Microscopic Characterization of Dental Materials and its Application in Orthodontics - A Review

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Abstract:- The characterization techniques used for dental material aids in a better understanding of the composition, density, physical properties, molecular weight, and surface properties of an object. It also helps to analyze and optimize the materials. The structure and properties can be understood by probing and measuring. Many techniques are available and different methods of characterization are being followed. The techniques and also instrumentation are in constant evolution. The objective of this article is to review the usage of various microscopic characterization techniques employed in orthodontics these days, through a careful and updated literature review.

Keywords:- *Micro Characterization, Microscopy, Spectrometry, Spectroscopy, Orthodontics.*

I. INTRODUCTION

Characterization in materials science refers to the broad and general method by which a material's structure and properties are probed and measured. It helps to increase the different degree of understanding of materials showing different behavior and properties. The characterization technique can be broadly classified into macroscopic and microscopic characterization as explained in Figure I. This review article focuses on the various microscopic, spectrometric and spectroscopic techniques used in orthodontics.

The macroscopic characterization of the material includes density calculation, thermal analysis ,and mechanical testing.[1] The microscopic characterization of the material includes various spectrometry, spectroscopes and microscopes. The microscopic methods for the measurement and characterization of materials used in accordance to the need of study done in dentistry and orthodontics are explained in figure II.

II. QUALITATIVE METHODS

A. Microscope

The technique uses a probe that maps the surface and sub-surface structure of a material. The various kinds of microscopes with their clinical implication in odontology are as follows:

➢ Optical Microscope

The optical magnifier usually uses visible light with the limited resolving power of visible light that keeps the maximum magnification power of optical microscopes limited to around 1000x.

- *Types:- There are 2 Basic Kinds of Optical Microscopes*

✓ *Simple Microscope*

A simple microscope uses the optical power of a single lens or cluster of lenses for magnification. It is not commonly used for research purposes.

✓ *Compound Microscope*

A light microscope uses a system of lenses (one set magnifying the image created by the other) to attain a higher magnification of the object.

➢ *Other Microscopic Variants are*

- *Scanning Electron Microscope (SEM)*

The SEM uses electrons instead of light to form an image. It is preferred over other microscopes because it allows the large depth of field and provides higher resolution. It uses electromagnets rather than lenses so as to provide more control over the degree of magnification. It provides a magnified visual assessment of tooth surfaces.

- *Applications*

✓ It generates high-resolution pictures of the shapes of objects and points out spatial variations in chemical compositions.
✓ It helps to determine phase-supported qualitative analysis and crystalline structure. The measurement of very small objects up to 50 nm in size is also accomplished using SEM.

- *Limitations*

✓ Samples should be solid and they should match the magnifier chamber.
✓ Maximum size in horizontal dimensions is within the order of 10^{-10} cm, and vertical dimensions are typically way more restricted and barely exceed forty metric linear units.

- *Transmission Electron Microscope(TEM)*

A transmission electron microscope (TEM) uses electron particles to visualize specimens and create highly magnified images.

A SEM creates an image by detecting reflected or knocked-off electrons, whereas a TEM uses transmitted electrons (electrons passing through the sample). Therefore, TEM provides information about the sample's inner structure, such as crystal structure, morphology, and stress state, whereas SEM provides information about its surface and composition.

➢ *Confocal Microscope*

By employing a spatial pinhole, the technique increases the optical resolution and contrast of a micrograph. It produces a point source of light and rejects out-of-focus light, which provides the ability to image deep into tissues with high resolution and optical sectioning for 3D reconstructions of imaged samples.

- *Limitations*

It is associated with a minimal number of excitation wavelengths and is expensive to produce in the ultraviolet region.

- *Atomic Force Microscopy (AFM)*

It is appropriate for characterizing nanoparticles and nanomaterials. It enables the imaging of surfaces including polymers, ceramics, composites, glass and biological samples. The range of particle size is 1 -8 µm.

B. Spectroscopy

The interaction between light and matter is the basis of this technique. Excitation and de-excitation occur when the light is absorbed by the matter resulting in the production of a spectrum.

➢ *Ultraviolet Visible Spectroscopy*

The production of distinct spectra as a result of the absorption of ultraviolet light or visible light by chemical compounds is the basic principle of UV-Visible Spectroscopy. It takes too much time to prepare to use the technique.

➢ *Fourier Transform Infrared Spectroscopy (FTIR)*

It enables qualitative and quantitative analysis for inorganic and organic samples. It produces an infrared absorption spectrum and chemical bonds can be identified.

The limitations are that only small items such as rings can be tested.

➢ *Raman Spectroscopy*

It is a non-destructive chemical analysis technique that provides detailed information about chemical structure, phase and polymorphy, crystallinity, and molecular interactions. It is based on the interaction of light with the chemical bonds within a material.

The limitations are that high-powered lasers and amplification sources are needed to get sensitive results and it is much more expensive.

➢ *X -Ray Diffraction (XRD):*

X-ray powder diffraction (XRD) is a technique that provides information on unit cell dimensions and is primarily used for phase identification of crystalline material. The analyzed material is homogenized, finely ground and the average bulk composition is decided.

➢ *Energy Dispersive X-Ray Spectroscopy*

It is a technique where an electron hits the sample inner shell electron and gets excited leading to ejection and the formation of an electron hole in the electronic structure of the element takes place.

C. Mass Spectrometry

It is a type of analytical tool used to measure the **mass-to-charge ratio** (m/z) of one or more molecules present in an object and calculate ate molecular weight of that object. After calculating molecular weight it quantifies the structural and molecular properties of the molecule.

➢ *Secondary-Ion Mass Spectrometry (SIMS)*

It analyzes the composition of solid surfaces and thin films by sputtering the surface of the specimen with a focused primary ion beam and collecting and analyzing ejected secondary ions.

❖ *Qualitative Methods*

➢ *Surface Profilometers*

It makes surface contact using profilometers with a material substrate. It notices various fluctuations and surface deformations. It can be used to determine roughness, waviness, and measurements.

➢ *Microradiography*

Unidirectional X-ray projection and absorption in thin slices of mineralized tissues is used and quantifies various mineralized areas in the tooth.

The limitation is that it is a transmission technique that implies an averaging of the calcium content over the thickness of the sample (i.e., over 100 µm) which seriously limits the resolution of the technique. The technique is not able to calculate whole mineral content in particular trabeculae.

➢ *Quantitative Light-Induced Fluorescence (QLF)*

It was used by van der Veen and de Josselin de Jong. According to him, fluorescence radiance in carious teeth decreases. The fluorescence loss in the carious lesion can be quantified.

It is a non-invasive technique that harnesses fluorescent light to detect the demineralization necessary for pathogenic microorganisms in plaque. It helps to diagnose white spot lesions by quantifying mineral content loss during demineralization. A modification of QLF is QLF- D stands for digitized QLF that provides the increased intensity of red-fluorescence.

It helps to quantify demineralization and remineralization in both therapeutic and experimental conditions. It acts as a supportive tool for monitoring the effects of dental clinic preventive care on patients. It also quantifies dentinal lesions and premature enamel lesions.

➢ *Optical Coherent Tomography (OCT)*

It is a high-resolution optical technique that allows the minimally invasive visualization of near-surface alterations in complex tissues. It provides real-time structural images of the enamel and of the soft tissue parts and is used to detect the morphological changes of oral tissues in vivo.

The applications of microscopic characterization of dental materials have been discussed in Table I and The latest studies using various methods of microscopic characterization in Orthodontics has been discussed in Table II.

III. CONCLUSION

The characterization techniques used for dental material aids in a better understanding of the composition, density, physical properties, molecular weight and surface properties of an object. The macroscopic characterization of the material includes density calculation, thermal analysis, and mechanical testing while the surface structural topography on a microscopic level utilizes SEM, TEM, X-ray diffraction , Confocal laser scanning microscope, and Atomic force microscopy that provides qualitative details of an object. This aids in better understanding and experimental exploration. Each given technique may have a limited range of applications. It is thus beneficial to employ more than one technique and combine results from several methods to get as much information as possible.

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Table 1 Applications of Microscopic Characterization of Dental Materials

S.no	Methods Of Dental Material Characterization	Applications In Dentistry
1.	Scanning electron microscope	1.Evaluation of surface topography and structural changes of prepared enamel and dentin surfaces after laser irradiation or by using different methods.
		2.Evaluation of macro and micromorphological features of the lesions (considering the enamel, dentin, enamel prisms, dentinal tubules, and pulp).
		3. Analysis of the external microstructure surface of a tooth by comparing the growth of dental plaque or microbiological contamination following the use of different cleaning method .
		4.Evaluation of in- vitro bacterial adhesion of Streptococcus mutans and Lactobacillus on dental surfaces.
		5. Visualization of biofilm and microorganisms associated with teeth with apical periodontitis.
		6. Evaluation and comparison of cleaning efficiency on root canal surfaces prepared with different rotary instruments.
		7. Estimation of the surface quality of polished dental composite after restoration.
		8.Examination of structural variations of the mini-implant surface of orthodontic implants.
		9.Examination of enamel surface before and after debonding of orthodontic brackets.
2.	Transmission electronic microscope	1.Examination of the dental hard tissue constituents at the ultrastructural level.
		2. Estimation of surface topography and adhesive interface .
		3.Presence of microcracks in the tissue surface and interfacial gaps.
		4. Mechanical wear of dental materials and tissues can be determined.
		5.Determination of adhesive and cohesive failures
3.	Confocal laser scanning microscope	1.Determination of surface roughness, Implant surface topography.
		2.Analysis of Bacterial Biofilm can be done.
		3. Visualization of the spatial structure of polymers, hybrid layer, and restoration-dentin interface or to assess the properties of new generation composite material
		4. Observation to visualize the spatial structure of polymers the infiltration zone of adhesive within the etched enamel
4.	Atomic force microscopy	1.Evaluation of surface topography, microhardness, and depth of remineralization can be done.
		2.Evaluation of Young's modulus of elasticity on the dentin–resin interface after application of the remineralization-inducing agent.
5.	Ultraviolet spectroscopy	1.Used to verify the purity and concentration of RNA and DNA.
		2.Quantification of various drugs like benzocaine, a local anesthetic, and chlortetracycline, an antibiotic, can be done.
		3. Used for bacterial culturing .
6.	Fourier transform infrared spectroscopy (FTIR)	1.Analyze chemical structural properties of natural materials.
		2.Directly extract molecular information regarding collagen's and hydroxyapatite's structural changes as dentin transitions from the transparent zone (TZ) into the normal zone (NZ).
7.	Raman spectroscopy	1.Determine the chemical composition of molecular species 2.Helps in characterizing hydroxyapatite crystals ($Ca_{10}(PO_4)_6(OH)_2$). Hydroxyapatite crystals undergo dissolution in acidic pH, and phosphate ions, $(PO_4)^{3-}$ and hydroxyl ions (OH−) react with the hydrogen ions (H^+) in the tooth–biofilm interface and form $H_2PO_4^{2-}$ 3. Used for Diagnosing Developmental Disorders of Enamel and Dentin 4. Determine crystalline phosphate-based minerals in dentin first human gingival crevicular fluid (GCF) biochemical characterization during the early phase of orthodontic tooth movement.
8.	X-ray diffraction	1.Used for the identification of enamel and dentin. crystalline materials.
		2.Identification of the crystal mineral phases in the exposed enamel
9.	Energy dispersive X-ray spectroscopy	1.Examination of surface and determination of chemical structure of the mini-implants
		2. measurement of nanoparticles can also be done.
10.	Secondary-ion mass spectrometry (SIMS)	1.Identification of Calcium and Hydroxyapatite crystals in cementum.
		2.Analyze the composition of solid surfaces like enamel , dentin.

11.	Quantitative light-induced fluorescence	1.Adhesive interface integrity can be determined.
		2.Presence and characteristic of dentinal smear layer can be determined.
		3.Depth of penetration of various materials in dentinal tubules can be determined.
		4.Invasiveness of etchants can be determined
12.	Optical coherent tomography	1.Used to detect qualitative and quantitative morphological changes of dental hard and soft tissues in vivo.
		2.Used for early diagnosis of dental diseases, including caries, periodontal disease, and oral cancer, because of the excellent spatial resolution.

Table 2 Latest Studies Using Various Methods of Microscopic Characterization in Orthodontics

S. No	**Methods Of Dental Material Characterization**	**Applications In Orthodontics**
1.	Scanning electron microscope	1.To assess the Copper addition influence on the corrosion resistance and biocompatibility, the surfaces of the materials NiTiCu (Ormco) and NiTi were evaluated by Scanning Electron Microscope (SEM) before and after the electrochemical tests. A complementary analysis was also done by confocal laser scan microscope (Zeiss LSM 880), to characterize the surface and corrosion morphology of the materials, before and after the electrochemical tests.[2]
		2.In another study, the effectiveness of diode laser-assisted bleaching on the shear bond strength (SBS) of different adhesive systems to enamel was investigated and the adhesive enamel interface was examined under scanning electron microscope [3]
		3.One study was performed to affirm surface characterization, as well as to compare the effect of coating of stainless steel (SS) orthodontic brackets and wires by **nanoparticles of Chitosan** (CTS) or Zinc oxide (ZnO) during friction. The Coating was analysed by SEM.[4]
2.	Transmission electronic microscope	1. In one study, Using TEM, crystalline evaluation of calcium hydroxyapatite nanoparticle size and shape was performed to evaluate polymerization and shear bond strength after addition of calcium hydroxyapatite nanoparticle for Heliosit adhesive.[5]
3.	Confocal laser scanning microscope	1.One study showed that confocal microscopy were used to evaluate autophagy (a conserved catabolic pathway) activation in tissues of loaded and control molars at time points after force application during orthodontic tooth movement[6]
		2.In another study, a three-dimensional laser confocal microscope (VK-X250K, Keyence, Japan) was used to determine the surface roughness of the archwires coated with carbon film. Using a Raman spectroscope (LabRAM HR Evolution, Horiba, Japan) the bonding structure configurations of carbon films can be obtained at a laser wave length of 532 nm and a spectrum region between 800 and 3,500 cm^{-1} and surface morphology and elemental composition of the uncoated and carbon film coated archwires were examined by using a scanning electron microscope.[7]
4.	Atomic force microscopy	AFM is used to evaluate the surface topography and roughness of orthodontic arch wire materials, including low-friction titanium molybdenum alloy (TMA), conventional TMA, and stainless-steel arch wires.[8]
5.	Ultraviolet spectroscopy	Polarization studies, AC impedance spectra, UV-visible absorption and fluorescence spectra, SEM and AFM were used to evaluate corrosion resistance of Ni-Ti alloy, 22 carat gold, SS 18/8 alloy, SS316L alloy, and thermoactive alloy in artificial saliva (AS) in the absence and presence of tea. [9]
6.	Fourier transform infrared spectroscopy (FTIR)	ATR-FTIR spectroscopy was utilized to characterize, at the molecular level, the chemical and color modifications in the surfaces of the appliances to evaluate the changes occurring in Invisalign® aligners, exposed in vitro to coffee, tea, Coca Cola® and UV radiation for 24 and 48 h.[10]
7.	Raman spectroscopy	Perillo L examined the biochemical and structural changes occurring in the periodontal ligament (PDL) during orthodontic-force application using micro-Raman spectroscopy.[11]
8.	X-ray diffraction	X-ray diffraction analysis was used to determine its phase structure and the element composition and valence state and the anti-corrosion behavior of NiTi and CuNiTi arch wires was simultaneously studied in artificial saliva under loading stress to simulate clinical conditions.[12]

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9.	Energy dispersive X-ray spectroscopy	In a study Quantitative analysis of enamel on debonded orthodontic brackets done by Nathan J.et,al the debonded brackets were examined by backscattered scanning electron microscopy with energy dispersive x-ray spectroscopy to determine the presence and area of enamel on the base pad.[13]
10.	Secondary-ion mass spectrometry (SIMS)	In a study, Formation of Nitrogen-Doped Titanium Dioxide Surface Layer on NiTi Shape Memory Alloy. Done by Tarnowski M,et,al The mesoscale linear distribution of the elements—oxygen, nitrogen, titanium, and nickel—in the surface layers was measured by secondary ion mass spectrometry (SIMS) analysis.[14]
11.	Quantitative light-induced fluorescence	**Banerjee** et.al, assess white spots with Quantitative Light-Induced Fluorescence in patients undergoing fixed orthodontics. He has done in vivo monitoring of mineral changes in incipient enamel lesions.
12.	Optical coherent tomography	Yusuke Koshimitsu conducted one study using Transverse Micro Radiography for the analysis of the effect of experimental Calcium-Containing Primer system on demineralized enamel and he observed the irregular surface of the adhesive which remained after bracket debonding.

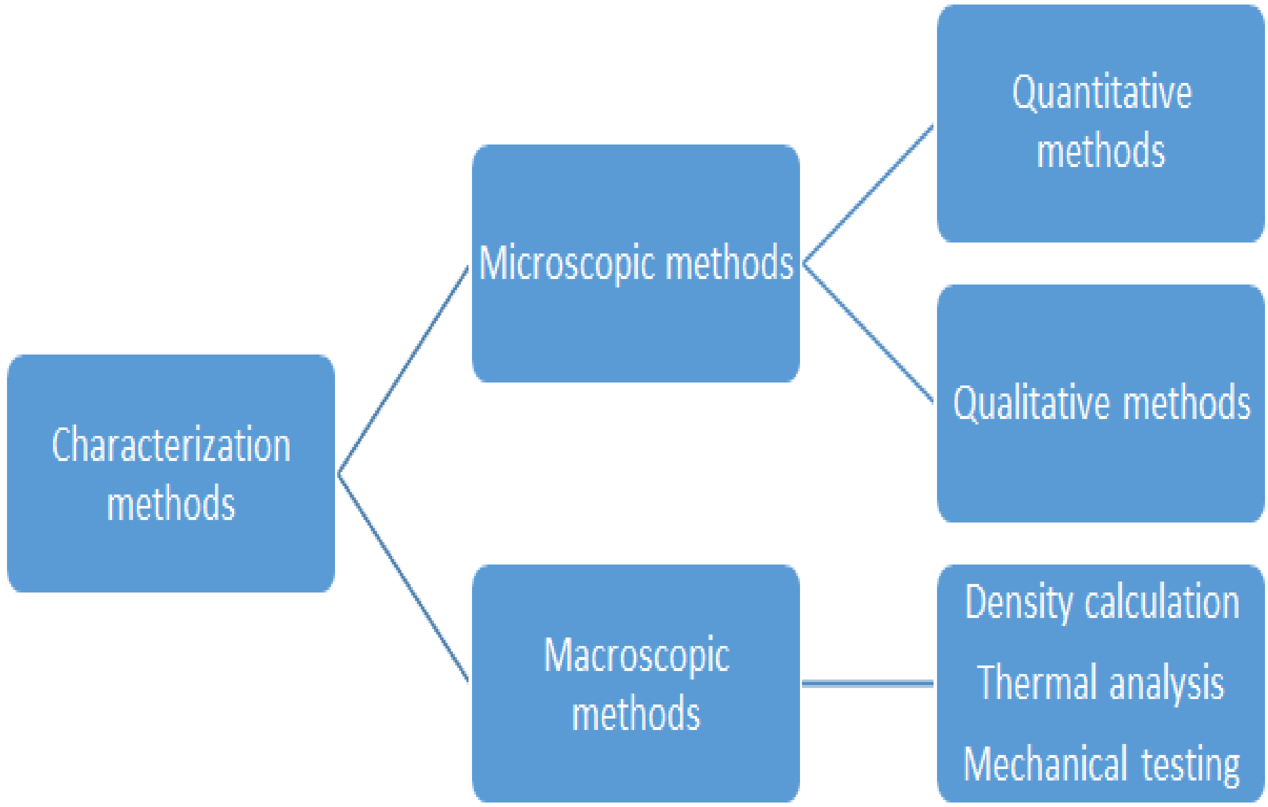

Fig 1 Describes Types Characterization Methods

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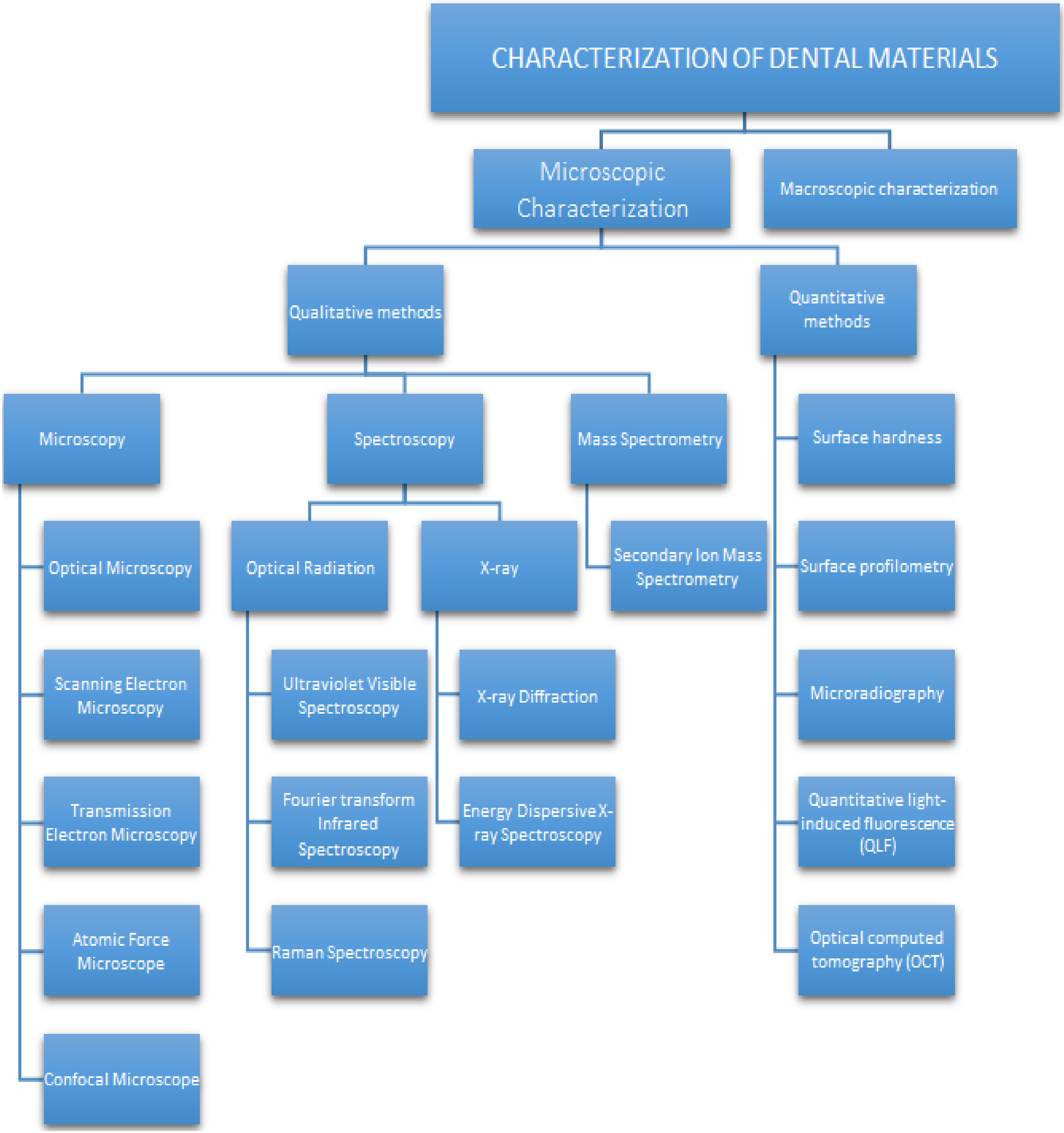

Fig 2 Shows Types of Microscopic Characterization Methods