Magnesium acetylacetonate-dipivaloylmethanate as a new precursor for MOCVD of MgO thin films

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Due to the high efficiency of the secondary electron emission thin films of MgO are valuable for the application in plasma displays with high luminosity. For this purpose smooth and highly transparent MgO films should be deposited below 450°C on large-area glass substrates. We demonstrate here that these demands can be satisfied by MOCVD with $Mg(thd)_2$ and new mixed ligand complex Mg(thd)(acac) as precursors, the latter is of lower cost.

The mixed ligand complex Mg(acac)(thd) has been possible to obtain by ligand exchange reaction

$$Mg(acac)_2 + Mg(thd)_2 \leftrightarrow Mg(acac)(thd)$$

The main stage of weight loss Mg(acac)(thd) occurs in temperature range of 150-250°C, and the total weight loss is equal to 98% that is more than total weight loss for $Mg(acac)_2$ (88%) or for mixture of parent complexes (Fig.1). The main argument of formation the mixed ligand complex has been obtained by mass spectrometry method. The mass spectra of Mg(thd)(acac) contain mixed ligand ions. Analysis of the relations between ion current intensities homoligand species and mixed ligand species and their temperature dependences provided reason enough to believe that new mixed ligand complex Mg(thd)(acac) sublimed congruently in the temperature range studied. The vapour phase over Mg(thd)(acac) consists of dimeric molecules $[Mg(thd)(acac)]_2$, which vapour pressure is equal to $1.6 \cdot 10^{-5}$ atm at 447 K. The standard enthalpy of sublimation for Mg(thd)(acac) was found to be equal 147±12 kJ/mol.

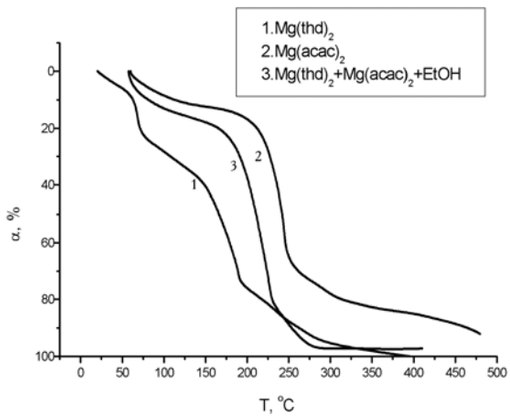

Figure 1. TG curves under vacuum (0.01 torr) for parent $[Mg(acac)_2]$ and $[Mg(thd)_2]$, and for mixed ligand complex.

We have found that the transparent colorless thin films of MgO can be grown in the kinetic regime in the temperature range 380-450°C. The activation energy for the deposition of MgO with $Mg(thd)_2$ is 60 kJ/mol what is typical value for the deposition controlled by the decay of the precursor molecule. Noteworthy $Mg(thd)_2$ provides essentially higher deposition rate as compared to $Mg(acac)_2$ and Mg(thd)(acac) (Fig.2). The behavior was explained by higher chemisorption of $Mg(thd)_2$ on the surface.

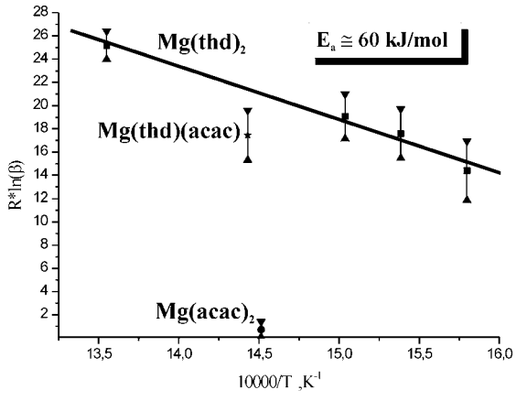

Figure 2. Deposition rates for different Mg-containing precursors

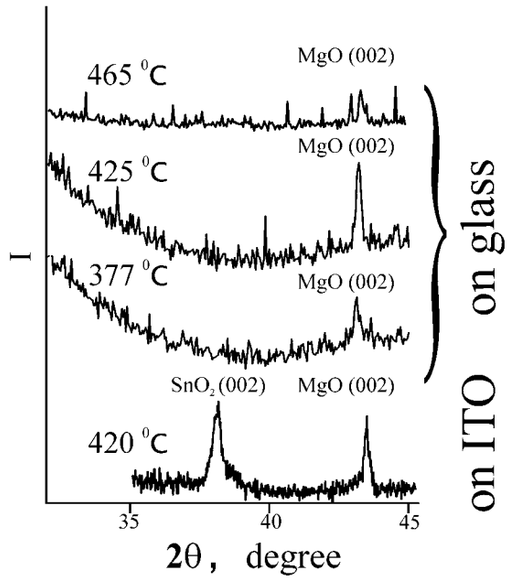

Figure 3. XRD patterns of MgO films deposited on glass and ITO at different temperatures (CuKα radiation). Decrease of the intensity at 465°C is due to the chemical interaction with glass.

XRD revealed that the films prepared were crystalline both on SnO_2 and on low-melting glass (Fig.3). The intensity of (002) reflection increased with the deposition temperature but for the films on low-melting glass the intensity decreased again above 450°C as a result of the chemical interaction between glass and MgO. The annealing experiments are in agreement with such supposition. After annealing at 460°C the reflection of MgO disappeared completely and $MgSiO_3$ was detected. This severe chemical interaction should be accounted in the technology of the plasma displays.

SEM observation showed that the surface of the MgO films is quite smooth, without precipitates and cracks. Also subsequent annealing produces no cracks in the MgO coating. AFM measurements revealed the same roughness of the MgO coating (0.5 μm thick) and underlying glass substrate what is in agreement with the kinetically controlled deposition process. Regular submicrometer grains were observed in MgO coating.