Deposition of Metallic Films by Electron Impact Decomposition of Organometallic Vapors

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The use of voltage variable capacitors in a similar application has been reported by Jennings and Tanttila[3]; however, the circuit depicted was a particularly simple marginal oscillator employing broad modulation to display strong resonances on an oscilloscope.

In this Laboratory the Robinson oscillator, in conjunction with the modifications herein described, has been used to produce line shape derivatives of pure quadrupole resonances in nitrogenous compounds possessing large asymmetry parameters and at low temperatures (Fig. 2). The system is sufficiently stable to conveniently search over long periods of time for unknown resonances and has been used by us to find several.[4]

[1] P. A. Casabella and P. J. Bray, J. Chem. Phys. **29**, 1105 (1958).
[2] F. N. H. Robinson, J. Sci. Instr. **36**, 481 (1959).
[3] D. A. Jennings and W. H. Tanttila, Rev. Sci. Instr. **30**, 137 (1959).
[4] C. H. Dutcher, Jr., and T. A. Scott (to be published).

Deposition of Metallic Films by Electron Impact Decomposition of Organometallic Vapors*

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AN electron beam with a current density of approximately 5 ma/cm^2 operated with potentials between 50 v and 1 kv has been used to decompose organometallic vapors to produce thin metallic films.

An electron gun and substrate are mounted in a bell jar which can be evacuated by a conventional diffusion pump and nitrogen cold trap.

The electron gun has a tantalum flat spiral filament, a screen grid, a deaccelerator electrode and a Pierce electrostatic axial flow lens system. The screen grid, held at a fixed accelerating potential, makes it possible to adjust the final accelerating voltage without appreciably affecting the beam current. The deaccelerator acts as an artificial cathode to the Pierce section by slowing the electrons to about 20 v before they enter the first Pierce electrode. The final accelerating potential can be adjusted from 30 to 1200 v.

Plain glass, glass with an evaporated metal layer, and metal substrates have been used. The latter two are used to eliminate charge effects when the beam potential would be below the first secondary emission crossover of the glass.

Compounds with vapor pressures between 10 μ and 100 mm of Hg have been used so that their vapor can be easily let into the vacuum system at room temperature. Tetrabutyltin, tetramethyltin, and tetraethyllead have been tried.

The system is first evacuated to a pressure of approximately 10^{-5} mm of Hg. Organometallic vapor is then let into the system until the pressure as read by a conventional ionization gauge is 0.5 μ of Hg. The electron beam is then allowed to spray the substrate with electrons thus producing a film on the substrate.

Two modes of operation have been investigated. A molecular starving is achieved by having more electrons arrive in unit time than are needed to decompose the molecules arriving at the substrate during that time. This mode gives films which do not show the nonuniformity of the electron beam. A second mode of operation can be realized by allowing more molecules to strike the substrate than the electron beam can accommodate. The films produced with this mode of operation show nonuniformities which appear to be due to the nonuniform beam density.

Films between 50 and 1500 A in thickness have been produced. Highly reflecting tin films with surface resistances from 50 ohms per square to about 1000 ohms per square have been made. Other films have resistances as high as 1 meg per square and have a brown appearance. The brown appearance and high resistance might be attributed to structural peculiarities caused by the presence of carbon which has been deposited by the decomposition of the carbon-hydrogen radicals of the organometallic molecules or from outgassing products from the rubber sealing gaskets.

Little has been done with the energetics of the process. The success of a number of the runs, however, might indicate that the radicals can be removed intact leaving only the metal atom on the substrate.

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Protection of Electrodes by Local Potting*

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PROTECTION of electrical components by resin potting has become commonplace in such applications as missile systems fabrication. The purpose of this note is

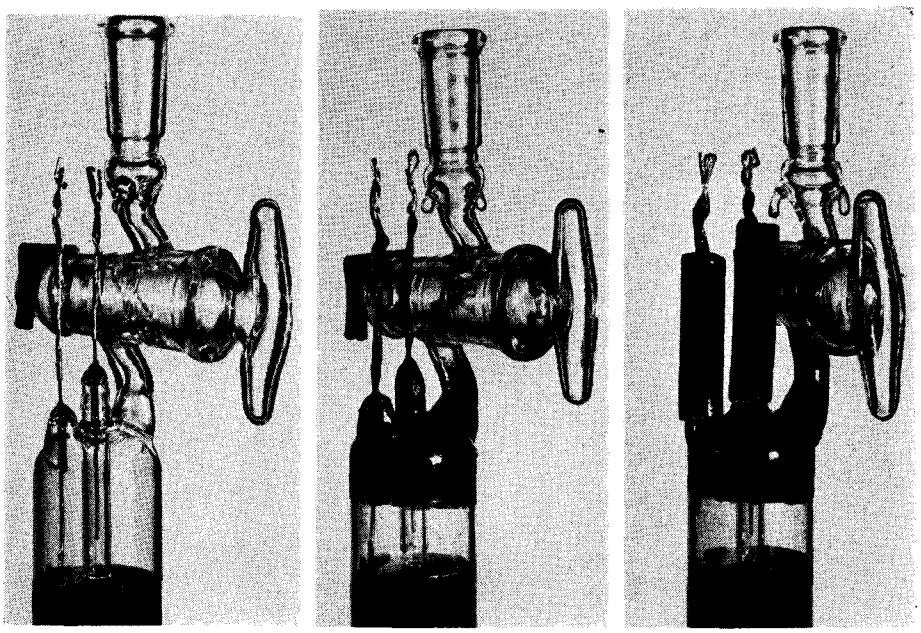

FIG. 1. Electrode bearing end, Bernstein-Ballentine type gas counting tube. (Left)—bare leads, as tube is purchased. (Middle)—three layers of a noninflammable, fluorinated, thermoset polymer have been applied to electrodes (except top $\frac{3}{4}$ in.) and contiguous glass surfaces. (Right)—plastic sleeves were sealed on the coated glass electrode-bearing nubs. After filling with epoxy resin and curing, the tube is ready for service.

TABLE I. Background and C^{14} counting performance of 3 sizes of Bernstein-Ballentine type gas-phase counting tubes (methane filling gas, atmospheric pressure). In excess of 10 000 total counts were accumulated for each observation.

Dimensions, external, length×diameter	Operating voltage	Counts/min Bare leads Background	Bare leads $C^{14}O_2$[a]	Coated leads Background	Coated leads $C^{14}O_2$[a]	Potted leads Background	Potted leads $C^{14}O_2$[a]
46.0×2.1 cm	2900	127	4049	127	4025	126	4040
45.9×2.4 cm	3100	114	4552	111	4545	110	4543
46.0×2.8 cm	3500	123	4144	122	4160	125	4101

[a] Net count. A single sample of $C^{14}O_2$ was used for the three observations with a given tube.

to call attention to the laboratory use of local potting, as illustrated by potting the electrodes of gas-phase Bernstein-Ballentine glass counting tubes.[1,2] Satisfactory mechanical protection of electrodes along with adequate electrical insulation has resulted from epoxy resin potting after coating with a fluorinated thermoset polymer. Potting of the entire servicing end of counting tubes was first tried but was abandoned because breakage of glass electrode-bearing nubs and/or gas-filling tubulation occurred as the epoxy resin expanded slightly in curing; individual potting of each electrode has worked well.

1. Coating: Electrodes and contiguous glass surfaces were washed with acetone. When dry, a thin layer of freshly mixed coating[3] was brushed on, air dried for 30 min, and then baked at 100°C for 30 min. Two additional layers were applied (Fig. 1, middle).

2. Potting: A $1\frac{1}{4}$ in. long plastic sleeve[4] was worked onto each coated glass electrode-bearing nub. Each sleeve was sealed to the glass coating by just filling the ring of contact with fresh liquid coating, and then air drying for 30 min. Freshly mixed epoxy resin[5] was heated to about 60°C in an electric oven so that bubbles introduced at mixing dissipated as the warming mix became less viscous. It was then possible to use a glass capillary pipette to fill the plastic sleeves with resin. Curing took place with the tubes held upright in a chemical fume hood (exhaust fan off) ambient temperature about 60°C, overnight. The end result is pictured in Fig. 1 (right).

We have not lost a tube through electrode breakage after potting. The adequacy of electrical insulation is attested by the count data presented in Table I.

* Work supported by grant from the National Institutes of Allergy and Infectious Diseases, U. S. Public Health Service, Department of Health, Education and Welfare, Bethesda 14, Maryland.

[1] W. Bernstein and R. Ballantine, Rev. Sci. Instr. **21**, 158 (1950).

[2] Obtained from C. A. Nawrocki, Box 458, Center Moriches, Long Island, New York.

[3] Coating 1510 A and B, Applied Plastics Division, Hexcel Products, Inc., 130 Penn Street, El Segundo, California.

[4] Imperial Poly-Flo tubing, 66P, $\frac{1}{4}$ in. i.d., $\frac{3}{8}$ in. o.d., Industrial Supply Company, Inc., 131 Social Hall Avenue, Salt Lake City, Utah.

[5] Epon 828 and Curing Agent D, Shell Chemical Company, 10642 Downey Avenue, Downey, California.