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Comparative study of zirconium and titanium oxide thin films obtained from metalorganic precursors by LP-MW plasma process

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Comparative study of Zr and Ti oxide nanocoatings obtained by low P°PECVD is the subject of this work. Zr (IV) tert-butoxide (ZTB, ZrO₄C₁₆H₃₆) and Ti (IV) isopropoxide (TTIP, TiO₄C₁₂H₂₈) have been used as Metal organic precursors (MOP). Thin films were grown in a multi-dipolar ECR microwave (MW) plasma. O₂ gas was used as an oxidant. Diagnostics such as OES, FTIR, XPS, SEM, AFM, XRR and ellipsometry porosimetry (EP) have been used to study the influence of 3 parameters on the plasma phase and the thin film characteristics: O₂ gas proportion in precursor/O₂ mixture, total gas pressure and addition of HMDSO to the gas mixture. First, O₂ percentage was increased in the MOP/O₂ mixture at constant total gas P° of 1 mTorr. Deposited ZrOCH and TiOCH thin films evolve from organic to almost inorganic. In O₂-rich plasmas, the growth mode changes and columnar morphology appears. Nevertheless, in both zirconia-like and titania-like films, the density increases significantly compared to the films deposited at 100% MOP when a compact and uniform morphology has been observed. Next, total gas P° has been varied from 1 to 10 mTorr in a 5% MOP/95% O₂ mixture. Thin film nanoporosity increases with total gas P° to reach 16% and 10% at 8 mTorr in ZTB and TTIP plasmas, respectively. In films deposited in ZTB/O₂ plasma, remarkable changes in morphological properties are observed: from closely stacked columns obtained at 1-2 mTorr to the disappearance of columnar structuration at 8 mTorr. Last, HMDSO was added to the 5% MOP / 95% O₂ mixture. Depending on the proportion of HMDSO, the morphology of the films can be uniform and compact with no observable structuration as well as columnar while chemical composition stays relatively stable. Thus, it has been demonstrated that these 3 parameters allow modifying and controlling the layers composition, morphology and properties over very wide ranges.

Keywords

low pressure PECVD

ZTB

TTIP

HMDSO

nanocoatings