**MASS SPECTROMETRY AS A TOOL TO STUDY CVD PROCESS**

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Application of two mass spectrometric (MS) techniques to study chemical vapour deposition from organometallic precursors is described. $CpCuPEt_3$ (Cp = $\eta^5$-$C_5H_5$, Et = $C_2H_5$) was used as a model precursor in this work.

*In situ* MS where a miniature hot wall CVD reactor built in the time-of-flight mass analyser ensures the fast and uninterrupted recording of temperature dependence of gas phase composition upon precursor vapors programmed heating. The detailed descriptions of how thermal decomposition mechanism of the precursor is deduced from the experimental data and how the films composition is predicted were demonstrated in [1].

In this report main attention is paid to the on line MS which was used to explore gas phase upon metal film formation in the cold wall CVD reactor. In this technique quadrupole mass spectrometer is connected to the reactor through the heated glass-coated capillary. This technique was also used for conditioning and cleaning the reactor and plasma assisted etching of the substrates.

Fig. 1 illustrates how on line MS may be used to choose temperature conditions of CVD process. It can be observed that the degree of the precursor decomposition is stringer enhanced by the increase of the evaporator temperature from 80 to 100 °C than by the increase of the substrate temperature from 200 to 240 °C.

Fig. 2 shows time related gas phase composition during the deposition experiment. Deposition starts *c.a.* 11 min after the beginning of the precursor feeding; it reaches maximum prior decreasing again. This behaviour is deduced from the decay of ion intensities corresponding to decomposition products ($Cp_2$ and $PEt_3$) and by the simultaneous increase of the signal from the molecular ion peak ($CpCuPEt_3$).

An on line MS experiment with programmable heating of the susceptor was performed with the aim to check the compatibility of data on gas phase composition provided by both MS techniques. The obtained results are compatible; i.e. the composition and the thermal behaviour of the gas phase are similar. Minor differences in the temperature range can be attributed to the use of hot wall reactor in the case of *in situ* MS (Fig. 3).

The correlation between the composition of the gas phase upon deposition and the resulting film properties are also considered in this contribution together with the mutual influence of precursors upon co-deposition of Al-Cu bimetallic films.

From the obtained results it can be concluded that the combination of the two MS techniques allows producing a convenient and relatively inexpensive approach for the investigation of the mechanisms of thin film deposition from molecular precursors.

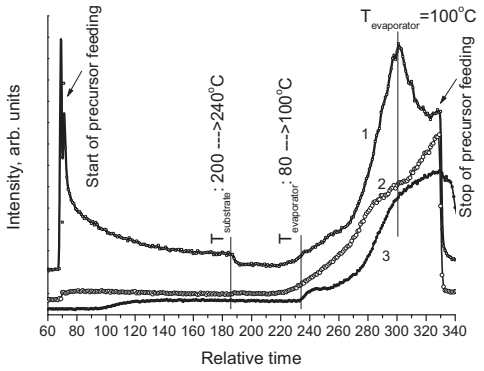


**Fig. 1:** Time dependence of intensity of ion peaks characterized the gas phase composition upon choosing the temperature conditions: 1- m/z 66 (fragmentary ion from $[CpCuPEt_3]^+$), 2- $[Cp_2]^+$, 3- $[PEt_3]^+$.

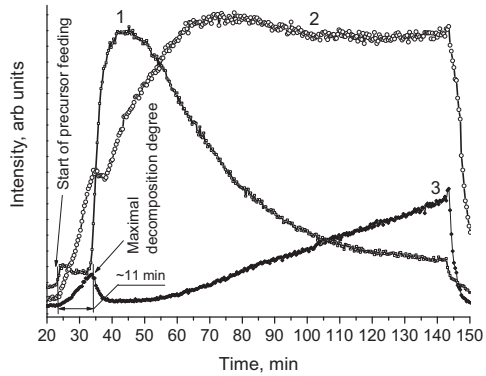


**Fig. 2:** Time dependence of intensity of ion peaks characterized the gas phase composition upon CVD: 1- $[Cp_2]^+$, 2- $[PEt_3]^+$, 3- m/z 181 (fragmentary ion from $[CpCuPEt_3]^+$).

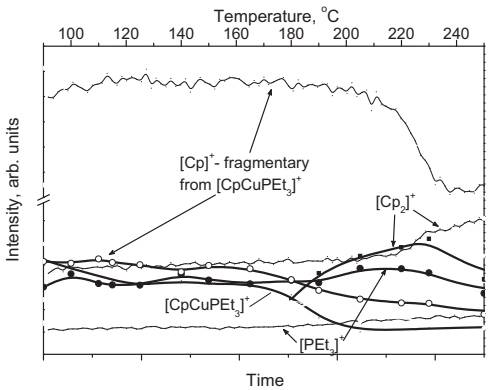


**Fig. 3:** Comparison of the data obtained by *in situ* (thick lines) and on line (thin lines) MS.

[1] F. Senocq, A. Turgambaeva, N. Prud'homme, U. Patil, V. Krisyuk, D. Samélor, A. Gleizes, C. Vahlas, *Surf. Coatings Tech.*, 201 (2007) 9131–9134.