Indian Streams Research Journal
ISSN 2230-7850
Impact Factor : 2.1506(UIF)
Volume-4 | Issue-10 | Nov-2014
Available online at www.lbp.world

APPLICATION OF SCANNING ELECTRON MICROSCOPY: A REVIEW

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ABSTRACT:

Scanning electron microscopy (SEM) is used to determine the morphological structure of a material or the oxidation process of a material. This paper reviews the morphology of zirconium in order to obtain accurate and detailed information as necessary to support the analysis of corrosion resistance of a material or zirconium material due to high temperature in the oxidation process using various methods in SEM analysis. In addition, this paper reviews the morphological composition of sugar factory waste and analyzes the relationship between volume fraction and weight fraction by observing the results of SEM on the composites.

KEYWORDS: Scanning Electron Microscopy, Morphological Structure, Material, Analysis, Composite.

1. INTRODUCTION

Scanning electron microscopy (SEM) is an electron microscope designed to directly observe the surface of solid objects. SEM has a magnification of 10 - 3,000,000, a depth of field of 4 - 0.4 mm and a resolution of 1 - 10 nm. The combination of high magnification, large depth of field, good resolution, ability to determine structure, and crystallographic information makes SEM widely used for research and industrial purposes. SEM focuses a beam of electrons on the surface of an object and captures images by detecting the electrons emitted from the surface of the object (Akhtar, K., Khan, S. A., Khan, S. B., & Asiri, A. M., 2018).

Conventional characterization techniques based on wavelengths of 650 nm and above, such as optical microscopy in metallographic analysis, do not have sufficient resolution to obtain the desired scientific information. We need other detection and characterization methods that can provide high resolution to provide visual aids to researchers to observe in detail what is happening in and around the interface or even in situ between the material and the oxide layer. Scanning Electron Microscopy (SEM) was considered as an accepted technique approved and recognized by the physical science community worldwide, for this reason, marked by the Nobel Prize (Bishop, J. M., 2004).

Identifying the microstructure of the oxide layer using SEM requires not only imaging but also appropriate techniques and procedures considering the imaging process. A physical process that is a corpuscular interaction between source electrons and atoms in a material. Although the resulting data signal is quite strong compared to optical microscopy because the observed objects are often relatively

small and contain non-conductive elements such as a passivation layer of oxide on the surface, SEM can provide relatively low contrast, especially at high magnification. Therefore, the SEM must be operated with electron parameter settings such as high voltage, spot size, bias and beam current as well as optical parameters such as contrast, precise focus and astigmatism to achieve scientifically optimal image results and not provide multiple interpretations. Additionally, the process of image capture and chemical analysis by SEM depends on the type of sample and how it is handled and the preparation technique as well as the operator's functional ability (Lyman, C. E., Newbury, D. E., Goldstein, J., Williams, D. B., Romig Jr., A. D., Armstrong, J., & Peters, K. R., 2012).

This paper reviews the results of the analysis and characterization of high temperature oxidation test specimens, using SEM, of a zirconium alloy developed for the manufacture of nuclear fuel cladding. This analysis is mainly intended to gain information about the extent to which SEM can answer the phenomena that occur in the oxidation process. In addition, researchers have been motivated to find and investigate the crystal structure of sugarcane waste based on many studies on the use of sugar mill waste as an energy source, absorbent and basic material for the production of many products. Scanning electron microscopy (SEM) and its effects on the environment.

2. LITERATURE REVIEW

2.1 SEM Working Principles

An SEM consists of an electron gun that produces an electron beam at an accelerating voltage of 2 - 30 kV. The electron beam passes through several electromagnetic lenses to form a 10 nm image on a sample displayed on photographic film or a display tube. A schematic diagram and a description of how SEM works are described below.

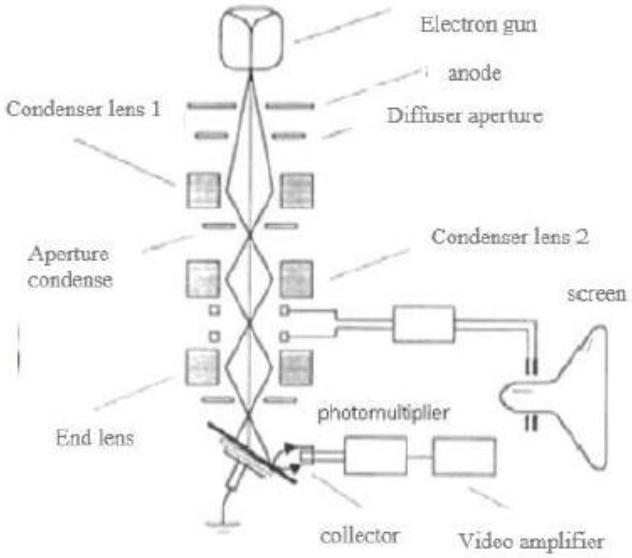

Figure 1. Schematic Diagram of Scanning Electron Microscopy (SEM) Working Principle (Anggraeni, N. D., 2008)

SEM is particularly suitable for use in situations where rough surfaces must be observed with magnifications of 20 times to 500,000 times. Before passing through the last electromagnetic lens, a scanning raster defines the electron beam to scan the sample surface. The scan results are synchronized with the cathode ray tube and the sample image will appear in the scanned area. Contrast levels seen on

a cathode ray tube result from different reflection effects in the sample (Vladár, A. E., & Postek, M. T., 2009).

When the electron beam hits the surface of the sample, some electrons are reflected as backscattered electrons (BSE) and others release low energy secondary electrons (SE). Electromagnetic radiation emission from a sample occurs at various wavelengths, but basically, the wavelengths that are most interesting to use are the visible light wavelength region (cathodoluminescence) and X-rays (Malik, T. A., 2020).

BSE and SE electrons reflected and emitted by the sample are collected by a scintillator that emits a pulse of light at the incoming electrons. The emitted light is converted into an electrical signal and amplified by a photomultiplier. After going through the amplification process, the signal is sent to the grid of the cathode ray tube (Inkson, B. J., 2016).

A scintillator usually has a positive potential of 5 - 10 kV to accelerate the low energy emitted by the electrons to release visible light upon striking the scintillator. It is necessary to protect the scintillator from the deflection of the main electron beam of high potential. A metal shield with open metal gauze facing the sample allows almost all electrons to pass through the scintillator surface (JIRÁK, J., Nedela, V., Černoch, P., Čudek, P., & Runštuk, J., 2010).

When a material is observed, the position of the surface of the object imaged by the electron beam of highest intensity is scanned over the entire surface of the observed material. By using the reflection of these objects, information can be detected using an image processing program on a computer.

2.2 Scanning Electron Microscopy Block Diagram

Figure 2 shows a schematic diagram of a standard SEM JSM-6510LA from the manufacturer JEOL used in this study with chemical composition analysis facilities in the form of an X-ray detector. Two pairs of scan coils are then scanned over the sample surface with variable frequency. The smaller the beam, the greater the lateral resolution achieved. The physical error in the electromagnetic lens in the form of astigmatism is corrected by a stigmatator device. SEM does not have a correction system for other distortion errors (Sujatno, A., Salam, R., Bandariana, B., & Dimyati, A., 2015).

The second is an electron source, usually in the form of a filament made of tungsten wire or a needle of Lanthanum Hexaboride LaB6 or Cerium Hexaboride CeB6 alloy, which can provide an electron beam that theoretically has a single energy (monochromatic). The third is the imaging detector, which works to convert the electron signal. Under the type of electron, this SEM has two types of detectors, namely SE detector and BSE detector (Ul-Hamid, A., 2018).

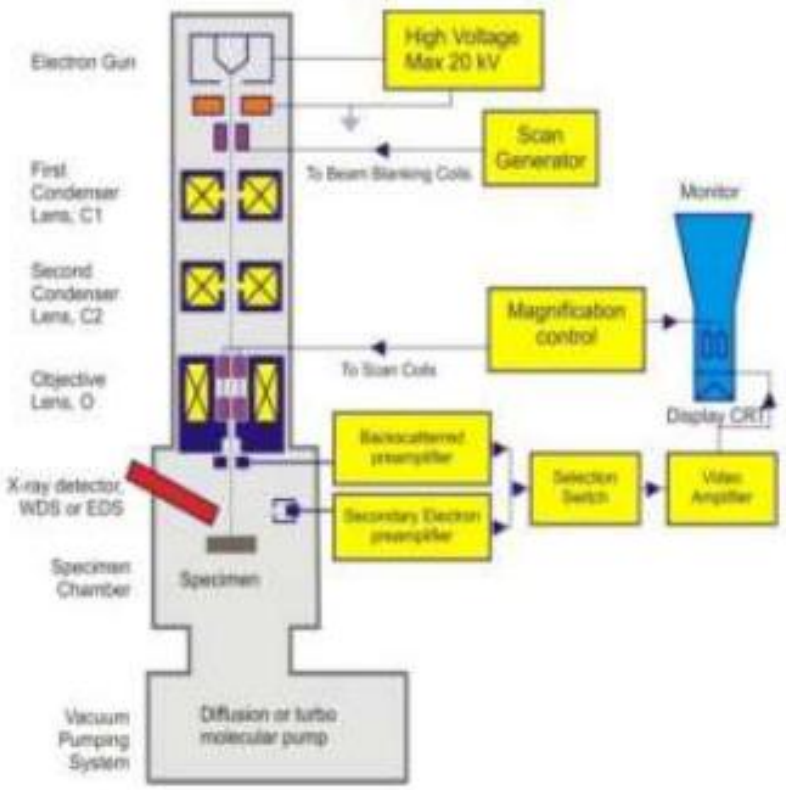

Figure 2. Scanning Electron Microscopy Block Diagram
(Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

To avoid interference of air molecules against the electron beam, the entire electron path (column) is vacuumed to 10-6 Torr. However, high vacuum increases the sensitivity of the instrument to detect non-conductivity, making it difficult to analyze non-conductive materials such as ceramics and oxides. To overcome this, the SEM has an option to operate with low vacuum, called low vacuum mode. With low vacuum techniques, we can also analyze non-conductive materials. The pressure in this mode ranges from 30 to 70 Pa (Danilatos, G., Rattenberger, J., & Dracopoulos, V., 2011).

2.3 Interaction between Materials and Electron

When an electron beam is scanned across the surface of a sample, the electrons interact with atoms on the surface and below the sample surface. As shown in Figure 3, due to this interaction most of the electron beam manages to re-emerge, these electrons are called backscattered electrons (BSE), a small fraction of the electrons enter the material and then transfer most of the energy to the atomic electrons. so that they are lifted from the surface of the material, i.e. secondary electrons (SE). Secondary electrons are always produced after the appearance of characteristic X-rays for each element so that it can be used to measure the content of the element present in the material under study.

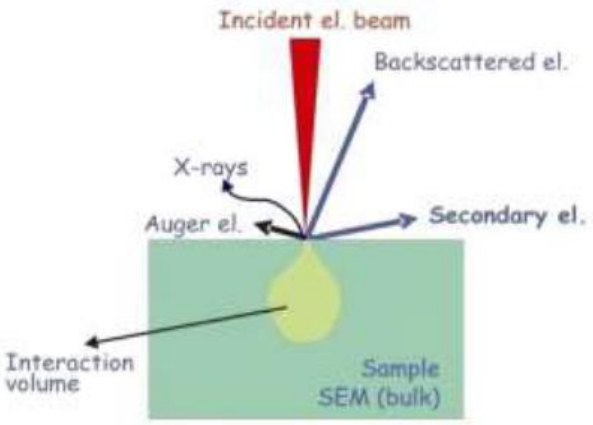

Figure 3. Interaction scheme between materials and electrons in SEM (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

The BSE formation process takes place in the atoms deep in the surface of the sample. This is caused by the collision of electrons between the source and the atomic nucleus as shown in Figure 4. Since the mass of the protons that make up the nucleus is 2000 times greater than that of the electrons, each collision will cause most of the electrons to bounce in 180o directions. That is, some will reflect in the direction from which they came, i.e. outside the surface of the material. These BSE electrons carry information about the atoms they hit and their bonds in phases. So that within certain limits the intensity in the image formed from BSE electrons can be seen as phase contrast.

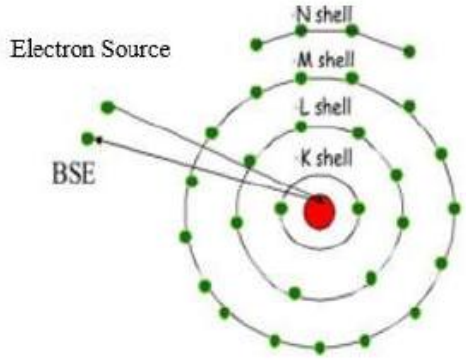

Figure 4. The Process of Backscattered Electron (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

If the source electrons passing through matter are only passing through an electron cloud or atomic orbital, they may transfer some of their kinetic energy to one or more electrons in that orbital. The electrons will become unstable and excited so that they leave their positions and come out of the surface of the material, so these electrons are known as secondary electrons (SE) or secondary electrons, Figure 5. Since electrons have low energy, only electrons that are on or near the surface of the material will be ejected can with the help of a special SE, an electron detector can be used to create a fine contour image of the material's surface. Surface structure and its characteristics, such as grain boundaries, edges, porosity.

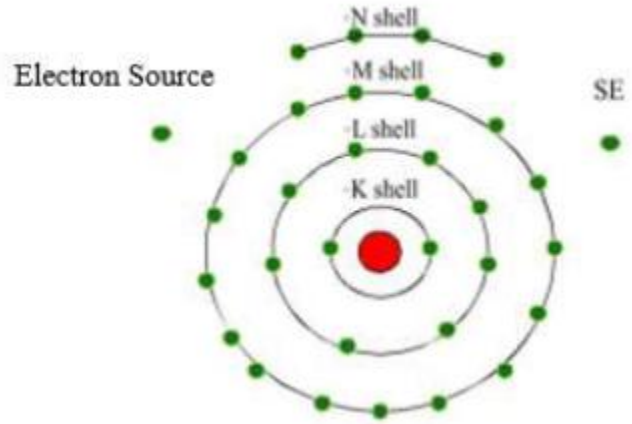

Figure 5. Secondary Electron Process
(Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

3. DISCUSSION

3.1 Morphological of zirconium

Figure 6 shows that there is an oxide layer on the surface which becomes a protective layer that determines the characteristics of oxidation. Strong growth shows up and down or wave graphs. This is because during the oxidation process, oxide layer growth and local flaking occurs. To support the oxidation analysis, SEM tests were performed to observe the microstructure of the oxide layer and reveal the processes that occurred during oxidation. Therefore, this SEM test is directed to observe the formation of oxide layer on the surface of the alloy sample.

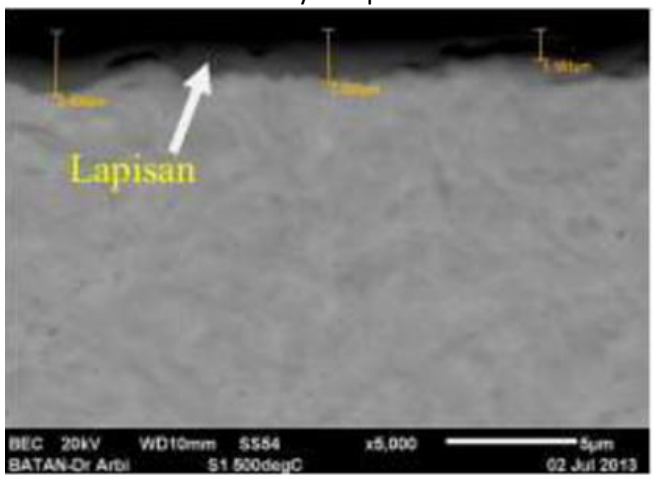

Figure 6. Micro Matrix Structure and Oxidation Layer of Zirconium Alloy
(Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

3.2 Characterization of SEM Morphological Structure of Sugar Factory Waste

Figure 7 shows a cross-section of an alloy sample after oxidation at 500 0C taken with a BSE detector. As discussed in the image formation theory section above, there is a clear distinction between the surface matrix and the oxide layer. The upper part, which is assumed to be a temporary oxide layer shows a darker imprint, while the matrix below is lighter. BSE signal formation analysis shows that the matrix portion has a higher density or a phase with a heavier atomic arrangement than the upper layer.

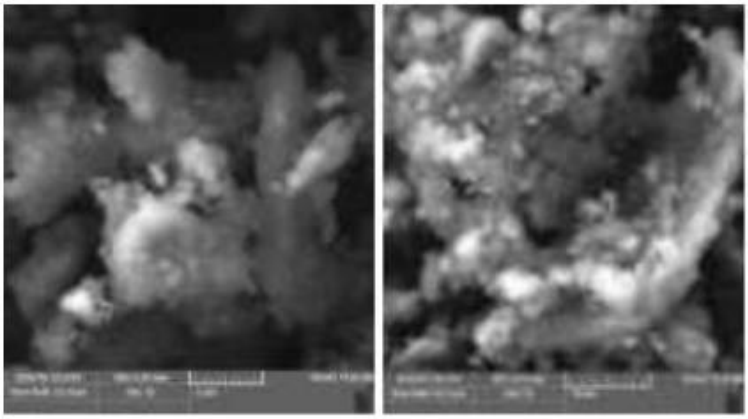

Figure 7. (a) Morphological Structure of Sugar Factory Waste on a 5 µm Scale Bar and (b) Factory Waste at a 10 µm Scale Bar (Hamriani, H., 2016)

In Figure 7, SEM image measurements on waste samples are performed with an SE (Secondary Electron) detector at an accelerating potential (HV) of 20.0 kV even with different fields of view and scale bars. It can be seen that the morphological condition of the sample shows irregular granules with different sizes. The brighter colors that appear more dominant in the sample are the elements with higher atomic numbers, while the darker colors that appear on the surface of the sample are the elements with lower atomic numbers.

3.3 Composite Surface Morphology

Figure 8 shows the morphology and microstructure of the composites with activated filler carbon. As a whole, carbon particles bind together to form fibers. This is most clearly seen in the 3% weight fraction where there is a long line of fibers formed.

The microstructure of activated carbon composites with a weight fraction of 1% indicates that the constituent elements, i.e. carbon, are less bound to each other in terms of distribution. The distribution of carbon particles throughout the matrix area is uneven. In other words, it only makes short fibers. The longest fiber formation value is 20.4 µm and the shortest is 12.8 µm.

At a weight fraction of 3%, the distribution of carbon particles can be uniformly distributed over the entire matrix area. Carbon particles can bond together so that they form rows to form long fibers. The longest fiber formation value is 96.2 µm and the shortest is 26.4 µm.

At 1% by weight fraction, above 6% by weight fraction, the carbon particles are less bound to each other. so that they make only short fibers. However, the results obtained in 6% weight fraction were better than 1% weight fraction. The longest fiber formation value is 40.6 µm and the shortest is 16.8 µm.

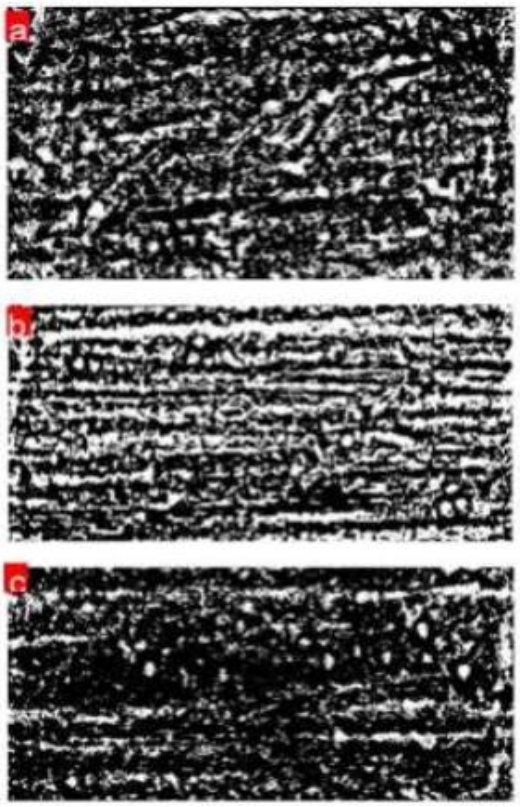

Figure 8. SEM observations of the Formation of Carbon Black Structure on Composites with activated carbon filler, (a) 1% weight fraction, (b) 3% weight fraction, (c) 6% weight fraction (Farikhin, F., Joko Sedyono, S. T., & Eng, M., 2016)

4. CONCLUSION

In summary, SEM testing produces microstructure images and chemical elemental composition in alloy materials. The oxide layer formed by the oxidation of zirconium alloys can also be clearly detected using a BSE detector. EDS test results can be used to predict the phase that occurs after oxidation and support the analysis results of alloy material characteristics and oxidation resistance. The morphology of the composites with activated carbon filler shows that the carbon particles bind to each other, forming fibers. This effect is most clearly seen in the 3% weight fraction.

Whereas in composites with inert carbon filler, the carbon particles form clusters of particles. This effect is most clearly seen in the 6% weight fraction.

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