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A Theoretical-Based Experimental Approach for Investigating the Charging of Insulators

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Abstract

A theoretical-based experimental approach has been presented as a simplified tool for investigating the charging of insulators. Throughout this work, the Polyethylene Terephthalate (PET) material is chosen as a case study. The experiment is carried out using the scanning electron microscope (SEM) with a primary energy of 30 keV, an electron current of 1.3 nA, and a working distance of 15 mm. The suggested approach has been initiated by the spacemen-current as the first step. Keeping in mind that this parameter is by default recorded by the SEM machine itself. The total electron yield emission is then determined during the irradiating time in accordance with this. Subsequently, the consuming time, electron-trapped lifetime, trapped electrons, leakage, and displacement currents are evaluated. Results have clearly showed that the knowledge of the spacemen current can be used to provide excellent information to SEM users. Indeed, many of the characteristics factors that define the insulators can be evaluated with no more complicated experimental setup.

Keywords: Charging effects; Electron irradiation; Insulators; Mirror effects; Scanning electron microscope.

1. Introduction

Imaging of insulating surfaces in scanning electron microscopy has always been a challenge due to the charging effects (CE) [1],[2]. Such a phenomenon involves accumulating the charges (electrons or ions) on the material surface. This is arising due to the excellent ability of these materials to build up charges in a microscope compared with conductors. The most important devices for investigating this phenomenon are the scanning electron (SEM) and focused ion beam (FIB) [3]. When insulators need to be investigated, CE is categorized as a disadvantage inherent in the SEM and FIB [4],[5]. Physically, the trapped charges lead to imaging the inner walls of the SEM and FIB instead of the specimen surface itself [6]. Hence, the apparatus fails to do its desired job [7].

For many years, CE has been observed and has attracted attention due to its strong connection to insulator properties [8]. Many theoretical, experimental, and computational [9]-[13] investigations for the CE have been presented since the seventeenth of the last century. Surprisingly, the phenomenon of CE of dielectrics is still not fully understood despite its long history [14]. However, the study of this phenomena is crucial for comprehending the dielectric strength, net charge density [15], microscopic charge characteristics [16], charging surface potential [17]-[19], flashover phenomenon [20] of insulators, and the self-origination of trapped charges within the surface [21]. Accordingly, CE may be explored as a tool for analyzing the insulators. Implementing the approaches related to CE studies usually requires a complicated experimental setup. [3],[5],[22], [23].

Current work, however, has been proposed to overcome this experimental complication. Therefore, it aims to provide SEM users with a simplified approach for investigating insulators. The ambition approach requires no more experimental preparation except for the specimen current, which is usually recorded by the machine itself. Hence, the user no longer has to prepare data to evaluate insulators.

2. Experiment Setup

The charging experiment was carried out with a 3D-Quanta Dual Beam scanning microscope. Using the specimen's current method, an invented procedure was used to estimate the consumption time. Polyethylene Terephthalate (PET) is used in the current work for a case study. The PET specimen was mounted on the grounded specimen holder, and the upper spacemen's surface was positioned perpendicular to face the electron beam. A metal cup with a circular hole made in its upper face of diameter 6.3 mm is located on the stage holding the sample, as shown in Fig. 1. Such a cup is used to ensure that the PET sample and its holder are isolated from the space of the machine chamber. Indeed, the metal cup prevents the backscattered, secondary, and Auger electrons from returning and adding to the recorded specimen current.

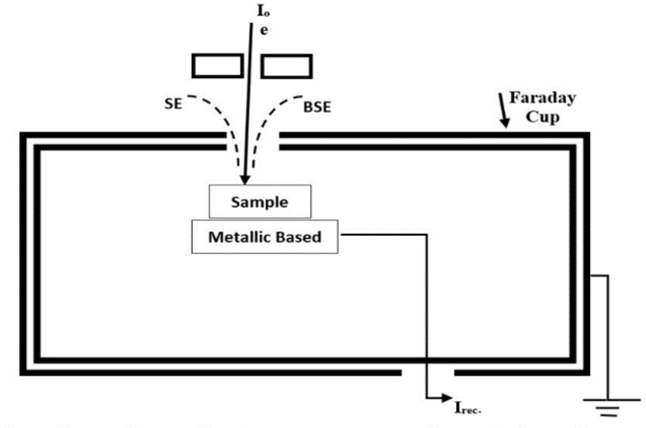

Figure 1. A schematic representation of the cup used to cover the spacemen and its holder.

It is important to mention that the PET sample is placed in contact with the specimen holder inside the cup. The cup is perfectly isolated from the holder by putting a Teflon ring in it to prevent electrical contact between them. The PET sample has been made into a flat circle with a diameter of 1 cm to fit in the specimen holder. Using distilled water, the surface of the sample was carefully cleaned. The experiments were carried out at room temperature and pressure of 2.0x10-5 Pa. Throughout this experiment, the working distance of the apparatus is set at 15 mm. A high-sensitivity pico-meter is connected to the apparatus sample holder, and its results are directly recorded on the apparatus's computer monitor. The current experiment is implemented by accelerating a bombardment beam of electrons with a potential of 30 KV and a current of 1.3 nA. The irradiation beam is oriented perpendicularly to irradiate an area of 18.39 mm2 from the PET specimen.

3. Recorded pico-meter current

When the PET specimen is irradiated with an electron beam, some of the electrons will be trapped in the PET's bulk. The trapped electrons either get stuck at the defect sites of the PET specimen or end up as self-trapped electrons (polarons) [24]. Trapped electrons gradually form a layer that steadily covers the irradiation area as long as the irradiation process continues for a period. Eventually, this layer forms an electric field that results in a detrapping process. Furthermore, this field tries to prevent incoming electrons from reaching the specimen. Indeed, the electric field acts as a mirror for the incident electrons. Consequently, incident electrons reflect towards the inner walls of the cup, and some of them escape across the hole-cup towards the inner walls of the apparatus chamber. Thus, the image appeared for these walls instead of the PET specimen, as shown in Fig. 2.

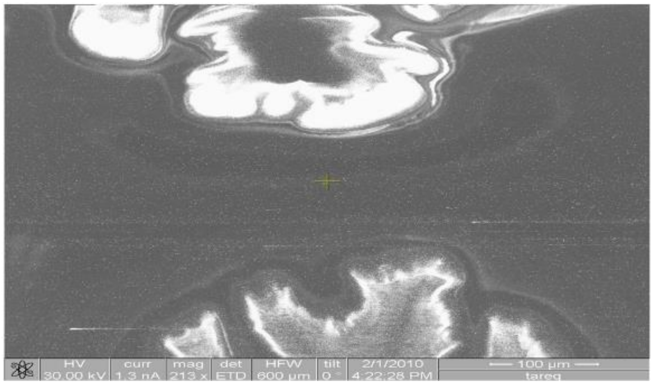

Figure 2. The electron mirror image for the PET specimen.

Fig. 3 shows the specimen current (I_s), as recorded at the apparatus screen, as a function of the irradiation time. There is no debate about a significant noise regarding the record of I_s. This noise could be caused by the backscattered (BSE) and secondary (SE) electrons incidents on the inner walls of the metal cup. This leads to the liberation of new SE of lower energy that moves towards the PET specimen. Additionally, low-energy SE that leaves the PET specimen may return to the surface in regions away from the irradiated area. These are the most likely reasons why the curve of I_s fluctuates around its actual values. Therefore, fitting I_s along with the irradiation time is useful. The dashed line in Fig. 3 represents the fitting curve of the specimen current. Another remark can be recorded from Fig. 3: the pico-meter starts recording the specimen current at an irradiation time approximately greater than 3s. This result clarifies that for the t≤3s, there is no significant concentration of trapped electrons within the irradiated area compared to its neighbors. Thus, the flow of electrons could not occur in these three seconds, which can be called the current damping time of the spacemen.

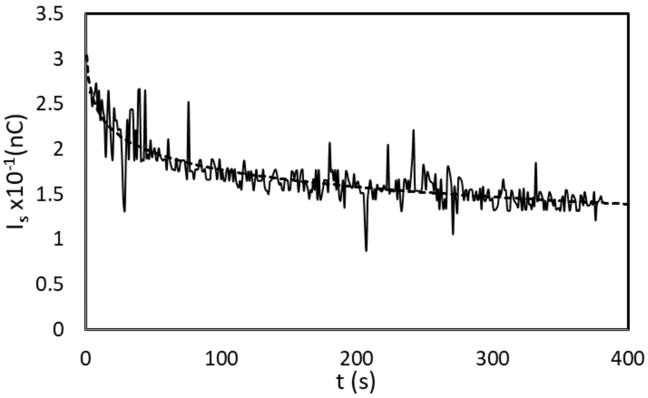

Figure 3. The specimen current is recorded from the screen of the apparatus as a function of the irradiation time.

Throughout the following contexts, attention will be focused on using the curve of I_s to analyze insulator materials. This adoption may provide a simple and straightforward approach to evaluating insulation materials. Furthermore, it may compensate for the complexity usually inherent in the investigation of insulators using SEM.

4. Determination of the Total Electron Yield

Indeed, while some electrons are trapped in the PET specimen, the rest are backscattered in different ordinations for the cover cup space. Some trapped electrons lead to the liberation of SE from the PET bulk during irradiation (t). The emitting of both SE and BSE forms an electron emission current $I_\sigma(t)$ related to the irradiation current I_i by the relation $I_\sigma(t) = \sigma(t)I_i$. Where $\sigma(t)$ is the total electron emission yield that is a sum of the emission coefficients of SE ($\delta(t)$) and BSE ($\eta(t)$), i.e., $\sigma(t) = \delta(t) + \eta(t)$.

The high concentration of trapped electrons $Q_t(t)$ throughout the irradiated area leads them toward neighboring regions. This movement ($dQ_t(t)/dt$), however, results in the generation of a displacement current $I_d(t)$. The diffusion of self-trapped electrons along the PET surface and the detrapping process leads to another current. Typically, this type of electron flow is called leakage current $I_L(t) = Q_t(t)/\tau'$, where τ' represents the lifetime of the trapped electron [6]. Thus, the law of charge conservation requires that [25];

$$I_b = \sigma(t)I_b + I_s(t) \qquad (1)$$

Where $I_s(t)$ is the entire current that flows within the specimen, i.e., the sum of displacement and leakage currents ($I_d(t) + I_L(t)$).

Concerning the usual uses of SEM, the SEM records the specimen and irradiation currents. Thus, equation (1) can be used directly to determine the total electron emission yield as follows;

$$\sigma(t) = 1 - \frac{I_S(t)}{I_b} \qquad (2)$$

Accordingly, the total electron yield is determined and plotted in Fig. 4 with the specimen current as a function of the irradiation time. It is seen that just after the damping time has passed, the spaceman current starts with a maximum value of 0.27 nA. Thereafter, the spacemen current gradually decreases concerning the irradiation time and eventually reaches almost fixed values of about 0.150 nA at $t > 350s$. Conversely, the curve of $\sigma(t)$ shows a reversal of behavior. The electron yield begins at a minimum of 0.792 after approximately 3s of irradiation and increases as the irradiation process continues. However, at $t > 350s$, the slope of $\sigma(t)$ approximately vanishes, indicating that the PET reaches the saturation case.

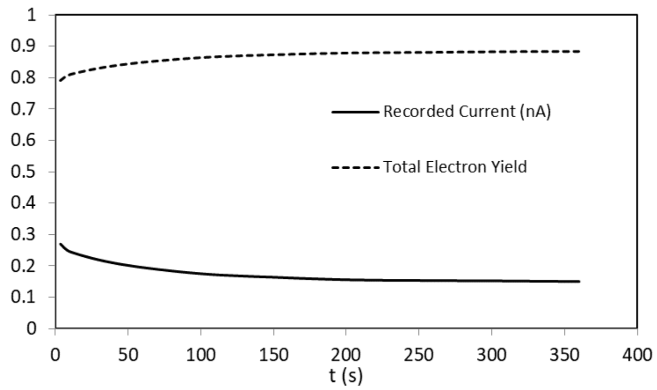

Figure 4. The specimen current and the total electron emission yield versus the irradiation **time.**

Indeed, the curve of $\sigma(t)$ can be used to reveal the behavior of PET material during the test process using SEM because two important parameters can be extracted from this curve in the sense of equation (2). Strictly speaking, these parameters are respectively the total yield at the beginning of bombardment ($\sigma(tb) = \sigma_o$) and its counterpart at the saturation limit ($\sigma(ts) = \sigma_s$). Just as these two parameters have been defined, the user of SEM could easily evaluate $I_d(t)$and $I_L(t)$, as illustrated in the following paragraphs.

Typically, the mathematical representation of trapped charges is given by the formula [4];

$$Q_t(t) = Q_s(1 - \exp(\frac{t}{\tau})) \qquad (3)$$

Q_s is the saturation value of the Q_t, i.e., $Q_t(ts)$, when the insulator can receive no more electrons. The symbol τ refers to the irradiation time when the trapping and detrapping processes become equivalent. Now, by relation ($I_s(t) = I_d(t) + I_L(t)$), one can easily find that;

$$I_s(t) = \frac{Q_s}{\tau} exp(\frac{t}{\tau}) - I_L^s(1 - exp(\frac{t}{\tau})) \qquad (4)$$

I_L^s represent the $I_L(t)$ value at the saturation limit. Remembering that the quotient $\frac{Q_s}{\tau}$ can expressed as ($\frac{Q_s}{\tau}$=$I_b(1 - \sigma_o)$ [26] and making use of equation (2), equation (3) can be reformulated as below;

$$\frac{t}{\tau} = ln(\frac{\sigma_s - \sigma_o}{\sigma_s - \sigma(\mathrm{t})}) \qquad (5)$$

With the aid of the curve of $\sigma(t)$ shown in Fig. 4, equation (5) has been plotted in Fig. 5 (labeled curve). The slope of this curve (dashed line) represents the reciprocal value of consuming time, which is $\tau = 63.68\ s$. Strictly speaking, at this instant of irradiation, the number of implanted electrons becomes equivalent to their counterpart dislocated from their own positions. Regarding the specimen's current information, several important parameters have been defined and summarized in Table 1.

Table 1. Several important parameters are by the specimen current.

t_b (s)	t_s (s)	σ_o	σ_s	τ (s)	I_L^s (nA)	Q_s (C)	τ' (s)
3.6	350	0.792	0.885	63.68	0.150	17.22	114.79

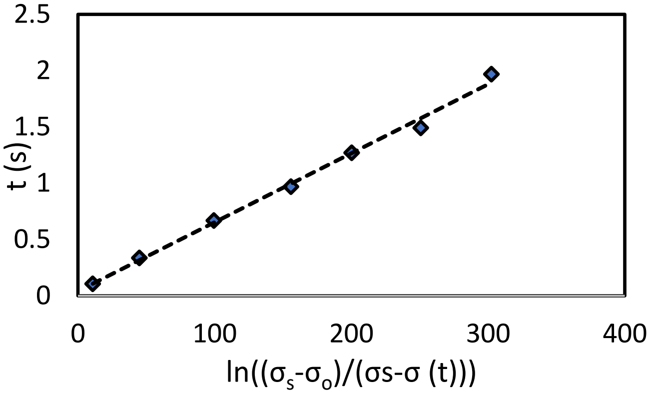

Figure 5. The time irradiation as a function of $ln(\frac{\sigma_s-\sigma_o}{\sigma_s-\sigma(t)})$.

5. Behavior of Displacement and Leakage Currents

Fig 6 shows the trapped electrons at the specified area of the PET specimen during the irradiation process. This curve has been deduced using equation (3) and the spacemen-current approach that leads to define Q_s and τ. Obviously, the PET specimen is neutral before initiating the irradiation process. Gradually, the PET becomes negatively charged, and this negativity increases until it becomes saturated at about t_b=350s. However, this means the irradiated area of the PET receives no more electrons beyond this critical time because the ability of PET to accumulate electrons has been exhausted due to its dielectric constant amount.

In other words, when the electron beam strikes the PET surface, the electron accumulates at the irradiated region. The accumulation grows progressively with time of irradiation until the detrapping process initiates. Thereafter, the growth of trapping reduces for the benefit of the detrrapping that progress sharply. This behavior is quite understandable, as shown by the curves of $I_d(t)$ and $I_L(t)$ in Fig 6. These two reverse processes continue until they become equivalent at $63.68\ s$. Fig 7 reveals that displacement and leakage current become equal at this irradiation time. In addition, Fig 7 shows that for $t > 63.68\ s$, the trapping process becomes less weight than detrapping. This means that the flows of trapped electrons are less than those of the detrapped electrons. Another loss for the trapped electrons occurs for the period $t > 114.797\ s$, where the lifetime of trapped electrons has expired. Finally, for the irradiation time of about $t > 350\ s$, the flow of trapped electrons (displacement current) almost vanishes. Hence, all electrons that flow throughout the PET spacemen come from detrapping. i.e., all spacemen current being for the leakage current, which is called saturation leakage current, see Fig 6.

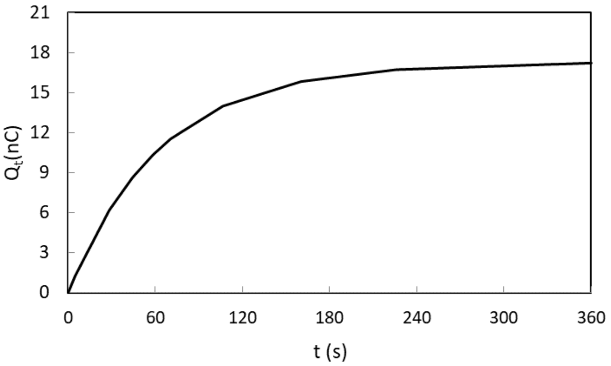

Figure 6. The amount of electron accumulation in the PET irradiated area is a function of time.

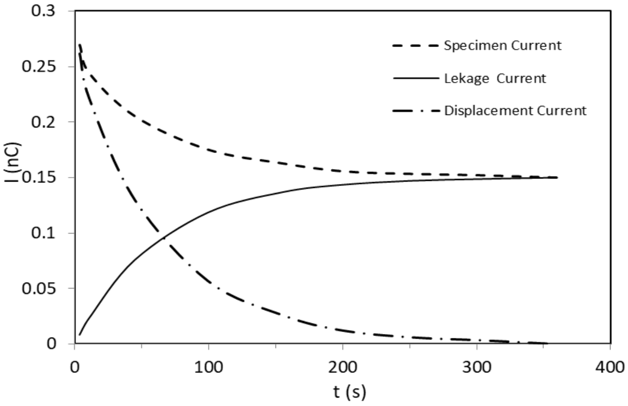

Figure 7. The specimen, displacement, and leakage currents are a time function.

For comparison purposes, the parameter Q_s has been determined according to the relation $Q_s = \int_{t_b}^{t_s}(I_s - I_L)$ [8]. However, the result is 17.61 nC, which differs by 2.22% from that evaluated throughout the present work (see Table 1). It's clear that the two outcomes are comparable and support the advantages of the current approach.

6. Conclusions

Several important remarks could be recorded according to the results obtained from the presented theoretical-based experimental approach. The knowledge of the spacemen can used to provide excellent information for the SEM users. Especially the SEM machine records the specimen almost by its default design. With the presented approach, the user of the SEM machine could quickly make a significant evaluation for any insulator material that needs to be examined. Implementing the insulator investigation using the current approach requires no more complicated setup. The algorithms followed in this work could be coded as part of the software used to operate the SEM machine. Hence, all the parameters and functions determined by the current approach can be revealed on the screen of the used SEM apparatus.

Conflict of interest

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

Author Contribution Statement

Author Tareq H. Abbood has proposed the research problem.

Authors Tareq H. Abbood and Hassan N. Al-Obaidi have developed the theory of the research.

Authors Tareq H. Abbood, Ali S. Mahdi and Wasan J. Kadhem have performed the computation of the research.

Authors Faten H. Mousa and Huda K. Hussein have compiled the necessary database for conducting the research.

All the sixth authors have collaborated in the discussion of the research results.

Authors Tareq H. Abbood and Hassan N. Al-Obaidi have verified the analytical methods, examined the impact of the outcomes, and supervised the findings of this work.

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