



Structural Properties of HfO₂ films deposited by spray pyrolysis technique

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Abstract

Structural, morphology and composition properties of Hafnium Oxide (HfO₂) films are reported. The films were prepared by spray pyrolysis deposition process, using chlorides as raw materials in deionised water as solvent. Corning glass was used as substrates at temperatures from 350°C to 600°C. X ray analysis shows an amorphous structure at low temperatures, while substrate temperatures higher than 400°C monoclinic phase of HfO₂ appear. This result is confirmed by TEM. Scanning electron microscopy was used to observe the microstructure of the films; rough surface with spherical particles are appreciated. The chemical composition was obtained by Energy Dispersive Spectroscopy (EDS), a rate H_f / O close to ideal (0.5) was obtained.

Introduction

The hafnium oxide (HfO₂) is a material with a wide range of possible technology applications because it's chemical and physics properties as high melting point, high chemical stability, and hardness near to diamond in the tetragonal phase. These properties make this compound attractive to be used as gas sensors and many electronic and optic applications [1]. In this work the structural features of HfO₂ coatings synthesized by the ultrasonic spray pyrolysis technique as a function of deposition temperature are studied.

Experimental details

The HfO₂ films were grown using the ultrasonic spray pyrolysis technique. Substrates of 1 x 1 cm² of corning glass were cleaning following a standard procedure [2] and heating on a tin bath. The starting reagents were HfCl₄ in deionised water as solvent; the initial solution was prepared to a 0.05 molar concentration. Deposition temperatures (Ts) were in the range from 350 °C to 600 °C. The carrier gas was filtered air at flow rate of 10 l/min. The deposition time was 10 minutes for all the samples in order to reach films with almost the same thickness.

Results and discussion

XRD spectra of HfO₂ films deposited at Ts from 350 °C to 600 °C are shown in the figure 1. At low deposition temperature, the hafnium oxide films are amorphous, but for higher Ts, these films show peaks which correspond to the hafnium oxide monoclinic stable phase (referenced JCPDS

431017). Sharper diffraction peaks at high Ts could indicate an increase in the crystal's size.

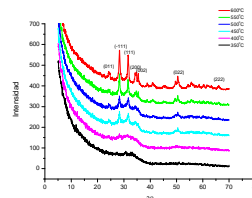
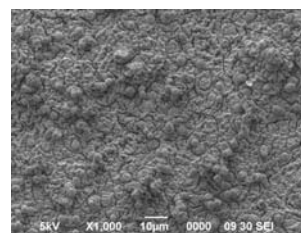


Figure 1.- XRD spectra of HfO₂ films deposited at Ts from 350 °C to 600 °C

The results of EDS measurements (not shown), were summarizes the relative atomic percentages of the oxygen, chlorine and hafnium present into the films as a function of deposition temperature. It was possible to observe an appreciable reduction in the relative content of chlorine with the temperature increase. A rate H_f/O close to ideal (0.5) was obtained.

Figure 2; show the SEM micrograph of the surface morphology of HfO₂ coatings, of the samples deposited at 500 °C. It is possible to observe rough but continuous films with good adherence to the substrate, the surface morphology of the films depends on the deposition temperature.



Acknowledgments

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References

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