CHAPTER 9

SCANNING ELECTRON MICROSCOPY
# SCANNING ELECTRON MICROSCOPY

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9.1 General Introduction

Scanning electron microscope (SEM) is a kind of electron microscope that produces images of a specimen by scanning it with a focused beam of electrons. SEM is a type of electron microscope that images the sample by scanning it with high energy beam of electrons which is used for examination and analysis of microstructural characteristics of the solid object. The signals obtained from electron-sample interaction reveal information about the sample including external morphology (texture), chemical composition, and crystalline structure and orientation of materials making up the sample.

This technique was first of all used by Knoll (1935) and Von Ardenne (1938). The SEM used for thicker substances was described by Zworykin et al (1942). Since then the advancement in this technique is continued and reaches to the resolution of 8000 times. The first commercial SEM was made in 1965. Modern SEM are equipped with digital store for images, computer screens, high resolution displays and convenient output methods. The recently variable-pressure SEM are used for wet and dry specimens. The three dimension image produced is continently interpreted and definitive measurements.

SEM delivers high-resolution surface data and greater materials contrast. They are widely used in electron microscopy sciences and application fields of Nanotechnology, Materials analysis, Semiconductor failure analysis, Life sciences and Quality assurance. Environmental SEM is especially useful for non-metallic and biological materials because coating with carbon or gold is unnecessary. It makes it possible to accomplish X-ray microanalysis on uncoated non-conductive samples.

Scanning electron microscopy uses a focused beam of high-energy electrons to produce a diversity of signals at the surface of solid specimens. The signals that derive from electron-sample interactions disclose information about the sample, including external morphology.
(texture), chemical composition, crystalline structure and orientation of materials.

SEM's advantages over traditional microscopes include:

- Large depth of field - This allows more of a specimen to be in focus at one time.
- Much higher resolution - Specimens can be magnified at much higher levels.
- Microanalysis - Advanced microscopes can tell researchers information about its composition.

### 9.2 Principle

The basic components of the scanning electron microscope are lens system, electron gun, and electron collector, visual and photo recording cathode ray tubes and associated electronics. The SEM components can be divided into electron column and control consol. The electron column consist often electron gun electron lenses. The accelerated electrons are generated having the energy of 0.1 to 30 KeV. The control consol consists of cathode ray tubes showing screen, knots and computer control systems.

The electron beams scanned across the surface of specimen and back scattered and detected. The output signals are synchronized and magnified and recorded. The electrons are focused from electromagnetic field.
The two signals are used to produce SEM images are secondary electrons and backscattered electrons. The detectors collect the both and amplify for display and formation of images.

SEM has the following capabilities

- Secondary electron images for topography, fractography, morphology, crystal structure, particle sizes and shapes
- Robinson electron back scatter detector to distinguish phases and areas based on atomic mass differences
- Large depth of field to provide well-focused images of rough and textured surfaces
- Electron beam energies of 2, 5, 10, 15, 20, 25, and 30 KeV
- High resolution – 5 nm capability
- Useful magnifications from 20X – 25,000X

9.3 SEM Samples and Materials

The following substances are used for the SEM analysis, samples should be carefully prepared for high vacuum compatibility excessively high out gassing and liquid evaporation is to be avoided. The best images are provided by electrically conductive materials because the SEM uses electrons to scan a sample surface. This technique is used in all engineering materials and solid chemicals, including metals, semiconductors, inorganic particles, glasses, ceramics, many polymers, and some organic particles. Many non-metals, ceramics, and organic materials may need to be coated with a conductive material. We have facilities for coating by sputter deposition. Semiconductors often do not need coating. Where coating will adversely affect the sample properties of interest, low electron beam energies can often be used instead for imaging the sample. Cross-section microscopy by SEM sometimes requires coating if sample edges are to be viewed at high resolution when the sample was potted in an epoxy for polishing. Sample size should be from less than 0.1 mm to 150 x 125mm x 50mm height, but samples with the widest dimensions can only be examined near their centers.

9.4 SEM Applications
The SEM can be applied to

- Observe surface topography and morphology at high resolution
- Observe rough or raised-feature surface with great depth-of-field
- Measure thin film and coating thickness with calibration standard reference
- Measure the size of surface features
- Measure particle sizes and determine particle shapes
- Metallographic characterization of metal alloys for precipitated phases, intermetallic phases, and carbides.
- Find number and density of surface defects
- Measure number and density of inclusions in metal alloy and determine their kind
- Examine intermetallic phase microstructures
- Determine appearance of stains and residues or deposits on surfaces due to contamination
- Determine whether fracture surface is intergranular or transgranular
- Observe evidence for locus of failure in adhesive bonding failures
- Observe the nucleation sites of the initial chemical attack starting a corrosion problem. Often the key to preventing corrosion is to prevent this initial attack, which may start at a small surface defect, such as an inclusion
- Observe defects in microelectronics dyes
- Observe contamination on electronic contacts with enhanced surface imaging at low electron beam voltages
- Examine precipitates at grain boundaries
- Observe damage and defects in micro- and nano-devices
- Observe agglomeration of particles
- Examine quality of surface finish
- Examine micro cracking in surfaces, thin films, and coatings
- Find pin holes in coatings
- Examine thin film growth modes, such as columnar growth
- Examine grain size in weld heat-affected zone
Discover evidence for chemical attack of a surface in very early stages

- Measure the grain size of polished and etched polycrystalline materials.
- Examine grain shape and orientation, often important in extruded and rolled materials.
- Examine micro-cutting features made by laser cutting techniques
- Examine residues left in sintered ceramics
- Examine pull-out of fibers in composite materials upon fracture for evidence of poor wetting and bonding.

**9.5 Observations and Conclusions**

It is found that the trichloroacetic acid is highly aggressive medium for the mild steel. The images in 0.5M and 1.0M trichloroacetic acid are shown in figures 8.3.1.0 and 8.3.2.0 and with the schiff bases in figure 8.3.1.1 to 8.3.1.4 and 8.3.2.1 to 8.3.2.4. The samples are prepared for the immersion of one hour in the acid. A large number of pits with large size and high depth distributed over the surface are seen in 1.0M trichloroacetic acid. Mild steel has a very porous structure after exposing to 0.5 M trichloroacetic acid solution in the absence of inhibitor.

The smooth mild surface was seriously damaged by the corrosion when placed in 0.5M trichloroacetic acid for one hour immersion exposure. The surface shows inclusions and the surface roughness. This is because of the attack of the acid on the metal surface and cause degradation of the metal. In presence of the schiff bases, it is observed that the surface of the metal was protected by the adsorption of the inhibitor. No pits and cracks except polishing lines or the schiff base layer were observed in image after the addition of Schiff base to the aggressive solution. It is seen that the surface was more strongly affected in 1.0M than in 0.5M trichloroacetic acid.

The inhibitor molecules involve in the interaction with reactive sites of the mild steel surface resulting in the decrease in the contact of the mild steel with the aggressive acid. The surface is more uniform in the
inhibited solution than the surface in the absence of the inhibitor except some hollows structures. The attacks of the corrosive ions can be prevented by forming a passive layer in the form of organometallic complexes on the surface of mild steel. The result indicate that the adsorption of the schiff bases are better in case of the 0.5M than in 1.0M trichloroacetic acid.

The result was an enhancement of surface coverage on the steel surface such that there was a decrease in contact between the steel and the aggressive medium. Thus, a good absorptive protection layer can efficiently inhibit the corrosion of steel.
Fig. 9.1.0: SEM image of the mild steel in 0.5M trichloroacetic acid

Fig. 9.1.1: SEM image of the mild steel in 0.5M trichloroacetic acid in presence of SB-I
Fig. 9.1.2: SEM image of the mild steel in 0.5M trichloroacetic acid in presence of SB-II

Fig. 9.1.3: SEM image of the mild steel in 0.5M trichloroacetic acid in presence of SB-III
Fig. 9.1.4: SEM image of the mild steel in 0.5M trichloroacetic acid in presence of SB-IV

Fig. 9.2.0: SEM image of the mild steel in 1.0M trichloroacetic acid
Fig. 9.2.1: SEM image of the mild steel in 1.0M trichloroacetic acid in presence of SB-I

Fig. 9.2.2: SEM image of the mild steel in 1.0M trichloroacetic acid in presence of SB-II
Fig. 9.2.3: SEM image of the mild steel in 1.0M trichloroacetic acid in presence of SB-III

Fig. 9.2.4: SEM image of the mild steel in 1.0M trichloroacetic acid in presence of SB-IV
References