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A Novel Method of making Metallic Catalysts

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There are several reports extant of the use of the exploding-wire technique for inorganic synthesis,[1,2] and for the preparation of metal aerosols.[3,4] We have applied this technique to the preparation of aerosols of metals which are of catalytic interest since, unlike the conventional methods,[5] it offers a method of preparing metal powders which are substantially free from contamination. It also provides a possible method of making catalysts *in situ* in reaction mixtures.

The explosion of 32 cm.-lengths of 0·008 in. diameter nickel, palladium, and platinum wires is carried out in *ca.* 200 mm. of spectroscopically-pure argon or purified hydrogen using energies of 1000—2000 joules (Ni or Pd), or 3200—4000 joules (Pt). The 20 μF capacitor bank, which can be charged up to 20 kv, is fired by ionizing the air gap between two stainless steel spheres. The explosion chamber is constructed from Q.V.F. Pyrex glassware and has a wall thickness of 4 mm. and a volume of 850 c.c. The wires are first outgassed at their evaporation temperature in an external apparatus and then *in situ* at 2·5 amps for 6 hours. At this stage the explosion chamber is at a pressure of 10^{-6} torr.

Explosion results in the formation of an incoherent film on the walls of the chamber, together with a smoke which settles out after a few minutes. After the explosion gas has been pumped away, the catalytic activity of the metal is tested with a 1 : 1 ethylene: hydrogen mixture. The results are summarized in the Table.

Experimental comparison of the activity of exploded palladium with that of evaporated palladium films shows that, on a weight-for-weight

Table

Wire	Explosion Conditions	Catalytic activity	Chemisorption H_2	Chemisorption C_2H_4
Pd	14 kv in 185—201 mm. Ar	No	Yes	No
Pd	14 kv in 180 mm. H_2	Yes	—	Yes
Pd	10 kv in 171 mm. H_2	Yes	—	—
Pd	13 kv in 165 mm. H_2	Yes	—	—
Pt	18·5—20 kv in Ar or H_2	No	—	—
Ni	12—15 kv in Ar or H_2	No	Yes	No

[1] M. F. Mahieux, *Compt. Rend.*, 1963, **257**, 1083.
[2] M. J. Jancich and D. G. Reu. "Exploding Wires, 3" (ed. W. G. Chace and H. K. Moore), Plenum Press, New York, 1964, p. 353.
[3] F. G. Karioris and B. R. Fish, *J. Colloid Sci.*, 1962, **17**, 155.
[4] C. P. Nash and R. P. Desieno, *J. Phys. Chem.*, 1965, **69**, 2139.
[5] G. C. Bond, "Catalysis by Metals," Academic Press, London, 1962, Ch. 3.

basis, the former are some ten times more active for ethylene hydrogenation at 20° c.

In an attempt to understand the inactivity of the argon-exploded wires, studies of the chemisorption properties and physical structure of the exploded wires are being undertaken. Electron-microscopic studies of argon-exploded nickel show that the metal particles are crystalline and have a mean diameter of around 250 Å. The preliminary results also suggest that the particles may be mono-crystalline.

Chemisorption studies indicate that argon-exploded nickel and palladium are both active for hydrogen chemisorption, but inactive for ethylene chemisorption. With hydrogen-exploded palladium films, ethylene chemisorption is accompanied by some hydrogenation. At 21°c the total uptake of ethylene is $1{\cdot}6 \times 10^{-5}$ millimoles of ethylene per mg. of palladium. At 22°c, the ratio of hydrogen : nickel atoms at saturation is ~1 : 800 for a 58 mg. exploded wire compared with ~1 : 65 for a 28 mg. evaporated film. With 53 mg. of argon-exploded palladium the corresponding ratio is ~1 : 16. The results for ethylene chemisorption are in apparent conflict with the results of Nash and Desieno[4] for acetylene adsorption on argon-exploded nickel and palladium. This anomaly may arise from the different heats of adsorption of ethylene and acetylene. Alternatively, it may be that our exploded wires are more sintered than those of Nash and Desieno, since our argon pressures are substantially lower than 760 mm. and the heat transfer from exploded metal to the surrounding atmosphere will be less efficient. The lower H : Ni ratio supports the suggestion that the exploded wire is highly sintered.

Further studies of these surfaces are being carried out in an attempt to further our knowledge of the factors affecting adsorption and catalysis.

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