Optimization Study of Charcoal Observation With Scanning Electron Microscope In Various Operating Conditions

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Abstract. Scanning electron microscopy (SEM) has been widely used in the development of science, especially in the field of materials science. The development of SEM as a tool of material analysis has also experienced very rapid development. However, in practice, the use of SEM for analysis requires setting parameters that cannot be generalized between materials. This study conducted to analysis charcoal morphology on various parameters of SEM operation. From the result of the study, it is known that some SEM operation parameters such as voltage accelerate, spot intensity, working distance and objective aperture influence the results of charcoal morphology analysis with SEM.

Keywords: SEM, accelerating voltage, aperture, working distance, spot intensity, charcoal.

1. Introduction

Scanning Electron Microscope (SEM) is a microscope that observes a high magnification’s object using electrons. SEM scans objects by scanning with high-speed electrons. SEM has a magnification of 10 - 300000 times and the resolution reaches below 1 nm even smaller depending on the type of SEM. The primary functions of SEM can be used to find out topography, morphology, composition of a material. The combination of high magnification, excellent resolution, ability to analyze the composition and crystallographic information make SEM widely used for research and industrial purposes.

SEM working principle is the electron beam produced by a filament on an electron gun fired at the test sample. The commonly used electron gun is a filament of tungsten hairpin which functions as a cathode. A high voltage is applied to the filament which results in the release of electrons in the filament. The released electrons will move towards the anode where the test sample is placed with speed depending on the given rating. In SEM there is a condenser lens that focused the electron beam to a point on the surface of the sample. Condenser lenses focused on the electron beam in a very small diameter, which is about 10-20 nm. When an accelerated electron interacts with there will be an interaction of electrons with the sample. Electron interactions with samples produced two new types of electrons which are secondary electrons and backscattered electrons. Secondary electrons have appeared when electrons from a filament hit an atom at the surface of the sample. Electrons in the certain atomic skin get enough energy to escape from the atom. The loose electron is called a secondary electron which is then captured by the SE detector. Secondary electrons are used to analyze surface information in the form of topography and morphology. The second type of electron is backscattered electron (BSE). BSE is an electron from the filament source which is reflected after the
electron hits the sample atom. BSE can be used to analyze the composition differences in a sample because each composition (molecule or atom) will reflect electrons with different intensity depending on the type of the composition of the sample itself. The electron interaction with the material causes secondary electron and scattering electron (BSE) from the surface of the sample. SE and BSE will be detected by the detector and displayed in the form of image images on a computer screen.

The attainment of high-quality SEM images requires testing samples with good conductivity, thermal stability, and a high secondary electrical yield[1,2]. In general, SEM image capturing requires the use of detectors that collect secondary electrons and backscattered electron signals[2-5]. The production of excess electrons or accumulation of free particles on a low conductive sample surface can result in a charge-discharge phenomenon and sensitive to radiation damage[6]. Charge-discharge is a phenomenon that the surface of the sample with poor conductivity force to produce a certain electrostatic charge accumulation under the irradiation of electron beam, which ultimately affects the electronic signal transmission and can generate image distortion problems such as image deformation, charged, electron drifting[2,7]. Gold plating with the surface of the test sample is carried out to improve non-conductive sample conductivity. The gold layer can reduce the accumulation of electrons and reduce damage to the test sample. However, if the gold plating is too thick, it can cause detailed changes in the sample topographic information, dimensional changes, and differences in sample composition which makes it difficult to observe [2,8]. The SEM operation process to produce high-quality images requires several parameter settings that are determined based on the type of sample tested, magnification, and information to be obtained. These parameters are the voltage of electron acceleration, Spot intensity, working distance, and Aperture.

A study of parameter optimization and sample characteristics in the SEM test has previously been carried out, but studies using charcoal have never been done. Previous research, Stein (1974) conducted a study on the effect of accelerating voltage in SEM on a biology sample, then Pretorius (2010) also conduct an investigate effect accelerating voltage with blood platelets samples, then the latest Dan he (2018) conducted study of FE SEM optimization with low conductive samples, namely hydroxyapatite, modified poly (vinylidene fluoride) and zinc oxide nanopillar. Based on a review of a previous study, this study discusses parameters that influence the operating condition of SEM to get the best test results on charcoal micrograph. Also, this study can also be used to reference SEM testing in other samples by determining the characteristics of the sample.

2. Materials and Methods
Charcoal obtained from Hitachi High-Tech at Tokyo Solution Laboratory (Kawasaki City, Kanagawa Prefecture, Japan). The surface morphology of charcoal obtained using SEM Hitachi SU3500 series with the different operating condition including spot intensity, voltage accelerates, working distance and appature. In this experiment used a secondary electron detector, because the focus of this study is on the morphology of charcoal which only can be analyzed with a secondary electron detector.

3. Results and discussion

3.1. Effect spot intensity
Spot intensity was showing probe current intensity electron beam from filament electron gun. Spot intensity was also showing the size of the electron beam spot. Spot intensity determined based on condenser lens current from highest to lowest [9]. A higher current intensity, resulting in a strong electron beam signal and low image noise. However, resolution of the image decreases, and there was distortion that was attributed to the charge which was the result of electron accumulated on the sample surface [2]. The result of various spot intensity shown in Figure 1.

Based on the observations of charcoal the sample was known if the spot intensity is higher then the image quality is lower. High image quality gets if using a low-intensity spot. However, the use of low spot intensity will be difficult in determining focus because Low-intensity spot causes high noise and number of electron decreases.
3.2. Effect Accelerating voltage

Accelerating voltage (Vacc) is the voltage difference between the electron source and the anode which accelerates the electron beam towards the anode. The accelerating voltage of a typical SEM ranges from 0 to 30kV. Changing the accelerating voltage alters the incident electron energy to a specimen. This entails changes area of electron scattering in the specimen, secondary electron emission yield, production charging up and depth penetration. In consequence, various image quality changes as shown in figure 2.

The most important thing about changing the accelerating voltage is the penetration depth. Penetration depth is a measure of the depth of the electron penetrating the sample. To get the high-quality image, it requires penetration depth with a certain value to get maximum sample surface information and resolution [10]. Penetration depth is determined based on the formula of Kanaya Okoyama.

\[ R = \frac{0.0276 A E_0^n}{(Z^{0.89})} \]

R -- Depth Penetration
A -- Atomic Weight (g/mole)
n -- A constant
E_0 -- Beam Energy (KV)
Z -- Atomic number
\( \rho \) -- Density (g/cm³)

Based on the formula above the penetration depth is determined by Vacc, density, and type of atom. Atoms with large or large density atomic numbers will need a large Vacc to get the penetration
depth compared to the density and smaller atomic numbers. In this study the sample used was charcoal. Test results with various Vacc variations are shown in Figure 2. charcoal is a sample that is included in a sample of small atomic numbers, and the density is also not too high so that the Vacc that is not too large has obtained a good image. Tests with high Vacc (15kv and 30 kV) have some morphological images that look transparent and light-emitting due to the charging up phenomenon. This happened because the depth penetration was too deep so that the secondary electron not only came from the surface of the sample but also the inside.

![Figure 2. SEM image of charcoal at the different accelerating voltage under working distance 5 mm and Spot intensity 30](image)

Images with Vacc 15 kV and 30 kV shows light exposure due to the charging up phenomenon. This charging phenomenon shows that there is a buildup of electron charges on the surface of this charcoal sample. The parameter that affects this phenomenon is the conductivity value of the sample. Conductivity values indicate the level of a sample flowing electric current (electron). The greater the conductivity, the greater the ability of the ocean to flow electric current. Carbon has a conductivity value of 3 Mohm. The higher the conductivity value, the smaller the risk of charging when the Vacc is enlarged. In the charcoal samples tested, the results with Vacc below 10 kV showed the charging phenomenon was decreasing and the best picture on the picture with 5kVVacc. Besides, the higher the Vacc, the risk of the sample being damaged when interacting with high-speed electrons. Understanding the type of sample is very important to determine the Vacc as needed.

### 3.3. Aperture effect

The objective lens aperture controls the divergence angle of the electron beam passing through it and affects the electron beam diameter, the probe current (number of an electron), focal depth. The aperture at Sem Hitachi SU3500 has five apertures with aperture number 4 having the smallest diameter aperture lens and other hands the aperture number 0 has the largest diameter aperture lens. Probe electron shows the number of secondary electrons generated. If the aperture lens diameter gets smaller, this causes the number of secondary electrons produced decrease. Focal depth D refers to a distance range in the direction of height where an image appears to be focused even when the focal point is deviated due to the surface unevenness of the image. This depth is determined by the
divergence angle. This signifies that the focal depth of SEM is longer when the size of the objective lens aperture is smaller.

SEM test results on charcoal with various Aperture parameters are shown in Figure 3. The image shows that the smaller aperture value of the resulting image the lower the quality. Theoretically, if the aperture gets smaller, this causes a large divergence angle resulting in shallow focal depth, and then the beam diameter gets bigger. Based on the resulting image shows the aperture number 4 produces the best image. The disadvantage of using a small aperture is that the current probe gets smaller so that the observation and finding focus process will be more difficult.

![SEM image of charcoal at the different Aperture under 3 kV accelerating voltage, working distance 5 mm and spot intensity 30.](image)

**Figure 3.** SEM image of charcoal at the different Aperture under 3 kV accelerating voltage, working distance 5 mm and spot intensity 30.

### 3.4. Working Distance effect

Working distance (WD) is the space between the bottom of the SEM column and the top of the sample. In general, the shorter the working distance will result in a higher resolution image and present a clearer surface detail. Changing the working distance bring about changes in the beam diameter, the focal length of the objective lens and the divergence angle of the electron beam. Extending the working distance enlarges the diameter of the electron beam but increase the focal depth. Also, when the working distance is extended, the scanning area of SEM widens and magnification decreases.

Test results with working distance variations are shown in figure 4. Based on working distance resulting image at a small magnification, it does not have too much effect. However, at large magnifications, working distance affects where the small working distance values focus better than the large working distance. The high working distance will make the diameter beam bigger, and therefore high magnification will be blurred. Working distance low contrast will make the small diameter beam, and that means the focus will be better.
4. Conclusion
The present study directly characterized a representative sample of charcoal using SU 3500 scanning electron microscope. The optimum conditions for these morphology materials observations were obtained following changes in the different working accelerating voltage, electron beam spot intensity, aperture, and working distance. Also, the application SEM on the direct morphology observation was systematically examined. The optimized study generated a clear improvement in the observation accuracy and detection efficiency of the sample.

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