Chapter 4: Instruments for Structural, Optical, Electrical Characterizations

4.1 Field Emission Scanning Electron Microscopy (FE-SEM) & Electron Dispersion X-Ray Spectroscopy (EDX or EDS)

Nanotechnology has strongly driven the development of recent electron microscopy, with demands not only for increasing resolution but also for more information from the sample. The field emission scanning electron microscope (FE-SEM) images a sample surface by raster scanning over it with a high-energy beam of electrons. The electrons interact with the atoms comprising the sample to produce signals that contain information about surface topography, composition and other properties, such as electrical conductivity. The function of the electron gun is to provide a large and stable current in a small beam. There are two classes of emission source: thermionic emitter and field emitter. Emitter type is the main difference between the Scanning Electron Microscope (SEM) and the Field Emission Scanning Electron Microscope (FE-SEM).

Thermionic Emitters use electrical current to heat up a filament; the two most common materials used for filaments are Tungsten (W) and Lanthanum hexaboride (LaB$_6$). When the heat is enough to overcome the work function of the filament material, the electrons can escape from the material. Thermionic sources have relative low brightness, evaporation of cathode material and thermal drift during operation. Field Emission is one way of generating electrons that avoids these problems. A Field Emission Source (FES); also called a cold cathode field emitter, does not heat the filament. The emission is reached by placing the filament in a huge electrical potential gradient. The FES is usually a wire of Tungsten (W) fashioned into a sharp point. The significance of the small tip radius (~ 100 nm) is that an electric field can be concentrated to an extreme level, becoming so big that the work function of the material is lowered and electrons can leave the cathode. The FE source reasonably combines with scanning electron microscopes (SEMs) whose development has been supported by advances in secondary electron detector technology. The acceleration voltage between cathode and anode is commonly in the order of magnitude of 0.5 to 30 kV, and the apparatus requires an extreme vacuum (~10$^{-6}$ Pa) in the column of the microscope. Because the electron beam produced by the
FE source is about 1000 times smaller than that in a standard microscope with a thermal electron gun, the image quality will be markedly improved; for example, resolution is on the order of \(~2\) nm at \(1\) keV and \(~1\) nm at \(15\) keV. To resolves a feature on the specimen surface, the beam diameter must be smaller than the feature (still containing high current density). Therefore it is necessary to condense the electron beam. To assist in the demagnification of the beam, electromagnetic lenses are employed. The table below shows the cross over diameter, final spot diameter and demagnification for thermionic and field emitter. Since the cross over diameter in the Field Emission Source is smaller, a lower level of the beam condensation is necessary to have a probe useful for image processing. This makes the FESEM the highest resolution instrument.

**Figure 1.** Schematic diagram of the near field emission scanning electron microscope

<table>
<thead>
<tr>
<th>Electron source</th>
<th>Cross over diameter</th>
<th>Final spot diameter</th>
<th>Demagnification</th>
</tr>
</thead>
<tbody>
<tr>
<td>Therminonic</td>
<td>(10-50) µm</td>
<td>1 nm - 1µm</td>
<td>10,000-50,000</td>
</tr>
<tr>
<td>Field Emission Source</td>
<td>10 nm</td>
<td>1 nm</td>
<td>10</td>
</tr>
</tbody>
</table>

**Table: 1** Specification of FE-SEM resolution

Chapter 4: *Instruments for Structural, Optical, Electrical Characterizations*
The size range in Nanoscience is typically from hundreds of nanometers down to the atomic level (approximately 0.2 nm), in which the materials (nano materials) can have different or enhanced chemical/physical properties compared to the same ones at a bulk size [1–5]. Two main origins for these features are an increased relative surface area and the dominance of quantum effects. An increase in specific surface area will result in a corresponding increase in chemical reactivity, making some nano materials useful as catalysts to improve the efficiency of fuel cells or batteries. As the size of matter is reduced to tens of nanometers or less, quantum effects can begin to play a role, and change the material’s optical, magnetic or electrical properties significantly [3]. For example, gold nano-particles can appear blue, red, or yellow in color as a function of their size. For intended applications using nano-particles, the crucial challenge has been to make all nano-particles the same size [2]. On the other hand, another emphasis has been placed on the shape control of nano materials because in many cases it allows one to fine tune the properties with a greater versatility than can be achieved otherwise [6–8]. In face-centered cubic (fcc) metal particles, for example, the crystallographic {111} and {100} surfaces are different not only in the surface atom densities but also in surface energies, so that single-crystalline silver or gold nano-particles with sizes smaller than ~10 nm show intriguing particle shapes such as truncated octahedra or cuboctahedra [9–10]. In addition, twinned metal particles are found [9–10]. Twinning is the result of two sub grains sharing a common crystallographic plane, and thus, multiple twinning on alternate coplanar planes produces cyclic twinned polyhedra (decahedra and icosahedra) where the twinned tetrahedral subunits are arranged around fivefold axes [11]. Furthermore, construction of superstructures (ordered assemblies or super lattices) of nano materials is of recent key interest not only in future electronics applications but also in fundamental Nanoscience [12–17]. Metal (gold and/or silver) nano-particles are ideal “building blocks” for two- (2D) and three-dimensional (3D) super lattice structures. Preparation of ordered assemblies often requires surface passivation of the building blocks to protect against modifications of their properties by their environment, as well as to inhibit sintering. The organic surface protection of nano-particles enables to self-assemble into their super lattices, so that controlling the chemical functionality of the organic monolayer allows the collective properties of the nano-particle super lattices to be engineered [12]. Electron microscopes use a beam of highly energetic electrons to probe objects on a very fine scale [9, 25]. Therefore, the FE scanning electron microscope (FE-SEM) is a very useful tool for high resolution surface imaging in the fields of nano materials science.
Applications of the field emission scanning electron microscopy (FE-SEM)

- Thickness measurement of thin coatings and films
- Correlation of surface appearance and surface morphology
- Characterization of size, size distribution, shape and dispersion of additives, particulates and fibers in composites and blends
- Measurement of height and lateral dimensions of nanometer-sized objects
- Characterization of cell size and size distribution in foam materials
- Elemental analysis of micron-sized features
- Fracture and failure analysis
- Defect analysis

**EDX or EDS:**

Energy Dispersive X-ray Spectroscopy (EDX or EDS) is a relatively simple yet powerful technique used to identify the elemental composition of as little as a cubic micron of material. The equipment is attached to the SEM to allow for elemental information to be gathered about the specimen under investigation. The technique is non-destructive and has a sensitivity of >0.1% for elements heavier than C. EDS works by detecting X-rays that are produced by a sample placed in an electron beam. The electron beam excites the atoms in the sample that subsequently produce X-rays to discharge the excess energy. The energy of X-rays is characteristic of the atoms that produced them, forming peaks in the spectrum. Please note individual elements may have more than one peak associated with them and some peaks from different elements may overlap to a certain degree. As the electron beam can be precisely controlled, EDX spectra can be collected from a specific point/particle on the sample, giving an analysis of a few cubic microns of material. Alternatively the beam can sweep over a selected area of the sample to identify the elements in that region. In addition, line profiles and X-ray maps can be acquired which depict the elemental distribution across the specimen.
Figure 2. Schematic diagram of EDX detector and its operation principle

Figure 3. Schematic diagram of an energy-dispersive spectrometer and its associated electronics instruments

Chapter 4: **Instruments for Structural, Optical, Electrical Characterizations**
4.2 Atomic Force Microscopy (AFM)

The invention of Atomic Force Microscopy (AFM) in the midsession from 1980 to 1986s, it was followed by continuous progress in instrumentation. A relatively new form of microscopy in which a sharp tip is scanned over the surface of a sample, while detecting the interaction force between the tip and the sample. Because AFM does not rely on an incident beam, as in electron or light microscopy, the specimen can be directly observed at high resolution in aqueous solution. The newly developed atomic force microscope is a valuable tool for studying physical and biological structures provides a unique window to the micro world of cells, sub-cellular structures, and bio-molecules. The AFM can image the three-dimensional structure of biological specimens in a physiological environment. AFM is a kind of scanning probe microscope where imaging of the sample is realized by interaction of the probe with the sample surface and no imaging beam (light or electron) is involved in the process. The tip of the probe is mounted on the end of a flexible cantilever [26]. Sample preparation and recording conditions, has revolutionized the way in which microscopes explore biological structures. This surface imaging technique involves scanning a sharp tip over the surface of a sample, while sensing the interaction force between the tip and the sample of biological specimens at sub-nanometer resolution under physiological conditions. A form of AFM in which the force acting on the tip is measured with piconewton (10-12 N) sensitivity as the tip is pushed towards the sample, and then retracted from it. Atomic force microscopes can be operated in various modes in the constant-force imaging mode, images are created by bringing the tip and sample into contact and scanning the tip across the surface while the sample height is adjusted using a feedback loop to keep the bending or DEFLECTION (The vertical bending of the AFM cantilever resulting from the tip sample interaction force) of the atomic force microscope cantilever constant. This yields a topographic image that gives calibrated height information about the sample. In tapping mode atomic force microscopy (TMAFM), an oscillating tip is scanned over the surface and the amplitude and phase of the cantilever are monitored near its resonance frequency. As the tip touches the sample surface only at the very end of its downward movement, lateral forces during imaging are greatly reduced, which is advantageous for imaging 'soft' biological samples. This is recorded as the tip is pushed towards the sample and retracted from it. Using appropriate corrections, a force versus separation distance curve is obtained. Such a curve can be exploited to gain insights into a variety of surface properties and molecular interactions and to manipulate single molecules. Importantly,
force distance curves can be recorded at multiple locations of the (x,y) plane to yield spatially resolved maps of properties and interactions.

**Figure 4.** Schematic representation of Atomic Force Microscope operation mechanism

**Figure 5.** Schematic diagram of image processing surface morphology through AFM

Chapter 4: *Instruments for Structural, Optical, Electrical Characterizations*
There has been recent success imaging individual proteins and other small molecules with the AFM such as collagen. Employing selective affinity binding procedures has successfully imaged smaller molecules that do not have a high affinity for common AFM substrates.

Figure 6. AFM image of the sample of Protein Surface layers

Figure 7. A. Three-dimensional AFM image of *Saccharomyces cerevisiae* Yeast cell immobilized in a porous membrane.

B. High-resolution deflection image of the surface of *Phanerochaet chrysosporium* Fungal Spores.
The recent innovation, such as Digital Instruments Bioscope system, which combines the high resolution of AFM with the ease of use and familiarity of inverted optical microscopes, has further added to the attractiveness of AFM for biological imaging. Bright-field, fluorescence and other optical techniques can be used to identify structures of interest while the AFM simultaneously generates nanometer-resolved images of the sample surface [27, 28, 30]. The AFM has been used to image living cells and the underlying cytoskeleton, chromatin and plasmids, ion channels, and a variety of membranes. Dynamic processes such as crystal growth and the polymerization of fibrinogen and physicochemical properties such as elasticity and viscosity in living cells have been studied. Nano manipulations, including dissection of DNA, plasma membranes, and cells, and transfer of synthetic structures have been achieved [29-30].

**Applications of Atomic Force Microscopy (AFM)**

- Surface ultra structure of living cells, Cell surface dynamics, and Morphology of biofilms.
- DNA and RNA analysis, Polymers and biomaterials.
- AFM has the ability to measure forces in the nanonewton range. This makes it possible to quantify the molecular interaction in biological systems such as a variety of important ligand-receptor interactions.
- AFM force measurements are to image or quantify electrical surface charge.

### 4.3 Small-angle X-ray Scattering (SAXS) / X-ray Diffraction

SAXS is a technique that is used for the structural characterization of solid and fluid materials in the nanometer (nm) range. It probes inhomogeneities of the electron density on a length scale of typically 1-100 nm, thus yielding complementary structural information to XRD (WAXS - wide angle X-ray scattering) data. It is applicable to crystalline and amorphous materials alike. Measurements are commonly performed in transmission geometry, using a narrow, well-collimated and intense X-ray beam. Scattering angles typically range between 0.1 and 5 deg. The smallest accessible angle determines the largest resolvable feature size. Samples can be easily prepared and are measured either in a capillary, as a thin layer between polymers foils, or free-standing. Some typical applications comprise the determination of nano-particle and pore size
distributions, of specific surface areas and the structure analysis in inhomogeneous (e.g. core-shell) particles. The technique may also yield information with respect to the aggregation behavior of nano-particles. Furthermore, SAXS is applied for analyzing structures in colloids, membranes, liquid crystals, detergents, micro emulsions, polymers etc. Under the condition that all particles essentially have the same size, it is possible to determine their shape and internal structure. As an example, SAXS is used to analyze the envelope shape of proteins and other bio-macromolecules in dilute solution. The measurement time on a laboratory instrument may range from a few minutes up to several hours. It depends on the type of instrument and optics used, on the sample concentration and on the electron density contrast. An evacuated beam path helps in reducing the effects of air scattering and in speeding up the measurements. However, even on a multi-purpose diffractometer in air, high-quality SAXS data can often be obtained for a large variety of samples. The result of a SAXS experiment is essentially the intensity of the Fourier transform of the electron density and must be interpreted in order to determine morphology. One fundamental problem with any scattering experiment is that two different morphologies can, in theory, give identical scattering patterns.

Phase identification in thin film

Phase identification in thin films refers to a diffraction technique that enhances the diffracted signal coming from the thin film. It is often referred as grazing incidence X-ray diffraction (GIXRD). In a GIXRD measurement, the angle of incidence of the X-rays with respect to the sample surface is kept fixed at a low value. This value has to be higher than the critical angle of total external reflection to insure penetration of the X-rays into the thin film. GIXRD measurements are performed in a parallel beam geometry, which makes it non-sensitive to sample height displacement.

Brief introduction & mechanism of X-ray powder diffraction analysis

X-rays are electromagnetic radiation of wavelength about 1 Å (10-10 m), which is about the same size as an atom. They occur in that portion of the electromagnetic spectrum between gamma-rays and the ultraviolet. The discovery of X-rays in 1895 enabled scientists to probe crystalline structure at the atomic level. X-ray diffraction has been in use in two main areas, for the fingerprint characterization of crystalline materials and the determination of their structure. Each crystalline solid has its unique characteristic X-ray
powder pattern which may be used as a "fingerprint" for its identification. Once the material has been identified, X-ray crystallography may be used to determine its structure, i.e. how the atoms pack together in the crystalline state and what inter atomic distance and angle are etc. X-ray diffraction is one of the most important characterization tools used in solid state chemistry and materials science. When a beam of X-rays illuminates a single crystal, many “spots” are generated. The positions of the spots are determined by the size and shape of the unit cell and the symmetry. The intensities of the spots are determined by the arrangement of the atoms within the crystal. After measuring the intensities of all of the diffraction spots (reflections), it is generally possible to determine the positions of the atoms in the unit cell (the structure) in a straightforward manner. Sometimes, however, the sample is more complex (twinning, aperiodic structure, diffuse scattering), and the structural analysis becomes a challenge for even the most skilled crystallographers.

Figure 8. Schematic representation of X-ray diffractometer and operational mechanism (Mechanism and working principle)
Most real solids are not single crystals (that is why we prize gem single crystals so much!), but are composed of large numbers of tiny crystals, so are described as polycrystalline. In the diffraction pattern, the effect is that each of the spots is spread out into a ring. If the crystallites are oriented randomly, the rings are uniform, and no information is lost by measuring along a radius vector of the complete 3-dimensional diffraction pattern. The result is a plot of diffracted intensity vs. angle - a powder pattern shown in detail analysis next chapter 5, (Result and Discussion).

**Figure 9.** Detection of well crystallinity of samples and distribution of crystal lattice diffraction pattern (white lattice spot around black surroundings)

**Applications X-ray diffraction (XRD)**

- Nano-Materials: Phase Composition, Crystallite size and shape, Lattice Distortions and Faulting, Composition Variations, Orientation, In-Situ Structure Development
- Catalyst evaluation
- Reservoir core analysis
- Fuel quality testing
- New materials research and development
- Polymers and Composites: Crystalline form, Crystallinity, Crystalline Perfection, Orientation
- Pharmaceuticals and Organics: Polymorphs, Crystallinity, Orientation

Chapter 4: *Instruments for Structural, Optical, Electrical Characterizations*
4.4 Ultraviolet Visible Spectroscopy (UV-Vis.)

Ultraviolet and visible spectrometers have been in general use for the last 35 years and over this period have become the most important analytical instrument in the modern day laboratory. In many applications other techniques could be employed but none rival UV-Visible spectrometry for its simplicity, versatility, speed, accuracy and cost effectiveness. An obvious difference between certain compounds is their color. Thus, quinone is yellow; chlorophyll is green; the 2,4-dinitrophenylhydrazone derivatives of aldehydes and ketones range in color from bright yellow to deep red, depending on double bond conjugation; and aspirin is colorless. In this respect the human eye is functioning as a spectrometer analyzing the light reflected from the surface of a solid or passing through a liquid. Although we see sunlight (or white light) as uniform or homogeneous in color, it is actually composed of a broad range of radiation wavelengths in the ultraviolet visible and infrared (IR) portions of the spectrum. As shown on the right, the component colors of the visible portion can be separated by passing sunlight through a prism, which acts to bend the light in differing degrees according to wavelength. Electromagnetic radiation such as visible light is commonly treated as a wave phenomenon, characterized by a wavelength or frequency. **Wavelength** is defined on the left below, as the distance between adjacent peaks (or troughs), and may be designated in meters, centimeters or nanometers ($10^{-9}$ meters). **Frequency** is the number of wave cycles that travel past a fixed point per unit of time, and is usually given in cycles per second, or hertz (Hz). Visible wavelengths cover a range from approximately 400 to 800 nm. The longest visible wavelength is red and the shortest is violet. Other common colors of the spectrum, in order of decreasing wavelength, may be remembered by the mnemonic: **ROY G BIV**. The wavelengths of what we perceive as particular colors in the visible portion of the spectrum are displayed and listed below. In horizontal diagrams, such as the one on the bottom left, wavelength will increase on moving from left to right.

![Figure 10](image.png)

**Figure 10.** Wave guide process from the bottom left to right and the wavelength absorbs particular colors in the visible portion.

Chapter 4: *Instruments for Structural, Optical, Electrical Characterizations*
When white light passes through or is reflected by a colored substance, a characteristic portion of the mixed wavelengths is absorbed. The remaining light will then assume the complementary color to the wavelength(s) absorbed. This relationship is demonstrated by the color wheel shown on the right. Here, complementary colors are diametrically opposite each other. Thus, absorption of 420-430 nm light renders a substance yellow, and absorption of 500-520 nm light makes it red. Green is unique in that it can be created by absorption close to 400nm as well as absorption near 800nm.

Early humans valued colored pigments, and used them for decorative purposes. Many of these were inorganic minerals, but several important organic dyes were also known. These included the crimson pigment, kermesic acid, the blue dye, indigo, and the yellow saffron pigment, crocetin. A rare dibromo-indigo derivative, punicin, was used to color the robes of the royal and wealthy. The deep orange hydrocarbon carotene is widely distributed in plants, but is not sufficiently stable to be used as permanent pigment, other than for food coloring. A common feature of all these colored compounds, displayed below in **Fig. 11**, is a system of extensively conjugated pi-electrons.

**Figure 11.** Ultraviolet absorption range of visible spectrum

- **Violet:** 400 - 420 nm
- **Indigo:** 420 - 440 nm
- **Blue:** 440 - 490 nm
- **Green:** 490 - 570 nm
- **Yellow:** 570 - 585 nm
- **Orange:** 585 - 620 nm
- **Red:** 620 - 780 nm
4.5 Photoluminescence Spectroscopy (PL)

Photoluminescence spectroscopy is a contactless, nondestructive method of probing the electronic structure of materials. Light is directed onto a sample, where it is absorbed and imparts excess energy into the material in a process called photo-excitation. One way this excess energy can be dissipated by the sample is through the emission of light, or luminescence. In the case of photo-excitation, this luminescence is called photoluminescence. Photo-excitation causes electrons within a material to move into permissible excited states. When these electrons return to their equilibrium states, the excess energy is released and may include the emission of light (a radiative process) or may not (a non-radiative process). The energy of the emitted light (photoluminescence) relates to the difference in energy levels between the two electron states involved in the transition between the excited state and the equilibrium state. The quantity of the emitted light is related to the relative contribution of the radiative process. When the chemical substrate undergoes internal energy transitions before relaxing to its ground state by emitting photons, some of the absorbed energy is dissipated so that the emitted light photons are of lower energy than those absorbed. One of such most familiar phenomenon is fluorescence, which has a short lifetime (10-8 to 10-4 s).

Figure 12. Energy-level diagram showing why structure is seen in the absorption and emission spectra and why the spectra are roughly mirror images of each other [31].
An excitation spectrum looks very much like an absorption spectrum. The greater the absorbance is at the excitation wavelength, the more molecules are promoted to the excited state and the more emission will be observed.

### 4.6 Dielectric Spectroscopy

Dielectric spectroscopy provides the short-time (AC) behavior of insulators and semiconductors. It measures the electrical susceptibility of the material with intrinsic (ionic/dipolar/atomic/electronic) and proximity (grain-boundary/space-charge) contributions, which are affected by its structural/order-disorder/kinetic transitions, as well as by various relaxations/arrests. Our broadband impedance analyzer (Novo Control) enables probing the electrical response at extreme timescales, covering a rather wide range of conductivities and dielectric losses, along with the dispersive/non-activated behavior. Dielectric Spectroscopy provides a powerful tool for investigating a variety of dielectric processes for both electrical and non-electrical applications. Also called Impedance Spectroscopy, the measurement separates molecular process on the basis of response time, providing a unique relaxation frequency along with a signature variation.
with frequency. Some common dielectric processes investigated by Dielectric Spectroscopy include:

- Small-molecule rotation in liquids - chemical and physical diagnostics.
- Large-molecule reorientation in polymers - physical diagnostics.
- Bulk conduction in solids & liquids and separation of electrode effects.
- Surface conduction and grain-boundary charge in porous materials.
-Interstitial ion effects in various solids.

We also performed in-depth analysis and interpretation as requested. We can collect data in common dielectric formats including complex permittivity as a function of frequency, complex impedance diagrams, and Cole-Cole plots. We model data using standard Debye, Cole-Cole, and Cole-Davidson models, to extract molecular-level information on parameters such as relaxation time, relaxation amplitude, and distribution of relaxation times. From this we draw conclusions on the state of processing of the material, for such properties as viscosity, percent reaction, chemical state of binding, etc.

Materials Science and Instrumentation (MSI) can perform Dielectric Spectroscopy and Impedance Spectroscopy continuously over the frequency range 10 Hz to 10 GHz. The frequency dispersion dielectric data were recorded in regular laboratory instrument at room temperature of frequency ranging from 10 Hz to 13MHz using a HP4192 Impedance Analyzer.

TDR Dielectric Spectroscopy uses an innovative approach to high frequency dielectric spectroscopy. The sensing electrodes are measured not with a continuous frequency wave, but with a rapid voltage pulse containing a broad range of frequencies at once [32-34]. The reflected signal is captured after an appropriate propagation delay, separating sensor response from connecting-line artifacts on the basis of delay time.

**Figure 14.** Schematic representation of data recording of high frequency dielectric Spectroscopy
The advantage of this approach is that the reflected signal is displayed in either time or frequency domain. In the frequency domain the signal is Fourier-Transformed to a dipole rotation spectrum, providing detailed quantitative information on the changing rotation spectrum. In the time domain the signal is monitored in its direct transient form, providing simple qualitative indicators of the changing rotation spectrum and allowing instrument artifacts to be diagnosed more easily. TDR Dielectric Spectroscopy is related to conventional Time-Domain-Reflectometry used in closed-circuit fault testing [35-36]. However, TDR Spectroscopy focuses on a time and frequency analysis from a lumped capacitance sensor while conventional TDR focuses on spatial differences along a distributed transmission line.

### 4.7 Polarizing Optical Microscope (POM)

Polarized optical microscopy is typically used to detect the existence of liquid crystal phases in a solution. The principle of this is corresponding to the polarization of light. A polarizer is a filter that only permits the light oriented in a specific direction with its polarizing direction to pass through. There are two polarizers in a polarizing optical microscope (POM) (Figure 15) and they are designed to be oriented at right angle to each other, which is termed as cross polar. The fundamental of cross polar is illustrated in Figure 16, the polarizing direction of the first polarizer is oriented vertically to the incident beam, so only the waves with vertical direction can pass through it. The passed wave is subsequently blocked by the second polarizer, since this polarizer is oriented horizontally to the incident wave.

![Instructoral analysis of Polarizing Optical Microscope](Copyright: Nikon)
Birefringent or doubly-refracting sample has a unique property that it can produce two individual wave components while one wave passes through it, those two components are termed as ordinary and extraordinary waves. **Figure 16** is an illustration of a typical construction of Nicol polarizing prism, as we can see, the non-polarized white light are splitted into two ray as it passes through the prism. The one travels out of the prism is called ordinary ray, and the other one is called extraordinary ray. So if we have a birefringent specimen located between the polarizer and analyzer, the initial light will be separated into two waves when it passes though the specimen. After exiting the specimen, the light components become out of phase, but are recombined with constructive and destructive interference when they pass through the analyzer. Now the combined wave will have elliptically or circularly polarized light wave, see **Figure 17(A and B)**, image contrast arises from the interaction of plane-polarized light with a birefringent specimen so some amount of wave will pass through the analyzer and give a bright domain on the specimen.
Figure 17. B schematic representation of elliptically and circularly polarized light waves
(Copyright: Nikon Corporation.)

The most common application of LCs is in liquid crystals displays (LCD) through polarizing microscopy techniques [37-39]. **Figure 18** is a simple demonstration of how LCD works in digit calculators. There are two crossed polarizers in this system, and liquid crystal (cholesteric spiral pattern) sandwich with positive and negative charging is located between these two polarizers. When the liquid crystal is charged, waves can pass through without changing orientations. When the liquid crystal is out of charge, waves will be rotated 90° as it passes through LCs so it can pass through the second polarizer. There are seven separately charged electrodes in the LCD, so the LCD can exhibit different numbers from 0 to 9 by adjusting the electrodes. For example, when the upper right and lower left electrodes are charged, we can get 2 on the display.

**Figure 18.** The images of liquid crystals phases different specimens. First order retardation plates are utilized in all of these images. Apparent contrasts are detected here in the image which corresponds to the existence of liquid crystal phase within the specimen.
Figure 19. Schematic diagram of experimental set up Polarizing Optical Microscope (POM). The glass support sheet is placed on a thermo-stage for control of the sample temperature with an accuracy of 0.1 K. Chemical structures of gelling agent and mesogen used in the experiments. The phase sequence of the pure mesogen is cryst. -28°C SmC* 73°C SmA* 77°C N* 85°C Iso.

Figure 20. Microscope images in polarized light with a first-order retardation plate inserted between the specimen and analyzer: (a) polyethylene glycol, (b) polycarbonate, and (c) liquid crystalline DNA (Copyright from Nikon).
The electro-optical measurements were carried out using the experimental setup described above section in detail analysis. The cell was set between two crossed polarizers so that the angle between the polarizer axes and the rubbing direction was 45°. The sinusoidal voltage 0-60 V (at frequency $f = 2$ kHz) was applied to the cell. The voltage was stepwise increased from 0 to 60 V and then decreased back to 0; the total measuring time, i.e., time of voltage application, was about 1 min. The transmittance of the samples was calculated as $\eta = (I_{out}/I_{in}) \times 100\%$, where $I_{in}$ and $I_{out}$ are intensities of the incident and transmitted light, respectively.

The effect of rotation of the polarizer at the angle between horizontal direction and polarizer transmission axis on the appearance of liquid crystal phase may be analyzed. In Figure 22 is shown images of an ascorbic acid (Figure 21) sample under cross polar mode. When the polarizer rotates from 0° to 90°, big variations on the shape of bright domains and domain colors appear due to the change of wave vibrating directions. By rotating the polarizer, we can have a comprehensive understanding of the overall texture.

![Figure 21](image1.png)

**Figure 21.** Cross polarized Microscope images of ascorbic acid specimen with polarizer rotation of (a) 0°, (b) 45°, and (c) 90°. Copyright: Nikon Corporation.

![Figure 22](image2.png)

**Figure 22.** The chemical structural formula of ascorbic acid
4.8 KEITHLEY Source Meter For (I~V) Characteristics

Keithley's Source Meter family is designed specifically for test applications that demand tightly coupled sourcing and measurement. All Source Meter models provide precision voltage and current sourcing as well as measurement capabilities. Each Source Meter instrument is both a highly stable DC power source and a true instrument-grade $6 \frac{1}{2}$ digit multimeter. The power source characteristics include low noise, precision, and read back. The multimeter capabilities include high repeatability and low noise. The result is a compact, single-channel, DC parametric tester. In operation, these instruments can act as a voltage source, a current source, a voltage meter, a current meter, and an ohmmeter. Manufacturers of components and modules for the communications, semiconductor, computer, automotive, and medical industries will find the Source Meter instruments invaluable for a wide range of characterization and production test applications.

![KEITHLEY source meter interfaces with GPIB card through PC](image)

The Model KEITHLEY 2611A SYSTEM solution is a 20W instrument that allows sourcing and measuring voltage from $\pm 5\mu V$ (sourcing) and $\pm 1\mu V$ (measuring) to $\pm 200V$ DC and current from $\pm 10pA$ to $\pm 1A$. It's well-suited for testing a wide range of devices, including diodes, resistors, resistor networks, active circuit protection devices, and portable battery-powered devices and components. It's also useful for systems power sourcing and IDDQ testing applications.
Device based applications of KEITHLEY’s source meter

- Discrete semiconductor devices
- Passive devices
- Transient suppression devices
- ICs, RFICs, MMICs
- Laser diodes, laser diode modules, LEDs, photo detectors
- Circuit protection devices: TVS, MOV, Fuses, etc.
- Airbags
- Connectors, DC switching, I–V characteristics.

4.9 Characterizations and Instrumental Specifications

Field Emission Scanning Electron Microscopy (FE-SEM) [Model: Hitachi- FE-SEM, S4800-JU,SE(M,LA0)] was used to record the scanning electron micrograph confirmation of nanostructure morphology analysis for CdS, TiO₂ and ZnO (e.g. ZnO-nanoparticles, nanorods, porous nanorods and nano-flakes itself only). Typical others nanostructures of ZnO nano-spikes formed microflower, nano-capsules bunch over nano-rods, nano porous surface evolved under OAFLCs (W-182) doped investigated by another instrument of E-SEM (Make FEI Model: Quanta mark II) and EDX also.

Scanning Electron Microscopy (SEM, JU-MET at 18kV) analysis of CdS nano doped liquid crystals Felix-017/100 mixture (Clairant, Frankfurt, Germany) study itself only.

Crystalline structure of the synthesized production with X-ray diffraction studies (XRD) using with a diffractometer (Model: Bruker D8 Advance) with Cu Ka line investigated for CdS, TiO₂, both pure and nano materials doped liquid crystals itself. The crystalline structure of the synthesized CdS nanowires doped liquid crystals (Felix) with X-ray diffraction studies by using another X-ray machine and data were recorded at a scan rate of 0.05° per sec by using a Seifert XRD 3000P diffractometer with Cu-Ka radiation (0.01418nm). Similar, highest crystalline peaks detected.
through another XRD machine (Model: X'pert Pro made by PANalytical). ZnO nano-spikes formed microflower, nano-capsules bunch over nano-rods, nano porous surface and doped OAFLCs (W-182) investigated itself only.

- Preparation of thin film and surface smoothness done by programmable spin coater [Apex Instruments Co. Model: SCU-2008C].
- The behavior of surface structure analysis, thickness of thin film surface area and roughness measured through Atomic Force Microscopy (AFM) [Model: Veeco, diInnova].
- Optical absorption and transmittance also measured through Ultra Violet visible scanning spectrophotometer (Model: UV-2401PC, TCC-240A, Shimadzu, Japan) was used to record the electronic absorption spectra of the films at room temperature.
- For high Luminous nano materials like: CdS, ZnO and TiO$_2$, the luminescence property also investigated by Photoluminescence spectroscopy applying slit width of excitation wavelength 15nm and emission wavelength 12 nm [Model: LS55, Fluorescence spectrometer, PerkinElmer] using excitation source of the 325 to 675 nm, line of a He-Cd laser, spectrum looks very much like an UV absorption region.
- The frequency dispersion dielectric data for CdS, Zno and TiO2 both pure nano materials and doped liquid crystals were recorded at room temperature of frequency ranging from 10 Hz to 13 MHz using a HP4192 Impedance Analyzer dielectric spectroscopy (Model: HEWLETT PACKARD 4192A LF IMPANCE ANALYZER ).
- DC biasing of bi-stable switching of current flow through (I~V) response characteristics measured by a source meter (Model: KEITHLEY 2611A SYSTEM).
- For electro-optic measurement done by Optical Polarizing Microscope (POM) with a (Model: HP 33120A) signal generator and a (Model: HP 54603B) oscilloscope were used. Different isotropic temperature of nano doped liquid crystals (Felix-017/100) texture (e.g. on and off state) kept snap sorts Cannon digital camera analogous to that polarizing microscopy.
Chapter 4: Instruments for Structural, Optical, Electrical Characterizations

References:


Chapter 4: **Instruments for Structural, Optical, Electrical Characterizations**


Chapter 4: *Instruments for Structural, Optical, Electrical Characterizations*

(30) [Www.chembio.uoguelph.ca/educmat/chm729/](http://Www.chembio.uoguelph.ca/educmat/chm729/) afm/applicat.htm Hong-Qiang Li: Biological Applications of AFM (1997).


(36) "Time Domain Reflectometry Clearinghouse": [http://www.itl.northwestern.edu/tdr](http://www.itl.northwestern.edu/tdr).

