ENVIRONMENTAL SCANNING ELECTRON MICROSCOPE -ESEM

Conventional SEM requires samples to be imaged under vacuum, because a gas atmosphere rapidly spreads and attenuates electron beams. Consequently, samples that produce a significant amount of vapour, e.g. wet biological samples or oil-bearing rock need to be either dried or cryogenically frozen. Processes involving phase transitions, such as the drying of adhesives or melting of alloys, liquid transport, chemical reactions, solid-air-gas systems and *living organisms in general cannot be observed.* The *accumulation of electric charge* on the surfaces of non-metallic specimens can be avoided by using Environmental SEM in which the *specimen is placed in an internal chamber at higher pressure* than the vacuum in the electron optical column. *Positively charged ions generated by beam interactions with the gas help to neutralize the negative charge on the specimen surface*. The pressure of gas in the chamber can be controlled, and the type of gas used can be varied according to need. Coating is thus unnecessary, and X-ray analysis unhindered.

The first commercial development of the Environmental SEM (ESEM) in the late 1980s allowed samples to be observed in low-pressure gaseous environments (e.g. 1-50 Torr) and high relative humidity (up to 100%). This was made possible by the development of a *secondary-electron detector* capable of operating in the *presence of water vapour* and by the use of pressure-limiting apertures with differential pumping in the path of the electron beam to separate the vacuum regions around the gun and lenses from the sample chamber.
The first commercial ESEMs were produced by the *ElectroScan Corporation* in USA in 1988. ElectroScan were later taken over by Philips (now FEI Company) in 1996.

ESEM is especially useful for *non-metallic and biological materials* because coating with carbon or gold is unnecessary. *Uncoated Plastics and Elastomers can be routinely examined*, as can uncoated biological samples. Coating can be difficult to reverse, *may conceal small features* on the surface of the sample and may reduce the value of the results obtained. X-ray analysis is difficult with a coating of a heavy metal, so carbon coatings are routinely used in conventional SEMs, but ESEM makes it possible *to perform X-ray microanalysis* on uncoated non-conductive specimens. ESEM may be the preferred for electron microscopy of unique samples from criminal or civil actions, where forensic analysis may need to be repeated by several different experts. Wet, oily, dirty, non-conductive samples may be examined in their natural state without modification or preparation. The ESEM offers high resolution *secondary electron-imaging* in a gaseous environment of practically any composition, at pressures as high as 50 Torr, and temperatures as high as 1500 °C.

All SEM's consist of an *electron column*, that creates a beam of electrons; a sample chamber, where the electron beam interacts with the sample; detectors, that monitor a variety of signals resulting from the beam-sample interaction; and a viewing system, that constructs an image from the signal. An electron gun at the top of the column generates the electron beam. In the gun, an electrostatic field directs electrons, emitted from a very small region on the surface of an electrode, through a small spot called the *crossover*. The gun then accelerates the electrons down the column towards the sample with energies typically ranging from a few hundred to tens of thousands of *electron volts*. The electrons emerge from the gun as a divergent beam. A series of *magnetic lenses and apertures* in the column *reconverges* and *focuses* the beam into a *demagnified image* of the crossover. Near the bottom of the column a set of *scan coils deflects* the beam in a scanning pattern over the sample surface. The final lens focuses the beam into the *smallest possible spot* on the sample surface. The beam exits from the column into the sample chamber. The chamber incorporates a stage for manipulating the sample, a door for inserting and removing the sample and access ports for mounting various signal detectors and

other accessories. As the beam *electrons penetrate the sample*, they give up energy, which is emitted from the sample in a variety of ways. There are two major ways of emission: *Secondary Electrons* (SE) are *sample atom electrons* that have been ejected by interactions with the primary electrons of the beam. They generally have very low energy (by convention less than fifty electron volts). Because of their low energy they can escape only from a very shallow region at the sample surface. As a result they offer the best

Imaging resolution.

Contrast in a secondary electron image comes primarily from sample topography. More of the volume of interaction is close to the sample surface, and therefore more secondary electrons can escape, for a point at the top of a peak than for a point at the bottom of a valley. *Peaks are bright. Valleys are dark.* This makes the interpretation of secondary images very intuitive. They look just like the corresponding visual image would look. *Backscattered Electrons (BSE)* are primarily beam electrons that have been *scattered back out of the sample* by elastic collisions with the nuclei of sample atoms. They have high-energy, ranging (by convention) from *fifty electron volts* up to the accelerating voltage of the beam. Their higher energy results in a larger specific volume of interaction and degrades the resolution of backscattered electron images. Contrast in backscattered images comes primarily from point to point differences in the average atomic number of the sample. *High atomic number nuclei backscatter* more electrons and *create bright areas* in the image. Backscattered images are not as easy to interpret, but properly interpreted, can provide important information about sample composition. Each emission mode is potentially a signal from which to create an image.

The new technology in ESEM

High vacuum conditions are required in the electron gas and throughout the column, where gas molecules can scatter electrons and degrade the beam. Instead of using a single pressure limiting aperture in conventional SEM, ESEM uses multiple Pressure Limiting Apertures (PLA's) to separate the sample chamber from the column. The column is still high vacuum, but the chamber may sustain pressures as high as 50 Torr.

ESEM uses a proprietary *Environmental Secondary Detector* (ESD) which can function in non-vacuum environment instead of *Everhart-Thornley (ET) detector* used in SEM. The ESD uses the principle of *gas ionization*. By applying a *positive potential* of a few hundred volts to the detector, the *secondary electron emitted* by the sample when interacts with electron beam is *attracted to detector*. As the *electrons accelerate in the detector field, they collide with gas molecules*. The resulting ionizations create *additional electrons, amplifying original secondary electron signal*, and positive ions. The *detector collects secondary electron signal* and passes it directly to an *electron amplifier*. In nonconductive samples *the positive ions* created in gas ionization process are *attracted to the sample surface* and they effectively suppress charging artifacts.

Advantages of ESEM

1. Gas ionization in the sample chamber eliminates the charging artifacts, typically seen with nonconductive samples. So the specimens do not need to be coated with a conductive film. ESEM gets rid of the preparation process.

2. The ESEM can image wet, dirty and oily samples. The contaminants do not damage the or degrade the image quality.

3. ESEM can acquire electron images from samples as hot as 1500ºC, because the Environmental Secondary Detector (ESD) is insensitive to heat.

4. The detector is also insensitive to light. Light from the sample, for example incandescence from heated samples, cathodoluminescence and fluorescence do not interfere with imaging.

5. ESEM eliminates the need for conductive coating, so delicate structure, which was often damaged during the sample preparation, can be imaged.

6. ESEM can acquire x-ray data from insulating samples at high accelerating voltage.

7. Eliminating the need for sample preparation, particularly the need for conductive coating, makes it possible to investigate specimen in dynamic processes, such as tension, compression, deformation, crack propagation, adhesion, heating, cooling, freezing, melting, hydration, dehydration and sublimation.