International Journal of Applied Science and Engineering Review

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

APPLICATION OF SCANNING ELECTRON MICROSCOPY: A REVIEW

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ABSTRACT

The application of the scanning electron microscopy (SEM) is used to determine the morphological structure of a substance or oxidation process of a substance. This paper reviews morphological of zirconium to obtain accurate and detailed information due to it is needed to support the analysis of corrosion resistance of a substance or zirconium material caused by high temperatures in the oxidation process using various methods in SEM analysis. Besides that, this paper reviews morphological structure at sugar factory waste and analyses the relationship between volume fraction and weight fraction by observing the results of SEM on the composite.

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

1. INTRODUCTION

Scanning Electron Microscopy (SEM) is an electron microscope designed to observe the surface of solid objects directly. SEM has a magnification of 10 - 3,000,000 times, 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, the ability to know the composition and crystallographic information makes SEM widely used for research and industrial purposes. SEM focuses a beam of electrons on the surface of the object and takes image by detecting electrons that emerge 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 expected scientific information. We need other methods of identification and characterization that can provide higher resolution to give the researchers visual assistance to be able to observe what is happening in and around the interface in detail or even in situ between the material and the oxide layer. Scanning Electron Microscopy (SEM) was understood as an acceptable technique approved and recognized by the worldwide material science community, for this reason, marked by the award of the Nobel Prize (Bishop, J. M., 2004).

Identification of the microstructure of the oxide layer using SEM is not just taking an image but must be done with the correct technique and operating method considering the image formation process. Physical

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

process which is a corpuscular interaction between the source electrons and the atoms in the material. Even though the resulting data signal is quite strong compared to optical microscopy because often the observed objects are relatively small and contain non-conductive components, such as a passivation layer of oxide on the surface, SEM can provide relatively low contrast, especially at high magnifications. Therefore, 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 in order to obtain scientifically optimal image results and do not provide multiple interpretations. In addition, the process of image capture and chemical analysis by SEM is strongly influenced by the type of sample and how to handle it and the preparation technique as well as the operational capability of the operator (Lyman, C. E., Newbury, D. E., Goldstein, J., Williams, D. B., Romig Jr, A. D., Armstrong, J., ... & Peters, K. R., 2012).

This paper review the results of the analysis and characterization of oxidation test samples at high temperatures using SEM, a zirconium alloy material developed for the manufacture of nuclear fuel cladding. This analysis is mainly intended to obtain information about the extent to which SEM can answer what phenomena occur in the oxidation process. Besides, to find out the morphological structure based on several studies on the use of sugar factory waste as an energy source, adsorbent and basic material for the manufacture of several products, researchers were inspired to find out and investigate the crystal structure of sugarcane waste using scanning electron microscopy (SEM) and its effects on the environment.

2. LITERATURE REVIEW

2.1 SEM Working Principle

SEM consists of an electron gun that produces an electron beam at an accelerated voltage of 2 - 30 kV. The electron beam is passed through several electromagnetic lenses to produce a 10 nm image on the sample displayed on photographic film or onto a display tube. The schematic diagram and how SEM works is described as follows.

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

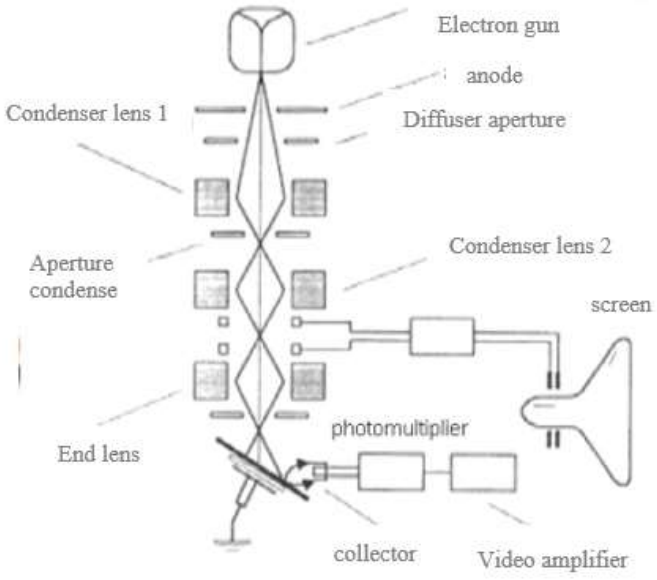

Figure 1. Schematic Diagram of Scanning Electron Microscopy (SEM) Working Principle (Anggraeni, N. D., 2008)

SEM is particularly suitable for use in situations requiring observation of rough surfaces with magnifications ranging from 20 times to 500,000 times. Before going through the last electromagnetic lens, the scanning raster defines an 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. The contrast level seen on the cathode ray tube arises because of the different reflection results from 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 liberate low energy secondary electrons (SE). The electromagnetic radiation emission from the sample occurs at various wavelengths, but basically, the wavelengths that are more interesting to use are the visible light wavelength regions (cathodoluminescence) and X-rays (Malik, T. A., 2020).

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

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

The scintillator usually has a positive potential of 5 - 10 kV to accelerate the low energy emitted by electrons enough to emit visible light when hitting the scintillator. The scintillator must be protected against deflection of the main electron beam which has a high potential. The metal shield containing an open metal gauze facing the sample allows nearly all of the electrons to pass through the scintillator surface (JIRÁK, J., Neděla, V., Černoch, P., Čudek, P., & Runštuk, J., 2010).

When an observation is made of the material, the location of the surface of the object being shot with the electron beam of the highest intensity is scanned over the entire surface of the material being observed. By utilizing the reflection of these objects, information can be found using an image processing program that is on the computer.

2.2 Scanning Electron Microscopy Block Diagram

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

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

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

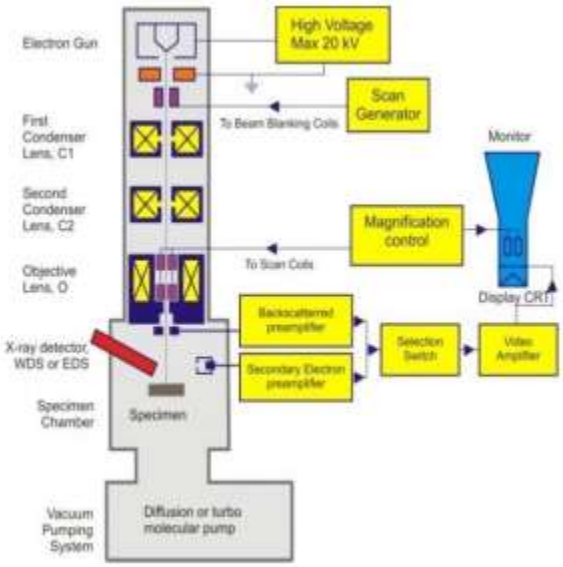

Figure 2. Scanning Electron Microscopy Block Diagram (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

To avoid disturbance of the air molecules against the electron beam, the entire electron path (column) is vacuumed up to 10-6 torr. However, the high vacuum causes increased sensitivity of the device's detection to non-conductivity, which makes it difficult to analyze non-conductive materials, such as ceramics and oxides. To overcome this, SEM has the option to operate with a low vacuum, which is called Low Vacuum Mode. With the low vacuum technique, we can even 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 the electron beam is scanned on the surface of the sample, there is an interaction of the electrons 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 come out again, these electrons are called Backscattered Electrons (BSE), a small portion of the electrons enter the material then transfer most of the energy on the atomic electrons so that they are bounced off the surface of the material, namely Secondary Electrons (SE). The formation of secondary electrons is always followed by the appearance of a characteristic X-ray for each element so that it can be used to measure the content of the element present in the material under study.

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

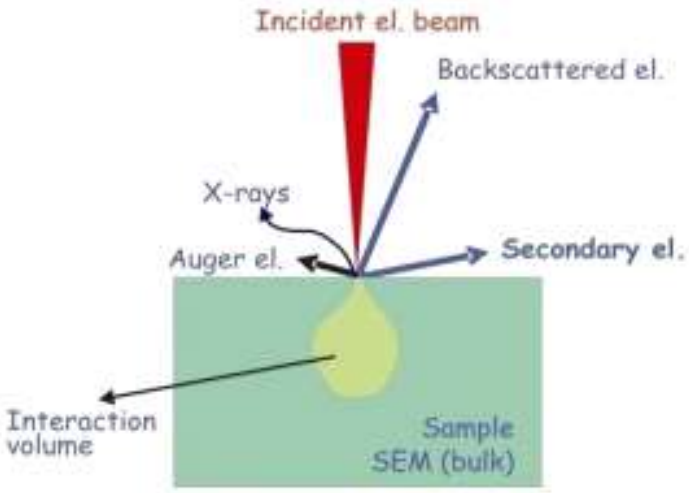

Figure 3. Interaction scheme between materials and electrons in SEM (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

The process of BSE formation occurs in atoms on the deeper part of the sample surface. This is due to the collision between the electrons from the source and the atomic nucleus, as shown in Figure 4. Since the mass of the protons that make up the nucleus is up to 2000 times larger than the electrons, each collision will cause most of the electrons to bounce towards 180o. That is, some will be reflected back in the direction in which they came, i.e. outside the surface of the material. These BSE electrons carry information about the atoms they strike and their bonds in phase. So that the contrast in the image formed from BSE electrons within certain limits can be viewed as phase contrast.

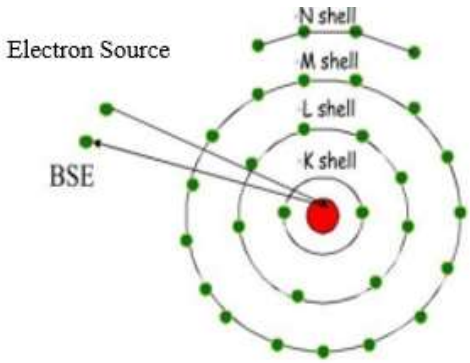

Figure 4. The Process of Backscattered Electron (Sujatno, A., Salam, R., Bandriyana, B., &

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

Dimyati, A., 2015)

If the source electrons on their way through the material pass only through the electron cloud or the orbitals of an atom, they may transfer some of their kinetic energy to one or more electrons in that orbit. The electrons will become unstable and in an excited state so that they leave their position 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 are only at or very close to the surface of the material that can escape. With the help of a special SE, electron detector can be used to form a good surface morphological image of the material. The surface structure and its characteristics, such as grain boundaries, edges, porosity.

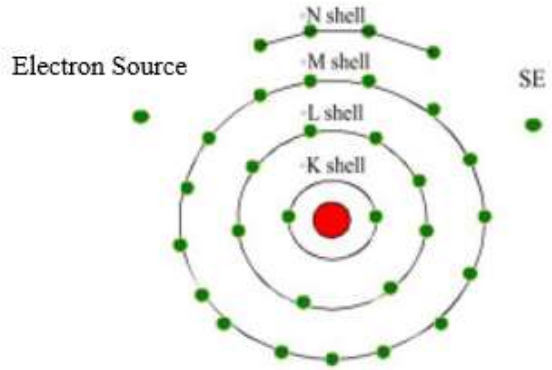

Figure 5. Secondary Electron Process (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

3. DISCUSSION

3.1 Morphological of zirconium

Figure 6 shows that there has been an oxide layer on the surface which has become a protective layer that determines the characteristics of the oxidation. Heavy growth shows an up and down or wavy graph. This is because during the oxidation process, there is growth and local flaking of the oxide layer. To support the oxidation analysis, SEM tests were carried out to observe the microstructure of the oxide layer and possibly reveal the processes that occurred during oxidation. Therefore, this SEM test is directed to observe the formation of an oxide layer on the surface of the alloy sample.

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

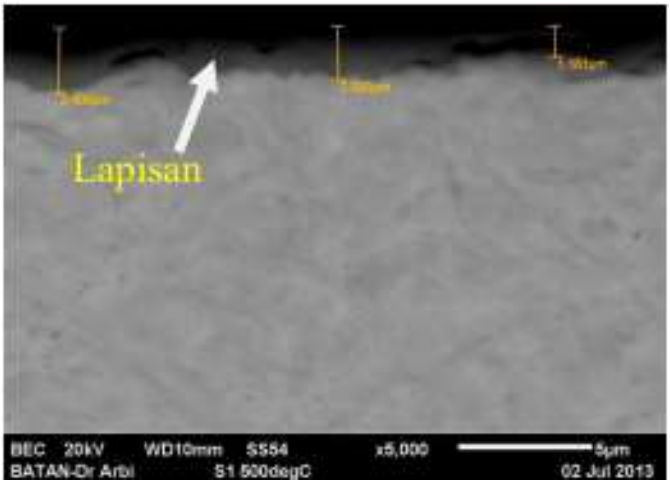

Figure 6. Micro Matrix Structure and Oxidation Layer of Zirconium Alloy (Sujatno, A., Salam, R., Bandriyana, B., & Dimyati, A., 2015)

3.2 Characterization of SEM Morphological Structure of Sugar Factory Waste

Figure 7 shows a cross-section of the alloy sample after oxidation at a temperature of 500 0C taken with a BSE detector. As discussed in the image formation theory section above, there is a clear contrast between the matrix and the oxide layer on the surface. The upper part, which is temporarily assumed to be an oxide layer shows a dark impression, while the matrix below is light. The BSE signal formation analysis indicates that the matrix part has a greater density or is a phase with a heavier atomic arrangement than the top layer.

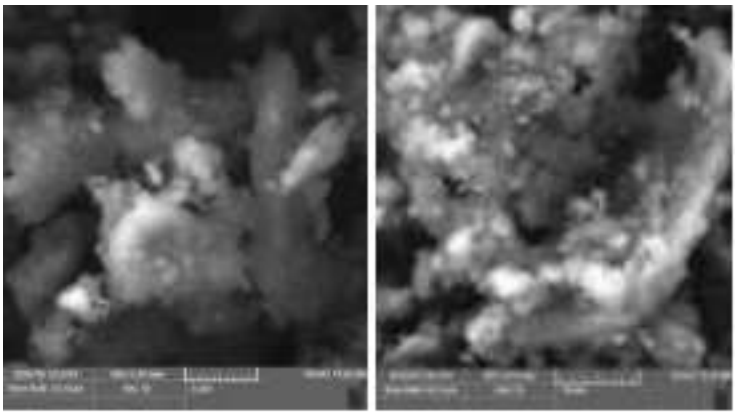

Figure 7. (a) Morphological Structure of Sugar Factory Waste on a 5 µm Scale Bar and (b)

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

Factory Waste at a 10 µm Scale Bar (Hamriani, H., 2016)

In Figure 7, SEM image measurements from waste samples are carried out at an accelerator potential (HV) of 20.0 kV with a SE (Secondary Electron) detector even though with a different view field and scale bar. It can be seen that the morphological condition of the sample shows irregular granules with varying sizes. Bright colours that appear more dominant in the sample are constituent elements that have a high atomic number, while dark colours that appear on the surface of the sample are constituent elements that have a low atomic number.

3.3 Composite Surface Morphology

Figure 8 shows the morphology and microstructure of composites with active filler carbons. As a whole, the carbon particles bind to each other leading to the formation of fibers. This is most clearly seen in the 3% weight fraction where the fibers formed have a long line.

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

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

Similar to 1% by weight fraction, at 6% by weight fraction, the carbon particles are less binding to each other. So that it only forms short fibers. However, the results obtained in the weight fraction 6% were better than the 1% weight fraction. The longest fiber formation has a value of 40.6 µm and the shortest 16.8 µm.

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

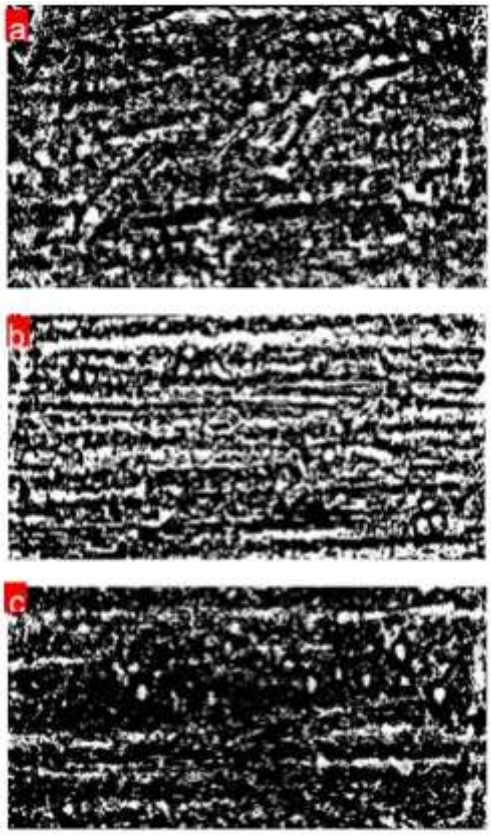

Figure 8. SEM observations of the Formation of Carbon Black Structure on Composites with activated carbon filler, (a) 1% weight fraction, (b) 3% weight fraction, (c) 6% weight fraction (Farikhin, F., Joko Sedyono, S. T., & Eng, M., 2016)

4. CONCLUSION

In summary, SEM testing produces microstructure images and chemical element composition in the alloy material. The oxide layer resulting from the oxidation of zirconium alloys can also be detected clearly using a BSE detector. The EDS test results can be used to predict the phase that occurs after oxidation and can support the results of the analysis of the characteristics and oxidation resistance of the alloy material. The morphology of the composite with activated carbon filler shows that the carbon particles bind to one another, leading to the formation of fibers. This result is most clearly seen in the weight fraction of 3%.

ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

Whereas in composites with inactive carbon filler, carbon particles form particle clumps. This result is most clearly seen in the weight fraction of 6%.

ACKNOWLEDGEMENT

Thank you for Lembaga Penelitian dan Pengabdian kepada Masyarakat (LPPM) Universitas Nasional and my colleagues at Faculty of Engineering and Science, Universitas Nasional, Jakarta, Indonesia.

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ISSN: 2582-6271

Vol.1 No.6; Dec. 2020

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