A Comprehensive Review of Features, Applications and Techniques in Materials Characterization

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Abstract – The realm of materials characterization is as vast and varied as the domains of materials and engineering put together. During the course of decades or centuries, several techniques went from being fairly simple to becoming quite complex. Mechanical, electrical, and thermal properties are all measured and tested, including numerous other materials, such as semiconductors, metals, insulators, ceramics, composites, and polymers; atomic, nanometer, micrometer, millimeter, and centimeter scales; picoseconds to many practice years; and simulation eons. Materials measurement may be performed in a variety of technical contexts, from pure research with murky connections to potential engineering uses to more pragmatic "real-world" field testing, which has the capacity to predict performance, and (potentially) avoid failure of components. Mastering the microstructure of materials expanded greatly in 20th century's second half, and this was made possible by enhancement of novel strategies for characterizing materials. Atomic penetration, X-ray diffraction, thermogravimetric analysis, electron heating and imaging are only some of the direct and indirect methods now used to analyze materials. This article discusses the scope, kinds, and applications of these methods.

Keywords – Materials Characterization, Differential Thermal Analysis, Thermogravimetry, Differential Scanning Calorimetry.

I.   INTRODUCTION

The physical, electrical, and chemical characteristics of materials are characterized via their study and comprehension. To determine which material is best suited for a given use, this step in materials engineering analysis is crucial. The methods utilized in materials characterisation are discussed in today's blog, along with the benefits and drawbacks of each method. Properties like mechanical strength, thermal stability, electrical conductivity, and corrosion resistance may all be analyzed with the use of modern material characterisation methods. By using these methods, engineers may better understand the material properties of system components and optimize their designs for increased performance and dependability. This enables them to make more informed choices on their usage in the development of safe and efficient goods, as well as novel materials whose behavior is tailored to their intended purpose.

Many materials may be characterized by means of microscopy and spectroscopy. Microscopy [Optical, Scanning Electron Microscopy (SEM) [1], and Transmission Electron Microscopy (TEM) [2]] is employed for microstructural studies. NMR, Raman, FTIR, XRD, XRF, and SEM are only few of the spectroscopic methods often used for chemical composition investigation (EDS). Modern society requires effective and reliable characterization procedures to effectively meet the demands of future obstacles to research and development. Several characterisation approaches for material analysis are required to verify practical work by academic and professional researchers. The significance of these methods stems from the fact that macroscopic structure is linked to bulk characteristics. These methods may be used to characterize a wide range of materials for many purposes. The investigated substance responds to a variety of stimuli in order to produce results. Several detectors are used to analyze these readings and draw conclusions about the underlying material structure (micro, macro, nano, etc.). Spectroscopy and microscopy are two typical tools for analyzing materials' properties.

Changes in our knowledge of the structure of materials have not kept pace with the scientific and technical developments in industry that occurred in the seventeenth century and continued until the late nineteenth century. It was not until the invention of X-ray diffraction in 1911–12 that indirect methods like dilatometry and heat analysis could be

employed in determining the crystalline construction of different nanomaterials. Later, the metallurgist could describe the phases and microstructure transformations that occur during metal processing, and the connection to the materials' attributes became intuitive. Microstructures and minute features were more easily detected with electronmicroscopy than with optical microscopes. **Table 1** displays popular methods for investigating microstructure, the size of'microstructural' features, and the magnification necessary to expose the feature.

Table 1: Micro-structural feature scale

Scale	Nanostructure	Microstructure	Mesostructure	Macrostructure
Characteristics microstructural elements	Interface and crystal structure	Dislocation sub-structures	Particle and grain sizes	Production defects
	Point-defect clusters and point defects	Precipitation aspect; phase and grain boundary	Phase anisotropy and morphology	Cracks, porosity and inclusion
Typical experimental methods	High-resolution transmission electronic microscopy	-	-	Ultrasonic inspectins
	Microscopy tunneling scanning	Atomic force microscopy	Scanning electronic microscopy	X-ray radiography
	X-ray diffraction	Transmission and scanning electronic microscopy	Light optical microscopy	Visual inspection
Normal magnification	x 1000000	x 10000	x 100 to x 1000	x 1

Traditional approaches of thermal analysis are first decoded in this paper. These include: evolved gas analysis, and evolved gas detection, mechanical dynamical analysis (DMA), termomechanical analysis (TMA), differential thermal analysis (DTA), thermogravimetry (TG/DTG), and differential scanning calorimetry (DSC). Examining mineralogical analysis using x-ray diffraction is the next stage. Atomic absorption spectroscopy (AAS) and X-ray fluorescence (FRX) are two of the chemical analysis methods that will be discussed in the last section. This is how this article has been organized: Section II presents a methodology of the research. Section III presents a critical analysis of the results and discussions of thermal analysis, microscopy analysis, mineralogical analysis, and chemical analysis. Section IV is the last section, which provides final remarks to the research.

II. METHODOLOGY

Methods for improving text disposition via planning were used in this work. A further bibliographic study of analytical chemical equipment and methods for materials characterisation was carried out. The primary goal was to learn about the practicality and unique qualities of the techniques used to define materials, rather than the specifics of the instruments used. Materials engineering, chemical analysis, and mineralogy were all included into the research for a well-rounded look at the topic.

III. RESULTS AND DISCUSION

Thermal Analysis

In terms of natural science history, it is not overstated to say that "TA is the late fruit of researchers' passion of analyzing matter". From Aristotle's day (384 B.C. to 322 B.C.) the doctrine "Fire is the universal analysator of matter" has been widely accepted. The claim that fire is a considerable analyser of matter due to the ideology that fire is considered destructive was denied by Robert Boyle (1626 to 1691), in his well-known work The Skeptical Chymist (in 1661). Joseph Black (1728 to 1799), who lived a century later, was the first to differentiate between heat and temperature (heat quality), including between sensible heat (capable to be felt) and latent heat (quantity of heat) [3].

Modern Thermal Analysis (TA) techniques have their roots in clay minerals, which are relevant to the chemicals under investigation. The first person who carried out a TA research, which would have met current standards, was possibly Antoine Lavoisier (1743 to 1794). He tested a clay mineral under heating while studying mass changes caused by oxidation; Cronstedt termed it Montmorillonite in 1788. Lavoisier focused primarily on the mass change, whereas Henri Le Chatelier (1850 to 1936), who published an influential study on clays in 1887, was more interested in the aspects of heat and temperature. Le Chatelier is also credited with developing the Pt-PtRh10 thermocouple, which is used to measure very high temperatures. Josiah Willard Gibbs' phase rule and the thermocouple's difference measurement concept are two more significant contributions to modern TA (1839 to 1903) [4].

The concept of TA is widely employed in characterizing analytical experimental processes, which examine a sample's behaviour as a temperature function. The employment of TA is growing in both academic and commercial applications, and it is now being used to study a variety of materials such as natural, pharmaceutical, inorganic, organic, petrochemical products, construction materials, coverings, glassware, catalysis, food, ceramics, grease, composite polymers, and surfactants. It is possible to classify the TA techniques according to three categories of physical factors that must be evaluated: mechanical parameters, heat flow or temperature flow, mass, and miscellaneous characteristics (see **Fig. 1**).
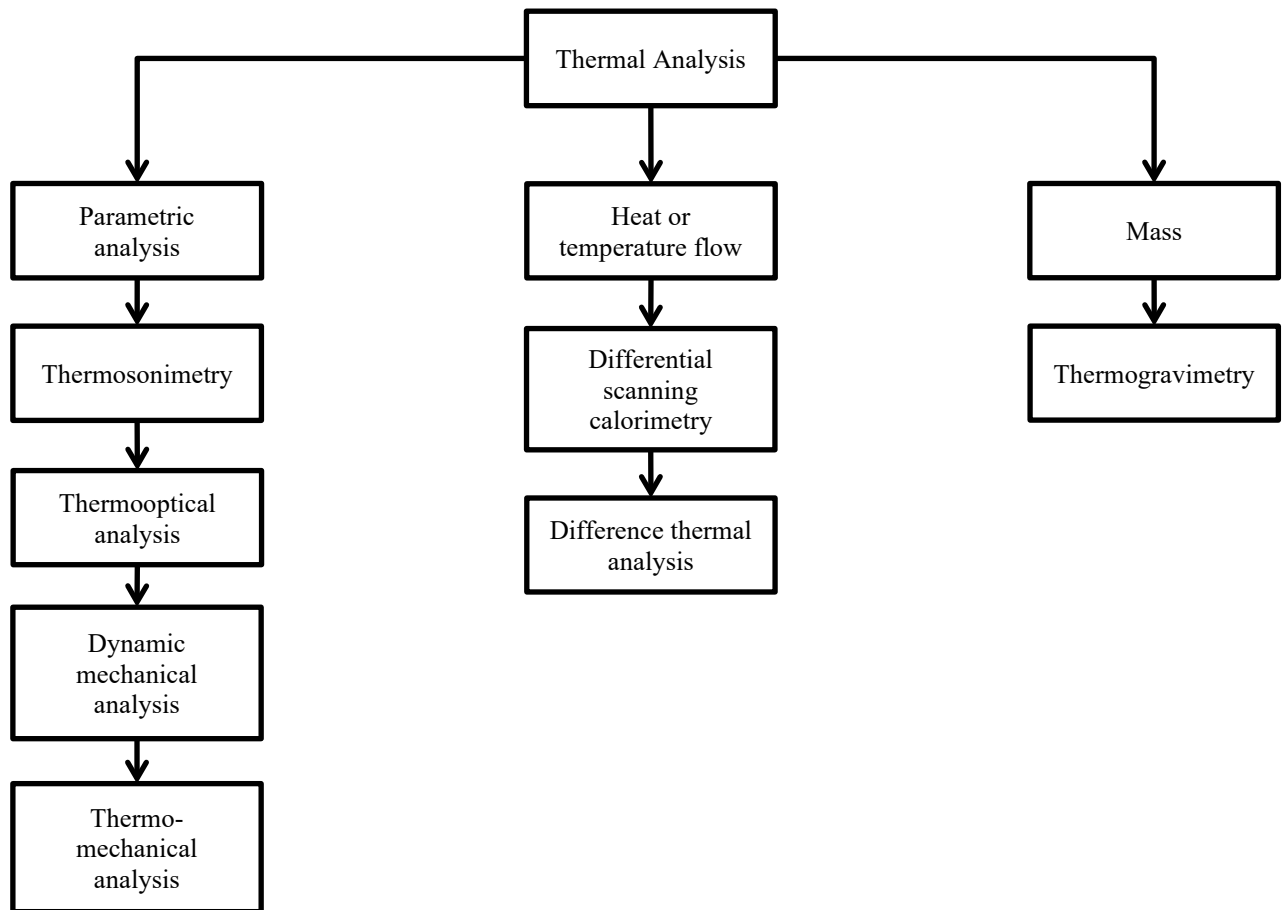
Fig 1. Methods and parameters in TA

The system does not include reaction calorimetry, for which several applicative solutions have been created. It is undeniably a part of thermal analysis techniques. As contrast to traditional TA or calorimetry, reaction calorimetry is conducted in significantly larger reaction containers (such as glass reactors used to create adiabatic conditions). Mostly, it helps bring chemical processes up to an industrial scale.
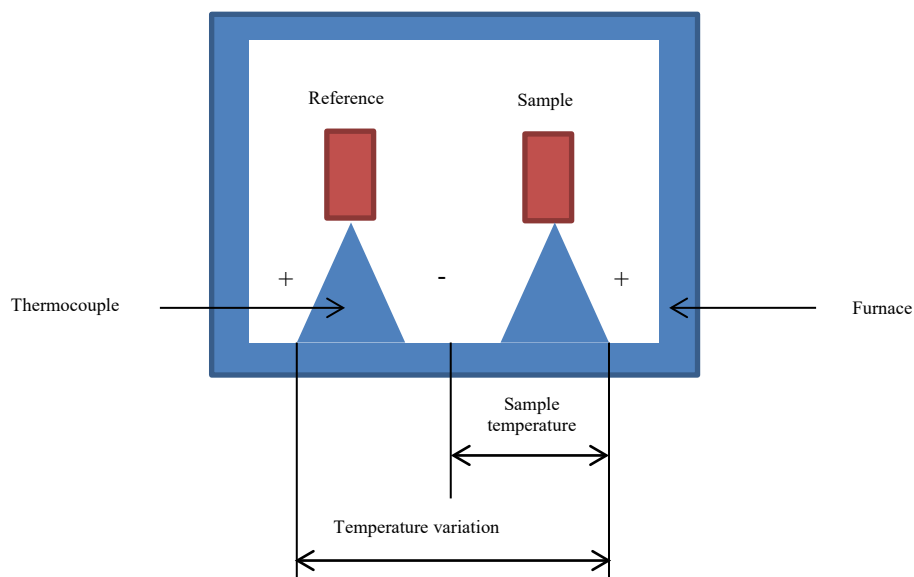
Differential thermal Analysis

Fig 2. DTA block representation

When a sample is cooled or heated at a constant temperature, the variation in temperature between a thermally inert substance and the sample may be recorded using a method called differential thermal analysis (DTA) [5] (see **Fig. 2**). The furnace's center is where the sample holding assembly will go. An inert reference material like alpha-alumina is placed in one holder while the sample is embedded in the other. The temperature programmer regulates the furnace's heat, and thermocouples placed in the holders record the range of temperatures experienced by the reference and the sample. The difference in temperature is shown as a temperature or time function on a DTA curve (isothermal mode).

The sample's ability to absorb or emit heat makes it impossible to keep the temperature ramp constant throughout a phase transition. The DTA curves may be used to learn more about the structure and arrangement of the material. Finally, the variations provide invaluable insight on the substance. Both the specimen and the standard are put into the furnace in a mirror-image fashion. Temperature control software modifies the furnace's heat output in response to changes in the sample and reference temperatures. A differential thermoelectric is used to measure the change in temperature from the sample to the standard. In addition, the specimen temperature is additionally determined using a thermocouple on the side of the sample.
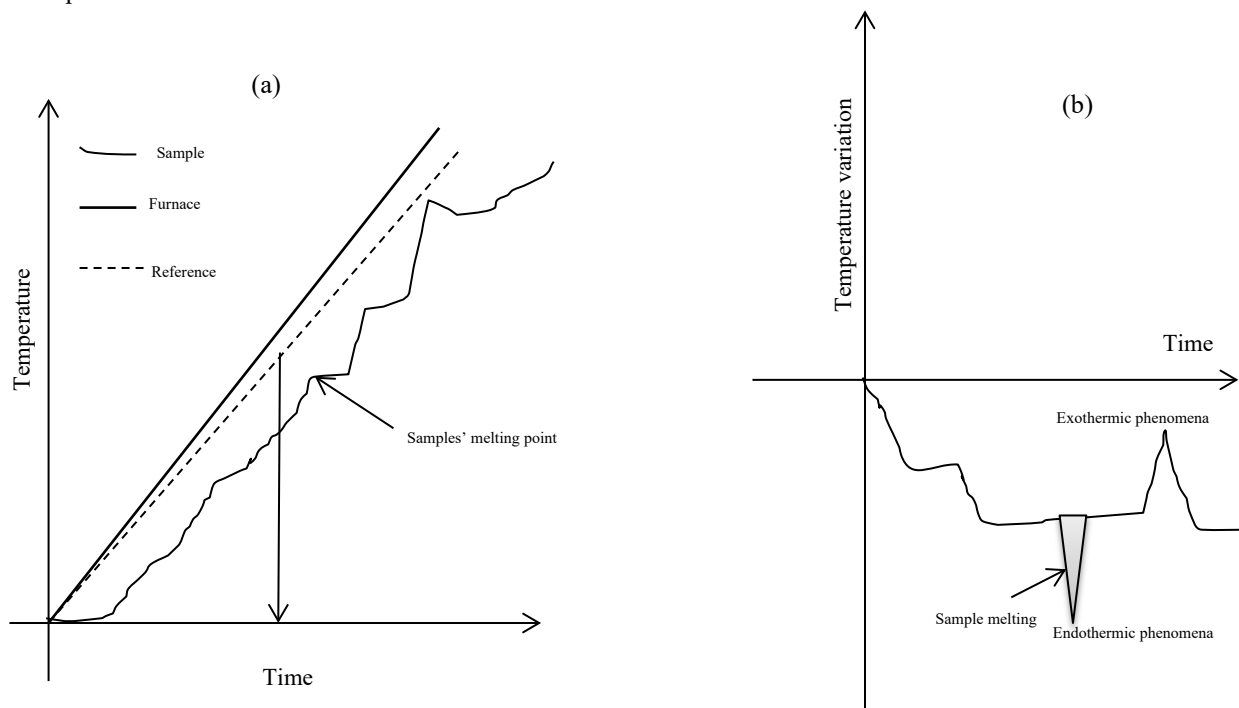

Fig 3. DTA principles of measurement

Graph in **Fig. 3** (a) displays the time-dependent rise and fall in furnace, reference, and sample temperatures. Differential thermocouple readings of temperature differences (T) over time are shown in **Fig. 3** (b). The DTA signal designates a T signal. Temperature-invariant substances (often a-alumina) are employed as standards. When the furnace is turned on, both the reference and the sample heat up gradually based on their individual heat capacities until reaching equilibrium with the furnace temperature. When the heating process has begun, T will fluctuate until it reaches a steady state, at which point it will rise by a predetermined amount to account for the disparity in heat capacity between the sample and the standard. The baseline represents the signal in its unchanging form.

As illustrated in graph **Fig. 3** (a), when melting begins in the sample the temperature increases no further and the T increases. The temperature curve quickly returns to the baseline after the melting stops. The graph shows that the T signal peaks right now **Fig. 3** (b). We may then use the resulting T signal to determine both the transition temperature and reaction temperature of the sample (DTA signal). Graph **Fig. 3** (b) depicts the difference in temperatures as a negative slope for the endothermic change in the sample and a positive slope for the exothermic change.

The transformation in the temperature measurement in the sample can be steered by different enthalpy reactions, such as vaporization, melting, oxidation, decomposition, and reduction among others. Generally, phase reductions, transitions and various decomposition activities produce an endothermic effect, whereas oxidation, crystallization and a variety of decomposition reactions produce an exothermic effect. Due to the fact that the peak size is proportionate to the change in heat entailed; the strategy may be used to make measurements that are semi-quantitative, and in certain situations, quantitative determinations, of heat reactions. It has been successfully used, for instance, to the estimation of maximumnucleation in glasses, and offers a number of benefits over the more traditional approach. Important crystallization characteristics, such as enthalpy and activation energy, are also examined using differential thermal analysis (DTA).

Differential scanning calorimetry

Differential scanning calorimetry (DSC) refers to a strategy, which integrates heating simplicity and cooling curve measurement with different calorimetry quantitative aspects. Temperature is evaluated constantly, and a differential methodology is employed in evaluating the flow of heat into the sample and to equalize the losses and gains of accidental heat with the sample. It is important to remember that calorimetry is not a direct approach of determining the content of heat. A DSC curve is significantly similar to the DTA curve, with the exclusion of the Y-axis catalogue. The DSC curve, like the DTA technical, is proportionate to enthalpy variation. Degradation, oxidation, and crystallization of polymers are all exothermal processes that may be examined using DSC. Past the measurements of a variable heat, the crystallization level, and crystal melting temperature, there are other instances, such as the transformation from a firm to glass transition and brittle rubber.

Differential scanning calorimetry (DSC) is a thermoanalytical strategy for calculating the predicted heat variation needed to raise the temperature of a sample. Throughout the process, the standard and the sample are made constant at the sample temperatures. In most cases, the temperature of the sample holder will rise linearly with time as part of the DSC measurement's temperature program. The technique involves measuring the variation in temperature between an empty pot and the sample. The necessary tool in this case is a differential calorimeter (DSC). Both energy-compensating and heat-flux variants of the DSC are commercially available. One of the most often used techniques for determining the transformation in polymers and their composites is the DSC procedure. The change from glassy to rubbery is associated with Brownian movements of the major chains and the relaxation of the relevant dipoles.

The DSC method has shown considerable gains in Tg, which has resulted in the development of nano-sized montmorillonite in numerous polymers. This phenomenon is often linked to the fact that intercalation of polymers is confined inside the silicate galleries, hence halting the chain's segmental motions. DSC may also be used to analyze the kinetics of polymer crystallization under isothermal circumstances, shedding light on how nanoparticles influence the nucleation and crystal growth processes. Polypropylene crystallization at low levels of organically modified montmorillonite (OMMT) [6] was inferred from a decrease in the duration of half-crystallization ($t_{1/2}$).

DSC is a flexible technique for studying the time and temperature dependence of a sample's structural characteristics. Heat amount that is highly absorbed or radiated by the sample as temperature changes is calculated by the instrument according to the differential in temperature between the reference material and the sample. There are two common DSCs utilized nowadays: power-compensated DSCs and heat-flux DSCs. The later one involves placing an empty reference pan and the sample pan on the thermoelectric disk within a linear-rate-heating furnace. Using a calibration method, thermocouples monitor the temperature difference between the two pots and provide a heat flow signal. The later kind involves using two separate furnaces to keep the reference and sample pans at a constant temperature while doing the analysis.

Thermal power needed to maintain a similar temperature is evaluated and indicated as temperature or time function. Endothermic events are often represented by positive power signals in the negative signals in heat flux DSCs and power balanced DSCs. To prevent unwanted interactions with the air, instruments are purged with inert gases like nitrogen or helium. Scanning speeds typically range from 1 to 10 degrees Celsius per minute. Although high scan rates increase the sensitivity of measurements due to rapid heat exchange, low scan rates are favored for peak resolution and sample study near equilibrium.

The impacts of six varied liquid lipids on the size of particles, drug loads, and encapsulation efficacy of resveratrol Nanostructured Lipid Carriers (NLCs) were investigated by Gu, Sun, Wang, and Xia [7]. To learn how liquid lipids and trimyristin (solid lipid), including resveratrol and liquid lipids, interact, a thorough DSC investigation was conducted. Trimyristin and resveratrol's melting points dropped when liquid lipids were added to the mixture. The research assisted in the selection of the best liquid lipids that in turn resulted to the production of stable, drug-soluble crystalline particles. This flexible technique (operated in a dry state) can be used to characterize crystallite sizes in the 1–100 nm range and provides insight into a variety of structural features (such as the configuration of the crystal elements and macroscopic dataset such as mean shape and size) in a crystalline sample. While DSC allows for the monitoring and calculation of even the minutest thermal events in a sample, the source of these events cannot be revealed directly. X-ray diffraction analysis (XRD) allows for an accurate determination of temperature transitions and a thorough examination of crystal structure.

Thermogravimetry

In the scanning mode of thermogravimetry (TG) [8], sample's mass transformation is measured against temperature, and in the isothermal mode, the mass change is measured against time. Thermogravimetry (TG) is employed in describing the thermal breakdown and stability of a material under the range of situations and to assess the physicochemical kinetics process occurring within the sample; nonetheless, not all events amount to the transformations in sample mass (for instance, glass transition, melting or crystallization). It set up a predetermined heating procedure and then monitored the sample's weight change as it occurred as a result of melting, expanding, contracting, or reacting with the environment. This results in discrete TGA or peak DTG points. Steps in the TGA curve occur when a sample loses or gains mass as a result of a variety of factors. These days, most people rely on their compensation funds. Electronic microbalance, temperature programmer, furnace, and equipment for capturing concurrent outputs are the major components of a microbalance. (TG) curves are of an empirical character, since their cause is largely dependent on the sample's parameters and the heating method used. Then, differences across labs made comparisons impossible; however, this problem has been solved with the use of modern thermobalances.

In order to perform a thermogravimetric analysis (TGA) [9], a special piece of equipment called a thermogravimetric analyzer is used. The sample's mass is progressively evaluated using the thermogravimetric analyser while the temperature is distinct. Thermogravimetric evaluation depends on time, temperature and mass as its fundamental measurements, from which a wide variety of other data may be generated. An accurate balance built-in with sample pans is kept in fire with tunable control temperatures to serve as a thermogravimetric analyzer. In most cases, a thermal reaction is induced by gradually raising the temperature (or, in rare cases, by controlling the temperature to produce a constant mass loss). There are several possible conditions for the thermal reaction, such as inert gas, corrosive gases, vapors in liquids, carburizing gases, reducing/oxidizing gases, or a "self-generated atmosphere"; and many possible pressures, such as regulated pressure, high pressure, high vacuum, or continuous pressure.

Mass or the starting mass percentage on the temperature vs. y axis or x axis for time constitutes the thermogravimetric plot generated by a thermal process. TGA curve is a plot of this data that has been smoothed out. Differential thermal analysis and in-depth interpretations may both benefit from plotting TGA curve first derivative (DTA curve). Materials may be characterized using a TGA by examining their unique patterns of degradation. Polymeric materials, including fields, coatings, paints, plastic films, fibers, composites, thermosets, elastomers, and thermoplastics, benefit significantly from this kind of analysis.

As shown in **Fig. 4**, the furnace houses both the sample and reference balance beams. The driving coils' calibrated sensitivities allow for independent measurements of the sample and reference masses. The TG signal is the mass discrepancy. The differential mass measurement neutralizes the buoyant force, convection currents, and beam expansion. This allows for the very precise thermogravimetric measurement to be taken. The TG baseline drift may be easily adjusted electrically by measuring the sample and reference masses using separate driving coils. Each holder has a thermocouple, allowing for simultaneous DTA signal output. Thermogravimetric analysis (TG) may be used for analyzing thermal breakdown, oxidation, dehydration, heat resistance, and kinetics. When used in conjunction with other methods of measurement, a greater range of data may be extracted from a single instance. Particularly widespread is the TG/DTA simultaneous measuring equipment.
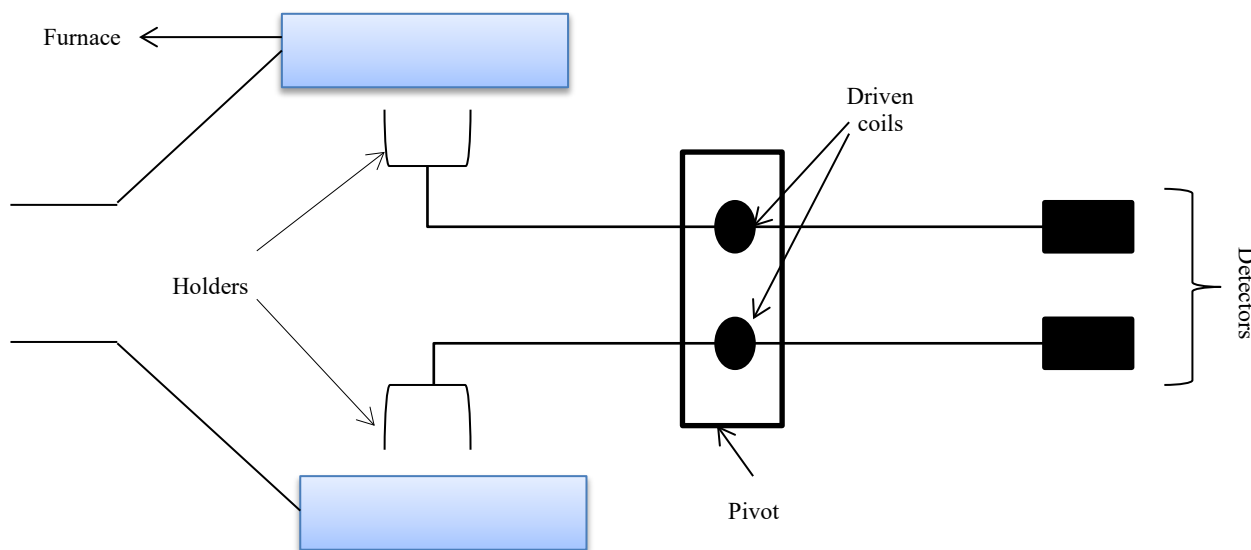

Fig 4. Horizontal TG/DTA representation

Thermomechanical Analysis
In thermomechanical analysis, strain and stress are the state variables. A bottom-mounted heater and coolant maintain a consistent interior temperature. The sample tube allows for manipulation of the surrounding atmosphere. The linearly variable differential transformer (LVDT) monitors the probe's location relative surface of the sample. TMA is typically employed in determining the glass transition temperature (Tg) by means of expansion mode or penetration. Dates of linear thermal expansion below and over the (TG) are also provided by the expansion mode. This approach is analogous to the (DSC) methodology. When a time- or temperature-dependent dye is applied to a sample, information on the sample's expansion or thermal contraction may be obtained using TMA.

TMA monitors the dimensions transformations of samples wherever it is cooled or heated in a controlled environment. Normal TMA curves show growth below the temperature of the glass transition, increment above the temperature of glass transition, growth again after the glass transition, and finally plastic deformation above the temperature of glass transition. Penetration, dilatometry, and Dynamic Load TMA (DLTMA) modes are all available for taking measurements.

Dilatometry

Expansion or contraction of a sample is what dilatometry is all about. This is why the applied force is minimal, but sufficient to keep the probe in place on the sample. The thermal expansion coefficient is the value obtained from the experiment. Figure 2 shows the output of a dilatometry test performed on a sample that was about 0.5 mm thick and was held between dual silica disks. To begin, it was heated to 90 degrees Celsius in the tool to erase any previous thermal memories. As it cooled, the ball-point probe was used to take readings between 30-310 °C at a heat degree of 20K in every minute suing very gentle 0.005 N of force.

Penetration

The majority of the data gathered during penetration measures is temperature-related. As the area of contact of probe with the sample transforms, according to the experiment, the thickness of the sample is usually not very important. Both the measurement force and the sample geometry influence the depth of penetration. The sample was put on a silica disk and its thickness was measured by placing a ball-point probe immediately on top of the sample. The temperatures ranged from 30 °C-300 °C, the rate of heating was about 20K per minute, and the measuring force was 0.1 N and 0.5 N. In this scenario, the sample was not warmed up first. Throughout the course of the penetration evaluations, probe will go deeper and deeper into the samples being tested. After the glass transition, the ordinate signal drops dramatically; after cold crystallization, it remains almost constant; and finally, upon melting, it drops once again.

DLTMA

When it comes to measuring physical characteristics, Dynamic Load TMA (DLTMA) is one of the most sensitive methods available. In contrast to DSC, mechanical behavior of samples is characterized. In DLTMA4, the sample is subjected to high and low forces at predetermined frequencies. This stimulates sample elasticity evaluation (i.e., Young's modulus), including expansivity and weak transitions. The amplitude has a negative correlation with the sample's rigidity.

Evolved gas analysis and evolved gas detection

Either evolved gas analysis (EGA) or evolved gas detection (EGD) refers to a kind of thermal analysis in which gases produced during a chemical reaction are identified and analyzed in relation to their temperature of generation. Before EGA, there was the EGD that just looked for the existence of evolved gases. For thermal analysis, EGA and EGD are defined as follows by the ICTA nomenclature committee: Enhanced gas detection (EGD). When a volatile product is created during thermal analysis, it may be determined with the use of a process called evolved gas detection.

Analysis of the gases that have been "evolved" during a thermal decomposition is called evolved gas analysis (EGA). Most often, these methods are employed in tandem with others, such as thermal analysis or several techniques such as thermogravimetric analysis (TG) and electron spin resonance (EGD) or nuclear magnetic resonance spectroscopy (MS). Both continuous and sporadic samplings of evolved gas pools are possible. Fourier transform infrared (FTIR) spectroscopy and Mass spectroscopy (MS) are the two most used EGA techniques, and they both involve collecting and analyzing purge gas over a certain period of time or temperature.

Microscopy Analysis

Both qualitative and quantitative determinations may be made with the use of microscopy analysis, which is accomplished with either an optical microscope or an electron microscope using either transmitted or reflected light. As a result, less attention is being paid to light transmission methods. For many years, standard photography cameras using emulsion-based films have been used to detect tiny pictures. Rapid advances in photography technology over the last twenty years have allowed electronic imaging to supplant traditional photomicrography. At the beginning, there were video rate cams. These cameras employ vacuum-tube light detectors to provide an ana-log output signal that is compatible with a wide variety of video formats. Video recorders are common examples of electrical signal storage devices, but a frame grabber is required before the signal can be transferred to a computer for image processing. A detector in solid state whole output is considered a digitalized signal has largely supplanted video rate cameras in recent years. The detectors that are considered a vacuum tube (photomultipliers) are employed exclusively in microscopy methods of laser-scanning. Scientific-grade digital cameras are expected to perform at a high level, but the properties of the device significantly impact their costs. The detectors in solid state are 2D photodiodes arrays, typically produced using charge-coupled device (CCD) and complementary metal-oxide semiconductor (CMOS) [10] technology.

There are a handful of essential aspects to think about depending on the interest application. The illuminated area of each photodiode's cross section is a key factor in determining resolution (pixel). Better resolution is achieved by using smaller pixels and thereby increasing the pixel density in the array. Many methods have been tried to increase sensitivity (intensified cameras, back-illuminated cameras, etc.) by adjusting the duration of exposure, area of pixel, and the CCD quantum efficiency. Readout noise is the primary cause of digital camera noise and varies with CCD production, readout speed, and the device's circuitry. The thermal noise of the camera may be mitigated by placing it in a cold environment. The dynamic range of a device is determined by the size of its pixels and the way in which it processes light, as well as the degree of noise it produces. The color values or gray number, which could be evaluated and digitally represented, is affected by dynamic range; normally, 256 levels (8-bit) or 4096 levels (12-bit) digital representation is chosen.

Optical Microscopy

In the fields of science, engineering, and medicine, the optical microscope is a critical instrument for nanostructure morphological characterization. Polarized light is the most typical contrast source used by the geologist, who polishes his mineralogical samples comprised with the thickness of less than 50 m and then mounts them on glass slides. This technique allows the geologist to learn about the spatial orientations and optical features of crystalline phases, which are available in samples. Thin slices of metallurgical samples were initially made for metallographic analysis in 1964 by Henry Sorby, utilizing a procedure developed previously for mineralogical specimens. Nonetheless, all the metallurgical samples should be analysed in reflection because the presence of charge transfer makes metals opaque to visible light.

As the system is primarily composed of the lighting system and lenses, the picture contrast is a consequence of the light mirrored light in different microstructure parts. Reflections and transmission imaging both work well for plastics and polymers, but the amorphous, glassy phases provide very little detail. Polymer matrix composites and filled plastics may be analyzed by reflecting light off of them, seeing their crystalline polymer phases, etc. Unfortunately, the phases that may be found with this method, as well as the final composition and structural analysis, are often unknown without using a different method like as x-ray diffraction. The greatest rise is also capped at about 2000 times, which is a significant restriction.

Electron Microscopy

High-resolution images of both in-organic and biological samples could be retrieved using microscopy (EM). Biomedical scientists use it to learn more about the internal organization of cells, organelles, and macromolecular complexes. The electrons used as the illumination radiation source have significantly shorter wavelengths, adding to the high resolutions in EM images. Different complementary approaches (such as immune-labeling, negative staining, and thin sectioning) are utilized tandem with EM to provide potential solutions to particular research issues. The structural foundation of cell function and cell illness may be deduced from EM images. Transmission electron microscopy is similar to the transmissions optical microscope – but normally "upside down," in the aspect that the electron beam source is below the microscope bottom rather than the top. This allows for a resolution that is orders of magnitude greater than that present for morphological researches when partial by visible light wavelength. Nevertheless, electronic microscopy is seldom used on thick sections, unless silhouettes are needed to understand the geometry of the material. As a result, ultrathin sections ($< 1\mu$) must be prepared for e-transmission inspection.

The scanning electron microscope (SEM) and the transmission electron microscope (TEM) are the two most compound varieties. The transmission electron microscope creates a projected picture by passing electrons through a thin material (tissue segment, molecule, etc.). The TEM is the same as the compound (common) light microscope in many respects. The negative staining approach is used in conjunction with TEM to see the organization of molecules inside viruses and cytoskeletal filaments, and TEM may be used to view the structure of proteins within cell membranes as well as the interior of cells in thin slices (by freeze-fracture).

Traditional scanning electron microscopy (SEM) uses a specimen's surface to emit secondary electrons. A SEM is the same as SLM (stereo light microscope) in the EM realm due to its high magnification and depth of field. It allows for more in-depth imaging of cell and organism surfaces than is feasible with TEM. Particle counting, size analysis, and process monitoring are all possible applications. The image is formed by sweeping a stream of electrons over the material surface, thus the name "scanning electron microscope." Particles are produced at every raster locality due to the interactions of main electron beams with the atoms at the surface level (such as low-energy secondary electrons, electrons with high energy reverse scatter, photons and X-rays). Counting them with different detectors gives us an idea of how bright each spot on a cathode ray tube really is. When viewed on a CRT, the final image seems enlarged because the bitmap size at the sample is increasingly smaller compared to the visual screen. With the right attachments (X-ray detectors, and backscatter), SEMs could be employed in the investigation of not just the surface dispersion of immune-labels, but also the topography and molecular composition of specimens.

Mineralogical Analysis

The research of minerals and other mineralized artifacts, including their chemistry, crystalline structure, and structural (including optical) qualities, is known as mineralogy, a subfield of geology. Mineralogy is the study of minerals and the methods by which they are formed, categorized, distributed geographically, and put to practical use.

X-ray spectroscopy (DRX)

The study of matter's atomic structure may be aided by X-ray diffraction (see **Fig. 5**). X-ray diffraction was originally used only for crystallographic purposes. However, as time went on, new applications were discovered, and now the technique is used for everything from stress quantification and chemical analysis to the phase equilibria surface and measurement of particle sizes to evaluate the single crystal orientations or ensemble of orientations within the polycrystalline aggregate. For a long time, x-ray spectroscopy was the go-to technique for identifying heavier elements like niobium, tantalum, and rare earths, which were notoriously hard to identify using other strategies. X-ray diffraction, neutron diffraction, and electron diffraction are only few of the modern methods of diffraction. Electron diffraction is utilized to examine gases and vapors, as well as surface coatings, whereas X-ray diffraction is the best for solids and liquidcrystals investigations. Electromagnetic waves with a wave length between 0.1 and 100, such as X-rays, light, and ultraviolet radiation, exhibit tremendous penetrative strength.

Journal of Computational Intelligence in Materials Science 2(2024)

X-ray diffraction is often used to analyze a sample's crystalline structure or chemical makeup. It may be used to figure out how the atoms in a sample of a bigger crystal, such a macromolecule or an inorganic substance, are arranged. Sample composition, crystalline nature, and phase purity may be established if the crystal size is small enough. X-ray beams are used in this method. In order to have diffraction angle affected by atoms spacing in molecules, X-ray beams are used instead of much greater wavelengths that would not be impacted by atoms spacing. After entering the sample, the x-rays "bounce" off the atomic structure, altering the beam's route at a particular angle, theta, relative to the original. The diffraction angle is at this point. Although some of the diffracted beams cancel each other out, positive interference may occur if the beams are of comparable wavelengths.
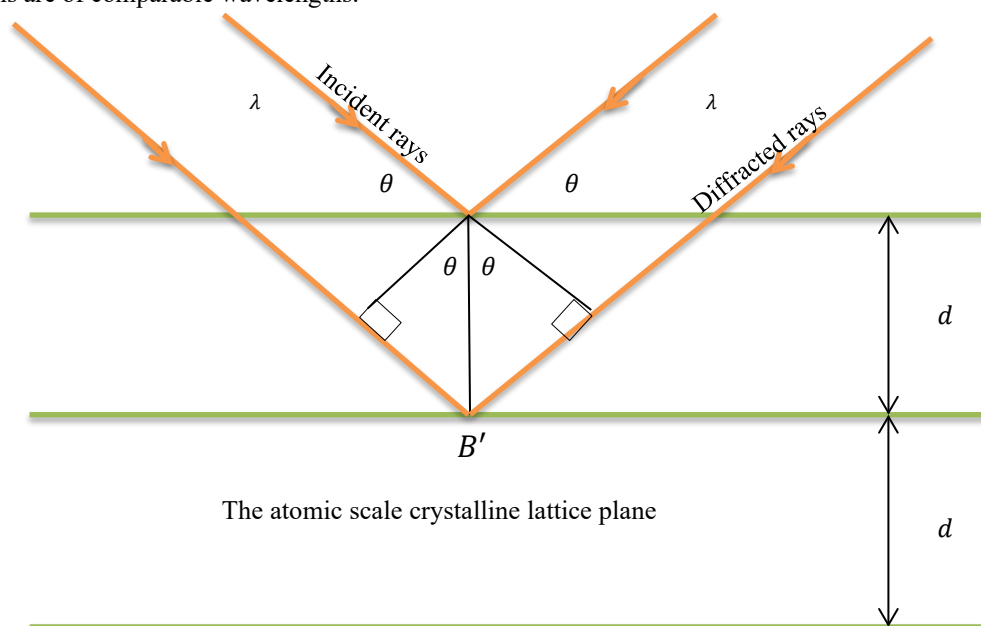

Fig 5. Representation of X-ray diffraction

When x-ray beams of similar wavelength integrate to create a novel beam with higher amplitude this phenomenon is known as constructive interference. With this diffraction angle, a higher signal corresponds to larger wave amplitude. Bragg's rule states that the distance between atomic planes may be calculated from the angle of diffraction using the following formula: $sin\theta = n\lambda/2d$, where λ (lambda) represents the wavelength added, θ (theta) represents the diffraction angle, and d represents the distance. Composition or crystalline structure may then be deduced from the gap between the atomic plates. Whenever the route separation of ABC and the A'B'C' is a wavelength (λ) integer number, the X-rays being diffracted will indicate a constructive interference.

Spectroscopic techniques reliably identify about 70 elements. Gases and a select few nonmetals are notoriously hard to detect, in large part because their sensitive lines are located in the elusive short-ultraviolet region of the spectrum. There are a variety of ways to introduce a qualitative sample to the electrode. The sample may be put in the cup electrode without needing to be weighed if it is a powder. In the powder strategy, the crystalline to be investigative is crushed to fine powder and the integrated in a monochromatic X-rays beam. Tiny metalfilings may also be put in a cup and the solvent is then vaporized. To confirm the existence of an element in an analytical sample, a spectroscopist often depends on the detection of three or four spectral lines. Each powder particle is a small assemblage of small crystals, leaned at random in reference to incoming beams. Typically, values around 0.05 to 0.10 while enough. Elements with yet-to-be-determined spectra are also used as a benchmark against the unknown sample. Spectralline wave length data are unnecessary for this method.

The krypton-84 red line, in which the wavelength of 6057.8021 has been assigned, was chosen as the standard for such measurements in 1960. The wavelength plates for iron lines that can act as secondary standards are based on this standard; however, it is not particularly useful for qualitative analysis. Practically, among the varied strategies for materials characterization, DRX is the most acknowledged for crystalline materials perseverance available in ceramics. Most solids (crystals) have their atoms arranged in crystalline planes, which allows the x-ray wavelength to penetrate all the way through.

Chemical Analysis
X-ray fluorescence spectroscopy
X-ray fluorescence (FRX) [11] is a nondestructive technique employed to determine the chemical composition of materials. It operates on the principle that absorption of radiation by a substance triggers the atomic excitation that underlies the emission of a secondary radiation known as x-ray fluorescence. The produced rays have a feature wavelength, which illustrated for every chemical component that integrates the irradiation materials (chemical qualitative evaluation), which can be determined by the bunch may dislodge one electron from the innermost layer of the atom,

prompting another electron to jump from the outermost layer to take its place. This process releases energy as x-ray fluorescence at wavelengths unique to each element.

The semiconductor industry now routinely employs X-ray fluorescence (XRF), a state-of-art strategy of materials investigation. X-rays or gamma rays are used to light the sample being analyzed, causing the electrons in the sample's core to be stimulated to higher energy levels. As these electrons return to their ground states through radiation, they emit secondary X-rays or fluorescent, which are unique to the levels of energy of their atomic species and could be employed as spectroscopic fingerprints. The size of the spots on the product wafers might be as little as 100 nm.

The X-ray fluorescence (XRF) signal could be employed as a direct evaluation of metals thickness and alloys since the signal strength for every atomic species is proportional to the atom number available. The capacity to effectively quantify the thickness of the thin-film and concentration of alloys without the need for numerical modeling approaches is XRF's fundamental strength as a measuring tool. Metals are able to be measured despite their thickness, since they are transparent at high frequencies. On the other end of the spectrum, XRF may theoretically be employed to evaluate thin layers of approximately 10 nm. The key challenge in evaluating such thin layers originates from the long duration of acquisition needed by XRF detectors to collect statistically relevant quantity of fluorescence counts, including the care require in de-convolving spectrum overlaps from underlayers, and substrates, among others.

XRF does have certain restrictions. The XRF signal should be calibrated over reference thicknesses for precise quantitative findings. Weak fluorescence from these species makes XRF less helpful for measuring components with a lower atomic number, generally Z11. Due to the films' X-ray transparency, XRF cannot be employed for depth profiling; it could evaluate the stacked film thickness, but cannot normally determine the film at the top. Lastly, it is important to be selective about the spectral lines to sample, since stronger spectral signals from underlayers or substrates could with the thin film signal being detected hence resulting to getting an inaccurate reading on their intensity.

Analytical atomic absorption spectroscope
The proportion of metal atoms or ions in a sample may be calculated with the use of AAS, an analytical method. Almost three-quarters (75% to be exact) of the elements on Earth are metals. Metals may be both desirable and undesirable characteristics of a certain material (poisons). This article will discuss the numerous uses for which accurate metal content measurements are essential. Let's just state that it has applications in quality assurance, toxicology, and environmental testing for the time being.

The following is a summary of the fundamental ideas of AAS. To begin, light of a certain wavelength may be absorbed by any atom or ion. For example, only the copper (Cu) atoms or ions in a sample containing both Cu and Ni will absorb light at the characteristic wavelength of Cu. Absorption at this wavelength is proportional to the density of the ions or atoms doing the absorbing. There are many different energy states that an atom's electrons may occupy. Atoms may absorb energy (photons) and transition from a ground state to an excited state when they are exposed to light of their own specific wavelength. The electrons' ability to absorb radiant energy is proportional to a transition that takes place throughout this process. As each element has its own distinct electronic structure, the amount of radiation absorbed is also a measurable, one-of-a-kind attribute of each element. These fundamental concepts are implemented in a quantifiable manner using an AAS. The atomization model, light source, detection model, and the monochromator are the four major parts of the AAS (see **Fig. 6**).
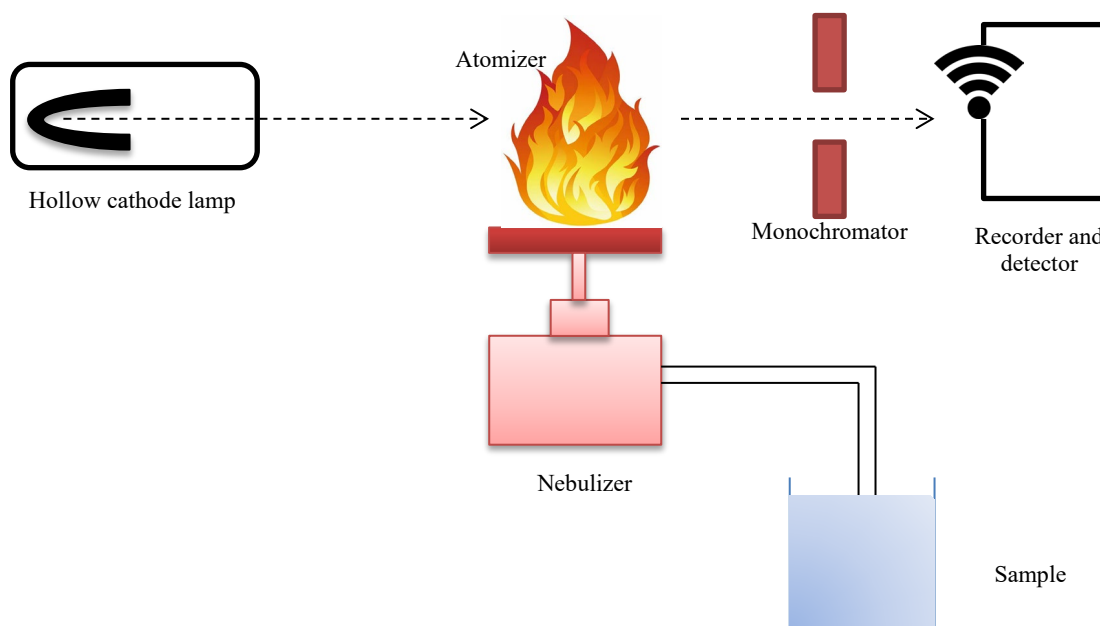

Fig 6. A sample atomic absorption spectrometer's schematic design

Atomicabsorption spectroscopy is based on the principle that atoms in their ground state absorb energy when they are in a gaseous state. Each element has its own unique absorption spectrum, and this particular wavelength is absorbed by another element. The Atomic Absorption Spectrometer (AAS) [12] employed this concept for quantitative evaluation of metals, particle non-metals, and semi-metals in a broad variety of materials, including biological samples, pharmaceutical samples, metallurgical samples, weather samples, and geological samples. While alternative photosensitive devices including the, vacuum phototube, barrier layer photocell, and photographic emulsions have been utilized, the photomultiplier tube is by far the most compound detector for AAS. The procedure consists of determining the availability and quantity of metals in chemical solutions, and the sample is typically a liquid; however advances with tantalum boats and carbon rods have rendered the usage of solid samples feasible.

IV.   CONCLUSION

In materials science, "characterization" is shorthand for the extensive investigation into and quantitative assessment of a material's structure and qualities. It is a crucial step in materials research, without which we would not be able to learn anything about the science behind the materials we use in engineering. Some definitions restrict the word to methods that examine the microscopic structure and qualities of materials, whilst other definitions extend the phrase to apply to any materials analysis procedure, including macroscopic methods like mechanical testing, thermal analysis, and density computation. Materials characterisation allows us to see structures from the atomic and molecular level all the way up to the centimeter level, as in the imaging of metal's coarse grain structures. In this article, we looked at the most popular analytic methods used to characterize materials. Although space constraints prevented a more in-depth examination of the topic, important details and potential uses of each approach were retained.

Data Availability

No data was used to support this study.

Conflicts of Interests

The author(s) declare(s) that they have no conflicts of interest.

Competing Interests

There are no competing interests.

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