Introduction

A Scanning Electron Microscope (SEM) is a powerful magnification tool that utilizes focused beams of electrons to obtain information. The high resolution, three dimensional images produced by SEMs provide topographical, morphological and compositional information makes them invaluable in a variety of science and industry applications

The scanning electron microscope (SEM) uses a focused beam of high energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron sample interactions reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-D image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques (magnification ranging from 20X to approximately 30,000X, spatial resolution of 50 to 100 nm).

The SEM is also capable of performing analyses of selected point locations on the Sample ; This approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions (using **EDS**), crystalline structure, and crystal orientations (using **EBSD**). The design and function of the **SEM** is very similar to the **EPMA**and considerable overlap in capabilities exists between the two instruments.

History

•The first electromagnetic lens was developed in 1926 by Hans Busch.

• The **earliest recognized work** describing the concept of an SEM is that of **M. Knoll (1935**) in Germany working in the field of electron optics. The **German physicist Ernst Ruska** and the **Electrical Engineer Max Knoll** constructed the **prototype electron microscope** in 1931, capable of 400X magnification; the apparatus was the first demonstration of the principles of electron microscopy. Two years later, in 1933, Ruska built an electron microscope that exceeded the resolution attainable with an optical microscope.

•**Ernst Ruska**for his fundamental work in electron optics, and for the design of the first electron microscope received **The Nobel Prize in Physics 1986**.

•A SEM that incorporates a demagnifying lens system was first developed by **Von Ardenne (Germany**) in 1938. The height of the optical column of this instrument was about 2 m, and a small electron probe with a diameter of 4 nm was produced by a demagnifying lens system comprised of a two-stage electrostatic lens. This instrument was dedicated to **Scanning Transmission ElectronMicroscopy** (STEM) and it was used for thin-film specimens.

•The SEM developed by **Prof. Dr. Charles Oatlev** with the assistance of graduate students in the 1950s, are one of the three types of Electron Microscopes (EM) which finally led to the marketing of the first commercial instrument by Cambridge Scientific Instrument Company as the "**Stereoscan**" in 1965 (delivered to **DuPont**).

• The improvement of the secondary electron detector was accomplished by **Everhart and Thornley** in 1960. The Everhart-Thornley Detector is a detector used in SEM, named after its designers, T Everhart and RFM Thornley. The Everhart-ThornleyDetector has been available since the fifties, but remains the most frequently used detector in SEMs.

• Also In 1953**, McMullan** succeeded in the development of a SEM producing a resolution of 50 nm at accelerating voltages from 15 to 20 kV, and his laboratory made five SEMs of this kind until 1965.

• In 1965, Cambridge Scientific Instrument (UK) and JEOL (Japan) first commercialized a SEM, individually. During the four decades after the first commercialization of the SEM, about several tens of thousands of SEMs have been manufactured.

• SEM is the next step to Optical microscope so first we need to know why we moved to a new Technology & what the various features of this new Instrument are-

Scanning Electron Microscope vs. Optical Microscope

S. No.	Scanning Electron Microscope	Light Microscope
1.	The biggest advantage is that they have a higher resolution and are therefore also able of a higher magnification (up to 2 million times).Electron microscopes allow for the visualization of structures that would normally be not visible by optical microscopy.	Light microscopes can show a useful magnification only up to 1000-2000 times. This is a physical limit imposed by the wavelength of the light.
2.	Depending on the type of electron microscope, it is possible to view the three dimensional external shape of an object (Scanning Electron Microscope, SEM).	It is always projecting image in 2-d of any object.
3.	The sample must be completely dry. This makes it impossible to observe living specimens.It is not possible to observe moving specimens (they are dead).	One big advantage of light microscopes is the ability to observe living cells.
4.	Illuminating source is beam of electrons.	Illuminating source is light.
5.	Vacuum is essential for its operation.	A vacuum is not required.
6.	It employs electro-magnets.	Light microscope uses glass lenses.
7.	It is very large and complicated.They may need a whole room.	Microscope is compact and handy.
8.	The object is ultrathin, 0.1μm or below.	The object is 5 μm or thicker.
9.	Resolving power is 0.5 -5.0 A^0, through theoretically it can be 0.25 A^0.	Resolving power is 0.25μm or 250nm (2500A^0).
10.	It is not possible to observe color. Electrons do not possess a color. The image is only black/white. Sometimes the image is colored artificially to give a better visual impression.	Image is colored.

11.	Image is seen only on a fluorescent screen.	Image can be seen directly. Specimen is coated with heavy metals in order to reflect electrons.
12.	There is risk of radiation leakage.	Radiation risk is absent.
13.	High voltage electric current is required (50,000 volts and above).	There is no need for high voltage electricity.
14.	Tungsten filament is used to produce electrons.	A filament is not used.
15.	Only dehydrated specimens are used.	The specimen need not be dehydrated.
16.	There are two different types of electron microscopes, scanning electron microscopes (SEM) and transmission electron microscopes (TEM).	The two most common types of microscopes are compound microscopes and stereo microscopes (dissecting microscopes).

Principle

A normal scanning electron microscope operates at a high vacuum. The basic principle is that a beam of electrons is generated by a suitable source, typically a tungsten filament or a field emission gun. The electron beam is accelerated through a high voltage (e.g.: 20 kV) and pass through a system of apertures and electromagnetic lenses to produce a thin beam of electrons., then the beam scans the surface of the specimen by means of scan coils (like the spot in a cathode-ray tube "old-style" television). Electrons are emitted from the specimen by the action of the scanning beam and collected by a suitably-positioned detector.

The microscope operator is watching the image on a screen. Imagine a spot on the screen scanning across the screen from left to right. At the end of the screen, it drops down a line and scans across again, the process being repeated down to the bottom of the screen.

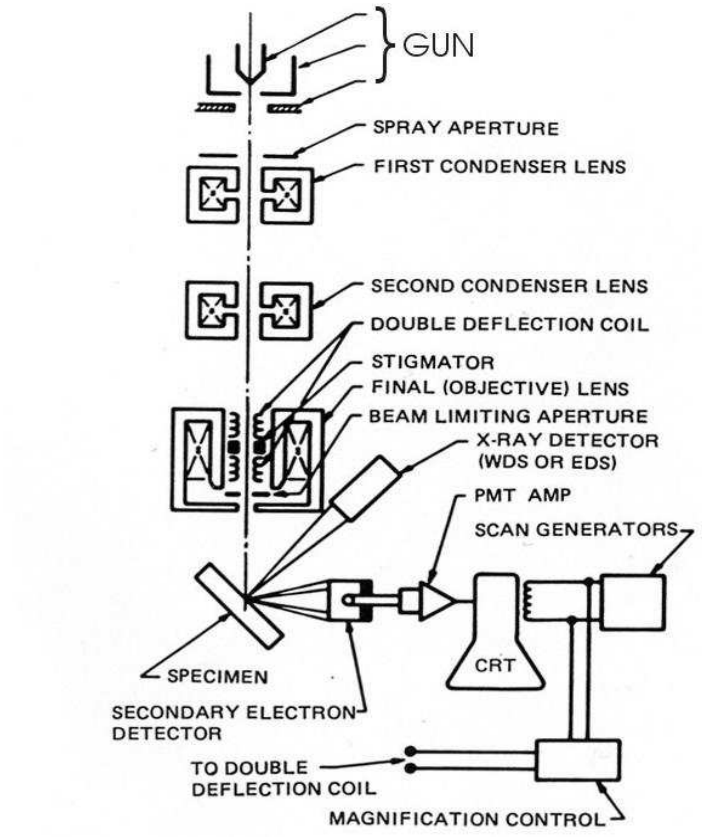

Fig 1- Schematic Model of SEM

Components

1. **Electron Column-**The electron column is where the electron beam is generated under vacuum, focused to a small diameter, and scanned across the surface of a specimen by electromagnetic deflection coils. The lower portion of the column is called the specimen chamber. The secondary electron detector is located above the sample stage inside the specimen chamber. Specimens are mounted and secured onto the stage which is controlled by a goniometer. The manual stage controls are found on the front side of the specimen chamber and allow for x-y-z movement, 360^0 rotation and 90^0 tilt however only the tilt cannot be controlled through the computer system thus there is no need to use all of the manual controls manipulate the orientation of the sample inside the sample chamber. Below is a diagram of the electron column and a description of each of the components of the electron column.

2. **Electron gun-** Located at the top of the column where free electrons are generated by thermionic emission from a tungsten filament at ~2700K. The filament is inside the Wehnelt which controls the number of electrons leaving the gun. Electrons are primarily accelerated toward an anode that is adjustable from 200V to 30 kV (1kV=1000V).

Types of Electron Guns-

- **Tungsten (W) Hairpin Electron Gun:** The typical tungsten electron gun is a "Λ" shape wire filament about 100 μm. To achieve thermionic emission, the filament is heated resistively by the filament heating current.
- **Lanthanum Hexaboride (LaB6) Electron Gun**: is a thermionic emission gun. It is the most common high-brightness source. This source offers about 5-10 times more brightness and a longer lifetime than tungsten, but the required vacuum conditions are more stringent.

Distinguishing Features When Using Single crystal of LaB_6 -

- Tip is ~100μm.
- Chemically reactive when it gets hot.
- Crystal is held by glassy carbon or graphite supports.
- Carbon not reactive with LaB_6.

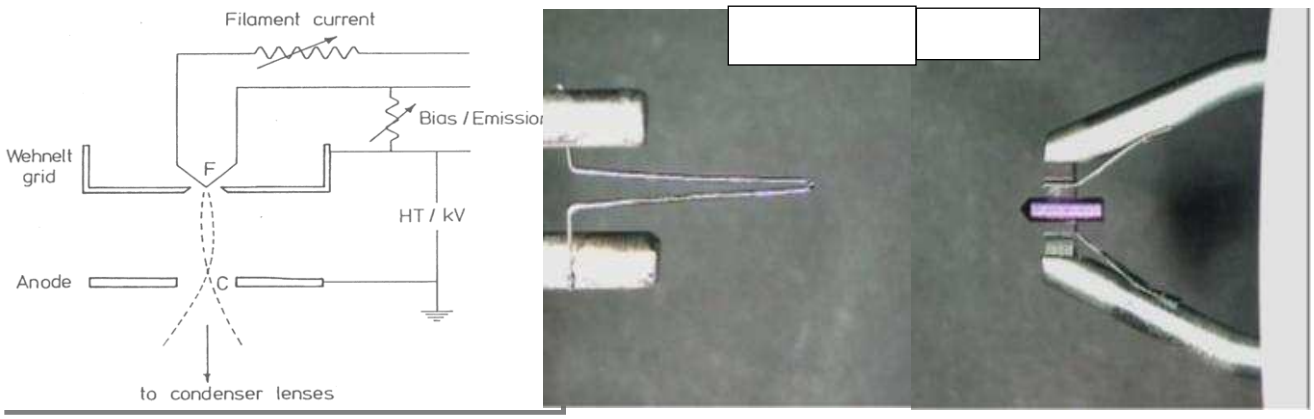

Fig 2- Electrode (a) Schematic (b) Tungsten (c) Ls-B_6

3. **Electron Beam Manipulation-** Electrons in motion are affected by only two things: electrostatic fields and magnetic fields. In the electron gun, electrons are controlled by an electrostatic field, while throughout the rest of the SEM the electrons are controlled by magnetic lenses. Electrostatic lenses are formed when negative and positive fields are near each other. This is the situation between the gun and the anode.

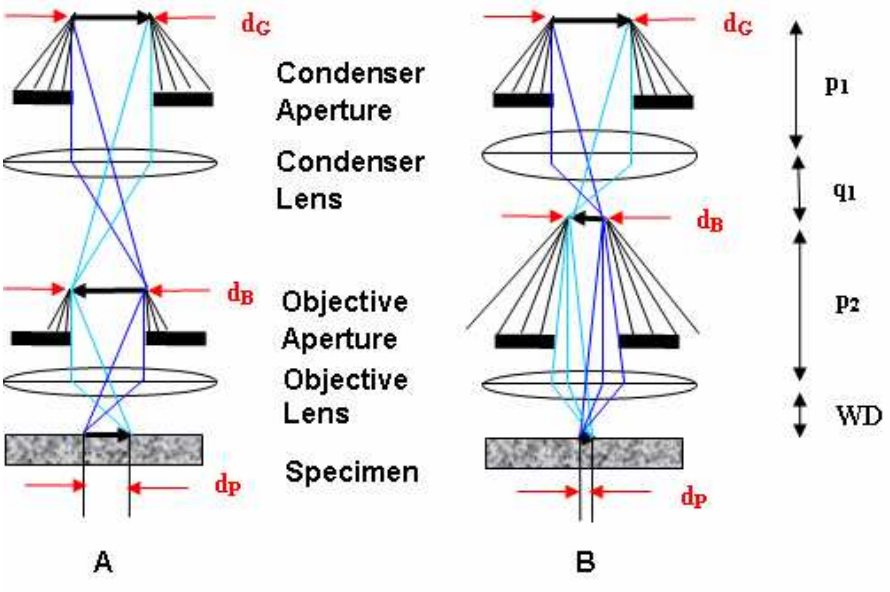

Fig 3- Aperture and Lens

Electrostatic lenses are not used in the microscope column because of the likelihood of arcing. Dirty apertures are undesirable because they will function as electrostatic lenses and thus, affects the electron beam. Magnetic fields are used to form electron microscope lenses by passing electric current through a copper wire. These lenses are known as electromagnetic lenses. Most SEMs use several electromagnetic lenses to reduce the size of the beam's cross-over spot. The lenses are known as **Condenser lenses**. All electromagnetic lenses have spherical aberration. Spherical aberrations is the inability of the lens to image central and peripheral portions of the electron beam at the same focal point (Figure-3).

Other types of magnetic lenses found on a SEM are for correcting astigmatism and alignment. Also, two very important magnetic fields are found within the final condenser lens. As the beam passes through the final condenser lens, two sets of magnetic scanning coils move the beam. These radially opposing, magnetic coils allow scanning in both the X and Y directions. The scan pattern is called a raster pattern and the coils are known as raster coils. Magnification in the SEM is controlled by the ratio of the dimensions of the Cathode Ray Tube (CRT) to the dimensions of the area being scanned. If the scanned length of the display CRT is 100 millimeters, and 1 millimeter of the specimen is scanned, then the magnification is 100 times. There are two ways to adjust the magnification on the SEM:

1) Use the magnification control to change the scanned area of the specimen

2) adjust the focal point of the beam and the Z-axis (working distance) until an appropriate magnification is reached.

Raising or lowering the focal point in relationship to the final lens will determine the magnification of the raster while the Z-axis will bring the sample into focus. Adjusting both the Z-axis and the focal point is the way to obtain a micrograph with an exact magnification. (Besides being able to move the sample up and down on the Z-axis there are four other axes of specimen movement with respect the detector:

1) The X axis, which is the side to side movement

2) The Y axis, which is the forward and backward movement

3) The tilt angle

4) The planar rotation

The last set of electromagnetic coils in the column is for correcting astigmatism. Astigmatism causes poor image quality by causing the beam spot (beam on the sample) to be noncircular, drastically decreasing resolution. There are two directional controls, labeled strength and azimuth, (magnitude and

angle or X and Y etc.....) to correct astigmatism. Most microscopes have 6 to 8 astigmatism coils located radially around the column after the lens and its aperture. This arrangement gives precise directional control. The azimuth control determines which pair of coils is being energized. It is possible to energize adjacent pairs of coils so that there is continuous directional control.

The last controls in electron beam manipulation are apertures. An aperture is just simply a very round hole than can come in a large range of sizes. Apertures control the passing through of scattered electrons from the optical center without moving the beam as electromagnetic lenses. A 20 μm aperture will only let electrons that have strayed less than 10 μm from the center of the beam to continue on. Those greater than 10 μm will be stopped by the aperture plate. The selection of aperture size is dependent on the type of work being done on the SEM. If high resolution is require, a small aperture should be used to minimize the scattering of the beam so that a smaller more precise area (spot size) on the sample is bombarded. If more electrons are needed to hit the sample and resolution is not important then a larger aperture should be used. For example large apertures will increase the number of low probability beam-specimen reactions (discussed later) thatoccur in the sample.

4.**Specimen Chamber-**At the lower portion of the column the specimen stage and controls are located. The secondary electrons from the specimen are attracted to the detector by a positive charge.

Sample Preparation for SEM

The specimen must meet the following requirements before it is loaded to the SEM stage:

a) The surface to observe is exposed.

b) The specimen is firmly fixed to the specimen mount.

c) The specimen has conductivity in principle.

Exposing Surface to Observe and Contrast Enhancement-After cutting the specimen with a suitable size for observation, expose a surface to observe. In principle, when you want to observe the specimen surface itself, special treatment is not required. But if necessary, you are required to remove films that may prevent observation.

When you want to observe internal structures, it is necessary to prepare a cross section. Actual methods are as follows-

Fracturing- If a specimen is hard, it is fractured to prepare a cross section. When a specimen is a structural object, such as semiconductor device that is grown on a Si or GaAs single crystal, it has a cleavage property in a specific direction of the crystal; therefore, fracturing the specimen in this specific direction enables you to obtain a flat cross section. If a specimen is soft at normal temperature but hard at low temperature, freeze fracturing is applied to this material in liquid nitrogen.

Cutting- If a specimen is soft like a polymer, it can be cut using an ultramicrotome, which is originally used to prepare a thin section for a TEM. A cross section planed off by this method is very flat. When only a low-magnification observation is performed, a specimen with a few scars may be acceptable. In such a case, a razor blade is used for cross-section preparation.

Mechanical polishing-For many metal or mineral specimens, mechanical polishing is applied. In this method, a specimen is embedded in a resin and polished.

In the mechanical polishing process, abrasives are gradually changed from rough to fine abrasives and finally, a polished cross section is fabricated to a mirrored surface.

Contrast enhancement- In many specimens, secondary electron images provide no contrast when their cross sections are very smooth. In these cases, various contrast-enhancement techniques are applied. Two examples are explained. The first is selective etching. Surfaces of cross sections are chemically or physically etched to form irregularity on the surface and internal structures are observed using secondary electron images. The second is called “staining.” Specific areas of a high polymer

specimen are stained by heavy metals, such as Os and Ru, and its compositional image is observed by using backscattered electron images. On the other hand, even when such specimen treatment (contrast enhancement) is not applied, if an original specimen has a difference in composition or crystalline property, a compositional image or an ECC image can be observed in backscattered electron mode.

Mounting Specimen- The specimen must be stably fixed to the specimen mount. In addition, the specimen must electrically connect to this specimenmount.

Bulk specimens- Bulk specimens are fixed to the specimen mount by conductive paste or conductive double-sided adhesive tape. If a bulk specimen has a relatively uniform shape, it is clamped with an exclusive specimen holder. If a bulk specimen is nonconductive, it should be coated by conductive paste as wide as possible while keeping an area to observe.

Powders and particles- These specimens are dusted on conductive paste or double-sided adhesive tape. In dusting powders or particles, they must disperse as wide as possible. On the other hand, some specimens can be subjected to the suspension method. In this method, specimens are suspended in the dispersion medium (organic solvent, water, etc.) and they are dropped on an aluminum foil or a Si-wafer, and finally they are dried.

Coating- If a specimen is nonconductive, its surface needs to be coated with a thin metal film so that the surface has conductivity. This technique is called coating, and ion sputtering and vacuum evaporation are typical methods. Ion sputtering is classified into two techniques.

The first technique uses an ion-sputter coater and the second one uses an ion-beam sputter coater. In general, the first technique (ion-sputter coater using diode sputtering) is used. In the ion-sputter coater, positive ions, which are produced by discharge in a low vacuum of about 10 Pa, sputter a target metal and the specimen is coated with the sputtered target metal.

Vacuum evaporation heats and vaporizes a material, forming a thin metal film on the specimen surface. Since the inside of the vacuum evaporator is a high vacuum of about 10-3 Pa, the number of the residual gas molecules in the device is small, causing the scattering of the evaporated material particles to be weak. As a result, the specimen surface cannot be coated with the evaporated material from every direction. In order to form a uniform film on the surface, the specimen is rotated and tilted.

As a coating material, a noble metal (Au, Au-Pd, Pt, Pt-Pd, etc.) is used because it is stable and has a high secondary-electron yield. For high-magnification observation, Au-Pd, Pt or Pt-Pd is used. For

some cases including elemental analysis, C or Al may be used. Pt and Pt-Pd are difficult to evaporate in vacuum. C and Al are difficult to sputter.

If a coated film is thick, this hides fine structures on the specimen surface; therefore, a thinner film is desirable. However, if the film is too thin, it may cause charging because the coating film loses its continuity. In general, a coated film is prepared to be a few to 10 nm in thickness.

Treatment of Biological Specimen- Water-containing specimens, such as biological tissues, are deformed if they are transferred to the SEM specimen chamber without any pre-treatment. In order to prevent the deformation, biological specimens are generally subjected to the following procedure, and then they are observed with their surfaces coated. Foods are also subjected to a process similar to the following procedure-

Removing and cleaning of tissues- In this process, a tissue is cut to make it with an appropriate size so that it can be preserved until the drying process. In order to prevent the deformation of the tissue, sufficient care is needed. Cleaning of its surface may be also necessary.

Fixation-Since a removed tissue starts to change its structure after its death, in order to prevent this change, it is chemically fixed by chemicals such as glutaraldehyde, formaldehyde, and osmium tetroxide. In this process, adsorbing much osmium metal to the tissue may give conductivity to the tissue (conductive staining). For some tissue specimens, rapid freezing (called physical fixation) is used to suppress a structural change of the tissue.

Dehydration-In the dehydration process, to prevent the deformation, the specimen is immersed in an ethanol or acetone solution for a certain period of time while the concentration of the solution is changed in several steps.

Drying-Ethanol or acetone in the tissue specimen is removed and then, this tissue is dried. If natural drying is applied, a surface-tension effect deforms the specimen. Thus, a special drying method, critical-point drying or freeze drying for example, is used.

Working of Scanning Electron Microscope

The key to how the scanning electron microscope works (and this is the clever bit) is that the beam scanning the specimen surface is exactly synchronized with the spot in the screen that the operator is watching. The electron detector controls the brightness of the spot on the screen - as the detector "sees" more electrons from a particular feature, the screen brightness is increased. When there are fewer electrons, the spot on the screen gets darker. These days, the screen is generally a digital monitor, not a glass **CRT**, but the principle is almost same.

The magnification of the image is the ratio of the size of the screen to the size of the area scanned on the specimen. If the screen is 300 mm across and the scanned area on the specimen is 3 mm across, the magnification is x100. To go to a higher magnification, the operator scans a smaller area; if the scanned area is 0.3 mm across, the magnification is x 1000, and so on.

There are different types of electron image. The two most common are the secondary electron image (SEI) and the backscattered electron image (BEI). The SEI is used mainly to image fracture surfaces and gives a high resolution image. The BEI is used typically to image a polished section; the brightness of the BEI is dependent on the atomic number of the specimen (or, for compounds, the average atomic number). For example, lead will appear brighter than iron and calcium oxide will appear brighter than calcium carbonate. The BEI is, in essence, an atomic number map of the specimen surface.

All SEM images are in black-and-white, although they may subsequently have **false colours** applied to them for aesthetic reasons or to aid interpretation.

A development of the normal high-vacuum scanning electron microscope is the ESEM, or Environmental SEM. The ESEM can operate with air in the specimen chamber - the pressure is lower than atmospheric pressure but higher that the high-vacuum of a normal SEM. This has the advantage that wet specimens can be examined without them dehydrating and is especially useful for biological specimens and other specimens containing water, such as freshly-mixed cement paste.

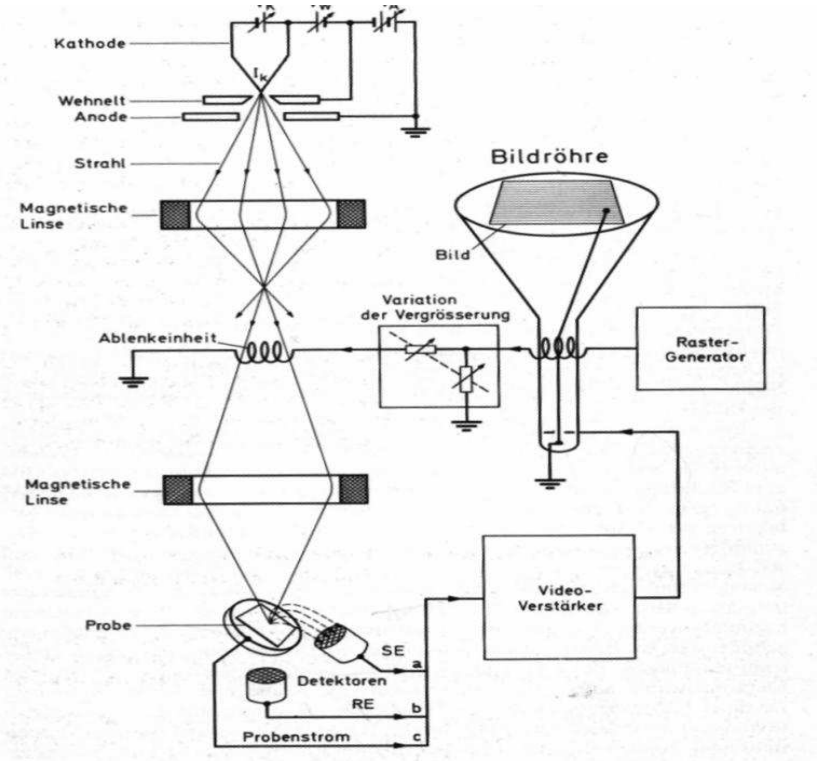

Fig 4- Working Model of SEM

Beam Interaction

Signal detection begins when a beam electron, known as the primary electron enters a specimen. When the primary electron enters a specimen it will probably travel some a distance into the specimen before hitting another particle. After hitting an electron or a nucleus, etc., the primary electron will continue on in a new trajectory. This is known as scattering. It is the scattering events that are most interesting, because it is the components of the scattering events (not all events involve electrons) that can be detected. The result of the primary beam hitting the specimen is the formation of a teardrop shaped reaction vessel .The reaction vessel by definition is where all the scattering events are taking place. Small reaction vessels tend to give better resolution, while large reaction vessels tend to give more signal. The volume of a reaction vessel depends upon the atomic density, topography of the specimen and the acceleration potential of the primary electron beam. For example low density material and higher voltages will result in larger reaction vessels since the electron beam can penetrate deeper into the sample.

Topography will also change the amount of emissions from a reaction vessel. An increase in the topography will increase the surface area of the reaction vessel resulting in more signal. Six or more different events occur in the reaction vessel. These events include:-

1. **Backscattered electrons-** A primary beam electron may be scattered in such a way that it escapes back from the specimen but does not go through the specimen. Backscattered electrons are the original beam electrons and thus, have a high energy level, near that of the gun voltage. Operating in the backscattered imaging mode is useful when relative atomic density information in conjunction with topographical information is to be displayed.

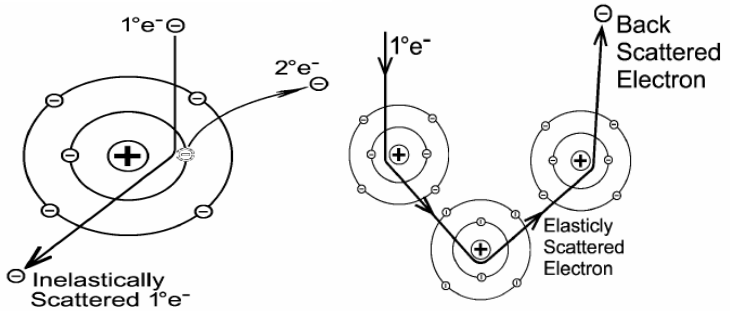

Fig 5- Action of Electrons

2. **Secondary electrons-**Perhaps the most commonly used reaction event is the secondary electron. Secondary electrons are generated when a primary electron dislodges a specimen electron from the specimen surface. Secondary electrons can also be generated by other secondary electrons. Secondary electrons have a low energy level of only a few electron volts, thus, they can only be detected when they are dislodged near the surface of the reaction vessel. Therefore, secondary electrons cannot escape from deep within the reaction vessel. Secondary electrons that are generated but do not escape from the sample are absorbed by the sample specimen atom and a potential expulsion of an electron from that atom as a **secondary electron (SE)**. SEs by definition are less than 50 eV. If the vacancy due to the creation of a secondary electron is filled from a higher level orbital, an X-Ray characteristic of that energy transition is produced.

   Two of the foremost reasons for operating in the secondary electron imaging mode are to obtain topographical information and high resolution. An excellent feature about imaging in the secondary mode is that the contrast and soft shadows of the image closely resemble that of a specimen illuminated with light. Thus, image interpretation is easier because the images appear more familiar. The use of secondary electrons to determine atomic number is not as reliable as with the backscatter model.

3.X-rays-When electrons are dislodged from specific orbits of an atom in the specimen, X-rays are omitted. Elemental information can be obtained in the X-ray mode, because the X-ray generated has a wavelength and energy characteristic of the elemental atom from which it originated. Problems arise when the X-rays hit other particles, they lose energy this changes the wavelength. As the number of hits increases, the x-rays will not have the appropriate energy to be classified as coming from the originating element and detection of these X-rays will be known as background. X-ray spectrometer detectors measure wavelength (wavelength Dispersive Spectrometer or WDS) or energy level (Energy Dispersive Spectrometer or EDS). These are the two types of detectors used in X-ray analysis.

4.Cathode Luminescence- Some specimen molecule's florescence when exposed to an electron beam. In the SEM, this reaction is called cathode luminescence. The florescence produces light photons that can be detected. A compound or structure labeled with a luminescent molecule can be detected by using cathode luminescence techniques. Few SEM are equipped with capability of detecting photons.

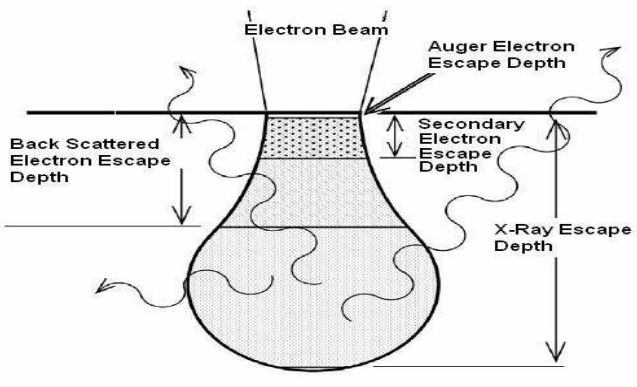

Fig 6- Interaction Volume

5. Specimen Current- When the primary electron undergoes enough scattering such that the energy of the electron is decrease to a point where the electron is absorbed by the sample, this is known as specimen current. The changes in specimen current can be detected and viewed. Like cathode luminescence, this type of detection is seldom available. In most samples, the induced current is just led to ground. If not, the region being bombarded by the beam will build up a negative charge. This charge will increase until a critical point is reached and a discharge of electrons occurs, relieving the pressure of the additional electrons.

6. Transmitted electrons- If the specimen is thin enough, primary electrons may pass through the specimen. These electrons are known as transmitted electrons and they provide some atomic density information. The atomic density information is displayed as a shadow. The higher the atomic number the darker the shadow until no electrons pass through the specimen.

7. Auger Electrons- The energy of Auger electrons is given by the difference between the original excitation energy and the binding energy of the outer shell from which electron was ejected.

Typical Auger electron energies are in the range of a few hundred eV to a few keV and are strongly absorbed within the specimen. An alternative to X-ray emission as anionized atom returns to ground state.

Signal Detection

The majority of the work done on a SEM is for topographical information. Topographical information is mainly provided by secondary electrons that are produced by the interaction of the beam with the specimen.

1. **Detection of Secondary Electron-** A secondary electron detector magnetically attracts emitted secondary electrons by a +200 volt potential applied to a ring around the detector (Faraday Cup).
   Upon entering the ring, the secondary electron is attracted and accelerated by the +10 kilovolt potential on the scintillator. The secondary electrons hit the scintillator causing photons to be emitted. Photons emitted from the scintillator travel down the light pipe hitting the photomultiplier (PM). The function of the photomultiplier is to increase or amplify original signal. Thus, for every photon generated several electrons will be produced, this will result in a significant amplification of the original signal. The amount of amplification of the photomultiplier tube is controlled by the PM voltage control (Contrast Control).

   The strength of the signal to be amplified by the PM depends upon the number of secondary electrons released from the reaction vessel. Since secondary electrons are low energy electrons, their generation is limited to a thin layer close to the specimen surface. Therefore, the number of secondary electrons generated is directly proportional to the area of the emitting surface. The density of the emissive surface will determine signal strength. For example, carbon is a low density material and will emit fewer electrons than a higher density material such as gold. Topological features such as flat surfaces, pointed structures, and edges significantly affect the area of the emissive surface. Flat surfaces (surfaces perpendicular to the beam) will emit fewer electrons because the surface emitting has the smallest surface area. While pointed structures (largest surface area) will emit more electrons. When secondary electrons are emitted behind a larger feature or in a depression some secondary electrons are lost due to re absorption by the specimen. The result is a shadowing effect. However, some detail can be observed because there will be a few electrons escape the shadowed area and are detected.

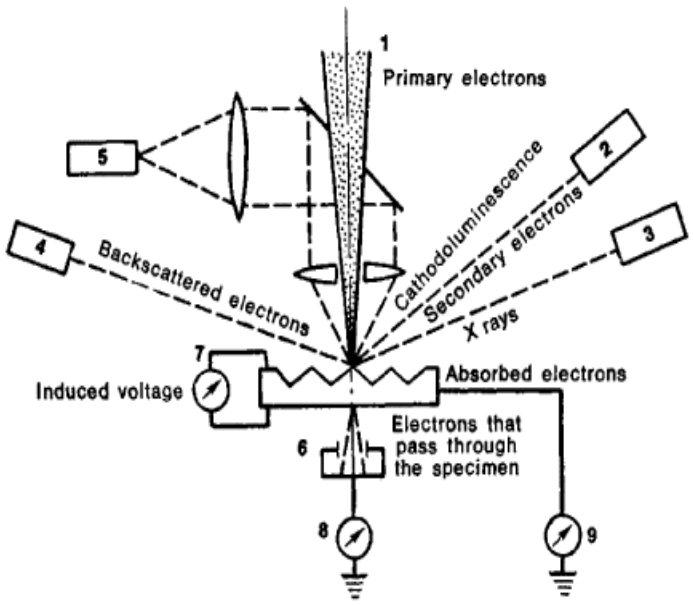

Fig 7- Signal Detection Unit

1) Primary electron beam

2 Secondary-electron detector

3) X-ray detector

4) Back-scattered-electron detector

5) Visible-radiation detector

6) Detector forelectronsthatpass through the specimen

7) Instrument for measuring the electric potential inducedinthespecimen

8) Instrument for measuring the currentduetoelectronsthatpassthrough the specimen

9) Instrument for measuring the current due to electrons that are absorbed by the specimen

2. **Detection of Backscattered Electrons**- Backscattered electrons are those primary electrons that have been scattered back to the surface. Backscattered electrons use a similar detector to that of a secondary detector, with one notable exception, there are no positive voltages applied to it. Because the backscatter detector is collecting those electrons that have bounced back, the placement of the detector needs to be optimized. If the analogy of a person throwing a ball against a wall is used the probability of the ball returning to the person is great. After many throws, however, several of the returns will be away from the thrower and very few if any will return parallel to the wall. The same principle applies with a backscatter detector. The detector should be as close to the primary beam as possible without interfering with the primary beam. Ideally the backscattered detector will surround the primary beam. This placement should maximize the detection on backscattered electrons. Backscattered electrons are detected when a sample's atomic contrast needs to be viewed. Atomic contrast is just a display of an atoms density. Carbon is not dense compared to gold which is very dense. Returning to our analogy, if a person throws a ball at a brick wall the ball will return more often than if the ball is thrown at a screen fence (few balls will go through a brick wall, while many balls will go through screen). As in secondary electron detection, topography will also add to the signal and will interfere with the interpretation of atomic contrast. Therefore when accurate atomic contrast is needed a sample must be as flat and smooth as possible.

3. **X-ray detector**- X-rays are very characteristic of the element from which they originated. If an X-ray hits and is scattered by another particle, the X-ray will lose energy and this will change the wavelength from when it was emitted. The probability of detecting an X-ray after losing some its energy will be most common at the low energy end of the spectra and gradually decreases towards the high energy end. This is called background x-rays or background. Background X-rays are also known as uncharacteristic X-rays because the energy and wavelengths they possess no longer are characteristic of the element they originated from. Background x-rays can interfere with elemental identification when elements are in low concentrations. Especially the low energy characteristic x-ray lines where the background is significantly higher. There are two types of detectors and they both obtain data differently. An EDS X-ray detector will gather the entire spectrum of X-rays from 0 eV to greater than 30 eV. The EDS X-ray detector is an excellent detector when dealing with unknowns or large concentrations but when more analytical work is required a WDS X-ray detector should be used.

4. **WDS detector**- A WDS detector is set to detect a specific range of wavelengths. Wavelengths outside this range will not be detected. Because the WDS is set to detect a small range, the sensitivity of the detector is greater and allows for greater accuracy in analytical work. Using this type of detector, however, is cumbersome when dealing with an unknown because of the many spectra that need to be gathered to determine the elemental composition of an unknown sample.
With any type of X-ray work it is important that the sample be as smooth as possible. Topography will influence the X-ray counts and gives a poor determination of the elemental composition. This is the main reason X-ray analyses are often difficult to use with biological samples.

After studying how the SEM works, The Beam interacts with the specimen and how the Signal is detected we are now going to see how the researchers are working in this small dimension, what are the different images formed and how to interpret the result from them.

Microstructure of Aluminium Matrix Composites

This work focuses on the effect of graphite particles addition on the microstructure of Al6082 metalmatrix composites manufactured by conventional stir casting process. The reinforcement content wasvaried from 0% to 12% in a step of 3%. The microstructures of the manufactured composites were analyzedby scanning electron micrographic test.

Chemical composition of base metal Al6082

Constituent	Al	Cu	Mg	Si	Fe	Ni	Mn	Zn
Content %	97.14	0.038	0.690	1.16	0.258	0.04	0.580	0.027

Fig8 (a) shows the SEM image of Gr particles. Fig. 8(b) showsthe microstructure of cast Al6082, and the microstructure contains solid solution of aluminium and inter-dendritic networkof aluminium silicon eutectic. The microstructure of cast Al6082 (Fig. 8(b)) reveals the formation of aluminium dendritic networkstructure which is formed due to super-cooling of casting during solidification, with less impurities present.

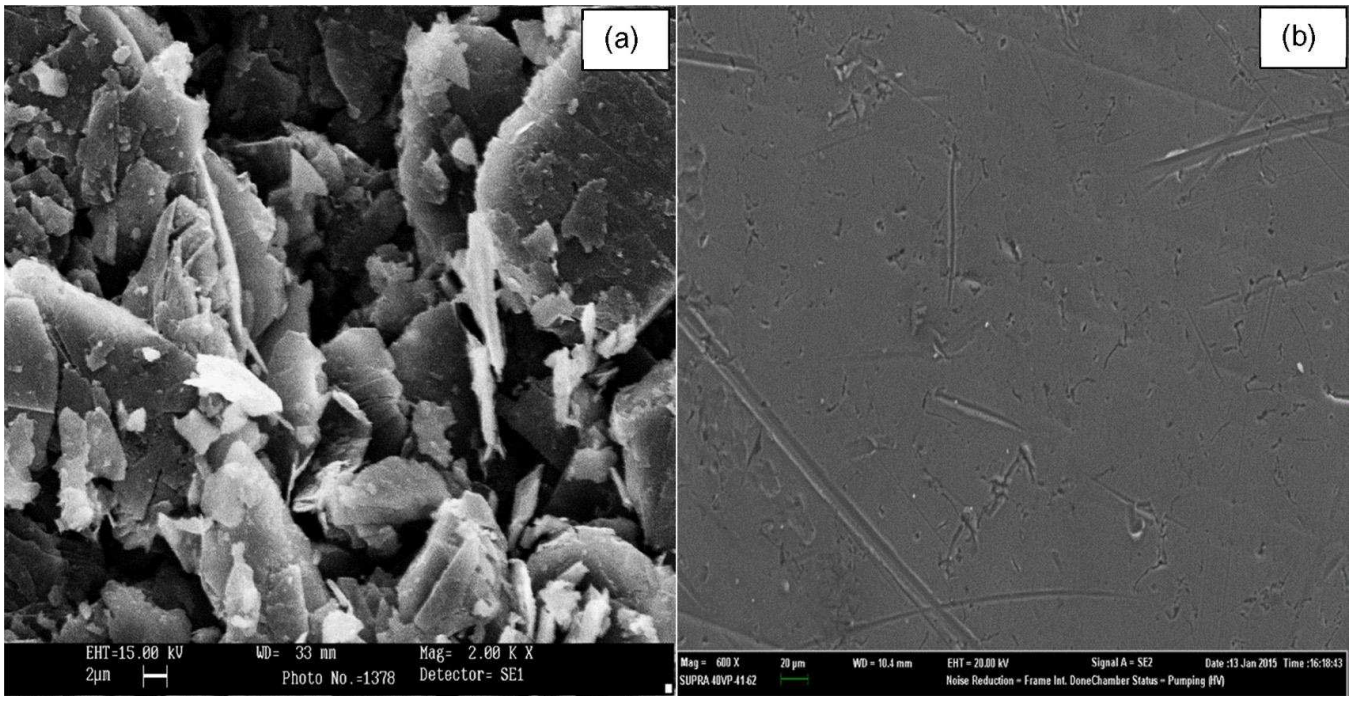

Fig 8 (a) SEM image of pure Gr reinforcement. (b) SEM image of cast metal 6082.

The SEM image ofmanufactured composites is shown in Fig. 9(a)–(d). The microstructure of all composites reveals that there are large impurities witha non-uniform distribution of Gr particles along with clusteringof Gr particles at some locations.

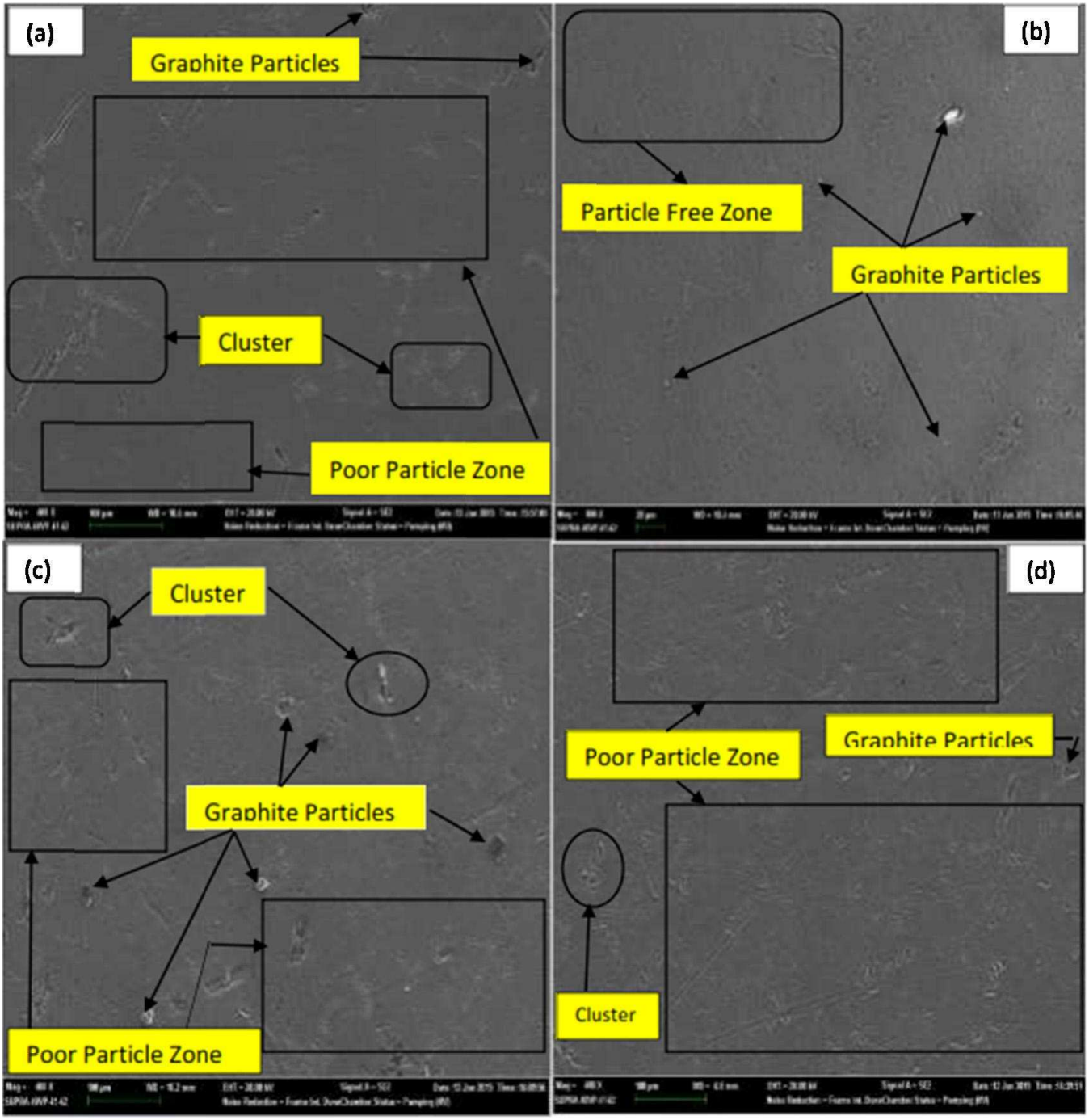

Fig 9- SEM image of Al–Gr composites: (a) 3% Gr, (b) 6% Gr, (c) 9% Gr and (d) 12% Gr.

The low density of Gr particlesas compared to that of Al6082 causes the Gr particles to float inthe aluminium melt resulting in non-uniform distribution. Themicrostructures of all composites contain solid solution of aluminium and inter-dendritic network of aluminium silicon eutectic.When the composites are solidifying, the Gr particles are prohibited in the direction of refined-Al grains. Because of this, thefurther refinement of Al grains takes place and Gr particles actas nucleus on which the Al grains solidify and Gr particles offerconfrontation against growing -Al phase during the solidificationprocess.

Elements present in Al6082 + 12% Gr reinforced composites

Element	Weight %	Atomic %	Compound(form)
Al	84.76	72.48	Al_2O_3
Si	0.59	0.49	SiO_2
Mg	0.41	0.39	MgO
Mn	0.47	0.20	Mn
C	13.77	26.45	$CaCO_3$

This showsthe presence of basic elements of Al6082 + 12% Gr reinforced composite in the compound form. The elemental maps confirm that themanufactured composite is reinforced with Gr particles.

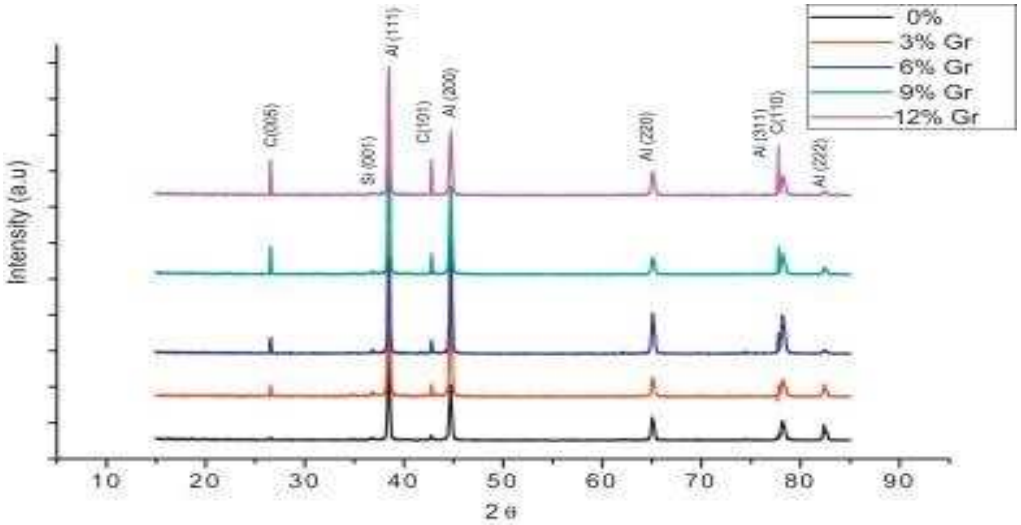

Fig 10- XRD pattern of composites

Fig 10 shows the XRD results of manufactured composites. XRDgives the details about the elements present in the manufacturedcomposites. The results of XRD reveal that the main elementspresent are Al (largest peak), C (second largest peak) and Si (lower-peak). The Al is present in the form of phase i.e. Al (1 1 1), Al (2 0 0),Al (2 2 0), Al (3 1 1), Al (2 2 2) and C in the form C (0 0 5), C (1 0 1)and C (1 1 0) whereas Si is present in the form of Si (0 0 1). Thesepeaks are identified by using **JCPDS software**. The peaks of Gr (inthe form of C) are clearly visible in all manufactured composites.The peaks of C are increasing with the increasing content of Gr inthe manufactured composites. The peaks of Al in the manufacturedcomposites are slightly shifted to 2 theta angles with the increasing content of Gr when compared to that of cast AA6082. The XRDresults also confirm the elemental map results which proves thatmanufactured composites are Gr reinforced composites.

The study was carried out to find some new results and to establish a new composite. For that purpose here are some of the conclusions as stated by research team is-

- Scanning electron micrograph, elemental mapping and XRD confirmed the validation of manufactured composites.
- The scanning electron micrograph revealed a reasonably non-uniform distribution of Gr particles in the matrix.

Several Factors Affecting the Scanning Process

- **Resolution of the SEM-**The spatial resolution of the SEM depends on the size of the electron spot, which in turn depends on both the wavelength of the electrons andthe electron-opticalsystem that produces the scanning beam. The resolution is also limited by the size of the interaction volume, the volume ofspecimen material that interacts with the electron beam. The spot size and the interaction volume are both large compared to the distances between atoms, so the resolution of the SEM is not high enough to image individual atoms, as is possible transmission electron microscope (TEM). The SEM has compensating advantages, though, including the ability to image a comparatively large area of the specimen; The abilityto image bulk materials (not just thin films or foils) and the variety of analytical mode available for measuring the composition and properties of the specimen. Depending on the instrument, the resolution can fall somewhere between less than 1 nm and 20 nm. As of 2009, world's highest resolution conventional (<30kV) SEM can reach a point resolution of 0.4nm using a secondary electron detector.

- **Effect of Accelerating Voltage-**The accelerating voltage on the Qunata 200 is adjustable from200V-30 kV. Choosing the right accelerating voltage is critical for obtaining a good clear image however the most suitable voltage level depends will dependon mostly on the type of material being examined. The more conductive thematerial the better it will behave under higher voltages. Higher voltages (15-30kv) generally allow for high resolution at high magnifications although, this candamage the specimen very quickly if it is not highly conductive. Thus, whenimaging polymers and ceramics it is more suitable to use voltages below 10 kV.

- **Effect of Working Distance-** Besides the accelerating voltage one's choice of working distance andspot size will greatly influence the image quality. As with accelerating voltage there exists a give and take situation when choosing the most suitable settingsfor working distance and spot size. Generally speaking a working distance of 10mm should be used and will allow for a good depth of field while maintaining good resolution. In most cases one may want to reduce the working distance to achieve better resolution especially when using lower accelerating voltages.

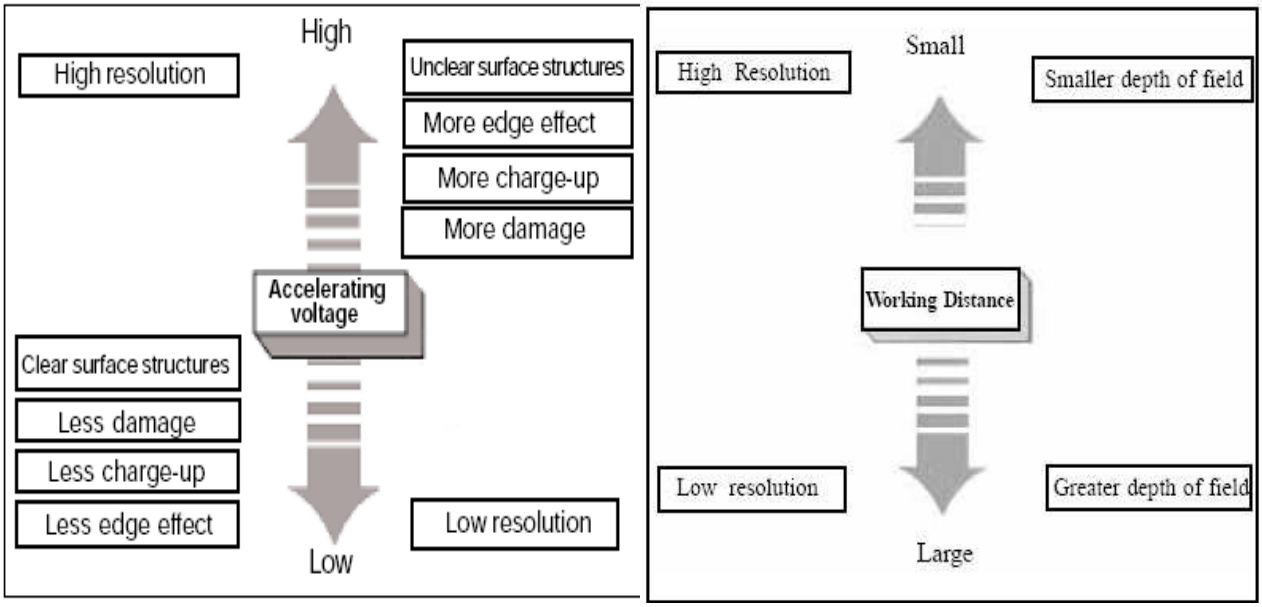

Fig 11 (a)Effect of Accelerating Voltage (b) Effect of Working Distance

- **Effect of Spot Size-**Spot size basically restricts the beam current and will thereby cause for brightness and contrast compensations. Smaller spot sizes will require higher brightness and contrast levels thus there can be a limits when using a small spotsize. Typically smaller spot sizes allow for higher resolution and a greater depth of field.

Strengths

There is arguably no other instrument with the breadth of applications in the study of solid materials that compares with the SEM-

1. The SEM is critical in all fields that require characterization of solid materials.

2. While this contribution is most concerned with geological applications, it is important to note that these applications are a very small subset of the scientific and industrial applications that exist for this instrumentation.

3. Most SEM's are comparatively easy to operate, with user friendly "intuitive" interfaces.

4. Many applications require minimal sample preparation.

5. For many applications, data acquisition is rapid (less than 5 minutes/image for SEI, BSE, spot EDS analyses).

6. Modern SEMs generate data in digital formats, which are highly portable.

7. Advantages of a Scanning Electron Microscope include its wide-array of applications, the detailed 3-d and topographical imaging and the versatile information garnered from different detectors.

8. SEMs are also easy to operate with the proper training and advances in computer technology and associated software make operation user-friendly.

9. Although all samples must be prepared before placed in the vacuum chamber, most SEM samples require minimal preparation actions.

Disadvantages

The disadvantages of a Scanning Electron Microscope start with the size and cost-

1. SEMs are expensive, large and must be housed in an area free of any possible electric, magnetic or vibration interference.
2. Maintenance involves keeping a steady voltage, currents to electromagnetic coils and circulation of cool water.

3. Special training is required to operate an SEM as well as prepare samples.
4. The preparation of samples can result in artifacts.
5. The negative impact can be minimized with knowledgeable experience researchers being able to identify artifacts from actual data as well as preparation skill.
6. There is no absolute way to eliminate or identify all potential artifacts.
7. In addition, SEMs are limited to solid, inorganic samples small enough to fit inside the vacuum chamber that can handle moderate vacuum pressure.
8. Finally, SEMs carry a small risk of radiation exposure associated with the electrons that scatter from beneath the sample surface.
9. The sample chamber is designed to prevent any electrical and magnetic interference, which should eliminate the chance of radiation escaping the system.
10. Samples must be solid and they must fit into the microscope chamber.
11. Maximum size in horizontal dimensions is usually on the order of 10 cm, vertical dimensions are generally much more limited and rarely exceed 40 mm.
12. For most instruments samples must be stable in a vacuum on the order of 105-106 torr. Samples likely to outgas at low pressures (rocks saturated with hydrocarbons, "wet" samples such as coal, organic materials or swelling clays, and samples likely to decrepitate at low pressure) are unsuitable for examination in conventional SEM's.
13. However, "low vacuum" and "environmental" SEMs also exist, and many of these types of samples can be successfully examined in these specialized instruments.
14. EDS detectors on SEM's cannot detect very light elements (H, He, and Li), and many instruments cannot detect elements with atomic numbers less than 11 (Na).
15. Most SEMsuse a solid state x-ray detector (EDS), and while these detectors are very fast and easy to utilize.
16. Theyhave relatively poor energy resolution and sensitivity to elements present in low abundances when compared to wavelength dispersive x-ray detectors (WDS) on most electron probe microanalyzers(EPMA).

Range of Materials for Investigation by SEM

- Metals, Glass and Ceramics
- Semiconductors
- Plastics and polymers
- Powders and Dust
- Composite Materials
- Fibers(Textile, fabric , man-made, natural, carbon fibers, glass fibers, Kevlar)

Software for Scanning Electron Microscopes

- Software available for SEMs by Carl Zeiss include:
- GSR packages from various EDS suppliers
- Image analysis for general applications
- 3D-reconstruction for measuring of depth and shape

Applications

The SEM is routinely used to generate high resolutionimages of shapes of objects(SEI) and to show spatial variations in chemical compositions-

1) Acquiring elemental maps or spot chemical analyses using EDS,

2) Discrimination of phases based on mean atomic number (commonly related to relative density) using BSE

3) Compositionalmaps based on differences in trace element "activators" (typically transition metal andRare Earth elements) using CL.

The SEM is also widely used to identify phases basedon qualitative chemical analysis and/or crystalline structure. Precise measurement ofvery small features and objects down to 50 nm in size is also accomplished using the SEM. Backescattered electron images (BSE) can be used for rapid discrimination of phases in multiphase samples. SEMs equipped with diffracted backscattered electron detectors (EBSD) can be used to examine microfabric and crystallographic orientation in many materials.

Typical Applications include-

- Identification of metals and materials
- Particle contamination identification and elimination
- Product and process failure and defect analysis
- Examination of surface morphology (including stereo imaging)
- Analysis and identification of surface and airborne contamination
- Powder morphology, particle size and analysis
- Cleaning problems and chemical etching
- Welding and joining technology quality evaluation and failure investigation
- Paint and coating failure and delamination investigation
- Paint, Adhesive, Sealant and Gasket Filler Fingerprinting
- Identification and elimination of corrosion and oxidization problems

- Contamination or stain investigation
- Structural analysis
- Reverse engineering of products and processes