

## Metal Oxide + Nanometal Composites

Metal oxide + nanometal composites are a new class of materials that have been demonstrated to be catalytically active in supporting cold fusion using deuterium fuel. A metal oxide + nanometal is a combination of small oxide crystals in interface contact with a nanometal form of metal, such that the contacting layer of metal adjusts to the lattice structure of the oxide. We call such material an "oxide-nanometal composite" (ONC). At present only one form of oxide-nanometal composite has been tested as a catalyst for cold fusion heat production. In the test a zirconium-palladium ONC generated a continuous 10 Watts of excess heat when used in a standard A-Z DS-cathode electrolysis cell. The test run was A-Z's first production of excess heat using a nanometal other than Pd-black. In a second study a "DS-cathode vessel" was filled with zirconium-palladium ONC and exposed to D<sub>2</sub> gas at elevated temperature and pressure. No electrolysis was involved. Continuous heat at an estimated 0.5 Watt was observed. The tested zirconium-metal ONC was a ZrO<sub>2</sub> nanometal Pd composite with 0.33 Pd/Zr atom ratio. In an unrelated program, gas permeation studies were carried out by Iwamura *et al.* in which a permeation flow of deuterium was forced through a Pd plate containing diffusion-impeding structures. These internal structures were produced by sputtering layers of calcium oxide (CaO) and Pd onto a Pd substrate. The sputtered layers are thought to be somewhat equivalent to a CaO-palladium ONC. Electrolysis driven permeation flow produced a reported >1.0 Watt of excess heat in five tests. The Iwamura "ONC catalyst" composition could be described as a CaO-nanoPd layer. It would seem that stable ionic crystals other than ionic oxides could be used to produce catalysts of the same general type.

As discussed later under THEORY, a metal oxide + nanometal composite is thought to be able to create a stable lattice interface between a very chemically stable ionic crystal (very negative Gibbs Free Energy) and a more morphable, electrically conductive metal material. A nanocrystalline metal is especially morphable and can adjust itself to fit commensurably onto the ionic crystal surface. An exact fit interface is called an epitaxy interface. An epitaxy interface layer provides a periodic environment of the type needed to make deuterium ions behave like the conduction electrons of a metal.

Information on metal oxide nanometal composites was first published in 2002 by Yamaura *et al.* from the Institute of Materials Science at Tohoku University in Sendai, Japan. Their paper provides details of the protocol used in producing the zirconium-palladium ONC. A molten alloy of Zr and Pd is rapidly frozen by a spin-cooling ribbon-forming process. In the next steps the thin ribbon of amorphous alloy is oxidized at a relatively low temperature and

pulverized into a powder before use. The authors characterized the powder's internal structure using x-ray and electron scattering. They used electron microscope imagery to "photograph" the internal distribution of nanometal in the  $ZrO_2$ , and its embedded shapes. The research team also carried out laboratory tests showing the material's remarkable ability to absorb hydrogen gas. Similar hydrogen absorption studies were carried out by A-Z prior to examining the composite's excess heat production properties at Osaka University. The high absorption capacity of the powder was confirmed.

At Osaka University a sample of  $ZrO_2 + nanoPd$  composite was tested for its ability to generate nuclear fusion heat. A-Z used the powdered composite to replace commercial Pd-black in their standard electrolysis test cell. It was their first use of a nanometal in a form other than commercial Pd-black. Using their standard electrolysis procedure to pressurize the sample with deuterium, they produced a stable output of cold fusion heat at a 10-Watt level for a period of 3 weeks. The amount of Pd in the sample was a few grams. Their work was published in 2002, the same year as the Yamaura *et al.* study. Their daily output of heat was remarkably steady. It did not seem to show the fluctuations in power that were seen in their Pd-black runs. The data may show a component of fluctuation that does not alter mean energy production, though the relatively rapid power fluctuations may be a metering problem or an illusion. In any case, the data indicate an improved stability in heat production. The data run is the same 2002 test run discussed in the preceding chapter. [See Supplement 1, Figure 5 on page A1-14]

The A-Z 2002 test showed that  $ZrO_2 + nanoPd$  composite provides an effective cure for two problems that have hampered other researchers in their use of nanoPd catalyst. Good repeatability has not been much of a problem for A-Z. The McKubre team, using a DS-cathode assembled, filled with Pd-black, evacuated, and sealed by A-Z, had the same excess heat results as A-Z had when they operated a control experiment prepared at the same time. Both experiments were run with the same protocol. This experiment duplication showed that the technology was transportable between laboratories. But other experimenters have handled their nanopowder differently from A-Z, and have had difficulty producing comparable fusion heat. The problem seems to be that new investigators have followed the normal instructions used by chemists and engineers in preparing catalyst for promoting chemical reactions. They have chemically reduced their catalyst before use. In contrast, A-Z vacuum clean their Pd-black before sealing it off under vacuum, but generally do not chemically reduce it. Commercial Pd-black has an oxide coating that subsequently gets chemically reduced when deuterium diffuses through the wall of the DS-cathode, leaving A-Z's Pd-black

nanocrystals coated with adsorbed D<sub>2</sub>O. Evidence for this is that substantial adsorbed water was found in a mass-spectrometer desorption analysis of post-run powder in a study by Oliver at the Pacific Northwest Lab. The oxygen in the water had to come from surface oxide on the sealed-off Pd-black, since there was no other oxygen available. The presence of water showed that the A-Z Pd-black had not been chemically reduced before being hermetically isolated. The adsorbed water present in Pd-black prepared using the A-Z protocol probably plays the same role as the ZrO<sub>2</sub> in the work using ZrO<sub>2</sub> + nanoPd composites.

An example of inexperienced cold fusion experimenters treating Pd-black prepared in accord with instructions for preparing chemical catalysts prior to use is found in a study by G. Schmidt and T. Chubb. Dr. Schmidt designed and built a system designed to study heat production from Pd-black at pressures as high as 30,000 psi (2000 atmospheres) and temperatures up to 350 °C. The search for fusion heat was unsuccessful, but the deleterious effect of rigorous chemical reduction of Pd-black was discovered. Before-run and after-run powder samples were sent to Asraf Imam at the Naval Research Lab (NRL) for x-ray diffraction study. Imam's Bragg diffraction spectra are shown below in Fig. 2.6,1. The before-run spectrum shows unusually broad line widths, which means that the effective grain size was a few nanometers, despite the manufacturer's characterization of the material size as 0.3 micron (300 nanometers). The after-run spectrum shows that the x-ray diffraction line widths had narrowed, which means that grain size had grown and that the nanometer properties had been lost. The powder was not essentially different from bulk Pd metal. This also means that the nanocrystal metal form is a higher energy configuration than bulk metal. As chemists and physicists know, such material seeks to go to lower energy. When 2 nanometal crystals make mutual contact, they transition to a lower energy state by merging together, losing such properties as being able to store hydrogen in anomalously high amounts, and losing the ability to promote cold fusion reactions. This crystal merging process also takes place when a clean nanocrystal Pd makes contact with ordinary Pd metal. The clean nanocrystals grow into the metal surface, and become part of the Pd bulk metal. This phenomenon does not occur when the nanoPd makes contact with stainless steel. The measurements and crystal merging observations are discussed in Supplement 2.

One concludes that the new composites have two important properties. First, they prevent the nanometal crystals from making with each other by surrounding them with inert metal oxide crystal. Second, they provide a highly periodic interface between the nanometal and ionic crystal, an interface that catalyzes the cold fusion reaction. Their fabrication protocol is clearly defined, and matches a

protocol suggested by Imam and Hubler at NRL. Prior to their reading the Yamaura *et al.* paper, Imam and Hubler explained how such material could be made.

The role of ionic solid + nanometal interfaces will be discussed further in the THEORY section. However, it is worth noting that two 2007 material science papers relevant to the interface layer have been published in *Physical Review Letters*. A paper by K.J. Franke *et al.* is titled "Achieving Epitaxy between Incommensurate Materials by Quasicrystalline Interlayers". The paper discusses the *locking into registry* that can occur when different materials make epitaxy contact. This locking into registry can lower system energy if perfect crystalline order by somewhat incommensurate partners does not extend to the actual interface contact. The other paper, by G. Barcaro *et al.* is titled "Epitaxy, Truncations, and Overhangs in Palladium Nanoclusters Adsorbed on MgO (001)". This paper is a solid state physics modeling paper that uses a standard procedure called density functional calculation. It calculates the minimal energy configuration for a number of perfect and imperfect metal clusters in epitaxial contact with a crystal phase of the metal oxide MgO. The metal clusters studied are smaller than 30 atoms, so they are smaller than nominal nanoPd crystals. For many of the imperfect metal clusters, energy is minimized when *locking in registry* occurs. Disorder in the deposited metal and strong ordering in the metal oxide are properties that make the new composites able to promote cold fusion.

Dr. Yamaura expresses his views as to where the anomalously large amount of hydrogen, (or deuterium), absorbed into his oxide-nanometal composite goes. His view is that the extra hydrogen is associated with the nanometal surfaces, and not with the nanometal interior. His views should be taken seriously. His view fits a picture in which most of A-Z's anomalous deuterium atoms occupy vacancy sites and interstitial locations in the somewhat imperfect region of metal adjacent to the actual interface. The metal monolayer that binds to the ionic crystal can be epitaxial (exact fit). As discussed in the THEORY section, the epitaxial interface can be shared with a geometrically ordered, quasiparticle form of deuterium, which is nuclearly reactive.

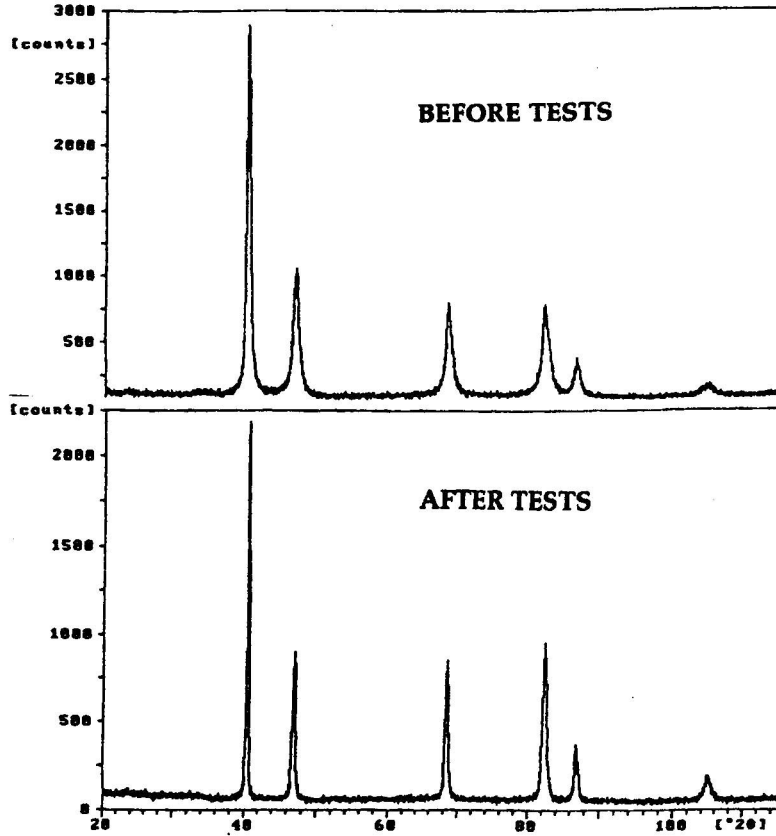


Fig 2.6,1 Bragg reflection spectrum of pre-run and post-run Pd-black used in high pressure studies by G. Schmidt and T. Chubb at the University of New Mexico. Bragg spectra were recorded by Dr. Imam of the US Naval Research Laboratory (NRL). The Pd-black was thoroughly pumped down, chemically reduced, and pumped down again before being pressurized with  $D_2$  gas and tested for fusion heat at elevated temperature. No heat at 1-watt level was observed. Broad spectral lines of purchased Pd-black indicate nanostructure. Post-run spectral show the same narrow lines that characterize bulk Pd, which means the nanocrystals had grown together to form bigger crystals. Study shows importance of **not** including chemical reduction in protocol for preparing Pd-black for use in DS-cathode studies.