Analytical Methods for Materials

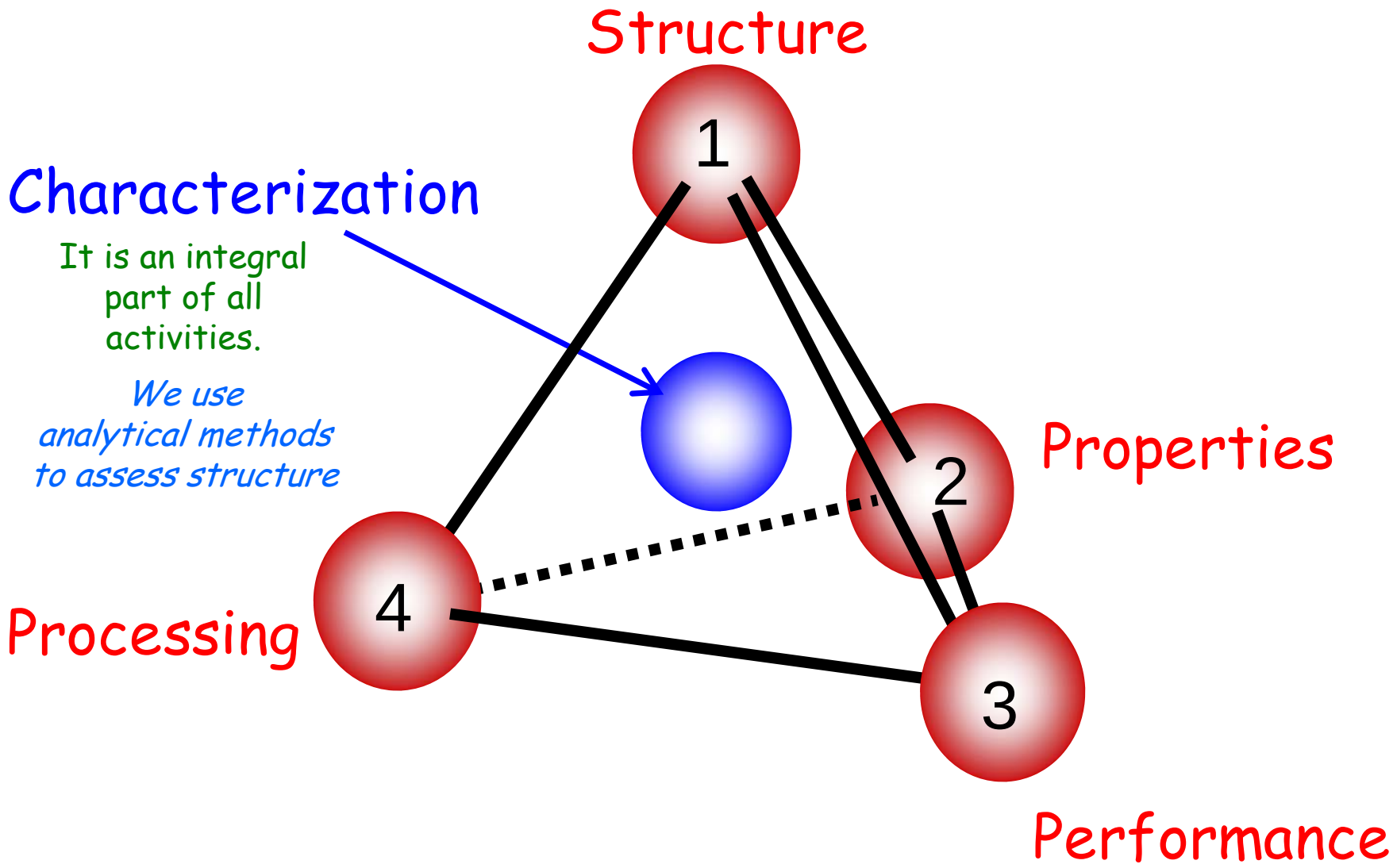
Lesson 1

Introduction to Materials Characterization

Reading Assignment

Materials Science & Engineering
Metallurgical & Materials Engineering
Ceramic Engineering
etc…

- All involve establishing relationships between:
  1. Structures of materials
  2. Processing of materials
  3. Properties of materials


Structure
1
Characterization
It is an integral part of all activities.
We use analytical methods to assess structure
Properties
2
Processing
4
3
Performance

Structure determines properties

Properties can be changed by altering composition and/or processing

Must know/understand "structure" and composition to exploit properties

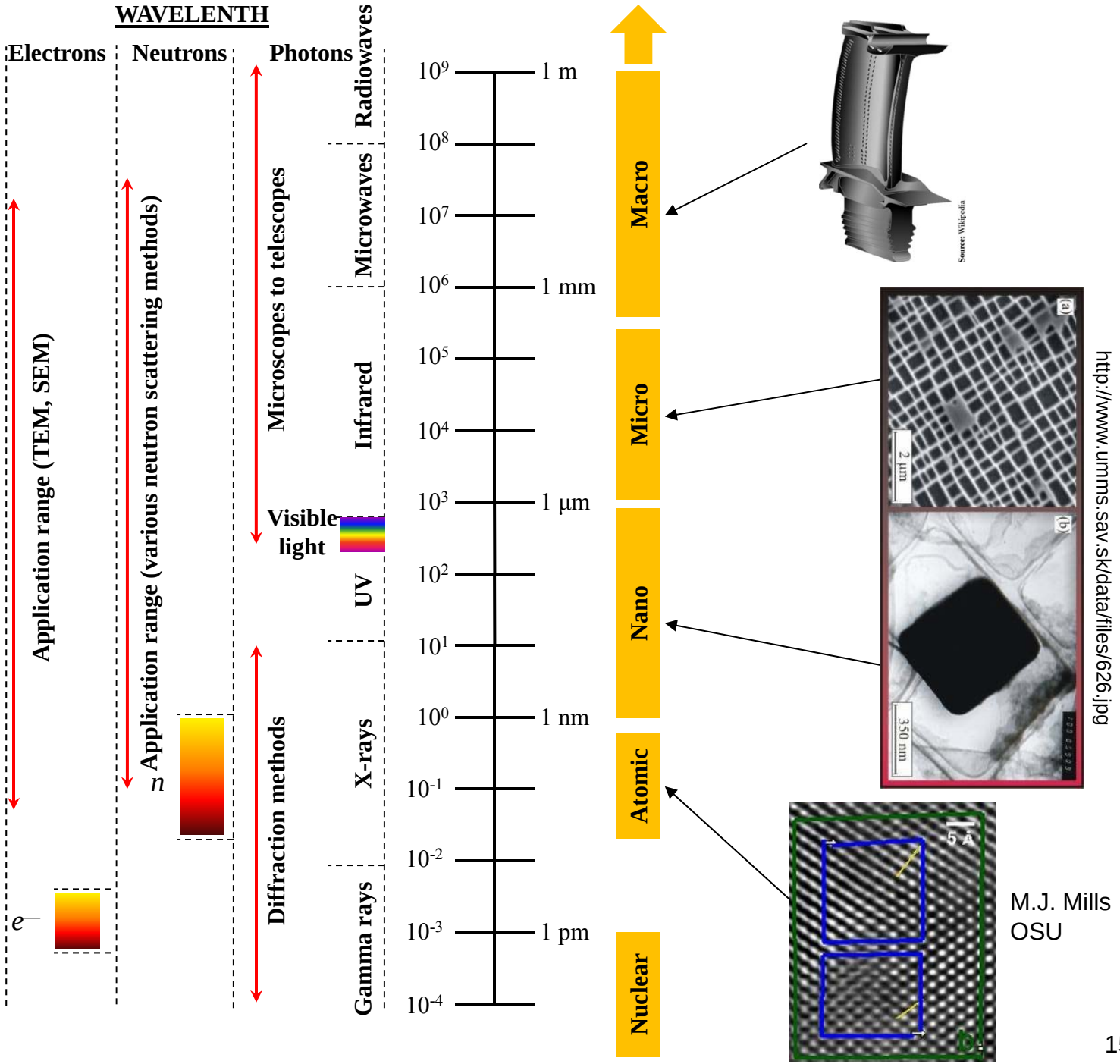


Figure adapted from M. De Graef and M.E. McHenry, *Structure of Materials*, Cambridge University Press, Cambridge UK (2007) p. 5.

Hierarchy of structure

- Macrostructure:
  - Objects can be observed with the naked eye.
- Mesostructure:
  - Objects are on the borderline of visibility.
- Microstructure:
  - Objects can be viewed by means of optical microscopy techniques. Objects are micron sized (~0.001 mm).
- Nanostructure:
  - Objects have sizes between 1 nm and 100 nm.

Table 1.2. The scale of 'microstructural' features, the magnification required to reveal the feature, and some common techniques available for studying the microstructure.

Scale	Macrostructure	Mesostructure	Microstructure	Nanostructure
Typical Magnification	× 1	× 100 - 1000	× 10,000	× 1,000,000
Common experimental techniques	Visual inspection	Light Optical microscopy	Scanning and transmission electron microscopy	X-ray diffraction
	x-ray radiography	Scanning electron microscopy	Atomic force microscopy	Scanning tunneling microscopy
	Ultrasonic inspection			High-resolution transmission electron microscopy
Characteristic microstructural features	Production defects	Grain and particle sizes	Dislocation substructure	Crystal and interface structure
	Porosity, cracks and inclusions	Phase morphology and anisotropy	Grain and phase boundaries	Point defects and point-defect clusters
			Precipitation phenomena	

What characterizes structure?

Microstructure

- Structure characterized by size, shape, volume fraction, and arrangement of grains of different phases or of a single phase.

Substructure

- Structure characterized by the type, arrangement, and density of line defects or by the size shape and orientation of subgrains.

Crystal/Atomic Structure

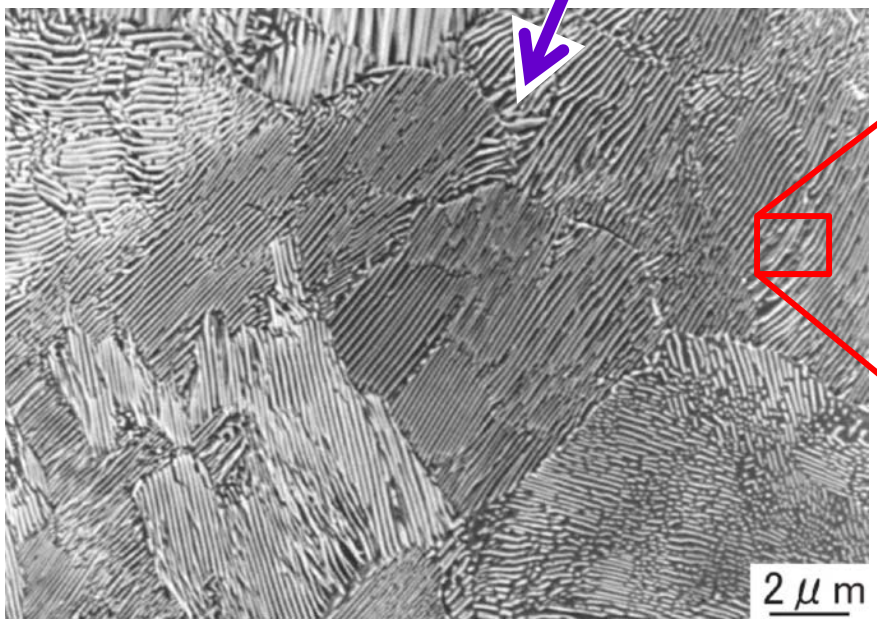
- Describes of the atomic arrangement within phases.

What do we want to identify?

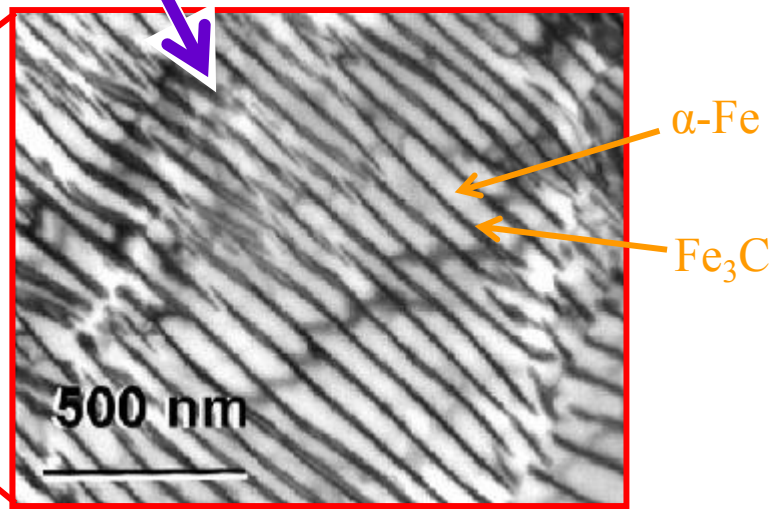
- Distinct crystallographic phases in materials.
  - E.g., cementite and ferrite in a steel.
- Phase morphology (i.e., size, shape, spatial distribution, etc.).
  - E.g., laths, spheroids, etc…
- Chemistries of bulk materials and/or individual phases.
- Can you think of any others?

Levels of Analysis

Qualitative Analysis	Phase identification	Microstructural morphology	Microchemical identification
Identification of: (1) phases present; (2) phase morphology (size and shape); (3) chemical constituents making up each phase.	Medium carbon steels consist of a mixture of ferrite and cementite	Ferrite and cementite have a lamellar morphology in med. C steels	Ferrite consists primarily of Fe. Cementite consists of Fe and C in a 3:1 ratio.



http://www.spaceflight.esa.int/impress/text/education/Images/Glossary/GlossaryImage%20032.png

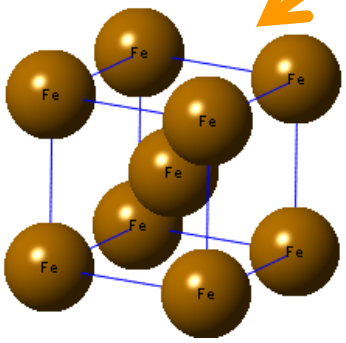


ww.msm.cam.ac.uk/phase-trans/2005/pearlite.JPG

Levels of Analysis

Quantitative Analysis	Applied crystallography	Stereology	Microchemical analysis
Determination of: (1) atomic arrangements (crystallography); (2) spatial relationships between micro-structural features (stereology); (3) micro-chemical composition (microanalysis).	(Bravais lattices) Ferrite is BCC; Cementite is primitive orthorhombic	How are the phases oriented relative to each other?	What is the chemistry of each phase?

http://neon.mems.cmu.edu/degraef/research.html



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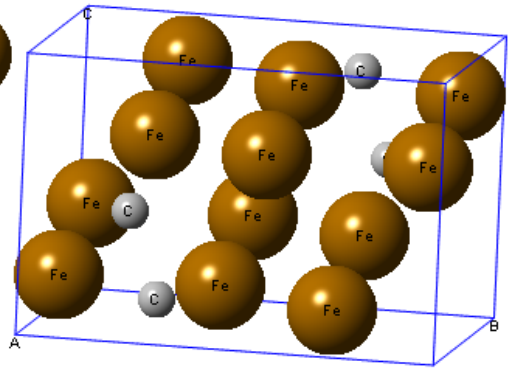

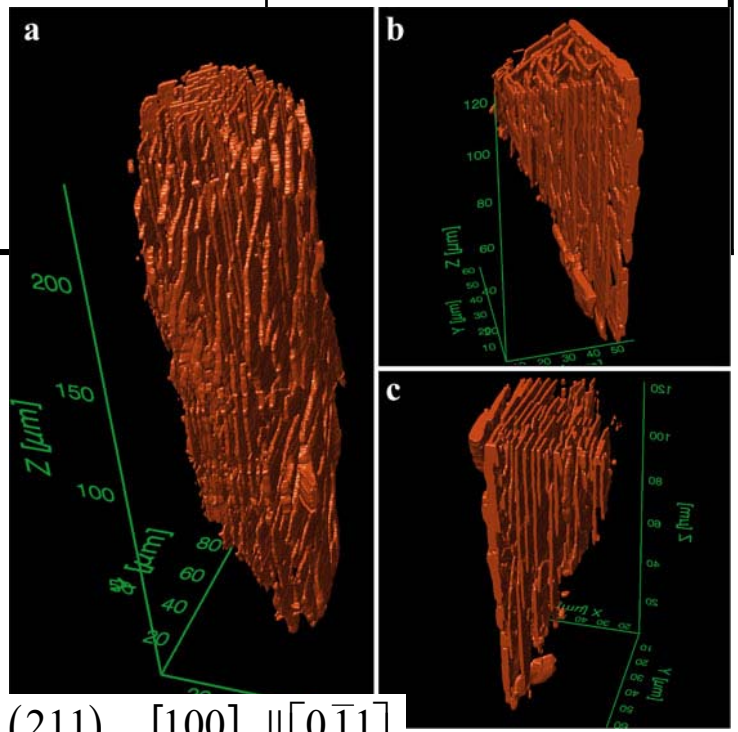

$$(001)_\theta \,||\, (211)_\alpha \,,\, [100]_\theta \,||\, [0\bar{1}1]_\theta$$
$$(101)_\theta \,||\, (112)_\alpha \,,\, [010]_\theta \,||\, [111]_\theta$$

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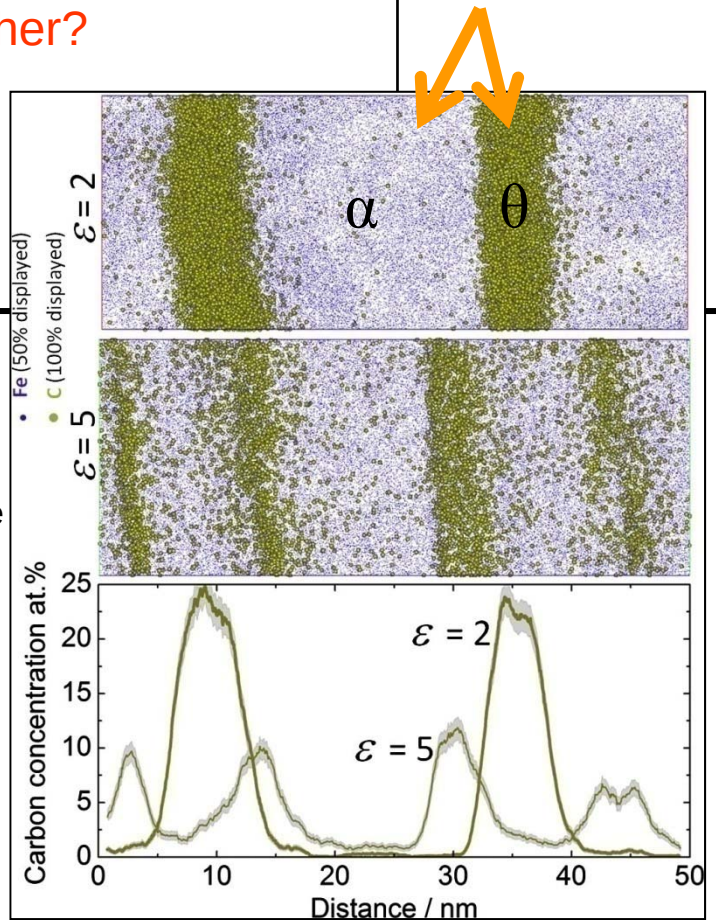
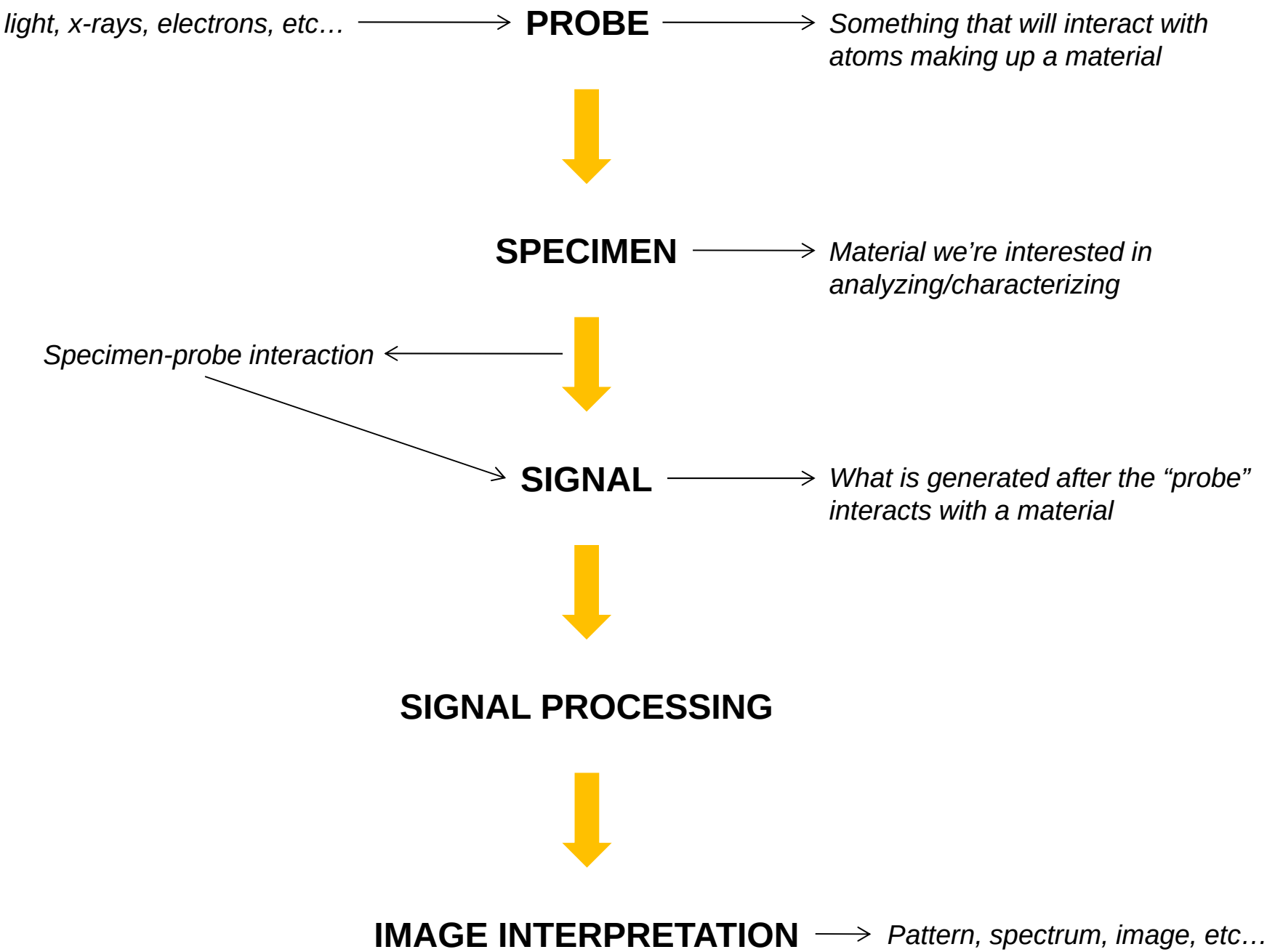


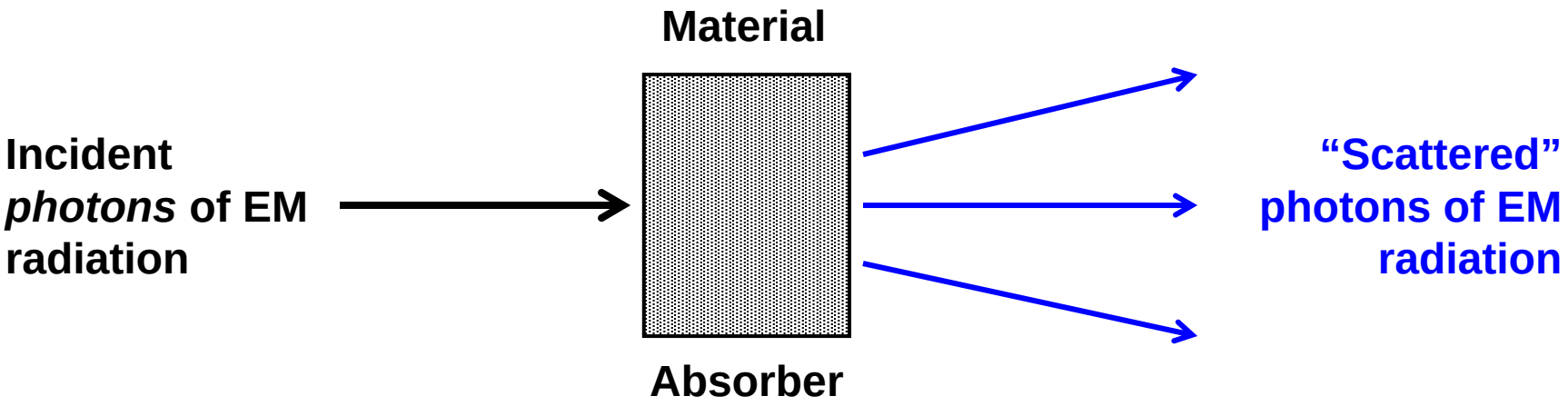
Fig. 4. Atom maps of the selected volume of 4 × 20 × 50 nm^3 in the cold-drawn pearlitic wires at $\varepsilon = 2$ (upper) and $\varepsilon = 5$ (middle). The large yellow dots and the small blue dots represent carbon (100% shown) and iron (50% shown), respectively. The bottom figure shows the corresponding 1D carbon concentration profiles along the direction perpendicular to the lamellar interfaces (also the probing direction). The error bars are marked in gray. Figure from Y.J. Li, P. Choi, C. Borchers, S. Westerkamp, S. Goto, D. Raabe, and R. Kirchheim, *Acta Materialia* 59 (2011) 3965-3977.


light, x-rays, electrons, etc…
PROBE
Something that will interact with atoms making up a material
SPECIMEN
Material we're interested in analyzing/characterizing
Specimen-probe interaction
SIGNAL
What is generated after the "probe" interacts with a material
SIGNAL PROCESSING
IMAGE INTERPRETATION
Pattern, spectrum, image, etc…

Some Common Characterization Methods

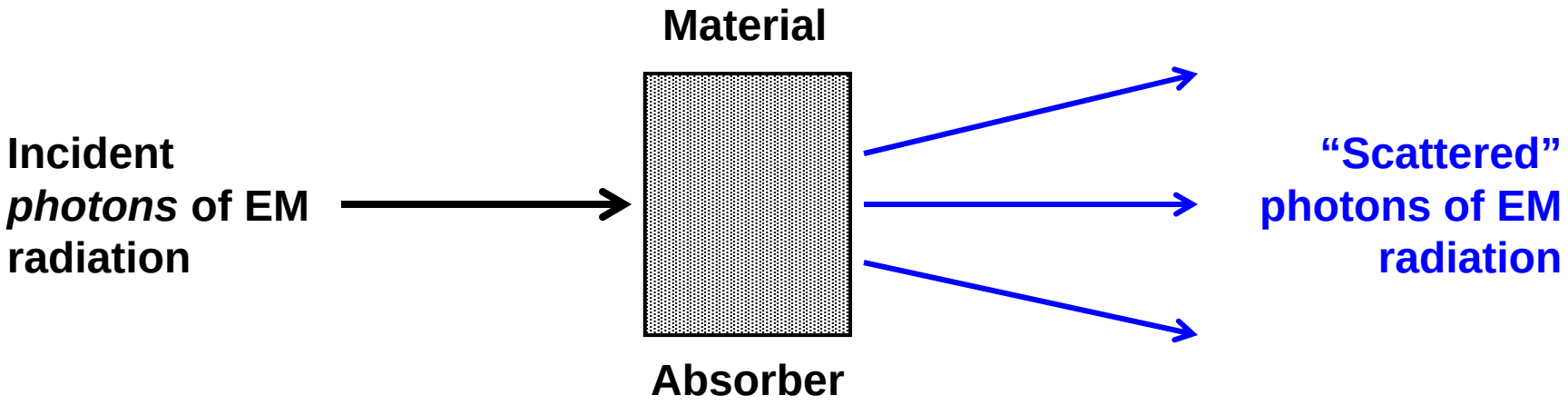
- **Visible light**
  - Optical *microscopy* (OM)
- **X-rays**
  - X-ray *diffraction* (XRD)
  - X-ray photoelectron *spectroscopy* (XPS)
- **Neutrons**
  - Neutron *diffraction* (ND)
- **Ion beams**
  - Secondary ion mass *spectrometry* (SIMS)
  - Focussed ion beam (FIB) *microscopy*
  - Cleaning and thinning samples
- **Electron beams**
  - Scanning electron *microscopy* (SEM)
  - Transmission electron *microscopy* (TEM)
  - Energy dispersive x-ray *spectroscopy* (EDS)
  - Wavelength dispersive x-ray *spectroscopy* (WDS)
  - Auger electron *spectroscopy* (AES)
  - Electron energy loss *spectroscopy* (EELS)

Probe is scattered by a solid.



We use this scattered signal to 'characterize' the structure of a material

Scattering – primary mechanism for reduction of the intensity of the incident beam.

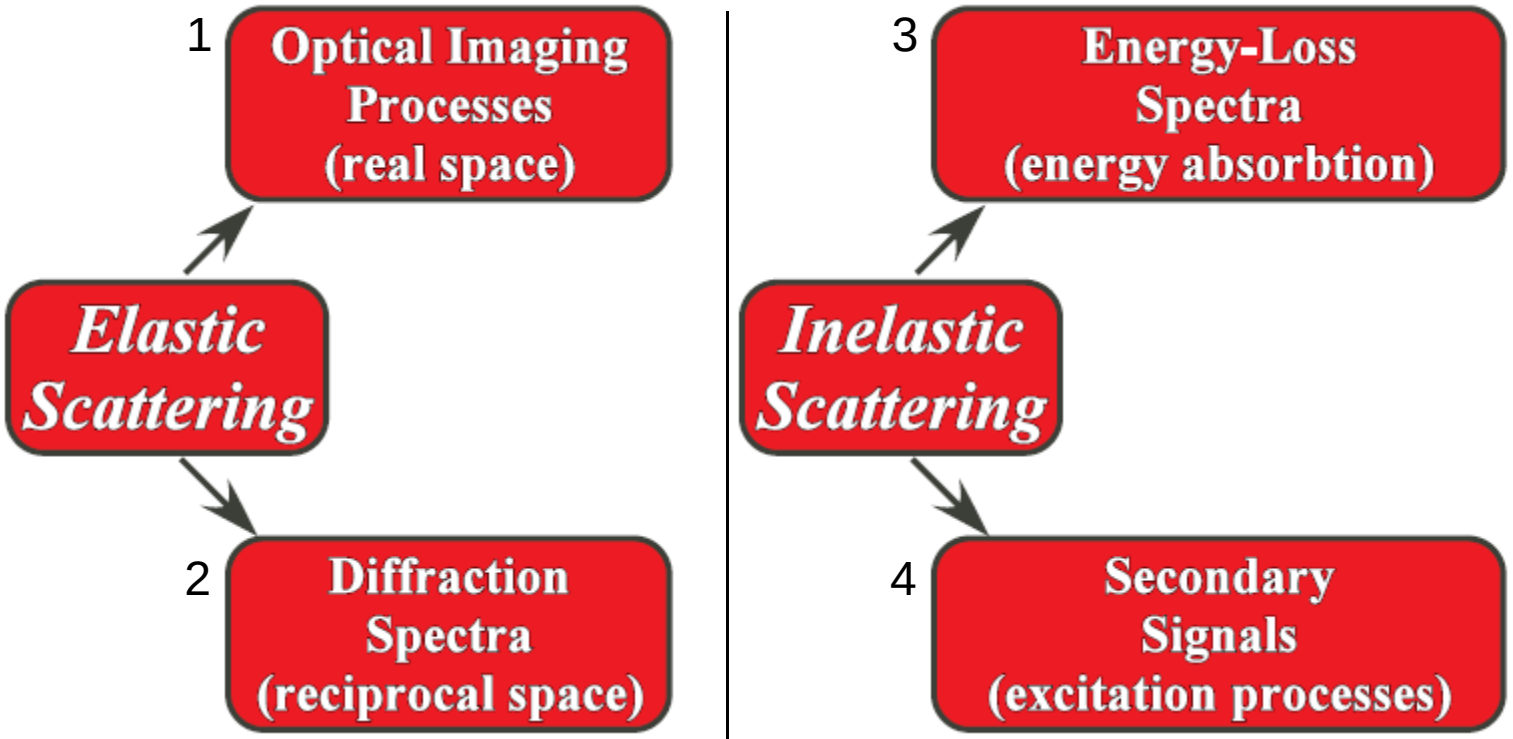


- Elastic scattering – little/no change in energy between the incident *photon* and the emitted photon.

- Inelastic scattering – significant energy loss between the incident photon and the emitted photon.

General Types of Experimental Techniques

- Microscopy
  - *Obtain a 2-D (or 3-D) image of a specimen.*
- Diffraction
  - *Incident signal is deflected w/o intensity loss. Scattered signal is displayed as a diffraction pattern or spectrum.*
- Spectroscopy / Microanalysis
  - *Some of incident signal intensity is lost. Collect signal intensity as a function of energy or wavelength.*



1. Signal can be focused → real space image (e.g., OM, SEM, TEM)

2. Scattering angles can be collected and analyzed in reciprocal space (e.g., XRD or SAD)

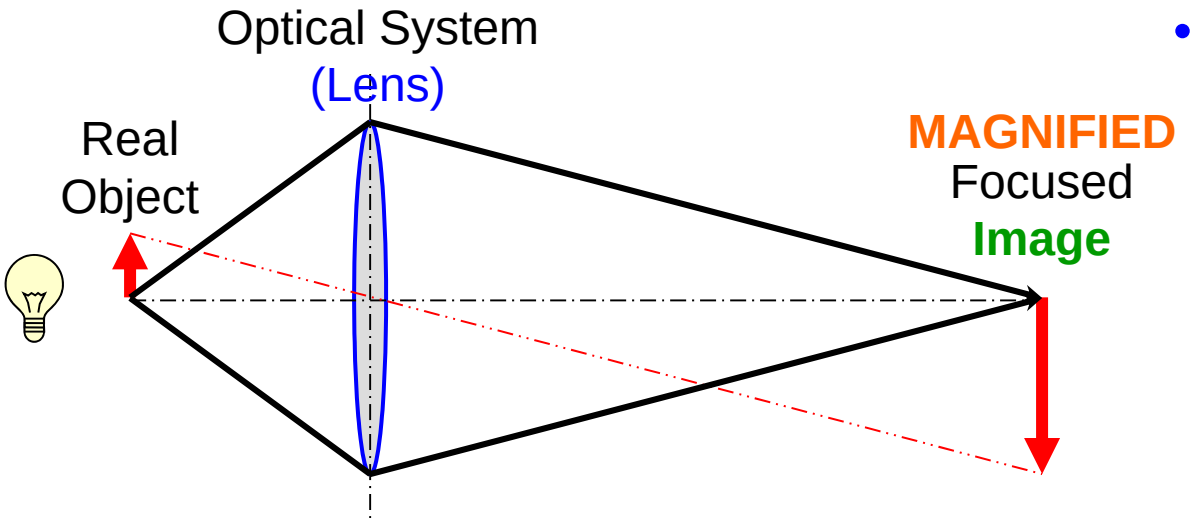
3. Energy loss spectra (*due to absorption of incident radiation*)

4. Secondary signals such as x-rays or secondary electrons (*due to excitation of electrons in material*)

(Figure from D.Brandon and W. Kaplan, *Microstructural Characterization of Materials, 2nd Edition*, Wiley (2008) p. 6)

Elastic Scattering

Images vs. Diffraction Patterns

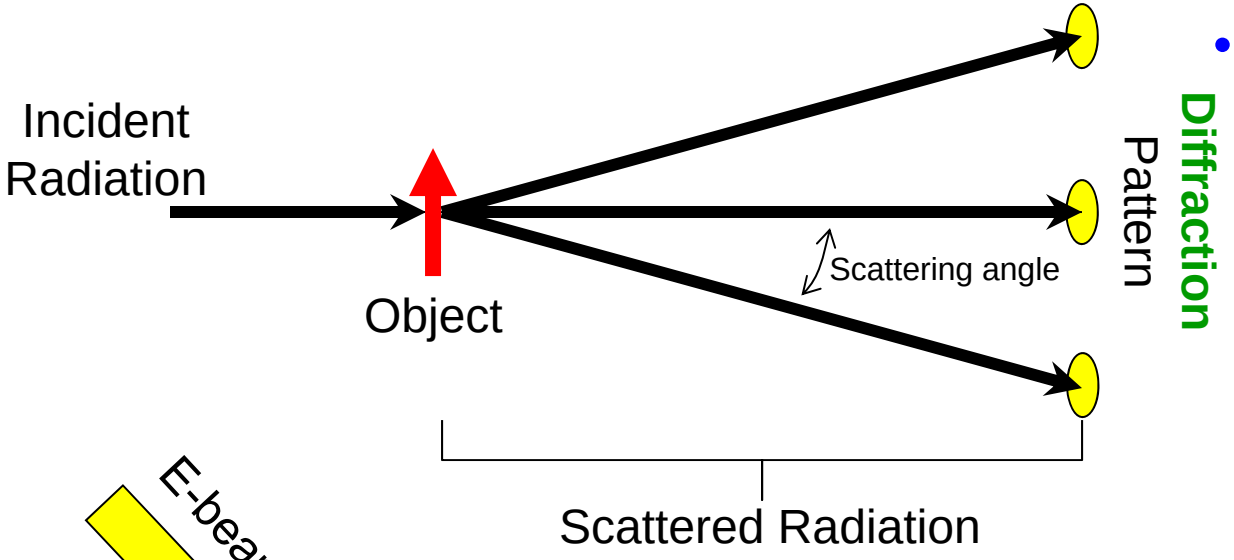


- Real space.
  - Distances in magnified image are directly proportional to distances in object.
  - $\text{Focused image} = M \times \text{Real object}$

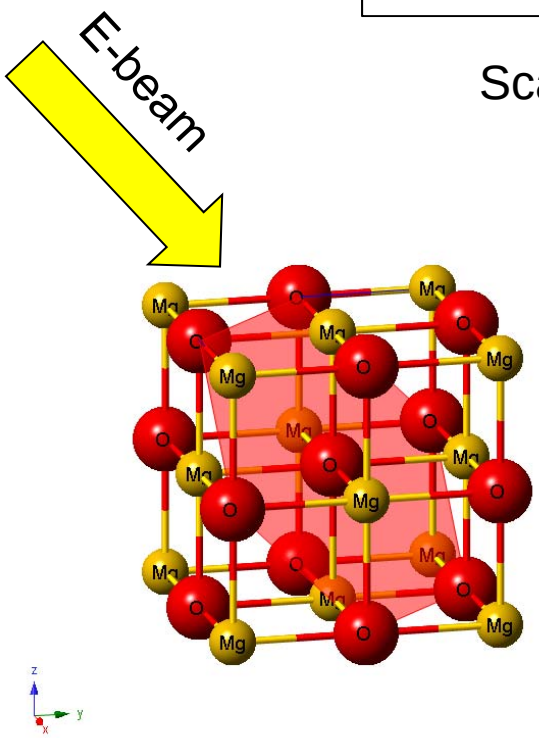
http://www.socialearth.org/wp-content/uploads/2009/12/magnifying-glass.jpg

Elastic Scattering

Images vs. Diffraction Patterns



- Reciprocal space.
  - Scattering angle is inversely proportional to separation of features in an object!
  - Angle $\propto d^{-1}$



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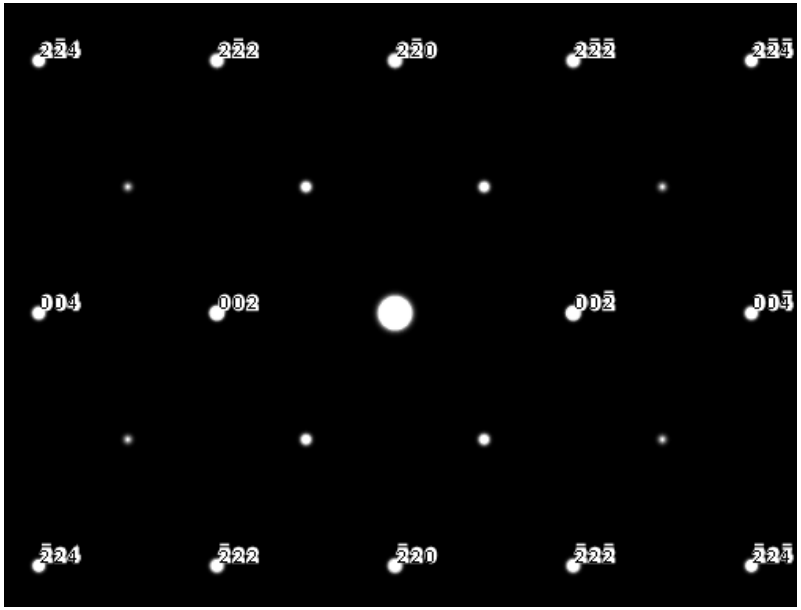


Inelastic Scattering

SEM as an example

Designed to scan surface and detect loss spectra or secondary signals (e.g., secondary electrons).

We use these signals to get image contrast (and to form high resolution images), determine chemistry, etc…

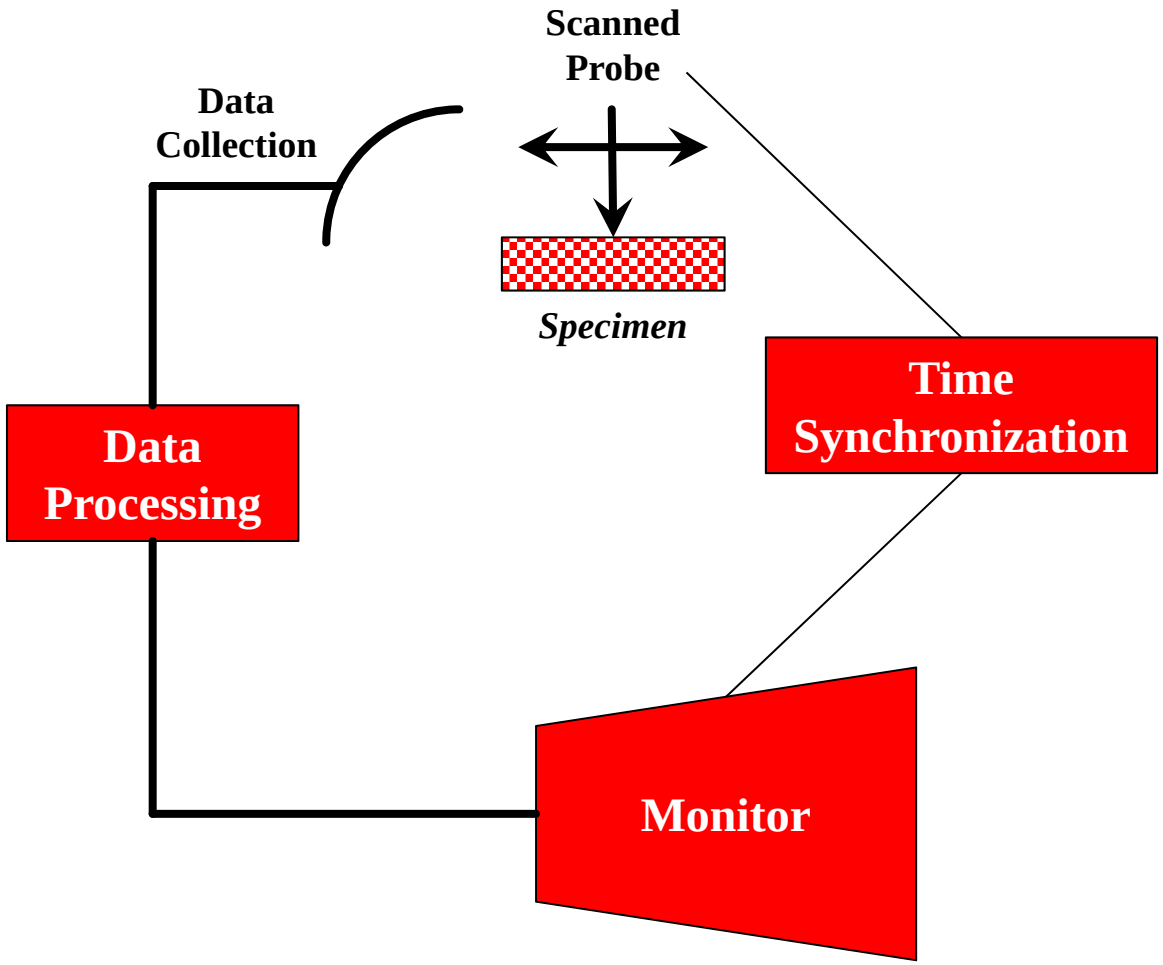


Figure 1.5 *A scanning image is formed by scanning a focused probe over the surface of a specimen and collecting a data signal from it. In an SEM, the signal is processed and displayed on a fluorescent screen with the same time base as that used to scan the probe. The signal may be secondary electrons, characteristic X-rays, or a wide variety of other excitation phenomena.* (*Figure from D.Brandon and W. Kaplan, Microstructural Characterization of Materials, 2nd Edition*, Wiley (2008) p. 6)

Structure-property relationships

Structure-insensitive

- Elastic constants.
- Thermal expansion coefficient.
- Specific gravity.
- Etc…

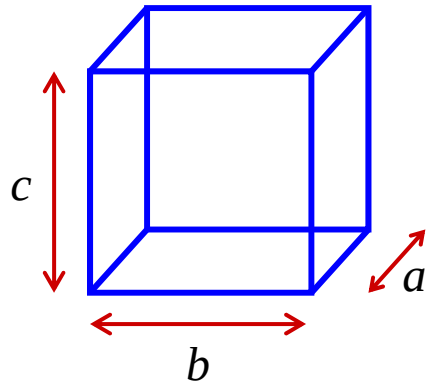
Structure-sensitive

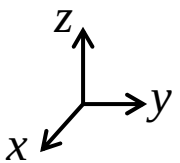
- Yield strength.
  - Grain size.
  - Dislocation density.
  - Vacancy or interstitial content.
- Conductivity (thermal and electrical).
- Fracture toughness.
- Etc…

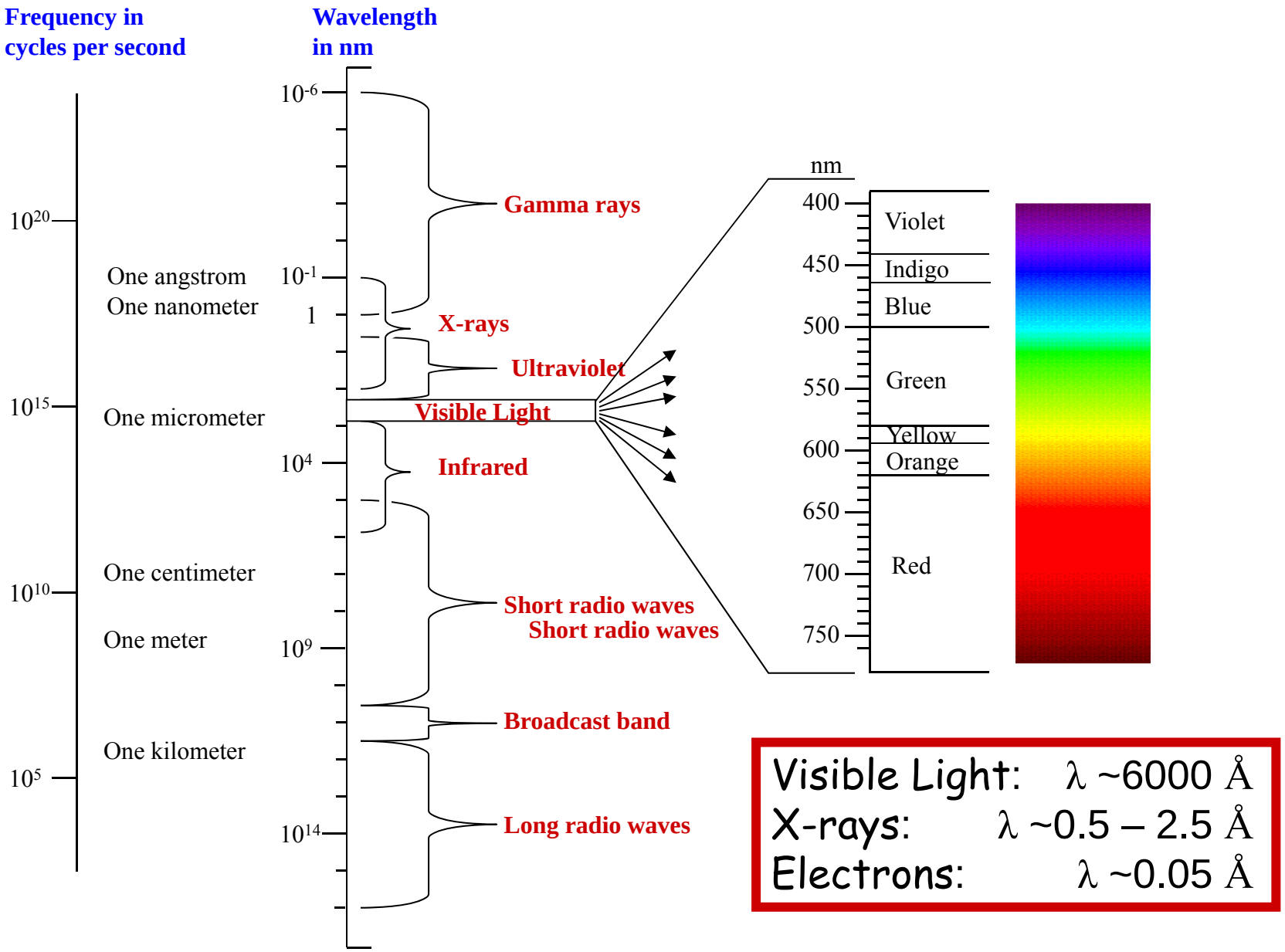
Common Units of Measure in Materials Characterization

	Unit	Equal to	Comments
Wavelength	**1 Å**	10^{-10} m	Traditional unit of measure for EM radiation. Covers visible portion of EM spectrum and x-rays.
	1 nm	10 Å or 10^{-9} m	SI units. Not very popular.
Energy	**1 eV**	1.602×10^{-19} J 1.602×10^{-12} ergs	Energy to move a single electron through a potential (Voltage) difference.

- Unit cell dimensions are defined in terms of Angstroms (Å)
- Approximately 3 eV or more of energy is required for self diffusion in solids!







For high-resolution characterization, x-rays and electrons are superior to light.

WHY?

Electromagnetic Spectrum

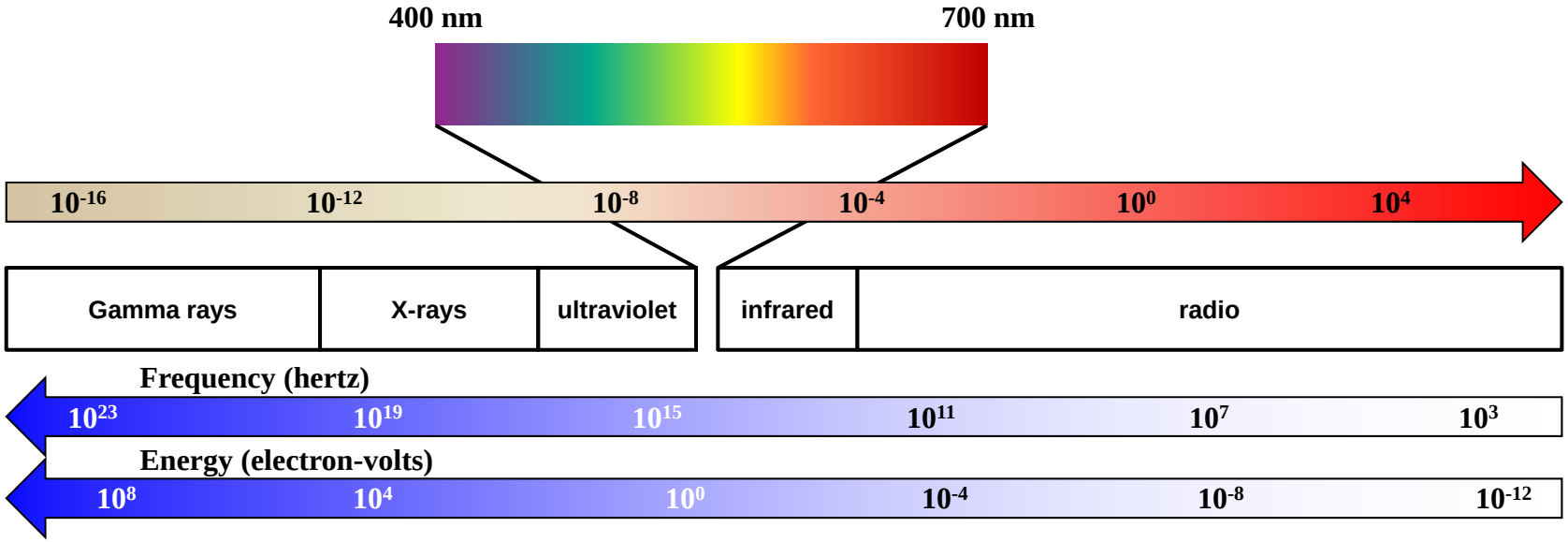


Figure modeled after
http://mrbarlow.files.wordpress.com/2007/09/em_spectrum.jpg

Start reading Chapter 1 in Leng.