



Effects of Nb on the TiO₂ Nanopowders: Synthesis and Characterization

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Abstract –Ti_{1-x}Nb_xO₂ nanopowders were prepared by polymeric precursor method. X-ray diffraction and micro-Raman measurements indicate that the presence of Nb ions in the TiO₂ network inhibits the phase transition (anatase-to-rutile). Preliminary electrical measurements show that the electrical conductivity increases rapidly with the Nb content.

Titanium oxide, TiO₂, is a semiconductor material most widely used for oxygen detection (commonly in a rutile phase). The oxygen detection mechanism implies the diffusion of oxygen ions in the bulk of the material and this occurs provided the material is operated at high temperatures (700-1000°C). On the other hand, TiO₂ showing an anatase crystalline phase has more free electrons than rutile phase of TiO₂ [1,2]. For anatase phase, oxygen detection can be associated to a surface reaction, which takes place at lower temperatures (~500°C)[1,2].

The presence of a suitable doping agent strongly affects the kinetics of this phase transformation process (anatase-to-rutile). Besides, the effect of Nb doping in titania and its importance for oxygen sensor has recently been reported by several authors [1-4], indicating higher device sensitivity at lower working temperatures [1-4]. In this work Ti_{1-x}Nb_xO₂ (x=0.0, 0.01, 0.03, and 0.05) were synthesized via the polymeric precursor method. This method is based on the polymerization of metallic citrate using ethylene glycol. A hydrocarboxylic acid such as citric acid is normally used to chelate cations in an aqueous solution. The addition of a polyalcohol such as ethylene glycol leads to the formation of an organic ester. Polymerization promoted by heating to around 100°C results a homogenous resin in which the metal ions are distributed uniformly throughout the organic matrix. The resin is then calcined to produce the desired oxide.

The samples were characterized by differential thermal analysis, X-Ray diffraction analysis, micro-Raman spectroscopy, and impedance spectroscopy. Rietveld refinement was performed on the samples. XRD measurements did not indicate evidences of niobium oxide phases. Moreover, from XRD and micro-Raman analysis, it was observed that the presence of Nb ions in the TiO₂ structure inhibits the phase transition from anatase-to-rutile (Figure 1).

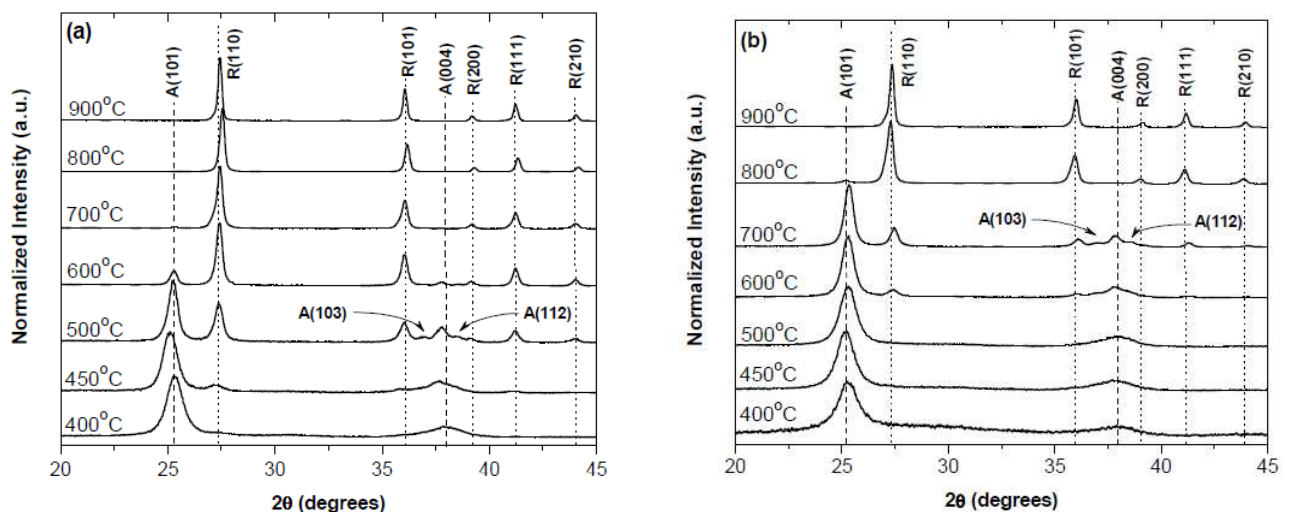


Figure 1: XRD patterns of powders calcined at different temperatures a) TiO₂ pure and b) Ti_{0.97}Nb_{0.03}O₂.

References

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