International Journal of Science and Research (IJSR)
ISSN: 2319-7064
Index Copernicus Value (2016): 79.57 | Impact Factor (2017): 7.296

Microstructural Characterization of Li-Ion Battery Separator using Scanning Electron Microscopy

Rekha L[1], Venkateswarlu M[2], Jagadish M[3]

[1, 2, 3]R&D, Technology Centre, Amara Raja Batteries Ltd, Karakambadi- 517520, Andhra Pradesh, India

Abstract: ***The present paper focus on identifying the optimal microstructure of a non conducting polyolefin separator material have been investigated by Scanning Electron Microscope (SEM) technique. In this study, experiments are conducted by varying the surface coating time from one minute to eight minutes and also varied the accelerated voltage from one to ten kilo volt in order to identify the optima conditions for desired microstructural separator material for lithium ion battery applications.From the studies, it was observed that the coating time on the specimenfromthree to five minutes and accelerating voltage appliedfrom five kilo volt to ten kilo volt was found to be preferred conditions for obtaining the desired microstructure of polyolefin based separator material.The observed SEM resultsare presented and discussed.***

Key words: Separator, Specimen Coating, Accelerating Voltage, Scanning Electron Microscope

1. Introduction

Lithium ion batteries are the preferred energy storage technology for portable applications, electrified vehicles and smart grids because of their high efficiency, high volumetric, gravimetric energy density and long cycle life [1, 2].Any batteries consists of cathode, anode, separator and electrolyte. Here the separator plays a vital role in preventing electronic contact between electrodes, in order to prevent electrical short circuits and it also functions as an ionic conductor to let ions pass freely in the charging and discharging cycles [3, 4].In general, separators are based on a homo polymer, copolymer or blends of ethylene and propylene in a number of combinations between high density polyethylene (HDPE) and ultrahigh molecular polyethylene.The manufacturing micro porous polyolefin can be divided into the dry process and the wet process[5]. Depending on the membranes and manufacturing process, the noticeable difference in the morphology is clearly visible.

The main aim of the study is to improve the microstructure quality of polyolefin based lithium ion separator. The importance of microstructural characterization of the materials has a strong influence on the battery performance alongside to the geometrical aspects. The key parameters of the microstructure of the battery separator are the shape, size and uniformity of the pores. Separators are made up of different materials and methods. For liquid electrolyte lithium ion batteries, it is broadly classified into microporous membranes, non-woven mats and composite membranes. Each separator type has inherent advantages and disadvantages which also influences the performance of battery [6].

Thus, by understanding the importance of microstructure of the separator and its influence with the surface coating time of the specimen and also the effect of accelerated voltage of the morphology of the separator material investigated by the SEM technique. The observed SEM results on the non-conducting separator isanalysed and consideringthe surface coating time andaccelerating voltages in order to identify the optima conditions of the microstructure of the separator arepresented and discussed.

2. Experimental

2.1. Sample Selection

Commercial sample of 20 micron thickness, biaxial oriented micro porous membrane separator is chosen for this study, and the characteristics of the separator are given in Table 1.

Table 1: Separator characteristics

Separator characteristics	Unit	Value
Thermal shrinkage (MD)	%	8
Thermal shrinkage (XTD)	%	1.5
Basis weight	g/m^2	10.5
Air permeability	Seconds	340
Tensile strength (MD)	kg/cm^2	875
Tensile strength (TD)	kg/cm^2	650
Tensile elongation at break (MD)	%	200
Tensile elongation at break (MD)	%	300
Porosity	%	41
Puncture resistance	Grams	450

The microstructure of the separator sample recorded by Scanning Electron Microscopy (hereafter referred as SEM).The SEMcan produce magnified images and in situ chemical information from any type of specimen. The imaging system depends on the specimen being sufficiently electrically conductive to ensure that the bulk of the incoming electrons go to ground.The formation of the image depends on collecting the different signals that are scattered as a consequence of the high energy beam interacting with the sample.

Backscattered electrons and secondary electrons are generated within the primary beam, sample interactive volume and these are the two principle signals used to form images.The backscattered electron coefficient increases with increasing in atomic number of the specimen, whereas the secondary electron coefficient is relatively insensitive to the atomic number. This fundamental difference in the two signals can have an important effect on the way of samples

to be prepared.The analytical system depends on collecting the X-Ray photons that are generated within the sample as a consequence of interaction with the same high energy beam of primary electrons that is used to produce images [7].

The SEM is designed to obtain information from the surface of specimens and this information may be obtained either from the natural surface of the specimen or from a surface that has been exposed by artificial means to reveal the interior of the sample. A quick decision need to be made early in the process of sample preparation about the proposed examination of the specimen. There are two approaches to deal the frequent impasse that may exist between the properties of the sample and the optimal operating conditions of the SEM. Sample preparation is an absolute prerequisite for microscopy and analysis as every specimen that goes into the SEM, needs some method of sample preparation. To obtain optimal image and analytical data from non conductive specimens, the whole sample need to be conductive.

2.2 Sample preparation

Sample preparation is a crucial step to get a good image for non conducting sample.When a nonconductive material is imaged,the electrons shot on the sample surface don't have a path to the ground potential and will accumulate on the surface.This will result in a progressively increasing brightness in the imageuntil all the details are no longer visible and in the worst case,the entire field of view will turn white.

Due to their non conducting nature, the surface acts as an electron trap.This accumulation of electrons on the surface is called charging and creates the extra white regions on the sample which can influence the image information. For this reason,if high quality electron image is required,the use of sputter coater is recommended. The conductive coating material act as a channel that allows the charging electrons to be removed from the material[8] .

To avoid this,coating of sample is required in the SEM to improve the imaging of samples. Creating a conductive layer of metal on the sample inhibits charging,reduces the thermal damage and improves the secondary electron signal required for topographic examination in the SEM.

As effective way to attach specimens to the specimen support is usuallygiven by a double sided adhesive tape and need to ensure there is a conductive pathway from the sample surface to ground which isimportant in transporting specimens to the sputter coated and microscope. The key to these collection and transport procedures is to maintain the sample in a condition as close as possible to its natural environment and quickly transport it in to the microscope.

2.3 Sputter Coating

By using a sputter coater,it is possible to create a thin layer of a conductive material on the sample surface. Sputter coating for SEM is the process of applying an ultra –thin coating of electrically conducting metal,such as platinum onto a non conducting separator sample.Sputter coating also prevents the charging of specimens which would otherwise occur due the accumulation of static electric fields.It also increases the amount of secondary electrons that can be detected from the surface of the specimen in the SEM which increases the signal to noise ratio[8].In the present study samples are sputtered by using Sputter coater (JEOL,JEC-3000FC).

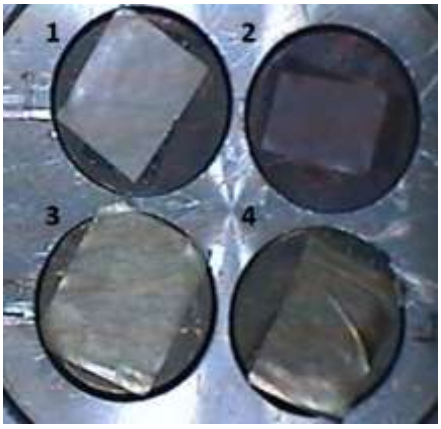

Figure 1: After coating sample images - 1) One minute coating time; 2) Three minute coating time; 3) Five minute coating time; 4) Eight minute coating time

The surface morphology of the separator was examined by Scanning Electron Microscope (SEM) (JSM6010PLUS, JEOL, Japan) The SEM images of the polyolefin based non conducting separator with sputtered coated at one minute, three minute, five minute and eight minutes and the images were recorded at different accelerated voltages one kilo volt, five kilo volt, and ten kilo volt captured the images at different magnification in the range of 2Xto20X for greater visibility of the sample.

3. Results and Discussion

The influence of specimen coating time, accelerating voltage and magnification on the surface morphology of lithium ion separator were examined (Fig.1 to Fig.4) in the SEM. The analysis showed that the coating time and accelerating voltage has an influence on the morphology of the separator and the images are captured at low magnification (2X) to high magnification (20X) for greater visibility. Image analysis has been used to characterize the pore structure, pore distribution, bulk material and fibrils arrangement of separator material.

By the specimen coating time from 1 minute to 8 minutes at 1.0KV, it is clear that the structure of the images are not visible from 5.0kv at 1.0minute specimen coating bulk material connected with fibrils are not visible and at 10.0kv it shows images are white in colour it indicates the charging effect. It is observed when the coating time is 3.0 minutes and 5.0 minutesat 5.0kv &10.kv, images shows interconnected non- oriented spherical and elliptical pores are visible with fibrous structure. Later with the 8 minutes specimen coating at 5.0kv &10.kv, images shows at higher magnification interconnected non- oriented spherical and elliptical pores are visible with fibrous structure. It is observed that, when the coating time is increased it is recommended to view the image in higher magnification.

Thus it concluded that with the optimized coating range between 3-5 minutes at 5.0-10.0 kV, the observed images shows the greater visibility and can considered this range to carry out the further studies.

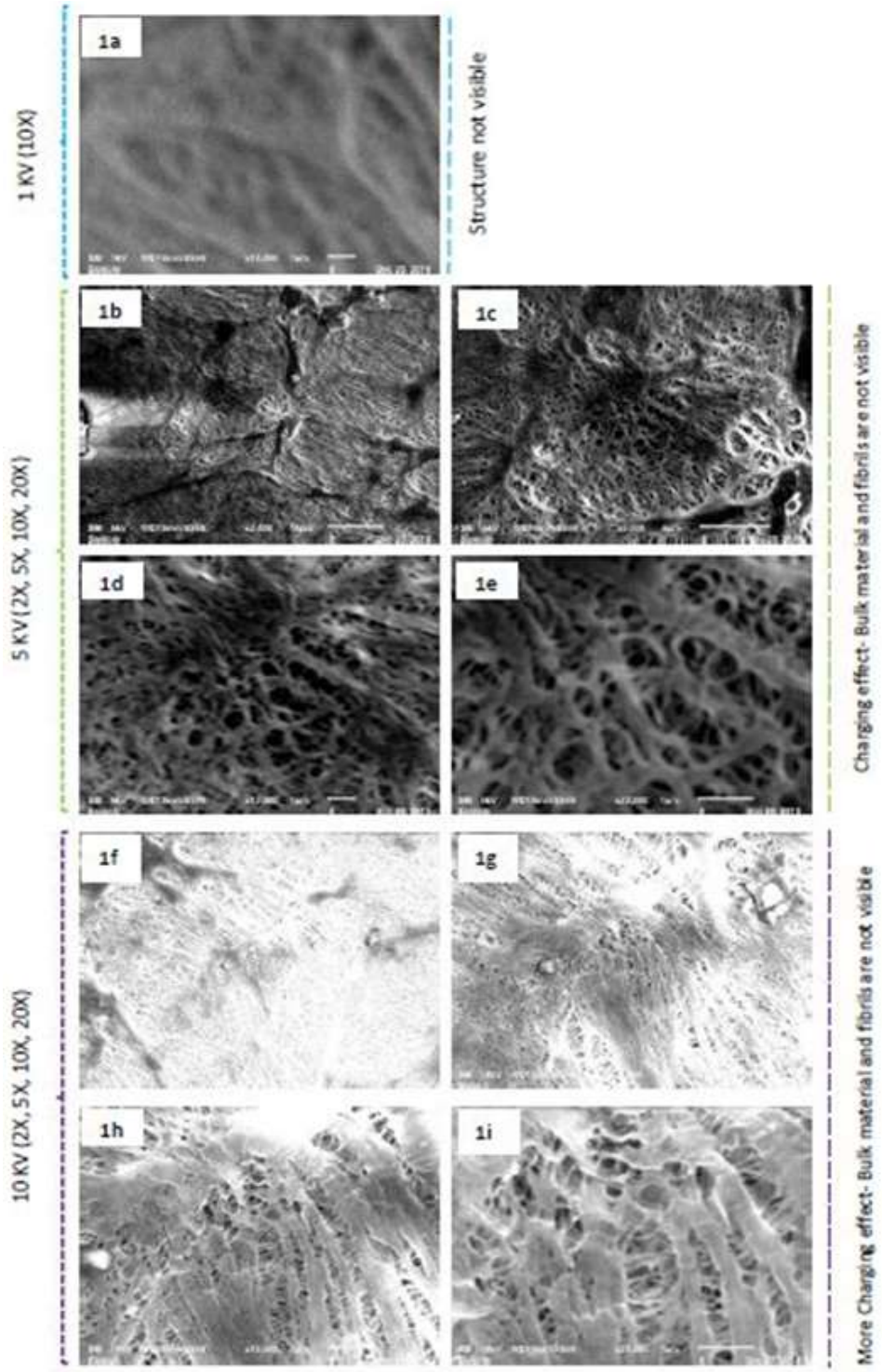

Figure 1: SEM images of the Separator sample of one minute platinum coating with varying KV (1kv, 5kv, and 10kv) and different Magnifications Images (2Xto20X) from 1a to1i

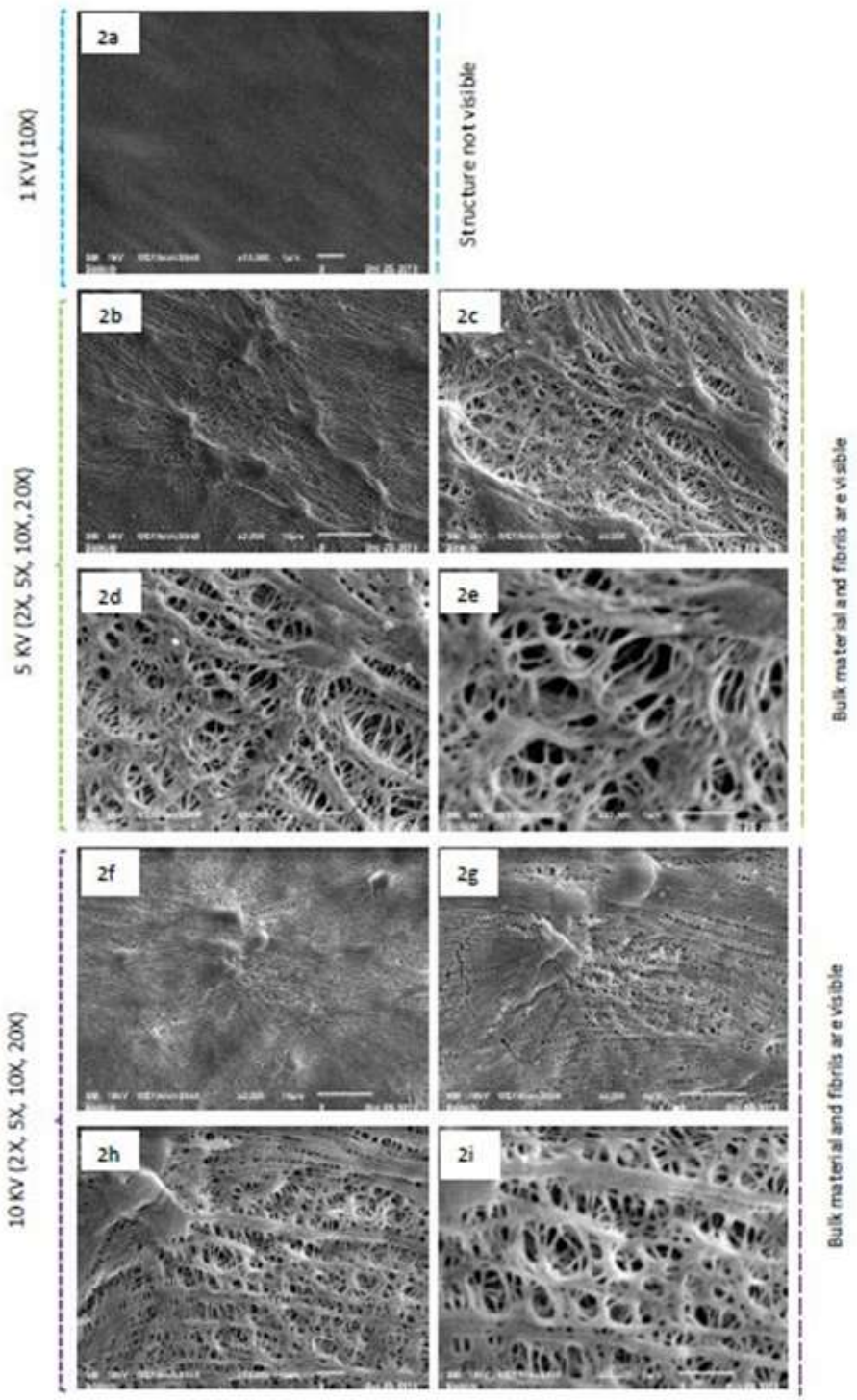

Figure 2: SEM images of the Separator sample of three minute platinum coating with varying KV (1kv, 5kv, and 10kv) and differentMagnifications Images (2Xto20X) from 2a to2i

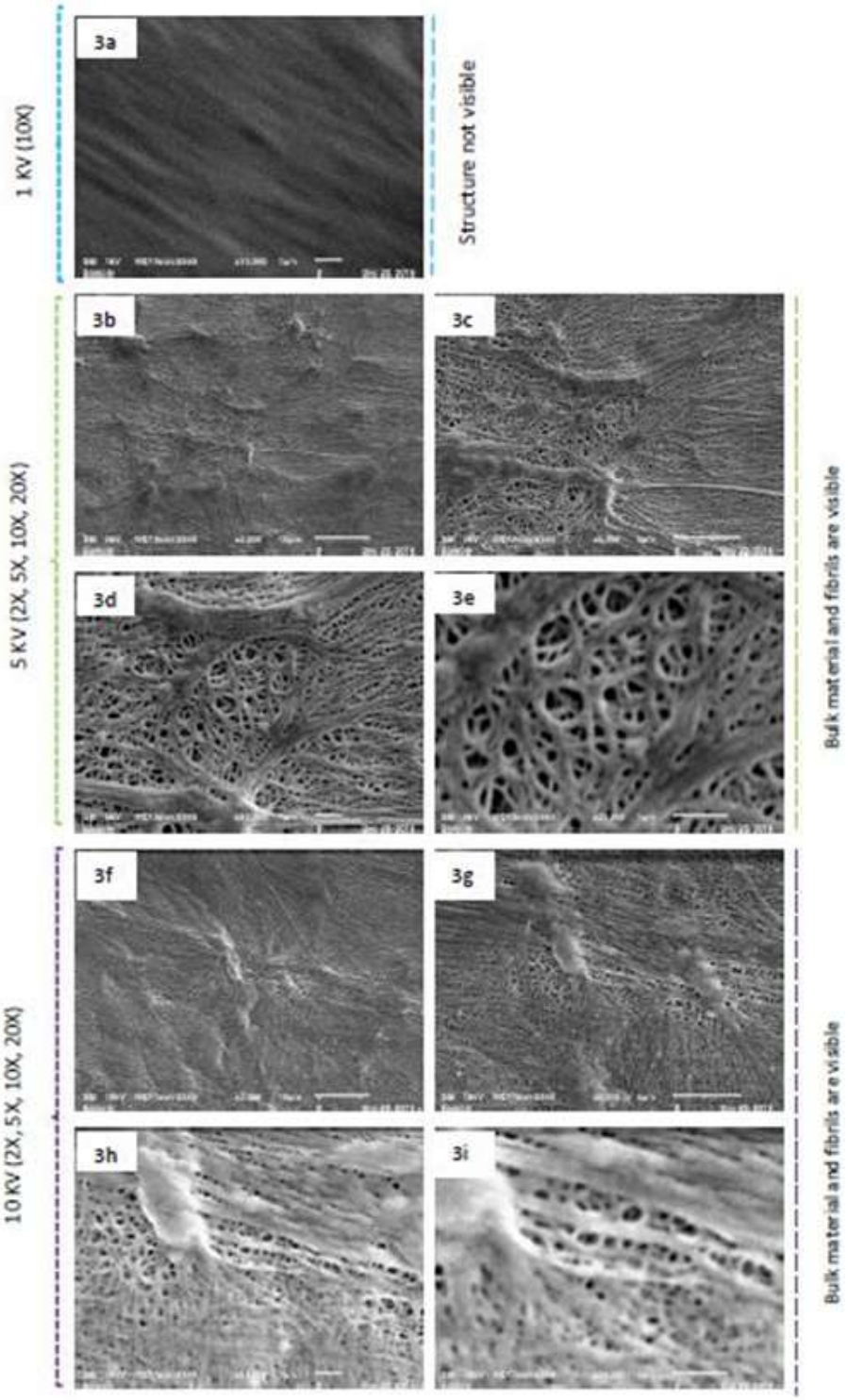

Figure 3: SEM images of the Separator sample of five minute platinum coating with varying KV (1kv, 5kv, and 10kv) And different Magnifications Images (2Xto20X) from 3a to3i

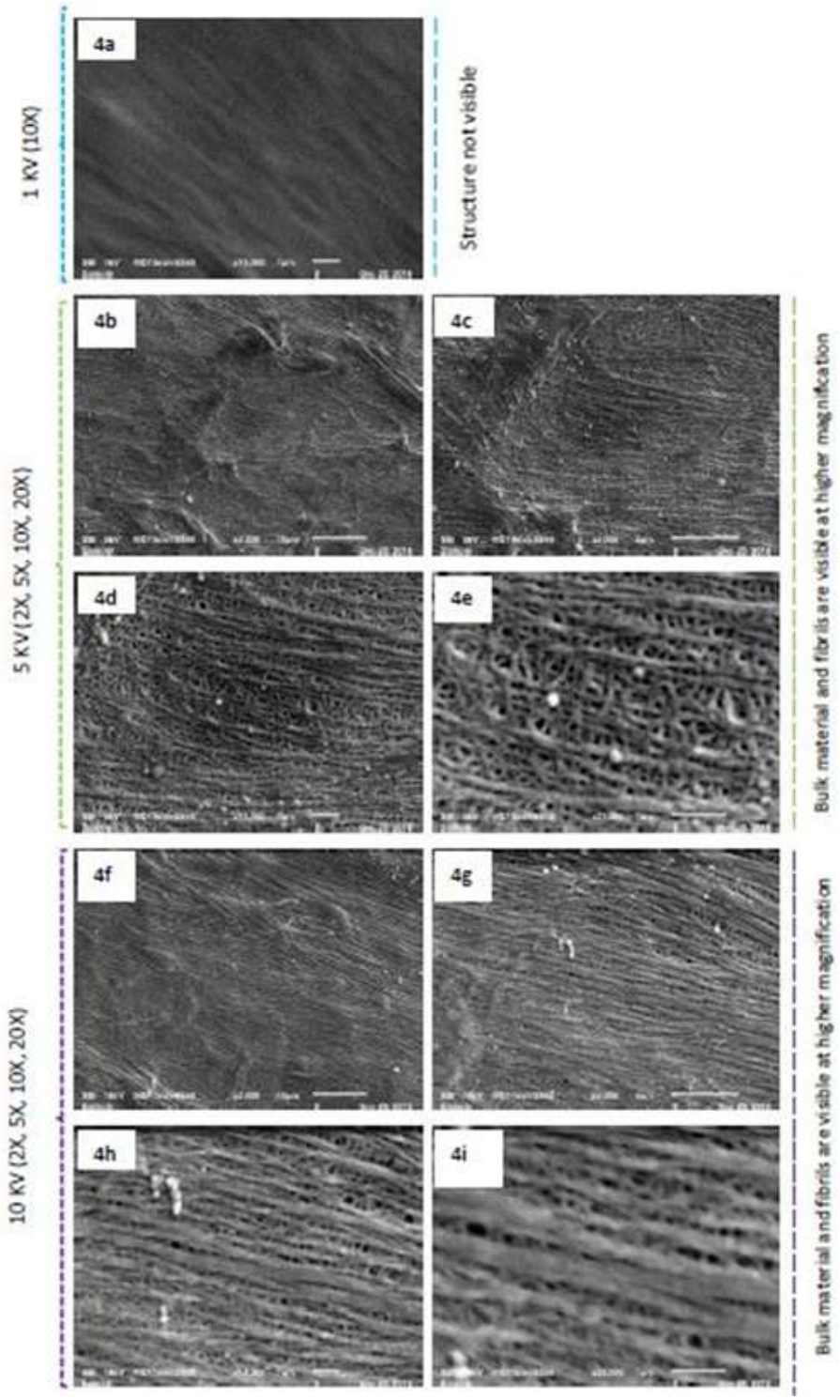

Figure 4: SEM images of the Separator sample of eight minute platinum coating with varying KV (1kv, 5kv, and 10kv) And different Magnifications Images (2Xto20X) from 4a to 4i

4. Conclusions

The effect of sample preparation, image recording condition and its influence on the structural morphology of the separator material have been studied. The observed results reveals that the increase of the surface coating time on the specimen and increase of the accelerating voltage of the SEM at the higher magnification provides the reveals are interconnected non-oriented spherical and elliptical pores are visible with fibrous structure.The optimal coating time on the specimen 3-5 minutes with accelerating voltage in between at 5-10kv recorded images at 10x -20x magnification are preferred conditions for understanding the polyolefin based non conducting separator material for lithium ion batteries .

5. Acknowledgments

The authors would greatly acknowledge to the management of Amara Raja Batteries Ltd., for their support and encouragement. Also would like to thank Mr.Muni Vishnu,Analytical Laboratory for his timely support.

References

[1] M. Kassem, C. Delacourt, Journal of Power Sources 235(2013)159-171.
[2] B. Stiaszny et al. Journal of Power Sources 258(2014)61-75.
[3] S.S. Zhang, Journal of Power Sources 164 (2007) 351–364.
[4] P. Arora, Z.M. Zhang, Chemical Reviews 104 (2004) 4419–4462.
[5] Hun Lee, et al. Energy &Environmental Science, Royal Society of Chemistry.
[6] Xiaosong Huang J Solid State Electrochem (2011) 15:649–662
[7] P. Echlin, Hand book of Sample Preparation for Scanning Electron Microscopy and X-Ray Microanalysis.
[8] White paper / a comprehensible guide to SEM technology –PHENOM WORD part of thermos fisher scientific.