

ane (containing 3% Ne as internal standard), at 180°C for 3 hours with 10 ml of triflic acid containing 2.0 mmol of mercury(II) triflate in a 50-ml high-pressure Autoclave Engineers Hastaloy-C reactor equipped with Desperi-Max gas liquid mixer. Quantitative analysis of the samples was challenging; GC analysis of the crude reaction mixtures was inadequate. Routine analyses were carried out with high-performance liquid chromatography to quantify the methanol produced by hydrolysis of a reaction aliquot. In selected cases, qualitative and quantitative ^{13}C NMR analyses of the crude reaction mixtures with acetic acid as an internal standard (added after reaction) were used to confirm the results. In reported cases, the mass balance on methane was >90%. This mass balance was obtained by accounting for unreacted methane, methyl bisulfate (as methanol after hydrolysis), and carbon dioxide. To obtain good mass balance, the methane-Ne mixture was dispensed from a known-volume, known-pressure reservoir. This procedure allowed the total moles of methane delivered to the reactor to be determined. The moles of methane remaining after reaction and carbon dioxide produced were determined by GC analysis of the gas phase by using Ne as an internal standard.

21. Reactions were carried out as described in (20) but with a 1-liter Hastaloy-C reactor and 300 ml of a 0.1 M solution of $\text{Hg}(\text{OSO}_3\text{H})_2$ in 100% sulfuric acid. Higher selectivities were achieved at lower conversions. The selectivity was observed to decrease at higher conversions.
22. Oxidative addition of methane to $\text{Hg}(\text{II})$ would be formally described as involving the formation of methyl mercury(IV) hydride, $(\text{CH}_3)(\text{H})\text{Hg}(\text{IV})$.
23. An intriguing possibility is that a complex involving coordinated methane and solvated $[\text{Hg}(\text{OSO}_3\text{H})]^+$ might be an intermediate on the reaction pathway to **1**. Such coordinated complexes have been proposed; see R. A. Periana and R. G. Bergman, *J. Am. Chem. Soc.* **108**, 7332 (1986).
24. We are deeply indebted to H. Taube, Marguerite Blake Wilbur Professor Emeritus at Stanford University and Scientific Advisor to Catalytica, Inc., for the insight and guidance provided during all phases of our investigation of selective methane oxidation. The results described here were obtained in the course of a joint research and development program among Petro-Canada, Technomico, Inc., a wholly owned subsidiary of Mitsubishi Oil Co., Ltd., and Catalytica, Inc.

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Production of Syngas by Direct Catalytic Oxidation of Methane

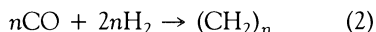
D. A. Hickman* and L. D. Schmidt

The reaction between methane and oxygen over platinum and rhodium surfaces in metal-coated ceramic monoliths can be made to produce mostly hydrogen and carbon monoxide (greater than 90% selectivity for both) with almost complete conversion of methane and oxygen at reaction times as short as 10^{-3} seconds. This process has great promise for conversion of abundant natural gas into liquid products such as methanol and hydrocarbons, which can be easily transported from remote locations. Rhodium was considerably superior to platinum in producing more H_2 and less H_2O , which can be explained by the known chemistry and kinetics of reactants, intermediates, and products on these surfaces.

Natural gas is our most abundant, clean, and easily extractable energy source. However, most methane (CH_4) is in locations far from consumption sites, and its transportation is expensive as gas and hazardous as liquid or compressed gas. Therefore, it is desirable to convert methane into liquid products for more efficient utilization. The conventional route to liquid fuel production is through a syngas intermediate, a $\text{CO} + \text{H}_2$ mixture, which can then be easily reacted to form either methanol (CH_3OH) (1) over Cu/ZnO catalysts:

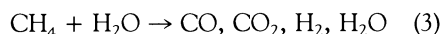


or to hydrocarbons through the Fischer-Tropsch process (1) over Fe and Ru catalysts:



Syngas is typically produced by reacting

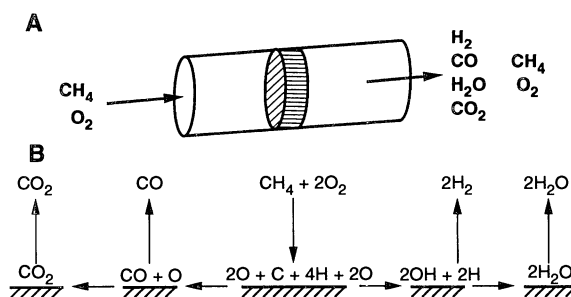
CH_4 with H_2O at $\sim 800^\circ\text{C}$ to produce a mixture of CO , H_2 , H_2O , and CO_2 in a process termed steam reforming (1):



The products are then converted into a 2:1 H_2/CO mixture (syngas) or into a $\text{H}_2 + \text{CO}_2$ mixture (hydrogen generation) by adding excess steam in the water gas shift reaction (1):



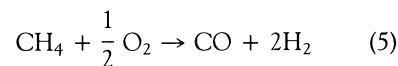
Fig. 1. (A) Sketch of the experimental reactor. (B) Schematic illustration of the adsorption, desorption, and surface reaction steps for which whose kinetic parameters are given in Table 1 and for which energies are shown in Fig. 3.



The conventional production of syngas or of H_2 therefore requires at least two reactors, a steam reformer and shifter, and excess CO_2 by-product is necessarily produced that must be vented. Residence times in each of these reactors are on the order of 1 s, so the size of a world-scale liquid fuel plant is very large.

An alternative method to produce hydrocarbons, particularly ethylene, is oxidative coupling (2, 3), in which CH_4 and O_2 flow over a mixed oxide catalyst which produces gaseous CH_3 radicals that ultimately form ethane and ethylene. Although this process is a topic of active research, it currently is limited by either low conversion of CH_4 or low selectivity to hydrocarbon products. Several publications have also appeared in which CO and H_2 are produced with only CH_4 and O_2 reactants (4–9). These processes were thought to involve first the oxidation of CH_4 primarily to H_2O , CO , and CO_2 followed by reaction of CH_4 with H_2O (reaction 2) and the water gas shift reaction (reaction 4), giving fairly long reactor residence times of 0.01 to 1 s. In fact, some H_2 may be formed initially in those experiments by the direct oxidation reaction (5). However, because the catalyst and reaction conditions are not optimized for this reaction, the formation of syngas by the direct oxidation reaction is not believed to be the primary route of H_2 generation (4–9).

We have produced syngas in nearly a 2:1 ratio by the direct catalytic oxidation of CH_4 :



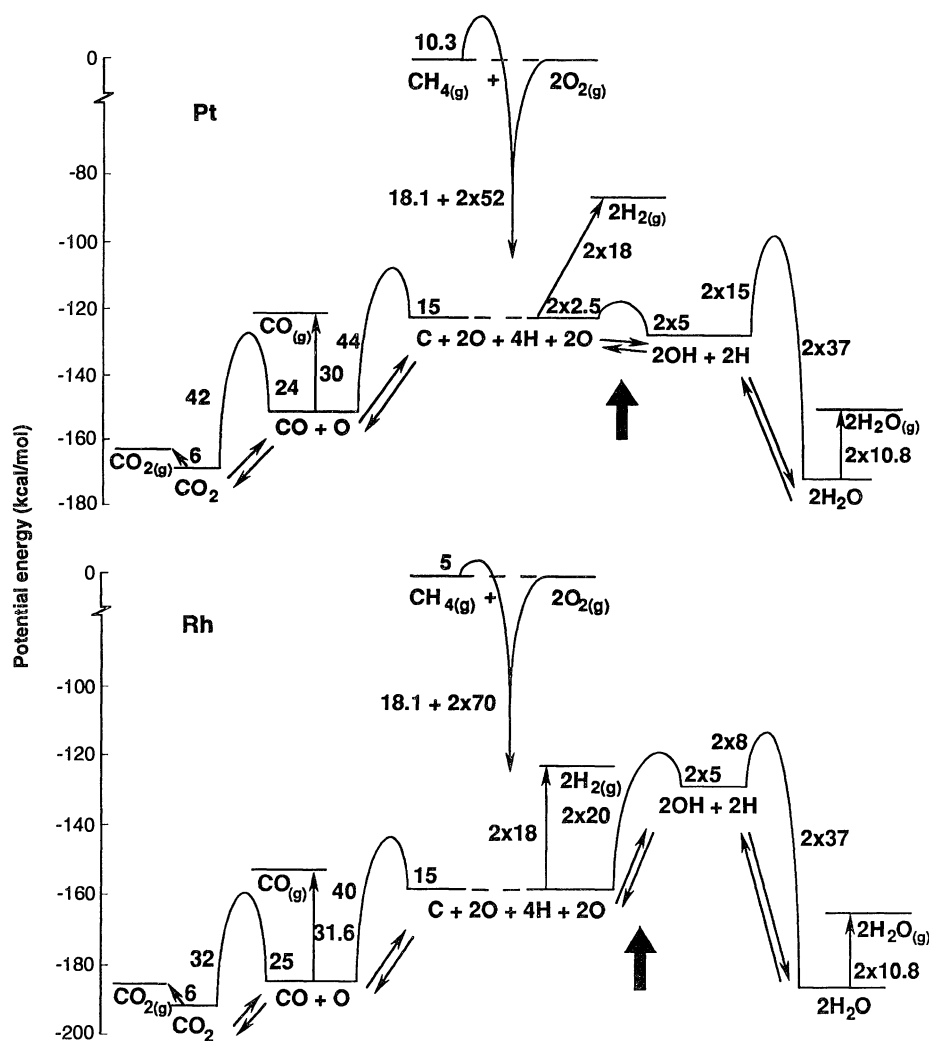
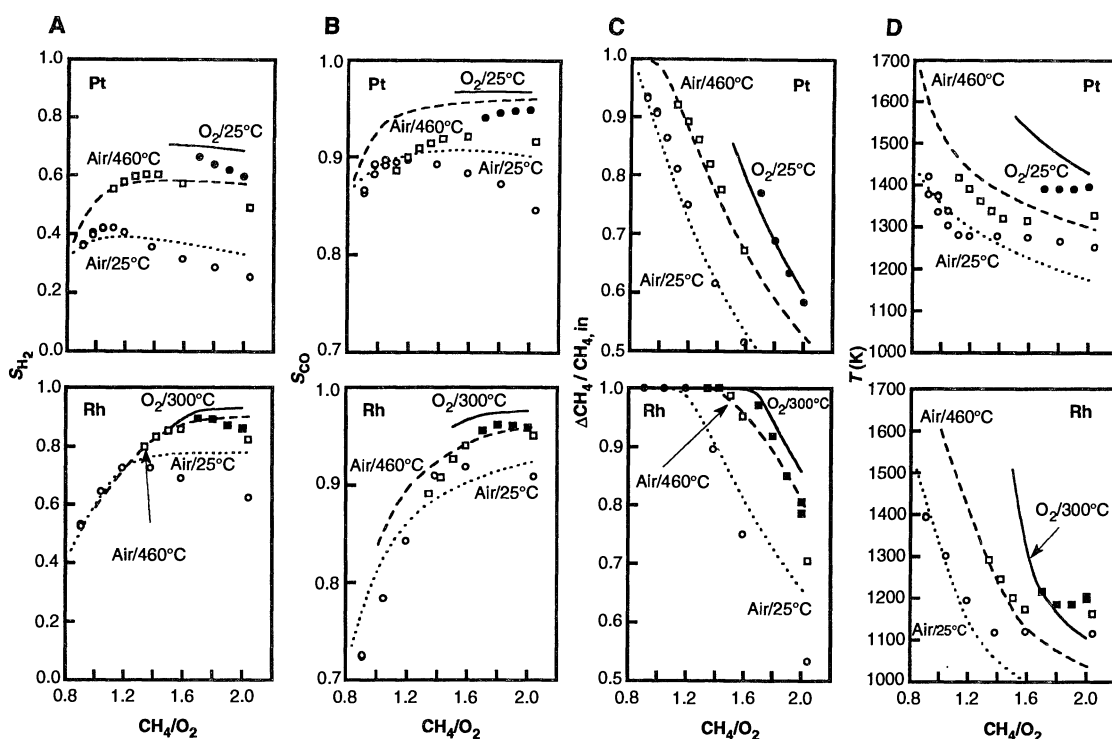
by using oxygen or air over Pt or Rh surfaces and feed gases near room temperature in a single monolith reactor with extremely short residence times. This reaction is entirely a surface process because homogeneous reaction in flames requires a combustible reactant mixture and would proceed by free-radical intermediates, thus generating very little H_2 .

It is, of course, very surprising that combustion of CH_4 in O_2 should produce H_2 because: (i) the $\text{H}_2 + \text{O}_2$ reaction is very fast at high temperatures, either homoge-

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Fig. 2. A comparison of experiments (symbols) and calculations (curves) for (A) H_2 and (B) CO selectivities, (C) CH_4 conversions, and (D) surface temperatures for methane oxidation over porous alumina foam monoliths coated with 10 to 12% Pt and Rh. Open symbols and dashed lines represent CH_4 -air feeds, closed symbols and solid lines represent CH_4 - O_2 feeds, circles represent feed temperatures of 25°C, and squares represent feeds preheated to 460°C (air) or 300°C (O_2).



neously or heterogeneously; (ii) CH_4 is difficult to dissociate and requires high temperatures, either homogeneously or heterogeneously; and (iii) graphitic carbon is predicted by thermodynamics to form in the reactor and deactivate all catalyst surfaces except at very high temperatures ($\sim 1000^\circ\text{C}$ in 2:1 CH_4/O_2 mixtures).

We constructed a small adiabatic flow reactor that closely approximates the conditions that would be used in a large-scale methane processing reactor. As sketched in Fig. 1A, gases flowed through a 18-mm diameter quartz tube containing a cylinder of porous alumina foam ~ 1 cm long that was impregnated with Pt or Rh salts to metal loadings between 1 and 20% by weight. Methane and either air or oxygen flowed through this foam at a rate of ~ 4 standard liters per minute at a pressure of 1.4 atm. The catalyst was ignited by preheating NH_3 and air to $\sim 200^\circ\text{C}$ (the light-off temperature); the system's steady-state temperature was 850° to 1150°C for inlet temperatures between 25° and 460°C . The gas velocity at reaction temperatures was ~ 1 m/s, giving a residence

Fig. 3. Potential energy surfaces for the CH_4 oxidation reaction system at low coverages and high temperatures on Pt and Rh surfaces. The energy values are activation energies from Table 1 and are calculated relative to $\text{CH}_4(\text{g})$ and $\text{O}_2(\text{g})$ at 298 K. Gas-phase species are denoted by the subscript g; all other species are surface species. The different barriers for $\text{H} + \text{O} \rightarrow \text{OH}$ on Pt and Rh correlate with the difference in H_2 selectivities.

Table 1. Rate parameters for Pt and Rh.

Reaction	Platinum			Rhodium		
	k_o^*	E_a (kcal/mol)	Ref.	k_o	E_a (kcal/mol)	Ref.
$\text{CH}_{4(g)} \rightarrow \text{C} + 4\text{H}$	5×10^4	10.3	(10)	3×10^4	5	(17)
$\text{H} + \text{O} \rightarrow \text{OH}$	1×10^{15}	2.5	(11)	7×10^{12}	20	(18)
$\text{OH} \rightarrow \text{O} + \text{H}$	1×10^8	5	(11)	1×10^{13}	5	(19)
$\text{H} + \text{OH} \rightarrow \text{H}_2\text{O}$	9×10^{16}	15	(11)	3×10^{17}	8	(19)
$\text{H}_2\text{O} \rightarrow \text{OH} + \text{H}$	1.8×10^{13}	37	(11)	5×10^{14}	37	(19)
$2\text{OH} \rightarrow \text{H}_2\text{O} + \text{O}$	1×10^{15}	12.3	(12)	4×10^{15}	15	(19)
$\text{H}_2\text{O} + \text{O} \rightarrow 2\text{OH}$	~ 0	31	†	~ 0	63	†
$\text{H}_{2(g)} \rightarrow 2\text{H}$	7.5×10^4 ($s = 0.05$)	0	(11)	2.25×10^5 ($s = 0.16$)	0	(19)
$2\text{H} \rightarrow \text{H}_{2(g)}$	5×10^{12}	18	(13)	5×10^{12}	18	(20)
$\text{O}_{2(g)} \rightarrow 2\text{O}$	1.25×10^3 ($s = 0.003$)	0	‡	3.5×10^3 ($s = 0.01$)	0	(21)
$2\text{O} \rightarrow \text{O}_{2(g)}$	5×10^{12}	52	(14)	5×10^{12}	70	(19, 22)
$\text{H}_2\text{O}_{(g)} \rightarrow \text{H}_2\text{O}$	5×10^4 ($s = 0.1$)	0	(11)	7.4×10^4 ($s = 0.16$)	0	(19)
$\text{H}_2\text{O} \rightarrow \text{H}_2\text{O}_{(g)}$	1×10^{13}	10.8	(12)	1×10^{13}	10.8	(23, 24)
$\text{C} + \text{O} \rightarrow \text{CO}$	5×10^{13}	15	‡	5×10^{13}	15	‡
$\text{CO} \rightarrow \text{C} + \text{O}$	1×10^{11}	44	‡	1×10^{11}	40	‡
$\text{CO}_{2(g)} \rightarrow \text{CO} + \text{O}$	~ 0	36	†	~ 0	26	†
$\text{CO} + \text{O} \rightarrow \text{CO}_{2(g)}$	1×10^{15}	24	(15)	1×10^{12}	25	(25, 26)
$\text{CO}_{(g)} \rightarrow \text{CO}$	3.21×10^5 ($s = 0.84$)	0	(16)	1.91×10^5 ($s = 0.5$)	0	(26)
$\text{CO} \rightarrow \text{CO}_{(g)}$	1×10^{13}	30	(13)	4×10^{13}	31.6	(27)

*All rates are in monolayers per second with coverages in monolayers. Rate coefficients are assumed to have the form $k = k_o \exp[-E_a/RT]$ (where E_a is the activation energy and R is the gas constant) with k_o in s^{-1} except for adsorption rates, which are in $\text{torr}^{-1} \text{s}^{-1}$ with sticking coefficients (s) indicated in parentheses. †Thermodynamic constraints. ‡This work.

time on the catalyst of ~ 10 ms. Products were analyzed for all species by a three-column gas chromatograph.

Stoichiometric ratios in air are 12.3% ($\text{CH}_4/\text{O}_2 = 1$) and 29.6% ($\text{CH}_4/\text{O}_2 = 2$) CH_4 for $\text{CO} + \text{H}_2\text{O}$ and $\text{CO} + \text{H}_2$, respectively, and 50 and 67% in pure oxygen. If all of the CH_4 and O_2 react for CH_4 feeds at the stoichiometric ratio for $\text{CO} + \text{H}_2$, then the selectivities must be 100%; lower selectivities require CH_4 breakthrough.

Figure 2 shows plots of H_2 and CO selectivities, defined in terms of product partial pressures as:

$$S_{\text{H}_2} = \frac{P_{\text{H}_2}}{(P_{\text{H}_2} + P_{\text{H}_2\text{O}})} \quad (6)$$

$$S_{\text{CO}} = \frac{P_{\text{CO}}}{(P_{\text{CO}} + P_{\text{CO}_2})} \quad (7)$$

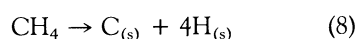
and the fraction of CH_4 converted over Pt and Rh catalysts versus the ratio of CH_4/O_2 in the feed. As expected, the temperature decreases as CH_4 in the feed increases because the heats of reaction to form CO and H_2 are less than those for CO_2 and H_2O . The selectivities attain maxima at CH_4/O_2 feed ratios of ~ 1.0 to 1.5 on Pt and ~ 1.2 to 1.6 on Rh, with significant CH_4 breakthrough for higher CH_4 feed concentrations.

It is seen that Rh gives consistently higher selectivities to H_2 , lower temperatures, and

higher CH_4 fractional conversions than does Pt. These results were repeated on several samples with reproducibilities within a few percent and no significant catalyst deactivation over at least several hours of operation. The temperature is lower over Rh than Pt because of the lower exothermicity of the CO and H_2 reactions. Oxygen also gives uniformly higher selectivities than air because of the higher temperatures without N_2 diluent.

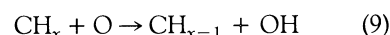
We have constructed a complete model that incorporates all of the elementary adsorption, desorption, and surface reaction steps in these reactions with experimental parameters taken from surface science experiments involving adsorption and desorption kinetics, identification of OH intermediate by laser-induced fluorescence (LIF), and measurement of kinetics of CH_4 dissociative adsorption and CO oxidation (summarized in Table 1). Figure 1B shows the steps implied by the proposed reaction mechanism, and the energy diagrams in Fig. 3 show activation energies for each step from Table 1.

For methane oxidation, we assumed that the first step was dissociative adsorption [where (s) indicates adsorption on a surface]:



Irreversible adsorption was assumed because the oxidation reactions are very fast, so that little CH_4 is expected to desorb under these reaction conditions. Reaction

8 also lumps the breaking of all C–H bonds into one step. This assumption does not eliminate other adsorption mechanisms in which H atoms dissociate sequentially but does eliminate the possibility of the abstraction of H atoms from adsorbed CH_x species by O adatoms:



The high selectivities for H_2 formation on Pt and Rh suggest that such abstraction reactions are not a dominant reaction step for CH_4 -rich oxidation at high temperatures because they should lead inevitably to formation of H_2O , which increases at lower metal loadings because of those reactions on the alumina foam.

It is seen in Fig. 2 that all data are fit by the model equations within a few percent and that in most situations the increases, decreases, and maxima in S_{H_2} and S_{CO} are closely reproduced by the model. The model predicts a higher S_{H_2} on Rh compared to Pt, in close agreement with experiments. This difference is almost completely due to the different rates in the $\text{H} + \text{O} \rightarrow \text{H}_2\text{O}$ sequence, steps 1 to 3 in Table 1, because the C oxidation rates are essentially identical on the two surfaces. On Pt there is a very small barrier toward formation of OH, step 1, whereas on Rh there is a large barrier, and OH exists near the top of the barrier between $\text{H} + \text{O}$ and H_2O (Fig. 3). The adsorbed OH species is much less stable on Rh than on Pt, which slows the surface reaction $\text{O} + \text{H} \rightarrow \text{OH} \rightarrow \text{H}_2\text{O}$ on Rh and makes the reaction $\text{H} + \text{H} \rightarrow \text{H}_2$, with an activation energy barrier of 18 kcal/mol on both surfaces, the favored reaction channel on Rh.

The essential features of these experiments that permit quantitative modeling are high temperatures and noble metals. All coverages are calculated to be small even at atmospheric pressure, and the coverage dependences of rate parameters and site competition between adsorbed species can therefore be ignored. Extension to lower temperatures and to other surfaces would require modification of parameters, but these low-coverage models represent the first step in accurate modeling of catalytic processes.

It is clear that CH_4 and O_2 can be reacted over noble metal catalysts to produce CO and H_2 with very high CH_4 yields and selectivities. Contact times of gases within the catalyst in these steady-state experiments are typically 10^{-3} s, and we have obtained nearly the same selectivities with metal gauze catalysts at residence times as low as $\sim 10^{-4}$ s. Our experiments and the model predict no significant homogeneous reaction contributions. We believe that homogeneous reactions can be neglected because the gas composition is far

from the flammability regime, residence times are very short, and catalyst walls inhibit gas-phase free-radical chain reactions.

The proposed mechanism of catalytic CH_4 pyrolysis followed by H_2 desorption and carbon oxidation appears to explain the high selectivity of syngas formation very well by using elementary steps from the surface science literature. The large differences between Pt and Rh also correlate well with their adsorption, desorption, and reaction properties. The ability to utilize models of catalytic processes such as these will be crucial in designing catalytic combustors and reactors for pollution control and efficient synthesis of chemicals with catalytic processes.

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Single Crystal Metals Encapsulated in Carbon Nanoparticles

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Single-domain microcrystals of LaC_2 encapsulated within nanoscale polyhedral carbon particles have been synthesized in a carbon arc. Typical particle sizes are on the order of 20 to 40 nanometers. The stoichiometry and phase of the La-containing crystals have been assigned from characteristic lattice spacings observed by high-resolution transmission electron microscopy and energy dispersive spectroscopy (EDS). EDS spectra show that La and C are the only elements present. Characteristic interatomic distances of 3.39 and 2.78 angstroms identify the compound inside the nanoparticle cavities as $\alpha\text{-LaC}_2$, the phase of LaC_2 that is stable at room temperature. Bulk $\alpha\text{-LaC}_2$ is metallic and hydrolytic. Observation of crystals of pure encapsulated $\alpha\text{-LaC}_2$ that were exposed to air for several days before analysis indicates that the LaC_2 is protected from degradation by the carbon polyhedral shells of the nanoparticles. A high percentage of the carbon nanoparticles have encapsulated LaC_2 single crystals. These carbon-coated metal crystals form a new class of materials that can be protected in their pure or carbide forms and may have interesting and useful properties.

The nested carbon nanotubes discovered by Iijima (1, 2) as a by-product of fullerene production have extended the dimensions and geometries of fullerenes into the domain of nanoparticles. Interest in the unique electrical and thermal properties of

these structures has resulted from several theoretical calculations which suggest that the structures have metallic or semiconductor properties (3–7). The discovery of a means for enhanced production of the tubes reported by Ebbeson and Ajayan (8) suggests that sufficient quantities of these particles can be made to permit their properties to be measured.

We have found that the tubes are produced together with a large variety of poly-

hedral carbon nanoparticles that frequently contain internal cavities. A typical transmission electron microscopy (TEM) scan of material obtained from the core of a growth on a carbon electrode in an arc discharge in 500 torr of He (Fig. 1) shows an assembly of tubules together with a variety of nano-dimension carbon particles that are evidently polyhedral shells. The particles consist of several nested layers of carbon enclosing internal cavities of various sizes. They can be considered to be super-giant nested fullerenes with their polyhedral geometries defined by the placement of 12 pentagons in a hexagon lattice. We report here the successful filling of these cavities with metals. We find that the carbon arc conditions which favor nanotube and nanoparticle growth can be exploited to completely encapsulate metals.

In analogy with the production of endohedral fullerenes, our first attempt to fill the cavities of these carbon nanoparticles made use of a graphite rod packed with an electropositive metal. The successful production of La endohedral fullerenes (9–12) suggested that La was a good candidate for incorporation into the nanoparticles. The 7.9-mm-diameter, 30.5-cm-long graphite positive electrode was drilled to a depth of 23 mm with a 3.2-mm drill and packed with La_2O_3 . The rod, which was weighed before and after packing with La_2O_3 , provided an La:C molar ratio of 0.02. The carbon arc conditions included a DC current of 150 A, a gap distance of about 1 mm between the stationary 12.7-mm-diameter negative electrode and the translating positive electrode, and an He pressure of 500 torr.

The deposited cylindrical growth on the end of the negative electrode was substantially more fragile than growths obtained without the La present. Extracting the central core was therefore difficult, and the cylinder often crumbled. The powder obtained was dispersed in ethanol by using an ultrasonic bath and deposited on carbon-coated grids for TEM studies. Conventional bright-field and high-resolution lattice fringe images were obtained with a JEOL 2000-FX TEM operated at 200 kV. Elemental analysis was performed with a high-angle Noran energy dispersive spectroscopy (EDS) analyzer with a Be window and Ge detector. For specific identification of the composition of low Z materials in the carbon nanoparticles, a Phillips CM-30 TEM equipped with a windowless Link EDS analyzer was used.

Figure 2 shows an example of a nanoparticle enclosing what appears to be a single crystal, as evidenced by its regular set of lattice fringes. The crystal boundaries conform to the interior carbon shell walls, which suggests that the particle is completely enclosed within the carbon nanoparticle. An EDS spectrum from the JEOL 2000-FX

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