Electrochromism of NiO-TiO_2 Sol Gel Layers and Devices Made of Them

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Films of NiO-TiO_2 with Ni concentration of 100, 90, 87, 83, 75, 66, 50 and 33 mol % have been obtained via the sol-gel route by dip coating technique and sintered in air between 250 and 500 °C using ethanolic sols of nickel acetate tetrahydrate ($Ni(CH_3COO)_2{\cdot}4H_2O$) and titanium n-propoxide ($Ti(O\text{-}CH(CH_3)_2)_4$) precursors. Xerogels obtained by drying the sols have been studied up to 900 °C by thermal analysis (DTA/TG) coupled to mass and IR spectroscopy. The crystalline structure and morphology of the layers in the as deposited, bleached and colored states was determined by X-ray diffractometry, scanning electron microscopy and transmission electron microscopy Their electrochromic properties have been studied in 1 M KOH aqueous electrolyte as a function of the layer composition, thickness and sintering temperature. Deep brown colour with reversible transmittance changes have been obtained using cycling voltammetry and chronoamperometry processes. The best composition to get stable sols, a high reversible transmittance change and fast switching times (10 s) was obtained with double NiO-TiO_2 layers 160 nm thick having 75 % Ni molar concentration, and sintered between 300 and 350 °C. The mechanism of coloration and morphology transformation of the layer during cycling are discussed in terms of an activation and degradation period. The results are in agreement with the accepted Bode model. 5 x 10 cm^2 brown coloring electrochromic windows with the configuration K-glass/NiO-TiO_2/KOH/CeO_2-TiO_2/K-glass have been prepared and characterized by optoelectrochemical techniques. The windows present a deep brown color due to the presence of Ni3+ (NiOOH) species, have a rather fast kinetics (<30 s) and are fully reversible up to more than 20000 cycles.