Chemical Vapor Deposition of Gold

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We have previously reported the deposition of high purity gold onto various substrates from dimethyl-2,4-pentandionato gold (III), $Me_2Au(acac)$, by localized, laser-induced (photothermal) chemical vapor deposition.[1,2] We now report preliminary results showing that high-purity gold films can also be deposited by large-area chemical vapor deposition (CVD) from this complex as well as from two fluorinated derivatives, dimethyl-(1,1,1-trifluoro-2,4-pentandionato) gold (III) and dimethyl-(1,1,1,5,5,5-hexafluoro-2,4-pentandionato) gold (III), $Me_2Au(tfac)$ and $Me_2Au(hfac)$, respectively. We discuss the conditions of deposition and the quality of the gold films obtained as well as the properties of the organogold precursors.

$Me_2Au(acac)$ was prepared and purified by the method of Brain and Gibson,[3] as previously reported.[1,2] The fluorinated derivatives were prepared by the same procedure. Room temperature vapor pressures of the pure complexes are reported in Table I. Each complex shows onset of decomposition (neat) at ~160°C by differential scanning calorimetry. CVD of gold from each complex was carried out in a stainless steel vacuum chamber evacuated by a turbomolecular pump to a base pressure of $< 10^{-6}$ torr. A flow of argon carrier gas through a Pyrex vessel containing the gold complex at room temperature is employed to deliver the organogold precursor into the chamber via stainless steel lines. Chamber pressure and carrier gas flow rate could be varied independently. Deposition proceeds on a substrate placed in the chamber in the flow path of the carrier gas and heated by a copper block heater temperature-controlled to within ±1°C.

We chose a single set of deposition conditions to compare directly the deposition rates and the quality of the gold films obtained from each complex. In all runs, the substrate temperature was 300°C, the chamber pressure was 0.5 torr, and the argon carrier gas flow rate was 15 std. cm^3/min. The rates of deposition obtained under these conditions for each complex on SiO_2 are shown in Table I. The deposition rate scales with the vapor pressure of the complex, as expected. The purity of the films, as measured by scanning Auger spectroscopy before and after annealing at 300°C in air, is also shown in Table 1. No carbon, oxygen, or fluorine contamination was observed for gold films obtained from $Me_2Au(tfac)$ and $Me_2Au(hfac)$, while contamintation is observed for gold films obtained from $Me_2Au(acac)$. The Auger results are reported for films that had been argon ion-sputtered to a depth of ~300 Å to remove surface contaminants. The excellent purity of the gold films is reflected in their resistivity values:

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after annealing, resistivity values are approximately twice the resistivity of bulk gold (2.44 μΩ-cm) for films obtained by CVD from the fluorinated complexes.[4] A significant drop in resistivity is observed for the $Me_2Au(tfac)$ film upon annealing, while less difference is seen for the $Me_2Au(hfac)$ film. This follows the trend observed in film morphology. The deposits obtained before annealing from both fluorinated derivatives show a grainy surface by scanning electron microscopy, but the grain size found in the $Me_2Au(tfac)$ films is noticeable smaller than that in the $Me_2Au(hfac)$ films (~0.5 μm vs. ~1 μm). Upon annealing the $Me_2Au(tfac)$ films, the grains clearly coalesce, while the $Me_2Au(hfac)$ films maintain a grainy structure. Significant improvement in morphology can probably be obtained by optimizing the deposition conditions for each complex.

In conclusion, $Me_2Au(acac)$, $Me_2Au(tfac)$, and $Me_2Au(hfac)$ have been shown to be excellent precursors for CVD of gold. We are currently working to optimize the deposition conditions on a variety of substrates.

Table I. Vapor pressures (mtorr) at 24°C, CVD rates (Å/min.), deposit contamination level (atom %) before (after) annealing, and deposit resistivity (ρ, μΩ-cm) before (after) annealing for $Me_2Au(acac)$, $Me_2Au(tfac)$, and $Me_2Au(hfac)$.

Complex	acac	tfac	hfac
vap. P	9	40	400
dep. rate	13	140	940
C,O,F	15,0,-	none	none
	(0,4,-)	none	none
ρ	-	21.2	6.4
	-	(4.7)	(5.1)

References.

1. T. H. Baum and C. R. Jones, Appl. Phys. Lett., 47, 538 (1985).

2. T. H. Baum and C. R. Jones, J. Vac. Sci. Tech. B, in press.

3. F. H. Brain and C. S. Gibson, J. Chem Soc., 762 (1939).

4. Unfortunately, films obtained by CVD from $Me_2Au(acac)$ could not be grown to a sufficient thickness to obtain resistivity values.

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