

Synthesis and Characterization of Electrospun WO₃/PVA nanofibers on FTO and their use as hydrogen gas sensors

B.T. Sone ^{(1), (2)*}, J.B. Kana Kana ^{(1), (2)}, R. Bucher ⁽¹⁾, M. Nkosi ⁽¹⁾ and M. Maaza ⁽¹⁾

(1) Materials Research Group, iThemba LABS, PO Box 722, Somerset West, South Africa. sonebert@gmail.com

(2) Chemistry Department, University of the Western Cape, PB X17, Bellville 7535, South Africa,

*Corresponding author

Abstract – WO₃ is a wide band gap semiconductor widely investigated for use in gas sensing, the design of smart windows and other electrochromic devices [1, 2]. In this paper we report the use of electrospinning to produce WO₃-PVA nanofibres applicable for H₂ sensing and use as an electrochromic device. SEM, AFM, XRD, FT-IR and Raman spectroscopy are used to establish the morphology, crystallinity, purity and structural characteristics of WO₃ before and after calcination. Optical characterization using UV-Vis-NIR radiation before and after H⁺ intercalation of the calcined WO₃-PVA-FTO substrate in 0.5M H₂SO₄ medium reveals optical modulation to be over 10% between the bleached and coloured states of the WO₃-FTO sample. The H₂ sensing capability within the 300-500°C window, of the fibres produced thus is also reported.

Various methods of synthesis including pulsed laser ablation, thermal evaporation, chemical vapour deposition and electrodeposition have been used to produce a wide variety of WO_3 structures ranging from the meso- to submicron scale [4]. In this paper, we present electrospinning of sol-gel prepared WO₃/PVA polymers unto F-doped-SnO₂ (FTO) as a simple and low cost method for producing FTO-supported WO₃ nanofibres that can find application in the area of H₂ sensing and use as an electrochromic device. WO₃/PVA coated FTO substrates produced by electrospinning[3] WO₃-PVA polymer unto FTO at DC voltages between 5-25kV, fixed distances 12-13cm, for 5min, were calcinated for 2h, between 500-600°C, resulting in the loss of PVA thus yielding PVA-free nanoscale WO₃ fibres. SEM (Fig.1(a) and (b)) revealed the non-calcinated WO₃-PVA fibres to be made up of randomly aligned ultrathin fibres, hundreds of micrometres in length, and average diameters = 134.61+/-23.51nm. Calcination at 500°C, 2hrs, showed a reduction of these diameters to the 57-120.0nm range. AFM confirmed an increase in surface roughness to 81.20nm after calcination. XRD confirmed the improvement in crystallinity of the samples upon annealing in air. The decomposition of PVA and the obtention of high-purity WO₃ nanofibres postcalcination was confirmed through FT-IR analysis. Optical characterization of the calcined sample (prepared at 20kV,12.5cm,for 5min) in the coloured and bleached state post H⁺ intercalation 0.50M H₂SO₄ medium (Fig.2(a) and (b)) showed optical modulation in the Vis-NIR range to be greater than 10%. An increase in the amount of WO₃ nanofibres deposited on the substrates is anticipated to yield improvements in the optical modulation of the samples post H+ intercalation, so too an increase in the H⁺ concentration of the electrolyte. More on these and the H_2 sensing performance of these calcined samples within the 300-500°C window is reported.



Figure 1: (a) WO₃-PVA fibres on FTO before calcination (b) WO₃ nanofibres on FTO after calcination.



Figure 2: (a) Cyclic Voltammogram for H⁺ intercalation of WO₃ on FTO, post-calcination at 500°C **(b)** Optical transmission before and after H⁺ intercalation.

References

- [1] G.N. Chaudhari, A.M. Bende, A.B. Bodade, S.S. Patil, V.S. Sapkal, Sensors and Actuators B 115 (2006) 297-302.
- [2] M.Gillet, R. Delamare, E.Gillet, Eur.Phys.J.D, **34** (2005) 291-294.
- [3] X. Lu, X. Liu, W. Zhang, C. Wang, Y.Wei, Journal of Colloid and Interface Science 298 (2006) 996-999.
- [4] A. Subrahmanyam, A. Karuppasamy, Solar Energy Materials & Solar cells 9 (2007) 266-274.