Growth Mechanism Of Zn/ZnO Core-Shell Nanostructures Obtained By DC Reactive Magnetron Sputtering With Variable Absolute Gas Flow Values At A Set Ratio

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In reactive magnetron sputtering thin films growth is strongly determined by the properties of working gas in the vacuum chamber. The influence of the composition and flow, the type of reactive gas as well as the ratio of argon to reactive gas have been intensively studied, however there are no investigations on the effect of absolute gas flow values at a set ratio. In previous experiments we have proved that by changing the total pressure and the ratio of argon to oxygen in a DC magnetron sputtering of a Zn target it is possible to obtain either dense or porous films [1]. In this communication we show that changing the absolute gas flow value strongly influences the morphologies as well as the properties of nanostructures in a DC reactive magnetron sputtering under porous growth conditions.

In this experiment we consider the growth mechanism of Zn/ZnO nanostructured thin films obtained by room temperature reactive magnetron sputtering of a Zn target at 80 W DC power, low 3 mTorr total pressure and variable absolute $Ar+O_2$ flow values in sccm at a set 10:1 ratio as follows: 3:0.3, 6:0.6, 8:0.8, 10:1, 15:1.5, 20:2, 30:3. The properties of the deposited films were investigated by means of complementary structural (SEM, TEM, XRD), electrical (resistivity) and chemical (XPS, EDX) measurements. Plasma properties were also examined using Langmuir probe and optical emission spectroscopy.

SEM images proved strong dependence of film morphology on absolute gas flow values – from dendritic/nanopetal for low flow to dense porous for high flow with decreasing sizes of grains and progressive amorphization according to XRD measurements. TEM imaging, XPS and EDX delivered more detailed information about composition – the nanostructures have a Zn core with a ZnO shell resulting from surface oxidation of metallic films to 4 nm thick after exposure to atmospheric air. This shell causes an increase in resistivity, especially for more porous films. The growth mechanism of Zn/ZnO nanostructures can be explained based on structure zone model by Mahieu et al. [2] and about the effect of oxygen impurities existing during deposition process. We assumed that the structural evolution of these films is determined by the influence of oxygen incorporating into the film surface during growth and acting as an inhibitor – decreasing the sizes of crystallites and amorphizing the structure. We have also proved that plasma properties do not influence the discussed changes due to relatively constant parameters during all processes. Despite detailed investigations the growth of the observed ZnO nanopetals remains unclear and requires further studies.

[1] M.A. Borysiewicz et al., *Phys. Status Solidi A* 209, No. **12**, 2463–2469 (2012)
[2] S. Mahieu, P. Ghekiere, D. Depla and R. De Gryse, *Thin Solid Films* **515** (2006) 1229 – 1249