MICROWAVE PROCESSING OF CYANOGEL COORDINATION POLYMERS TO YIELD BIMETALLIC ALLOYS

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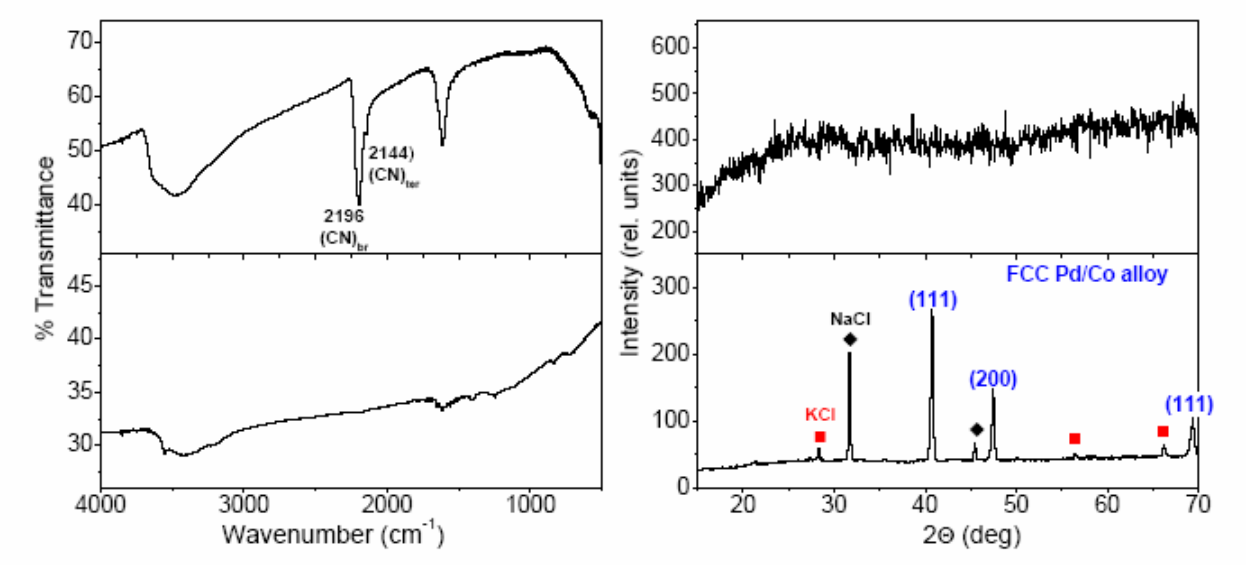
Cyanogels are a class of inorganic gels made of coordination polymers, in which transition metal centers are bridged by cyanide ligands. They are formed as hydrogels via a reaction of tetrachlorometalates with transition metal cyanometalates in aqueous solution. Dehydrated cyanogels can thermally self-reduce under an inert atmosphere to metal alloys at temperatures as low as 350 °C depending on the type of cyanogel. It was shown that a complete thermal reduction of a $Na_2PdCl_4/K_3Co(CN)_6$ cyanogel can be achieved in a domestic microwave oven in 4 minutes [1]. The cyanogel polymer directly absorbs microwave radiation, which leads to sufficient heating of the sample to cause the reduction of the metal centers. The reaction in the microwave oven occurs through similar cyanide-containing intermediates as in the furnace, but on a much shorter time scale. The solid state products of the reaction are identical to the products of thermal reduction in a furnace: Pd/Co face-centered cubic alloy, KCl, NaCl (detected by X-ray powder diffraction), and $CoCl_2$. The cyanide ligands, with characteristic infrared absorption at 2196 and 2144 cm^{-1}, are removed from the system as cyanogen gas, $(CN)_2$. Similar results were obtained for a Pd/Fe cyanogel, where the self-reduction leads to the formation of a face-centered cubic Pd/Fe alloy. The procedure has been applied to a variety of polymetallic systems.



Infrared spectra (left) and X-ray powder diffraction (right) of a Pd/Co cyanogel before (top) and after (bottom) irradiation.

[1] Vondrova, M., Majsztrik, P. W., Gould, S., and Bocarsly, A. B. *Solid-state chemistry in a microwave oven: Preparation of Pd/Co alloy from cyanogel coordination polymers.* Chemistry of Materials **17**(19), 4755-4757 (2005).