Scanning Electron Microscopy

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Contents

Abstract

In this experiment, the basic working principles of the Scanning Electron Microscope (SEM) were studied. Initially, the specific charge of electron e/m_e was calculated by analyzing the path of electron beam in a teltron tube under the influence of magnetic field produced by the Helmholtz coils. The ratio e/m_e was found to be $(1.79 \pm 0.33) \cdot 10^{11}\ As/Kg$ which is in accordance with the theoretical value of $1.759 \cdot 10^{11}\ As/Kg$. Then, the SEM was operated to analyze different specimen samples. The different modes and controls of SEM were used to obtain detailed image as well as to understand its working principle.

1 Introduction and Theory

1.1 Determination of specific charge of electron

When an electron with charge e and rest mass m_e is subjected to an acceleration due to potential difference U, it moves with kinetic energy E_{kin} given by:

$$E_{kin} = \frac{1}{2} m_e \cdot v^2 = e \cdot U \tag{1}$$

where v is the velocity of electron. In presence of magnetic field of strength B, the moving electron experiences Lorentz force $\vec{F_L}$ given by:

$$\vec{F_L} = e \cdot (\vec{v} \times \vec{B}) \tag{2}$$

As a result, the electron follows a helical path with the pitch of the helix depending on the angle between $\vec{v}$ and $\vec{B}$. In a special case when the velocity of electron, $\vec{v}$ is perpendicular to the magnetic field, $\vec{B}$, the path becomes circular as the Lorentz force provides the centripetal force.

$$m_e \cdot \frac{v^2}{r} = e \cdot v \cdot B \tag{3}$$

where r is the radius of the circular path followed by electron. Combining equations (1) and (3), the specific charge of electron can be calculated as:

$$\frac{e}{m_e} = \frac{2U}{(B \cdot r)^2} \tag{4}$$

A pair of Helmholtz coils, each with radius R produces nearly homogeneous magnetic field at position $z = 0$ between them given by:

$$B = \left(\frac{4}{5}\right)^{\frac{3}{2}} \cdot \mu_0 \cdot n \cdot \frac{I}{R} \tag{5}$$

where I is the current, μ_0 is the permeability of free space and n is the number of turns in the coil.

From equations (4) and (5), the linear relationship between U and I^2 for a fixed radius r can be established as:

$$U = \left[\frac{32}{125} \cdot \frac{e}{m_e} \cdot \mu_0^2 \cdot n^2 \cdot \frac{r^2}{R^2}\right] I^2 \tag{6}$$

Using the slope, m of the U vs I^2 line, the specific charge of electron can be calculated as:

$$\frac{e}{m_e} = \frac{125}{32} \cdot m \cdot \left(\frac{R}{\mu_0 \cdot n \cdot r}\right)^2 \tag{7}$$

1.2 Physical Principle of the SEM

The Scanning Electron Microscope (SEM) produces magnified image of a sample by focusing a beam of electrons over the specimen and then systematically scanning over the surface of specimen in a rectangular pattern. The strength of signal generated in the SEM image corresponds to the topographical or compositional differences in the sample.

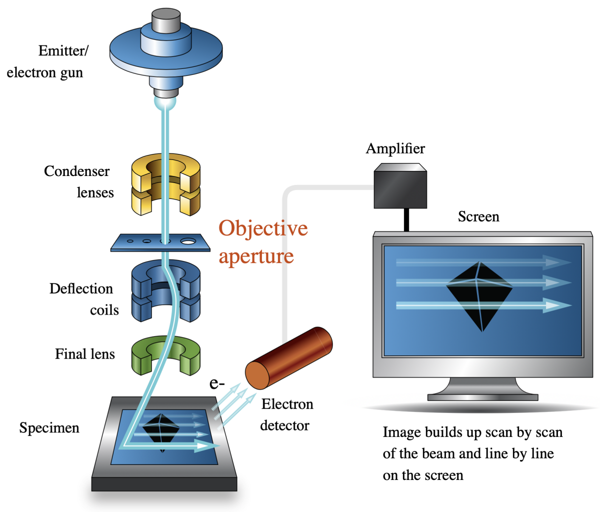

Figure 1: SEM layout and function[1]

The major components of an SEM are: the electron gun, the vacuum system, the water chilling system, the column, the specimen chamber, the detectors and the imaging system. The electron gun produces a source of free electrons via thermal emission from heated tungsten wire, called the filament and accelerate them. The filament is surrounded by the Wehnelt cylinder that has a hole in the center through which the electrons exit towards the anode. The portion of the cohesive beam that leaves the anode through the hole is termed the beam current. The filament load current of the thermionic electron gun has to be set correctly. Too low a current yields an image of inadequate brightness, whereas excessive current reduces the working life of the filament.

High vacuum mode is the normal mode of operation for the SEM to minimize scattering of the electron beam before it reaches the specimen. The vacuum is achieved by the action of a rotary pump (for rough evacuation) followed by a diffusion pump. The rotary pump expels the gases by creating alternating high and low pressure sequences due to rotation of vane in cylindrical chamber. In the diffusion pump, the vapour, formed by heating the oil, collects gas molecules in the pump. The vapour condenses on the walls, releasing the gases which are then extracted by rotary pump. The SEMs also include a water chilling system to maintain a constant temperature of 20°C for the operation of the magnetic lenses in the microscope.

The electron column focuses the beam and illuminates the specimen through a series of electromagnetic lenses. The condenser lens converges the cone of the electron beam to a spot below it, before the cone flares out and is converged again by the objective lens down onto the sample. The condenser lens current is referred to as the spot size control since the diameter of the initial convergence affects the final diameter of the spot the beam makes on the sample. Two pairs of electromagnetic deflection coils, called the scan coils, are used to construct the image point-by-point and line-by-line by deflecting the electron beam horizontally and vertically over the specimen surface.

When the electron beam strikes the surface, the interaction with the sample causes some electrons to bounce back (backscattered electrons), while others knock into atoms and displace electrons which in turn, come out of the sample (secondary electrons). X-rays as well as light and heat in the sample are also possible. The secondary electron detector are used to obtain topographical information while backscattered electron detector is ideal to obtain physical and chemical characteristics as well as information from below the surface of the sample.

2 Experimental set-up and procedure

2.1 Determination of specific charge of electron

The experimental setup is shown in Fig. 2.

Figure 2: Experimental set-up for determining the specific charge of electron[2]

The electrical connection of the Helmholtz coils is shown in the diagram in Fig. 3 and Fig. 4. As shown in Fig. 2, the two coils coils are turned towards each other in Helmholtz arrangement. Since the current must be the same in both coils, a serial connection was established between them . It was made sure that the ammeter reading didn't exceed 5A.

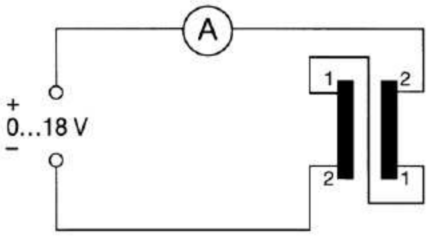

Figure 3: Wiring diagram for Helmholtz coils

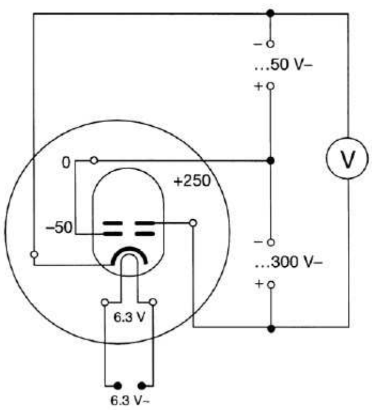

Figure 4: Wiring diagram for Narrow beam tube

The room was darkened for the experiment to observe the curved luminous trajectory with the correct polarity of the magnetic field. The radius of the orbit was adjusted by varying the magnetic field (current through Helmholtz coils) and the velocity of the electrons (acceleration and focusing voltage), such that it coincided with the luminous traces inside the tube. The radius of the circle was then obtained 2, 3, 4 or 5 cm. Also, at times, the trace obtained in the form of helix was eliminated by rotating the narrow beam tube around its longitudinal axis.

2.2 Scanning Electron Microscope

The picture of SEM used for the experiment is shown in Fig. 7

Figure 5: Image of Scanning Electron Microscope used

Initially, the given sample number 14 was placed inside the chamber and the chamber was shut down. Water cooling of the SEM was started. The the rotary (RP) and diffusion (DP) pump were turned on to evacuate column and sample chamber. The control panel was then checked again for all the operating elements. After the green V.L indication near DP was turned on, the operation was started with the display meter switched to emission mode. The X and Y positions of the microscope were adjusted to get the image in spot. Magnification of the SEM was used till 2000x to observe the sample. The focus knob was adjusted to obtain the sharp image. The specimen was also changed. For that, first the operation was turned off with emission knob turned to left, then the sample chamber was opened and the specimen was changed by unscrewing and screwing back the specimen holder. After the specimen was analysed, the operation was closed and vacuum was turned off. DP was switched off and the system was left to cool for about 20 minutes and then the water cooling and main power supply was also switched off.

3 Results and Data Analysis

3.1 Determination of Specific Charge of electron

The following information were noted for the setup shown in the figure(2):

$$\text{Number of turns in Helmholtz coils (n)} = 154$$

$$\text{Radius of Helmholtz coils (R)} = (0.2 \pm 0.05)\, m$$

After the measurement of current(I) and voltage(U) from multi-meters, the magnetic field (B) was calculated by using the formula provided in the manual[2]:

$$B = \left(\frac{4}{5}\right)^2 \cdot \frac{\mu_0 \cdot n \cdot I}{R} \tag{8}$$

Then, the specific charge of electron (e/m_e) was calculated using equation (4). It was observed that there was huge discrepancy between the calculated and the theoretical values. To account for this error, the whole set-up of the experiment was re-examined. After measuring the magnetic field in between the two Helmholtz coils manually using a portable Cassy, it was noticed that the calculated value of B from equation (8) had considerable error. Upon analysis, it was found that the correct expression for B due to Helmholtz coils should have been as stated in equation (5). Hence, equation (5) and equation (7) were used in the analysis that followed.

The plot of voltage (U) as a function of current squared (I^2) at different radii of circular path was obtained as shown in figure(6):

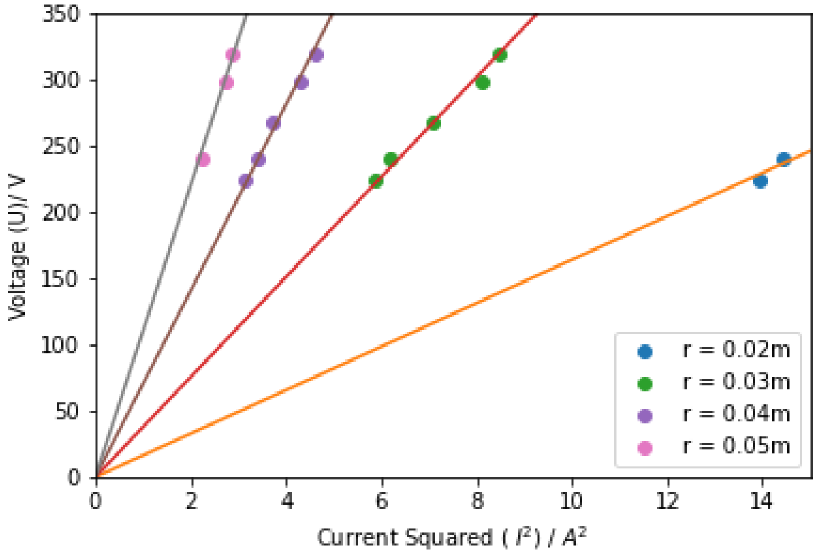

Figure 6: Variation of Voltage with square of current for different radii

For each straight line in figure (6), their slopes and the corresponding value of specific charge of electron were calculated using equation (7).
For r = 0.02 m :

$$\text{Slope} = 16.37V/A^2 \implies \left(\frac{e}{m_e}\right) = (1.71 \pm 0.26) \cdot 10^{11} As/kg$$

For r = 0.03m:

$$\text{Slope} = 37.66V/A^2 \implies \left(\frac{e}{m_e}\right) = (1.75 \pm 0.34) \cdot 10^{11} As/kg$$

For r = 0.04m

$$\text{Slope} = 70.12V/A^2 \implies \left(\frac{e}{m_e}\right) = (1.83 \pm 0.60) \cdot 10^{11}\ As/kg$$

For r = 0.05

$$\text{Slope} = 109.88V/A^2 \implies \left(\frac{e}{m_e}\right) = (1.83 \pm 0.91) \cdot 10^{11} As/kg$$

Hence, the average value of specific charge of electron was calculated to be:

$$\frac{e}{m_e} = (1.79 \pm 0.33) \cdot 10^{11} As/kg$$

3.2 Investigation of samples with SEM

The sample number 14 provided in the lab was observed under various magnifications as shown in the figures (7):

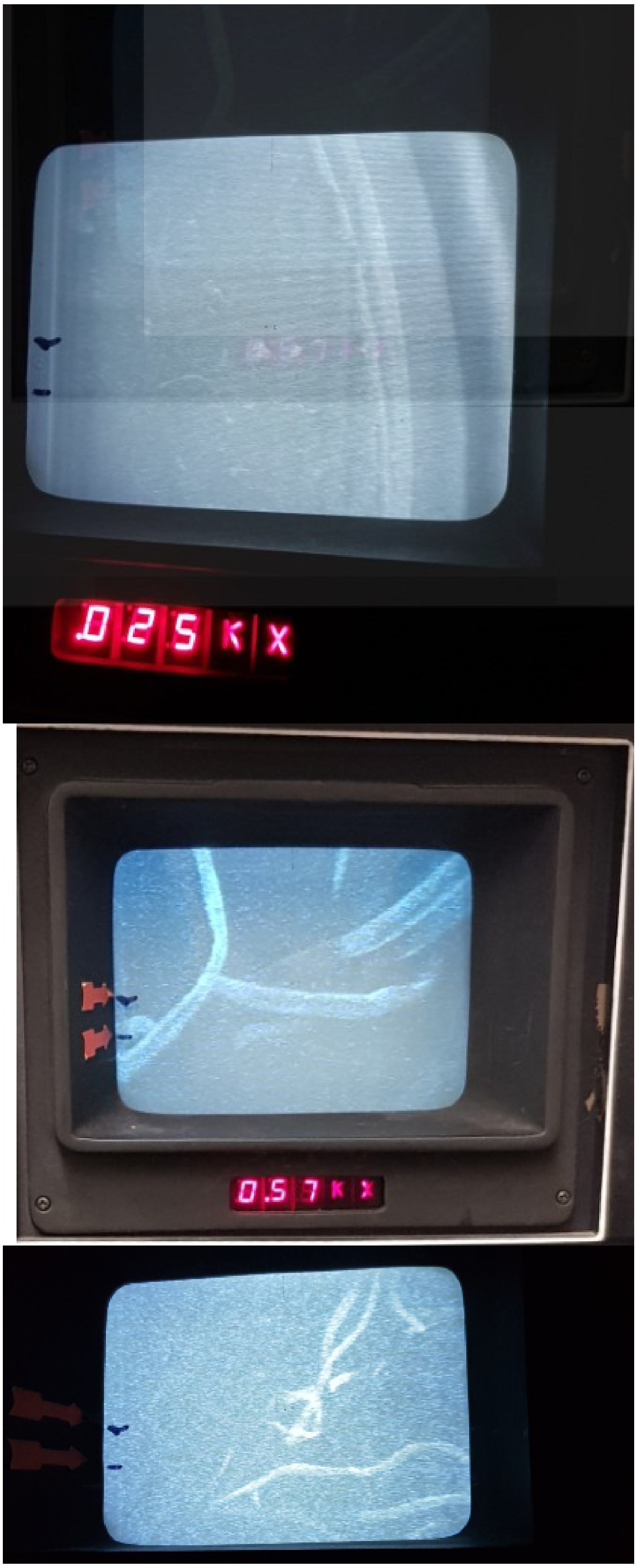

Figure 7: Sample as seen on screen with [top]: no magnification; [middle]: ×700 magnification; and [bottom]: ×2000 magnification

4 Discussion and Conclusion

In the first part of the experiment, the specific charge of electron e/m_e was determined. Electrons were accelerated in an electric field and entered the magnetic field at right angles to the direction of motion. The specific charge was then calculated from the measured accelerating voltage, the magnetic field strength and the radius of the electron orbit as shown in Table 1 in Appendix. Since the calculated values were not in the range of theoretical values, the corrected formula as given in equation (5) was used to calculate the magnetic field provided by a pair of Helmholtz coils.

The measured value for the specific charge of electron was found to be $(1.79 \pm 0.33)\cdot 10^{11}$ As/kg which was in agreement within the literature value of $(1.759) \cdot 10^{11}$ As/kg within the error intervals calculated. One of the primary sources of error experienced in the experiment could be the uneven focusing of the electron beam on the luminous bars for different radius. From the calculation shown in section 3.1, it can be seen that for the radius of 3 cm, the calculated value is very close to the literature value. However, with the increase in radius of the electron beam, the errors got higher. This indicates the systematic error associated with the measurement of the radius of the electron beam. There could also have been error due to the slight changes in magnetic field along the length of the tube. Another possible source of error could be that the electrons were not injected perfectly perpendicular to the magnetic field so that they might had the tendency to move in spiral instead of circle.

The second part of the experiment was about the investigation of the samples with the use of SEM. The specimen was observed at magnification up to 2000x. However, the image was resolved with better clarity at a magnification of 700x. The higher magnification and resolution is one of the advantages of using SEM over optical microscopes. However, nonconducting samples for SEM invariably need some sort of coating with gold or carbon before they can be examined and analyzed. The coating is necessary to eliminate or reduce the electric charge that builds up rapidly in a nonconducting specimen when it is scanned by a beam of high-energy electrons.

References

[1] Article retrieved from https://myscope.training//SEMlevel_3_1
[2] Prof. Dr. Arnulf Materny and Dr. Vladislav Jovanov, Advanced Physics Lab I, CO-486-A, Fall 2020