Welcome to

MLL 104

Characterization of Materials Lab

2022-23 Even Semester

3 – 0 – 3

Team

Krishna Balasubramanian

- Assistant Professor, Department of Material Science and Engineering
- Webpage: https://web.iitd.ac.in/~bkrishna
- Email: bkrishna@mse.iitd.ac.in
- Ph: 01126548559

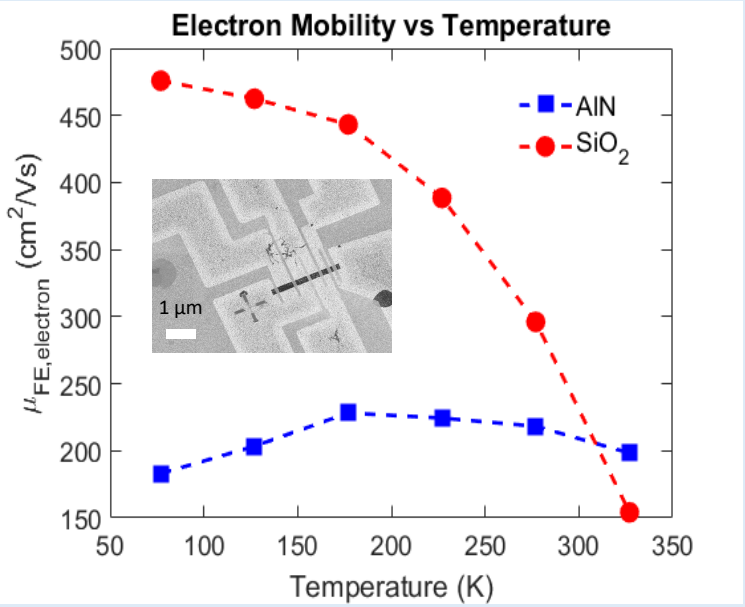
Leena Nebhani

- Associate Professor, Department of Material Science and Engineering
- Webpage: https://sites.google.com/view/surfchem/home
- Email: lnebhani@mse.iitd.ac.in
- Ph: 01126596691

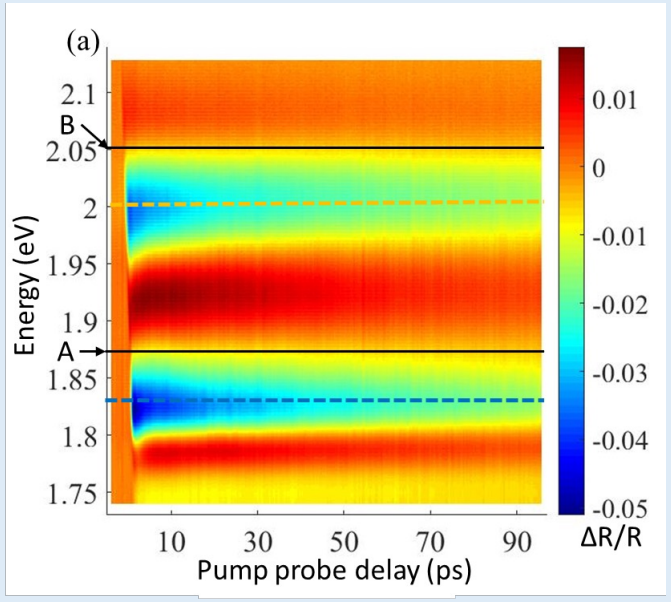
Teaching Assistants: (11)

Dr. Nagaraj, Durgesh Banswar, Ingita Indu, Priyanka Goel, Malar Vadani, Subhashree Subhasmita Pradhan, Chhotrai Soren, Mahesh Ranjan Dash, Monomoy Chatterjee, Sarthak Rautela

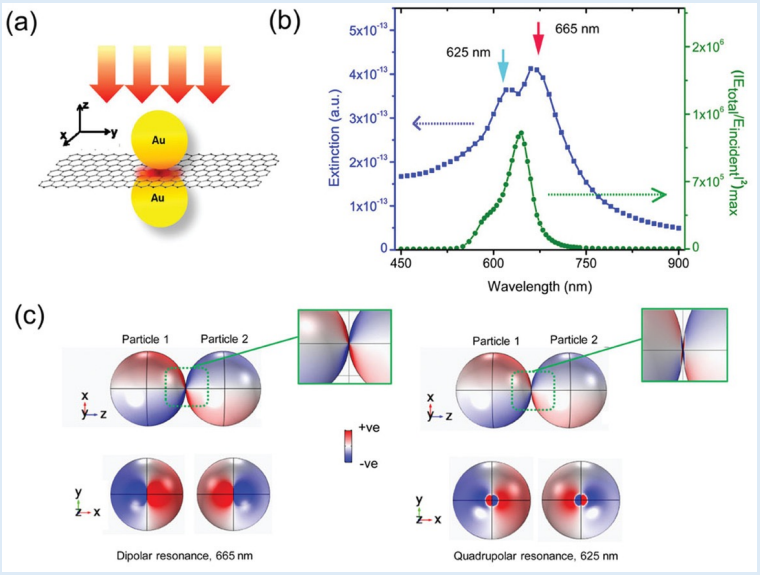
Graphene on III-V substrates



Ultra-fast dark exciton dynamics – TMD MoS_2

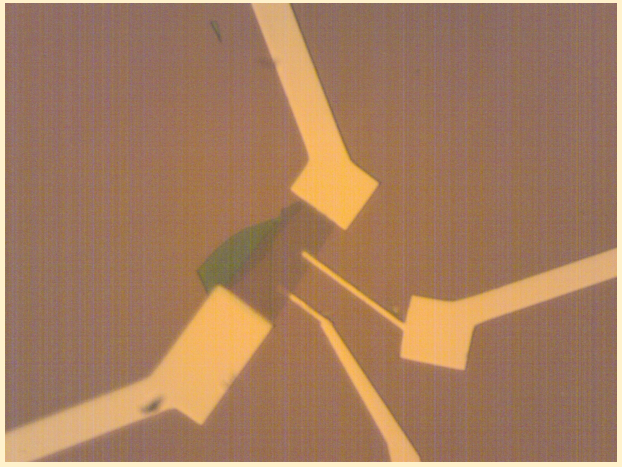


2D-nanoparticle plasmonics

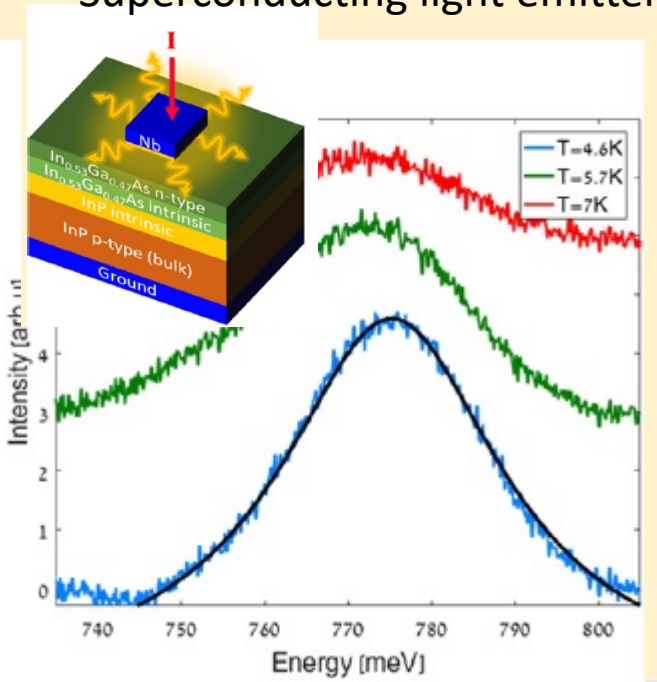


2D Electron transport and optics

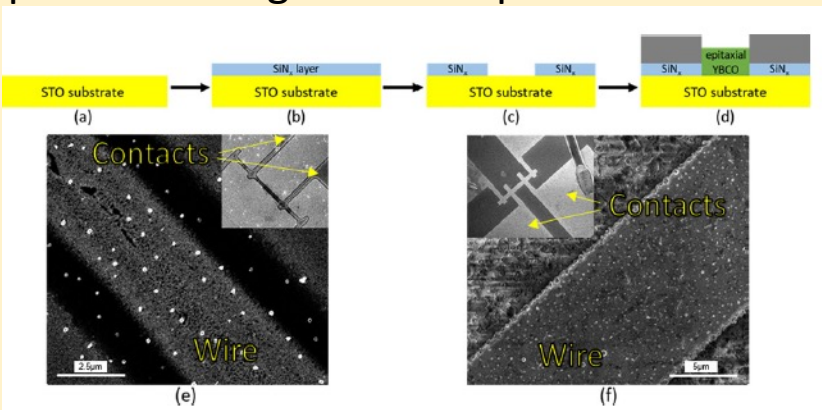
2D – Superconductor interfaces



Superconducting light emitters



Superconducting nanowire photon detector



Superconductivity

Nature communications 10 (1), 1-9
Optics Express 27 (23), 33427-33435
physica status solidi (a) 217 (16), 1900949
Journal of Physics D: Applied Physics 49 (26), 265301
Nanotechnology 27 (20)

About the course

- Introductory course on material characterization techniques.
- Introduce the concept of measurement, observations, qualification and characterization.
- Start with fundamental principles – measurement technique - Instrumentation
- The measurement techniques will involve several fundamental physical properties which every engineer, particular a materials engineer should master.
- The course has **three hours of lecture and three hours** of lab session per week and has a massive **4.5** credits.

Content

- Fundamental of measurement
- Optical Spectroscopy Techniques
- Optical microscopy
- X-ray diffraction and applications
- Scanning electron microscopy
- Thermal analysis
- Electrical characterization

Course Information

Course Timing:

- Lectures
  Slot E, Tuesday, Wednesday, and Friday 10 AM – 11 AM
- Labs
  Mondays 2 – 5 PM. All students will need to assemble in the lab hall (to be decided)

Prerequisites:

- Basic first year Math, Physics – Calculus, Electromagnetics.
- Inquisitiveness to learn!

Lecture Hosting

- Lecture delivery:
  - All sessions in-person at LH604
  - Attendance will be recorded. In fact participation in lectures is mandatory.
- Homework to be submitted before the class sessions. Could involve simple coding exercising.
  - Submissions are binary: either you submit before time or leave without marks. Sorry, no concessions for any amount of delay!
- All exams and evaluation will be offline. The answer sheets will be evaluated by TA assigned.
- Dishonesty will be very seriously dealt with. We are a huge team of very experienced professionals. So, any malpractice will be identified, and the student will be given a ‘F’. Remember, this is much worse than just dropping the course!

Lab Hosting

- Experiments will happen at different places in the department/Institute
- A lab file is to be maintained by all. Each lab you will bring the file with sheets.
- In lab session,
  - We will start with an introductory briefing by a faculty member (Either myself/Prof. Leena)
  - A question list will be provided which needs answering
  - We will divide you ppl into sufficiently small groups and each group will be independently taken for experiments.
  - The group will return back and complete the lab sheet.
  - The lab sheets are individual exercises. They are to be treated as exam papers and filled individually.
  - You are to bring several A4 sheets, graph papers, stapler, calculator and necessary stationaries.
  - No one is allowed to leave before 5 PM.
  - The sheets are expected to be completed on the same day and submitted to the TA. It will not be collected later on.
  - After evaluation, the sheets will be returned back and it needs to be filed.
  - A final viva-voce will be conducted on the last day of the course and your lab file be collected for evaluation.

References

- Multiple references for each topic. Will be announced during the lecture time. General references are as follows:

- Pavia, Lapman and Kris, Introduction to spectroscopy, Brooks Cole 2000
- B. D. Cullity and S. R. Stock, Elements of X-ray Diffraction, Prentice Hall; 3 ed. 2001R
- J. Goldstein, Scanning electron microscopy and X-Ray Microanalysis
- R. Haynes, Optical Microscopy of Materials, Springer, 1984
- Michael E. Brown and Patrick K. Gallagher, Handbook of Thermal Analysis and Calorimetry, Elsevier, 2003.
- Peter J. Haines, Thermal methods of Analysis, Principles, Applications and Problems, Springer

Evaluation policy

Evaluation:

- Relatively graded course with the following split up:

Lecture part:

- **35% minor, 45 % major, 10% homework/assignments and 10 % in-class interactions**

- Practical part:
- **60% - lab sheet evaluations, 30% Viva-Voce and 10% participation**

- **Final score: 60% Lectures and 40 % Practical**

- **Audit Pass is 35%**

Academic Honesty

All students registering to this course voluntarily commit to the academic integrity, research and Ethics policy, IIT Delhi (https://academics.iitd.ac.in/sites/default/files/registration/forms/10_FORM%20H.pdf). This is an under-graduate level course in the country's most premier institute, and I expect the students to know their responsibility. If any document submitted to us is plagiarized (copied verbatim either from textbook/ online resources/ fellow students…), the student will be deregistered and promptly handed to institute authorities.

Discussion is okay for homework, but the submission needs to reflect the student's work. No discussion/references are allowed in lab sheets, quizzes/minors/final exams.

Characterization, Measurement and Metrology

Character:

person: thinks, feels and behaves

Material: ?

Physical: density, thermal/electrical conductivity, optical transparency/reflectivity, hygroscopicity

Mechanical: Hardness, tenacity, elasticity, ductility, malleability,

Chemical: Reactivity, catalytic property, corrosion resistance, flammability

Measurement:

An act of quantification of the character

The International Bureau of Weight and Measures states that: “Metrology is the science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology.”

Measurement

I believe in evidence. I believe in observation, measurement, and reasoning, confirmed by independent observers. I'll believe anything, no matter how wild and ridiculous, if there is evidence for it. The wilder and more ridiculous something is, however, the firmer and more solid the evidence will have to be.[31] (Isaac Asimov)

An experiment is a question which science poses to Nature, and a measurement is the recording of Nature's answer.[46] (Max Planck)

If someone separated the art of counting and measuring and weighing from all the other arts, what was left of each (of the others) would be, so to speak, insignificant.[47] (Plato)

Count what is countable, measure what is measurable and what is not measurable, make measurable (Galileo Galilei)

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 meager and unsatisfactory kind: it may be the beginning of knowledge, but you have scarcely advanced to the stage of science whatever the matter may be [2]. (Lord Kelvin)

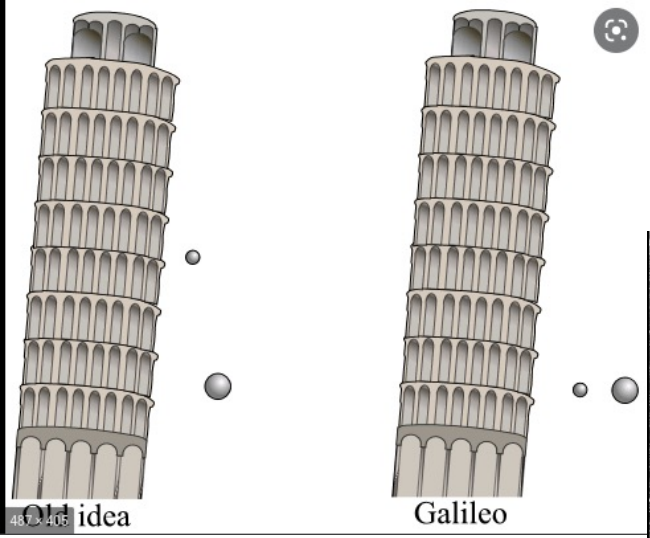
What is measurement (Historical Perspective)

Among the earliest known measurement is of length and weight,
As early as 2900 BC.

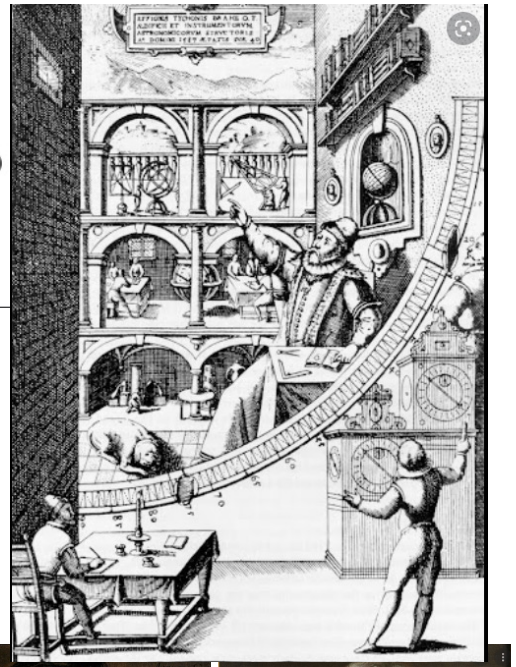
No side of the Pyramid's square base deviated from the 9000 inch value by 1/20 Inch

The right angle never deviated greater than 3/1000 a degree.

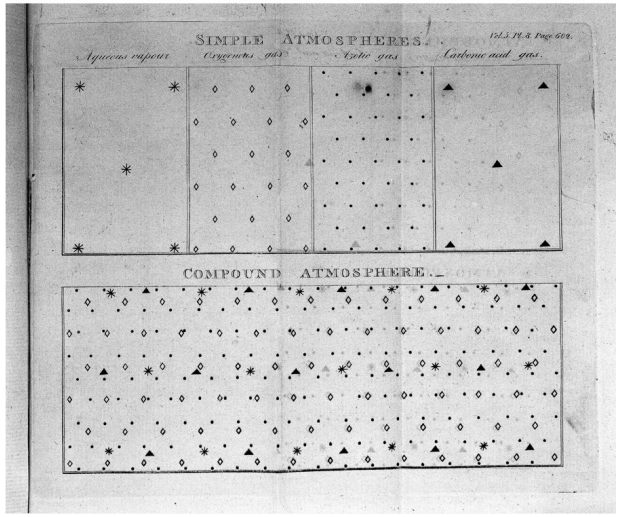
750-year-old sun dial in Konark, Odisha



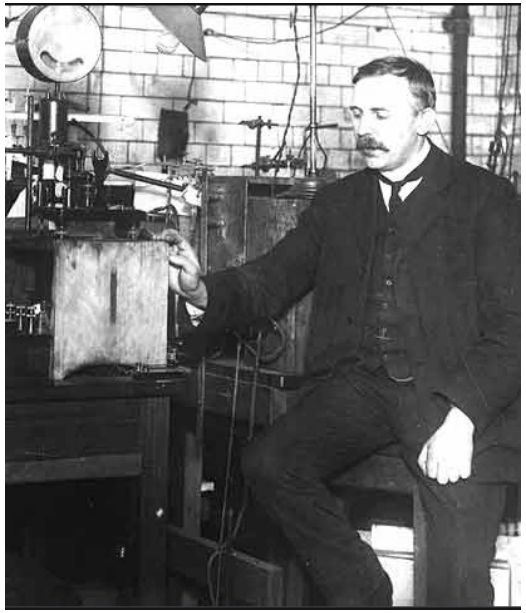
Galileo's gravitational experiment



Astronomical observations
– Tycho Brahe



Observation of chemical reactions
J. Dalton



X-rays through Au foil, Atomic structure

What is measurement ?

- Experimentally assign to a quantity a number for comparison and calculations.
- Many a times, we define a ‘standard’ for a particular measure and compare the object to the standard (unit)
- Necessary conditions of measurement:
  - The act of measurement does not affect the object
  - Preferably, the measuring apparatus does not degrade and can be reused infinitely.
- Qualities of a measurement:
  - Accuracy
  - Precision
  - Resolution
  - Sensitivity
  - True Value
  - Error

Types of measurement

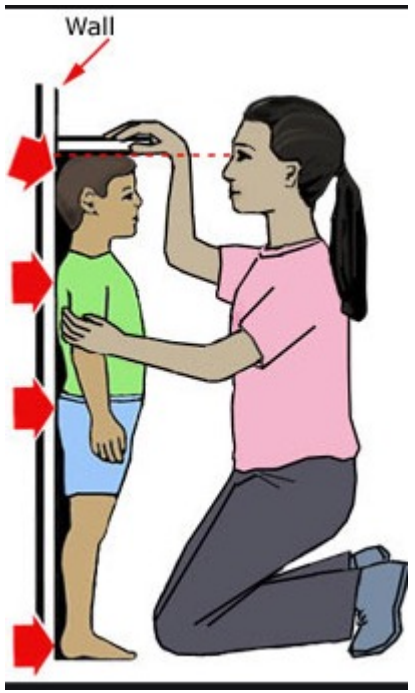
SCALE	DEFINITION	EXAMPLE
Nominal	Only the presence/absence of an attribute; can only count items	go/no go; success/fail; accept/reject
Ordinal	Can say that one item has more or less of an attribute than another item; can order a set of items	taste; attractiveness
Interval	Difference between any two successive points is equal; often treated as a ratio scale even if assumption of equal intervals is incorrect; can add, subtract, order objects	calendar time; temperature
Ratio	True zero point indicates absence of an attribute; can add, subtract, multiply and divide	elapsed time; distance; weight

DIY technique

Call a friend

Go to a professional

Call a Scientist



What ever you do, you will finally come out with a singular value.

Say the exact height of the person is 5 feet, 10 inches.

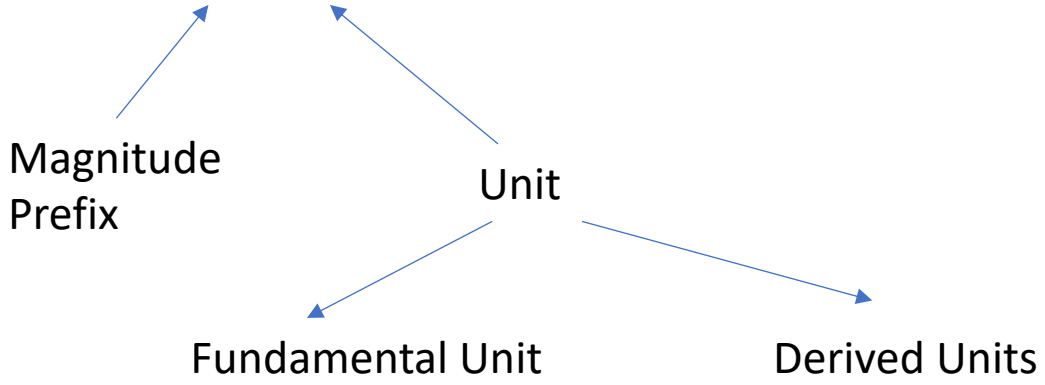
Images from web

The act of measurement on an object's attribute

- The act observes the right attribute (validity)
- The act observes the attribute to the closest value possible (accuracy)
- The act does not alter the object
- Repeated acts results in similar results (repeatability, stability)
- An object (n-times the original) should preferably result in n-times the originally observed (linearity)

Lets break the measurement down

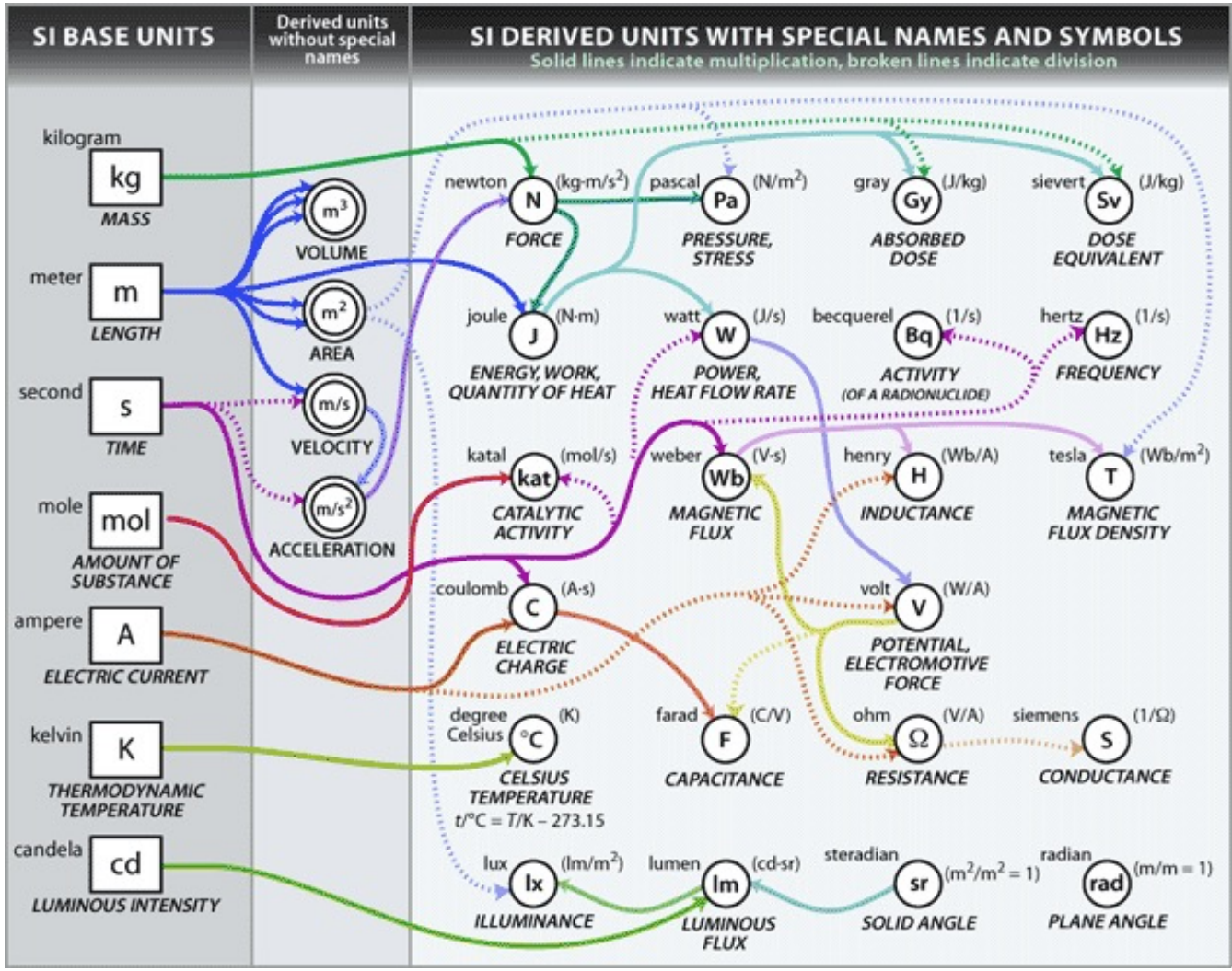
The height is 5 ft 10 inches



Any measure is expressed as a product of numeric with a measurable quantity

Units and Standards

- Earlier units like foot, inch, yard, furlong are very hard to compare.
  - First introduced by Egyptians, 1ft = 12 inches, yard = 3 ft and mile about 5000 ft. [things were later changed]
  - Defeats the purpose of measurement
- In 1960, Systeme International Denominations (SI) gave the standards for the units

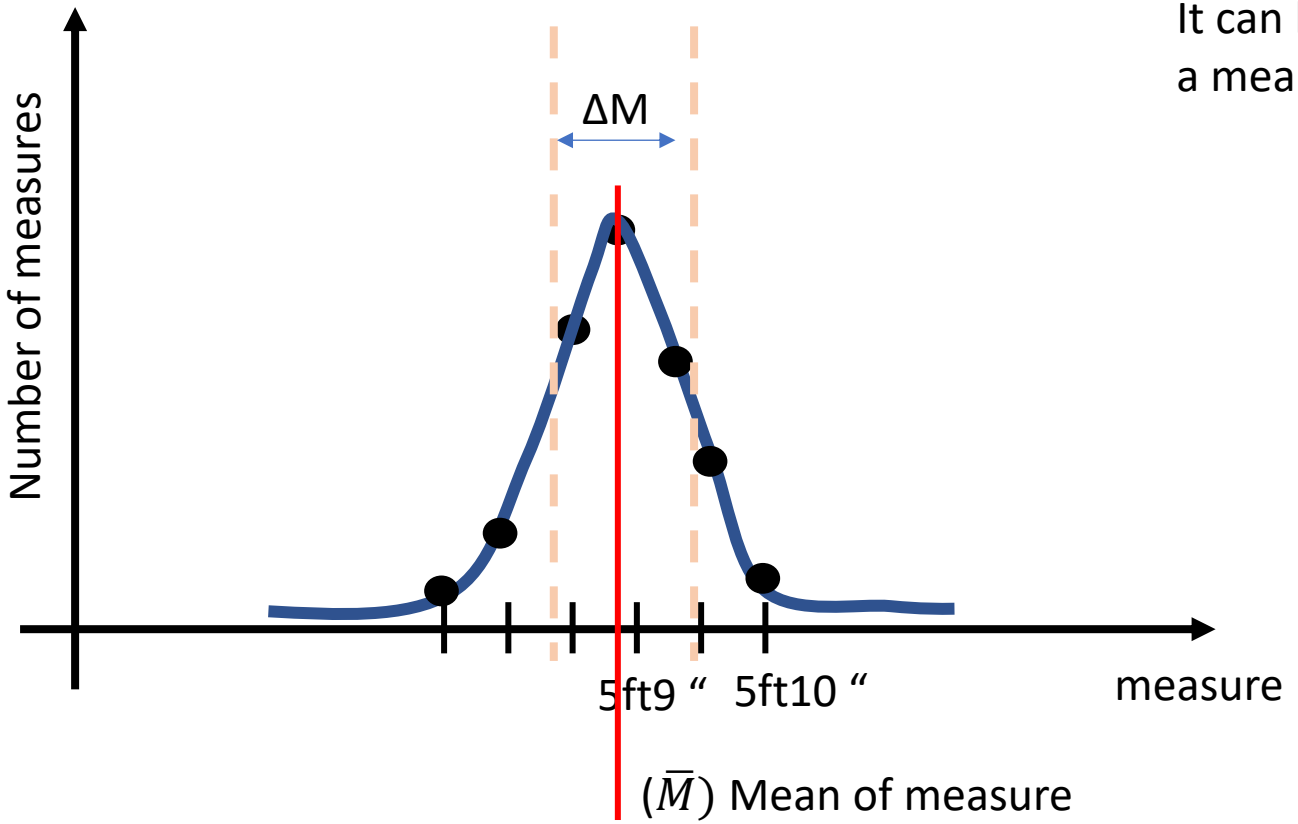


Fundamental Units

- These are fundamental jumps in our understanding
- **Meter (**m), is the length of the journey path from the light in the vacuum in a range of 1/299 792 458 seconds;
- **Second (s)**, is the duration of 9,192,631,770 periods of radiation corresponding to the transition between two hyperfine levels of the fundamental state of the Cesium Atom 133;
- **Kilogram (kg)**, is the mass unit; It is equal to the mass of the international prototype of the kilogram ≫ prototype made of Iridium Platinum in 1889 and conserved in Sevres;
- **Amperes** (a), is the intensity of a constant electric current which, kept in two parallel straight conductors of infinite length, of negligible circular section, placed at the distance of one meter from each other in the void, would produce between these conductors A force equal to 2 x 10-7 Newton on each metre of length;
- **Kelvin (K**), thermodynamic temperature unit, is the fraction 1/273,16 of the thermodynamic temperature of the triple point of the water;
- **Mole (MOL**), is the amount of substance of a system that contains as many elementary entities as there are atoms in 0.012 kg of carbon 12. When using the mole, the elementary entities must be specified; They can be atoms, molecules, ions, electrons, other particles, or specified groupings of such particles;
- **Candle (CD**), is the luminous intensity, in a given direction, of a source emitting a monochromatic frequency radiation 540×1012 hertz and whose energy intensity in that direction is 1/683 watts to the Steradian.

Quality of a measure

- How reliable is your prefix/magnitude ?
- Q1: Is your measurement valid ?
- Q2: How accurate is your measurement
- Q3: What now happens if there were multiple attempts at the measurement using the same equipment and the object ?



If each of the measurement event is uncorrelated (independent)
It can be proved that the outcome will be normally distributed with a mean and variance

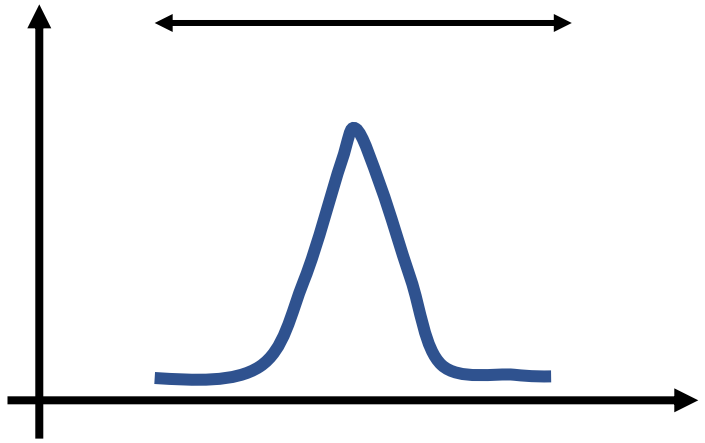
A single act of measurement gives a value (say M)

Least significant digit of your measure,
A band of values which you cannot distinguish (δM) is called as resolution.

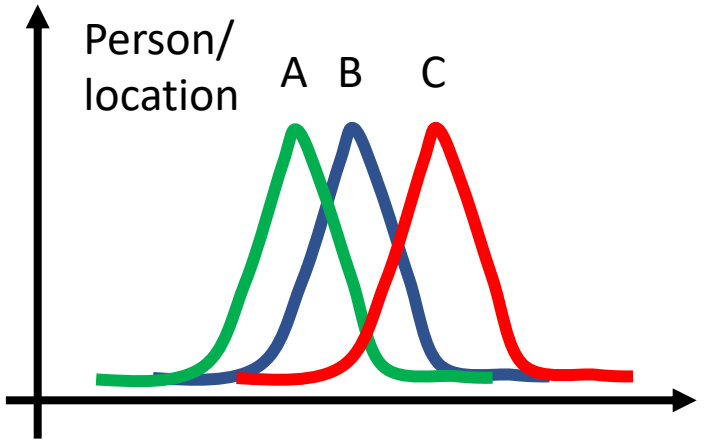
If numerous such measurements are made, (say M_1, M_2 …. M_n

One can plot a histogram and obtain a mean of all the measurements $(\bar{M})$ $\bar{M} = \frac{\sum M_i}{n}$

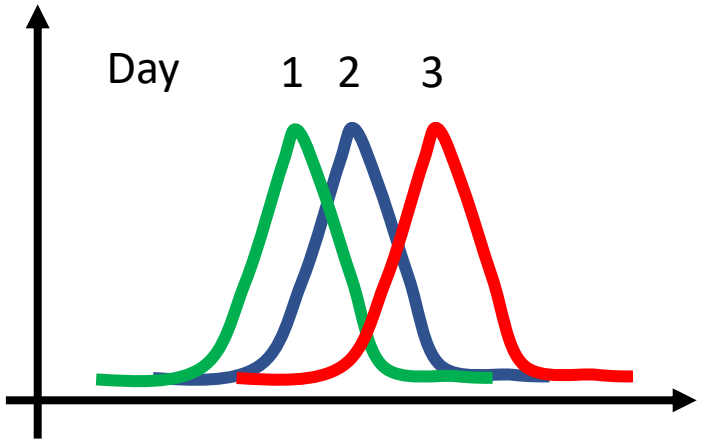
Precision of the measure is given by the width of the normal distribution $(\Delta M) = \pm\sqrt{\frac{\sum(M-\bar{M})^2}{n}}$



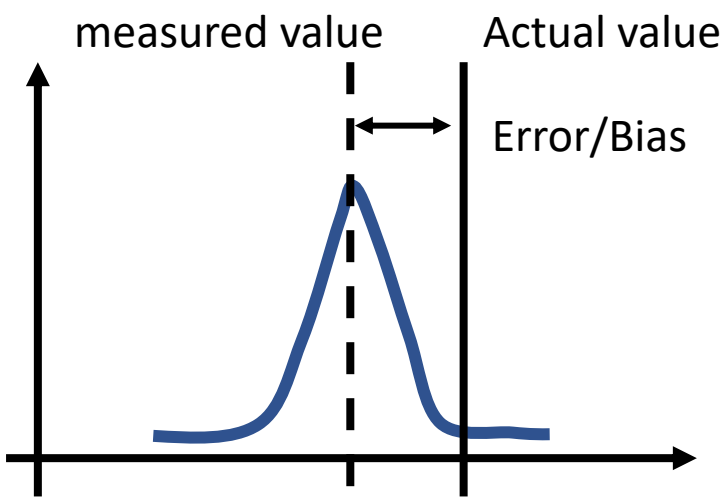
Repeatability of a measurement:



Reliability of a measurement:

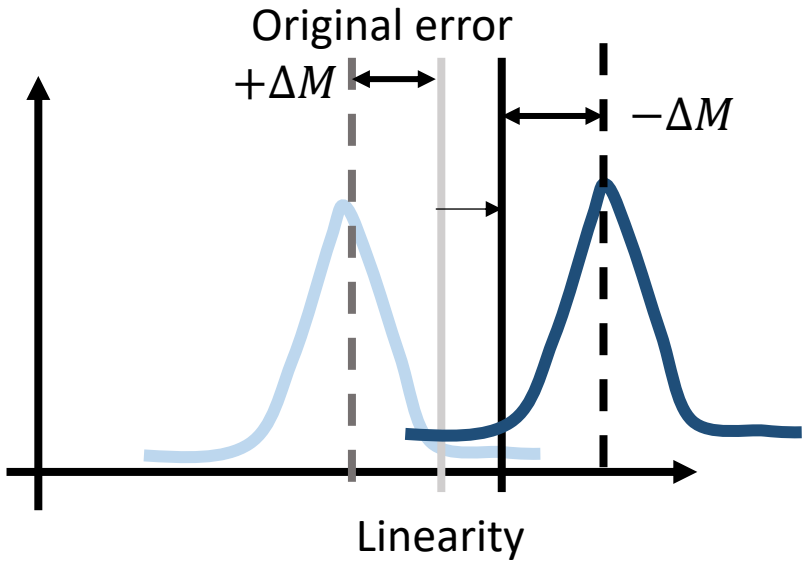


Stability of a measurement:



Error in the measurement

Difference between the measured and actual value is the error in the measurement



Linearity

If the actual value is changed by a small value, linear measurement will have a proportional change in the measured value and hence the error

Remember all these aspects as we conduct several measurements using very different techniques.