National Physical Laboratory

Introduction to metrology

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Materials Division
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National Physical Laboratory, Teddington, UK

UK thermoelectric Network -20 April 2016

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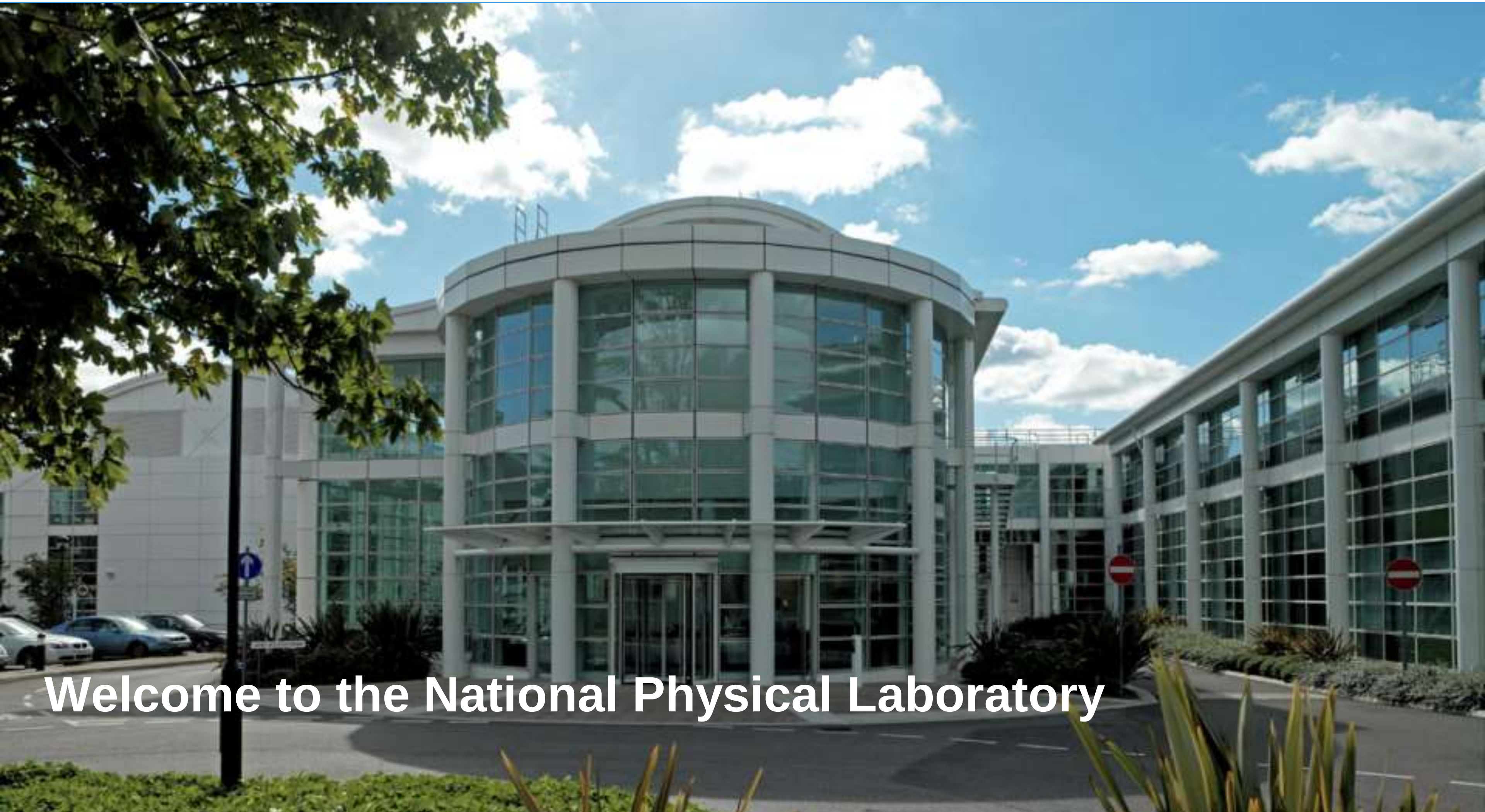

Welcome to the National Physical Laboratory

What do we do

NPL National Physical Laboratory

- Develop & disseminate UK's measurement standards, ensure they are internationally accepted
- Multidisciplinary R&D and technical services for public and private sector
- Knowledge transfer and advice between industry, government and academia
- Promotion of science and engineering




The importance of being … quantitative

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NATURE SERIES

POPULAR LECTURES

AND

ADDRESSES

BY

SIR WILLIAM THOMSON, LL.D., F.R.S., F.R.S.E., &c.

PROFESSOR OF NATURAL PHILOSOPHY IN THE UNIVERSITY OF GLASGOW, AND FELLOW OF ST. PETER'S COLLEGE, CAMBRIDGE

IN THREE VOLUMES

VOL. I.

CONSTITUTION OF MATTER

WITH ILLUSTRATIONS

SECOND EDITION, WITH ADDITIONS AND CORRECTIONS

London

MACMILLAN AND CO.

AND NEW YORK

1891

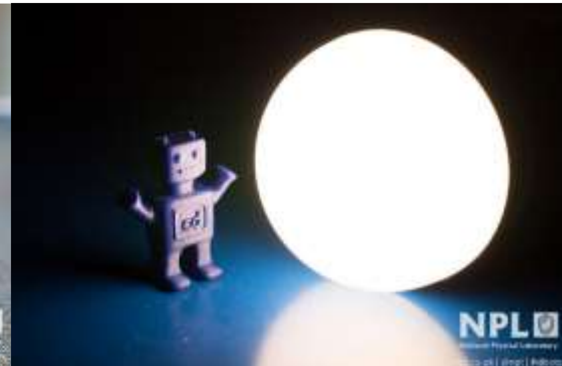
https://ia600205.us.archive.org/35/items/popularlectures10kelvgoog/popularlectures10kelvgoog.pdf

ELECTRICAL UNITS OF MEASUREMENT.

[*A Lecture delivered at the Institution of Civil Engineers on May 3, 1883; being one of a series of Six Lectures on "The Practical Applications of Electricity."*]

IN physical science a first essential step in the direction of learning any subject is to find principles of numerical reckoning and practicable methods for measuring some quality connected with it. I often say that when you can measure what you are speaking about and express it in numbers you know something about it; but when you cannot measure it, when you cannot express it in numbers, your knowledge is of a meagre and unsatisfactory kind: it may be the beginning of knowledge, but you have scarcely, in your thoughts, advanced to the stage of *science*, whatever the

NPL is the UK home of the SI



The NEW SI (2018) : from seven base units to seven fundamental constants

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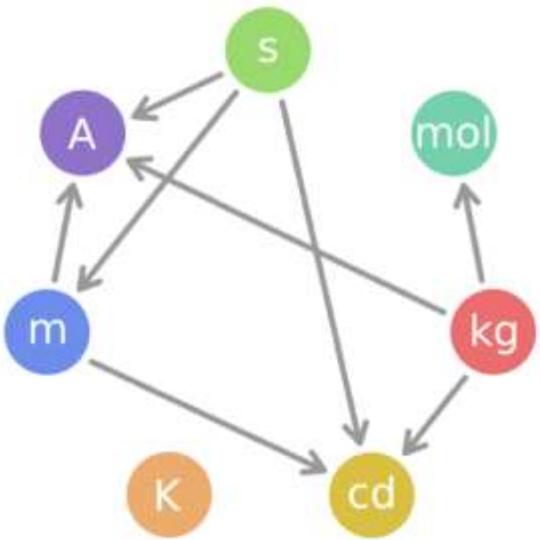
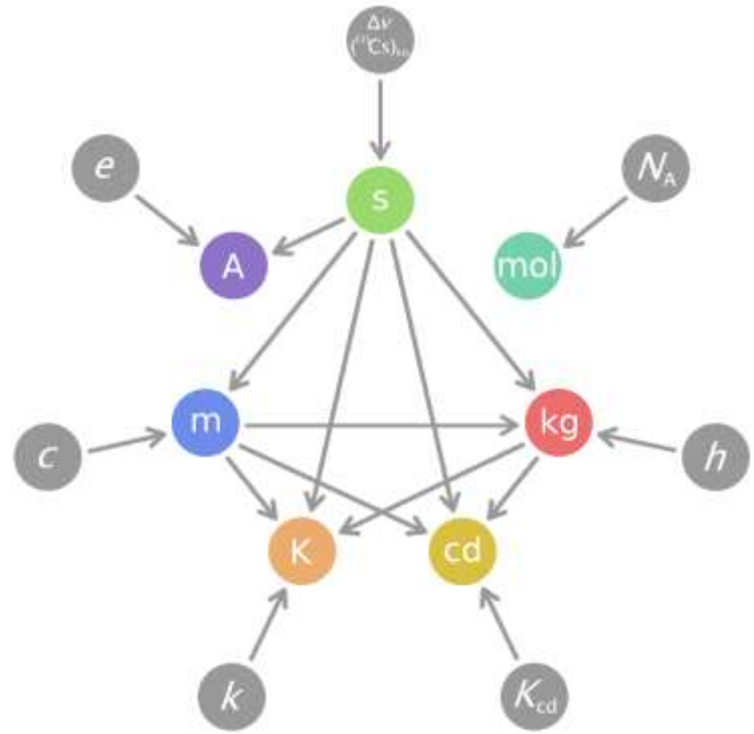


Current (2016) SI system: Dependence of base unit definitions on other base units for example, the metre is defined in terms of the distance traveled by light in a specific fraction of a second



New SI system: Dependence of base unit definitions on physical constants with **fixed numerical values** and on other base units that are derived from the same set of constants.

NPL’s Position

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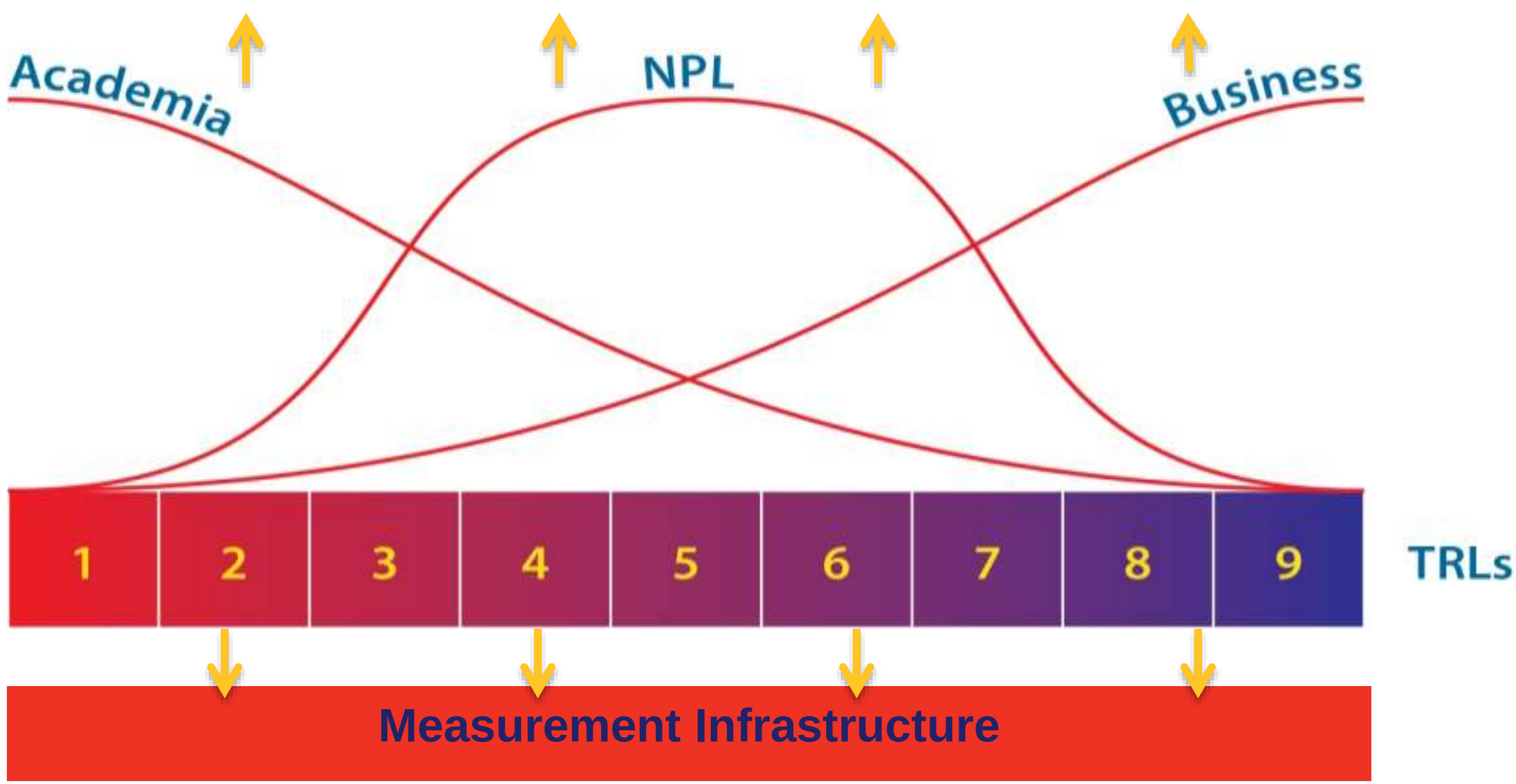
Supporting innovation

Concept — Characterisation, Leading-edge science, Facilities

Development — Prototype, Testing, Validation

Manufacturing — Quality assurance, Process, Optimisation

Trading — Standards, Regulation



The objectives of metrology

- Measurements that are stables

    Long-term trends can be used for decision making

- Measurements that are comparable

    Results from different laboratories can be brought together

- <u>Measurements that are coherent</u>

    Results from different compounds and from different methods can be brought together

These are achieved through providing the infrastructure to support traceable measurement results with uncertainties.

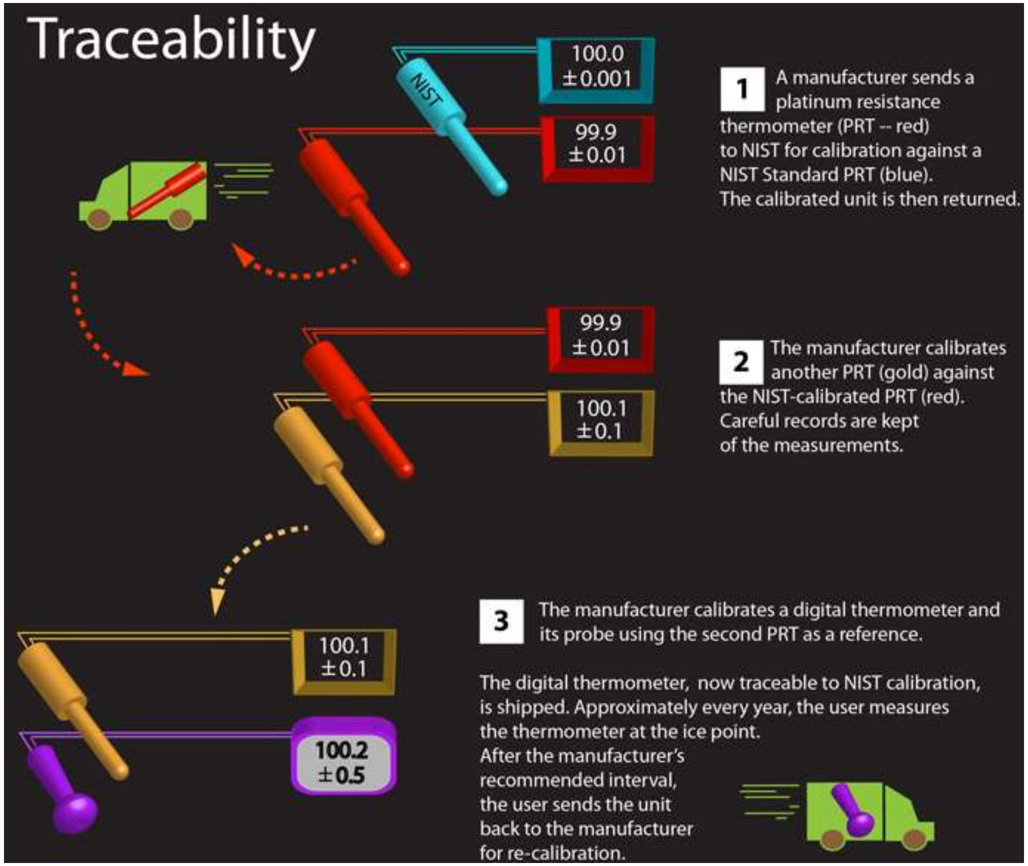
Traceability

Metrological traceability:

the <u>property of a measurement result</u> whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty

International Vocabulary of Basic and General Terms in Metrology; VIM, 3rd edition, JCGM 200:2008

An example (NIST) of traceable measurement.
Note that the uncertainty always increase at each step



Good measurement practice help businesses to improve: design processes; product and service specifications; quality control; and manage waste.

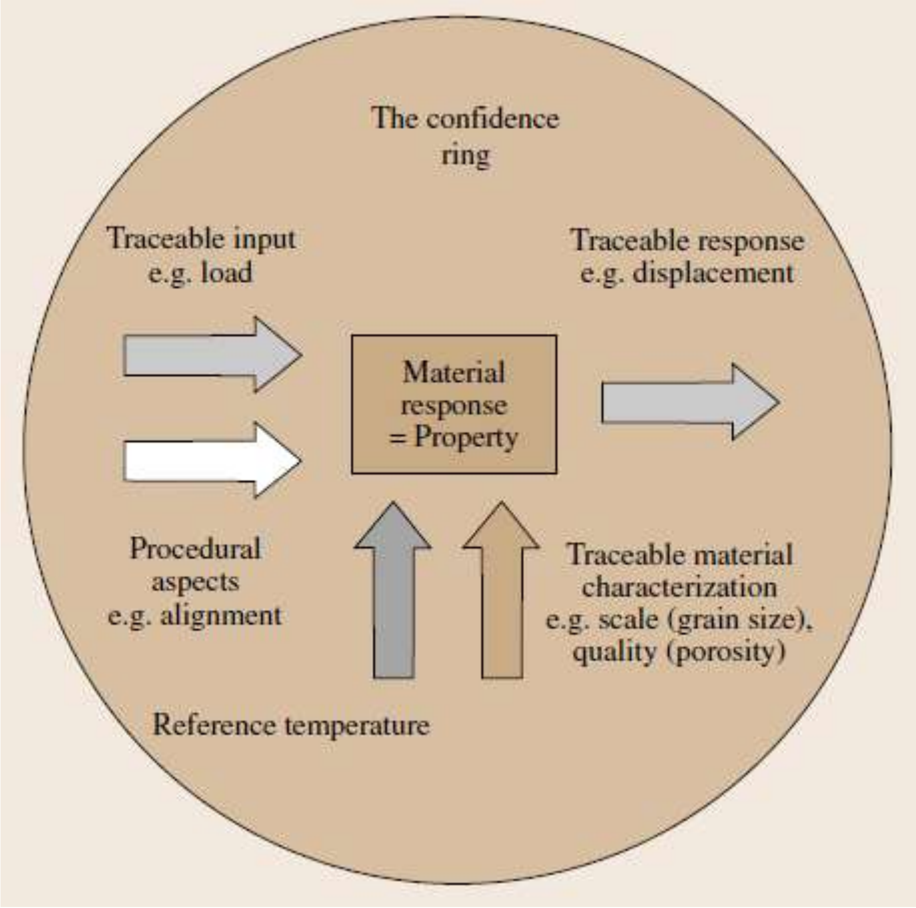
economic growth

Every aero-engine requires hundreds of thousands of exacting measurements and use over 200,000 measurement devices

Rolls-Royce has trained over 1,500 of its staff in NPL Measurement training and works closely with the NMS to implement best practice

Materials metrology

- Materials metrology is problematic: maybe **procedural**
- Link to mole and metre to define materials, then measure Kelvin or Ampere?



Confidence ring for material property combined measurement and testing – note that separate traceability requirements apply to applied stimulus (load), response (displacement), and material characterization (grain size, porosity)

Chemical Analysis

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- Mass, Molecular or Atomic Spectrometry

  SIMS: Secondary ion mass spectroscopy, **QMS:**quadrupole mass spectroscopy

  Accuracy dependant on calibration samples (5-30%)

  Eds J C Vickerman and I S Gilmore, Surface Analysis: The Principal Techniques, 2nd Edition, Wiley,2009

- Analytical Electron Microscopy: EELS, XEDS, EDS

  EDS: energy-dispersive x-ray spectrometry; **EELS:** electron energy-loss spectroscopy

  Often complementary. Through the use of standards and the measurement of empirical detector sensitivity factors (Cliff–Lorimer *k*-factors), measurements can be made quantitative

  D.B. Williams, B.C. Carter: *Transmission Electron Microscopy: A Textbook for Materials Science* (Kluwer/Plenum, New York 1996) pp. 1–306

  R.F. Egerton: Electron energy-loss spectroscopy. In: *The Electron Microscope* (Kluwer/Plenum, New York 1996) pp. 0–306

- Scanning Auger Electron Microscopy
- Infrared and Raman Microanalysis

Quantitative results possible, not always traceable (Uncertainty ~5-30%)
Beware most techniques measure chemical composition not electrical activity
Ultimately traceable to the Mole and the Metre

Dopant concentration

Semiconductor properties are critically dependant on dopants and impurities concentration

Hall measurements are generally made on **uniform samples** from which an average carrier density is derived.

For uniformly doped samples the true density is obtained, but for non-uniformly doped samples an average value is determined. Occasionally one wants to measure spatially varying carrier density profiles. The Hall technique is suitable through differential Hall effect (DHE) measurements. Layers can be stripped reliably by anodic oxidation and subsequent oxide etch.

One of the most accurate methods for dopant profiling is Electrochemical CV profiling ECV

I. Mayes, "Accuracy and Reproducibility of the Electrochemical Profiler," Mat. Sci. Eng. B80, 160–163, March 2001.

P. Blood, "Capacitance-Voltage Profiling and the Characterisation of III–V Semiconductors Using Electrolyte Barriers," *Semicond. Sci. Technol.* **1**, 7–27, 1986

Reproducibility of the ECV method, the standard deviation of the measurement, for uniformly doped epilayers, can be reduced to around 2%

Dopant concentration is usually specified with a carrier concentrations around ± 20%

Microstructural Analysis

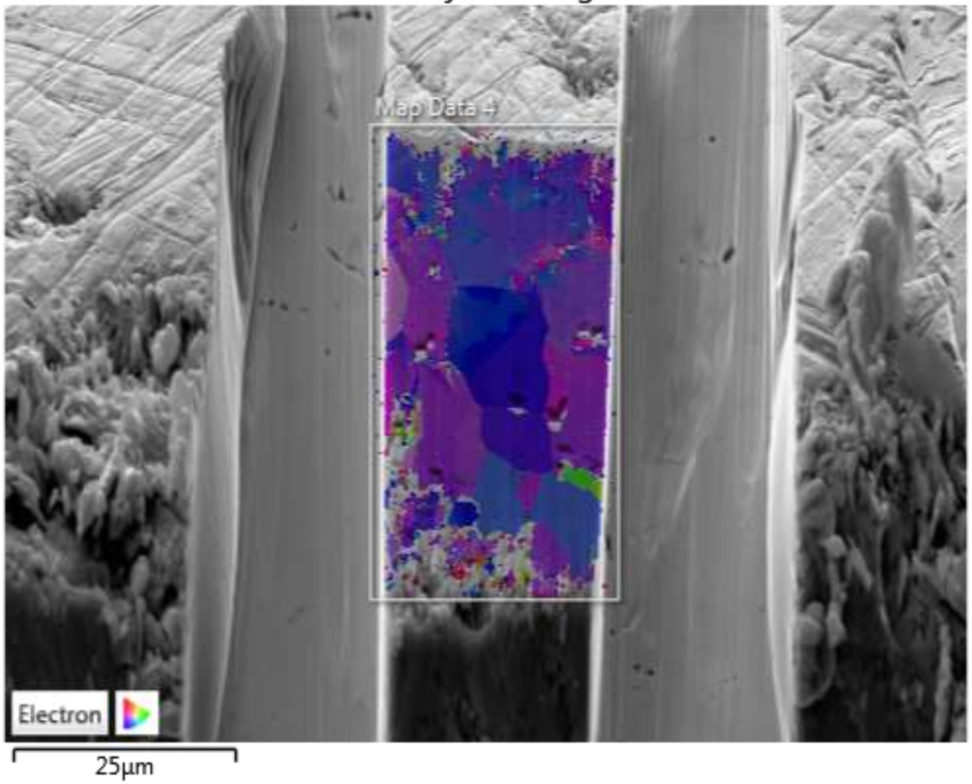
Most physical properties of materials depend on their geometric architecture, on scales ranging from the atomic to the microscopic.

Some properties are governed only by an elementary atomic group others are brought about by cooperative functioning of multiple phases or microscopic structures in different dimensions.

Corresponding to the vast variety of materials and their properties, a wide range of experimental techniques are available,

Electron beam scattered diffraction of Bismuth Telluride samples

EBSD Layered Image 4



Materials understanding may lack behind measurement capability

Electrical & Thermal metrology

National Physical Laboratory

- See Pablo's and Ekaterina's presentation

Beware:
inhomogeneities and anisotropy in samples can reduce as well as enhance apparent physical properties in samples
Polycrystalline samples are NOT necessary isotropic

P. Blood, J.W. Orton: *The Electrical Characterization of Semiconductors: Majority Carriers and Electron States* (Academic, New York 1992)

C.M. Wolfe, G.E. Stillman
Ch apter 3 Apparent Mobility Enhancement in Inhomogeneous Crystals. Semiconductors and Semimetals
Volume 10, 1975, Pages 175–220

Statistical evaluation of results

- A measurand has **a true value** which is unknown - and in general unknowable.
- Each measurement is an **observation** that provides an ***estimate*** of the value of the measurand
- An **observation** is the sum of the measurands value and an ***error***

NB: The error is usually a sum of several contributions from different sources and with different behaviour.

1) error constant for all experiments (*systematic* error) + *random* error.

2) In collaborative study: method bias + contribution from individual laboratory …

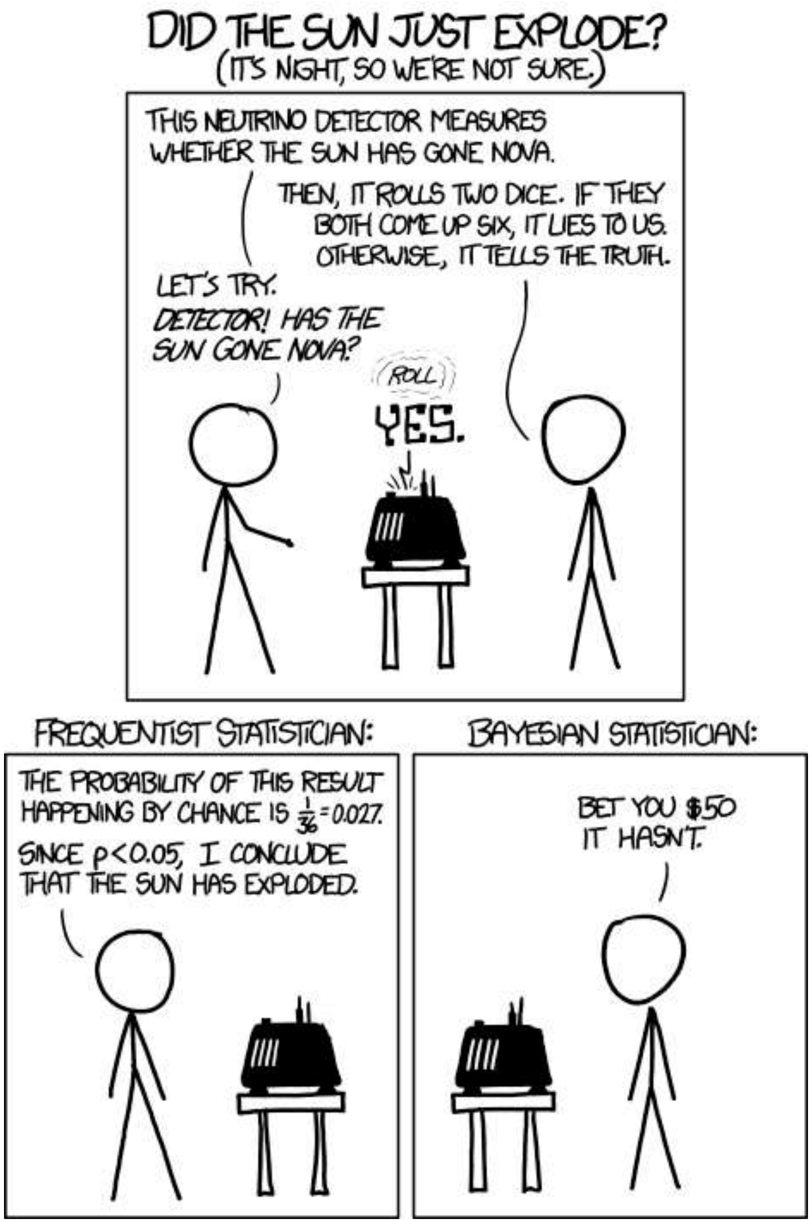
Studying the distribution of the observations allows **inferences** to be drawn about the **probable value of the measurand**. Ultimately, this provides:

a range of values that can reasonably be attributed to the measurand.

uncertainty (of measurement):
parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand

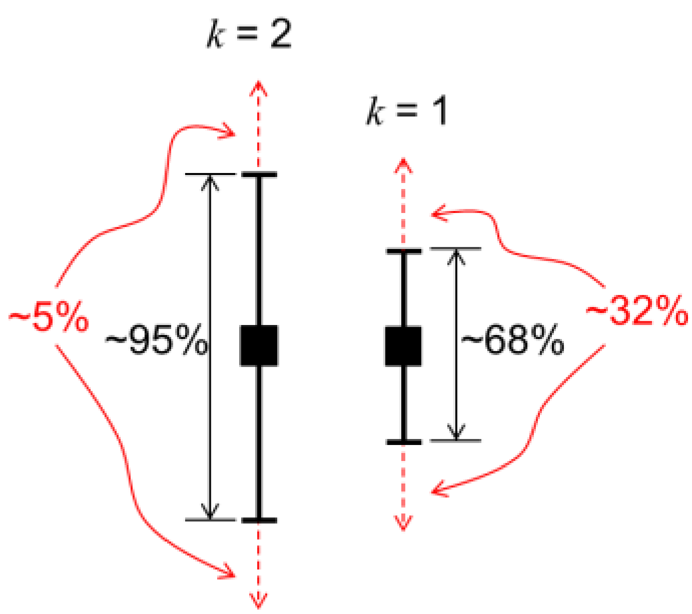
Limitations: multiplicity of true value,
real world may not follow theoretical distribution
Outside information can be used

Previous approach is purely "frequentist"

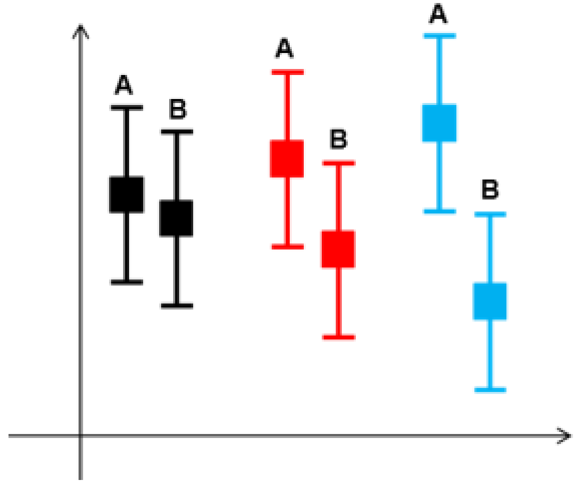


What's the meaning of an error bar?

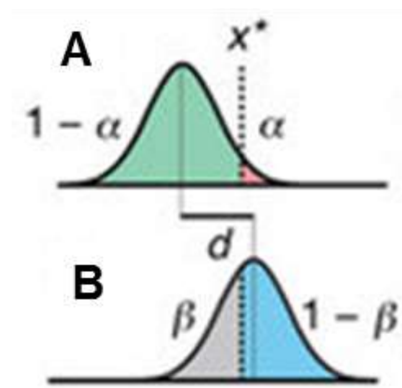
- Graphical representation of the uncertainty
- Uncertainty does not mean error!
- Probability that the true value lies in the given interval.

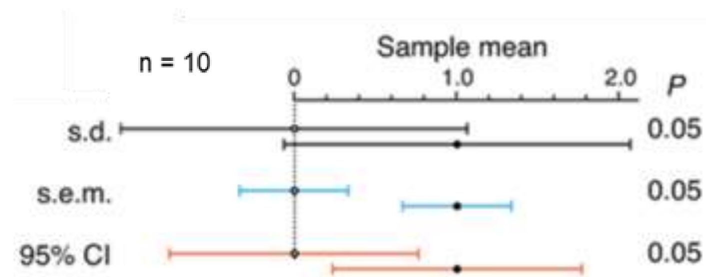


When can we say A is different from B?



Statistical significance!



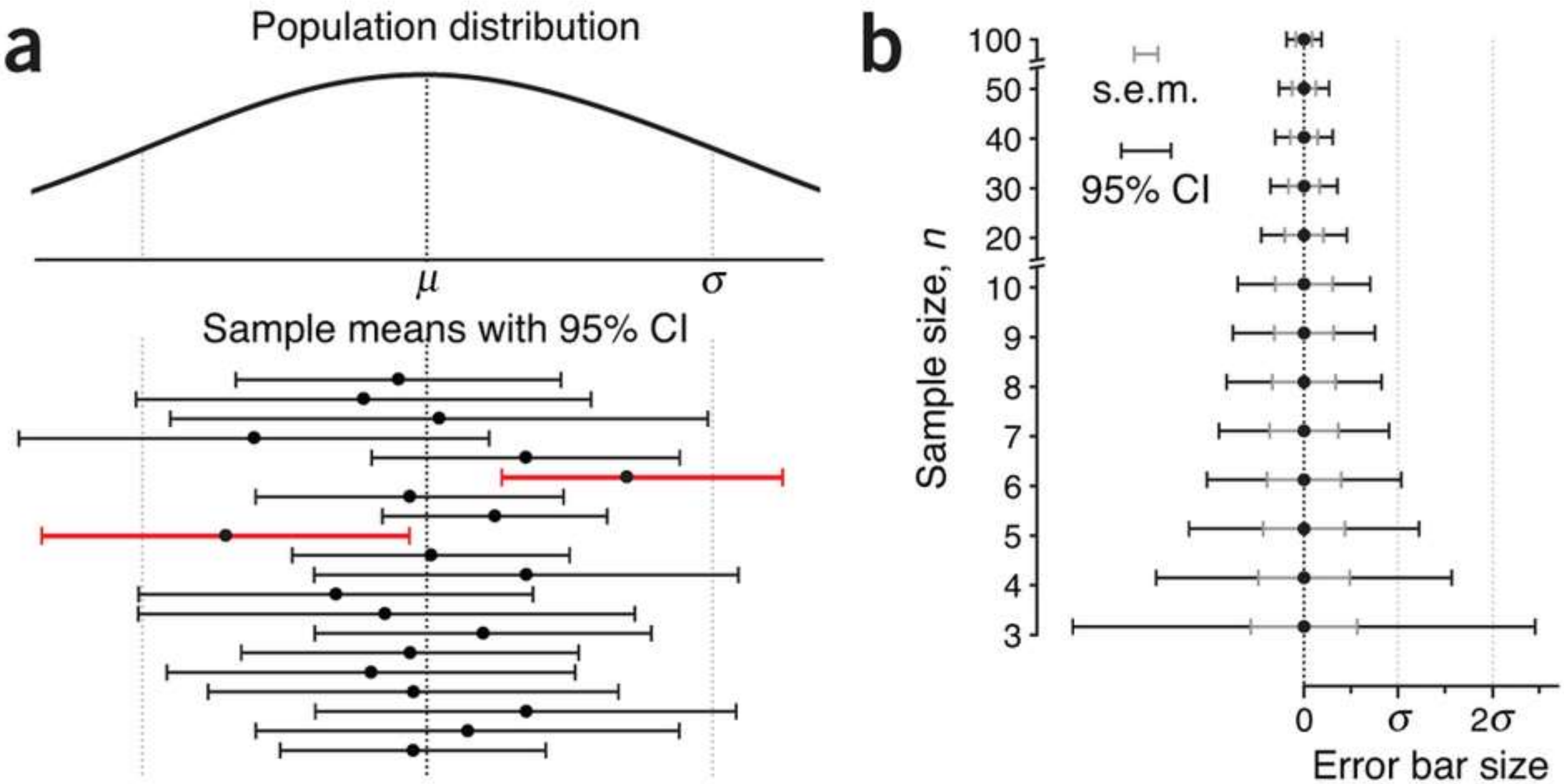


Three type of error bars:

- Standard deviation of the population (s.d)
- Standard deviation of the mean (s.e.m)
- Expanded uncertainty (level of confidence, confidence interval…)

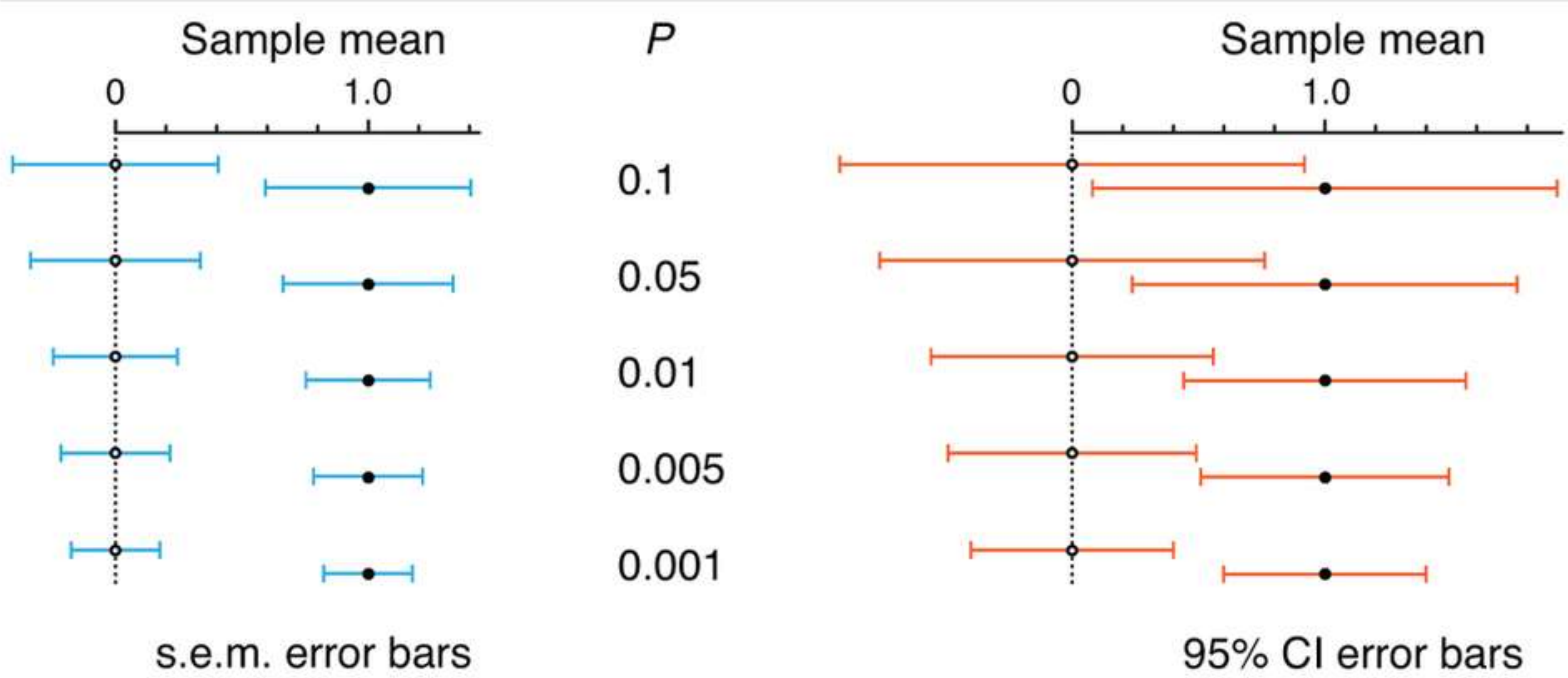
M. Krzywinski and N. Altman, Nature Methods, **10,10** (2013) 921-922

Standard error of the mean vs Confidence interval



M. Krzywinski and N. Altman, Nature Methods, **10,10** (2013) 921-922

P value for sample mean 1 and 0 (n=10)



M. Krzywinski and N. Altman, Nature Methods, **10,10** (2013) 921-922

Key points

- Every measurement is subject to some uncertainty.
- A measurement result is incomplete without a statement of the uncertainty.
- When you know the uncertainty in a measurement, then you can judge its fitness for purpose.
- Understanding measurement uncertainty is the first step to reducing it
- Confidence interval are usually more meaningful

Thermoelectric and Electrothermal Metrology

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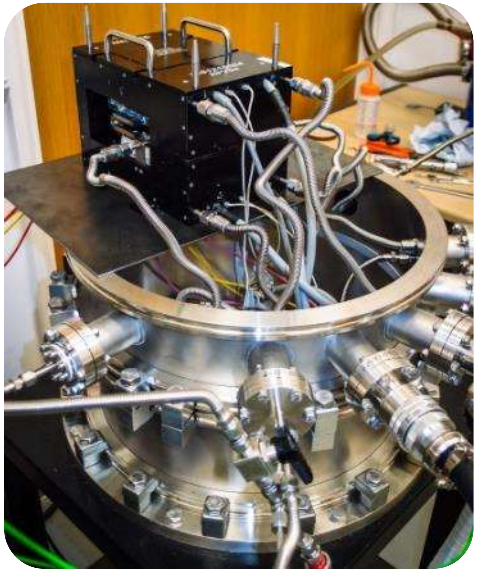
Accurate measurement of **heat flux** and energy **conversion efficiency** in solid state materials across length scales

Energy transport and dissipation in materials at the micro-nanoscale are key to

- **Direct Energy conversion**: thermoelectric, electrocaloric, photovoltaic,…
- **High current-density devices**: power electronics or emerging logic devices



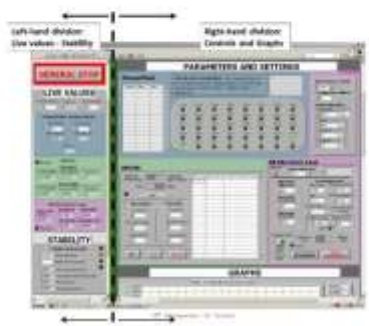
Accurate characterisation of thermoelectric generators up to 900K

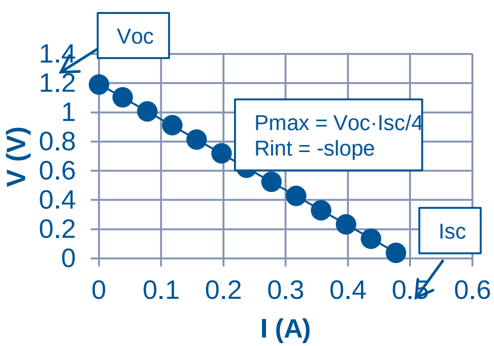
SPM measurement of transport properties at the nanoscale
Cuenat et al, Nanotechnology 23 045703 (2012)

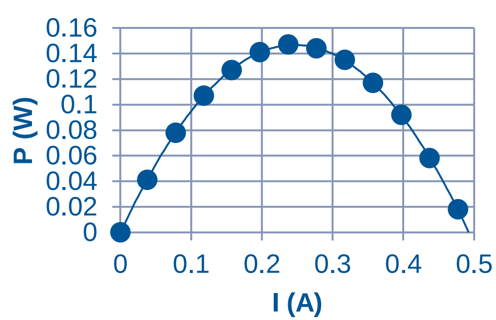
We are developing new traceable **nanoscale methods** to measure materials properties that link directly to **power conversion** in devices

Thermoelectric device characterisation

Repeatability	$u_c = 0.1\%$	Level of confidence: 68%
Combined uncertainty:	$u_c = 2.9\ \%$	Level of confidence: 68%
Extended uncertainty:	$U = 5.8\ \%$	Level of confidence: 95%



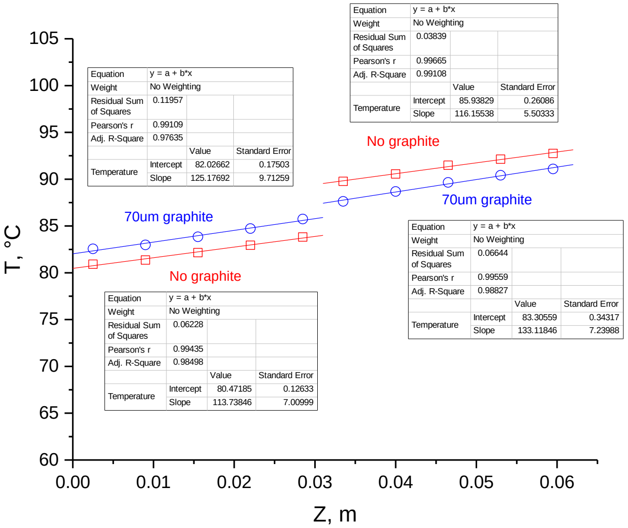




Heat flow meter based on ASTM-5470

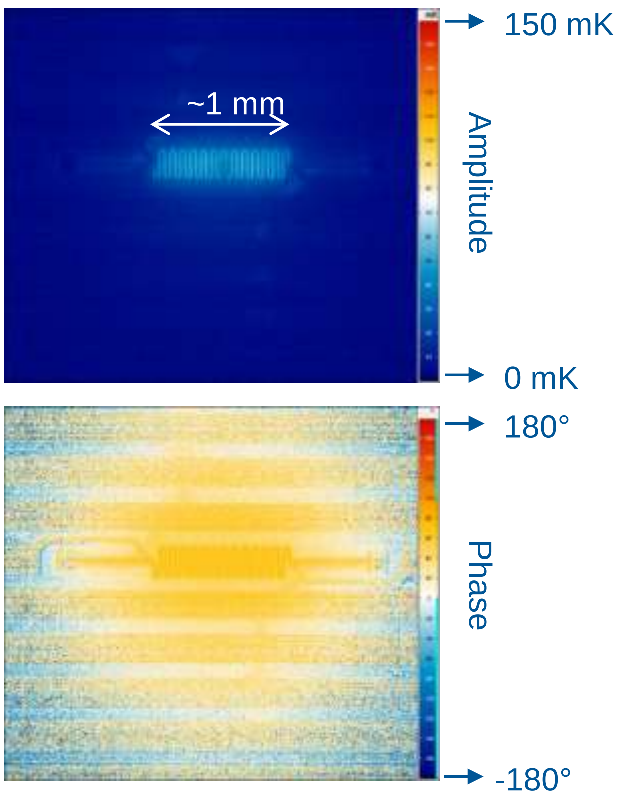
Measurement capabilities

- Heat flux
- Interface thermal resistance
- Thermal resistance and conductivity of a specimen

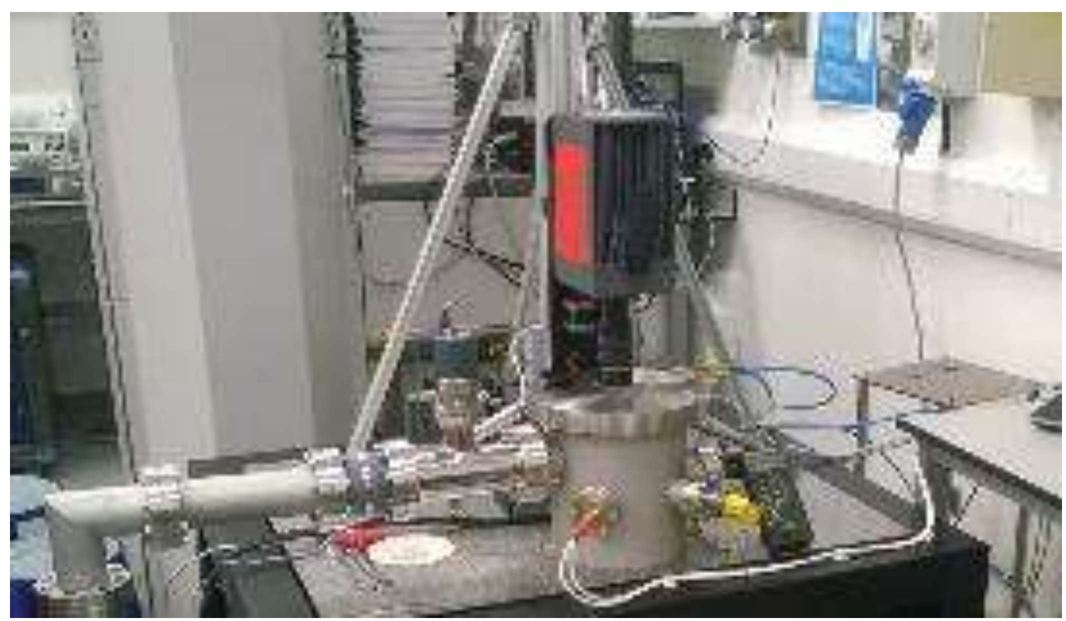


Lock-in infrared thermography

Measurement capabilities



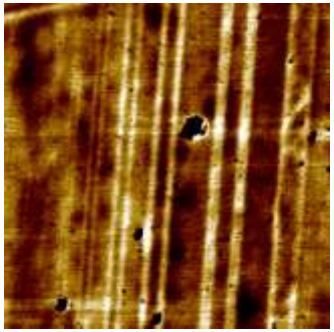
- Traceable temperature measurement
- mK change sensitivity
- Environmental enclosure with temperature control
- Capture rate up to 1.2 kHz



Metrology for high efficiency multi junction solar cells -MJSC

EMRP Energy 51: June 2014-May 2017 http://projects.npl.co.uk/solcell/

Stacking of multiple junction solar cells allows increased efficiency
up to 44% for III-V MJSC, a proven spatial technology



5 by 5 μm AlGaAs/GaA
Kelvin probe

- Experimental development is too long and expensive
- Predictive numerical modelling is required
- Large discrepancy (~30%) observed between measured and predicted efficiencies

NPL Objective 1: To develop accurate and spatially resolved metrology to determine the complete material properties layer by layer.

NPL Objective 2: Extension to novel material such as nitride and solar thermoelectric

Metrology for manufacturing 3D stacked integrated circuits

EMPIR – Industry June 2015-May2018

3D-devices will combine logic, memories, imagers and MEMS **from different wafers of various foundries using different manufacturing processes** optimized at the right node.

- ✓ **Traceability of the measurement and standardization will be mandatory**

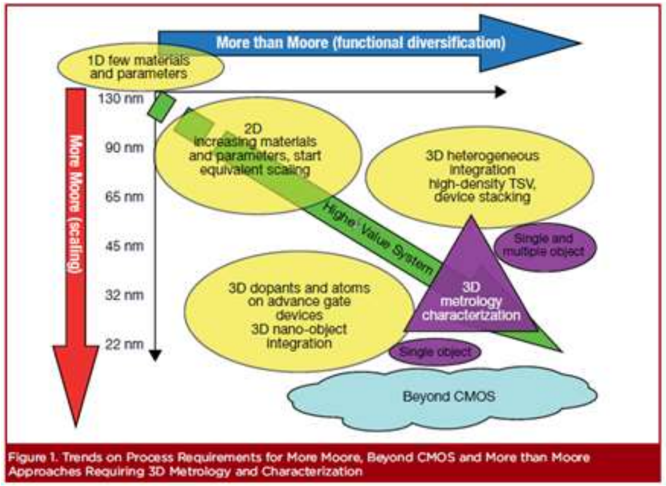


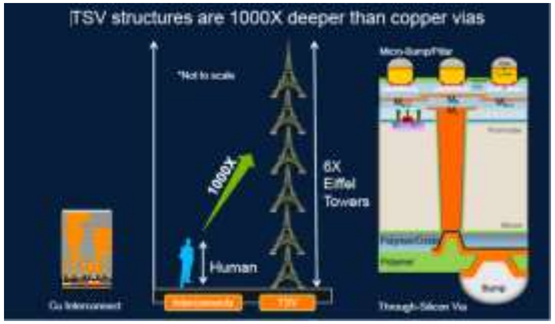
Figure 1. Trends on Process Requirements for More Moore, Beyond CMOS and More than Moore Approaches Requiring 3D Metrology and Characterization

European Industrial Roadmap 2014

Devices are “stacked” and connected with Through Silicon Vias (TSV)

Increased integration, means

- Increase Cu resistivity (smaller grains)
- Increase heat (higher current density)
- Larger Thermomechanical stress



NPL will develop tools to measure energy dissipation with better lateral resolution, better accuracy and develop new procedures for conformity assessment at the wafer level

Conclusions

- Metrology is the science of measurement, it is underpinning all physical sciences and trade .

Take home message: be “suspicious” and critical of your results, but do not overdo it. The first quality of good metrology is to be fit for purpose

Department for Business Innovation & Skills

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SolCell

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