

CHAPTER 8

SURFACE STRUCTURE

CHAPTER 8: Surface structure of chitosan-lithium acetate complexes with Al_2O_3 through Scanning Electron Microscopy (SEM)

8.1 Introduction

Scanning Electron Microscopy (SEM) is used for surface structure and morphology study. Thakut et. al (1998) reported their studies on polyethylene oxide (PEO) based composite polymer electrolytes comprising of PEO- $NaClO_4$ complex dispersed with SnO_2 . Effect of dispersion of SnO_2 morphology and structural properties were reported. The morphology of the film changes substantially on addition of different percentages of the SnO_2 additive. Linford (1988) also reported his study on the morphology of the polymer electrolyte films using SEM technique. In this chapter, surface morphology and structure will be discussed.

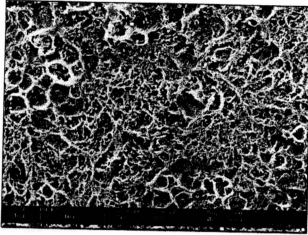
Linford (1988) also reported his work in microstructure studies using Variable Temperature Polarising Microscope (VTPM). He viewed PEO₇:LiClO₄ polymer electrolytes film under plane polarized light where the sample is illuminated by light polarized in one plane, and the transmitted light is passed through another polarizer which is set with its plane of polarization at right angles to the first plane.

SEM studies of polymer electrolyte films reveal the same morphology that can be seen by optical microscopy. The magnification can be substantially greater, but it is difficult to study the electrolyte films at other than the operating temperature of the electron microscope (Linford, 1988).

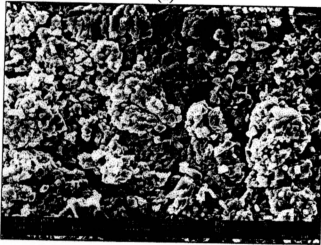
8.2 Scanning Electron Microscopy (SEM)

SEM study was carried out on three samples. They are A0 (chitosan-lithium acetate electrolyte), A2 (chitosan-lithium electrolyte with 2 %wt of Al_2O_3 added, which exhibits the highest conductivity among all samples) and A7 (chitosan-lithium electrolyte with 7 %wt of Al_2O_3 added, with precipitation of Al_2O_3 visible with naked eye). The micrographs are illustrated in figure 8.1.

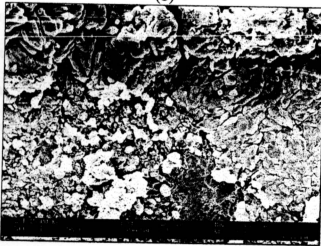
Figure 8.1. SEM micrographs of sample A2 and (b) sample A7.



(a)



(b)



(c)

Figure 8.1. SEM micrograph at 500x magnification for (a) sample A0; (c) sample A2 and (c) sample A7.

It can be seen that the surface morphology of sample A0 (with no Al_2O_3 added) is full with grain boundaries. This explains further its lower room temperature electrical conductivity. The grain boundaries which might extend into the bulk can impede ion migration. For sample A2, which is chitosan-lithium acetate electrolyte with 2 wt% of Al_2O_3 , the surface is full of grains of size about 4 micron. There are no grain boundary compared to A0. No crystalline fractals are observed. The structure of sample A7 shows that the surface is full of flakes of powder like layers, which explains the precipitation of the dispersoids that can be seen with naked eye. The presence of flakes and the boundaries between the flakes impede ionic motion and thereby explains low conductivity at room temperature.

According to Shahi and Wagner (1982), the conductivity enhancement strongly depends on the particle size and the concentration of dispersoids. Al_2O_3 leads to a maximum enhancement (by $\sim 10^3$ times) in their studies. The SEM studies has suggested that the enhancement is due to bulk rather than grain boundary or surface conditions. In this study the grain size is smaller for A2 suggesting that the dispersoid generate excess lattice defects and thereby increases the conductivity.