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Characterization of Materials Using Scanning Electron Microscope (The effects of composition, magnification and coating material)

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ABSTRACT

Scanning Electron Microscopy (SEM) is a versatile tool used to investigate the microstructures of materials such as ceramics, polymers, and semiconductors. It is invaluable for a wide range of investigations, including microstructural development, structure–property correlations, process control, failure analysis, quality control, surface characterization, grain orientation, and elemental determination of materials. SEM is employed for both qualitative and quantitative analyses through the use of attachments such as an Energy Dispersive X-ray Spectrometer (EDX). Imaging in SEM is influenced by various factors; it depends on the type of signal used, as well as the composition and surface characteristics of the material. Imaging defects in non-conducting materials, such as charging, can be prevented by coating the sample with a conductive material (e.g., Au or Au-Pd) or by maintaining the sample chamber under variable pressure (VP) conditions. The objective of the current study is to demonstrate the principles, operation, and applications of SEM. The effects of composition, magnification, and coating materials were also investigated.

Keywords: Material characterization, SEM, EDX, EDXRF.

Introduction

The Scanning Electron Microscope (SEM) is a type of electron microscope that images a sample surface by scanning it with a high-energy electron beam in a raster scan pattern. The electrons bombard the atoms that make up the sample, producing signals that contain information about the sample’s surface topography, composition, and other properties, such as electrical conductivity [1]. A wide range of magnifications is possible, ranging from approximately ×25 (comparable to that of a powerful hand lens) to about ×250,000, which is nearly 250 times the magnification limit of the best optical microscopes.

The signals produced by an SEM include secondary electrons, backscattered electrons (BSE), transmitted electrons, X-rays, Auger electrons, cathodoluminescence (light), and absorbed specimen current. Each of these signals requires specialized detectors, which are not usually all available on a single instrument. These signals result from interactions between the electron beam and atoms at or near the surface of the sample [2].

In the most common detection mode, secondary electron imaging (SEI), the SEM can produce very high-resolution images of the sample surface, revealing details as small as 1–5 nm. Owing to the image formation process, SEM micrographs exhibit a large depth of field, giving them a characteristic three-dimensional appearance that is particularly useful for understanding surface structures [3].

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Backscattered electrons are primary beam electrons that are reflected from the sample through elastic scattering. BSE imaging is frequently used in analytical SEM, often in conjunction with characteristic X-ray spectra. Because the intensity of the BSE signal is strongly related to the atomic number (Z) of the specimen, BSE images provide information about the distribution of different elements within the sample. For this reason, BSE imaging is especially effective for visualizing colloidal gold immunolabels with diameters of 5 or 10 nm, which are otherwise difficult or impossible to detect using secondary electron imaging in biological specimens [4].
Characteristic X-rays are emitted when the electron beam ejects an inner-shell electron from an atom in the sample, allowing a higher-energy electron to fill the vacancy and release excess energy. These X-rays are used to identify elemental composition and analyze the structure of materials [5].

Materials and Methods

Six samples composed of different materials and conditions were prepared. These included mixtures of aluminum oxide (Al_2O_3) and zirconium dioxide (ZrO_2) powders; metal matrix composites (MMC: Ti–Al–W); silver-reinforced polymer matrix composites; uncoated polymer foam; Au–Pd-coated polymer foam; and alumina nanoparticles subjected to heat treatments at 900 °C, 1,000 °C, and 1,100 °C. Each sample was mounted on a sample holder and placed inside the SEM sample chamber. The instrument was then switched on, and the chamber was maintained under vacuum conditions. Images were acquired at different magnifications, and various signals were detected during the demonstrations. Non-conductive samples were either coated with conductive materials or examined using the variable pressure (VP) technique to minimize charging effects.

Results and Discussion

Types of image in SEM.

Backscattered electron (BSE) imaging is useful for distinguishing elements because the yield of collected backscattered electrons increases monotonically with the atomic number of the specimen. BSE imaging can differentiate elements with atomic number differences of at least three; that is, materials with atomic number differences ≥ 3 will appear with good contrast in the image. This behavior arises from the probability of elastic scattering: materials with higher atomic numbers absorb or stop more incident electrons and therefore exhibit smaller interaction volumes. Consequently, materials with higher atomic numbers (Z) generate more backscattered electrons and appear brighter than materials with lower Z values [6].
Figure 1 illustrates the effect of atomic number on image contrast in metal matrix composites (Ti–Al–W). The atomic numbers of the constituent elements are Al ($Z = 13$), Ti ($Z = 22$), and W ($Z = 74$). As a result, tungsten-rich regions appear the brightest, followed by titanium, while aluminum-rich regions appear darker in the image.
In contrast, topographic contrast is particularly advantageous for materials with a constant average atomic number and for single-phase samples. This mode is commonly applied in the study of fracture and fatigue surfaces, as well as for measuring particle size and fiber diameter in reinforced polymers. Topographic contrast arises from both secondary and backscattered electron signals; however, it is more strongly dependent on the generation of secondary electrons and on the position of the detector relative to the sample surface.

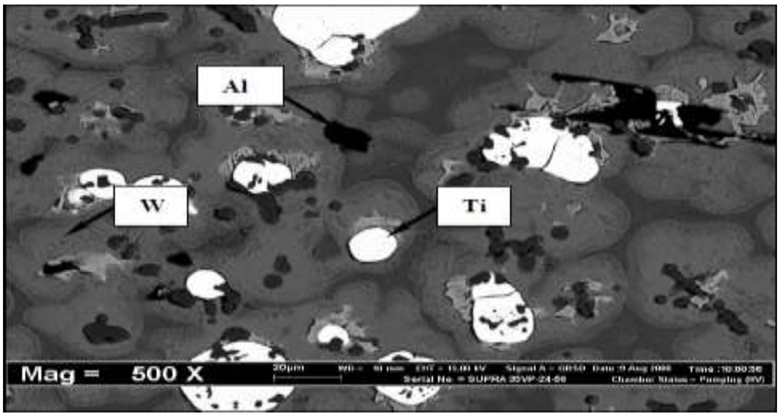

Fig.1: Metal Metric Composites samples showing the effect of atomic number on image illumination

The most common imaging mode collects low-energy (< 50 eV) secondary electrons that are ejected from the outer atomic orbitals of specimen atoms as a result of inelastic scattering interactions with the incident electron beam. Owing to their low energy, these electrons originate from within only a few nanometers of the sample surface. The secondary electrons are detected by an Everhart–Thornley detector, which is a type of scintillator–photomultiplier system. The secondary electrons are first collected by attraction toward an electrically biased grid and then further accelerated toward a positively charged phosphor or scintillator. Upon impact, the accelerated secondary electrons cause the scintillator to emit flashes of light (cathodoluminescence), which are transmitted to a photomultiplier located outside the SEM column via a light pipe and a window in the wall of the sample chamber.

The brightness of the image depends on the number of secondary electrons reaching the detector. When the electron beam enters the sample perpendicular to the surface, the interaction region is symmetrically distributed about the beam axis, allowing a certain number of electrons to escape from within the sample. As the angle of incidence increases, the escape path length on one side of the interaction volume decreases, resulting in the emission of a greater number of secondary electrons. Consequently, steep surfaces and edges appear brighter than flat regions, producing images with a well-defined three-dimensional appearance. Because secondary electrons have a very small escape depth (approximately 1–5 nm), they do not provide compositional contrast; instead, they are primarily sensitive to surface features. Figure 2 shows a mixture of aluminum and zirconium imaged using secondary electron and backscattered electron signals.

The emission of secondary electrons is influenced by several factors: (a) accelerating voltage; (b) surface morphology and the angle at which incident electrons strike a given surface site; (c) surface density, which affects beam penetration and secondary electron absorption; (d) surface chemistry and crystallography; and (e) local surface charge accumulation.

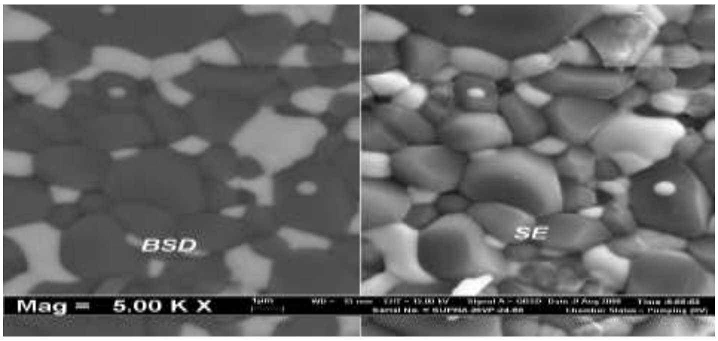

Fig. 2: Image showing the mixture of Aluminum oxide and Zirconium dioxide powders using secondary electron signals (L) and backscattered electron signals(R).

Effect of specimen type

During SEM operation, the specimen is continuously bombarded by the incident electron beam. Over time, these electrons may accumulate on the specimen surface, resulting in a buildup of negative charge that degrades image quality. In addition, surface charging can deflect both incident and emitted electrons, further compromising image accuracy. Therefore, specimens must be electrically conductive so that the deposited charge can be effectively dissipated to electrical ground through the specimen stage. This accumulation of charge on the specimen surface is referred to as *charging*. Charging can produce bright spots in the image that may be misinterpreted as significant sample features, leading to erroneous results.

For metallic samples, surface charging is generally not a concern. However, for high-resolution imaging, it is often advantageous to apply a thin conductive coating to the metallic surface in order to enhance signal stability and image quality [7].

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The metal coating used in SEM must be continuous, electrically conductive, stable, and possess a high secondary electron emission coefficient. Additionally, the coating should exhibit a smooth and fine-grained structure so that it is not resolved during imaging. Gold was originally used as a coating material; however, with continuous improvements in SEM resolution, gold–palladium (Au–Pd) alloys became preferred due to their finer grain size. Gold and palladium are known for their chemical stability and biocompatibility and have been used for several decades in dentistry and medicine [9]. Studies have shown that gold complexes exhibit activity against both Gram-positive and Gram-negative microorganisms, similar to antibiotics such as piperacillin and chloramphenicol. Palladium, in its ionic form and at sufficiently high concentrations, can exhibit toxic and allergic effects; however, in its metallic state, these risks are minimal due to its very low dissolution rate.

Non-conducting materials, such as ceramics and polymers, are particularly susceptible to surface charging. In addition, prolonged exposure of non-conductive materials to the electron beam may lead to structural degradation. Applying a thin metallic coating to the surface of insulating materials can effectively prevent charging and also assist in dissipating heat during imaging. In the case of relatively thick coatings, enhanced secondary electron emission from the sample has been observed [10]. Figure 3(A) shows an image of uncoated polymer foam, while Figure 3(B) presents the image of coated polymer foam. Notably, more surface details are visible in the coated sample due to improved image contrast. The polymer foam exhibits a porous structure, which contributes to its low density and lightweight characteristics.

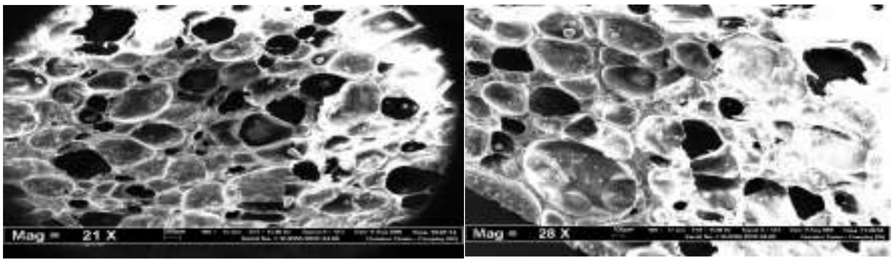

Fig. 3: (A) Showing the effect of surface charging on uncoated Polymer foam. Taken at 28X magnification. (B) Showing uncoated Polymer foam with high illuminate.

Effect of increasing magnification

Magnification in SEM can be controlled over a range of approximately five orders of magnitude, from about ×25 or less to ×250,000 or more. Unlike optical and transmission electron microscopes, image magnification in SEM is not determined by the power of the objective lens. Although SEMs are equipped with condenser and objective lenses, their primary function is to focus the electron beam to a fine spot rather than to form a magnified image of the specimen [11]. In principle, provided that the electron gun can generate a sufficiently small beam diameter, an SEM could operate without condenser or objective lenses; however, such a configuration would be less versatile and would not achieve very high resolution.

In SEM, as in scanning probe microscopy, magnification is determined by the ratio between the dimensions of the raster scanned on the specimen and the corresponding raster displayed on the viewing screen. Assuming a fixed display size, higher magnification is achieved by reducing the size of the raster on the specimen, while lower magnification corresponds to a larger raster area. Consequently, magnification is controlled by the current supplied to the X–Y scanning coils rather than by the objective lens power.

Increasing magnification during imaging allows for improved observation of surface features. At higher magnifications, a smaller area of the specimen is scanned, enabling the visualization of finer surface details [12]. As shown in Figure 4(A) and (B), features such as surface failure and particle pore size can be clearly identified at higher magnifications.

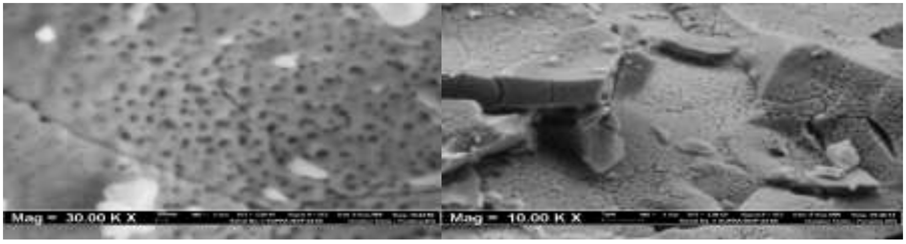

Fig. 4: (A) Showing surface of Alumina nanoparticles at 10,000X magnification. (B) Showing pores of Alumina nanoparticles at 30,000X magnification.

Compositional analysis via Energy Dispersive X-ray Spectrometry (EDX)

Energy dispersive X-ray spectroscopy (EDS, EDX, or EDXRF) is an analytical technique used for elemental analysis and chemical characterization of samples, providing both weight percentage and atomic percentage data, as shown in Figure 5(A) and (B). EDX is one of the common attachments to SEM that enables quantitative compositional analysis. Its operating principle is similar to that of wavelength-dispersive X-ray fluorescence spectrometry (WDX); however, instead of using an external X-ray source, the primary electron beam employed for SEM imaging is used to excite the specimen. This interaction results in the emission of characteristic X-rays with distinct and quantifiable energies, allowing for elemental identification and quantification [13].

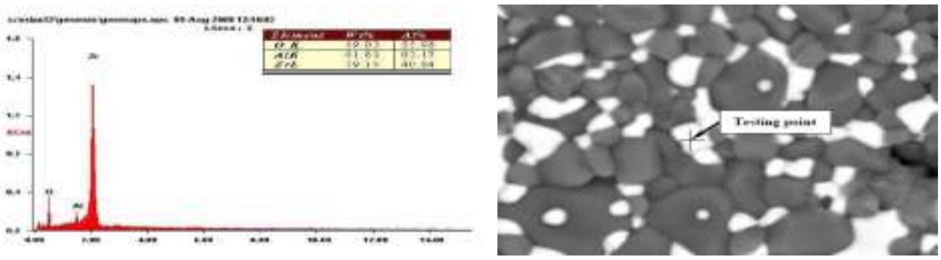

Fig. 5: Showing (A) The mixture of Aluminum oxide and Zirconium dioxide powders under testing by EDX technique, (B) The value of composition by EDX technique. (Testing by fixed point)

An EDX system consists of four primary components: the electron beam source, the X-ray detector, the pulse processor, and the analyzer. Although standalone EDX systems are available, EDX is most commonly integrated with scanning electron microscopes (SEM–EDX) and electron microprobes. In SEM–EDX systems, the scanning electron microscope is equipped with an electron source (cathode) and magnetic lenses to generate and focus the electron beam onto the specimen.

The X-ray detector converts the energy of emitted characteristic X-rays into electrical voltage signals. These signals are transmitted to a pulse processor, which measures and processes them before forwarding the data to an analyzer for display and interpretation. As illustrated in Figure 6(A) and (B), EDX is used for elemental analysis or chemical characterization of a specimen within a localized or limited area [14].

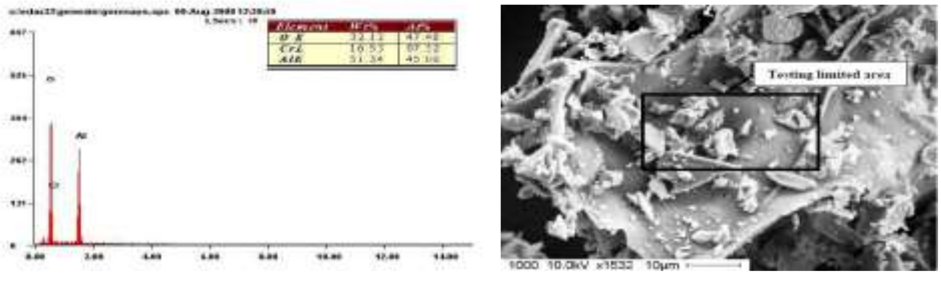

Fig. 6: Showing (A) The mixture of Alumina and Chromium nanoparticles under testing by EDX technique, (B) the value of composition by EDX technique.(Testing by limited area)

The observed microstructures in term of their properties.

Material characterization can be achieved using various techniques, depending on the type of property being investigated. SEM is primarily used to identify and analyze physical and morphological characteristics of materials. In contrast, EDX is used for elemental analysis and is typically applied to conductive or metallized samples, as shown in Figure 7(A) and (B).

The mixture of Aluminum oxide and Zirconium dioxide powders.

Aluminum oxide (alumina) is an amphoteric oxide of aluminum with the chemical formula Al_2O_3. It is commonly referred to as alumina and is widely used as an abrasive due to its high hardness and as a refractory material because of its high melting point. Aluminum oxide is an electrical insulator but exhibits relatively high thermal conductivity. Its combination of hardness and thermal stability makes it particularly suitable for abrasive and high-temperature applications [15].

Zirconium dioxide (ZrO_2), commonly known as zirconia, is highly valuable in its stabilized form. It exhibits excellent resistance to corrosion and high-temperature degradation and is widely used to enhance the quality and durability of coatings and advanced ceramic materials [16].

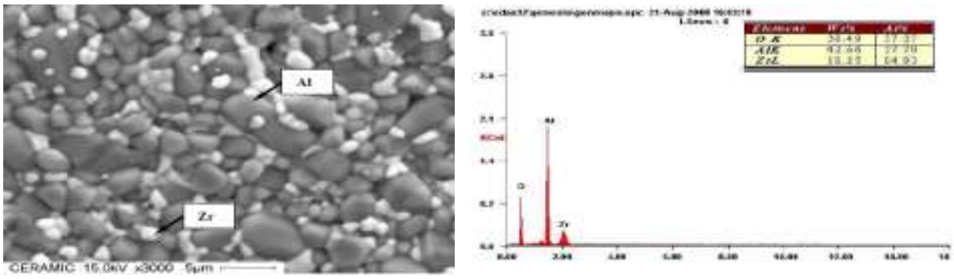

Fig. 7: Aluminum oxide and Zirconium dioxide powders (A) Taken by SEM, (B) analyzes by EDX.

Metal Matrix Composites (Ti-Al-W)

Aluminum is a soft, lightweight, durable, and malleable metal with good thermal and electrical conductivity. It has approximately one-third the density and stiffness of steel and is highly ductile, making it easily machined, cast, and extruded. Its corrosion resistance is enhanced by the formation of a thin surface layer of aluminum oxide.

Titanium is a light, strong, lustrous, and corrosion-resistant metal, including resistance to seawater and chlorine. It has a low density and a high strength-to-weight ratio. High-purity titanium has traditionally been produced in small quantities due to the complexity of its extraction and processing.

Tungsten is a hard, dense, and durable metal that maintains its hardness at high temperatures and has an exceptionally high melting point. Elemental tungsten is commonly used in high-temperature applications. Its high melting point makes it suitable for aerospace, electrical, heating, and welding applications, notably in the gas tungsten arc welding (GTAW) process [17].

As shown in Figure 8(A) and (B), tungsten and titanium are incorporated into aluminum matrices to enhance hardness, strength, elongation, corrosion resistance, and thermal stability.

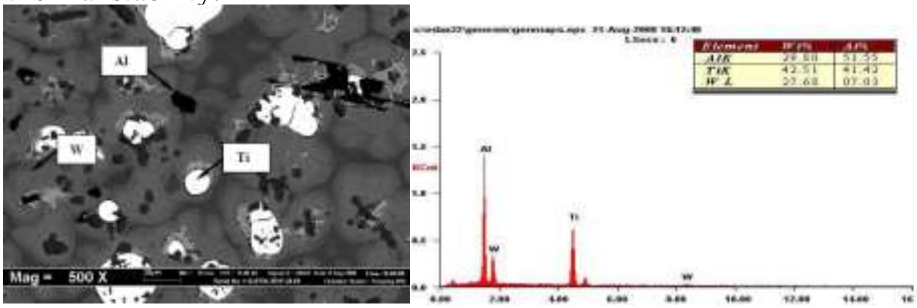

Fig. 8: Metal Matrix Composites (Ti-Al-W) (A) Taken by SEM & (B) analyzed by EDX

Silver Polymer Matrix Composite (PMC: Ag)

Silver is a highly ductile and malleable metal. It can be used to reinforce polymer matrix composites, as shown in Figure 9. For effective reinforcement, silver particles must be evenly distributed throughout the epoxy and form a strong adhesive bond with the polymer matrix. This uniform dispersion and strong interfacial bonding enhance the tensile strength and hardness of the composite material.

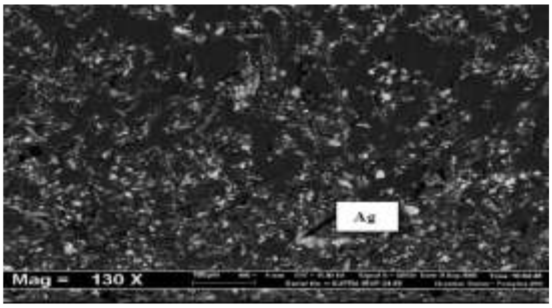

Fig. 9: Silver Polymer Matrix Composites.

Polymer foam.

The polymer foam has porous structure that may lead to light weight, low density also poor mechanical properties as shown in fig. 10.

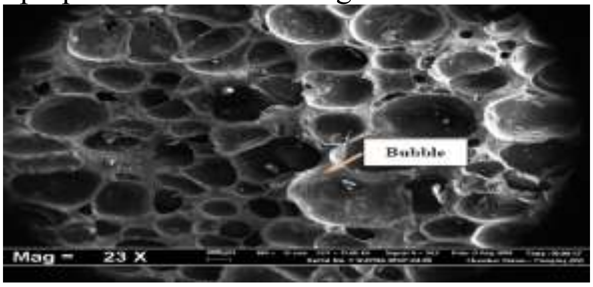

Fig. 10: Polymer foam.

Conclusion

Image formation in SEM is dependent on various factors. Some of these are the type of signal to be used, the composition of the material, and the electrical conductivity of materials. Backscattered electrons would appear with good contrast in the image. It uses for identify various materials in the components. Secondary electrons will be emitted. Thus, steep surfaces and edges tend to be brighter than flat surfaces, which results in images with a well-defined, three-dimensional appearance. It is used for structural analysis, failure analysis, surface characteristic and grain orientation. Quality of the image can be improved by polishing the sample surface, especially for metallic surfaces, and by coating with a conductive material or maintain samples chamber with the variable pressure (VP) technique to prevent charging for insulators like polymers and ceramics. Increasing the magnification leads to a smaller area of focus. However, finer details of the specimen surface can be resolved. EDX can be used for elemental analysis or chemical characterization of samples.

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