

New way to overcome on the mirror effect for imaging the surface of insulator

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Abstract: This work use the ion beam of a focused ion beam (FIB)/scanning electron microscope (SEM) microscope to investigate the ion mirror effect (IME) on the poly-methyl-methacrylate (PMMA). From study of parameter that influence the ion mirror effect (IME), we find easy and fast way to imaging the surface of the sample. To our knowledge this is the first observation of what can be called 'Imaging by Different Currents (IDC)'. This way is based on the measurement of the trapped charge (Q_t) as well as to evaluate the charge landing (Q_{sc}) through imaging the ion mirror effect (IME) by using different currents (I_{sc}). We observed begin overcome on the mirror effect when $Q_{sc} \geq Q_t$ and it allows to imaging the surface of insulator. This way (IDC) enables of imaging the surface of the sample in all the scanning potential of imaging (V_{sc}), all the scanning potential of irradiation (V_i) and also at any time radiation (t).

Keywords - Anomalous contrast, Focused Ion Beam (FIB), Scanning Electron Microscope (SEM), Electron Mirror Effect (EME), Pseudo mirror Effect (PME), Ion Mirror Effect (IME) and Trapped charge.

I. Introduction

The term "electron optics" refers to the study of the behavior of electron beams in electrostatic or magnetic fields, especially when the beams are use for the purpose of optical imaging; it is so named in analogy to light optics [1]. The electron optics experienced a rapid development stimulated by strong industrial needs, especially electron microscopes, cathode ray tubes, plasma generation, accelerators and television. The idea of developing an electron microscope, for investigating compact or bulk sample, had recognized by Max Knoll in 1935 through his pioneering work to set up the first original design for scanning electron microscope (SEM). The design was further advanced by Manfred von Ardenne to create an electron probe microscope in 1938. That used electro-optical lenses to focus the beam [2]. Actually this idea originated from the fact that resolution-limiting chromatic failures do not play a big role in such a microscope. Then the first scanning electron microscope was developed by Charles Oatley around 1953, and the first commercial SEM model was marketed in 1965 by the Cambridge Instrument Company [3].

Nowadays, the SEM is without doubt one of the most widely used in many fields of research and industrial production throughout the world. SEM combines high-resolution imaging with a large depth of field. In addition, the strong interaction of electrons with matter produces a wide variety of useful 'signals' that reveal all kinds of secrets about matter at the microscopic and even nanoscopic level [4]. The SEM is one of the most powerful tools to investigate the microscopic characteristics of a wide variety of samples. Typical applications of these machines involve conductive samples. In fact, when a conductive sample is irradiated with electrons or ions, the excess of charge quickly flows away from the beam spot. In contrast, in a non-conductive sample, an excess charge is spatially trapped within the sample generating distortions in the beam path. In this condition an anomalous contrast can be detected [5]. 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 [6]. Similar image have also been observed by Shaffner et al., and Le Gressus et al., are realized to be attributed to the deflection of the primary electron by electrostatic (negative) field formed on the insulator surface [7, 8]. Clark and Stuart were first who used the term "electron mirror" when they investigated the charging effect. It is proposed that the image observed is the result of a two-step process. The first one is the charging up of part of the surface of the material until it acts as an electron mirror of sufficient strength to deflect the electrons of the primary beam towards the electron collector. The second step is the building up of an image of the detector as the reflected beam scans over it [9]. Electron mirror effect (EME) is a physical phenomenon occurs inside the chamber of the SEM when the irradiating electron beam scans a dielectric sample. For seemingly inexplicable reasons, an image of the interior of the SEM appears instead of the image of the surface of interest [10]. However, the physics behind mirror effects is that when an insulating sample is irradiated in a first phase in a SEM at a high voltage, Negative charges will trapped in the insulator sample during the injection process. The trapped charges produce an electric field in the vacuum chamber of the SEM. If the sample is observed later (in a second phase), with a lower energy electron beam, the electric field can be strong enough to deflect the probing electron in the same manner as a convex mirror dose with light [11]. Recently a similar effect involving ions in a focused ion

beam microscope (FIB) has been observed by [5]. The electron mirror phenomenon in a scanning electron microscope (SEM) system has been reported by electron microscopists since the 1970s. For seemingly inexplicable reasons, instead of imaging a surface of interest, an image of the interior of the SEM appears. Traditionally, much effort has been extended to make sure this effect does not occur [5]. (10)

II. Experimental Procedure

1. The FIB/SEM system

All the results presented in the paper were obtained by a FIB/SEM (FEI, Quanta 3D) microscope. The dual beam is a kind of scanning electron microscope with an electron and ion columns mounted into the same specimen chamber. The electron beam being vertical while the ion beam held up off vertical by 52 degrees. Both beams are aiming at the same point (coincidence point) on the specimen surface and the system is engineered to work with all possible accessories at the coincidence point, without affecting the performances of both columns. Actually, it is a combination of two systems as shown in fig. (1) [12]. The Focused ion beam (FIB) is a tool that performs basically three functions: ion imaging (secondary electrons or ions from primary ions), milling (precision down to 10 nm) and deposition (with the insertion of a small needle, delivering special gases).

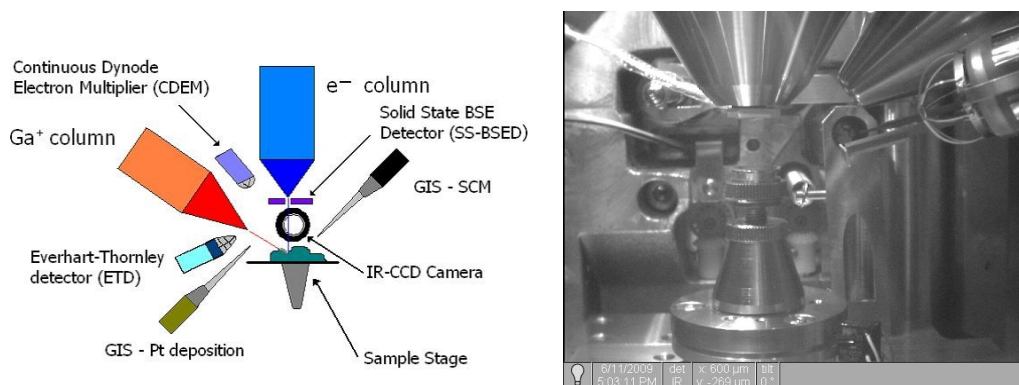


Fig.1.(a) A scheme of the components of the FIB-SEM microscope, **(b)**. CCD optical image of the specimen chamber.

2. Mirror Materials

Plastic samples are inexpensive and easy to find. As a result, they make an ideal insulating material to synthesize an electron mirror. Sample of poly-methyl-methacrylate (PMMA), Flat sample of this plastic material was collected as well as Teflon™ (Poly-tetra-flour-ethylene (PTFE)). It is important to recall that an electron mirror does not have to be an insulator as long as it's well insulated from ground.

3. Charging the Mirror

When an insulating sample is irradiated in a first phase in a SEM at high voltage selected as; $10 < V_i < 30$ kV. Negative charge trapped in the insulator during the injection, Q_t , produce an electrical field in the vacuum chamber of the SEM. If the sample is observed later (in a second phase) with a lower energy electron beam (defined by imaging scanning potential which can vary from a few hundreds to a few thousands of volts), the electric field can be strong enough to deflect the probing electron in the same manner as a convex mirror does with light. The amount of trapped charge must be carefully chosen: high enough to produce low-energy electron beam deflection, but not too high in order to avoid damage under electron beam irradiation [13, 14]. The effect of the trapped charge on incoming electrons is repulsion, so that electrons with sufficiently small kinetic energy are reflected back to different points of the SEM chamber, depending on the incoming direction and beam parameter. The consequent interaction between these electrons and various instruments, objects, and the inner walls of the SEM chamber can generate secondary electrons, which characteristics depend on the chamber materials and primary electron energy. The secondary electrons are then detected by the active detector within the chamber to form the mirror image of the SEM chamber itself as illustrated schematically in fig. 2.

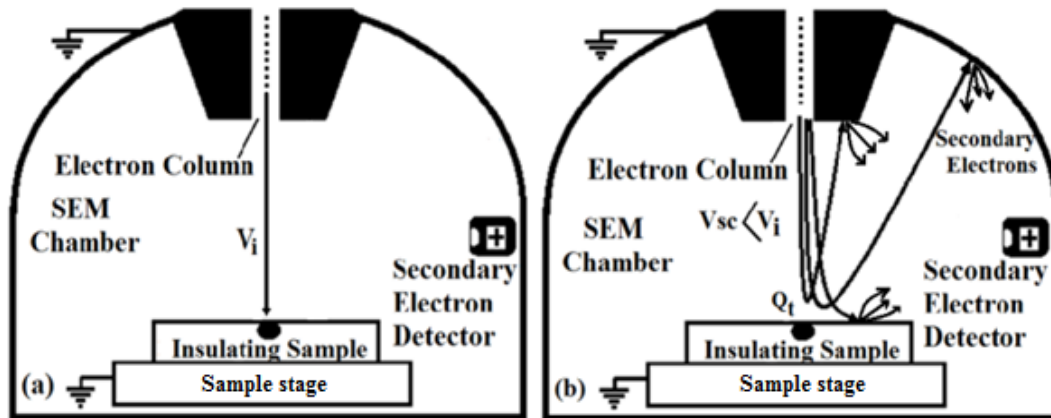


Fig. 2. (a) Schematic illustration of the charging process, (b) the SEM mirror image formation.

4. Detector Considerations

The choice of detector drastically effects the type of output experienced when using an electron mirror, it is seen that, electron–matter interaction can lead to the emission of secondary electrons (SE) and backscattered electrons (BSE). These are distinguished by their energy, with electrons having energies $<50\text{eV}$ being considered as SE, while those with energies close to that of the incident electron beam are labeled BSE. Both can be used in the imaging process in the scanning electron microscope, while several different types of detectors are required to differentiate between them [15].

III. Results And Discussion

1. Imaging the Chamber

Following the procedures outlined above, ion or electron mirrors were created using various plastic samples as well as plastic samples sputter coated with metal. In Fig.3 an IME image is obtained after saturation of the sample by irradiation with 30kV ions and successive observation with 5 kV ions. The mirror image is compared to a photograph of the inside of the FIB/SEM. It's easy to recognize the main elements of the machine: namely, the two columns (electron and ion), the four detectors, an Everhard-Tornley Detector (ETD), a channel detection electron multiplier (CDEM), a solid state back-scattered electron detector (SSBSD) and a large field detector (LFD), and also one of the two injectors (one for platinum deposition and the other for selective carbon milling).

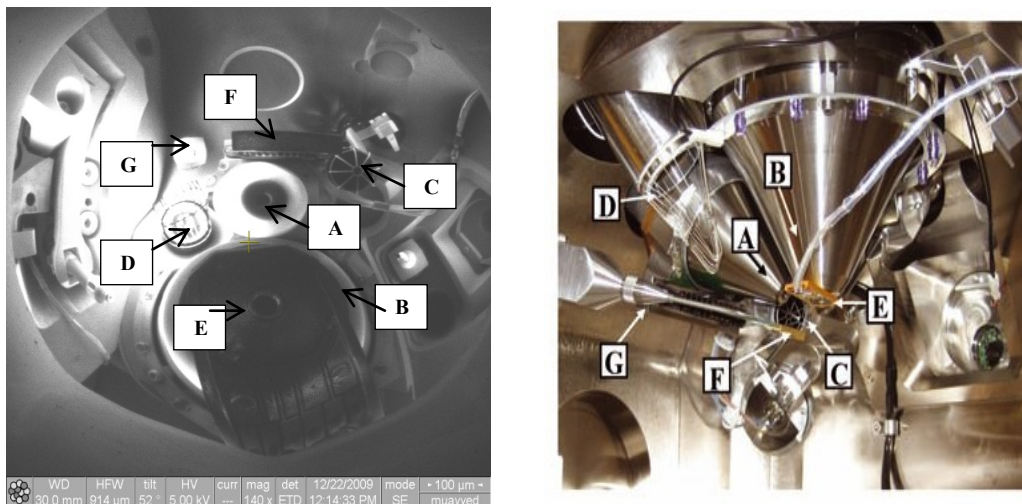


Fig.3. IME image from a PMMA sample vs. photograph of the FIB/SEM chamber. In both cases the principal components can be clearly identified: (A) iongun, (B) electron gun, (C) CDEM sensor, (D) ETD sensor, (E) SSBSD sensor, (F) LFD sensor, (G) platinum injector.

Ions gradually accumulate on the sample surface until they reach a high density and an electric field is established on the surface, generating a charged layer. Consequently when the energy of the primary beam is lowered then the electrical field energy from the charged layer may be higher than the actual primary beam one and prevents the charged particles from reaching the sample surface that acts as a mirror and “reflects” (or, to

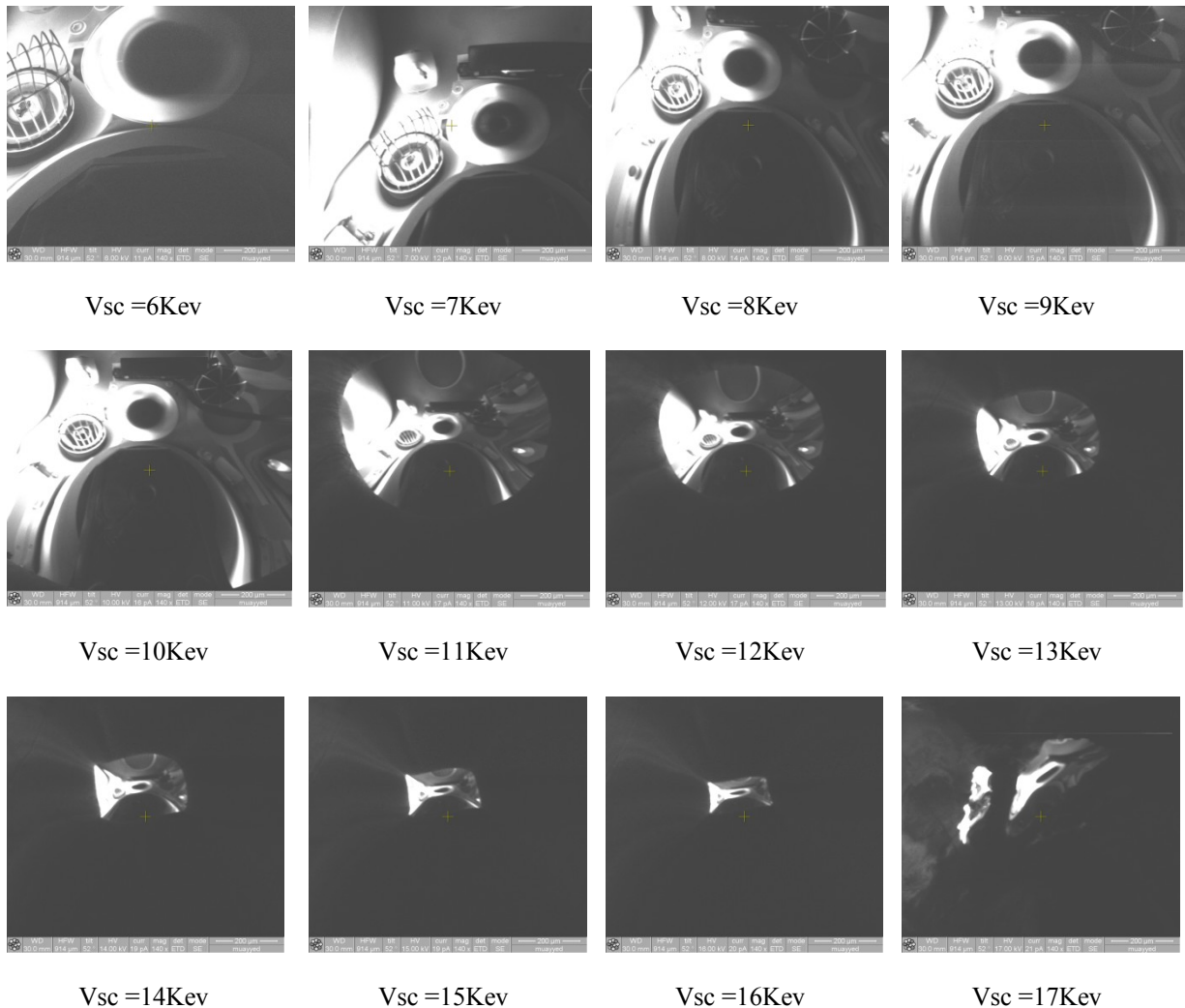
say better, “redirect”) ions somewhere else in the specimen chamber. The inner part of the specimen chamber is therefore imaged. The phenomenon (originally termed “anomalous contrast”) was explained in terms of something very close to what happens to photons hitting an optical mirror (Ion Mirror Effect-IME and Pseudo Mirror Effect-PME – when a mirror image is superimposed, or “mixed” to sample image).

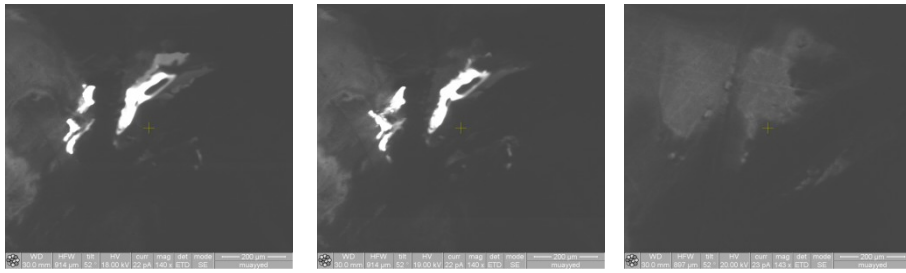
This effect (ion mirror effect or electron mirror effect) is a possible analytical tool for imaging or obtaining information about sample dielectric properties as used by [16, 17]. On the other hand Much effort has been devoted to prevent the appearance of this effect since it is detrimental of the image quality. The present work focus on imaging the surface sample use Imaging by Different Currents (IDC) way.

2. Imaging the sample

It possible is imaging the surface of insulator by using a relatively low scanning potential also at short time radiation [17]. In the present work was the study of the ion mirror effect (IME) as follows:

The sample (poly-methyl-methacrylate (PMMA)) was first irradiated with $V_i = 30\text{kV}$ ions with a current of $I_i = 30\text{pA}$ for a total time of about $t = 300\text{s}$ providing an injected charge of about $Q_{inj} \approx 9\text{nC}$. The subsequent image has been taken with different scanning potential (V_{sc}) with a different current (I_{sc}) for a total time of $t = 304\text{s}$. Where the parameters of this irradiation are: magnification is $\times 140$, Dwell Time = $3\mu\text{s}$, $\text{HFW} \approx 914\mu\text{m}$, voltage grid 250, Tilt = 52° and $\text{WD} = 30\text{mm}$. Here, the radius of the charged zone has been considered $R \approx 0.457\text{mm}$, corresponding to half the size of the image during the charging procedure. The fig. (4, 5) show the results of this study.





Vsc =18Kev

Vsc =19Kev

Vsc =20Kev

Fig. 4. Ion mirror images captured at different scanning potential capture when PMMA sample irradiated by 30kV.

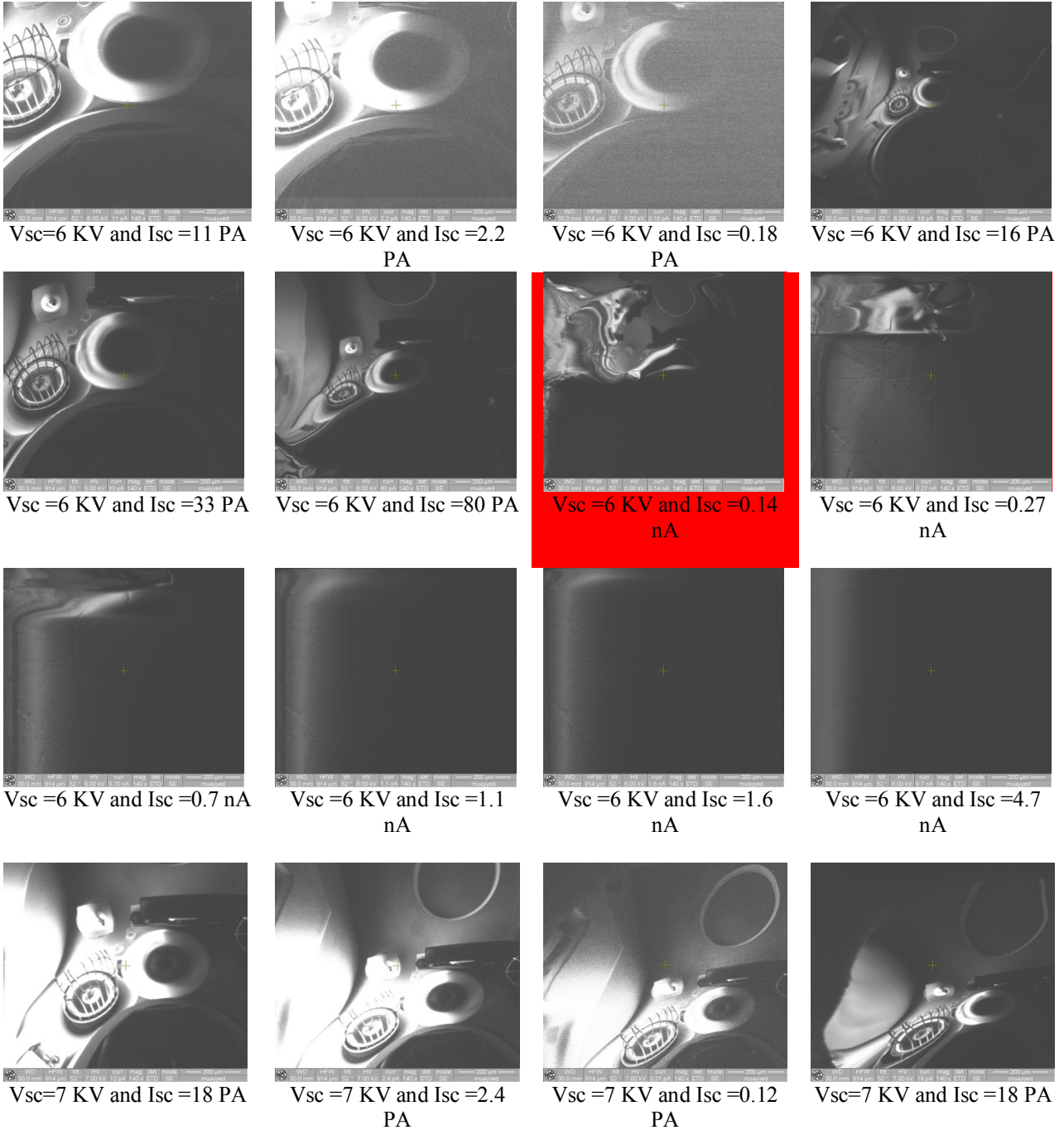
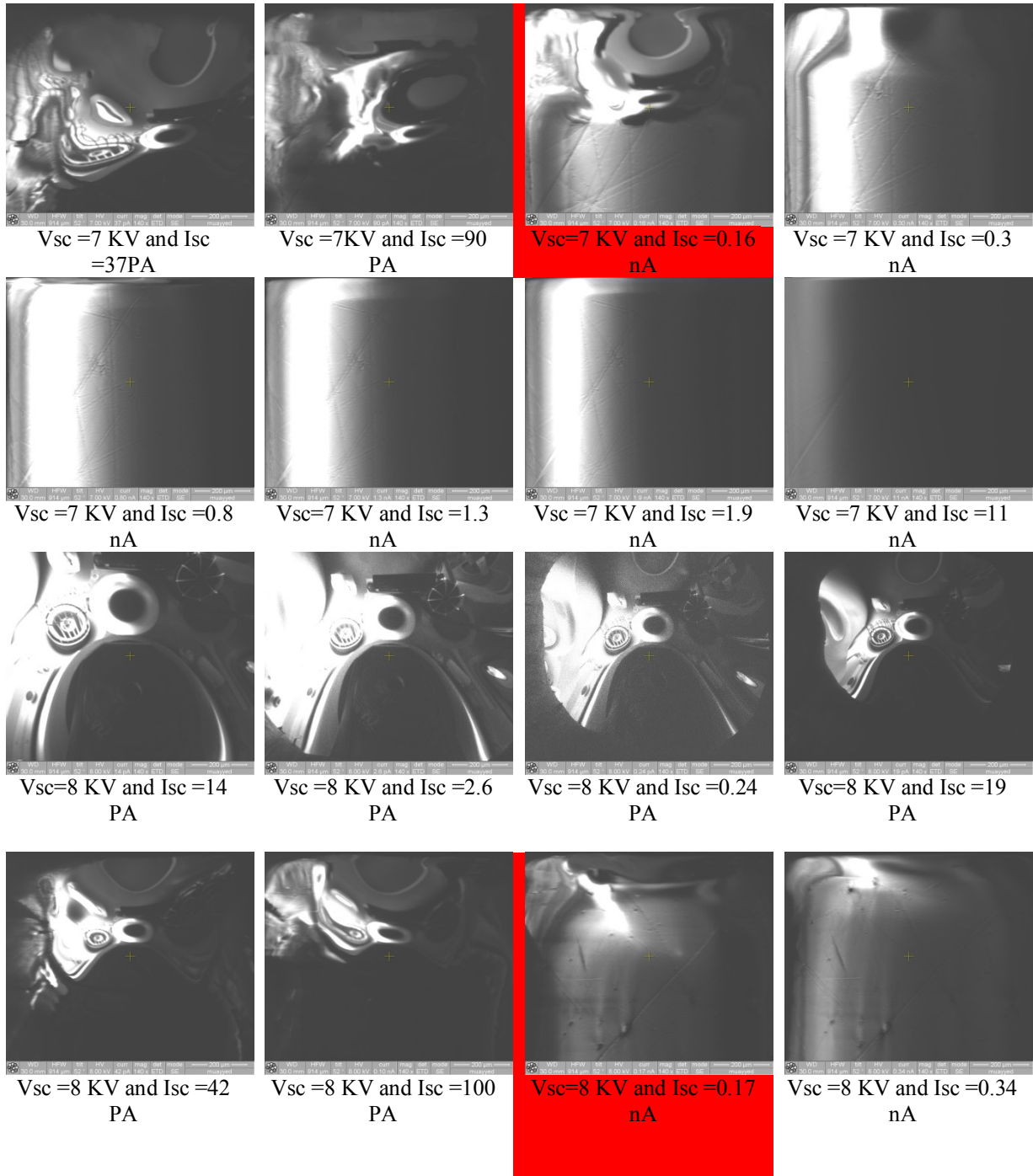
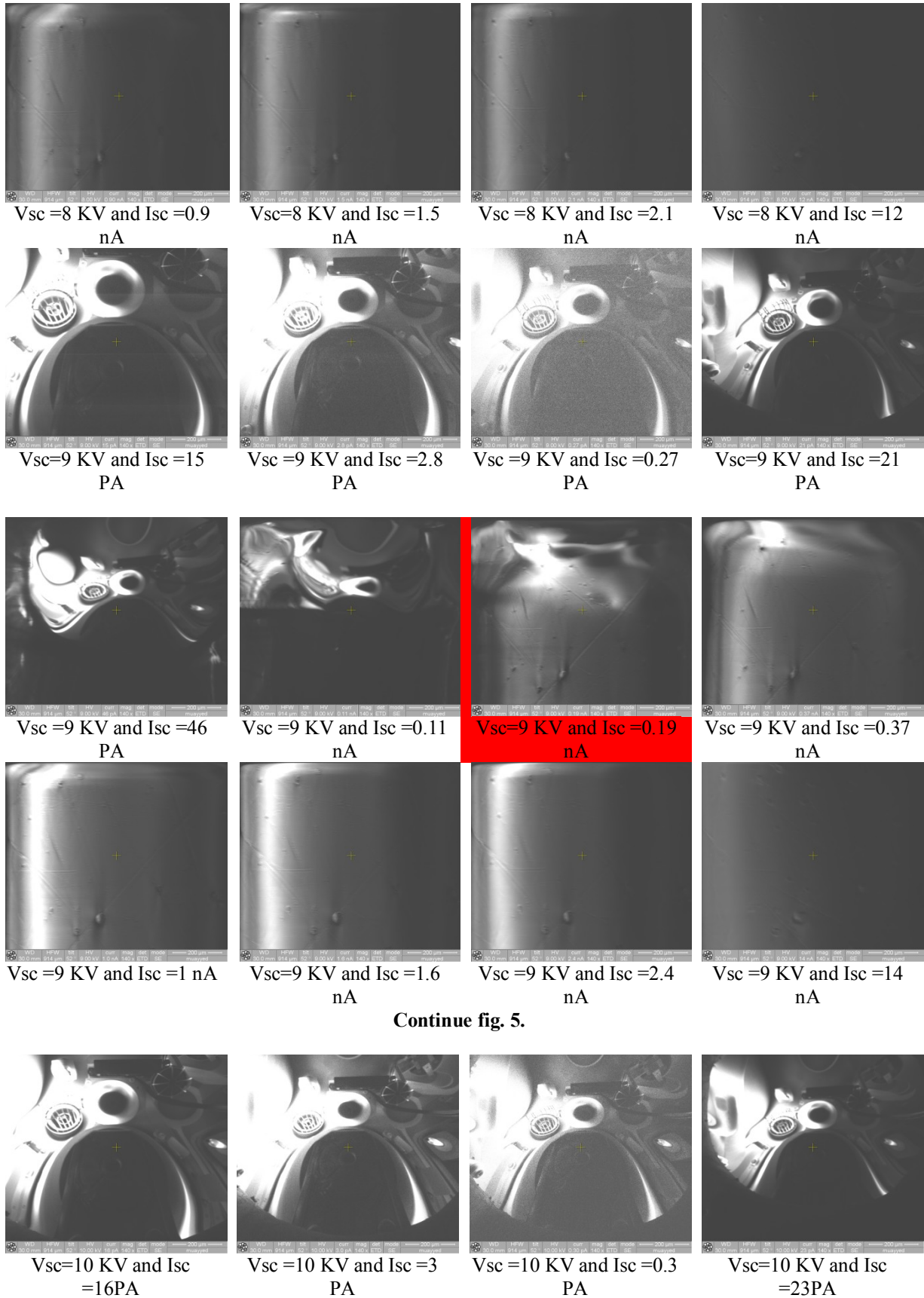
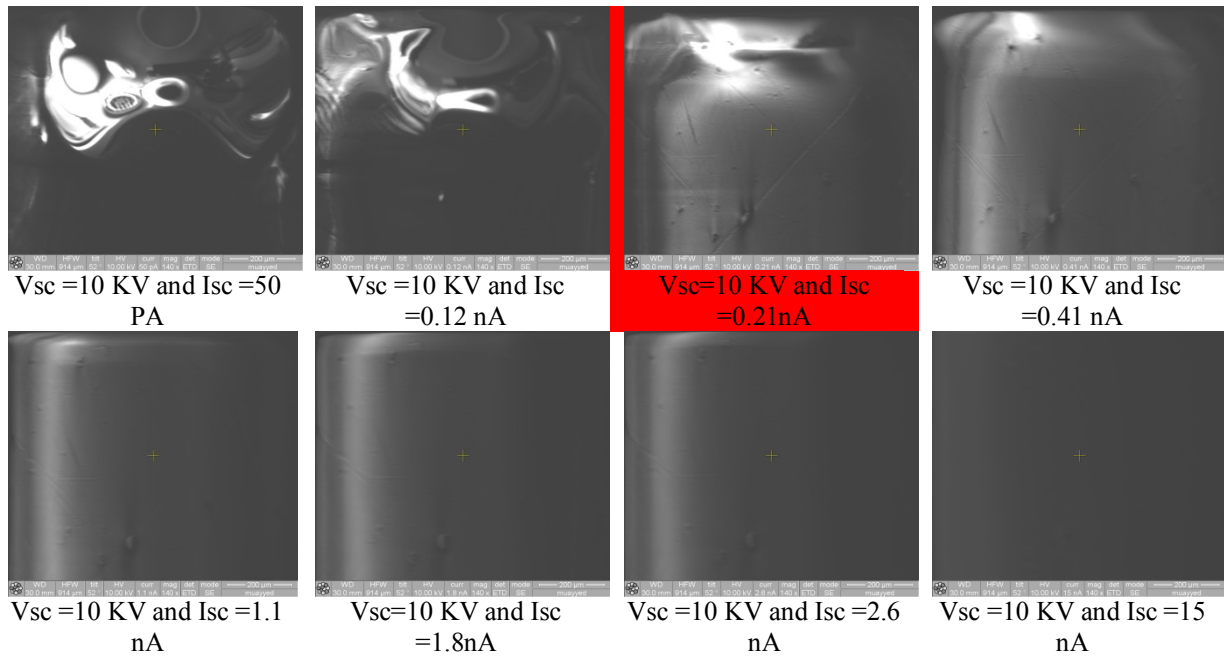


Fig. 5. Ion mirror images captured at different scanning potential and different current capture when PMMA sample irradiated by 30kV.



Continue fig. 5.





Continue fig. 5.

The determination of charge number that is trapped on the surface of insulator may lead to important information about the mirror effect phenomena and the insulator properties [16, 17]. The starting point for computing the trapped charges Q_t is the following relation [5]:

$$Q_t = 2\pi R \epsilon_s V_{sc}^{max} \quad (1)$$

Where V_{sc}^{max} is the maximum stopping potential. Figure (4) show the IME is observed to disappear for accelerating potentials larger than $V_{sc}^{max} \approx 20kV$, from which a trapped surface charge of about $Q_t \approx 508pC$ can be calculated.

Fig. (5) show the way to imaging the surface of insulator by using different current (I_{sc}) for the different scanning potential (V_{sc}). We observe from through shapes at increasing in the number of ions landing, the mirror effect begins distorted into disappear and we can get the imaging to the sample surface.

Regions shaded in red color shows the initial values of the currents that lead to imaging the surface. It is possible to calculate the value of the charge landing for these regions through the following equation:

$$Q_{sc} = I_{sc} \times t \quad (2)$$

Where Q_{sc} is the charge landing, I_{sc} is the different current using capture imaging and t is the time necessary to capture imaging in the present working value of four seconds.

Table1. Values of charge landing (Q_{sc}) for regions shaded in red color at different scanning potential.

V_{sc} KV	I_{sc} nA	Q_{sc} pC [Eq.(2)]
6	0.14	560
7	0.16	640
8	0.17	680
9	0.19	760
10	0.21	840

Table (1) shows the values of the charge landing (the number of electrons or ions landing through the unit time) where we note that these values are greater than the value of the charge trapping on the sample surface (i.e. $Q_{sc} > Q_t$). Therefore the number of ions (Q_{sc}) landing overcome ions trapped on the sample surface (Q_t) and then be able to these ions of access to the surface of insulator. In other words, the electric force landing overcome charge builds up on the sample (overcome on the force of repulsion between the ions in flight and the ions trapped on the sample). therefore these ions are able on the deformation the mirror effect into disappear and imaging the surface of insulator as shown in the fig. (5).

IV. Conclusions

The influences on the ion mirror effect (IME) exerted by the different scanning potential and the different current imaging, allow us to reach the following general conclusions: The Imaging by Different Currents (IDC), way described here permits to imaging the surface of insulator in any experimental conditions. We found it possible imaging the surface of insulator based only upon the measurement of the trapped charge (Q_t) and imaging by different currents (I_{sc}). Finally we observe when the currents are using to imaging large it can be to obtain imaging the surface of insulator clearly and overcome ion mirror effect.

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References

- [1] Al-Obadi H. N., (2009), "Electron Lenses", 2nd SUMMER SCHOOL on Electron and Ion Microscopy and Micromanipulation, 7th Sept- 6th Oct., Milano, Italy.
- [2] Michler G. H., (2008), "Electron Microscopy of Polymers", Springer-Verlag Berlin Heidelberg.
- [3] Ducati C., (2011), "Material Science: Electron Microscopy", University of Cambridge,
- [4] Stokes D. J., (2008), "Principles and Practice of Variable Pressure/Environmental Scanning Electron Microscopy", John Wiley & Sons Ltd.
- [5] Croccolo F., and Riccardi C., (2008), "Observation of the ion-mirror effect during microscopy of insulation materials", J. Microsc., Vol. 229, Pt1, pp.39-43.
- [6] Clark D. R., and Stuart P. R., (1970), "An anomalous Contrast effect in the Scanning Electron Microscope", J. Phys. E: Sci. Instrum., Vol. 3, pp. 705-707.
- [7] Shaffner T. J., and Van Veld R. D., (1971), "Charging effects in the scanning electron microscope", J. Phys. E: Sci. Instrum., Vol. 4, pp 633-637.
- [8] Le Gressus C., Valin F., Henriot M., Gatier M., and Duraud J. P., (1991), "Flashover in wide-band-gap high-purity insulators: Methodology and mechanisms", J. Appl. Phys., Vol. 69 (9), pp. 6325-6333.
- [9] Clark D. R., and Stuart P. R., (1970), "An anomalous Contrast effect in the Scanning Electron Microscope", J. Phys. E: Sci. Instrum., Vol. 3, pp. 705-707.
- [10] Milani M., Abdul-Wahab H. N., Abbood T. H., Savoia C., and Tatti F., (2010), "Rear window: looking at charged particles hitting a charged target in a FIB/SEM", Micr. : Sci., Tech., Appl. and Edu. , pp 1741-1754.
- [11] Ghorbel N., and Kallel A., (2010), "Charge Measurement in electron irradiated ceramic MgO: Induced current and Mirror effect methods", 2010 Annual Report Conference on Electrical Insulation and Dielectric Phenomena., IEEE, pp. 1-4.
- [12] Milani M, Bigoni D, and Savoia C. Electron Mirroring: Control of electron transport and understanding of physical processes from SEM images. Proceedings of ITP2009. Interdisciplinary Transport Phenomena VI, Volterra, Italy 2009 6.
- [13] Vallayer B., Le Gressus C., Blaise G., and Tréheux, D., (1994), "Theoretical possibility of the mirror method to measure breakdown relevant parameter", Electrical Insulation and Dielectric phenomena, IEEE, Annual Report Conference, pp. 761-766.
- [14] Liebault J., Zarbout K., Moya G., and Kallel A., (2003), "Advanced measurement techniques of space-charge induced by an electron beam irradiation in thin dielectric layers", Journal of Non-Crystalline Solids, Vol. 322, pp. 213-218.
- [15] Scheu C., and Wayne D., (2012), "Electron Microscopy: Application in Physics, Chemistry and Material Science", 1st ed., Wiley-VCH Verlag GmbH & Co. KGaA., pp. 16-35.
- [16] Zorry, M.J (2011), "Mirror Effect Investigation for Focused Ion Beams", Ph.D. thesis, University of Al-Mustansiriyah, College of Science, Baghdad, Iraq.
- [18] Abbood, T.H (2011), "Formal Investigation of the Mirror Effect in SEM", Ph.D.thesis, University of Al-Mustansiriyah, College of Educati