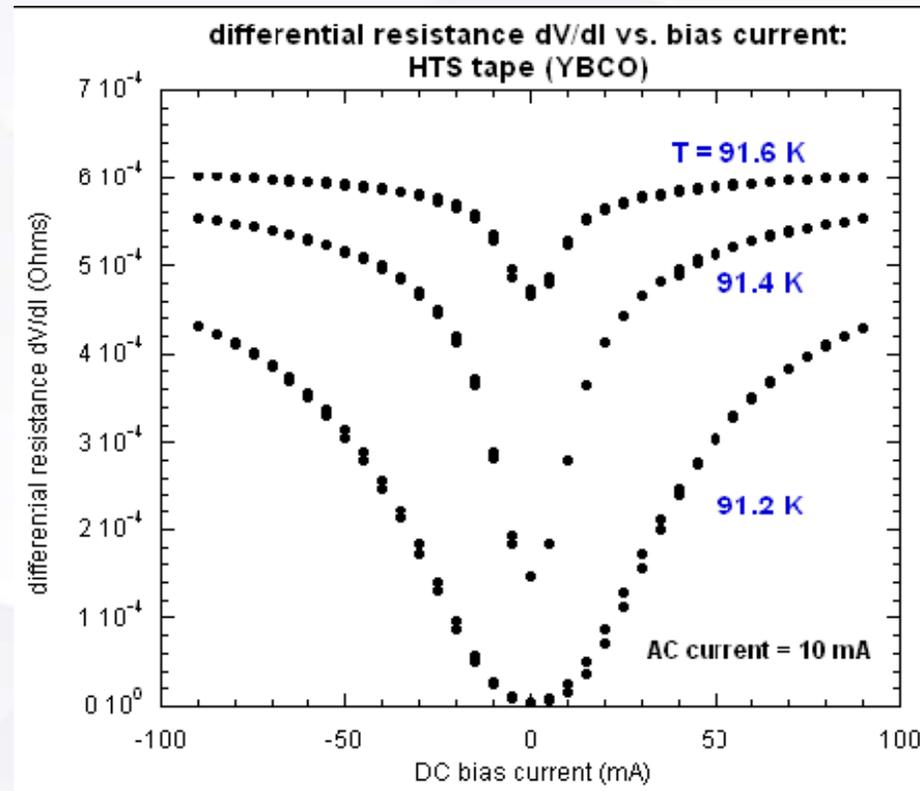
0.2% (R > 200 kΩ)

相对灵敏度：± 10 nΩ RMS (typical)





ETO结合了稀释制冷选件测量样品的超导转变



ETO对高温超导样品进行微分电阻测量





样品要求：小于 $3*3\text{mm}$, $1\text{-}20\text{mg}$,
如果要加场测试，样品磁各向异性弱



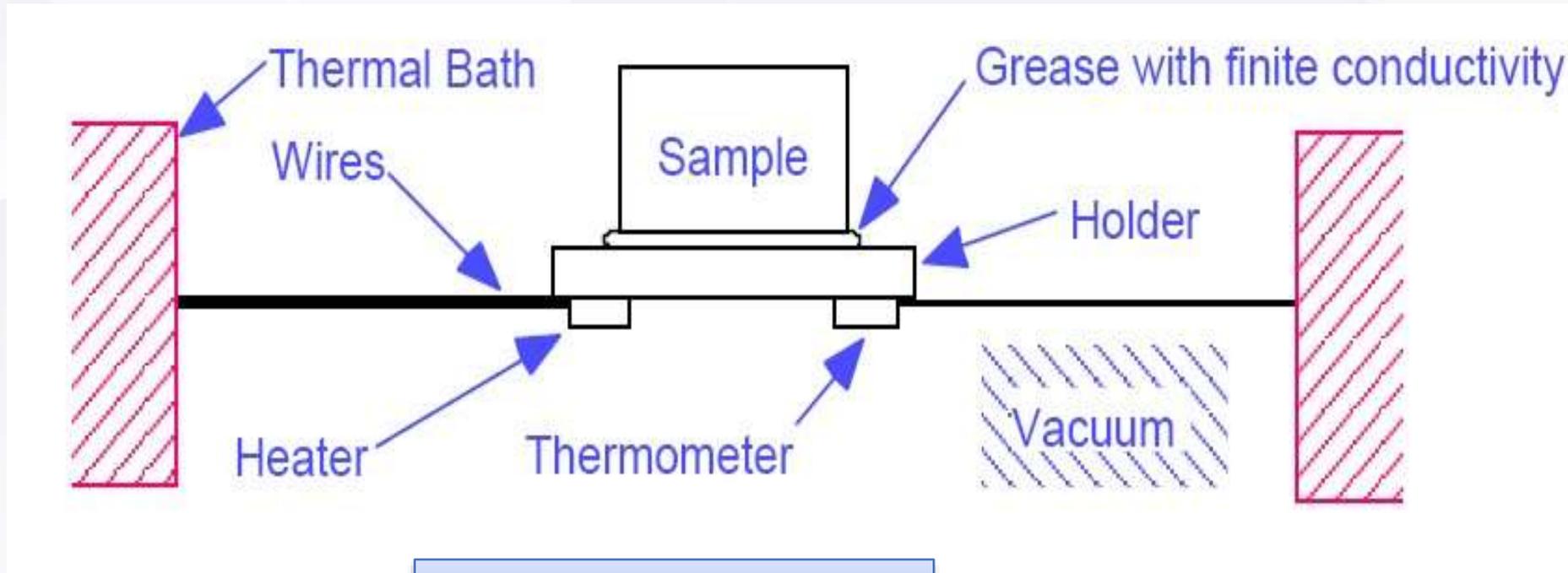
Puck



上样专用夹具



抽气泵



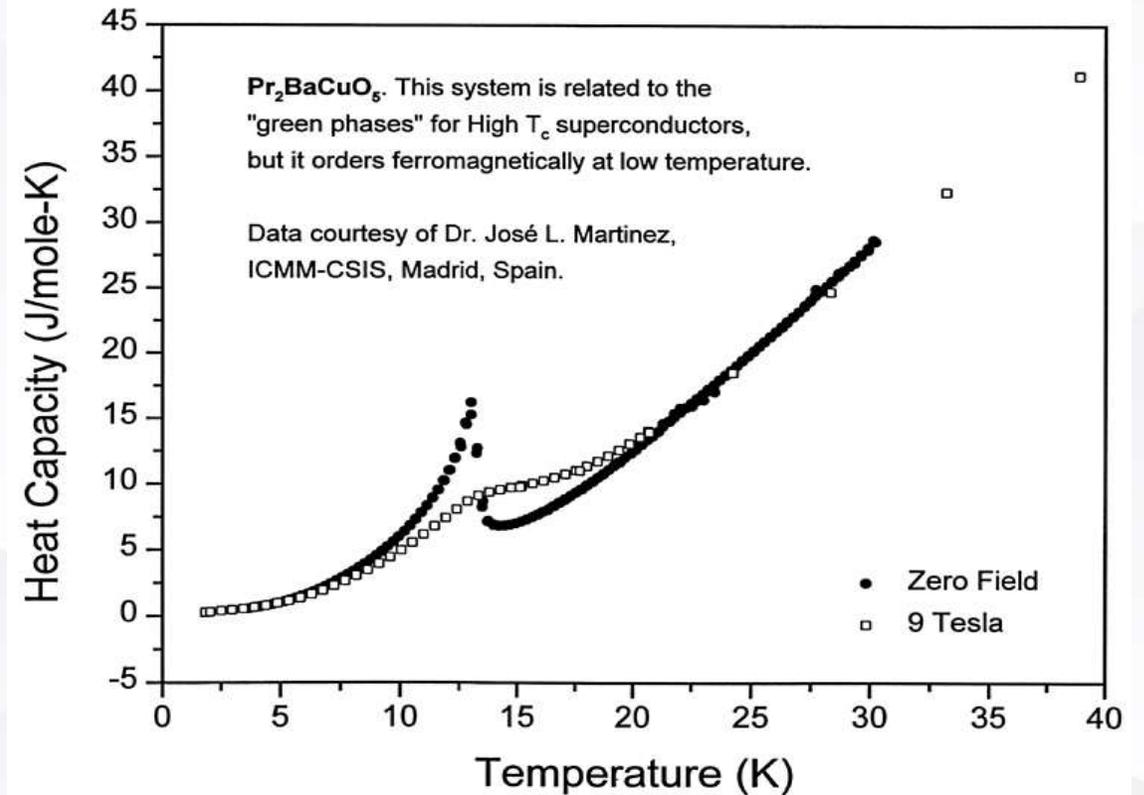
$$C = dQ/dT \approx \Delta Q/\Delta T$$

- 测量样品时背景的比热能够自动减除
- 每个测量点自动计算和记录德拜温度
- 采用专用样品安装工具极大简化了样品的安装
- 需要配合高真空选件(10^{-5} torr)



技术参数

- ◆ 温度范围：1.9-400K
- ◆ 温度控制精度：0.5% @ 2 K
- ◆ 样品尺寸：小于3*3mm,
- ◆ 样品质量：1-200 mg (典型值20 mg)
- ◆ 可测比热大小范围：1 μ J/K – 100 mJ/K
- ◆ 测量灵敏度：10 nJ/K @ 2 K
- ◆ 测量精度：< 5% @ 2 – 300 K (典型值< 2%)
- ◆ 样品腔最小压力： 10^{-5} torr

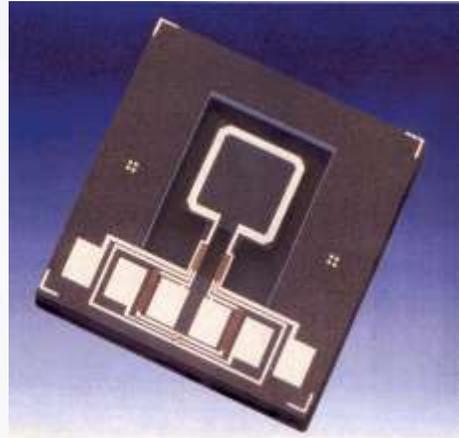


- 与IBM公司共同开发，专用测量具有磁各向异性的小样品 (单晶、薄膜, <10 mg)

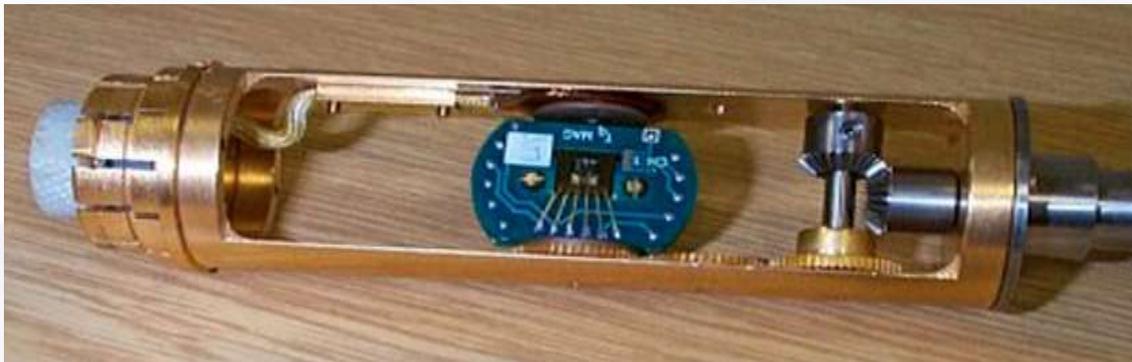
- 测量精度可与SQUID相媲美

- 利用压电传感器测量扭矩

- 用高精密惠斯通电桥测量压电传感器的电阻

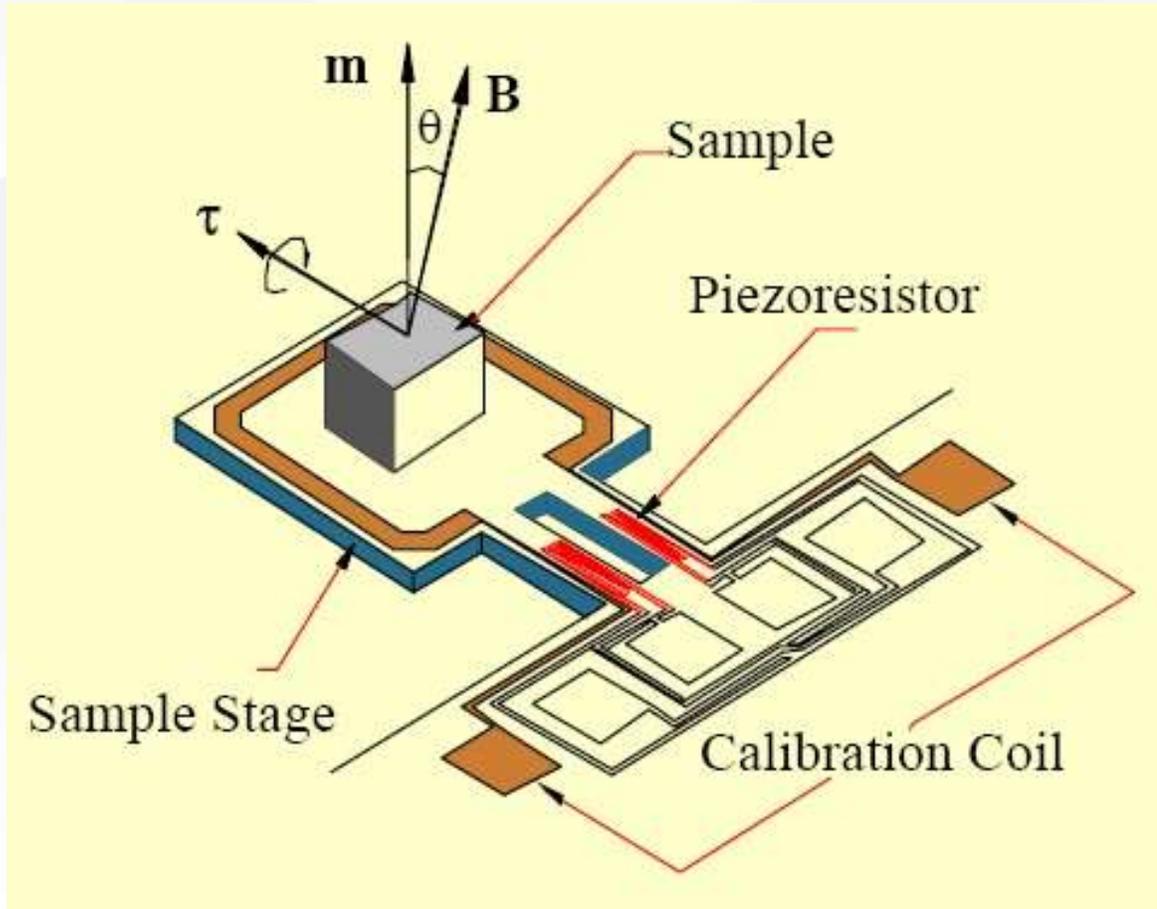


- 灵敏度: $7 \times 10^{-8} \text{emu @ 14 T}$
- 扭矩测量范围: $\pm 10^{-5} \text{Nm}$
- 扭矩背景噪音: $1 \times 10^{-9} \text{Nm}$
- 芯片尺寸: $6 \times 6 \times 1 \text{mm}^3$
- 安装样品区域: $2 \times 2 \text{mm}^2$
- 最大样品尺寸: $1.5 \times 1.5 \times 0.5 \text{mm}^3$
- 最大样品质量: 10 mg





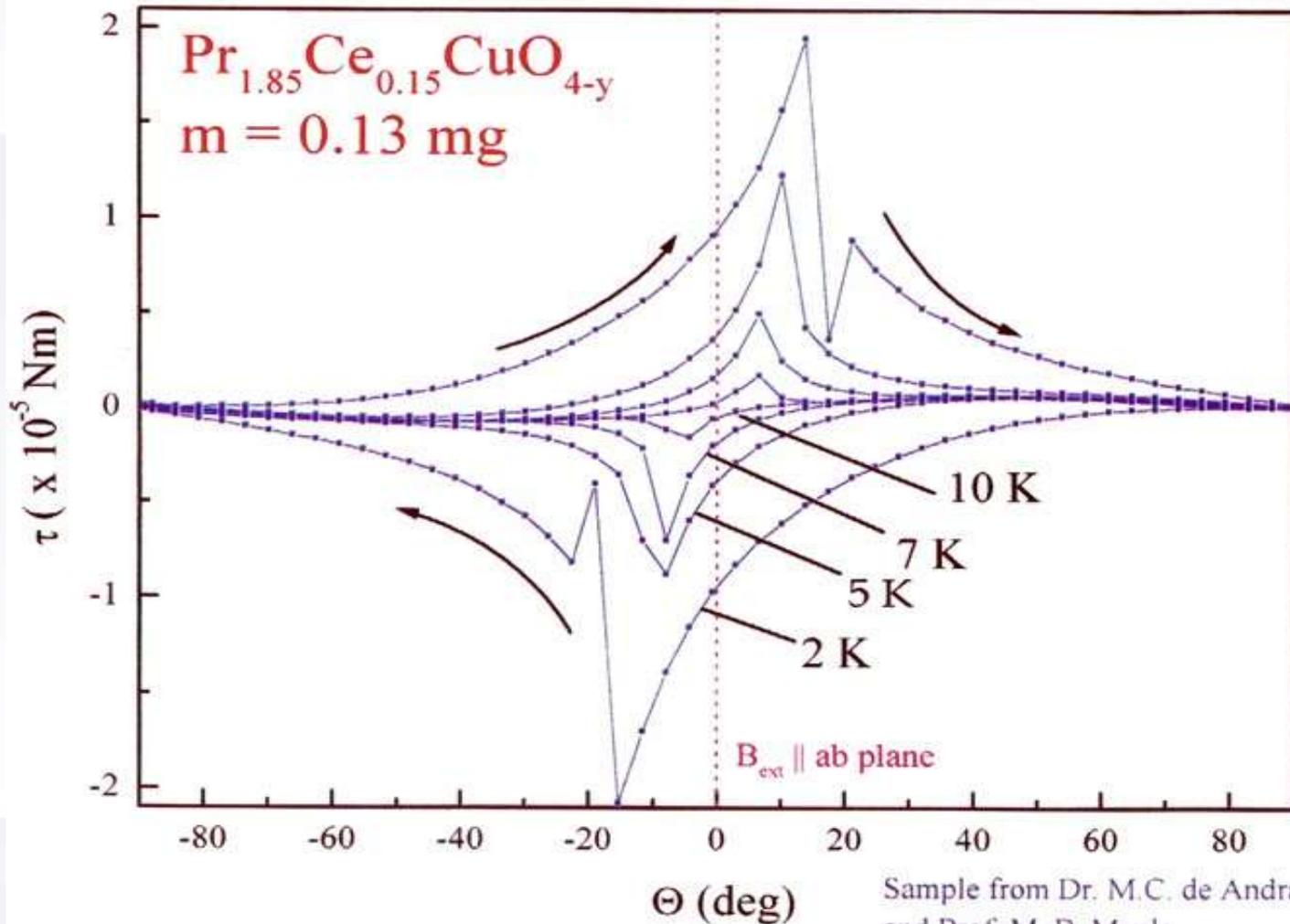
扭矩磁强计选件(Tq Mag)——工作原理



- 仅对非平行于外场的磁矩敏感
- 高场下灵敏度更高
- 集成在扭矩杆上的电流环，产生标准大小的磁矩，用来校准扭矩杆产生扭力的大小。
 - 消除重力作用的影响。
 - 最小化温度对测量的影响



Rotation Magnetization Hysteresis Loops



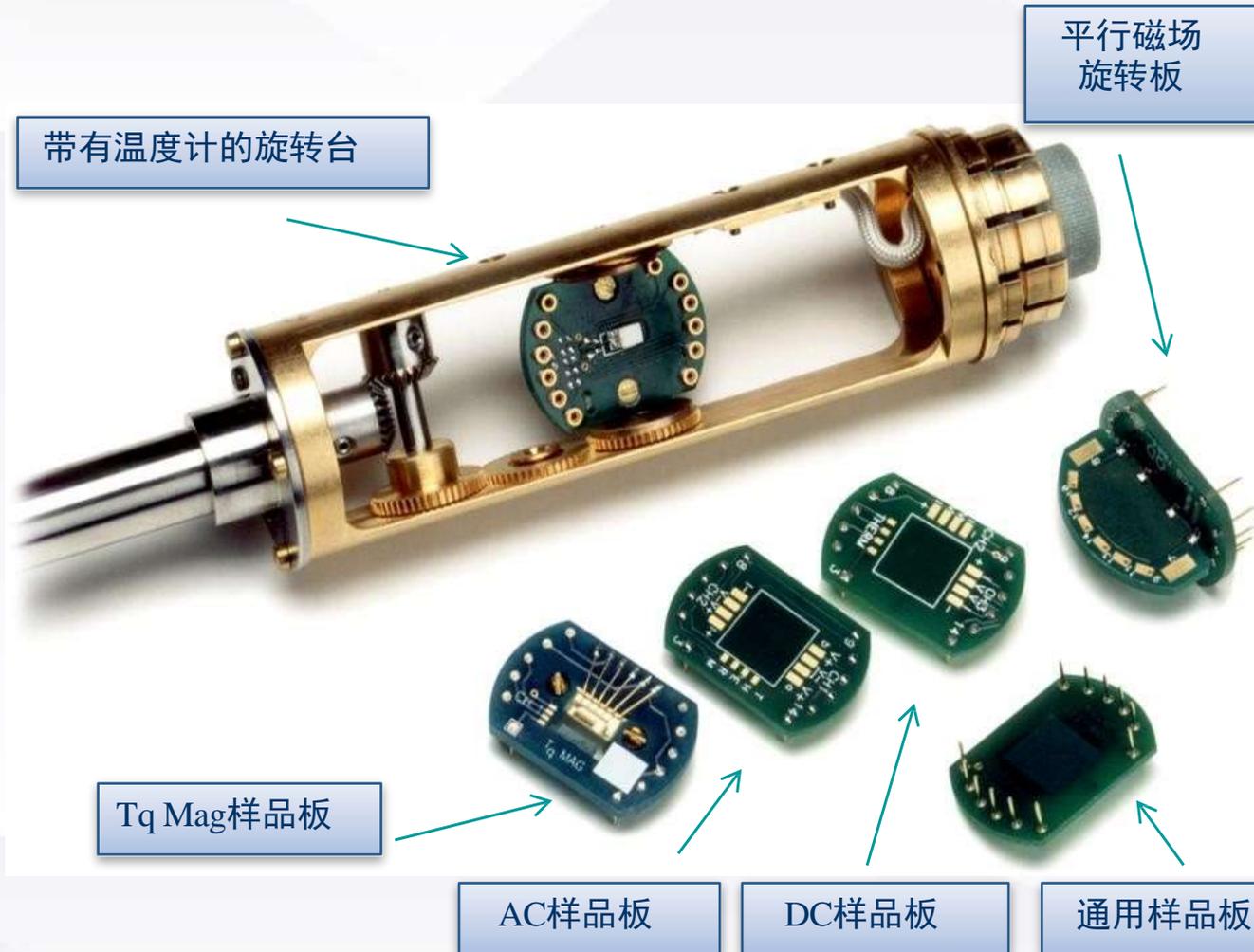
Sample from Dr. M.C. de Andrade
and Prof. M. B. Maple

不同角度下磁扭矩与角度的关系





Horizontal Sample Rotator (通用型和高精度型)



可用于测量与角度相关的电
输运和磁学性质

- AC & DC 电阻系数
- 霍尔效应研究
- I-V 特性
- 临界电流
- 扭矩磁强计
- 转角范围: $-10^{\circ} - 370^{\circ}$
- 转动步长: 0.05° 或 0.0045°
- 转动速度: $10^{\circ}/s$ 或 $1^{\circ}/s$





- 便于实验引进光照、微波以及连在样品上的附加电路引线。
- 样品杆上留有足够的空间让大尺寸电缆穿过，可传输较大的电流或者多路信号线。
- 通过三个轴向端口可以安装光纤、微波导管等。
- 样品安装在即插即用的小支板上。
- 测量杆上集成了校正温度计。
- 可实现铁电、介电、以及激光照射条件下的电输运等测量功能





平台PPMS 能测量的物理性质及样品要求



	PPMS选件	物理性质		样品要求
磁学	ACMS	交/直流磁学性质		块状或大颗粒样品，不超过 2x2x6 mm
	磁扭矩	磁各向异性		块状（形状规则），1-10mg，不大于 1.5*1.5mm
电学	直流电输运	直流电阻	磁电阻、霍尔系数 伏安特性、临界电流	固体薄片状，厚度不超过5 mm, 长宽不超过 10x10 mm（交流输运），长条形比较好。
	高级电输运	交流电阻率 微分电阻		
热学	比热	比热、热容量、磁熵		块状（有一个平面），1-20mg
其他	多功能样品杆 + 第三方设备	铁磁共振、磁电耦合、铁电、介电、光照下电、磁致伸缩等		





The screenshot shows the MultiVu software interface with several components labeled:

- Menu Bar**: Located at the top left, containing File, View, Sample, Sequence, Measure, Graph, System, Utilities, Window, and Help.
- Tool Bar**: Located below the menu bar, containing various icons for file operations and control.
- Control Center**: Located below the tool bar, containing buttons for Run, Pause, Abort, and Lock.
- Sequence Command Bar**: Located at the top right, containing a list of sequence commands such as Beep, Call Sequence or Script, Chamber Operations, Remark, Scan Field, Scan Temperature, Scan Time, Sequence Message, Set Field, Set Temperature, Standby, Wait, and Measurement Commands.
- Sequence Window**: Located in the upper left, showing the selected sequence (Sequence1.seq) and its status (Sequence Idle). It also displays the sequence steps: Selected Line 2, Scan Temp from 1.6K to 400K at 1.2K/min in 2 steps, Uniform, Fast Wait For Temperature, Delay 0 secs, No Action; End Sequence.
- Data Window (graph view)**: Located in the lower left, showing a plot of Resistance (Ohm) vs Temperature (K). The plot shows a red curve with data points and error bars, indicating a transition around 2.5 K.
- Data Window (record view)**: Located in the lower right, showing a table of field names and values. The table is as follows:

	Field Name	Field Value
1	Time Stamp (seconds relch)	5090.361
2	Temperature (K)	3.52983522415161
3	Field (Oe)	39.9323474121094
4	Resistance Ch2 (Ohms)	8.929399E-09
5	Resistance Std. Dev. Ch2 (K)	2.320396E-07
6	Phase Angle Ch2 (deg)	70.64775
7	Frequency Ch2 (Hz)	21.3623
8	Averaging Time Ch2 (s)	4.952011
9	AC Current Ch2 (mA)	100
10	DC Current Ch2 (mA)	0
11	Voltage Ampl. Ch2 (V)	2.6946E-08
12	In Phase Voltage Ampl Ch2 (V)	9.929227E-09
13	Quadrature Voltage Ch2 (V)	2.542352E-08
14	Gain Ch2	90000
15	2nd Harmonic Ch2 (V)	4.029424E-05
16	3rd Harmonic Ch2 (V)	0
17	ETO Status Code	411440512
18	ETO Measurement Mode	0
- Status Bar**: Located at the bottom, showing various system parameters such as Sequence Idle, 246.47 K Stable, 0.11 Oe Hold, 0.00 mTol, Tank: 643 Torr, and others.

Figure 3-2. MultiVu Main Window.



PPMS软件介绍—— Menu

File Edit View Sample Sequence Measure Graph Instrument Utilities Window Help

File Edit View Sample Sequence Measure Graph

- New Sequence Ctrl+N
- Open... Ctrl+O >
- Close
- Save Ctrl+S
- Save As...
- User Configuration...
- Print... Ctrl+P
- Print Preview...
- Print Setup...
- 1 T153MT-20240426.dat
- 2 D:\LinFei Liu\20240423.dat
- 3 T153MT-20240425.dat
- 4 D:\LinFei Liu\...\20240418.dat
- 5 eto_default.dat
- 6 ETO_00001.dat
- 7 D:\LinFei Liu\...\20240422.dat
- 8 D:\Guorui Chen\LZX\ETO-.seq
- Exit

File Edit View Sample Sequ

- Undo Ctrl-Z
- Cut Ctrl-X
- Copy Ctrl-C
- Paste Ctrl-V
- Delete Del
- Enable
- Disable

File Edit View Sample Sequence Measure

- Status Bar >
- Control Center
- Tool Bar
- Sequence Command Bar

File Edit View Sample Sequence M

Install...

File Edit View Sample Sequence Measure Graph

Run

Abort

Pause

Lock

Advanced >

Selected Sequence:
ETO-.seq

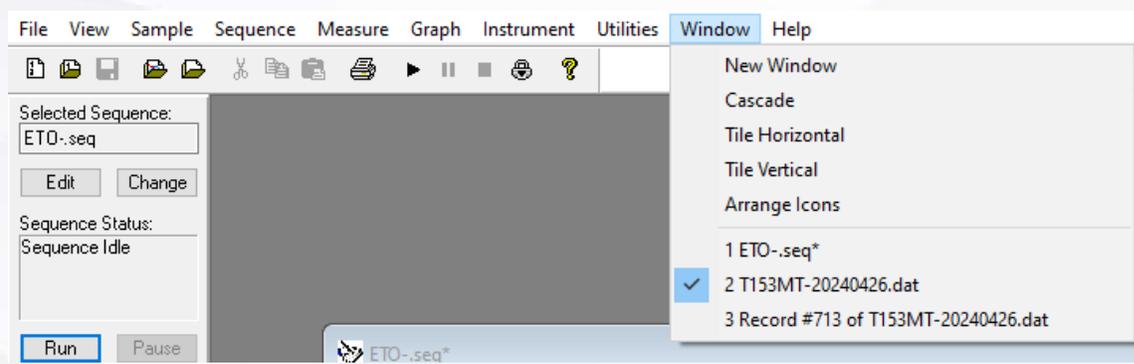
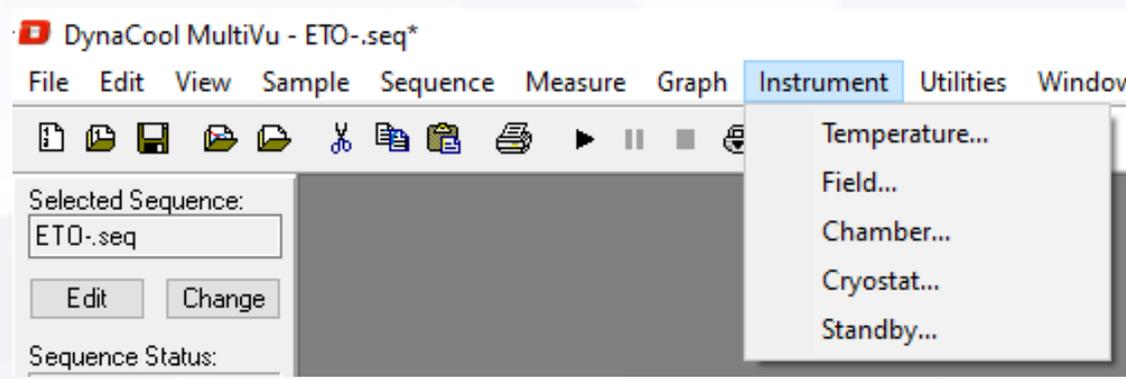
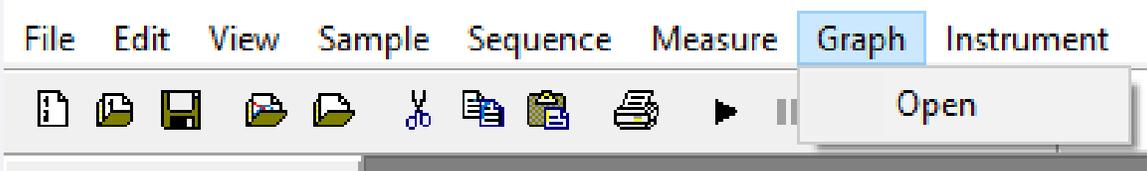
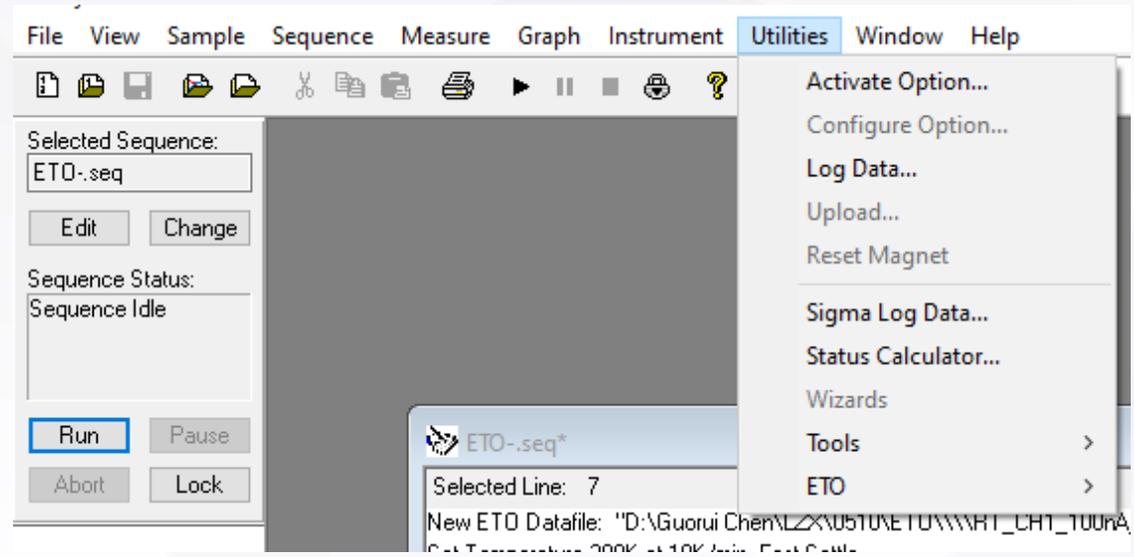
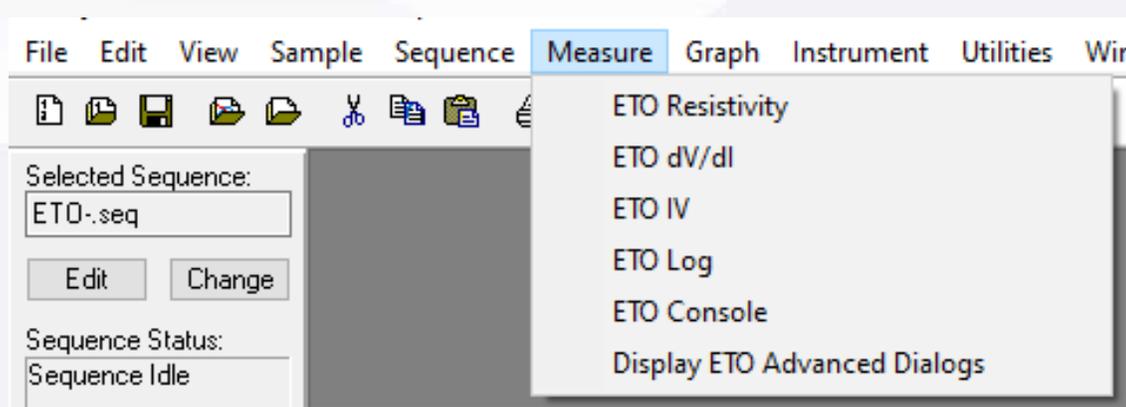
Edit Change

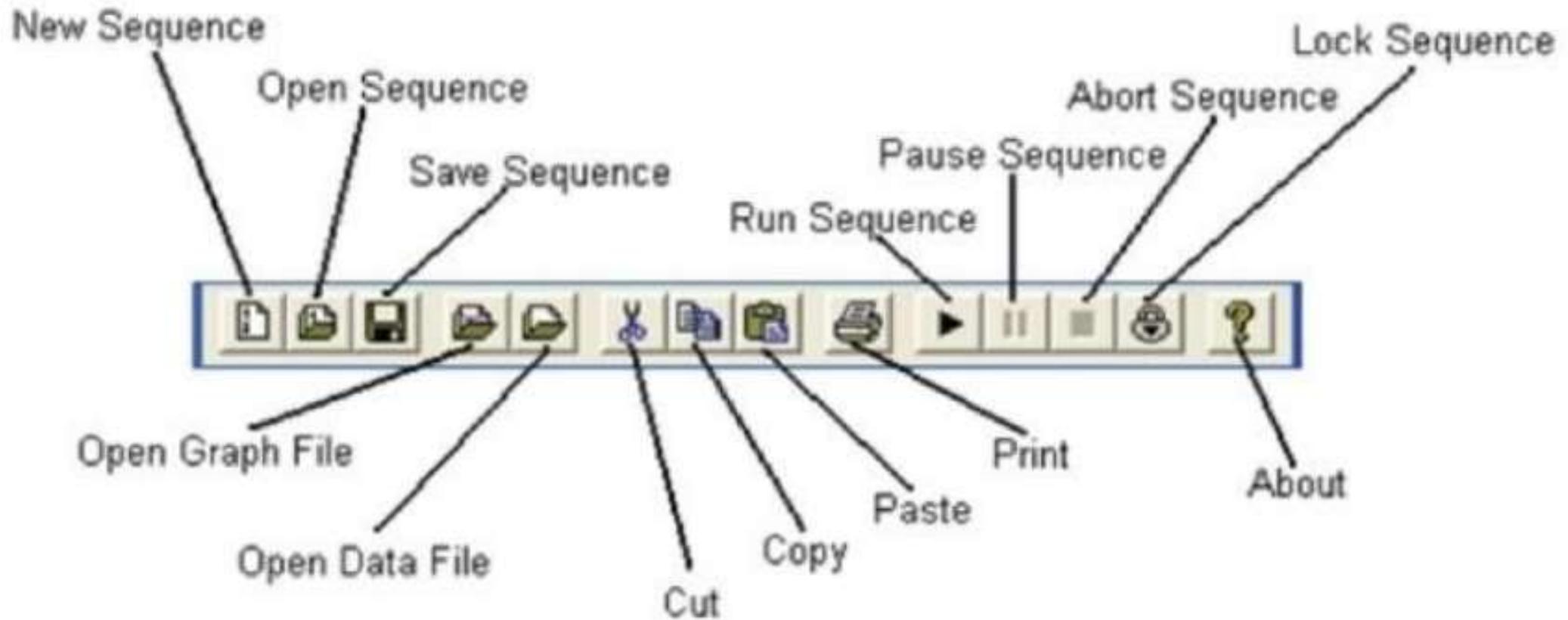
Sequence Status:
Sequence Idle

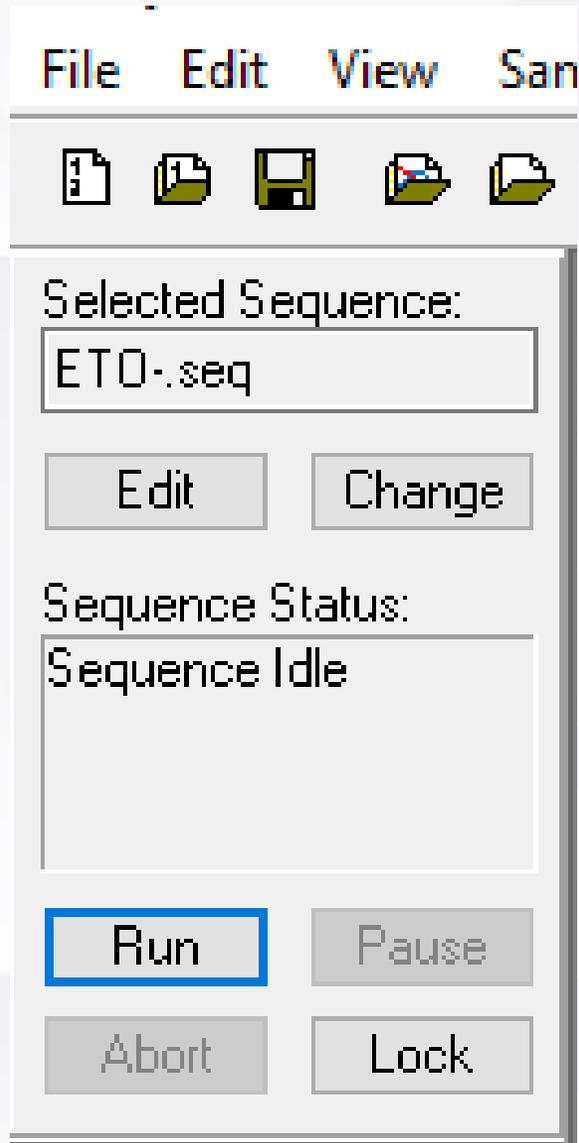




PPMS软件介绍—— Menu







显示目前选中的程序名称和运行状态。

Change按钮用来改变当前程序，还有编辑，运行，暂停，取消和锁定按钮



PPMS软件介绍——状态栏

The screenshot displays the PPMS software interface. It features several control panels:

- Temperature - System:** Shows current Temp (300.00 K) and State (Stable). Control options include Set Point (300.00 K), Rate (5.00 K/min), and Mode (Fast Settle).
- Field:** Shows current Field (-0.30 Oe) and State (Holding). Control options include Set Point (0.00 Oe), Rate (100.00 Oe/sec), and Approach (Oscillate).
- Chamber:** Shows current Pressure (10.0 Torr) and State (Purged). Control buttons include Seal, Purge/Seal, Vent/Seal, Pump Cont., Vent Cont., and HVac.
- Cryostat:** A table showing various parameters: Cryocooler (Running), Magnet (4.32 K, Holding), 4K Plate (4.22 K, 0.84 W), Shield (42.48 K), Loop (673.95 Torr, Circulating, Liquid Full), and Case (Purged).

 A vertical toolbar on the right contains buttons for Refresh Liquid..., System Standby..., Warm Up..., Shutdown..., Restart Cryostat..., and Rapid Shutdown... The status bar at the bottom provides a summary of the system's current state for each component.

Sequence:

- status
- file name
- execution time

Temperature:

- sample temp and status
- setpoint
- set rate and mode

Magnetic Field:

- field and status
- setpoint
- set rate and mode

Sample Chamber:

- pressure
- state

Cryostat and Cryocooler:

- circulation loop pressure
- status
- magnet temp.





Sequence Commands:

System Commands

- Beep
- Call Sequence or Script
- Chamber Operations
- Remark
- Scan Field
- Scan Temperature
- Scan Time
- Sequence Message
- Set Field
- Set Temperature
- Standby
- Wait

Measurement Commands

- Log Data
- Sigma Log Data

Beep – Causes the computer to make the Windows default beep sound.

Call Sequence – Suspends execution of parent sequence file and begins execution of selected child sequence file or script file. When execution of child sequence file or script is complete, execution of parent sequence will continue with the next line.

Chamber Operations – Changes the state of the sample chamber atmosphere (Figure 3-1). See Section 4.5.1 for a description of chamber operations.

Remark – Serves as a message, comment, or visual break for the user only. Does nothing during sequence execution.

Scan Field – Creates a program loop for executing repeated commands at user-defined magnetic field increments. All commands between the “Scan Field...” line in the sequence and the “End Scan” line in the sequence will be repeated at each magnetic field specified by the scan field command. Set the initial and final fields and the scale on which the field increments should appear uniform (linear, H^2 , $H^{1/2}$, $1/H$, $\log(H)$). Also set the total number of field steps. (For uniform linear spacing, you may alternatively set the field increment.) Finally, specify the rate and approach mode used by the magnet controller to achieve each set point:

Scan Temperature – Creates a program loop for executing repeated commands at user-defined temperature increments. All commands between the “Scan Temperature...” line in the sequence and the “End Scan” line in the sequence will be repeated at each temperature specified by the scan temperature command. Set the initial and final temperatures and the scale on which the spacing of the temperature steps should appear uniform (linear, $1/T$, $\log(T)$.) Also set the total number of temperature steps. (For uniform linear spacing, you may alternatively set the temperature increment.) Finally, specify the rate and approach mode used by the temperature controller to achieve each set point:





Sequence Commands:

System Commands

- Beep
- Call Sequence or Script
- Chamber Operations
- Remark
- Scan Field
- Scan Temperature
- Scan Time
- Sequence Message
- Set Field
- Set Temperature
- Standby
- Wait

Measurement Commands

- Log Data
- Sigma Log Data

Scan Time – Creates a program loop for executing repeated commands at user-defined time increments (or immediate repetitions with no time increment.) All commands between the “Scan Time...” line in the sequence and the “End Scan” line in the sequence will be repeated at each time specified by the scan time command. Set the total time in seconds and specify whether the spacing of events should be uniform in time or logarithmic in time. And specify the number of steps. This is the number of times the loop will be repeated. (For uniform spacing in time, you may alternatively specify the time increment.) If the total time is set to zero seconds, then the number of steps defines how many times the loop will be repeated in rapid succession.

Sequence Message – Displays a message on the computer screen and pauses sequence execution until the message is acknowledged or until a timer expires. If the computer is set up with network access and access to a mail server, a message can also be emailed with attachments such as data files.

Set Field – Sets the instrument’s magnetic field. Specify the field, the charging rate, and the approach:

Set Temperature – Sets the sample temperature. Specify the temperature, the rate, and the mode:

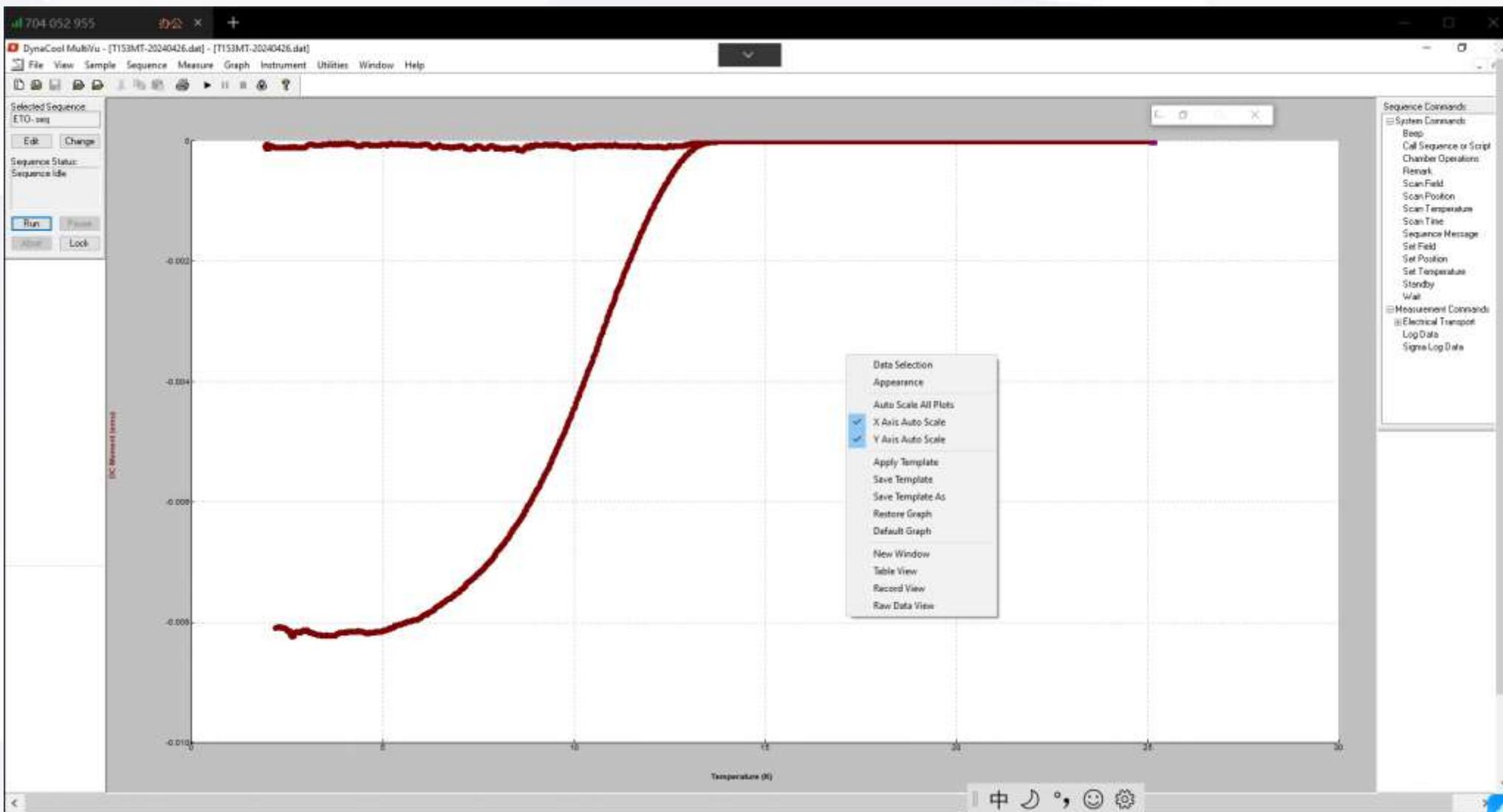
Shutdown – Places the instrument in shutdown mode to conserve resources. The field is set to zero, the magnet controller is turned off, the chamber heaters are turned off, and the chamber is sealed.

Wait – Waits for specified conditions to be achieved, then delays a specified amount of time before continuing with sequence execution. Conditions that can be specified to wait for are temperature stability, field stability, and chamber state. This command is usually used immediately after another command in order to make sure the desired outcome of the first command is achieved before proceeding. For example:





PPMS软件介绍——数据的查看





PPMS软件介绍——数据的查看

Select one X-axis and up to four Y-axes

Specify the data to plot on each axis

Click to apply additional labeling and scaling to axes

Check for logarithmic axis scaling

Select auto scaling for each axis or specify the min. and max. value for each axis

Axis	Log	Data	Auto	Min	Max
X	<input type="checkbox"/>	Magnetic Field (Oe)	<input checked="" type="checkbox"/>	-100000	250000
Y1	<input type="checkbox"/>	Moment (emu)	<input type="checkbox"/>	-0.6	0.8
Y2	<input checked="" type="checkbox"/>	M. Std. Err. (emu)	<input checked="" type="checkbox"/>	1E-010	0.0001
	<input type="checkbox"/>		<input checked="" type="checkbox"/>	0	0
	<input type="checkbox"/>		<input checked="" type="checkbox"/>	0	0

NOTE: Log scales show only positive values

Data Group: All

Show Time In: Minutes Seconds

Time Display: Relative Absolute

Display Range: All Records Last 539 records From # To # 4539 Prev 4539 Next Records

Filter...

OK Apply Cancel

Display time data as absolute time or relative to first data record in data file in minutes or seconds

Limit the data records plotted to the last few records (like a chart recorder) or to a range of specified records

Click to limit plotted data based on certain data values





- ❁ <https://phy-eshare.sjtu.edu.cn/customer/index/index.html>
- ❁ 校内用户登录Jconut账号，既可以送样预约，预约好之后，联系我，18818218860，我来给大家安排测试。
- ❁ 经过培训获得自主操作授权的用户，可预约自主上机操作。
- ❁ 校内：200/小时，
- ❁ 校外：300/小时
- ❁ 按天计费：对校内用户，经培训合格后（培训费另计），可按天计算收费，2000/天。原则上单次连续使用机时不超过5天。院内用户享统一折扣。



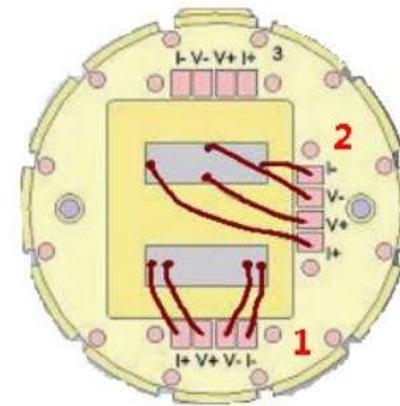
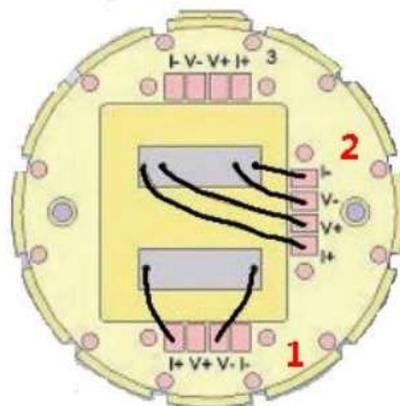


数据的传递

- 通过邮件发送，设备已联网
- 不可通过U盘copy数据。

配件价格

- 磁性铜管，石英杆，1000
- 比热puck，15000
- 电阻puck，8000
- 样品腔，80000



一般要求自己做电极，
平台提供金线，银胶，
四个电极在一条线上
焊点要小

导线应力尽量小，避免降温电极脱落

谢谢!