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Temperature and polarization dependence of growth processes of Zirconium metal-organic layers deposited by MMP-DECR reactor using zirconium tert-butoxide/O₂ mixture.Rick Verhoef¹, Raphael Cozzolino¹, Patrice Raynaud²¹LAPLACE laboratory, Toulouse, France ²CNRS, LAPLACE (Laboratoire Plasma et Conversion d'Energie), Toulouse, France

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ZrO_xC_yH_z metal-organic thin films were deposited by low pressure microwave DECR plasma of zirconium tetra-Tert-Butoxide (ZTB) as metal-organic precursor mixed with oxygen as oxidant gas. X-Ray Photoelectron Spectroscopy (XPS), Fourier Transform Infrared Spectroscopy (FTIR), and Spectroscopic ellipsometry were used to investigate the deposited films. Moreover the plasma phase was probed by Optical Emission Spectroscopy (OES). The plasma was created in a Microwave Multipolar reactor excited by Distributed Electron Cyclotron Resonance (MMP - DECR). The films were deposited for temperatures ranging from -5°C to 110°C, the total gas pressure was 1mTorr (0.133 Pa), the microwave power was fixed at 400W and the oxygen ratio varied from 60% to 95%. Moreover, an RF Bias (From 0.0V to -100.0 Volts) was applied to the Sample holder to control the ion bombardment of the substrate. The influence of sample temperature, polarization and gas mixture ratio [O₂/ZTB+O₂] on deposition rate, density, chemical bonds, atomic composition and micro - and meso-structure of the deposits are presented.

At high temperature (110°C) and room temperature and 80% of O₂ in the mixture, microstructure analysis shows a columnar growth, while at low temperature (-5°C) the columns disappear. At fixed temperature an increase of polarization enhances the columnar growth but for high values of polarization (80V) the columns are removed.

Correlation between process parameters and structure of the deposited films will be presented. The understanding of columnar growth allows the control of the optical and barrier properties of the deposited films.

Keywords

Plasma polymerization
Microwave plasma
zirconium tert butoxide
structure and composition
columnar growth