STRAW Gauge: from UHV to XHV in One Package… R&D through Beta Testing

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Funding

Department of Energy SBIR Grant DE-SC0004437

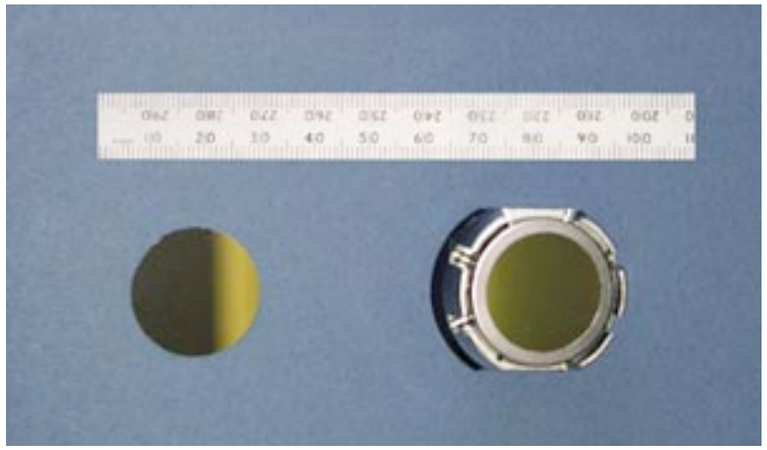
SBIR: Small Business Innovation Research → Phase I = 9 months: proof of principle
Phase II = 2 years: completion of project

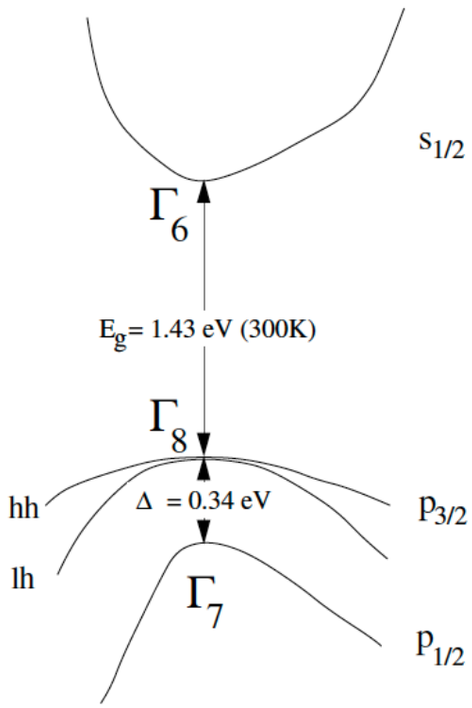
Outline

- Motivation
  - Nuclear physics e^- sources
  - Improved vacuum monitor
- Instrument components
  - Hardware
  - Software
- Instrument operation
  - In use
  - Calibration and data
- Sensor
  - Construction and operation
  - Details, details, details…
- Add-ons/spin-outs
  - Software
  - Hardware
- Summary

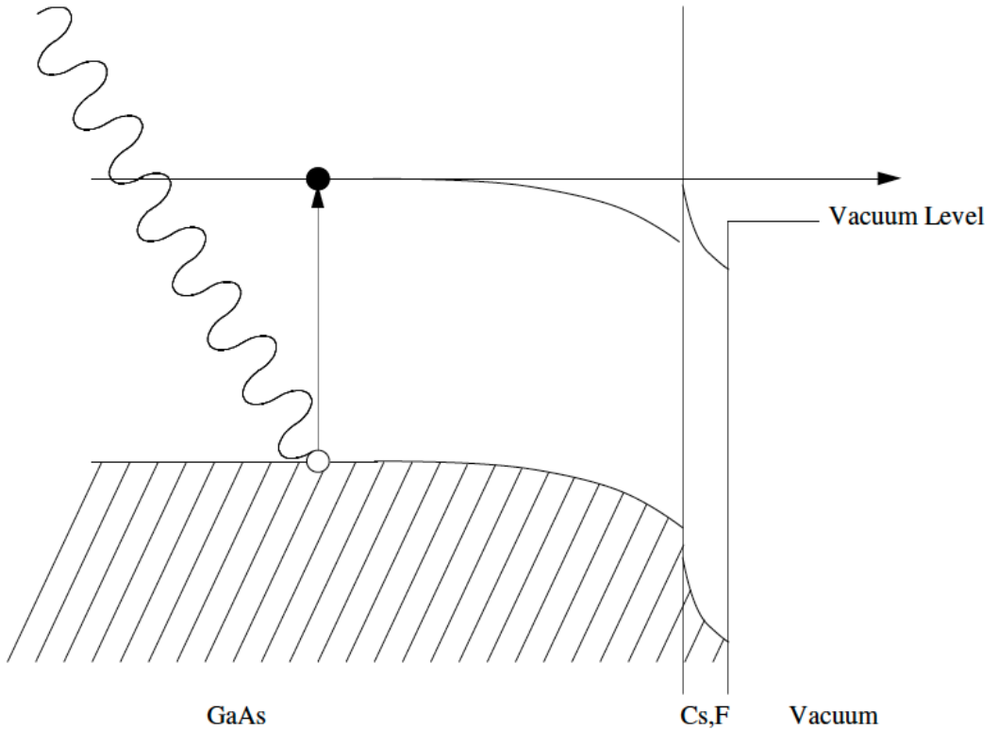
STRAW Vacuum Gauge

- Motivation: Monitor environment in nonperturbing manner
  - Photocathode
    - * Source of free electrons
    - * Electrons ejected upon photon absorption
    - * Anything with sufficiently high *hv*
  - Semiconductor (NEA GaAs)
    - * Activation allows bandgap energy excitation to generate free electrons
    - * Very sensitive to background gas contamination (shortened lifetime)
    - * Manipulation of crystalline symmetry enables high electron spin-polarization





GaAs band structure in vicinity of Γ point



NEA Surface

Gas + charged particles = damage!

Kinetic Damage

Chemical Changes

Gas + charged particles = damage!

Kinetic Damage

Chemical Changes

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Kinetic Damage

Chemical Changes

How to monitor extremely low pressures (UHV-XHV)?

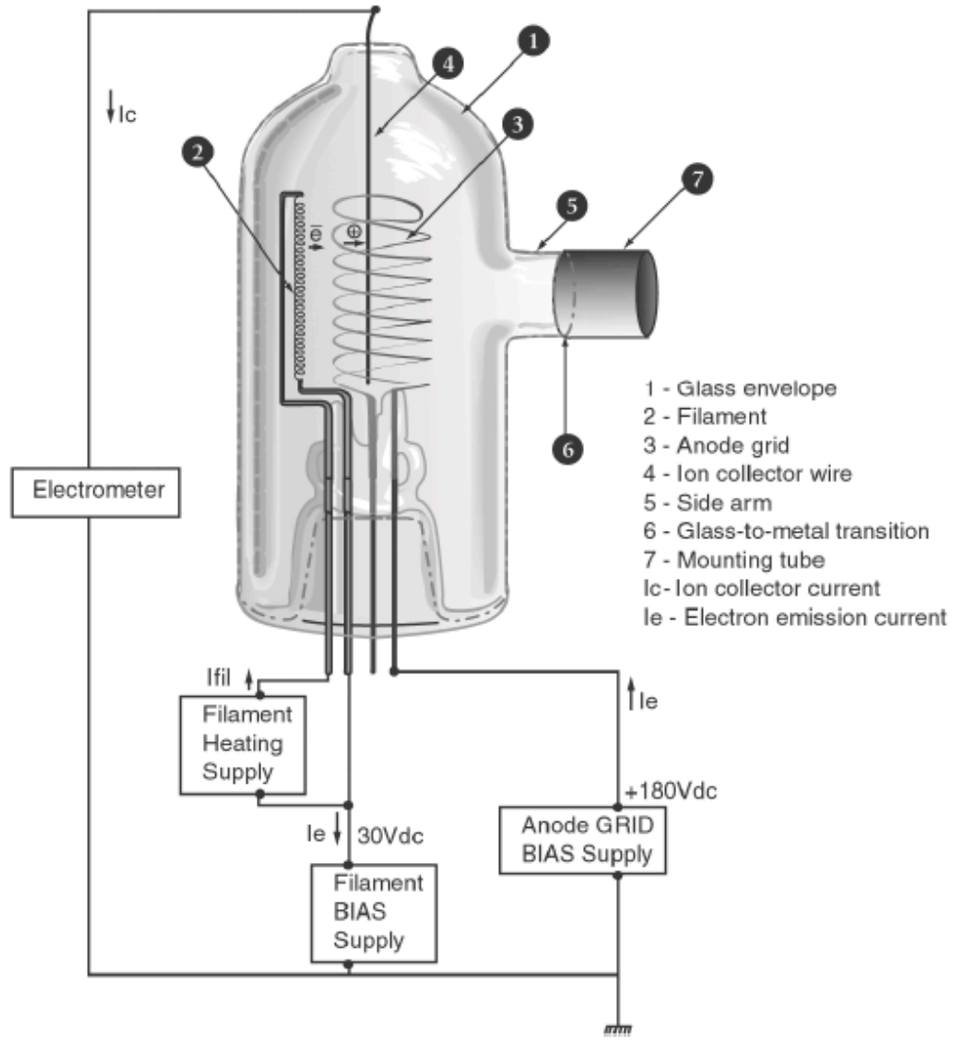
- Pressure ranges of interest: lower pressure = longer photocathode lifetimes
  - UHV (Ultra-high vacuum)

    $1x10^{-12}$ Torr < P < $1x10^{-9}$ Torr
  - XHV (Extreme-high vacuum)

    P < $1x10^{-12}$ Torr

- Ion gauge: particle and heat generation
  - Standard (Bayard-Alpert, $1x10^{-11}$ Torr, x-ray limit)
  - Extractor (Redhead 1960s, extends to $1x10^{-12}$, Leybold, Ulvac)
  - Radioisotope powered (no heater)

- Cold cathode gauge: particle generation
  - Commercial UHV (Balzers, $7.5x10^{-12}$ Torr)
  - Ion pump (Very low current monitoring: leakage currents, pump 'sleep')

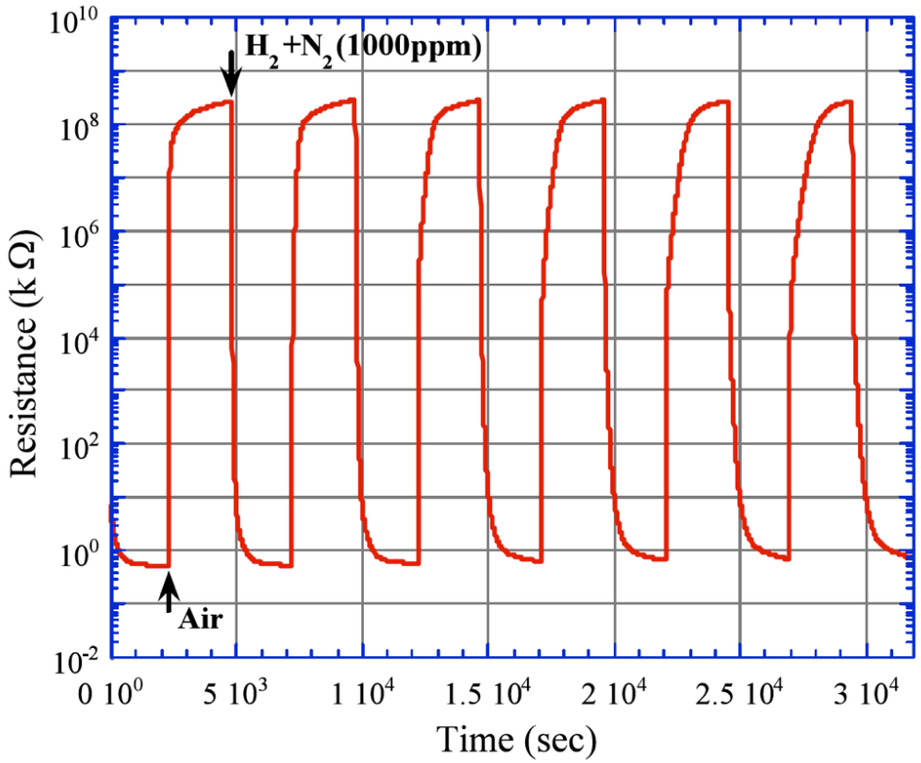


SRS Ion gauge diagram (IGC100 manual)

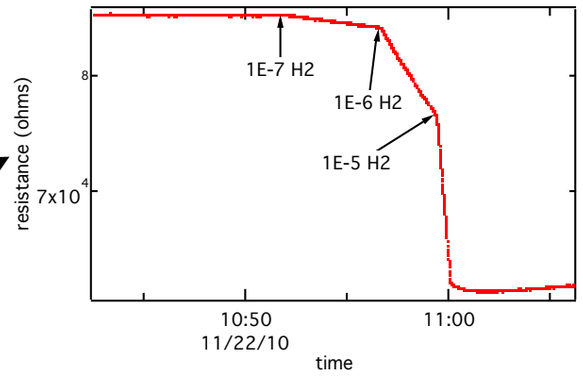
UHV/XHV gas primarily hydrogen

– Titania (titanium oxide) nanotube arrays have very strong response to hydrogen at STP

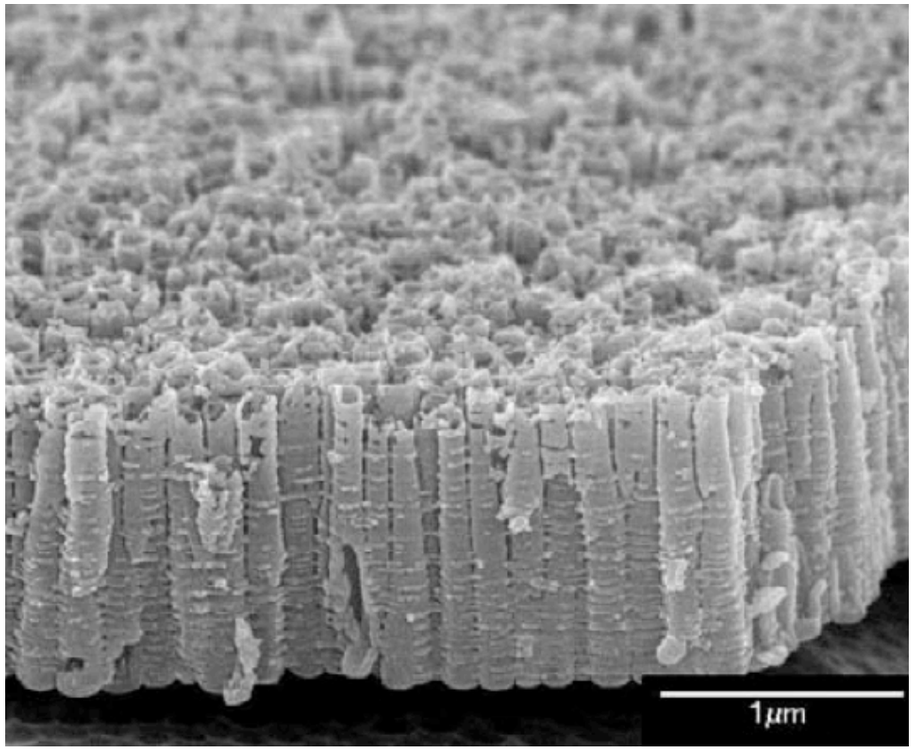
– Phase I result: even stronger than anticipated in vacuum



Time response of resistance for hydrogen purged (dry air) and hydrogen dosed (H_2 + N_2) using 1000 ppm of hydrogen [figure 3 of M. Paulose, O.K. Varghese, G.K. Mor, C.A. Grimes and K.G. Ong, Unprecedented Ultra-High Hydrogen Gas Sensitivity in Undoped Titania Nanotubes, *Nanotechnology* **17**, 398(2006)]



Vacuum hydrogen response of titania nanotubes. Rate is proportional to pressure.



SEM cross section of titania nanotubes

- Instrument components

– Components
  * Sensor head
  * Custom designed control unit
  * Software for DAQ

Similar form factor to modern RGA systems, e.g. MKS Microvision.

– Hardware

* Sensor head

  Vacuum feedthrough with electrical and optical transport

  Device bias/measure, thermocouple and illumination

* Control box

  Custom designed circuit board: source/measure

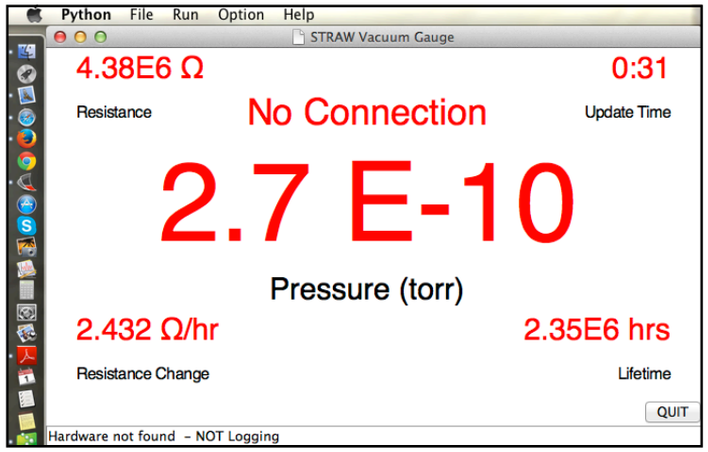
  using 32 bit PIC running @ 20 MHz

* Power supplies/cabling

  Separate power conversion box (AC to DC) to minimize noise

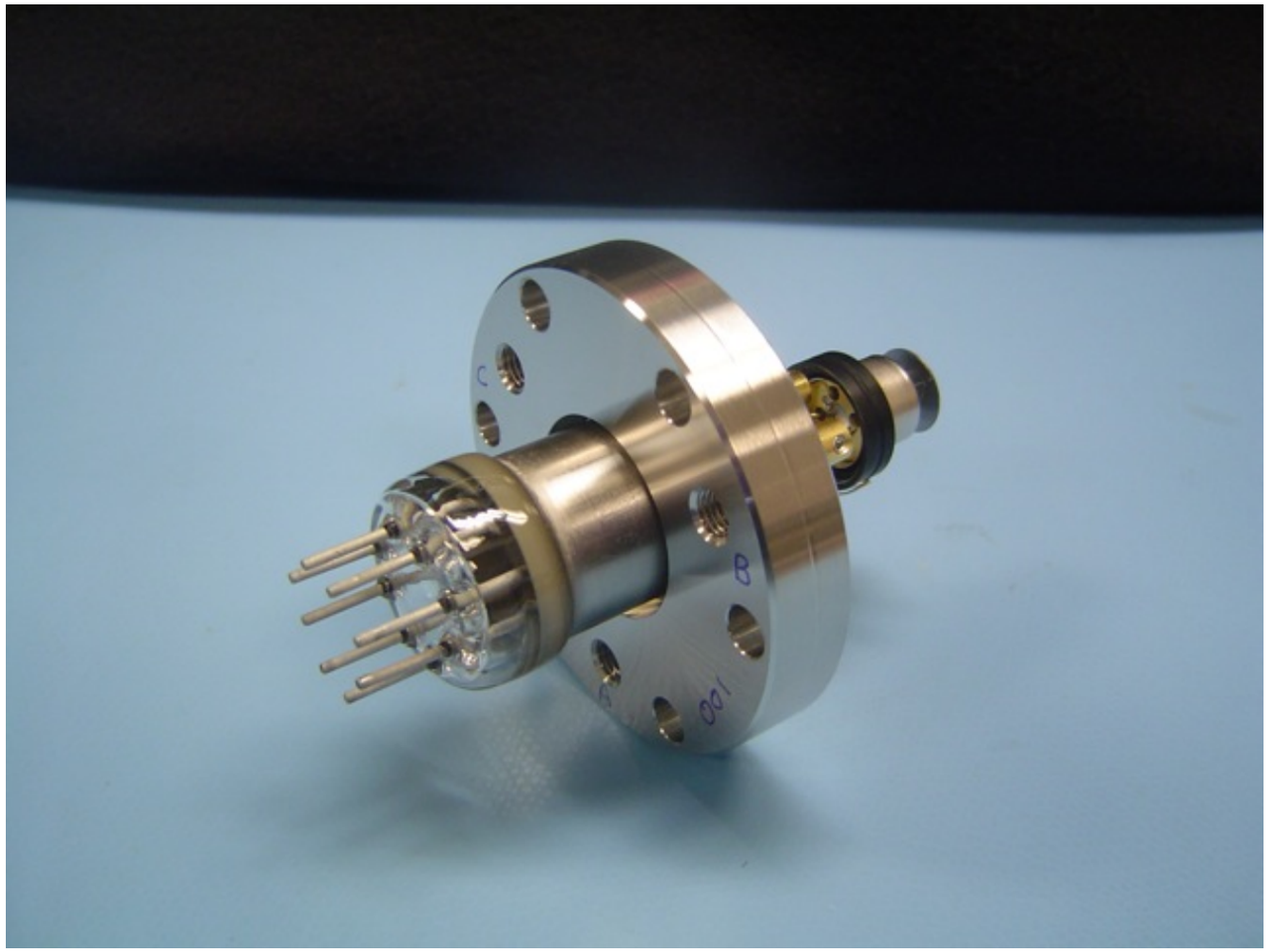
– Software

* Onboard controller firmware (programmed in C)
* Python for computer interface (NumPy + SciPy)
* Packaged installer (Windows via Py2exe)
* Manual install for Mac or Linux



STRAW Control Box Disassembled
(optic plate not shown)

* Sensor head flange
  -- SST 304L 2-3/4” Conflat type: Larson Electronic Glass Products
  -- Glass feedthrough functions as optical window to back of nanotube array
  -- Pin configuration is that of the old Loctal vacuum electron tube
  -- Air side threaded holes for control box support rods



STRAW gauge sensor head flange
Air side is to left, vacuum to right

Loctal socket (front)

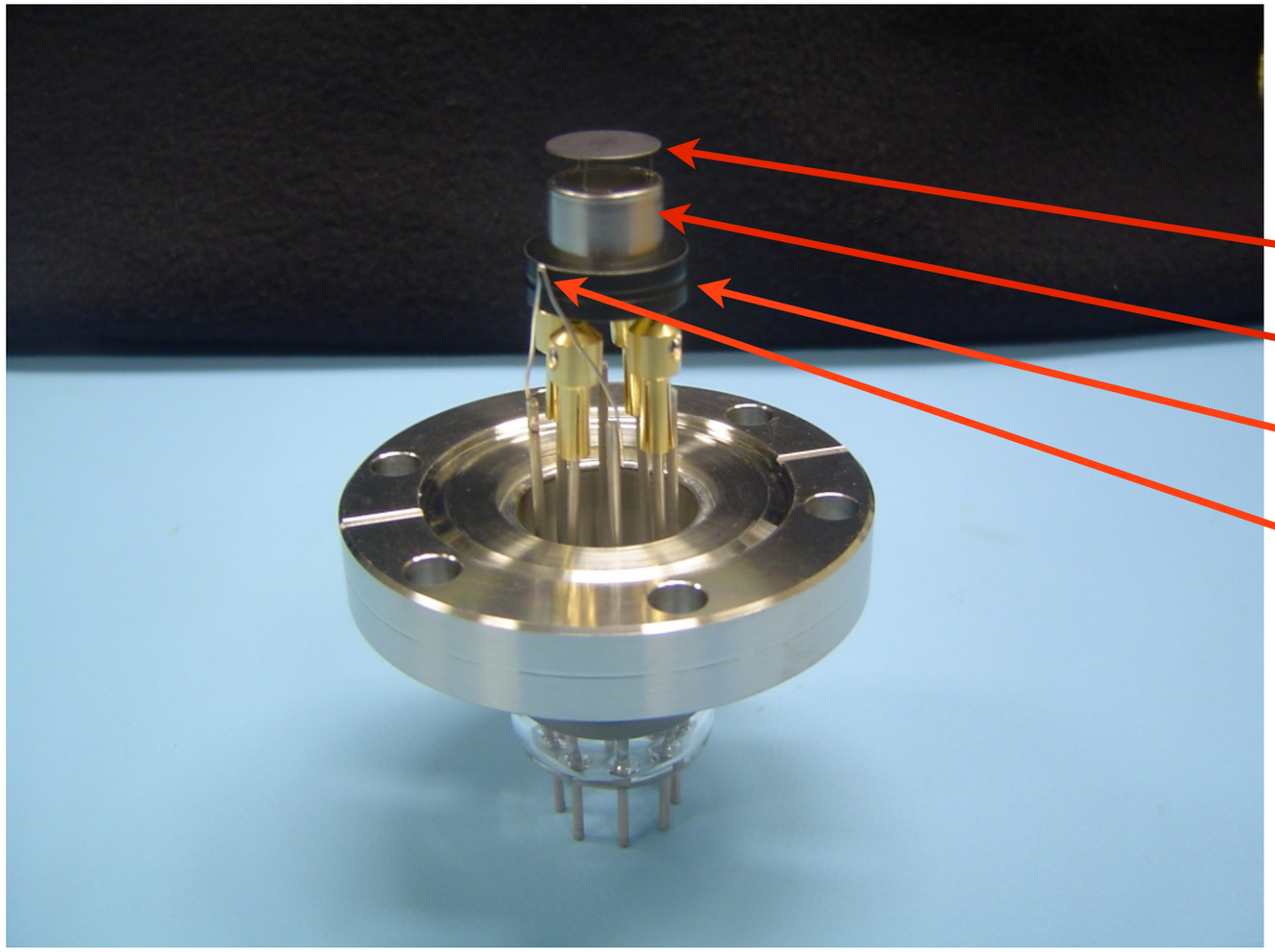
* Sensor head flange connections

-- Titania nanotube array sensor is mounted on TO8-550 header

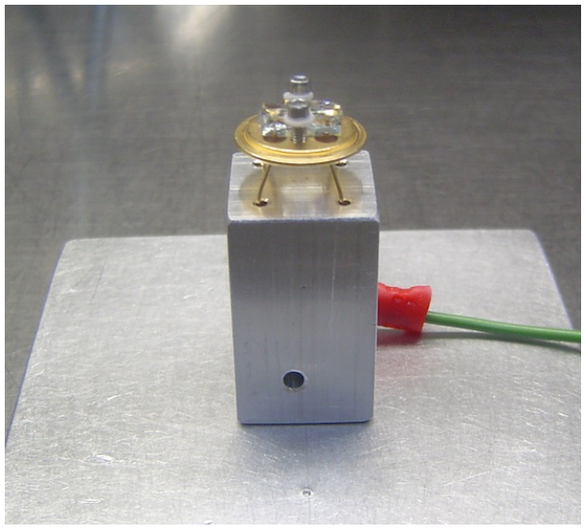
-- Sensor in mounted inside of physical shield with top hat light baffle

-- Near-device temperature is monitored by type K thermocouple

-- All four legs of the header are wired to the air side; only one pair used at a time



STRAW gauge sensor head flange
Sensor is mounted inside of shield



Leg alignment jig

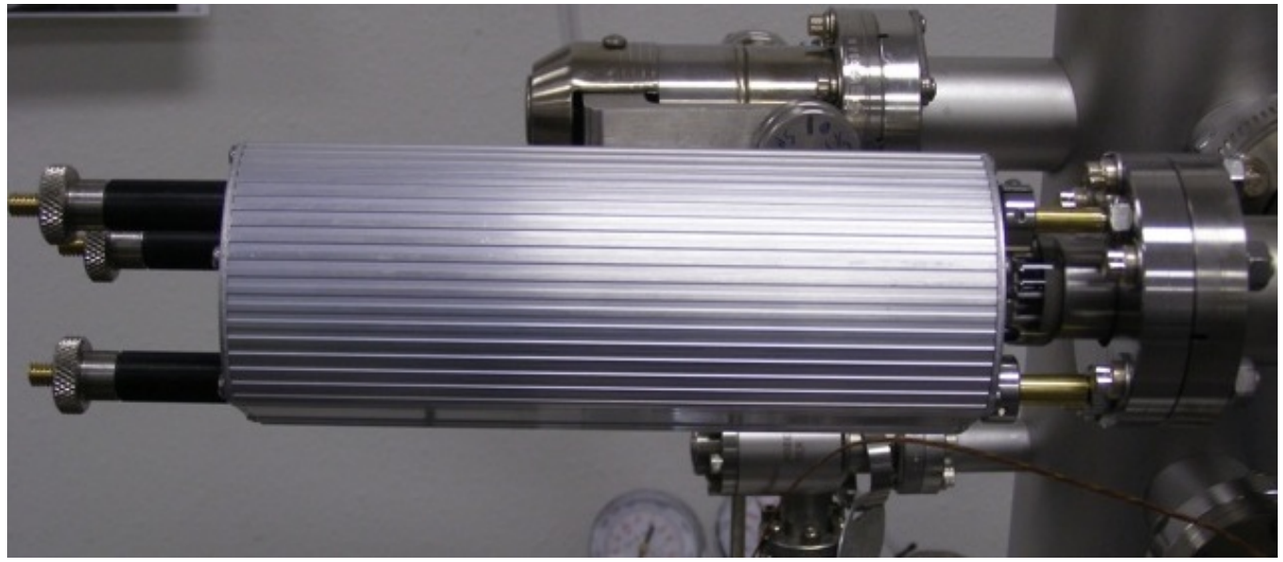
* Control box

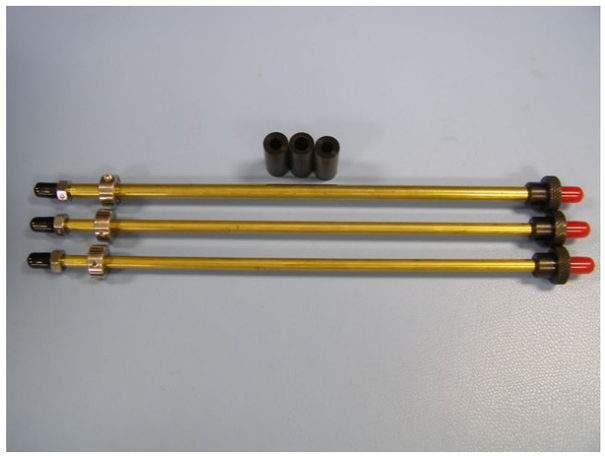
-- Externally powered, flange mounted chassis

-- PIC controlled, USB-to-serial communicating measurement board (COM emulation)

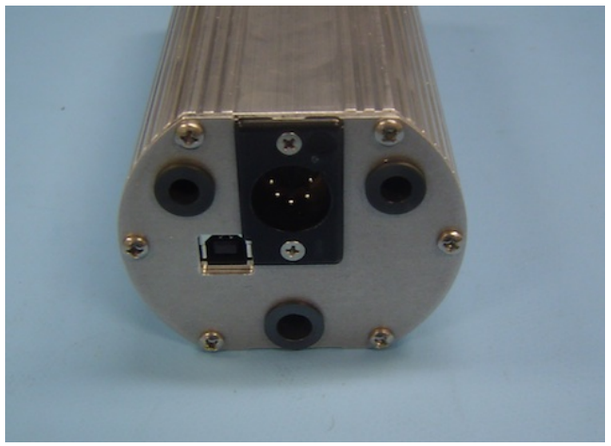
-- Onboard thermocouple readout

-- Optical boost LED output with photodiode monitor

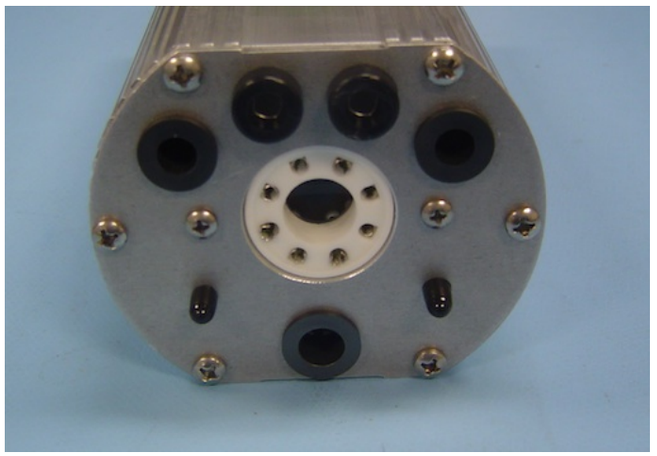




Support rod assembly

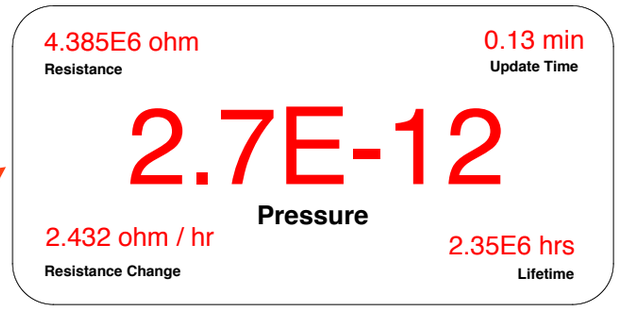


Rear of control box



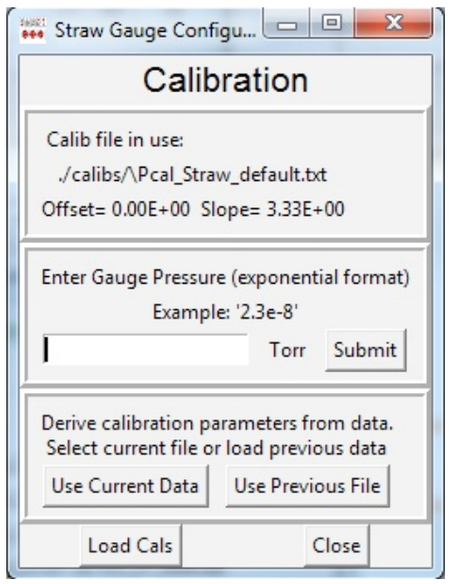
Front of control box

* Software
  - -- PIC firmware developed in C
  - -- Host computer control program developed in Python
  - -- Data logging as standard
  - -- Cross calibration data input by end user
  - -- Conductance replaces resistance (X vs. 1/X)

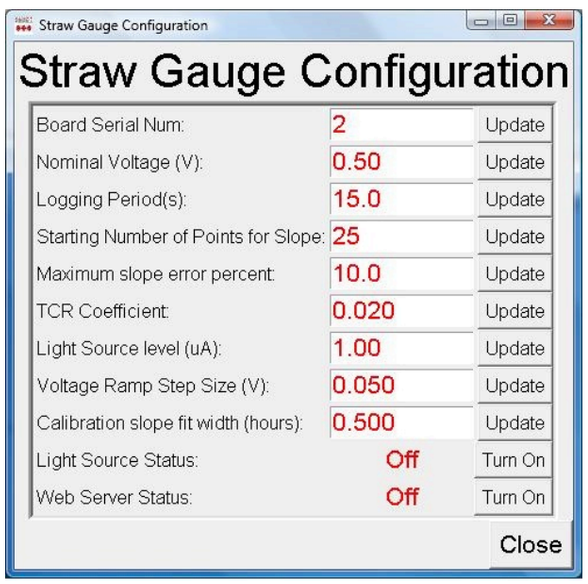


Mockup of main display window from Phase II proposal

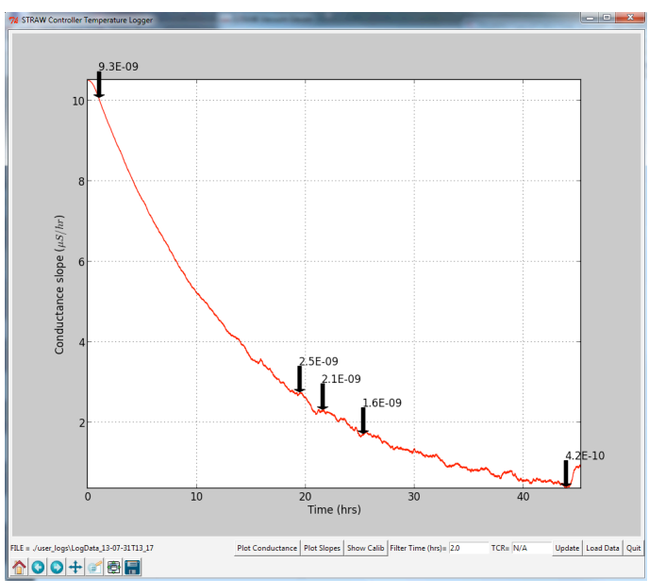
Main Display



Calibration panel

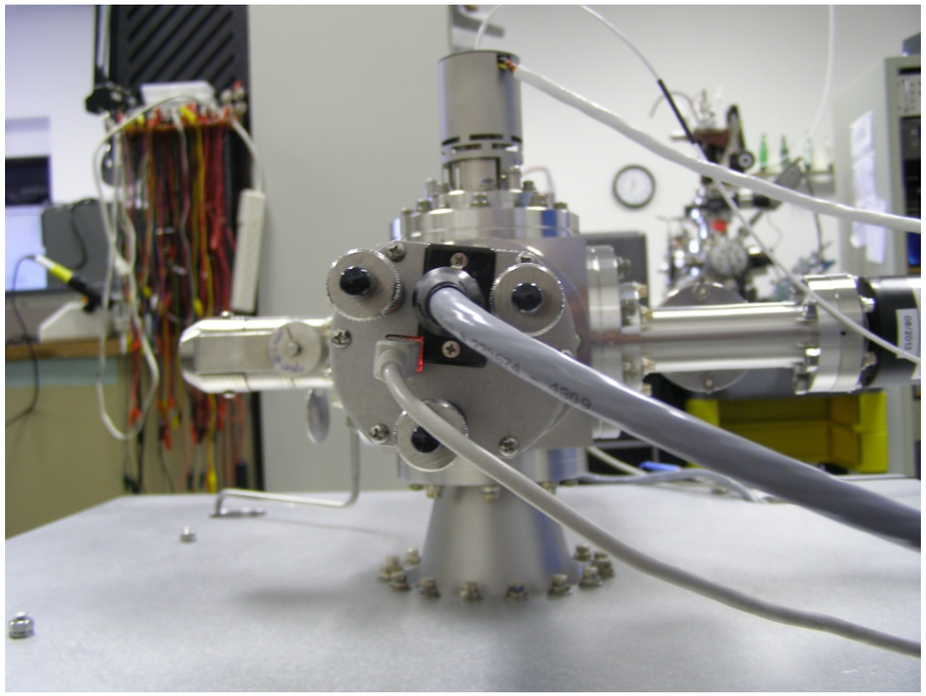


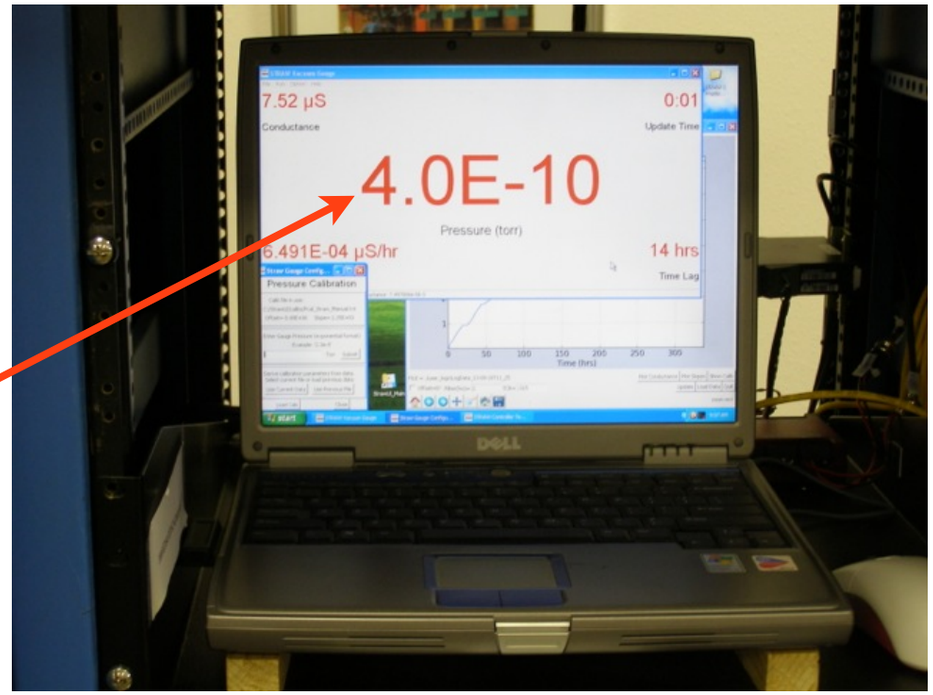
Configuration panel



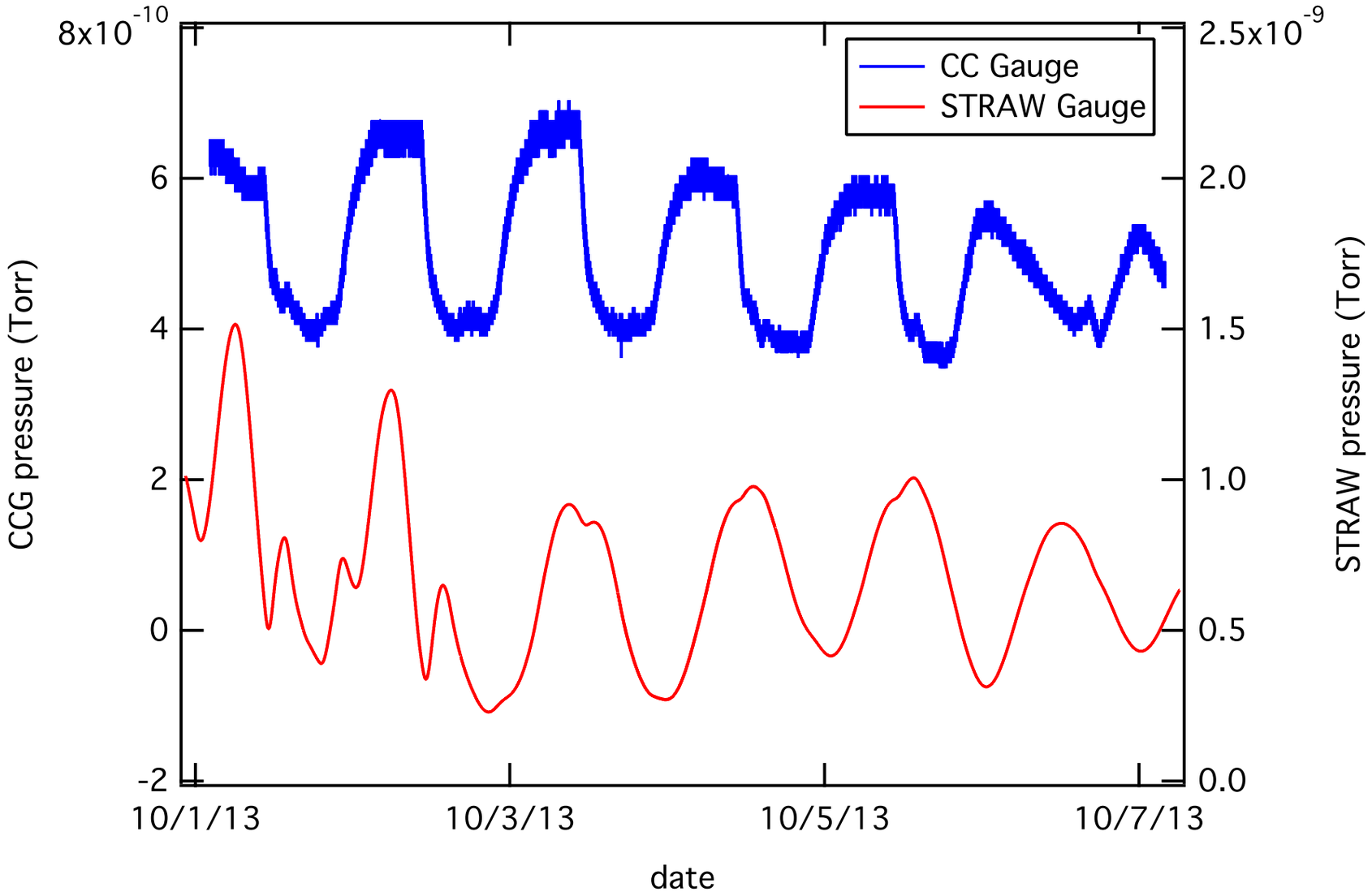
Conductance slope vs time

- Instrument in operation





STRAW UI on low end laptop running Windows XP



Comparison of pressure vs time for the STRAW vs. CCG

- Sensor
  - Construction/nanotube array

    * Titania (TiO_2) nanotubes anodically grown from Ti film

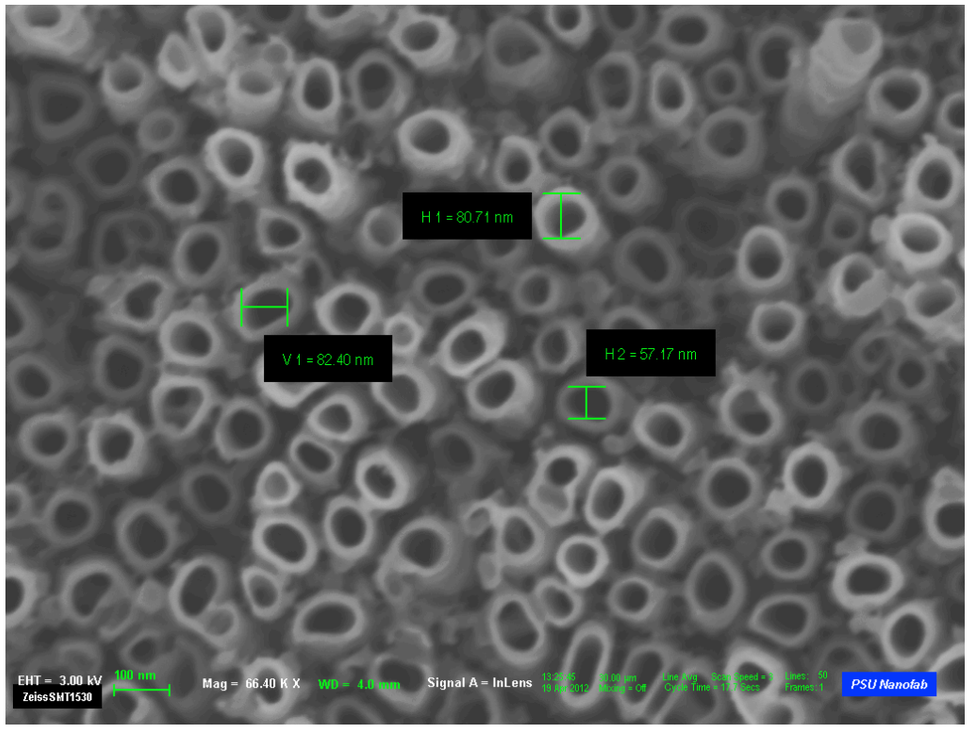
    * Wall thickness comparable to space charge length (all surface, no bulk)

    * Conduction along nanotubes dependent on surface chemistry

    * Absorption of hydrogen facilitated by Pd nanoparticles + Pt contacts

    * Resistance change proportional to hydrogen absorption

SEM photograph of the titania nanotubes end-on from the devices with pores an average size of 70 nm Better hydrogen response in vacuum is obtained from the devices with smaller pore sizes.



– Construction/connectorizing

* Glass substrate clamp mounted on TO8-550 header (Kovar, Au, glass)

* Anodized films are ~transparent

* Pt contact pads Au wire bonded to header pins

– Operational electrical characteristics

* Signal conversion very simple! V = IR (or V=I/G)

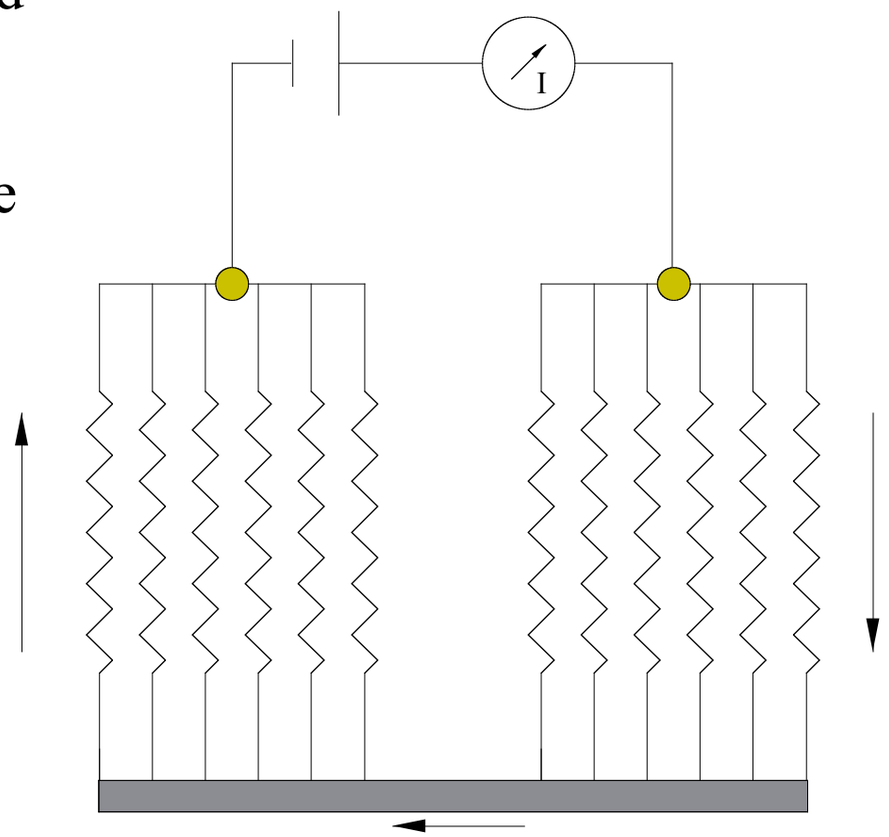
* Operational parameters: Bias = 0.40 V, I ≤ 20 μA

* Tolerant device, but best if handled as ESD sensitive

* Single point grounding used to eliminate ground loop noise

* Most other electrical noise eliminated by simple signal averaging (100x)

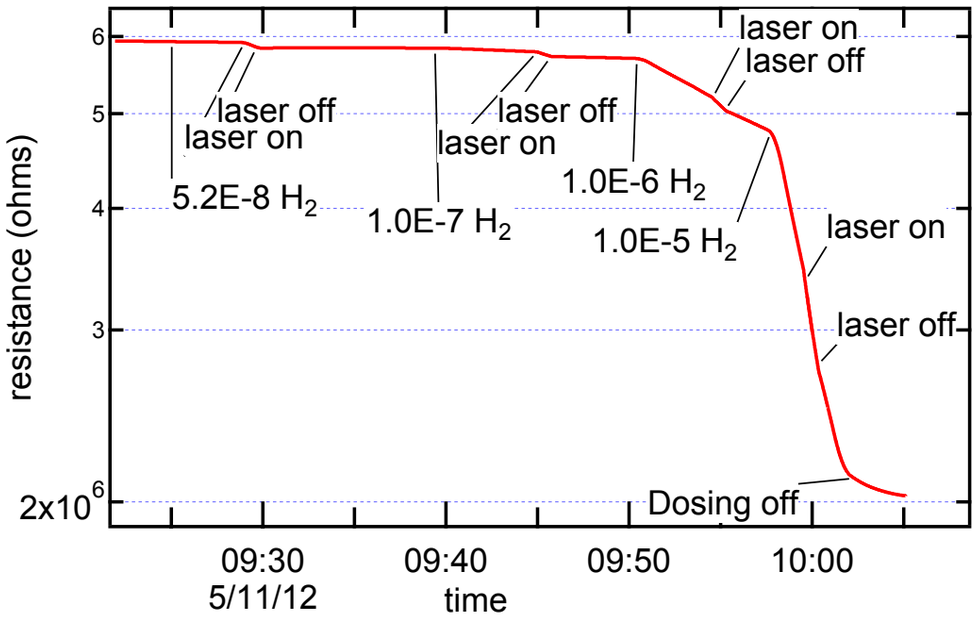
* Stability of high quality off-the-shelf measurement instruments sufficient



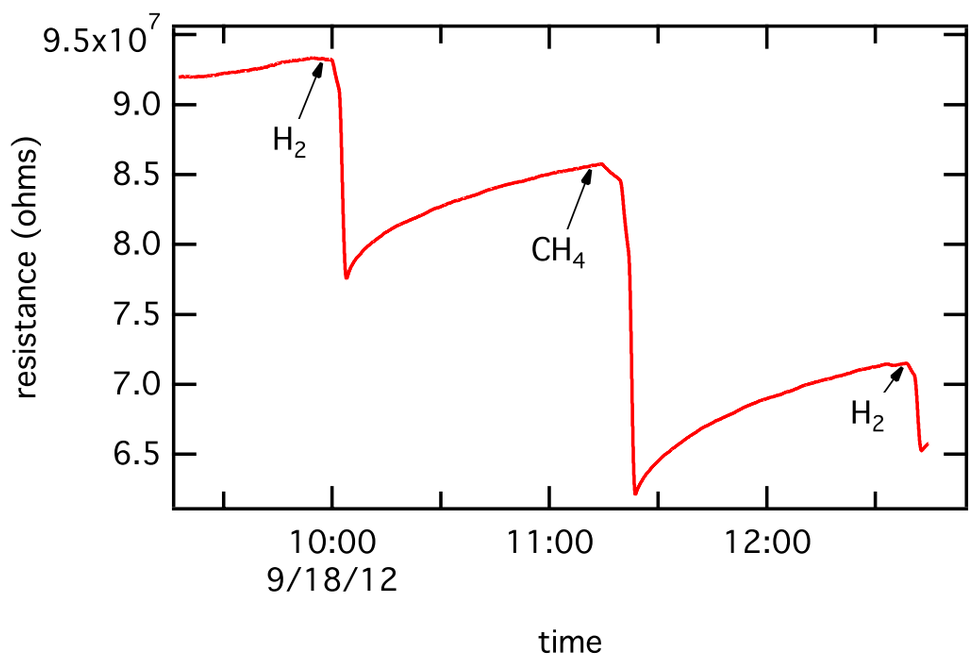
Simplified 1D diagram of nanotube array electrical circuit. Each parallel cluster is made up of the nanotubes under the individual Pt contact pads.

- Details, details, details

* Sensitivity boost for lowest pressure operation



Enhanced sensitivity via illumination

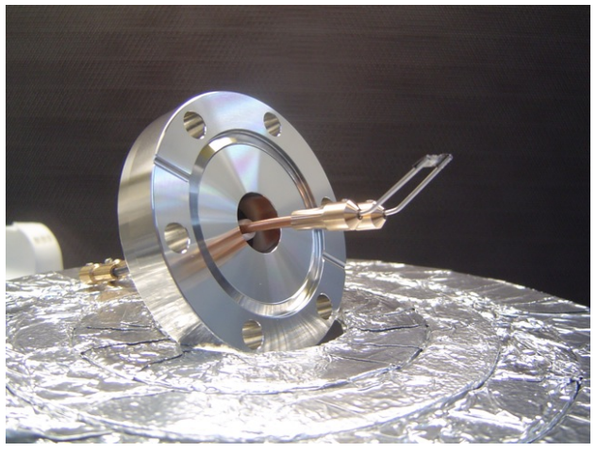


* Other gasses influence sensitivity

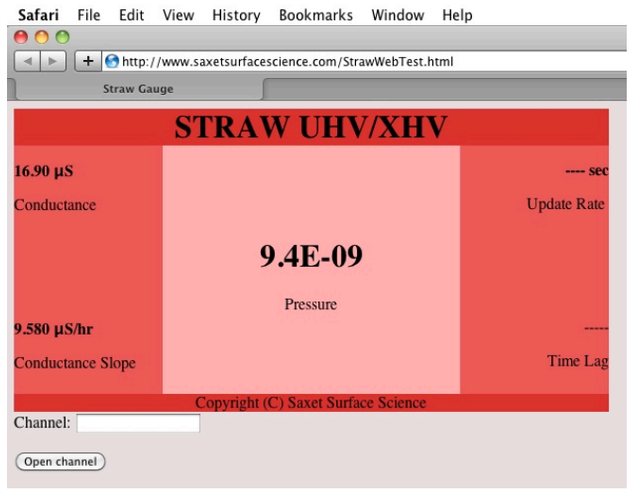
CH_4 yes, CO yes, CO_2 no

* Titania is semiconducting, large TCR

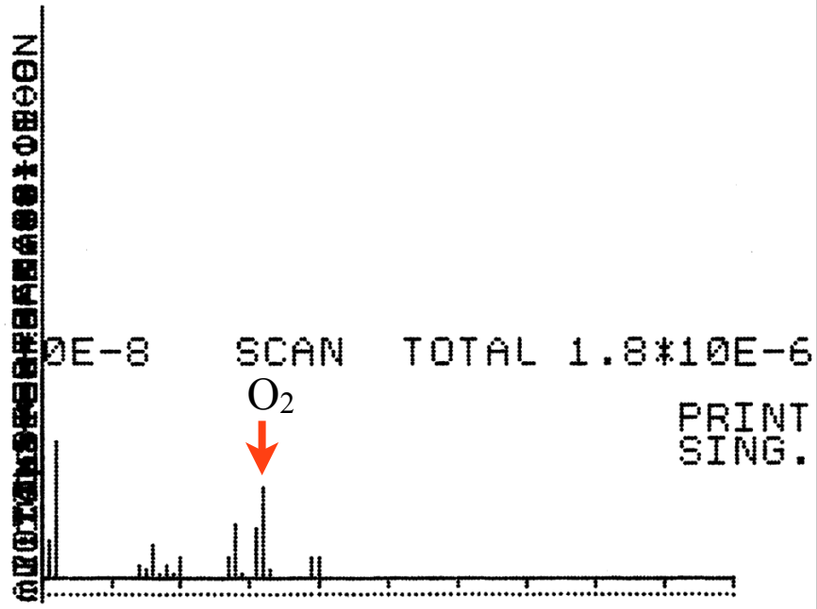
- Add-ons/Spin outs
  - Integral oxygen source for hydrogen absorption reversal. Silver oxide in stainless steel sleeve. One shot only!



  - Web-based interrogation of host computer



  - Remote readout of pressure with ‘stale’ data watchdog



RGA spectrum while operating oxygen source

  - Market control board as low cost low voltage alternative for semiconducting nano-device I-V characterization
    Stability meets or exceeds that of commercial instruments (multi-task, so much higher $$$)

Saxet

Saxet is a sole proprietorship founded in 2002

– Who did the work?

Greg Mulhollan

Program management

Robert Kirby

Vacuum technology expert

John Bierman

System construction/measurement

Andrew Milder

Control system

– Where was the work done?

– 1,982 ft^2 in the Pecan Business Park in south Austin

– Texas is the 28th state and was a republic prior to statehood

– Austin is home to the Texas state capital, the University of Texas and many high-tech companies including Samsung (flash memory chips & mobile device processors)

Summary

- STRAW UHV/XHV vacuum gauge system constructed
  - Does not perturb system
  - On-flange controller, host computer readout

- Titania nanotube sensor technology
  - High hydrogen sensitivity, with optical boost even better
  - Sensor technology tested, tested and tested some more!

- One unit is out currently for beta testing

- Future
  - Phase IIB or not to Phase IIB?
  - Commercialization
    * Direct sales
    * Two entities shown interest as product line

- Measuring pressure at UHV and XHV is not easy!

Atmosphere to 10^{-12} Torr requires a drop of 15 orders of magnitude in pressure.

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Going beyond normal limits…

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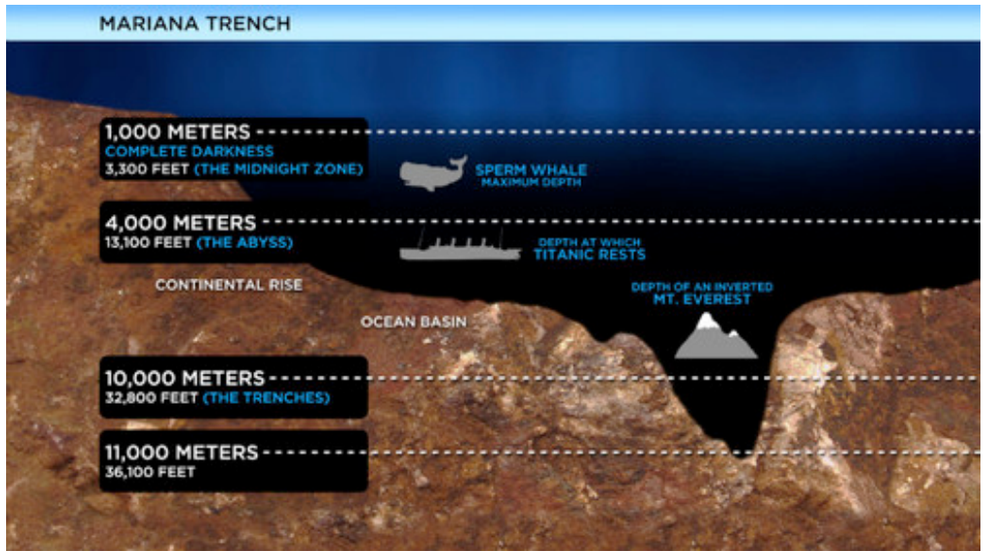

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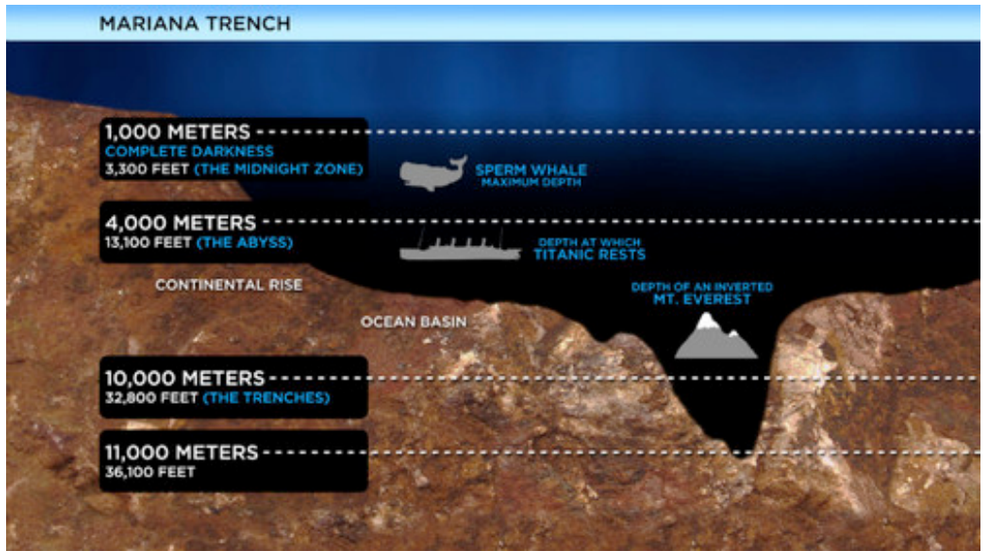

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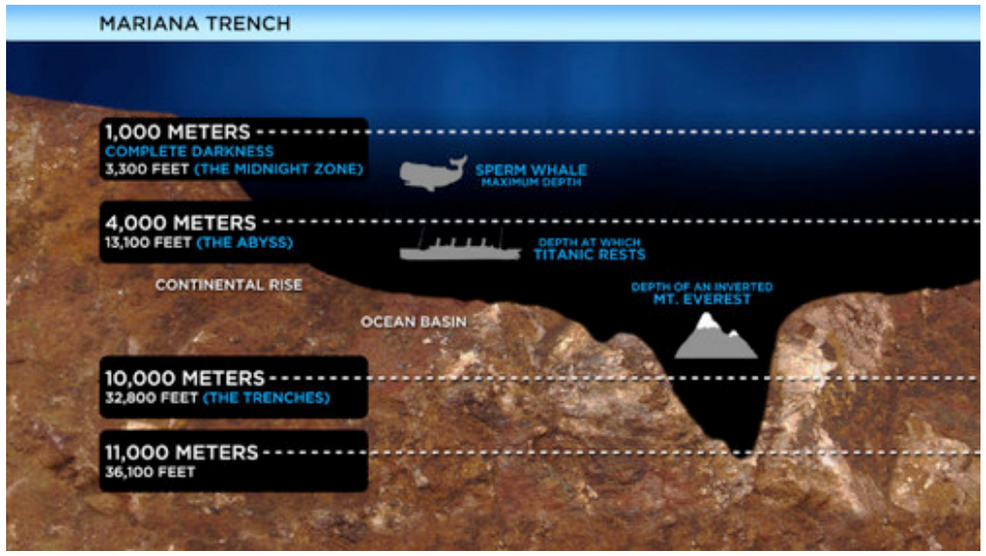

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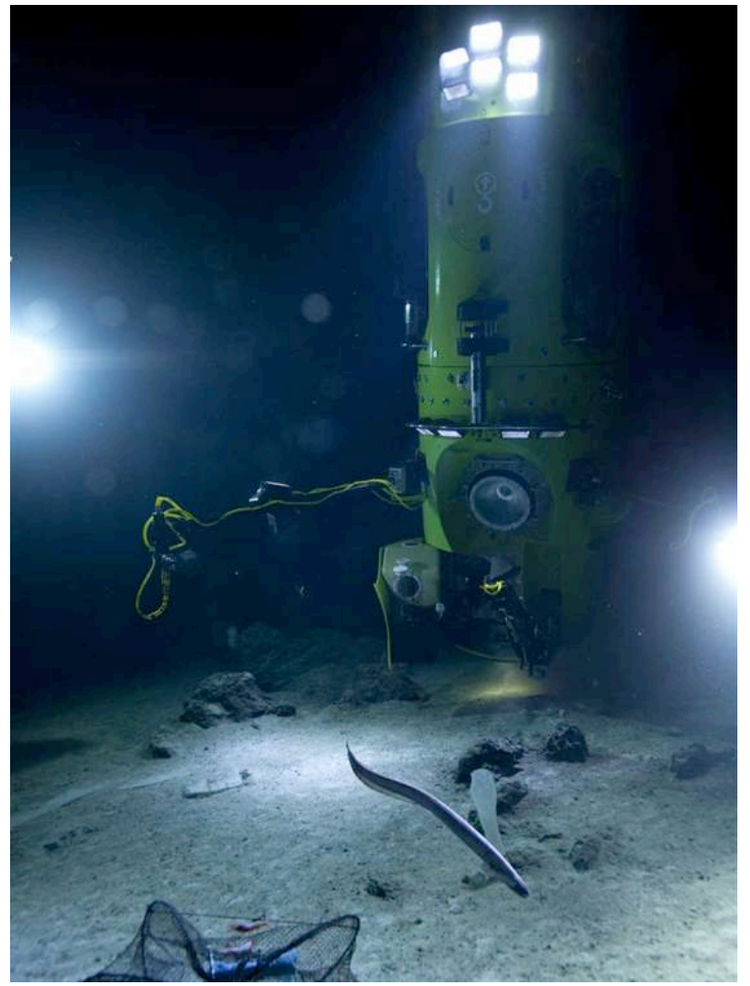

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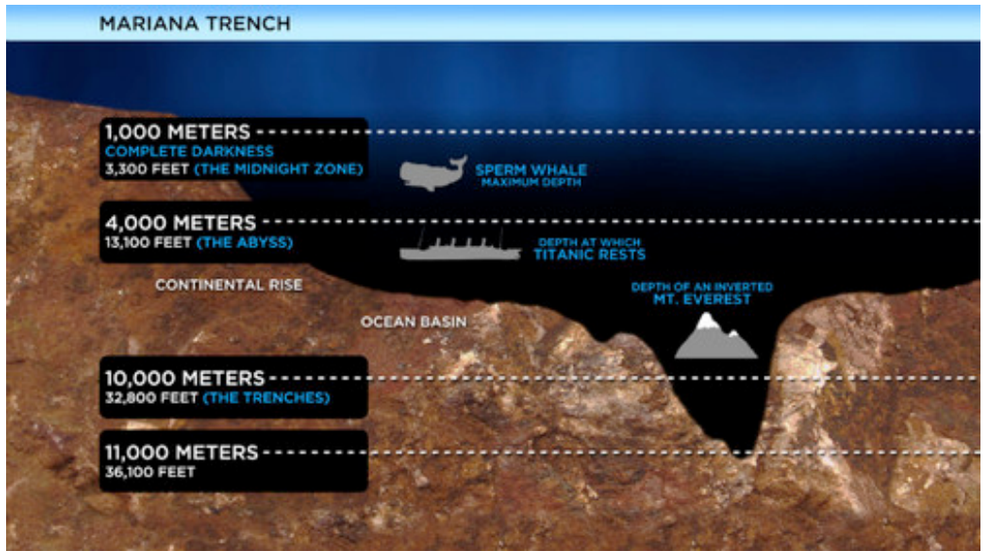

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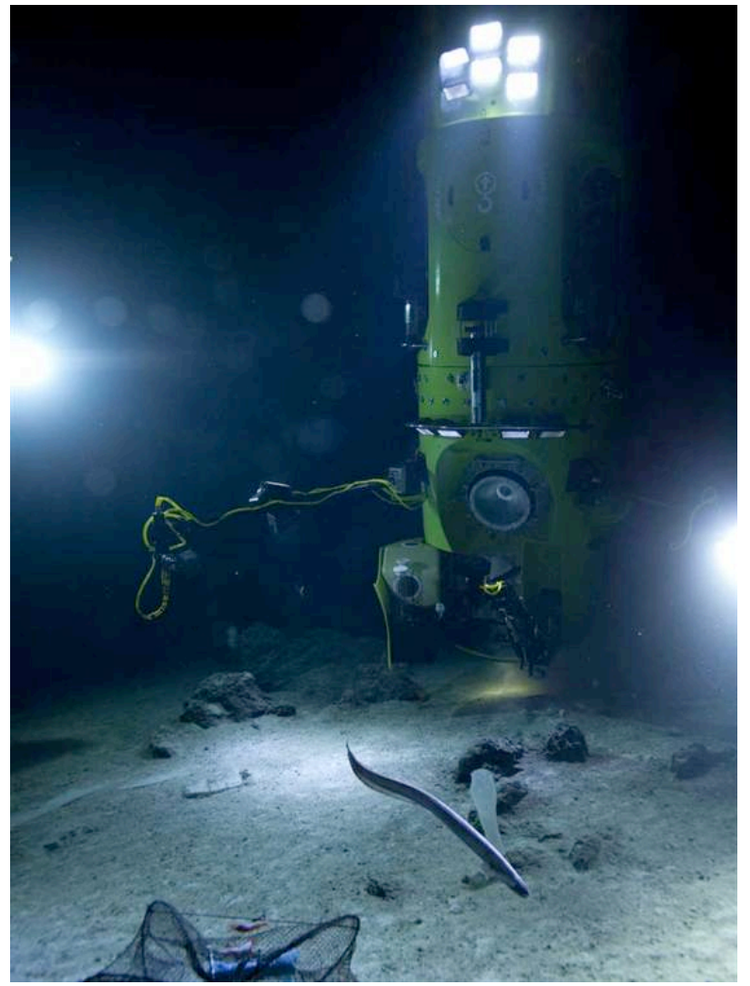

Requires specialized equipment…

And a good deal of nerve!

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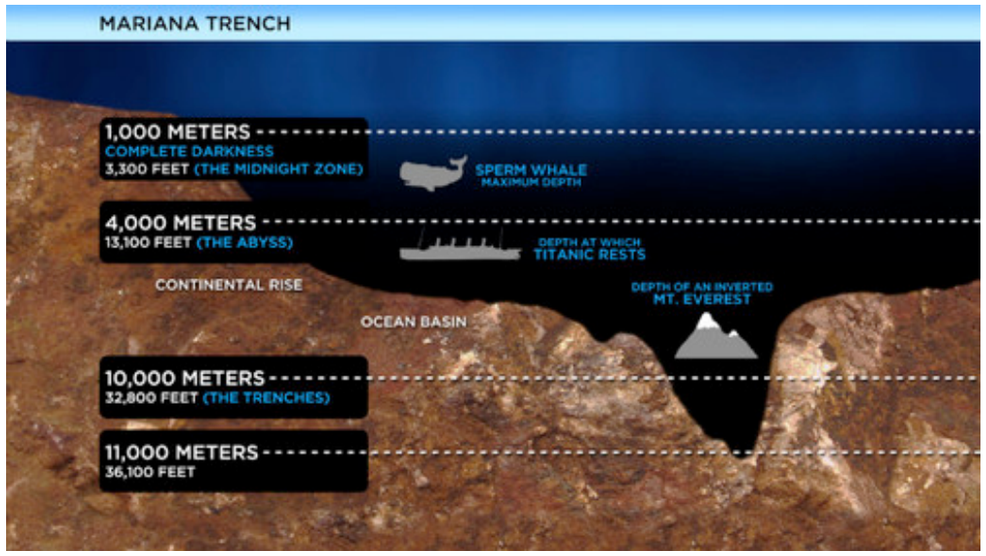

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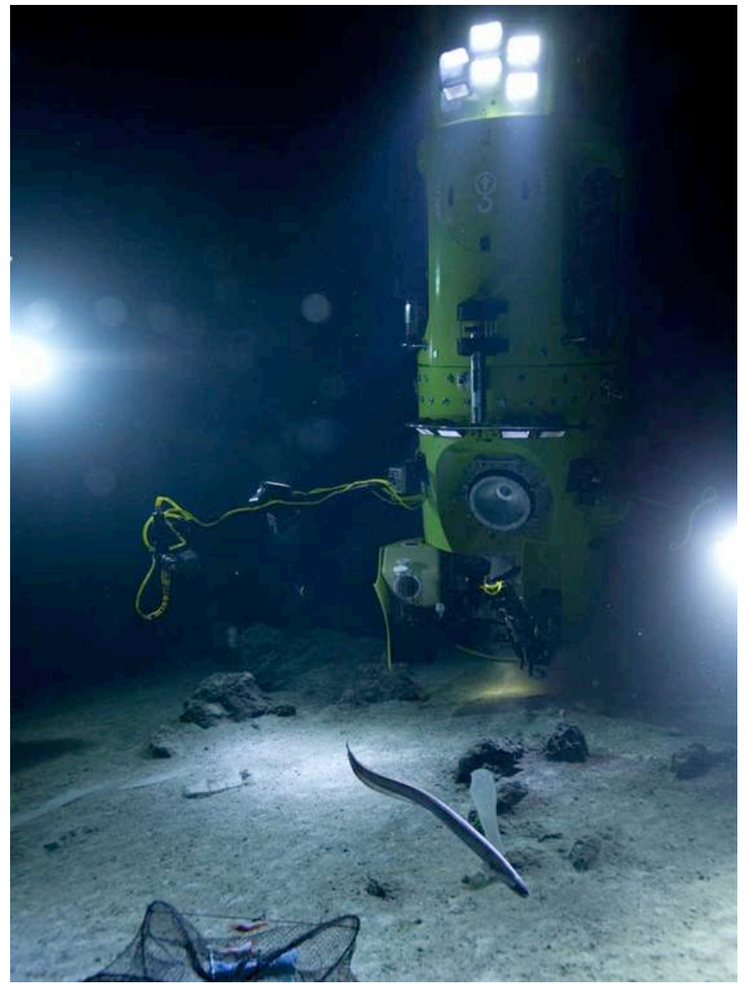

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