Synthesis of Nano Crystalline TiO_2 powders by soft chemistry method

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ABSTRACT:

Nanocrystalline Titanium dioxide (TiO_2) powder was prepared using sol-gel process. The phase and the structural evaluation of the heat treated gel were monitoring respectively by XRD and FTIR techniques. The crystallite size was calculated using the Scherrer equation, the average crystallize size found to be ~9 nm. The DSC curve of the gel sample is confirms the removal of water molecules. Hence, the observed XRD, FTIR and DSC results confirmed the formation of nanocrystalline TiO_2 structured powder for developing nanocomposite polymer solid electrolytes for lithium battery applications.

Key Words: Sol-gel process; nanocrystalline TiO_2; XRD; FTIR; DSC

INTRODUCTION

Nanoparticle science and technology have a major impact on various aspects of advanced material research. Wide range of synthesis methods have developed to prepare different size, morphologies (size and shape, cluster composition, dispersion) of nanopowders. Titanium oxide (TiO_2) nanoparticles are an attractive key material for a wide variety of industrial processes, such as photocatalysis, catalysis, charge separating devices, chemical sensors, solar cells, microelectronics, and electrochemistry, due to its varied physical as well as chemical properties [1-4]. Mechanical, optical and electrical behavior of nano-TiO_2 have been widely studied, in order understand its physical properties [2, 3].

Recently, it has been reported that the addition of inorganic fillers like TiO_2, SiO_2 and Al_2O3 of nano sized particles to the polymer matrix improved its ionic conductivity and mechanical stability. Hence, the present work investigates the low temperature synthesis and also characterization by XRD and FTIR of Titanium oxide (TiO_2) **n**anoparticles to develop nanocrystalline composite polymer solid electrolyte films for lithium based rechargeable microbattery applications

EXPERIMENTAL

Synthesis:

Analytical grade precursor chemical of $TiCl_4$ was diluted with ice cooled distilled water and 200ml ethanol under continues stirring in ice bath. The prepared solution was labeled A. Required quantity of urea was dissolved in 100ml water is labeled solution B. Solutions A and B were mixed under continues stirring at 353K to form the gel as well as removing of an alcohol. The prepared gel was washed with water and centrifuged. The obtained gel was heated at 473K and 523K for 4 hours. The phase and the structural evaluation of the heat treated gel were monitoring respectively by XRD and FTIR techniques.

STRUCTURAL STUDIES

XRD

Rigaku miniflex, Japan, X-ray diffractormetrer with Cu-Kα radiation of wavelength (1.5418 A °) was used to record the XRD patterns for the TiO_2 sample between 80° and 3°, 2θ values at a scan rate of 2° per minute. Fig. 1 shows the XRD patterns for dried gel heat treated from 423K to 523K for 4h at each temperature. From Fig 1, the observed peak free XRD patterns of gel heated at 423K and 473K confirm the amorphous nature. Further heating of the gel at 523K, the XRD showed the broad peaks,

TiO_2 structure and also the removal of water molecules [6]. The DSC curve of the gel sample also confirms the removal of water molecules.

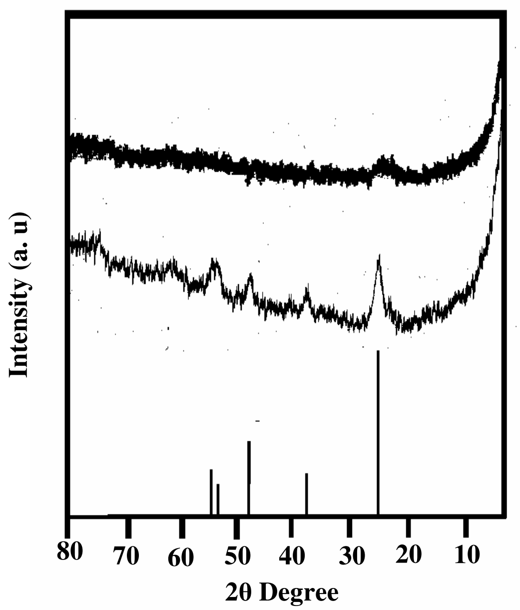

Fig. 1. XRD patterns for nano crystalline TiO_2 sample

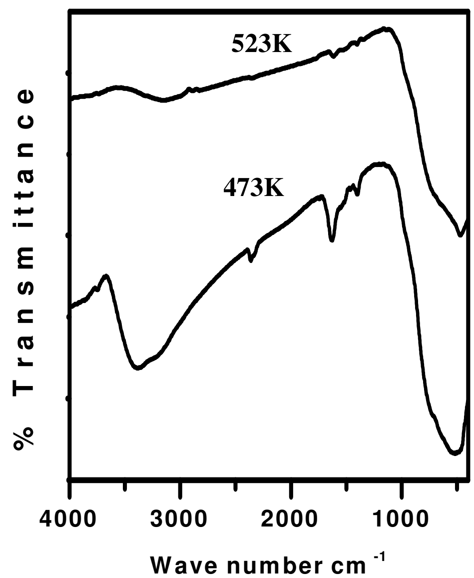

Fig. 2. FTIR spectra patterns for nano crystalline TiO_2 sample

revealing the formation of characteristic of nanocrystalline TiO_2 phase, which was conformed by comparing the JCPDS data (77-0446) with the observed XRD pattern[5]. The crystallite size was calculated using the Scherrer equation, $D=k\lambda/\beta\cos\theta$, where D is the crystallite size, k is a constant (=0.9 assuming that the particles are spherical), θ is the wavelength of the X-ray radiation, β is the line width (obtained after correction for the instrumental broadening) and θ is the angle of diffraction. The average crystallize size obtained from XRD data is found to be ~9 nm

FTIR

FTIR spectra were recorded using a Shimadzu FTIR-8300/8700 spectrometer in the range 4000-400 cm^{-1} at 40 scan for the pellets of TiO_2 sample. All pellets were made with KBr powder and TiO_2 sample using a KBr mini press. Fig. 2 shows the FTIR spectra of the heat treated gel at 473K and 523K. In Fig. 2, the observed variation of IR band positions and also band intensities revealed that the structure of sample changes when it was heated at different temperatures. For the sample heated at 523K, the observed IR band positions revealed the formation of characteristic of

DSC

DSC curve was recorded using a Mettler Toledo Store System under nitrogen atmosphere by heating the thin bottom cell type TiO_2 sample. Placing the TiO_2 sample in an aluminium pan covered with a lid and pressed using the micro pelletizer made thin bottom cells. Fig. 3, shows the DSC curve for dried TiO_2 gel. From fig. 3, the DSC

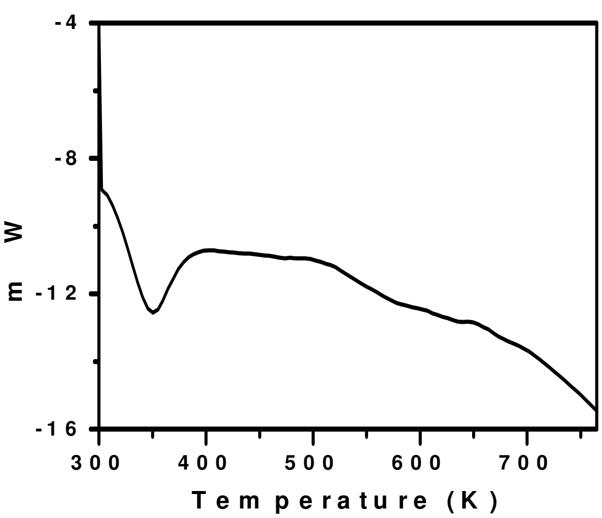

Fig. 3. DSC curve for nano crystalline TiO_2

curve showed a broad endothermic peak between 353K – 450 K corresponds to the loss of water molecules present in the dried gels capillaries [6]

CONCLUSION

Nano Crystalline TiO_2 powder was synthesized by soft chemistry method. The completer synthesis process was monitored by XRD and FTIR techniques. XRD patterns confirmed the nanocrystalline phase and the calculated crystalline particle size is found to be ~9nm. The FTIR spectra confirmed the structure of the TO_2.

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