Analytical Investigation of Charging Ability of Insulators under Electron Beam Irradiation inside SEM Chamber

Hassan N. Al-Obaidi[1], Ali S. Mahdi[2]

[1]*Deparment of Physics, College of Education, Al-Mustansiriyah University, Baghdad, Iraq.*

[2]*Ministery of Education, Baghdad, Iraq.*

Abstract: An analytical procedure has been carried out to measure the charge that may trapped in an insulator sample and related electrostatic surface potential in sense of mirror effect phenomenon. In fact, scanning electron microscope mirror method (SEMME), sometimes called electron mirror method (EME) and/or magnification factor method (MFM), has been used to accomplished that purpose. However, this work has been carried out concerning the theoretical point of view, the mirror plot curve has been adopted as an evaluation scale for the quality of the mirror image. Therefore, this procedure had been used to investigates the experimental mirror plot curves for PMMA material with different accelerating potential and studying the most important parameters that affects in these curves. Results have clearly shows that the radius of irradiated area play an important rule in the shape of mirror plot figure and then the quality of the image.

Keywords: electron optics, scanning electron microscope, mirror effect.

1. Introduction

Many investigations have been accomplished concerns with electron trapping in insulators due to its own importance when a dielectric material is inspected by means of scanning electron microscope (SEM). The most critical issue regarding the observation of insulating material by (SEM) is charging due to electron-beam irradiation. Such charging causes deflection of the incident and emitted electrons, resulting in various phenomena such as image contrast variation, magnification variation, and image drift [1; 2]. These effects have been observed particularly by textile microscopists, and are frequently referred to in the literature as "charging" effects [3; 4]. The study of insulator charging effect in a (SEM) has led to several interesting observations. For instance Clark et al. observed a distorted image of the electron collector grid instead of the specimen surface, while tilting the certain uncoated insulating specimen [5; 6]. However, this distorted image as to be seen of the observation inside SEM chamber so called later mirror effect. Mirror effects occur when a primary electron beam scans an insulating sample and the charges on its surface accumulate to a high density. When the energy of the electrical field becomes higher than the primary beam one it prevents the charged particles from reaching the sample surface, reflecting them somewhere else in the vacuum chamber whose walls act as a mirror. The inner part of the specimen chamber can be therefore imaged. The phenomenon was explained in terms of something very close to what happens to photons interacting with an optical mirror [7; 8]. Actually, the inspection process behind insulator irradiation by a charged beam is a very complicated problem. This complication arises from several physical and geometrical situations for the sample to being. Where, the sample may be coated by a metallic material or not, grounded with the stage or not, separated from the stage by special distance or not… etc [9]. Consequently, various experimental techniques had been presented for measuring the trapped charges that suitable for specific situation. Most of the experimental techniques, including the thermal pulse method, the pressure wave propagation method, the Kerr electro-optic method, and the mirror image method, measure the total trapped charge and its distribution in the insulator [10].

Concerning with mirror image method one can be mention several literatures which adopted

this method to investigating the charging process and so the trapped charges. For example this technique is employed for the investigation of charging in different cuts of a α-SiO$_2$ [11]. Furthermore, the scanning electron microscope copper-detector technique is employed for the investigation of charging on different faces of single-crystalline α-quartz. However, these results are confirmed by the well-established mirror-image method [12]. Another authors developed this method to measure the charge distribution volume in insulators [13]. Where an electrostatic potential expression is derived by assuming the dipolar approximation and hemispheroid distribution. Dielectric samples with different relative permittivities are employed in charging experiments to justify this approach. The charging ability of insulator under electron beam irradiation have been investigated using a time-resolved current method. They derived a formula which related the measured current to the sample irradiation condition and the material properties. They found that measured current decreases with time exponentially at the initial stage and maintains a constant value when the saturation charge trapped in the sample is reached [10]. An electrostatic model has been developed to describe the mirror image formation in the scanning electron microscope mirror method and to calculate the trapping ability by using a semi-infinite sample [14]. In addition to that, observation of Pseudo–Mirror Effect had been reported and given an explanation concerning the factors that could be influence [15]. Other authors had been presented a reviewed investigation for the charging effects occurring when an insulating material is subjected to electron irradiation by means of SEM. However, they proposed a method to deduce the trapped charge inside the insulator (wither it is coated or not) and so the corresponding internal or external electric field [16]. The Electron Mirror Effect, (EME), images was discussed by using the passive Solid State Backscattered Detector (SSBSD). Where a non conductive PET sample irradiated by high energy electrons [17].

On the other hand a basic model to calculate the inversion point of electrons of the primary beam launched against the dielectric in connection with simple measurements is presented [8]. This investigation had been showed the importance of the analytical properties of EME (and IME) associates with the investigation of insulator charged surfaces, the whole family of experiments can be played a relevant role in the understanding of the basic features of electrodynamics of charged particles in a simple and controllable way. The electric charging phenomenon i.e. trapping-detrapping is studied by Scanning Electron Microscope Mirror Effect (SEMME) coupled with the Induced Current Method (ICM). The two complementary techniques were developed for insulator characterization for the study of charging properties of ceramics. It is shown that SEMME characterization can regarded to be a good method for study of residual stress and changes in properties of material developed in metal\ceramic joint [18]. The behavior of an accelerated probing electron that orientated towards a charged insulator sample and hence producing mirror images is investigated analytically [19]. Where, the distribution of the trapped charges at the sample surface is approximated as a point charge. Hence, analytical derivation for the path equation of this electron has been introduced. Thereafter a comprehensive investigation is curried out to inspect the influence of trapped charge, scanning potential, working distance and dielectric constant on the images by means of this model [6].

The forward line of this article is to explore the results that could be achieved from this experimental method to simulate the potential distribution due to the trapped charge. Eventually, detect the real profile that in accordance trapped charge extends over a volume of an insulator sample. Therefore the essential goal of this manuscript is to investigate the influence of trapped charge on the properties of the mirror images. So, attention will be focused on the mechanism by

which charges accumulates on the sample

2. Theory

Determination of trapped charge, and hence its related potential, within an insulator material may consider to be an ambition goal for many scientists, researchers, engineers and designers. Recently, such a task get additional interest in the field of electron microscopy and micro analysis due to the effect of electron mirror. Because trapped charge is the most effective parameters that controls the characteristics of an electron mirror image. Accordingly, when an insulator sample material of permittivity ε_d is irradiated be means of electron beam, one may regards that an arbitrary charge distribution $\rho(\vec{r}')$ be embedded in infinitesimal volume V' within

$$\varepsilon_d \nabla^2 U = \rho$$

Since, the quantity of charge Q_t is embedded inside dielectric material, the associated potential in the vacuum is equivalent to that of a quantity of charge KQ_t at the same position,

$$U(\vec{r}) = \frac{K}{4\pi\varepsilon_o} \int_{V'} \frac{\rho(\vec{r}')}{|\vec{r} - \vec{r}'|} dV'$$

Actually, an expansion for the potential in equation (2) may be carried out by means of several ways. In this work the binomial theorem has been adopted to do that concerning the spherical coordinates. In fact, the charge distribution of $\rho(\vec{r}')$ will assumed to be look

surface where the chamber of SEM involves.

the bulk of this sample. Indeed, any point within this volume can be located at a point $\vec{r}'$ which is in the neighborhood of the origin. Such assumption definitely indicate that the considered real volume V of the insulator material has infinite extend over the infinitesimal volume V', and hence no confliction should be arises. Strictly speaking, the charge distribution can regards to be of a dimension less point in comparison with the dimension of the sample, and hence such a point has a charge amount (Q_t) equivalent to $\int \rho(\vec{r}_1')dV_1'$. Therefore, the Poisson's equation is given by [20; 21; 22];

$$\dots\dots\dots(1)$$

where K is equal to; $(2/\varepsilon_r + 1)$ and ε_r is the relative permeability ($\varepsilon_d/\varepsilon_o$). Then the solution of equation (1) can takes the following expression;

$$\dots\dots\dots(2)$$

like a sphere of radius r' which is small compared with the distance $\vec{r}$, where the potential needs to be calculates. Accordingly, the denominator of equation (2) can be written as follows;

$$|\vec{r} - \vec{r}'|^{-1} = \left(r^2 - 2\vec{r}\cdot\vec{r}' + (r')^2\right)^{-1/2} \qquad\dots\dots\dots(3)$$

Using the definition of scalar product and make assuming the angle between the observation distance ($\vec{r}$) and distribution point distance ($\vec{r}'$) is ϑ', equation (3) convert to the following form;

$$|\vec{r} - \vec{r}'|^{-1} = r^{-1}\left(1 + \left(\frac{r'}{r}\right)^2 - 2\left(\frac{r'}{r}\right)\cos\vartheta'\right)^{-1/2} \qquad\dots\dots\dots(4)$$

The using of binomial expansion for the right hand side of equation (4) leads to the following expression;

$$|\vec{r} - \vec{r}'|^{-1} = r^{-1}\left\{1 + \left(\frac{r'}{r}\right)\cos\vartheta' + \left(\frac{r'}{r}\right)^2(3\cos^2\vartheta' - 1)/2 \right.$$
$$\left. + \left(\frac{r'}{r}\right)^3(5\cos^3\vartheta' - 3\cos\vartheta')/2 + ...\right\} \qquad \ldots\ldots\ldots(5)$$

The careful inspection of equation (5) reveals that the polynomial inside the parentheses is simple the Legendre functions. So, such a formula can be written as follows;

$$|\vec{r} - \vec{r}'|^{-1} = \sum_{n=0}^{\infty}\frac{1}{r^{n+1}}(r')^n P_n(\cos\vartheta')\rho(\vec{r}')dV' \qquad \ldots\ldots\ldots(6)$$

Now, the substitution of equation (6) in equation (2) leads ultimately to the following relation;

$$U(\vec{r}) = \frac{K}{4\pi\varepsilon_o}\sum_{n=0}^{\infty}\frac{1}{r^{n+1}}\int_{V'}(r')^n P_n(\cos\vartheta')\rho(\vec{r}')dV' \qquad \ldots\ldots\ldots(7)$$

Indeed, the nth term in the last equation represent the nth order multipole moment of the charge distribution of $\rho(r_1')$. Strictly speaking, the terms of n=0, 1, 2, 3, 4,…etc. are respectively monopole, dipole, quadrupole, octopole, …etc. moment of the charge distribution.

Regarding the charges being uniformly distributed within the spherical volume V' and extended equation (7) for n=0,1,2 and 3, then performing the integrations, the following formula could be obtain;

$$U(\vec{r}) = \frac{KQ_t}{4\pi\varepsilon_o r} - \frac{3KQ_t}{32\pi\varepsilon_o}\cdot\frac{r'}{r^2} + \frac{KQ_t}{64\pi\varepsilon_o}\cdot\frac{r'^3}{r^4} \qquad \ldots\ldots\ldots(8)$$

Indeed, equation (8) shows the electrostatic potential, at any point ($\vec{r}$) in the space of SEM chamber, due to a volumetric spherical distribution of Q_t charges embedded in the bulk material of the sample. The main task of this article is to find a relationship between 1/r as a function of scanning potential in which what so called "Mirror plot Figure". Therefore, the point charge approximation i.e. the first term in equation (8) can be written as follows;

$$\frac{1}{r} = \frac{4\pi\varepsilon_o V_{sc}}{KQ_t} = \frac{1}{R} \qquad \ldots\ldots\ldots(9)$$

Where the sample ($U(\vec{r})$) and scanning (V_{sc}) potentials have been equated at this coordinate point $\vec{r}$. i.e. at such a point the potential of incoming electrons become equivalents to the sample potential and hence they are reflected back toward the inner walls of the chamber producing mirror image. Actually, R in equation (9) represent the radius of an equipotential surface of a potential V_{sc} due to a trapped charge Q_t in a dielectric sample of permittivity ε_r, which in this case equal to the actual Gaussian surface radius r.

The using of equation (9) in equation (8), considering only the first two terms, the following formula can be set up;

$$\frac{4\pi\varepsilon_o V_{sc}}{KQ_t} = \left[\frac{1}{R} - \frac{3r'}{8R^2}\right] \qquad \ldots\ldots\ldots(10)$$

The right hand side of the last equation refer to the reciprocal of the actual radius of the Gaussian surface, i.e.$1/r$. However, it is being corrected now by an amount equal to $3r'/8R^2$ considering the dipole term. So this equation may be written as follows;

$$\frac{1}{r} = \left[\frac{1}{R} - \frac{3r'}{8R^2}\right] \qquad \ldots\ldots\ldots\ldots(11)$$

Equation (11) reveal the correction of mirror plot equation up to the first order. Anyway, the above manipulation in principle provide a correction approach for mirror plot formula up to any required order. Therefore, the mirror plot

correction up to the nth order, in sense of equation (8) remembering the uniform distribution of charges through the volume V', can be expressed as follows;

$$\frac{1}{r} = \left[\frac{1}{R} - \frac{3r'}{8R^2} + \frac{r'^3}{16R^4} + \ldots + \frac{1}{V'R^{n+1}} \int_{V_1'} (r')^n P_n(\cos\vartheta')dV'\right]$$

$$\ldots\ldots\ldots\ldots(12)$$

3. Results and Discussion

Actually one may summarize the ideas presented in the previous section by two different forms for the distribution profile of the specimen's surface potential. Indeed these

forms are included in equations (12). For simplicity these models, that present work is mainly concerned with, can be extracted individually, to be as follows;

$$\frac{1}{r} = aV_{sc} \qquad \ldots\ldots\ldots\ldots(13)$$

$$\frac{1}{r} = aV_{sc} - a^2(3r'/8)V_{sc}^2 + a^4(r'^3/16)V_{sc}^4 \qquad \ldots\ldots\ldots\ldots(14)$$

Where a refers to the expression; $4\pi\varepsilon_o / KQ_e$. Indeed, such distributions could simulates the accumulation of the charges over the surface of the sample and its related potential of course. These models, however, needs to be verified, regarding experimental data, to determine the appropriate format for each of these formulas at a specific circumstance. Therefore, some of the experimental data that published in appropriate literatures have been adopted to attained that.

Therefore, according to the literature [13] a sample of Poly Methyl Methacrylate (PMMA), with the dielectric constant 2.6, is irradiated by an electron beam of different accelerating potentials namely (25, 30, 35 and 39 kV) for a same period of time. The accumulation of charge is assumed to extent over a hemispheroid profile of radius r'. The experimentally measured trapped charges and their corresponding penetration depths are recorded to be as shown in Table-I.

Table-I: Trapped charge and penetration depth for various irradiation potential [13].

V_i (kV)	25	30	35	39
Q_t (nC)	0.064	0.082	0.108	0.126
r' (μm)	13.7	17.7	23.1	29.3

Figure (1) represents a typical mirror plot of this circumstance as it reported by the mentioned reference. Actually, curves in this figure represent an experimental points that fitted by means of the following equation [13];

$$V_{sc}(r) = \frac{K_{exp}Q_e}{4\pi\varepsilon_o r} - \frac{(\varepsilon_r + 2)Q_e}{32\pi\varepsilon_o\varepsilon_r} \cdot \frac{r'}{r^2} \qquad \ldots\ldots\ldots\ldots(15)$$

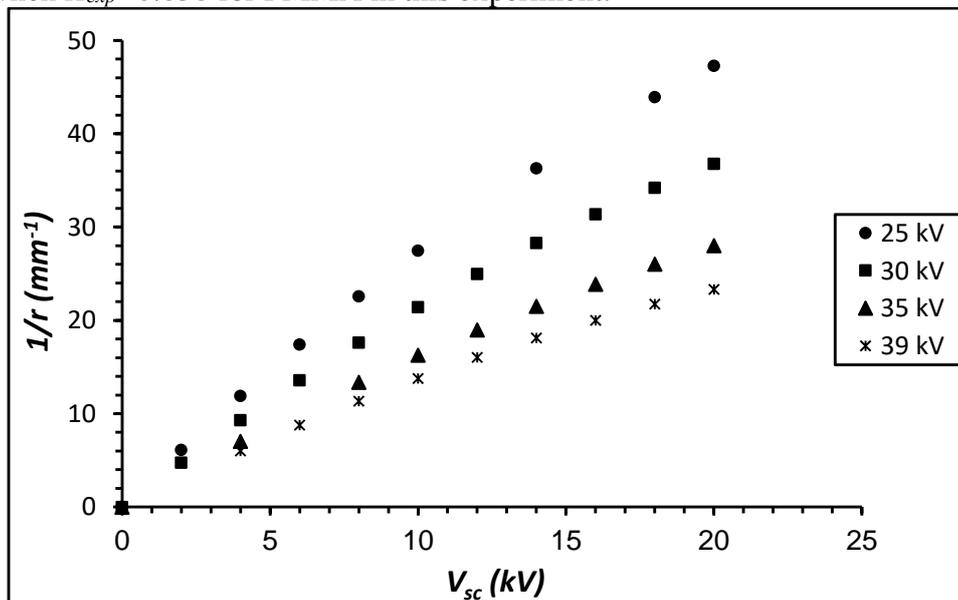For the case when $K_{exp}=0.656$ for PMMA in this experiment.

Fig. (1): Experimental mirror plot for different accelerated potentials [13].

Figure (2) describe a simulation for these curves of mirror plot using equations (13). It is quite obvious that equation (13) does matches with the experimental data, except for the values of $V_{sc}<5$ kV. Such result apparently indicate that the approximation, which suggest the trapped charge accumulated as charged-point at the sample surface, being fails as long as the scanning potential increases. In other word, the dimensional distribution of the trapped charge can never being neglected when a higher scanning potential is used to receive the electron mirror image. Indeed, this result become in parallel with those may be found in [23; 24; 25 and 19]. Alternatively, terms of higher orders, must be take into account in order to get a real simulation for the trapped charges distribution.
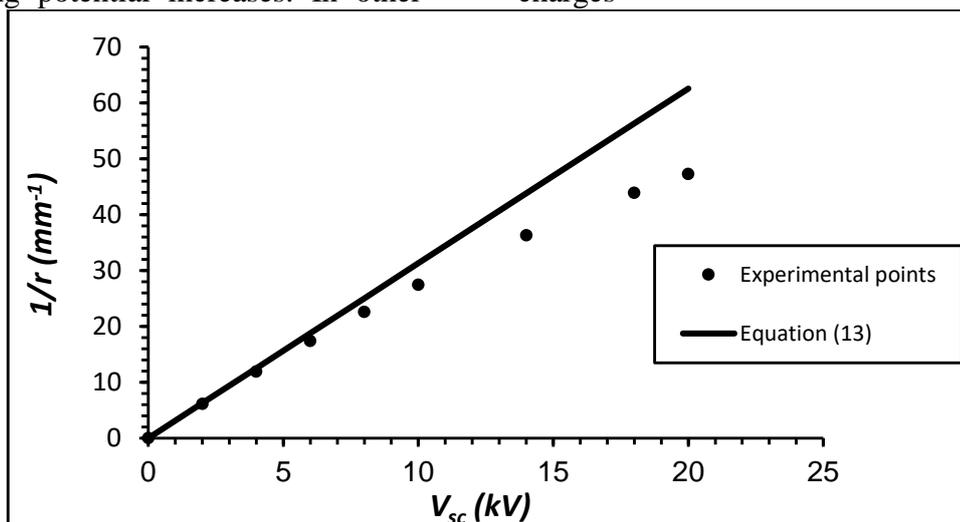

Fig. (2a): Variation of *1/r* as a function of the scanning potential for the irradiation potential 25 kV.
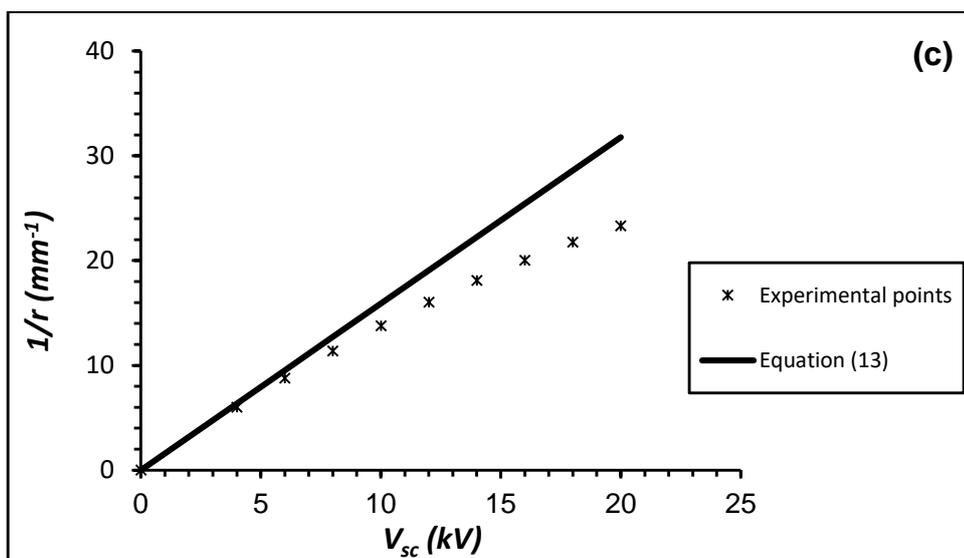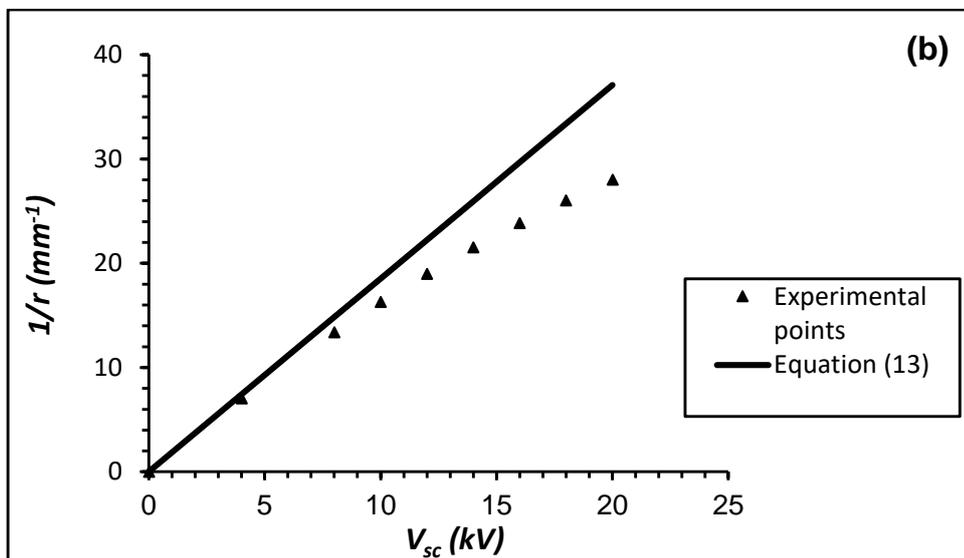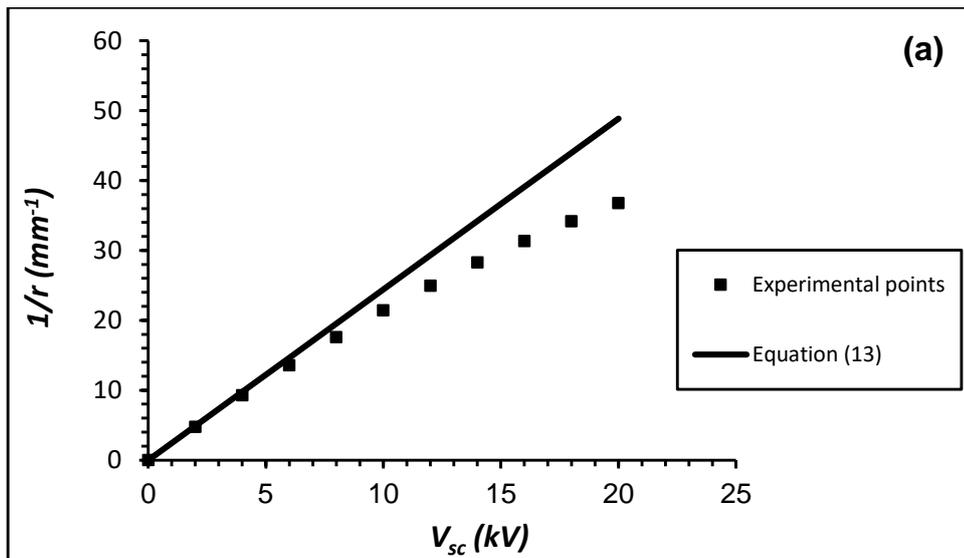
Fig. (2b): Variation of *1/r* as a function of the scanning potential for the irradiation potential a) 30 kV, b) 35 kV and c) 39 kV.

The mirror plot curve shown in figure (1) is simulated again by means of equation (14), for this case instead of equation (13), and the result presented in figure (3), where r' in which represents the radius of irradiated area and takes approximate values 10.6, 14.4, 18.6 and 22.3 μm, respectively, for each irradiated potential. Concerning with equation (14) it is seen that this sort of models reveals excellent matching with the experimental points for all of the values of the scanning potential and all of the considered irradiated potential. Therefore, the uses of potential expansion up to third order, gives raise to well approximation for the sample potential. Indeed, such announcement lay with the framework by means the experimental point being outcome.
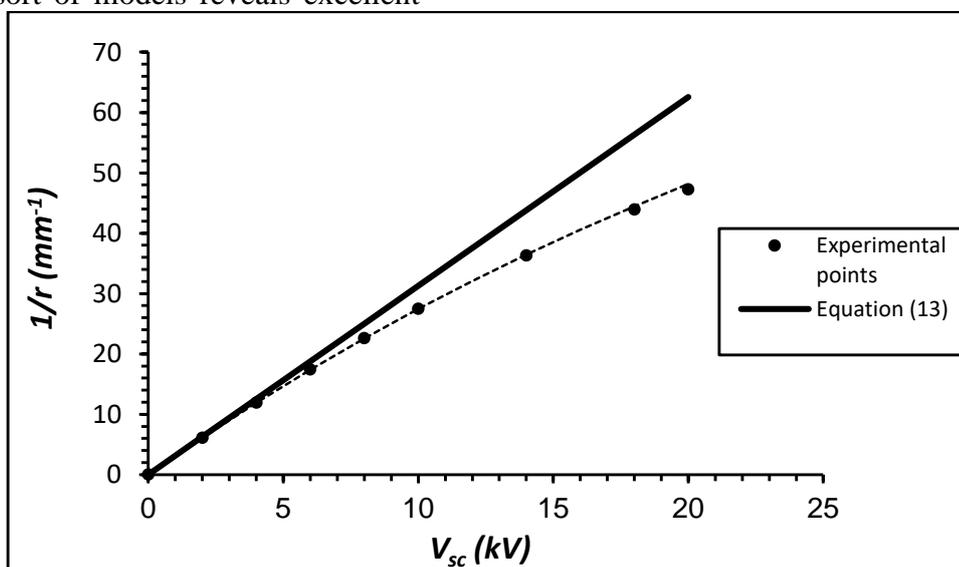

Fig. (3a): A simulated mirror plot by means of equations (13) and (14) for a PMMA material irradiated by a potential 25 kV.
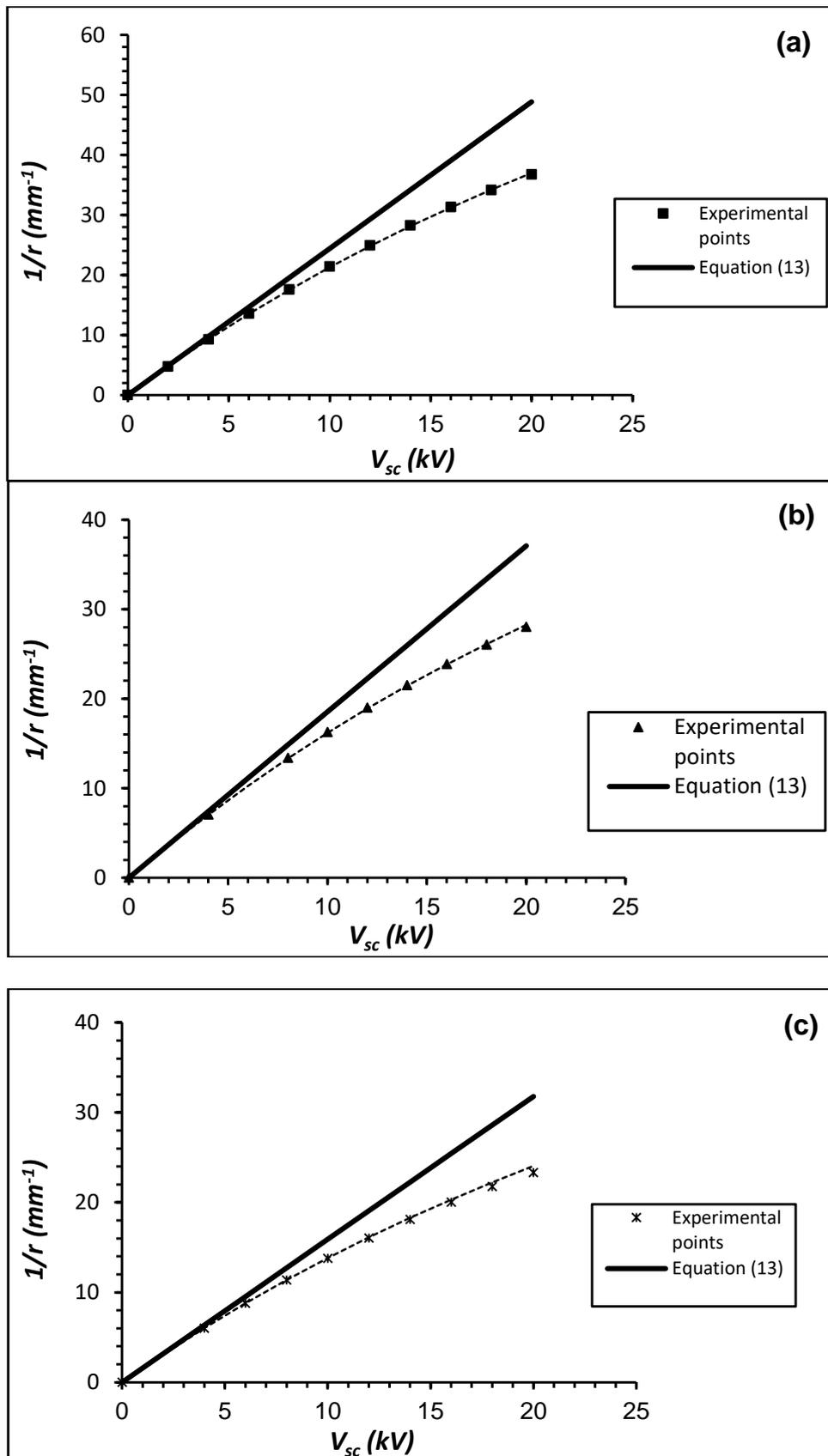
Fig. (3b): A simulated mirror plot by means of equations (13 and 14) for a PMMA material irradiated by a potential a) 30 kV, b) 35 kV and c) 39 kV.

4. Conclusion

It can be considered that the experimental mirror plot curves as an evaluation scale for the quality of the mirror image. Therefore, it had been used the suggested model of this work (i.e. multipolar expansion) to obtain an excellent matching for any experimental curve. However, the contributing of higher order poles term to the total potential will be decreased as the increases of poles number. Hence, it can be said that the higher order poles than octopole does not valid anymore, because the effect like this on the potential becomes not perceptible. Actually, the divergence of the experimental points from the linear behavior will be increased whenever the radius of the irradiated area and verse versa. Therefore, this parameter can be the most important rule which control to the shape of mirror plot.

5. Reference

[1] Rau E. I., Fakhfakh S., Andrianov M. V., Evstafeva E. N., Jbara O., Rondot S., and Mouze D., (2008), Nucl. Instr. and Meth. in Phys. Res. B, Vol. 266, pp. 719-729.

[2] Okai N., and Sohda Y., (2012), Jpn. J. Appl. Phys., 52: 06FB11(7pp).

[3] Oatley C. W., Nixon W. C., and Pease R. F. W., (1965), Advances in Electron Physics, Vol. 21, pp.181-247.

[4] Sikorski J., Moss J. S., Newman P. H., and Buckley T., (1968), J. Phys. E: Sci. Instum., Vol. 1, pp. 29-31.

[5] Clark D. R., and Stuart P. R, (1970), j. Phys. E: Sci. Instrum., Vol. 3, pp. 705-707.

[6] Khaleel I. H., (2013), "*An Attempt for Inspection of Electrons Behaviour inside SEM Concerning Mirror Effect Phenomenon*", Ph.D. Thesis, College of Education, University of Al-Mustansiriyah, Baghdad, Iraq.

[7] Milani M., Savoia C., and Bigoni D., (2009), Proceedings of ITP2009, Interdisciplinary Transport Phenomena VI: Fluid, Thermal, Biological, Materials and Space Sciences, Volterra, Italy.

[8] Milani M., Abdul-Wahab H. N., Abbood T. H., Savoia C., and Tatti F., (2010), Micr.: Sci., Tech., Appl. and Edu., pp 1741-1754.

[9] Abbood T. H., (2011), "*Formal Investigation of the Mirror Effect in SEM*", Ph.D. Thesis, College of Education, University of Al-Mustansiriyah, Baghdad, Iraq.

[10] Song Z., Ong C., and Gong H., (1996), J. Appl. Phys., Vol. 79 (9).

[11] Gong H., Le Gressus C., Oh K. H., Ding X. Z., Ong C. K., and Tan B. T. G., (1993), J. Appl. Phys. Vol. 74, No. (3), pp. 1944.

[12] Gong H., Taijing L., and Ong C. K., (1994), J. Appl. Phys., Vol. 76, No. 2, p.p 1352-1354.

[13] Chen H., Gong H., and Ong C. K., (1995), J. Appl. Phys. Vol. 78, No. 6, p.p. 3714-3718.

[14] Bigarré J., and Hourquebie P., (1999), J. Appl. Phys., Vol. 85, No. 10, pp. 7443-7447.

[15] Belhaj M., Jbara O., Odof S., Msellak K., Rau E. I., and Andrianov M. V., (2000), Scanning, Vol. 88, pp. 352.

[16] Jbara O., Fakhfakh S., Belhaj M., Rondot S., (2004), Microsc. Microanal. Vol. 10, pp. 697.

[17] Croccolo F., Riccardi C., (2008), Microscopy Today, Vol. 16, pp. 40.

[18] Hattali M. L., Mesrati N., and Treheux D., (2012), J. Europe Ceram. Soc., Vol. 32, pp. 717-725.

[19] AL-Obaidi H. N. and Khaleel I. H., (2013), Micron, Vol. 51, p.p. 13-20.

[20] Reitz J. R. and Milford F. J., (1960), "*Foundations of Electromagnetic Theory*", ADDISON-WESLEY PUBLISHING COMPANY, INC., USA.

[21] Jackson J. D., (1999), *"Classical Electrodynamics"*, Third ed., John Wiley, New York.

[22] Greiner W., (1998), "*Classical Electrodynamics*", 1st ed., Springer- Verlag New York, Inc.

[23] Vallayer B., Blaise G., and Treheux D., (1999), Rev. Sci. Instr., Vol. 70, No. 7, pp. 3102.

[24] Wintle H. J., (1999), J. Appl. Phys., Vol. 86, No. 11, pp. 5961-5967.

.

[25] Mady F., Renoud R., Attard C., Bigarré J., Ganachaud J. P., and Hourquebie P., (2002), Eur. Phys. J., AP 20, pp. 41-53.