

CVD OF TRANSITION METALS FROM METALORGANIC COMPLEXES

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Because of the progress in microelectronics, the devices have to be more and more efficient. In other terms the devices have to be miniaturized to reach the demands of the user. To built integrated circuits on e. g. silicon wafers the electric conducting, semi conducting and insulating layers consisting of metals and metal oxides have to be of maximum purity and deposited very precise on the desired areas. The smaller or thinner the structures are the more important it is to keep the temperature of the deposition process low in order to avoid diffusive mass transport out of the layer into the base material or reverse. Also the deposition of metals or metal oxides onto polymers is of great interest and needs also low deposition temperatures.

Using CVD, iron and ruthenium films with high purity are commonly produced from metal carbonyl compounds. However, the use of metal carbonyls is limited by their toxicity.

The aim of the German Research Foundation (DFG) project is to develop a technology for the deposition of transition metal and metal oxide films under mild conditions starting from new synthesized precursors with an optimized MOCVD process.

The precursors for the MOCVD experiments are prepared in laboratory scale at the Institute of Anorganic Chemistry of the University of Erlangen.

The preparation of the iron precursor [(butadiene)(toluene)Fe(0)] requires two steps (Fig. 1).

A metal vapour reaction leads to the highly reactive intermediate [bis(toluene)Fe(0)]. Iron is evaporated at temperatures of about 1700°C in high vacuum and co-condensed at the cold walls of the reaction container with toluene. After warming up the walls to around -100°C, the apparatus is flooded with butadiene gas. Around -60°C, a butadiene substitutes one toluene ligand to result in the formation of the desired product [(butadiene)(toluene)Fe(0)]. Based on the evaporated iron, the yield is about 60%. A vacuum distillation step at 100°C leads to analytically pure material.

Under steady state conditions the decomposition of the iron complex starts at about 150°C. NMR analyses of the thermal decomposition products indicate pure toluene and butadiene, only (Fig. 2). No significant decomposition of the ligands could be observed (detection limit 4%).

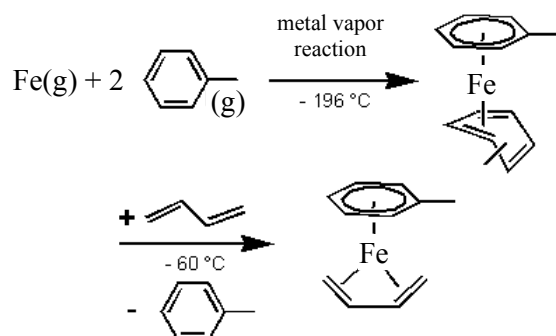


Fig. 1: Preparation of [(butadiene)(toluene)iron(0)]

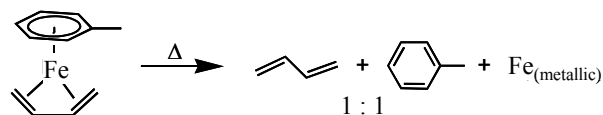


Fig. 2: Thermal decomposition of [(butadiene)(toluene)iron(0)]

The preparation of [(1,5-COD)(toluene)Ru(0)] is given in Fig. 3.

A threefold excess of 1-methyl-1,4-cyclohexadiene is reacted with RuCl₃ in refluxing ethanol and the binuclear arene complex [(toluene)Ru(II)Cl₂]₂ is formed effectively. Its reduction in the presence of 1,5-cyclooctadiene (1,5-COD) leads to the target compound [(1,5-COD)(toluene)Ru(0)] in good yield.

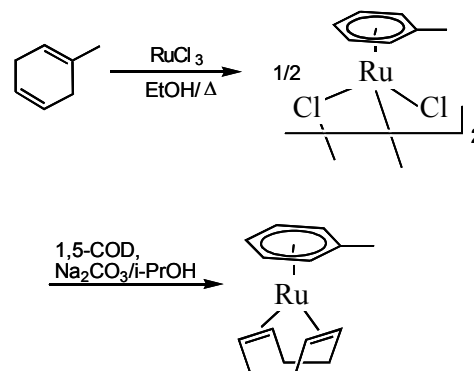


Fig.3 : Preparation of [(1,5-cyclooctadiene)(toluene)ruthenium(0)]

MOCVD experiments need only relatively low temperatures (< 250°C) to deposit iron and ruthenium. The possibility to deposit closed layers is given. As a result of the layer examination the layers are contaminated with carbon. Reducing the residence time suppresses the possibility of adsorption of the ligands and the consecutive decomposition of the ligands on the surface. This leads to very low carbon contaminated layers (< 3 at.%).

All together the complexes [(butadiene)(toluene)Fe(0)] and [(1,5-cyclooctadiene)(toluene)Ru(0)] are suitable as precursors for MOCVD processes. Further experiments towards analysis of the adsorption and decomposition procedures of the ligands on the surface will be done. From these results and from varying the ligands, a further reduction of the carbon part in the deposited layers can be realized.