

THERMAL PROPERTIES OF Ir(I) PRECURSORS:
ACETYLACETONATO(1,5-
CYCLOOCTADIENE)IRIDIUM(I) AND
(METHYLCYCLOPENTADIENYL)(1,5-
CYCLOOCTADIENE)IRIDIUM(I)

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The goal of the present work is complex investigation of thermal properties of perspective iridium(I) chelates for the purposeful selection of the parameters for the deposition of iridium coatings by means of CVD.

Melting temperatures that were measured are 155°C and 36°C for (acac)Ir(cod) and (MeCp)Ir(cod) respectively. Thermogravimetric analysis for (acac)Ir(cod) and (MeCp)Ir(cod) was carried out in inert atmosphere (He) and in vacuum. Thermogravimetric curves of mass losing were obtained.

The temperature dependencies of saturated vapor pressure of iridium(I) complexes (MeCp)Ir(cod) and (acac)Ir(cod) were investigated by means of Knudsen's effusion method within temperature ranges 20-56°C and 60-100°C respectively (figure 1). Thermodynamic parameters of vaporization processes for the complexes were calculated. The following values of ΔH°_T (kcal mol⁻¹) and ΔS°_T (cal mol⁻¹K⁻¹) for equation $\lg P_{\text{torr}} = - (A/T) + B$ were obtained for sublimation of (MeCp)Ir(cod) and (acac)Ir(cod) respectively: 29,78±1,19, 66,57±3,94; 26,72±0,41, 47,17±1,24; and for evaporation of (MeCp)Ir(cod): 21,04±0,32, 38,57±1,24.

Thermal decomposition of the vapors of compounds was investigated in vacuum within temperature range 200-570°C and in the presence of oxygen in the reaction region within temperature range 200-450°C. The temperature dependencies of the changes of the composition of gas phase during thermolysis of Ir(I) chelates at the initial vapor pressure in the reactor 0,001 torr are shown in figures 2 and 3. On the basis of experimental data on the composition of gas-phase products of thermal decomposition of the compounds schemes for the deposition of metal phase were offered.

The analysis of the results shows that the investigated complexes possess high volatility at not very high temperature and high thermal stability. Transport of vapor into the deposition region at using in CVD processes does not cause any special difficulties. Thus, these complexes can be used to obtain iridium coatings in LPCVD processes, as well in CVD processes in the presence of reagent gases, for example, oxygen. In our opinion, a more preferable compound for the deposition of metal Ir in CVD processes is (MeCp)Ir(cod), because it possesses higher volatility, and thermal decomposition of its vapor gives thermally stable products.

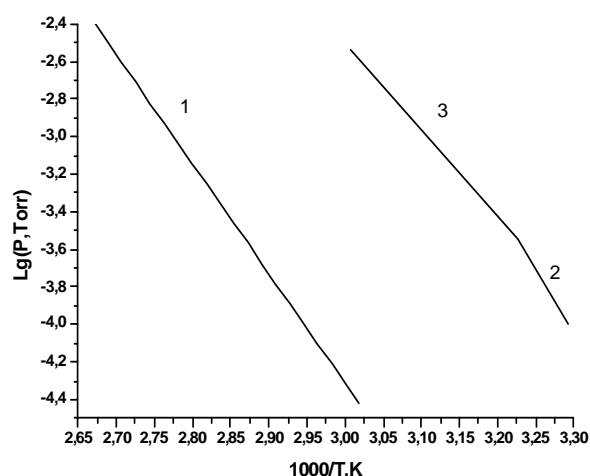


Figure 1. The dependencies of the logarithm of saturated vapor pressure of complexes *versus* inverse temperatures for (acac)Ir(cod) (1); (MeCp)Ir(cod) (2 – sublimation process and 3 – evaporation process).

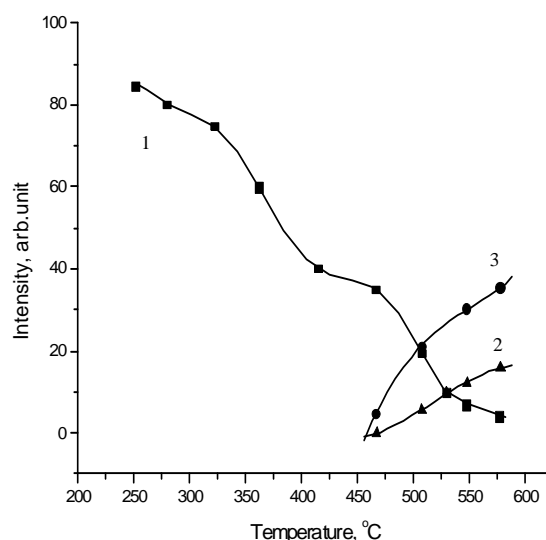


Fig. 2. Temperature dependence of intensities of ion peaks at decomposition of (MeCp)Ir(cod) vapor: 1 – [(MeCp)Ir(cod)]⁺, 2 – cod⁺, 3 – C₆H₆⁺.

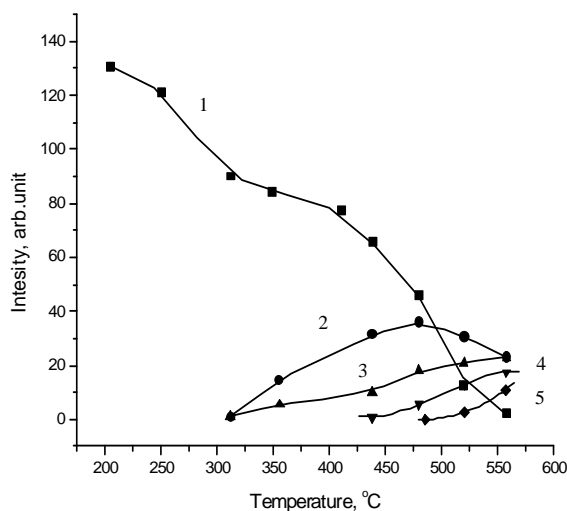


Fig. 3. Temperature dependence of intensities of ion peaks at decomposition of (acac)Ir(cod) vapor: 1 – [(acac)Ir(cod)]⁺, 2 – Hacac⁺, 3 – cod⁺, 4 – C₈H₈⁺, 5 – C₅H₅O⁺.