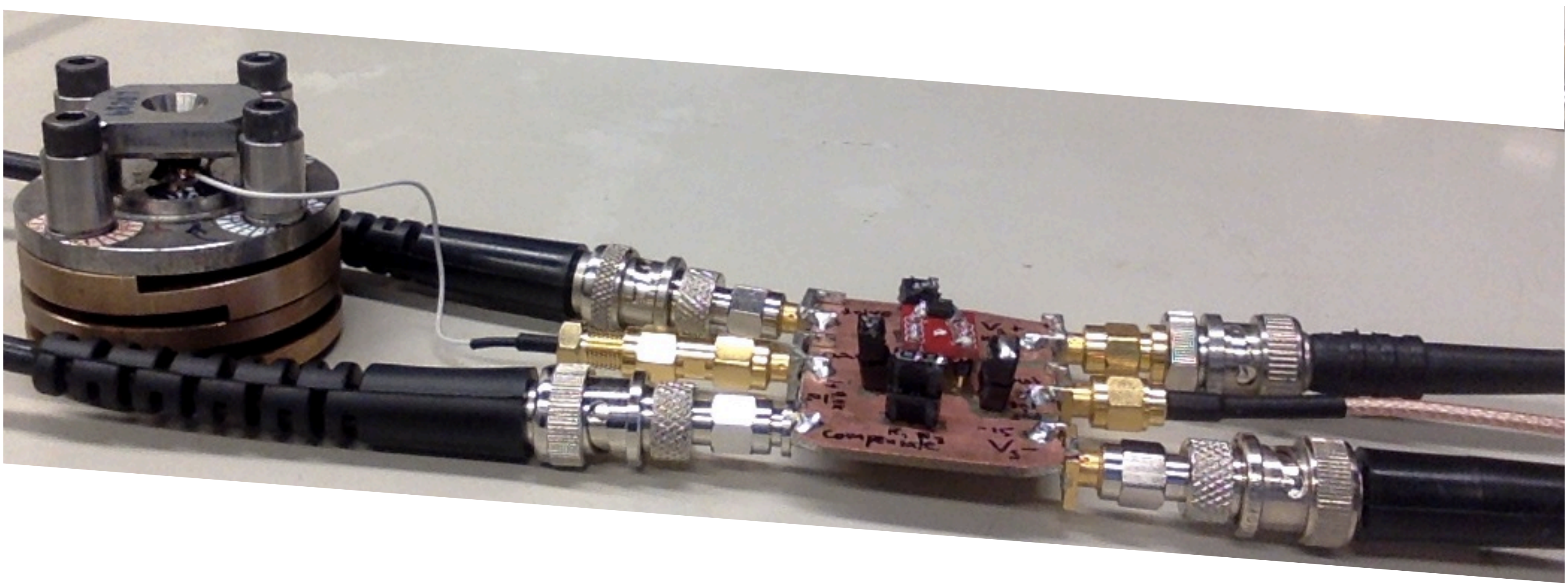
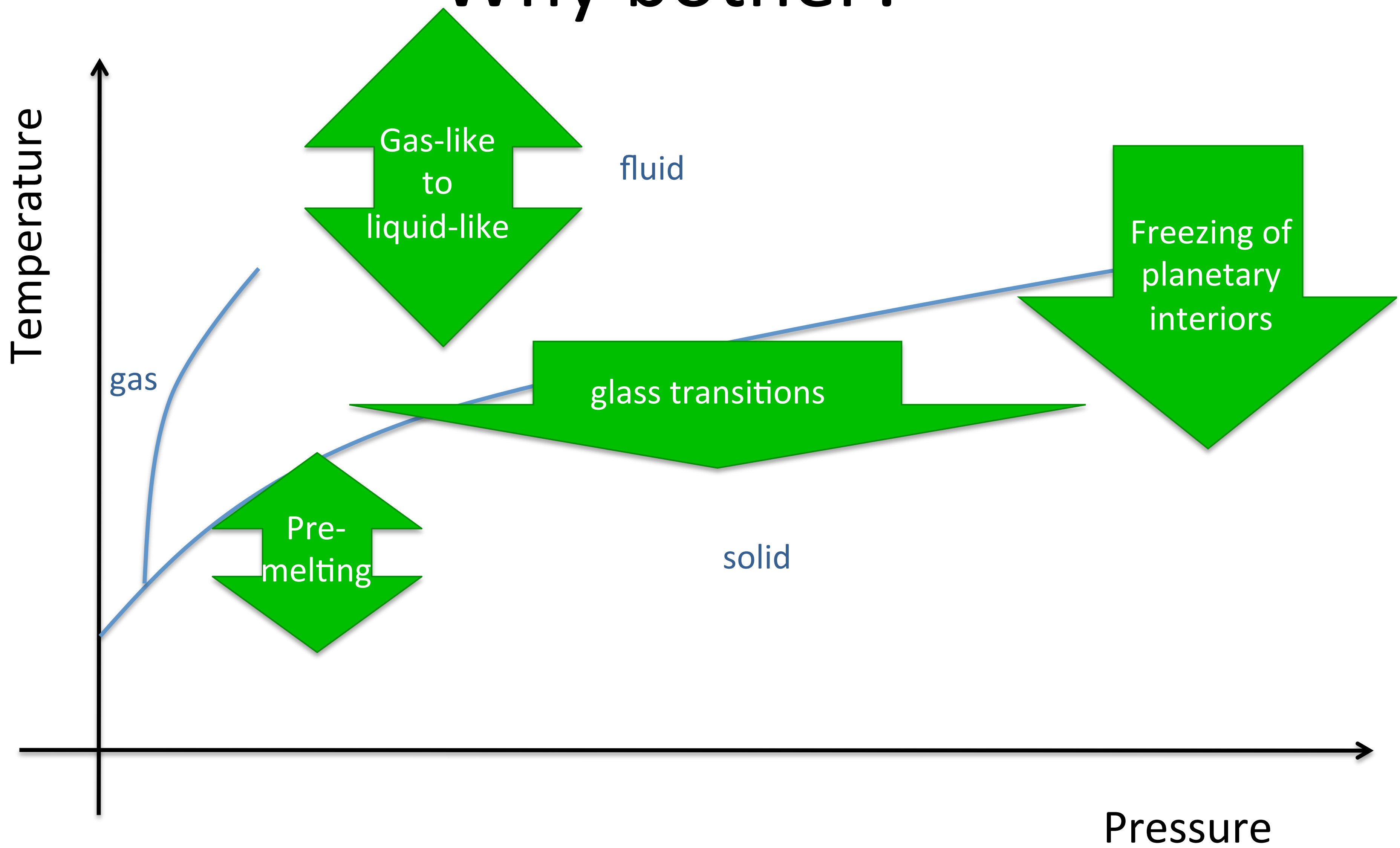
High-pressure calorimetry: design and prototypes

Zack Geballe



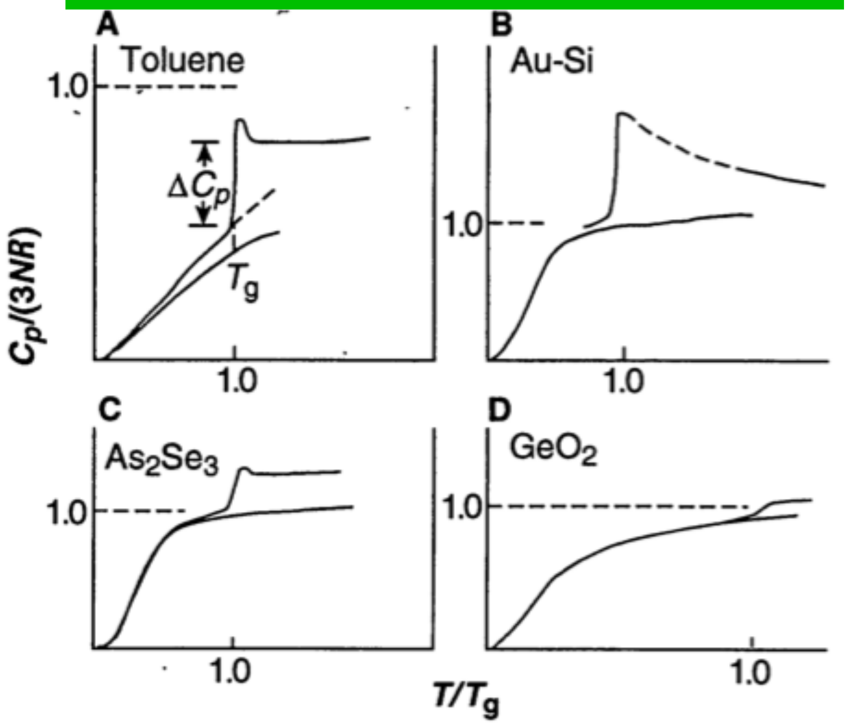
Why bother?



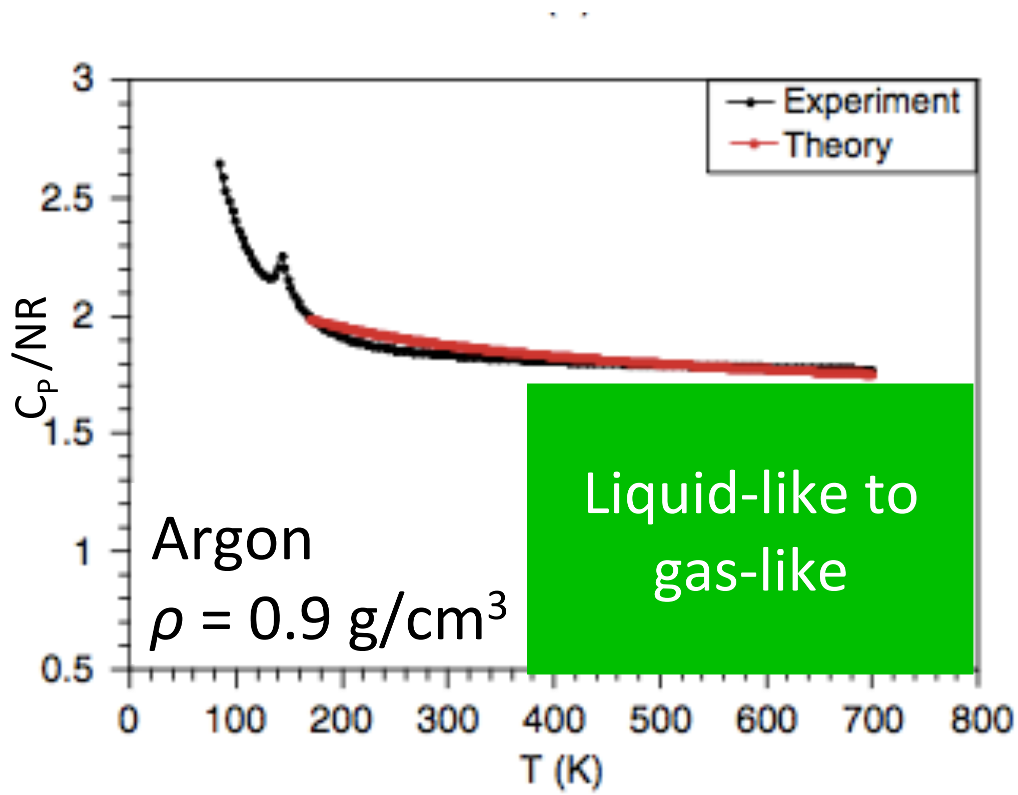
Temperature
Gas-like
to
liquid-like
fluid
Freezing of
planetary
interiors
gas
glass transitions
Pre-
melting
solid
Pressure

Potential applications of $C_p(P,T)$

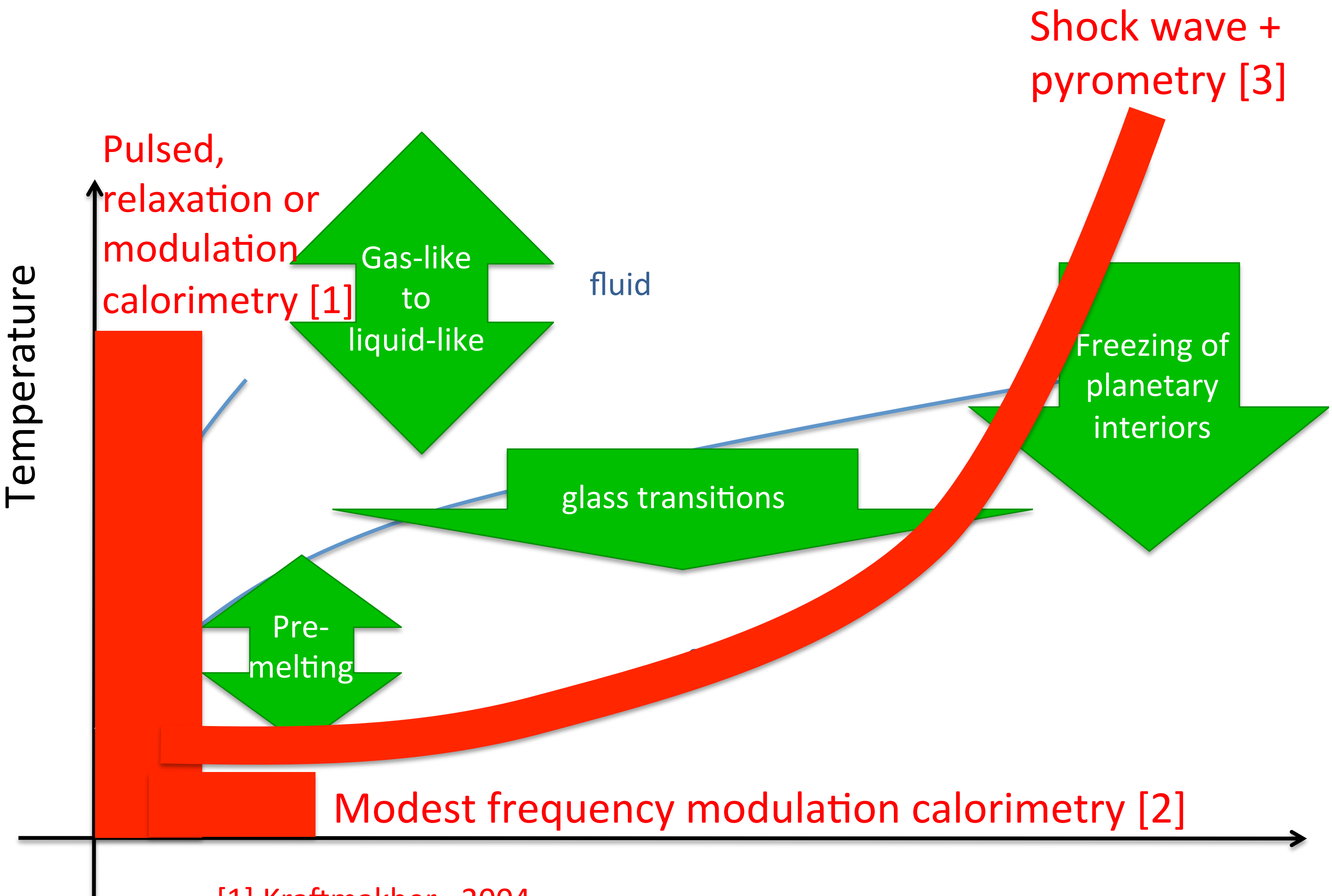
Glass transitions



Angell 1995



Bolmatov et al., 2013



[1] Kraftmakher, 2004
[2] Bouquet et al., 2000 ; Sidorov et al., 2010
[3] Hicks et al., 2006

A recipe for C_P in diamond-cell

Doable

1. Make circuit through diamond cell

Easy

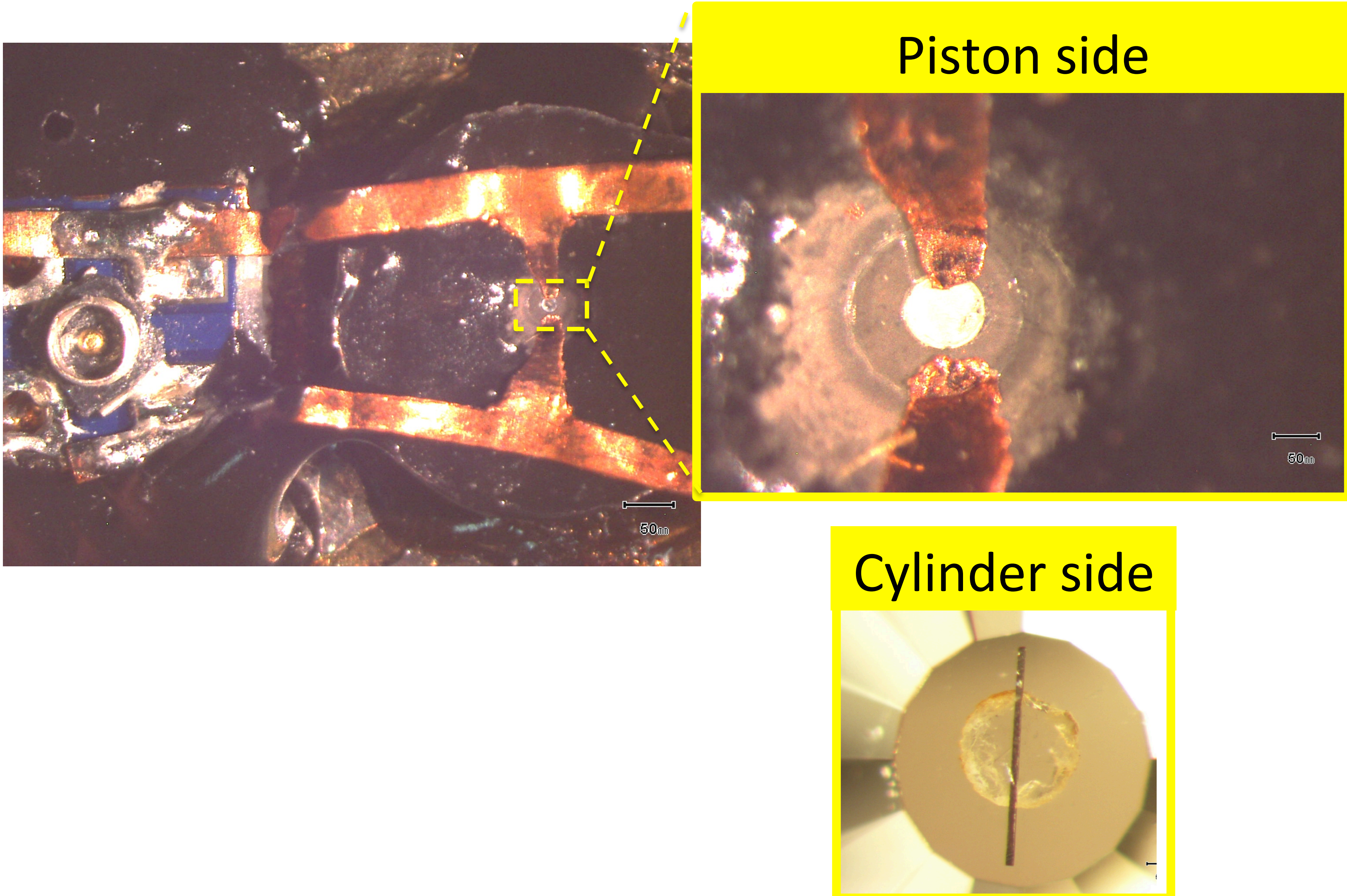
2. Drive circuit with ± 5 V

Hard

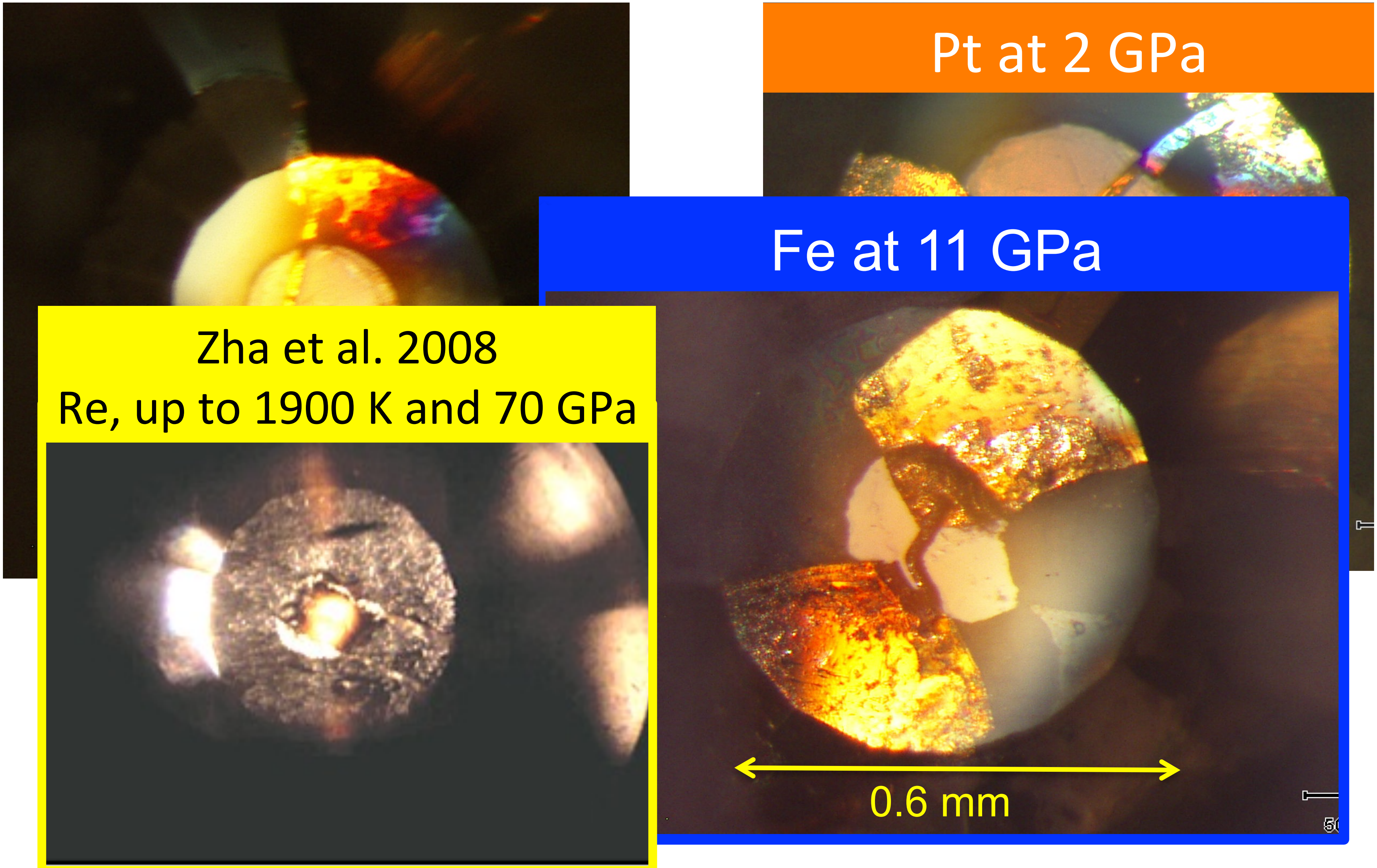
3. Measure V, R, dR/dT and 3rd harmonic voltage
4. Infer $p_{2\omega}$, $T_{2\omega}$, then $C = p_{2\omega}/2\omega T_{2\omega}$

…or $(kC)^{1/2}$, or k & C

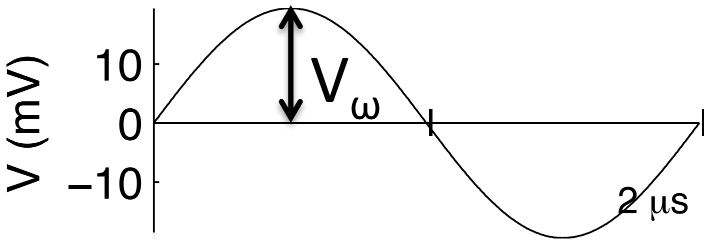
Step 1a: Metal foil between diamond tips

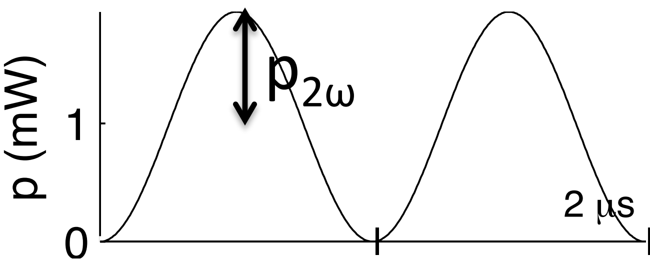


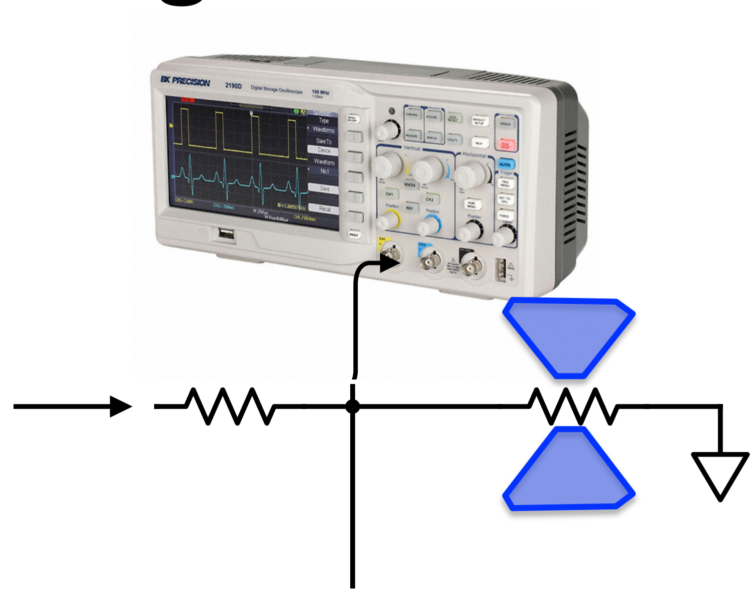
Step 1b: Press



Step 1c: Design circuit



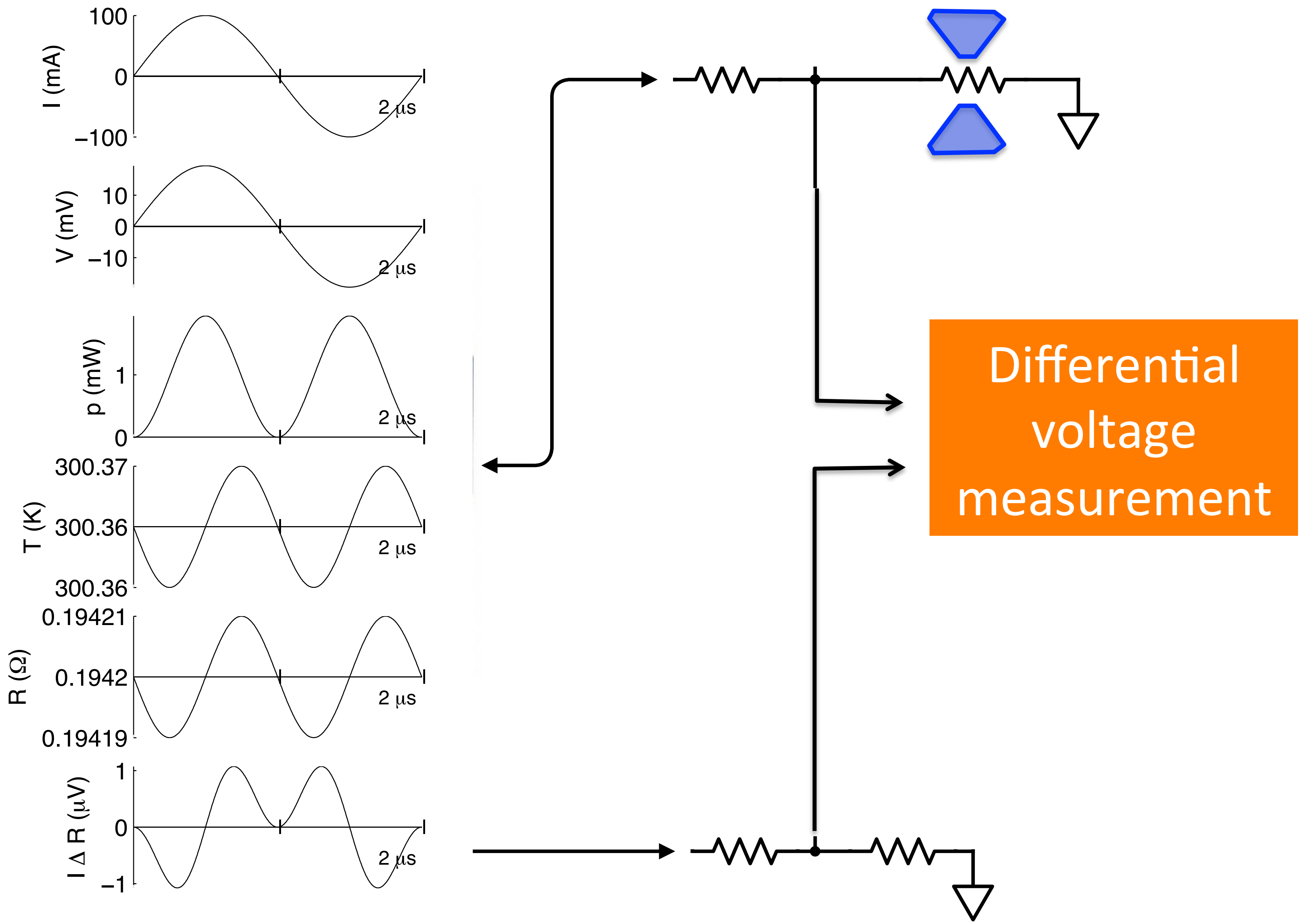




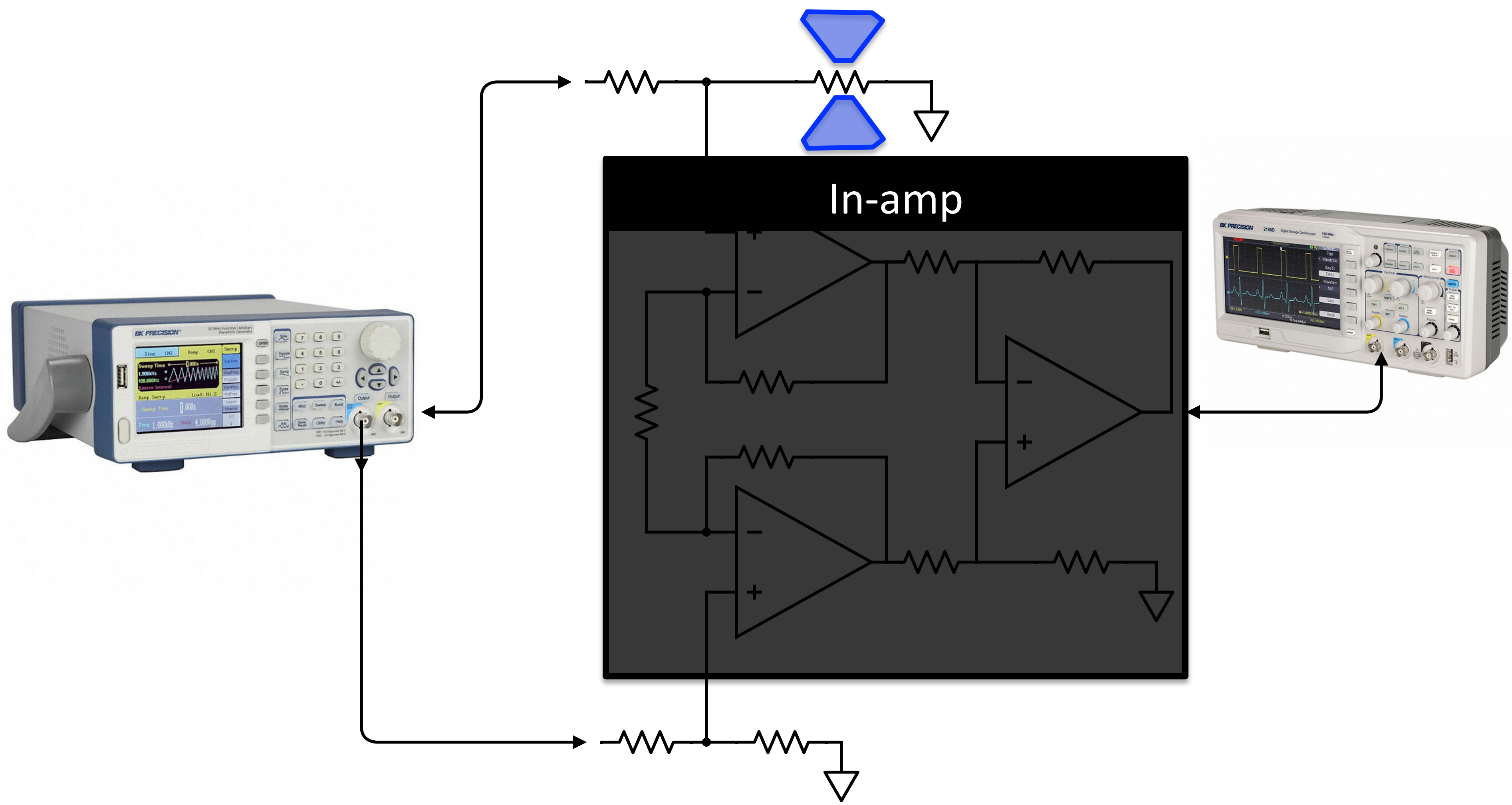
$$p_{2\omega} = \frac{1}{2}\frac{V_\omega^{\;2}}{R_S}$$

$$C = \frac{p_{2\omega}}{2\omega T_{2\omega}}$$

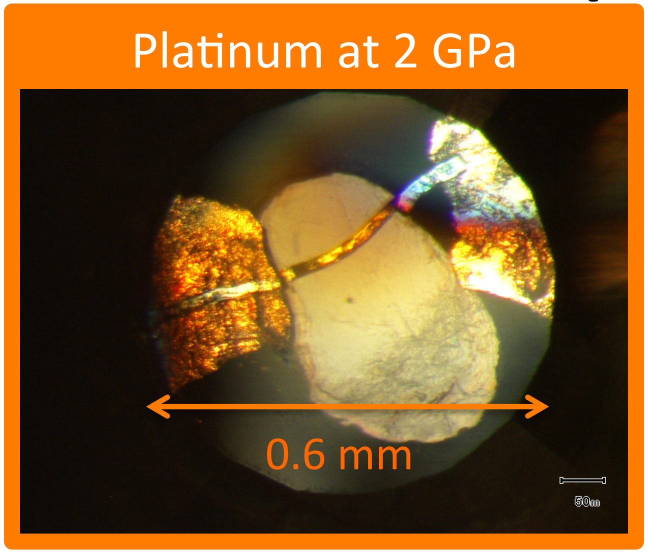
Step 1c: Design circuit

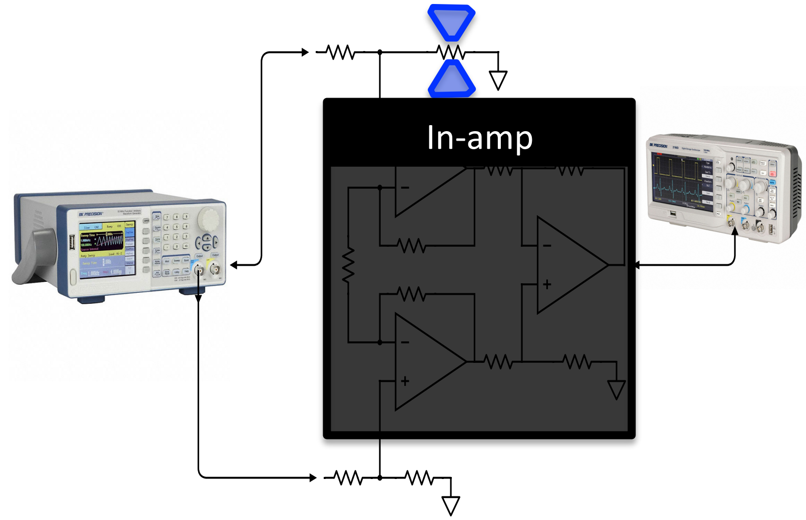


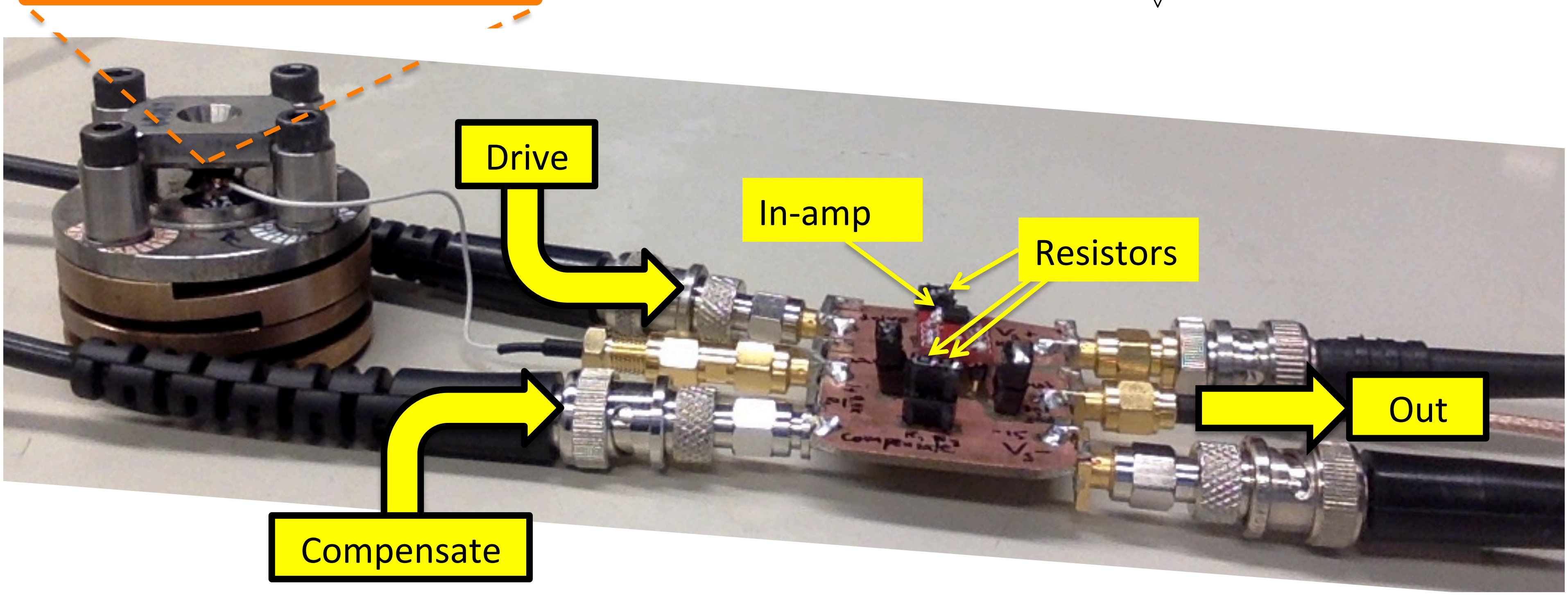
Step 1c: Design circuit

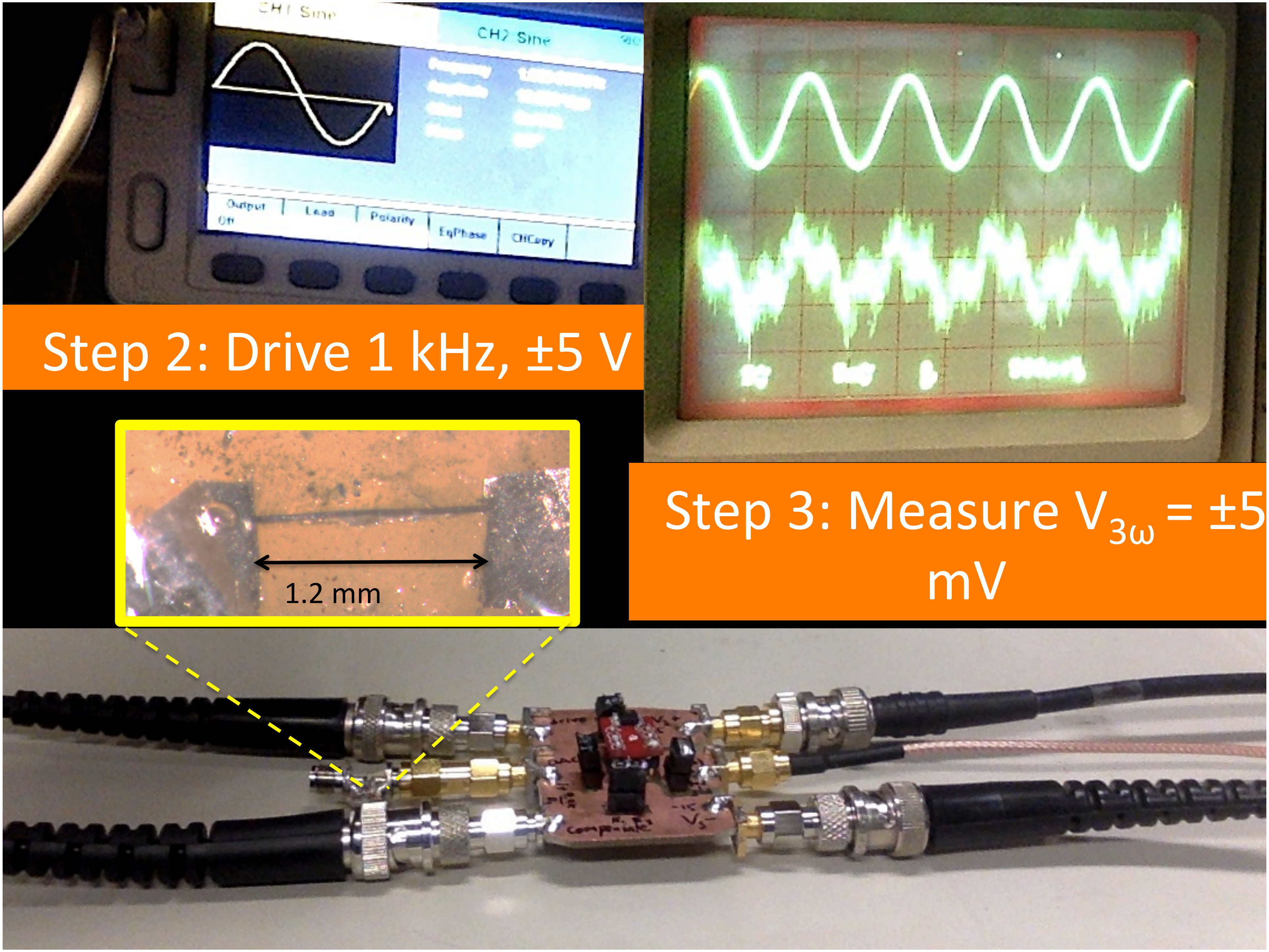


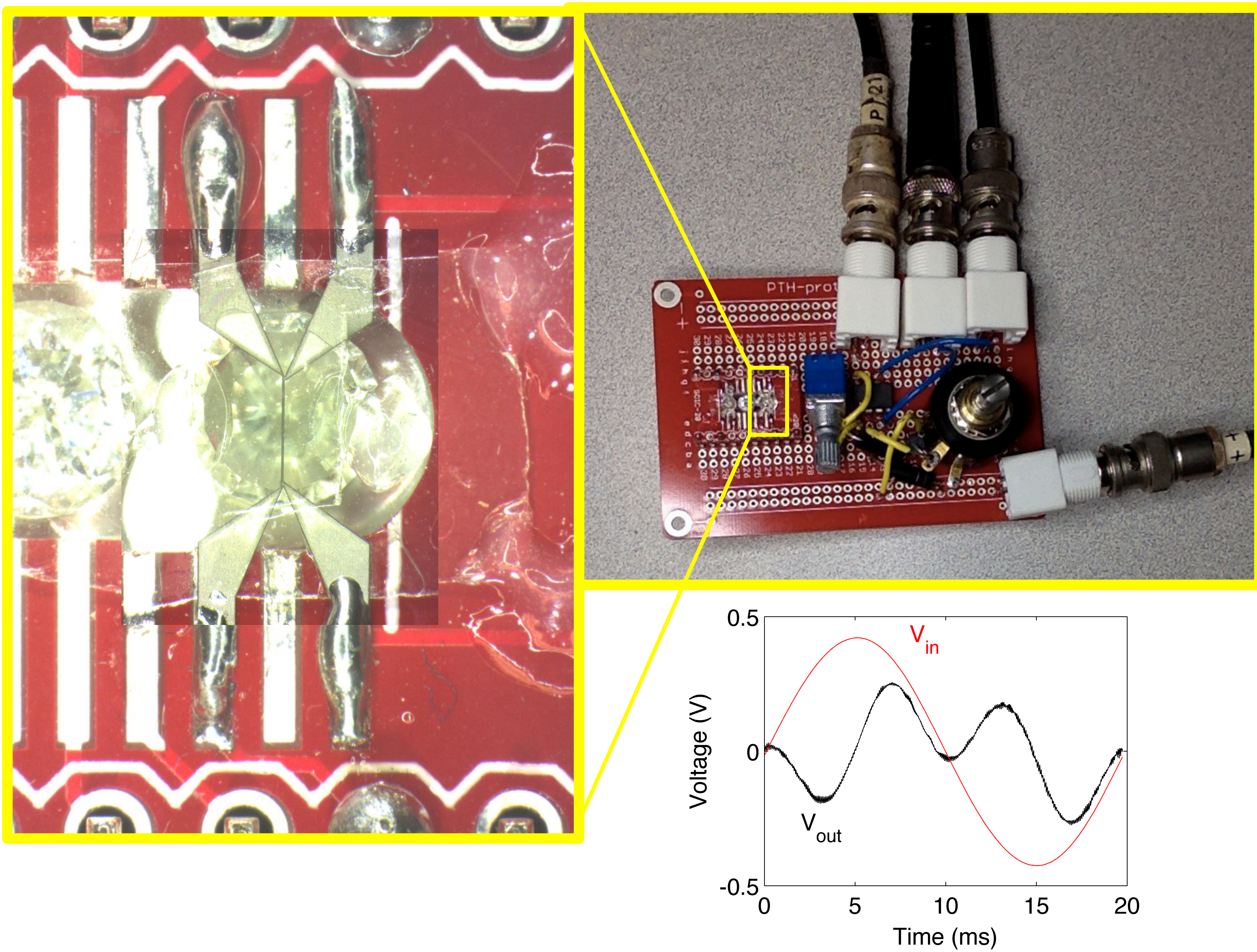
Step 1d: Make circuit





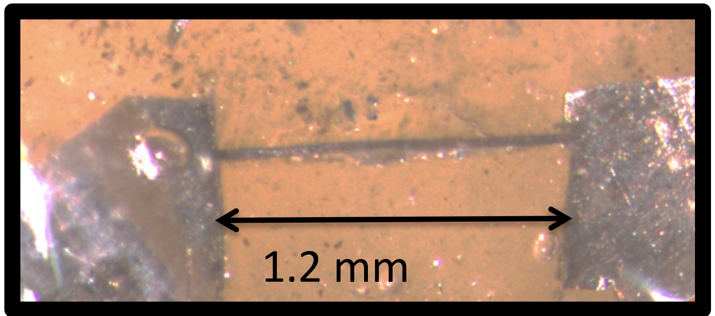


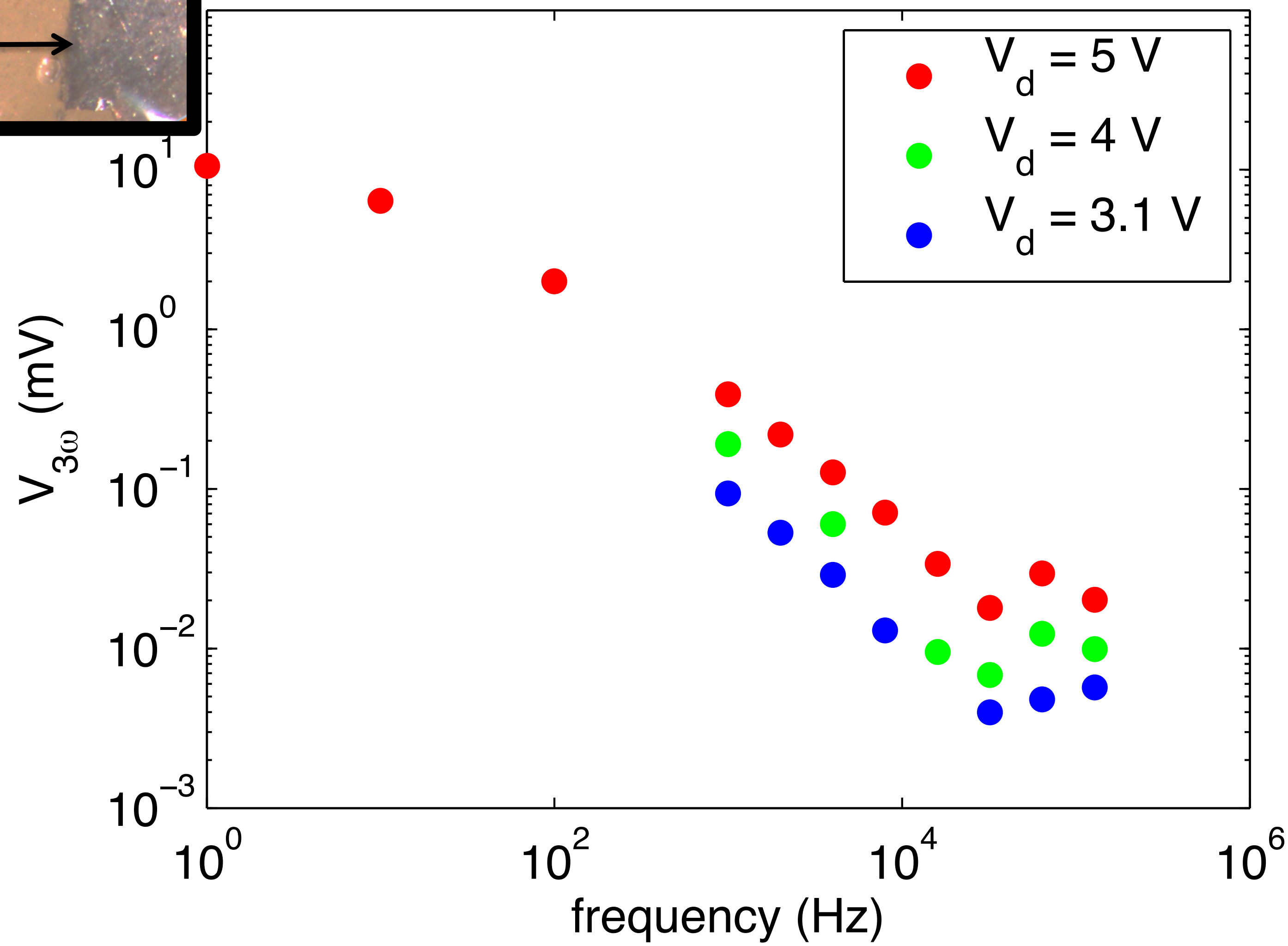

CH1 Sine
CH2 Sine
Output
Off
Load
Polarity
EqPhase
CHCopy
Step 2: Drive 1 kHz, ±5 V
1.2 mm
Step 3: Measure $V_{3\omega}$ = ±5 mV



PTH-prot
V in
V out
Voltage (V)
0.5
0
-0.5
0
5
10
15
20
Time (ms)

Step 3: measure $V_{3\omega}$





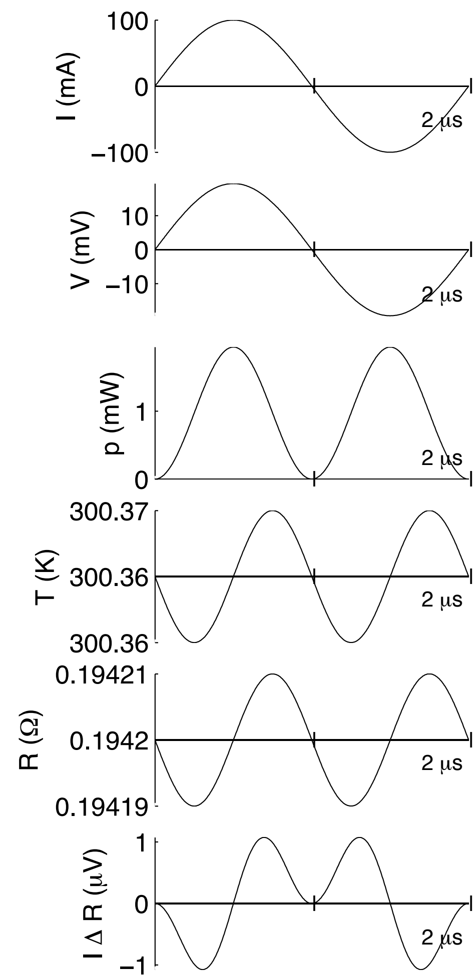
Step 4: Infer $p_{2\omega}$, $T_{2\omega}$, C_P

$$C = \frac{p_{2\omega}}{2\omega T_{2\omega}}$$

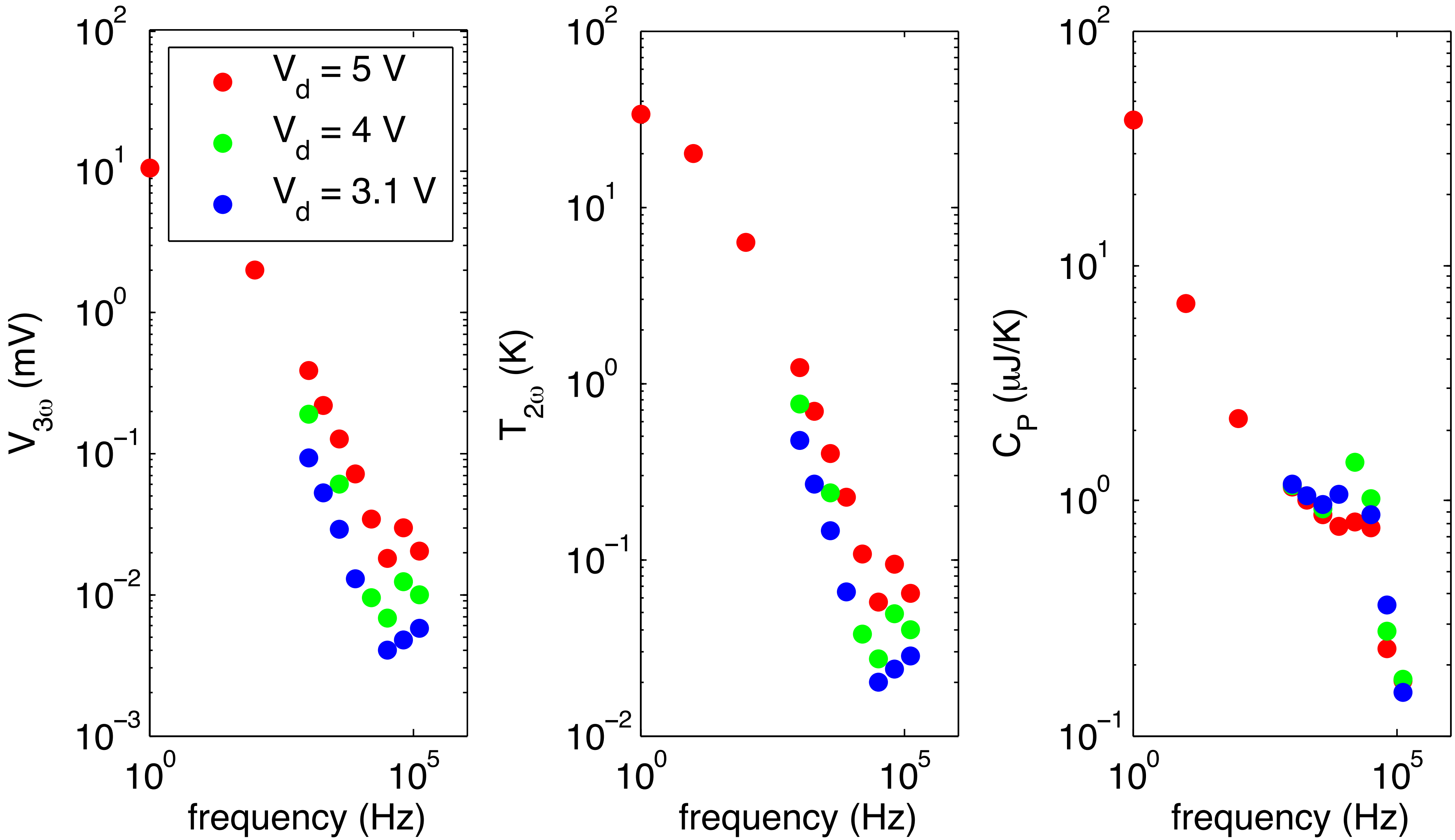
$$p_{2\omega} = \frac{1}{2} I_\omega^2 R_S$$

$$V_{3\omega} = G_{3\omega} V_{3\omega}^{induced}$$

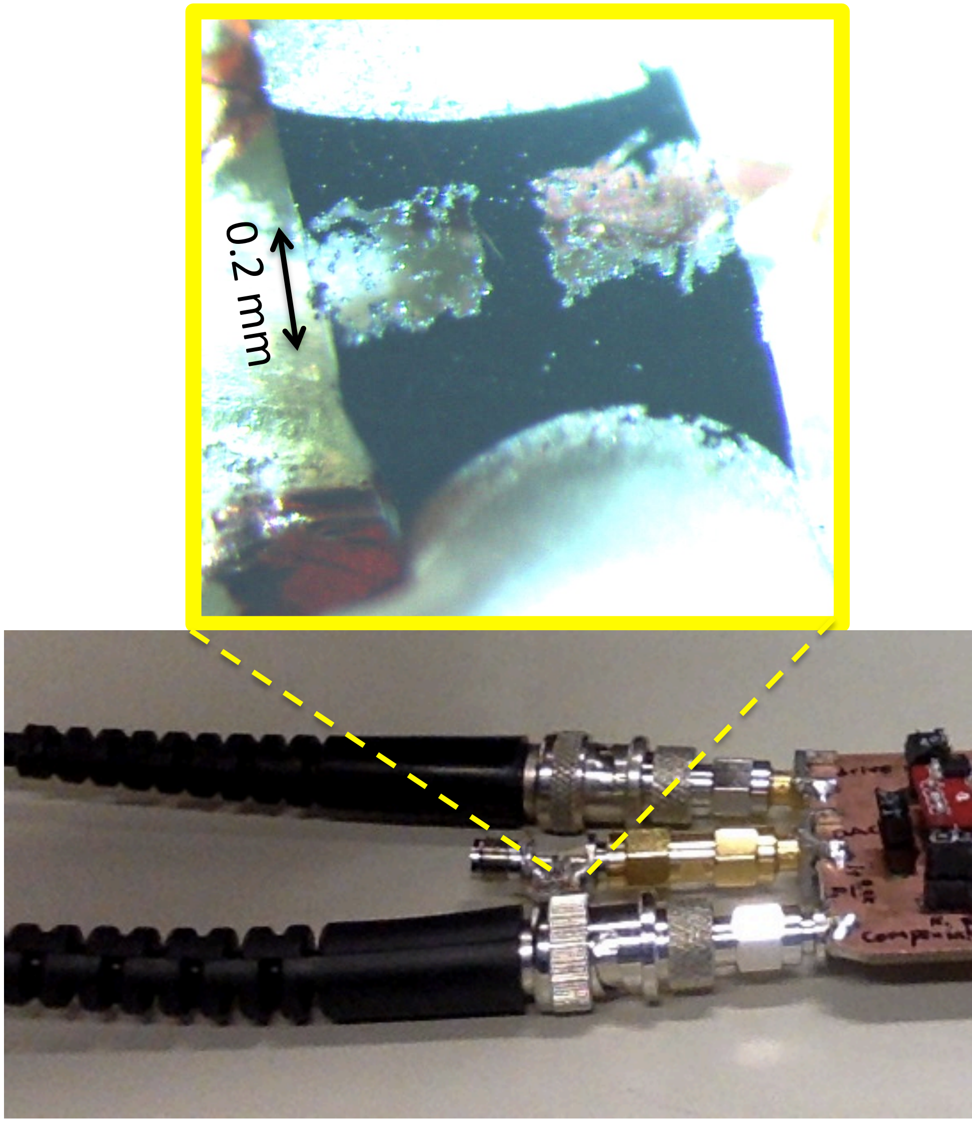
$$V_{3\omega}^{induced} = \frac{1}{2} I_\omega R_S \alpha T_{2\omega}$$

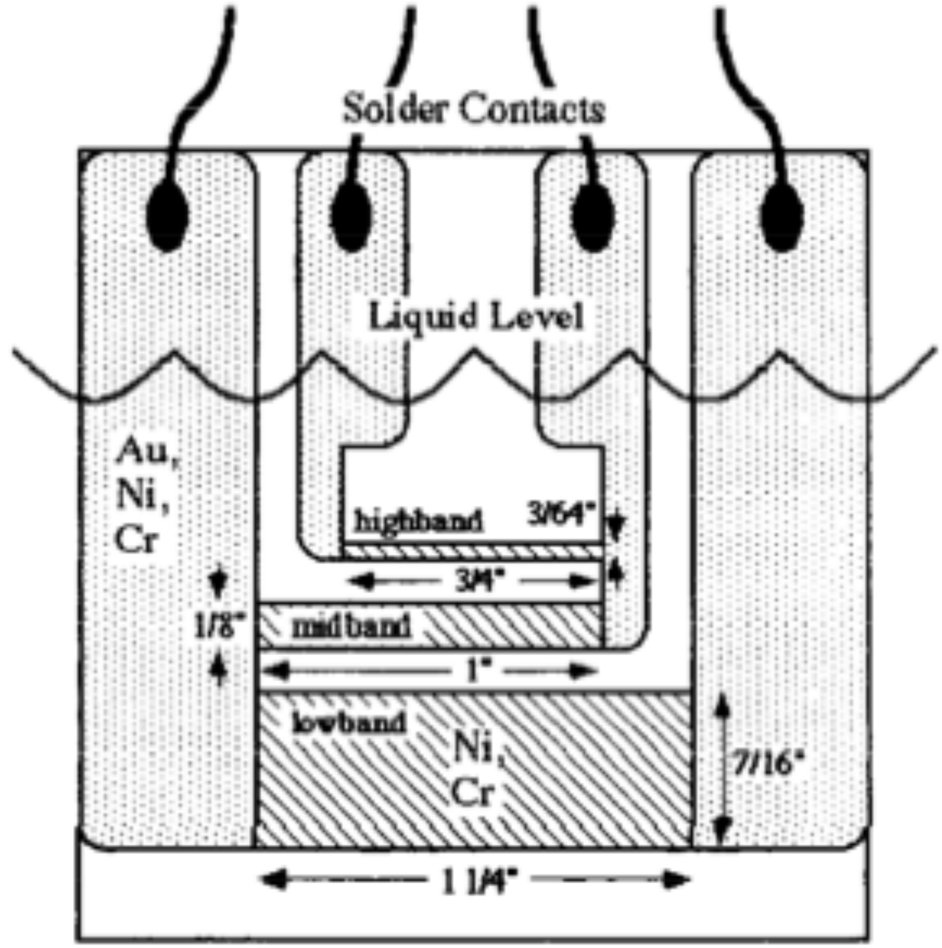


Step 4: Infer $p_{2\omega}$, $T_{2\omega}$, C_P



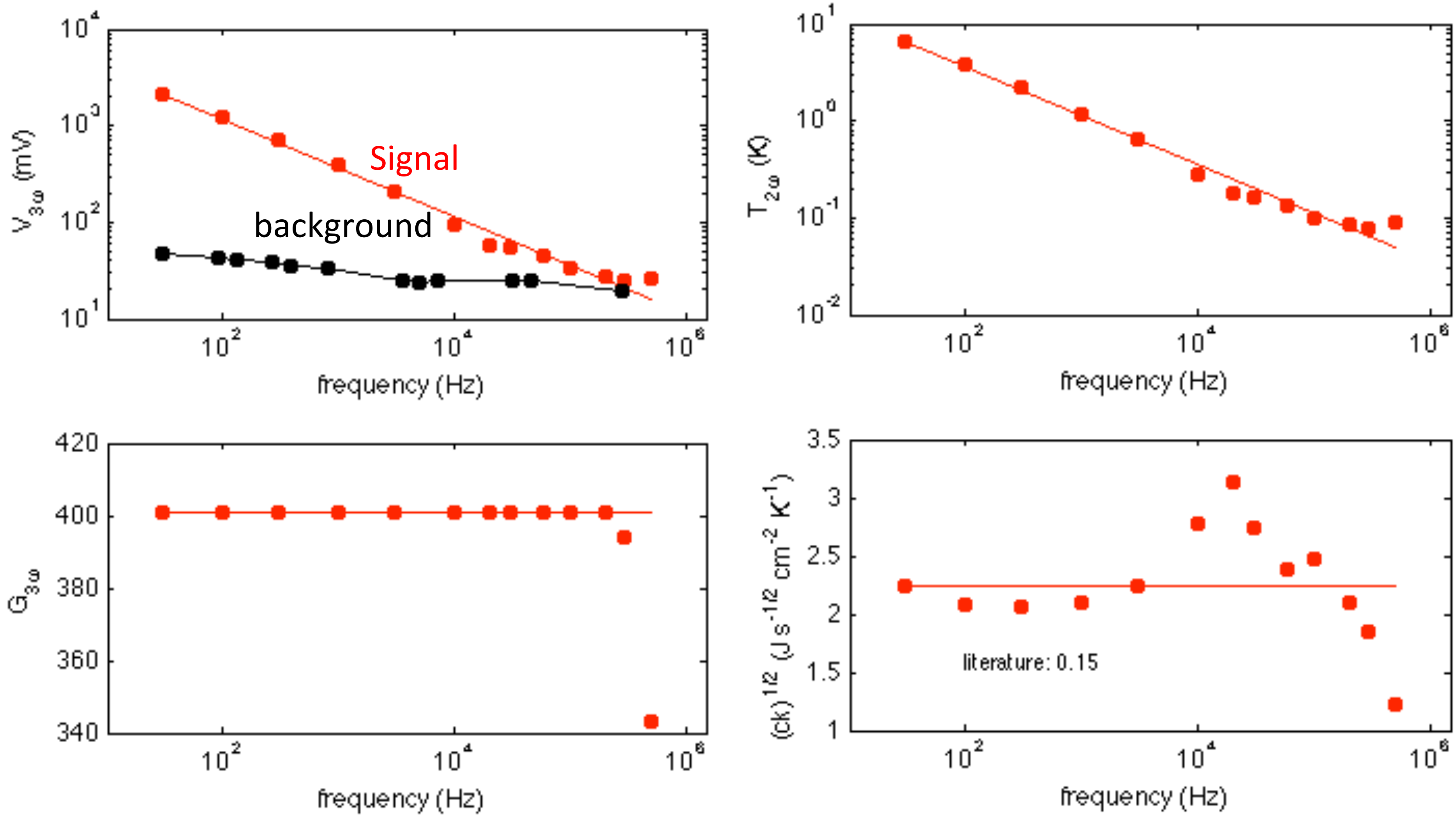
Alternative design: thin film heater



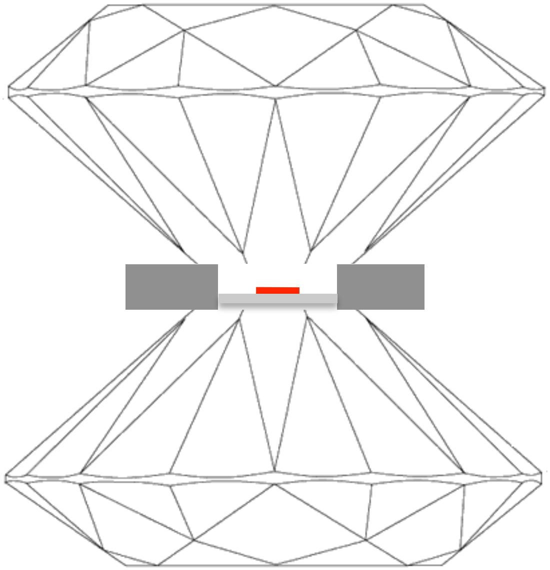


Birge et al., 1997

~10 nm Pt on glass cover slip

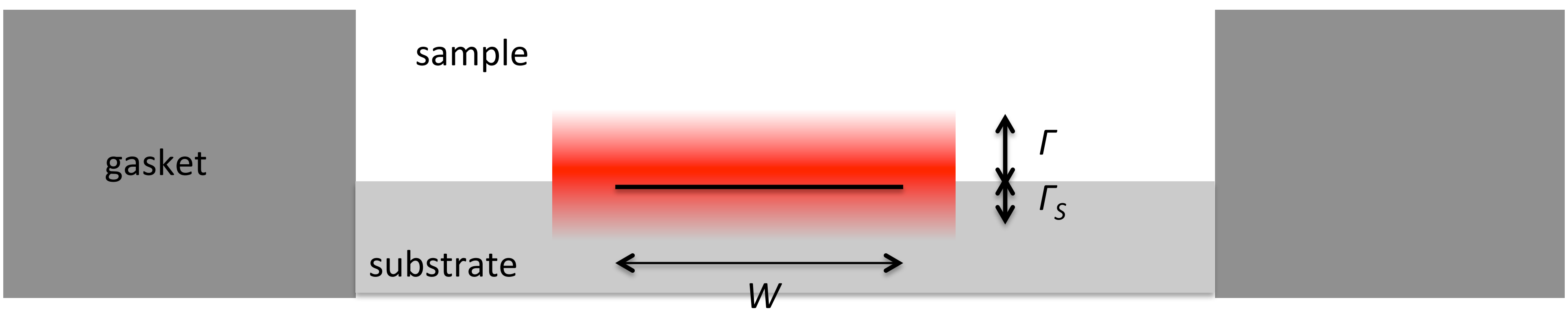


Possibilities with thin film heaters

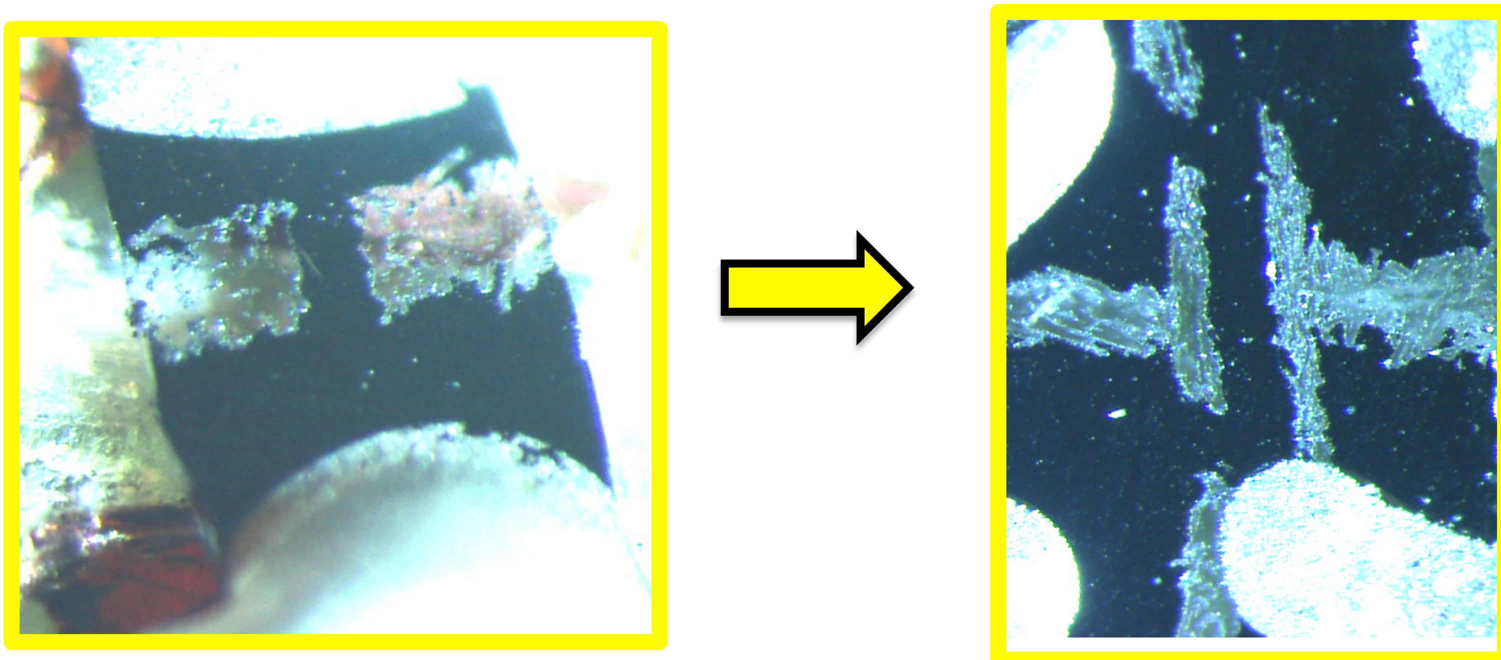


$$Z = \frac{e^{i\pi/4}}{\sqrt{c_{ps}\kappa_s}+\sqrt{c_p\kappa}}[1-\Delta\phi_{SL}]e^{i\Delta\phi_{SL}}$$

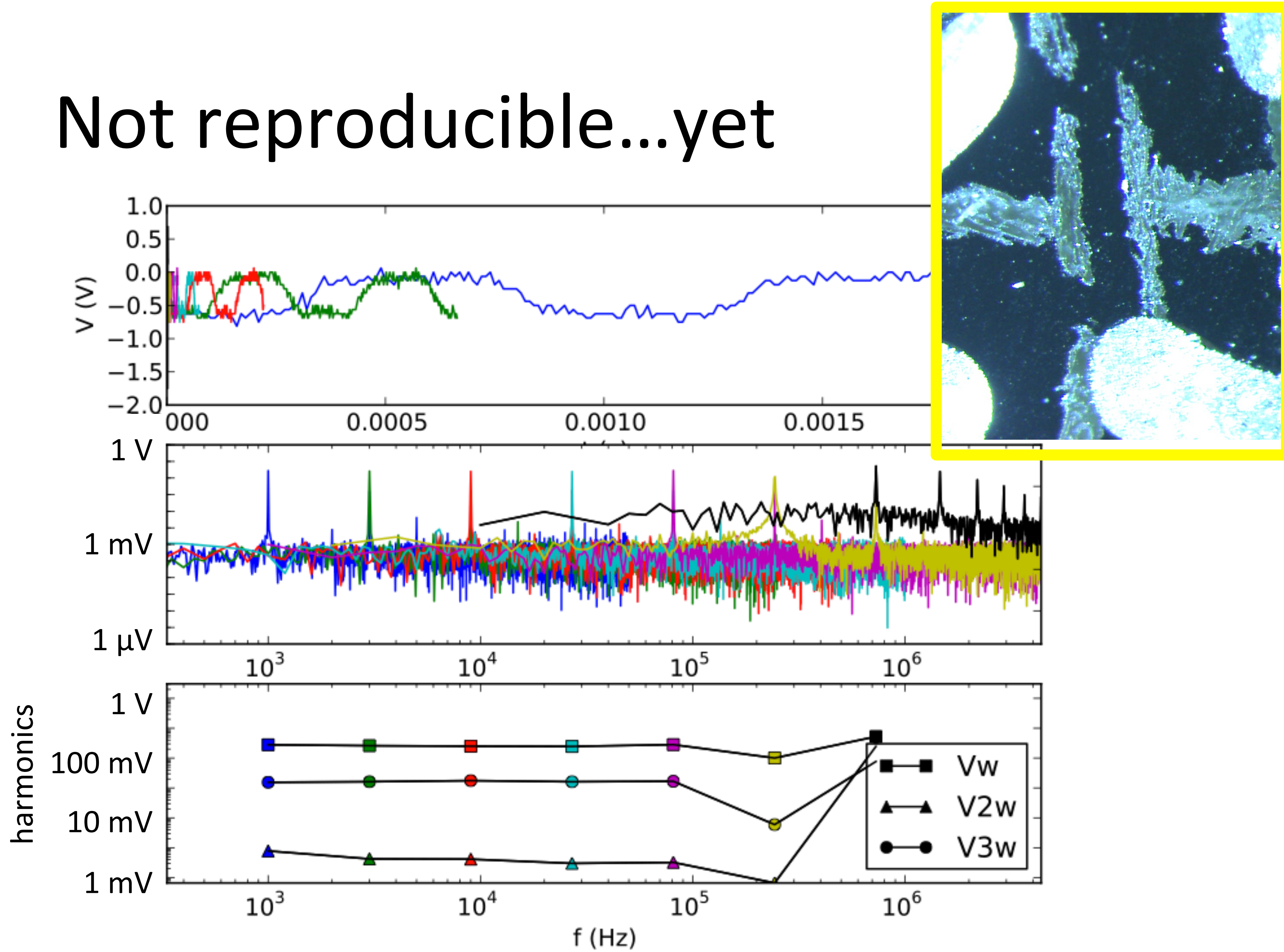
$$\Delta\phi_{SL} \equiv \frac{\kappa_s+\kappa}{\frac{\kappa_s}{\left(\frac{\Gamma_s}{W}\right)}+\frac{\kappa}{\left(\frac{\Gamma}{W}\right)}} = \frac{1}{W\sqrt{2\omega}}\frac{\kappa_s+\kappa}{\sqrt{\frac{\kappa_s}{c_{ps}}}\kappa_s+\sqrt{\frac{\kappa}{c_p}}\kappa}$$



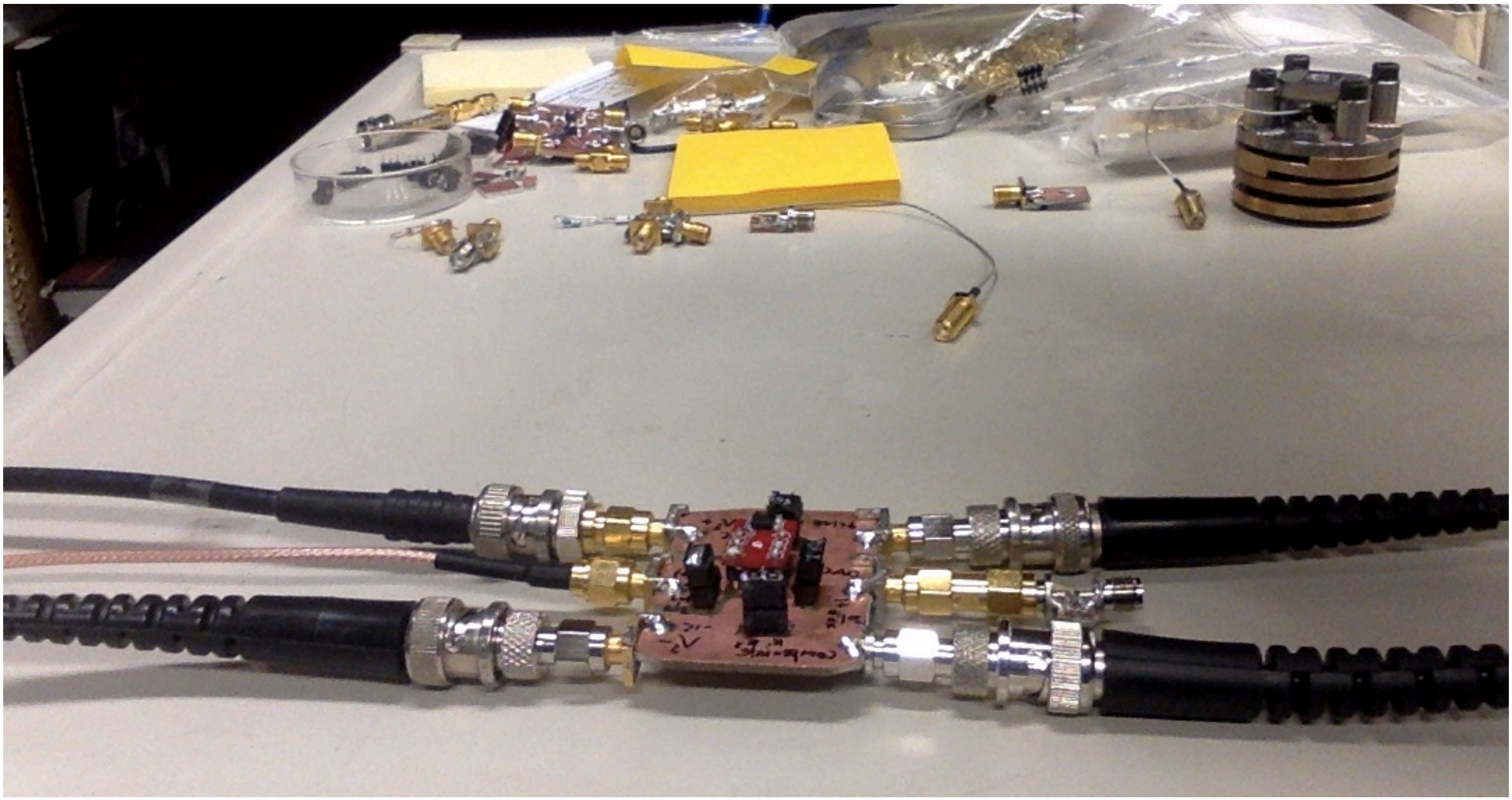
Reproducibility?



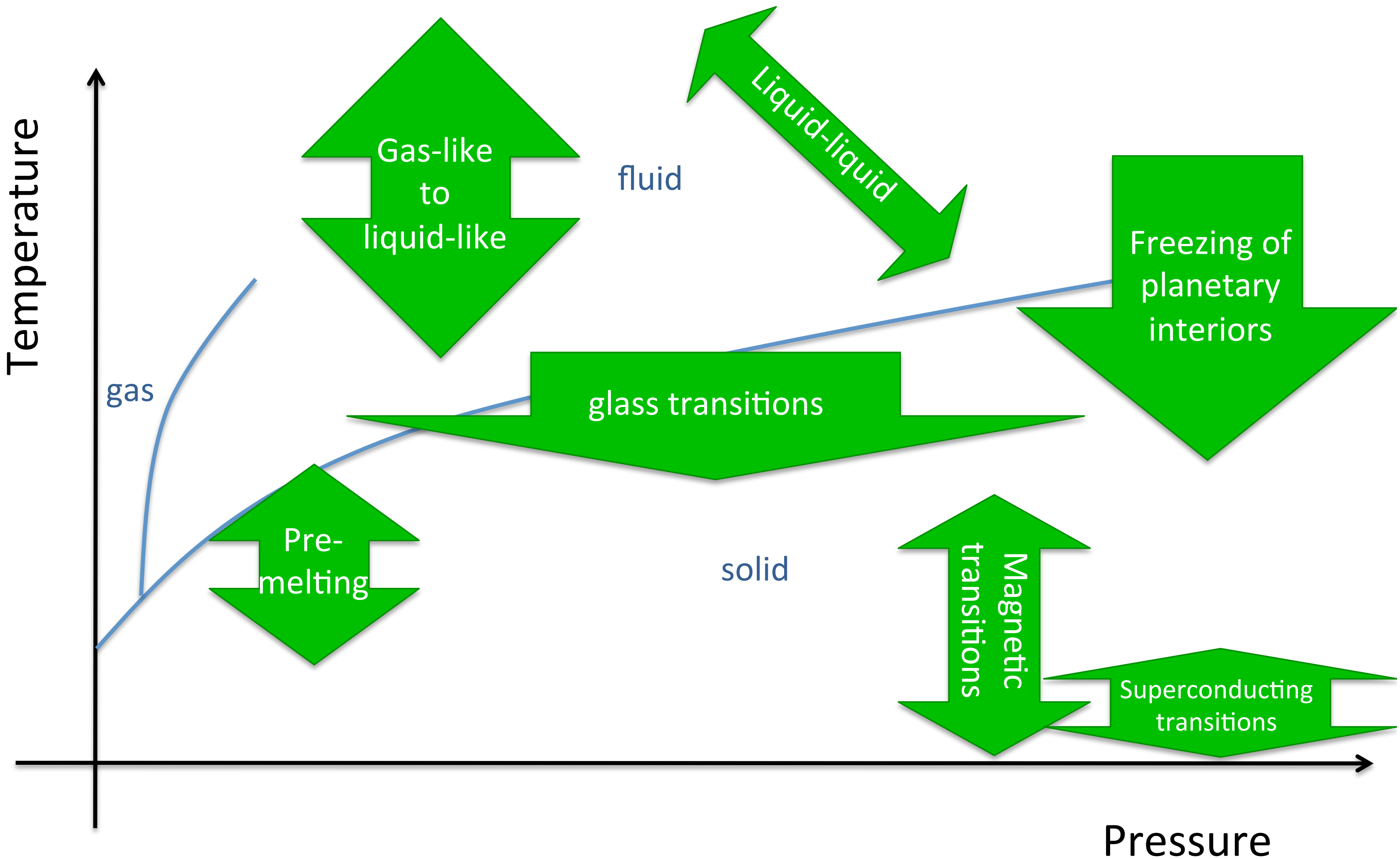
Not reproducible…yet



Ongoing work



Future work


Temperature
Pressure
gas
fluid
solid
Gas-like to liquid-like
Liquid-liquid
Freezing of planetary interiors
glass transitions
Pre-melting
Magnetic transitions
Superconducting transitions