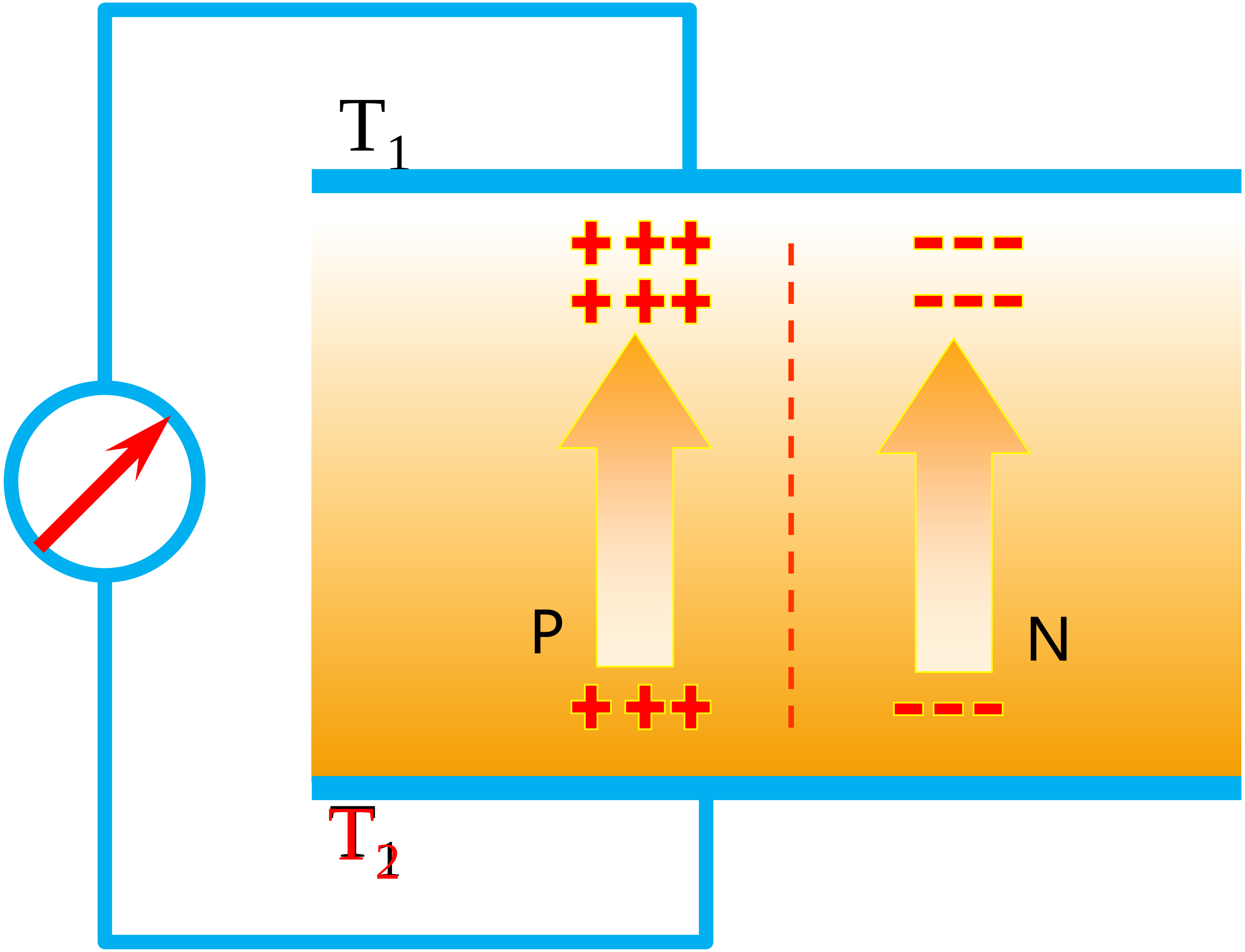
上次课程知识点回顾

- 静电力显微镜（EFM）
  - 原理、应用
  - 频率、相位、振幅调制
- 开尔文探针力显微镜（KPFM）
  - 原理
  - 应用
  - FM-KPFM vs. AM-KPFM
  - PeakForce KPFM
- 磁力显微镜（MFM）
  - 原理
  - 磁探针的选择（磁矩、矫顽场）
  - 应用
- 扫描电容显微镜（SCM）
  - 原理
- 扫描扩散电阻显微镜（SSRM）
  - 原理
- 压电力显微镜（PFM）
  - 原理、应用

视频展示

SThM的基本原理

热电势的产生



Scanning Thermal Microscopy (SThM)

- Scanning Thermal Microscopy (SThM) is an advanced SPM mode intended for simultaneous obtaining nanoscale thermal and topography images.
- SThM is able to visualize temperature and thermal conductivity distribution at the sample surface.

SThM methods

operate by :

• bringing a sharp temperature sensing tip in close proximity to the solid surface to be characterized.

• using the variation of the tip temperature depending on the changes of the heat transfer between the tip and sample surface.

SThM

=

Scanning Probe Microscopy Technique

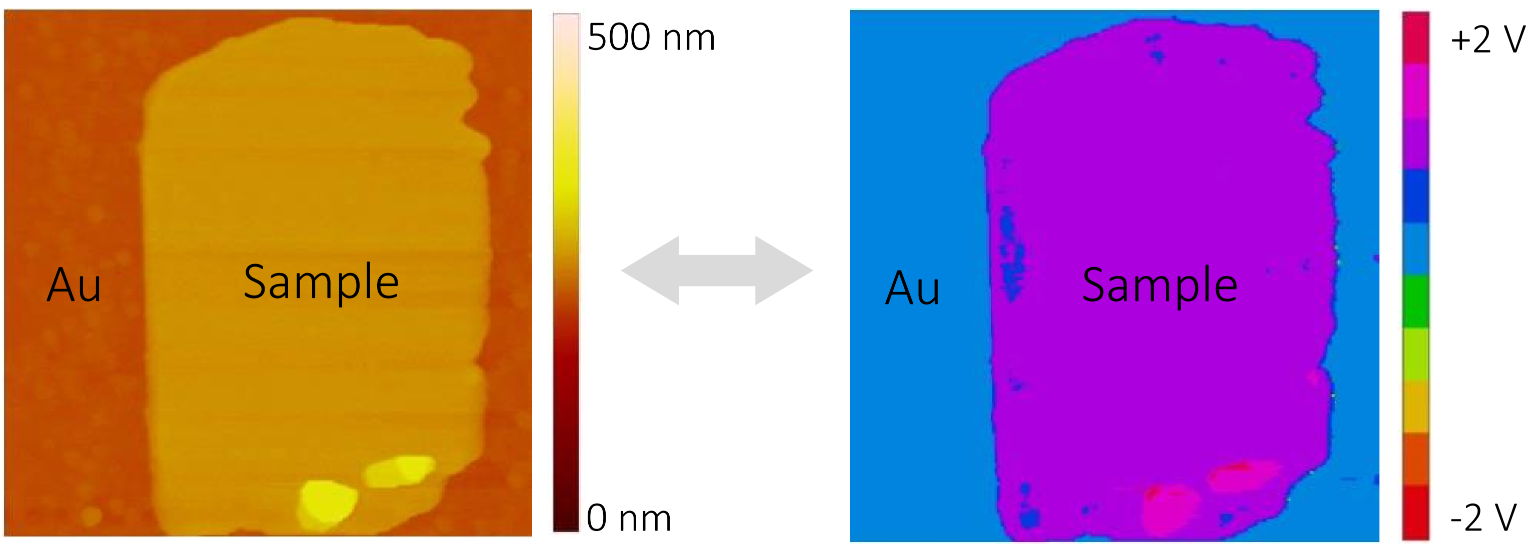
+

Small probe used as thermal sensor

SThM的基本原理

主要工作步骤

- SPM的导电针尖和金属电极接触待测样品两端
- 温度控制和测量系统在样品两极之间施加温差并测量势差 定量研究
- 热电压放大器将样品产生的热电势信号放大并输出



Topography

Thermoelectric voltage

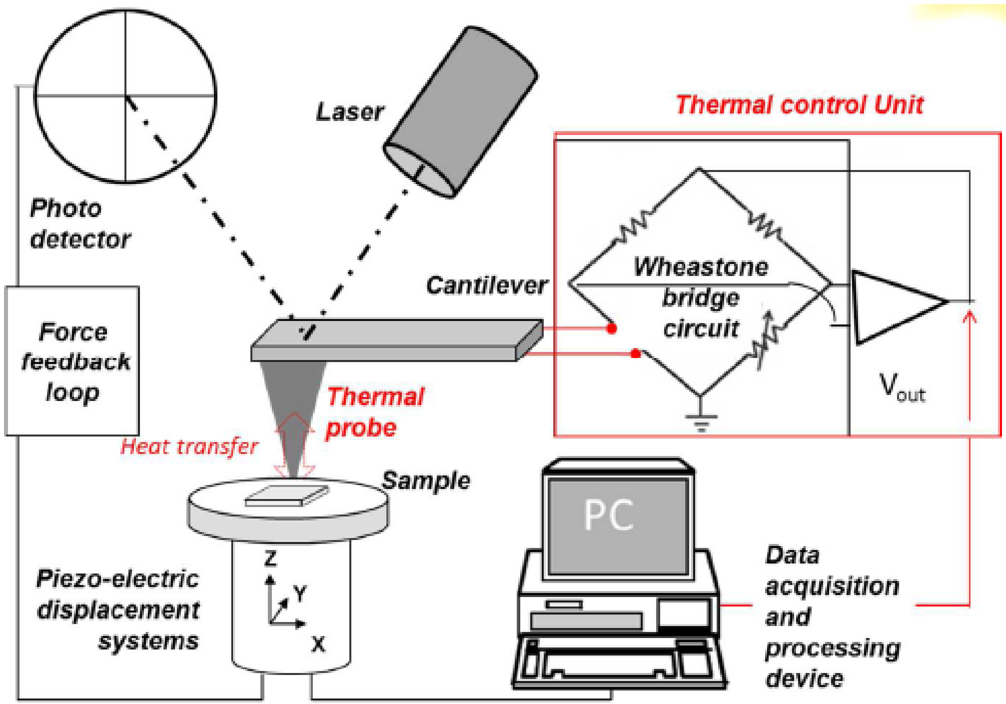
SThM的基本原理

- Observation of all kind of materials

- Able to obtain :

➢ **Force-distance curves**

➢ **Surface topography with the control of sample-probe contact force**



and independently:

➢ **Thermal signal versus probe–sample force & distance**

➢ **Thermal imaging and measurement according to the principle of thermal probe**

SThM的基本原理

Since 1993,
various thermal methods based on the use of different thermosensitive sensors or phenomena

Classification according to the temperature-dependent mechanism used:

- **Thermovoltage:** - Tunneling thermometry and point contact method
  - Built-in thermal sensors: **Thermocouple**
    Schottky diode

- **Change in electrical resistance: Built-in thermal sensors**

- **Fluorescence:** Fluorescent nanocrystal glued at the end of a AFM tip

- **Thermal expansion:** - Bilame effect
  - Measurement of the dilatation of the sample surface induced by its internal or external heating

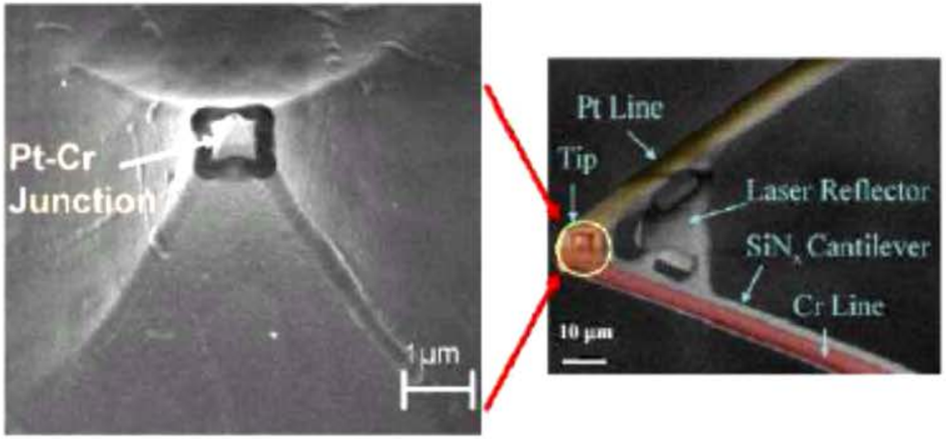
->focus on thermovoltage-based and resistive methods.

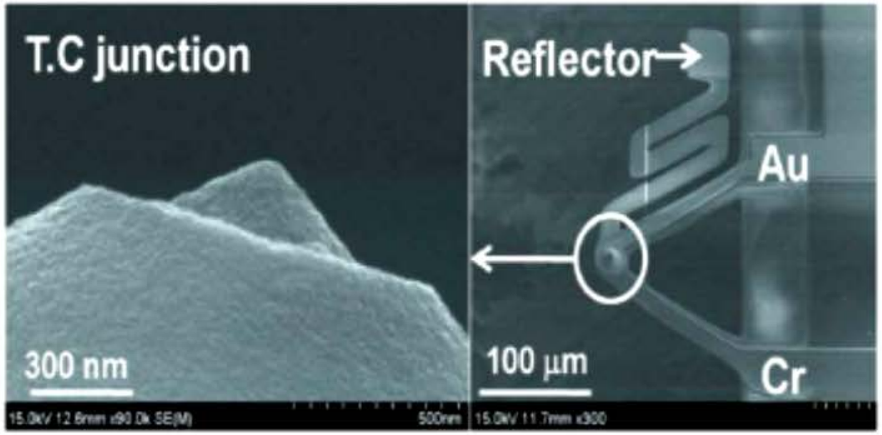
Cantilevers and thermocouple probes

Thermoelectrical voltage =g(T)

Lateral spatial resolution < 50nm

[Shi et al.2001, J Microelectromechanical Sys 10, 370]





Au–Cr thermocouple SThM probe.

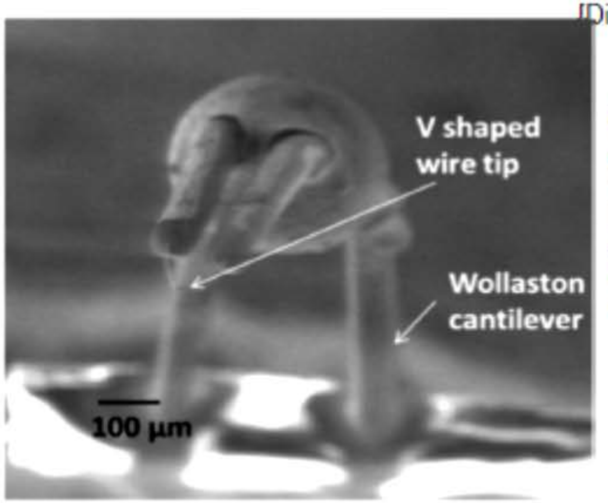
The miniaturization of the cantilever, the tip and the junction at the tip end could lead to a decrease of the probe thermal time constants and to an improvement of the spatial resolution.

[Kim et al. 2008., *Applied Physics Letters* **93**, 203115.]

Cantilevers and resistive probes

Electrical resistance =g(T)

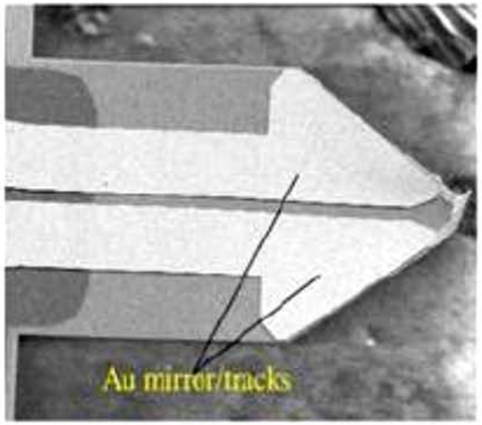
[Dinwiddie et al. E, Thermal Conductivity, vol. 22, 1994]

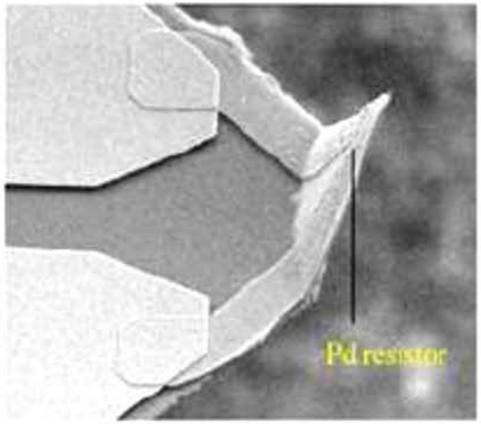


Wollaston cantilever with a resistive Pt/Rh$_{10\%}$ wire probe
Section : 5 µm
Length: 200 µm
V shape - curvature radius : 15-20 µm
Spatial resolution: micrometric

[Kelvin Nanotechnology, Glasgow Univ.]

Thin film Pd resistor located at the tip of a contact mode AFM probe
Cantilever made of Silicon Nitride (Si_3N_4).
Tip radius lower than 100 nm.
Spatial resolution < 80 nm





Operating modes

All resistive metallic probes can be used in two modes

At first order, electrical resistance of the probe resistive element at a temperature T_p :

$$R_p(T_p) = R_p(T_0)[1+\alpha(T_p - T_o)]$$

R_{p0} : electrical resistance of the resistive element at T_0
α : temperature coefficient of its electrical resistivity

Passive Mode for thermometry	Active Mode for thermophysical properties analysis
very small current through the probe **→ minimal Joule self-heating and enables the measurement of the electrical resistance.** During a scan, heat flows from the hot sample to the probe and changes the electrical resistance R_p of the probe	**larger electrical current through the probe** **→ significant Joule heating of probe** **Probe = heat source + thermal sensor** Part of the Joule power flows into the sample, depending on its thermal properties. The probe temperature is monitored by measuring the probe voltage. This temperature can be related to the thermal conductivity of the sample.

Under both modes, dc, ac or both measurements
ac regime → improved signal-to-noise ratio, since lock-in detection is possible.

Operating modes

SThM methods

Enable :

➡ <u>Thermal contrast imaging & measurement</u>

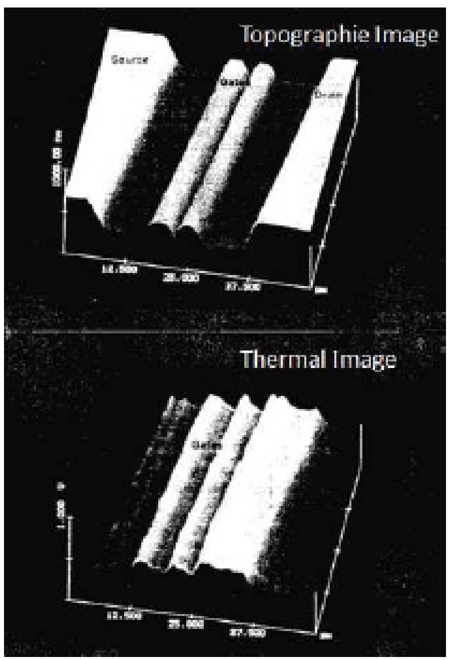
Temperature field (sample surface)

when the tip comes in local equilibrium with the sample

Temperature map of a MESFET

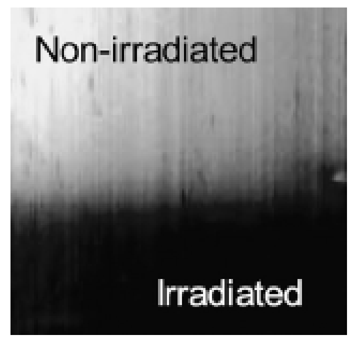
(metal/semiconductor field-effect transistor)

Majumdar et al. -1993



Thermal conductivity contrast

if the temperature change is determined for a known heat flux



SThM image obtained at the level of the interface between irradiated and non-irradiated areas of meso-PSi (85% porosity)

S. Gomes et al. MicroelectronicsJournal 44 (2013)

Spatial resolution depends on three main factors :

- Tip sharpness
- Thermal design of the probe
- Tip-sample heat transfer mechanisms (surrounding environment)

SThM的特点

SThM的主要优势：

- 空间分辨率高；可以实现对样品进行微区热电性质分析，适用于薄膜、纳米线、纳米颗粒、以及单分子热电性质的表征。
- 表面形貌与热电性质表征同时完成，热电势像和形貌像相互对应，可实现热电性质原位分析。

SThM的关键技术问题：

- 样品两接触面 (导电探针和金属电极) 之间温差的控制和准确测量
- 针尖-样品的接触状态控制

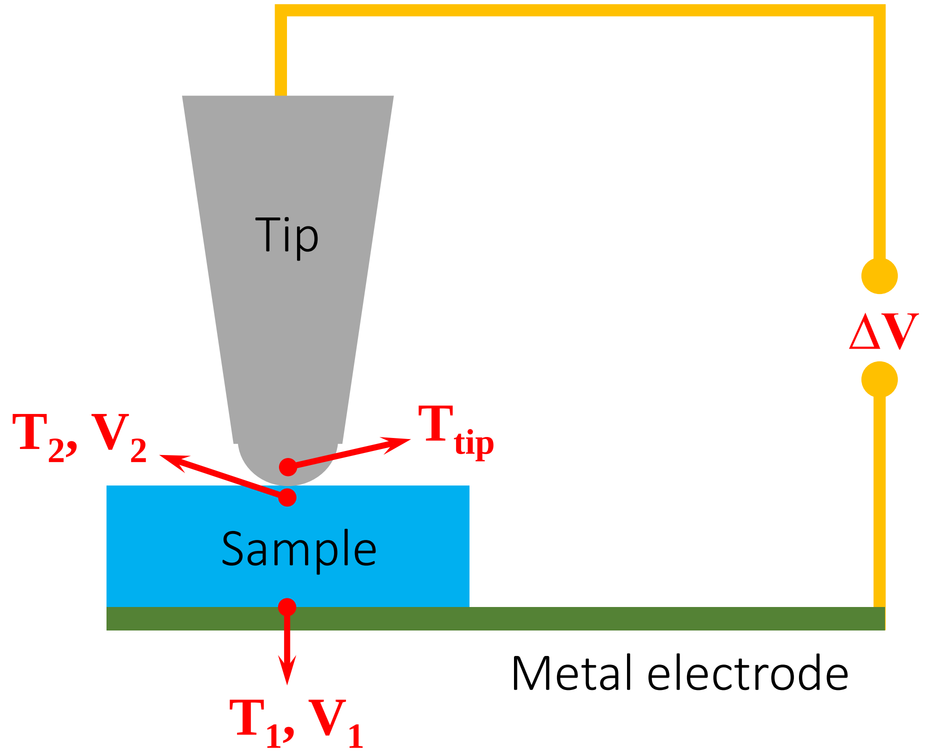
SThM的定量测量研究

SThM用于样品Seebeck系数的定量测量

$$-\nabla V = S\nabla T$$

$$-\boxed{(V_1 - V_2)} = S\boxed{(T_1 - T_2)}$$

ΔV **ΔT**



关键问题：ΔT 的准确测定

扫描热显微镜 (SThM) 的温度定量测量

Kim, K., et al. ***Appl. Phys. Lett.*** 2008.

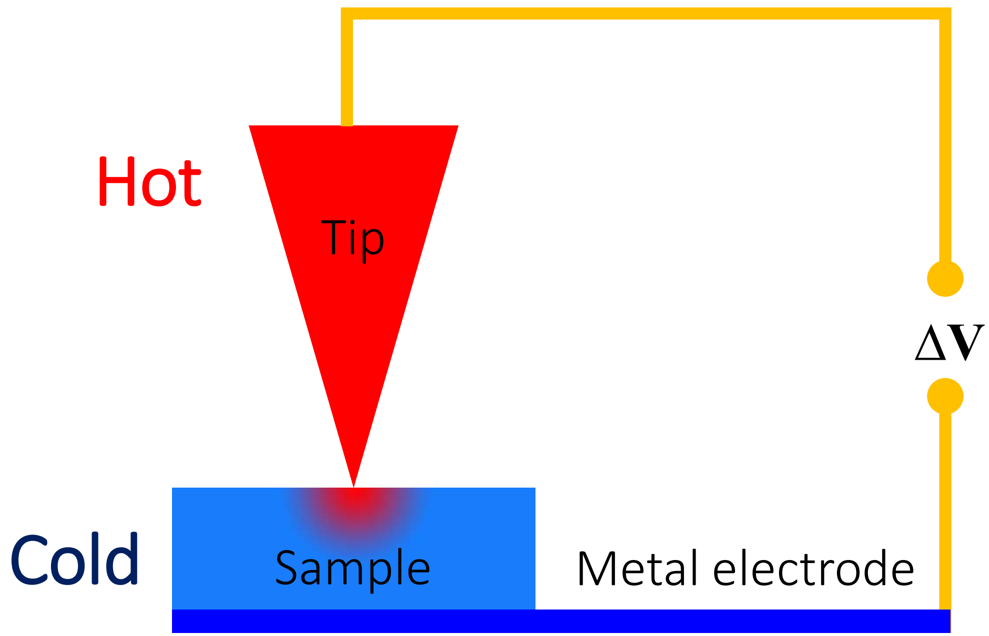
Kim, K., et al. ***ACS Nano*** 2011.

Kim, K., et al. ***ACS Nano*** 2012.

主动加热式SThM

共同特点：

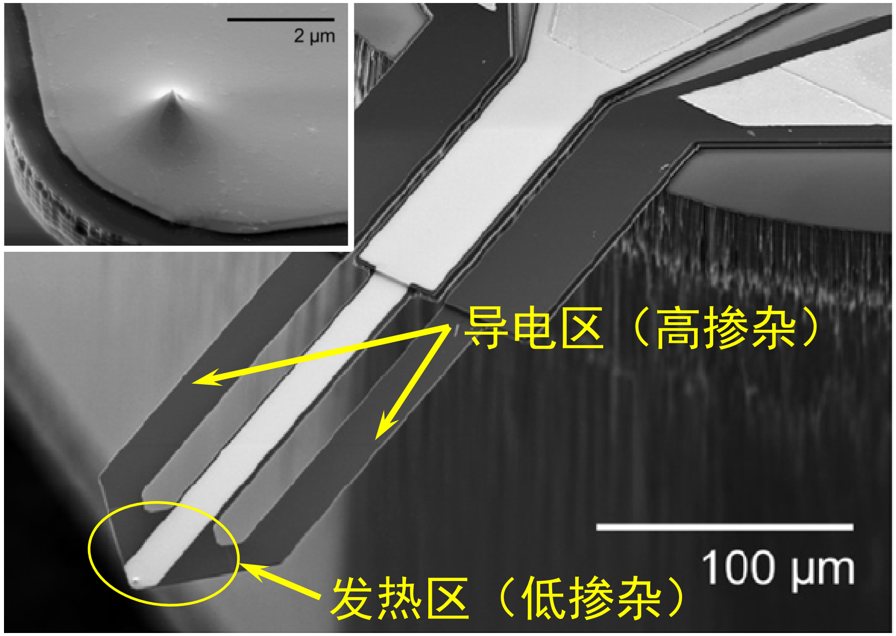
使用特殊制造的探针，依靠焦耳热效应让针尖发热，当针尖接触到较冷的样品时，样品两电极之间形成温度差，由此产生热电势。



主动加热式SThM

典型探针结构

掺杂硅悬臂加热式

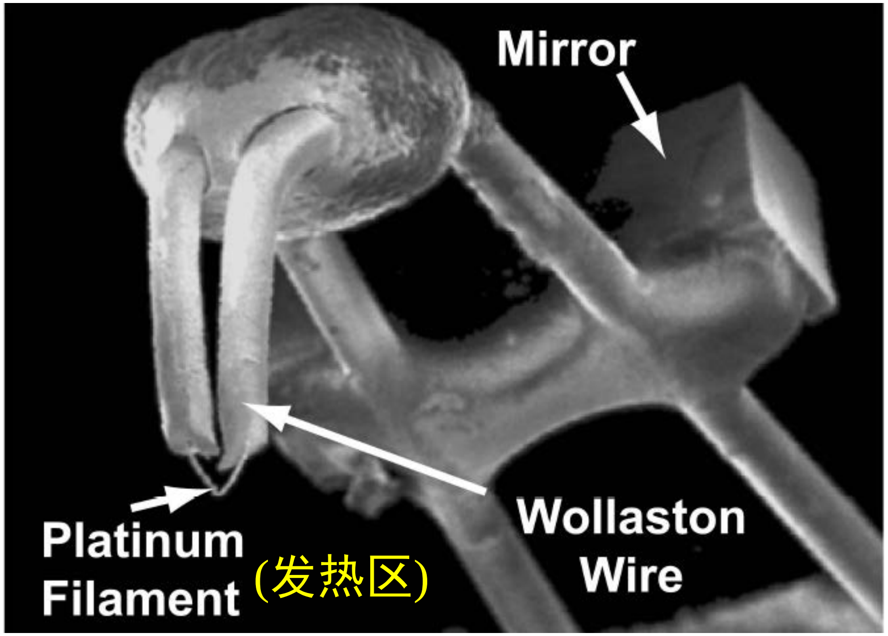


Fletcher, P. C., et al. ***Nanotechnology*** 2012.

Menges, F., et al. ***Nano lett.*** 2012.

Park, K., et al. ***Rev. Sci. Instrum.*** 2007.

电阻丝加热式



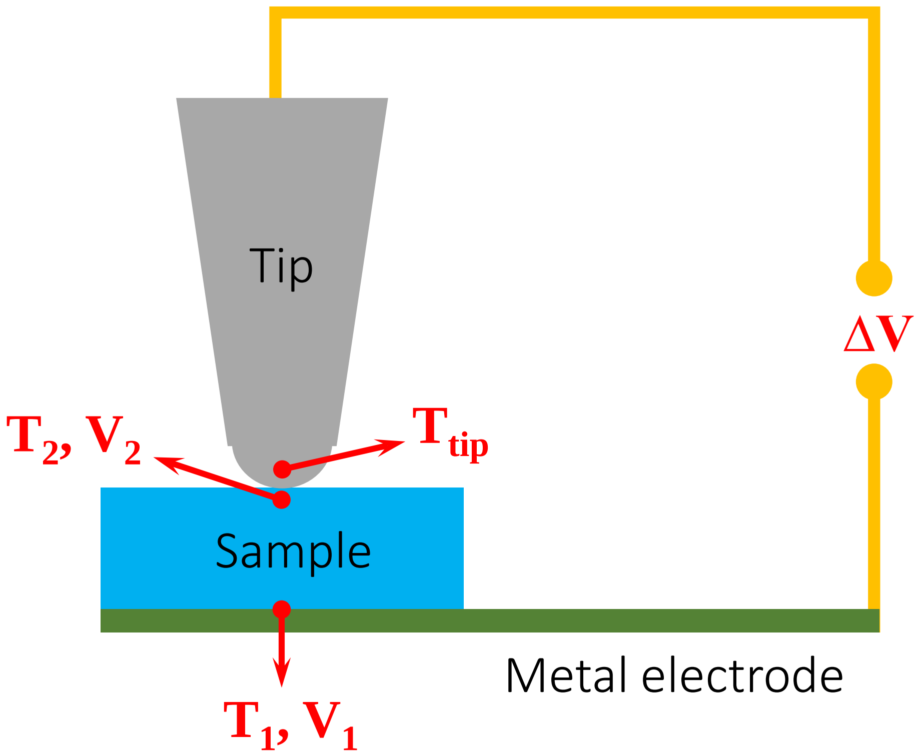
Zhang, Y., et al. ***Appl. Phys. Lett.*** 2010.

Zhang, Y., et al. ***Rev. Sci. Instrum.*** 2011.

主动加热式SThM

温差测定

- T_1 固定为已知温度：
  温控台、热电制冷片、空气
- T_2 无法直接测定：
  目前是SThM研究的难点
- T_{tip} 能直接测定：
  热敏电阻法、热电偶法



忽略针尖-样品接触热阻 → $T_{tip} = T_2$ → $\Delta T \approx T_1 - T_{tip}$

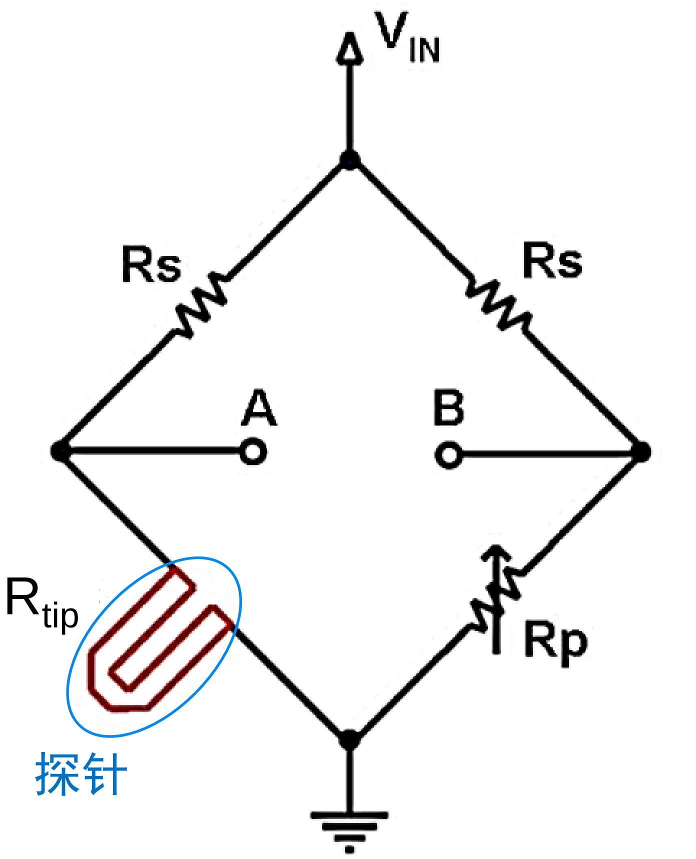
主动加热式SThM

针尖温度测定

- 热敏电阻法 (使用最广泛)

$$R_{tip} = R_0\left[1+\alpha\left(T_{tip} - T_0\right)\right]$$

$$\alpha = \frac{1}{R}\frac{dR}{dT}$$ 电阻温度系数需要事先标定



惠斯通电桥测定探针电阻

特点：

- 针尖的加工相对较容易
- 测得的温度代表针尖附近区域的平均温度

Park, K., et al. ***Rev. Sci. Instrum.*** 2007.

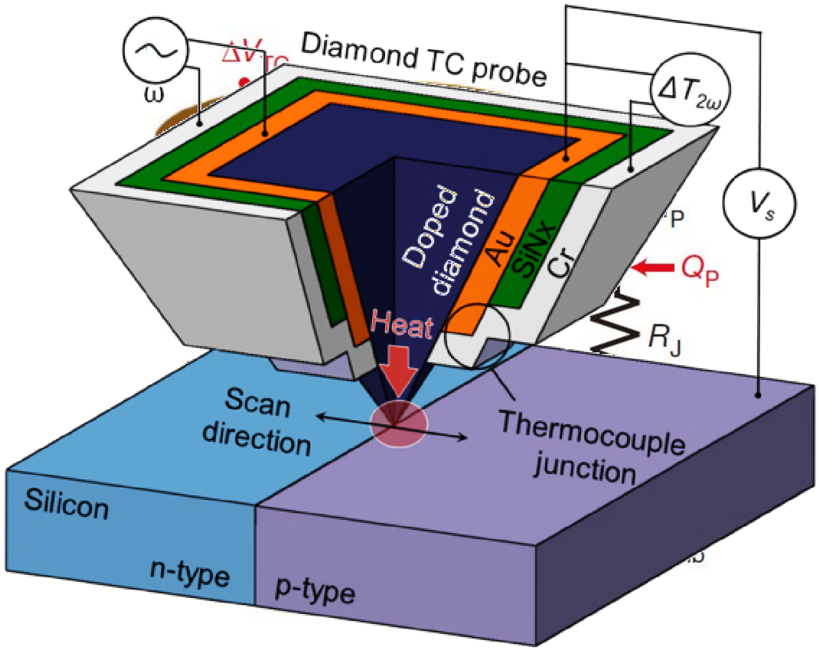
Zhang, Y., et al. ***Rev. Sci. Instrum.*** 2011.

主动加热式SThM

针尖温度测定

热电偶集成式探针

- ■ 热电偶法
- ➢ 热电偶集成在针尖直接测量针尖温度
- ➢ 热电偶同时充当焦耳热源和温度计



特点：

- ■ 针尖的温度测量更局域、更准确
- ■ 保证针尖和样品接触良好而又不磨损针尖
- ■ 针尖的加工难度较大

Lee, B., et al. ***Nano lett.*** 2012.

Lee, W., et al. ***Nature*** 2013.

系统特点及功能

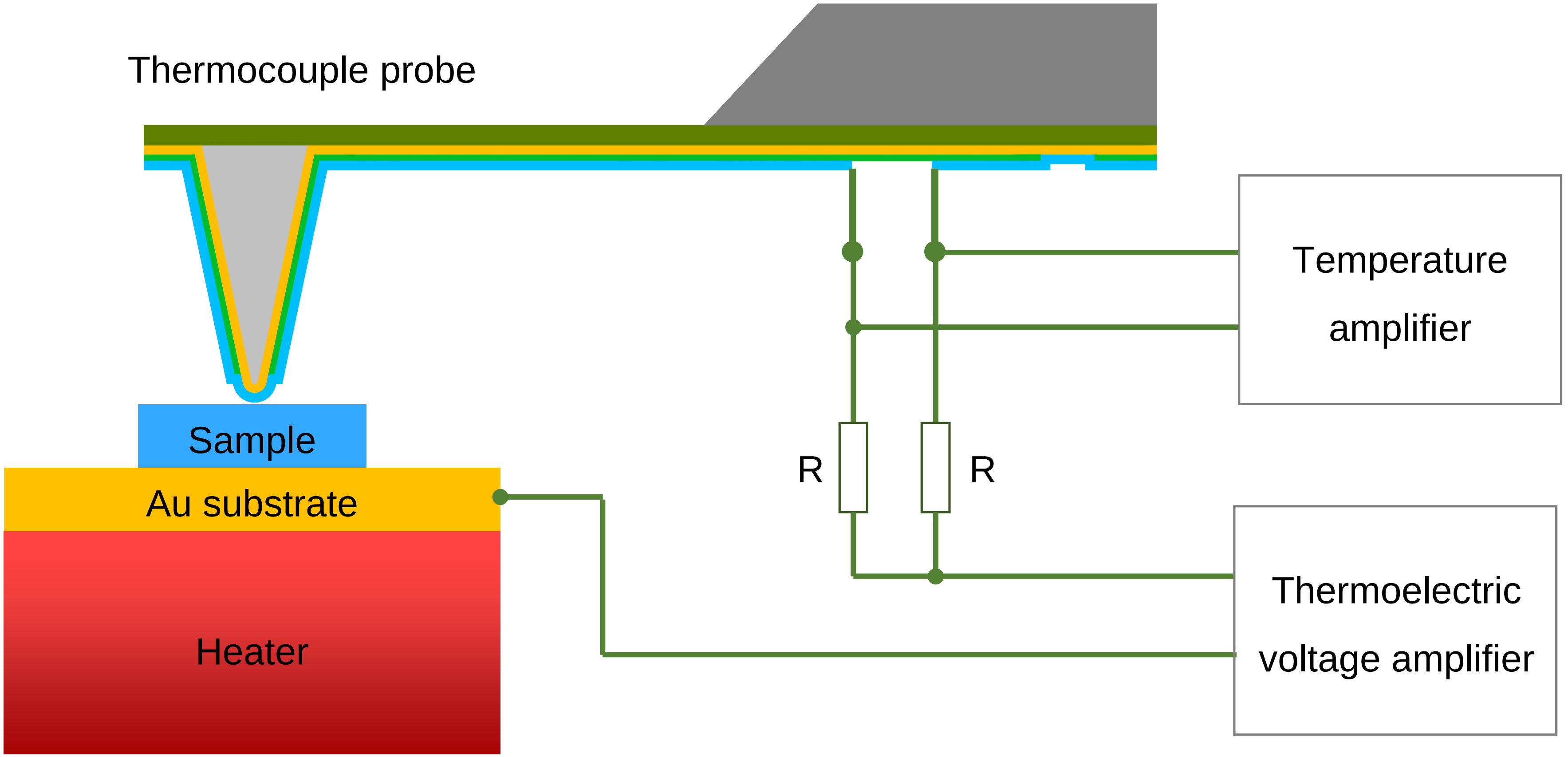
热电偶探针SThM

温度测量精度：<100mK

空间分辨率：20~50nm

测量范围：室温~700℃

- 接触模式下工作
- 形貌和温度同步成像
- 温度、热电势同步测量
- 形貌、热导率(定性)同步成像



Next class

- AFM高阶模式
- 微纳电子大厦443室
- 4月25日周一下午1-3点，分4组