Introduction to Materials

The outcome of a LENR experiment depends on several major things, the scientist conducting the experiment, the procedures employed, the equipment for the experiment and the materials within the experiment. All of these things are important. The skill, experience, intuitions and hard work of the experimenter are clearly critical. The protocols used during an experiment, such as the time history of applied voltages, also largely determine the results. The performance, stability and precision of the apparatus greatly influence the outcome. And, materials are absolutely central to any LENR experiment. What happens to the matter and energy put into the system depends on the contents of the experimental system. Adequately characterized materials constitute one of the most critical and most vexing aspects of LENR. It is not agreed if the LENR effects arise only on surface or at boundaries or if they are a bulk property or even what is the appropriate scale of consideration – nuclear, atomic or both.

Whether the LENR experiment is electrochemical, gaseous, plasma or a beam in terms of loading hydrogen isotopes into lattices, the elemental and molecular materials within the central cell can vary widely, and are both difficult to know and control. Knowledge and manipulation of low levels of impurities are particularly difficult.

Beam experiments are conducted in vacuum systems that can be pumped to very low pressures and heated to drive off many impurities. They, along with gas permeation experiments with one side of the Pd or other foil in a vacuum, are generally the cleanest LENR experiments. The incident beams are carefully controlled in direction and energy. Hence, even though they carry much kinetic energy, they usually do not make the experiment “dirty”. Spectroscopic and other tools can be used to perform qualitative and quantitative analysis of impurities *in situ* before, during and after runs.

Plasma experiments are also conducted in vacuum vessels. They are first pumped down, and then filled with low pressures of the gases, which will be ionized to form the plasma. In LENR experiments, the plasmas are commonly a glow discharge but sometimes an arc discharge. Many of the analytical tools applicable to vacuum systems can be used for analysis of plasma loading experiments. Others, like optical spectroscopy, are very useful for monitoring plasmas. However, the ions and photons in and from plasmas go off in most directions. Their impact with nearby solids, including the materials to be loaded with H or D, leads to sputtering and contamination of the experiment.

Gas loading experiments are usually conducted in a strong vessel that can be pressurized and heated. Such vessels can be purged, cleaned and filled with high purity gases prior to experimental runs. The thermal energies in such experiments are relatively low compared to beam and plasma experiments. Here again, many *in situ* diagnostics can be brought to bear. However, some incisive analytical methods require vacuum conditions for their operation, so they are excluded from use in gas experiments when the gas is present.

Electrochemical LENR experiments can be relatively clean if great care is taken to start with clean equipment and high purity chemicals are used. Both light and heavy water with high purity are available. And, the salts that dissolve to form the electrolyte can also be obtained with few impurities. However, the container that forms the cell and any other materials in contact with the electrolyte can provide contamination. The range of diagnostics that can be used within electrochemical cells during their operation is more restricted than in the other types of loading. There is no agreement on what purity is required. For example, the original experiments suggested that materials that were too pure did not produce FPE heat. Subsequent work has found that stress induced by loading hydrogen causes the metal to crack and that low levels of alloying elements can prevent cracking.

The "joker" in all of these types of loading approaches is the solid material(s) put into the heart of the experiments, the materials in or on which the LENR occur. They vary greatly in their composition, that is, the elements present and their spatial distributions, both of which can change with time. And, the structural arrangements of the atoms on nano-, micro- and macro-scales before, during and after an experiment also vary widely. The interactions of the experimental protocols with the composition and structure of the cathodes, other electrodes and probes within experiments are both central to the outcome and poorly known in most cases.

Impurities from any source might either promote or degrade production of excess power. They could be deleterious to the point of poisoning an experiment. Or, they might be beneficial to the extent of enabling power production. These very fundamental possibilities are little explored and essentially not understood at this time.

That features of key materials in LENR experiments are not known adequately should not be surprising. After all, nuclear reactions take place on size scales of femtometers, the metric for nuclear dimensions. This scale is 100,000 times smaller than the scale of atoms. Nanometers are the scale at which proper conditions for the occurrence of LENR probably take place. But, the tools of nanotechnology for determining the type and locations of atoms and molecules are yet to be widely brought to bear on LENR experiments. Part of the reason is lack of funding. But, even if atomic force microscopes and other atomic-scale probes were available for analyses before, during and after runs, there is the problem of their small fields of view. It is likely that LENR do not occur uniformly over the surface of a cathode in, say, an electrochemical experiment. Repeated scanning of a small patch, typically 100 micrometers square or smaller, with an AFM could miss the locations of LENR, either by bad luck or because the probe destroys the critical conditions. The situation could be like the old story of looking for lost keys at night only under the street lights. In short, the composition and structure of cathode materials on the nano-meter scale is probably critical to production of LENR, but very difficult to access experimentally in a meaningful way.

Three papers on materials were given at ICCF-14. They are the result of collaborations between scientists from three laboratories, ENEA in Italy and the Naval Research Laboratory and SRI International in the US. The collaboration of these three laboratories, which reported their current results in an on-going program, represents the most thorough approach to the study of materials in this field.

The first paper on materials was an invited presentation by Violante *et al*, which involved correlations between impurities, metallurgy of Pd foils, loading (D/Pd) and production of excess heat. It was found that contaminants control the crystal orientation during annealing of the foils, and the effects of subsequent chemical etching. The power spectral density (PSD) is a measure of the magnitudes of roughness on the surfaces of the foils. It was measured after etching. Samples that produced excess power had peaks in the PSD in the range of 10^6 to 10^7 m^{-1}. That is, surface roughness in the range of 0.1 to 1 micrometer favored production of excess heat. Further, it was reported that the surface crystallite orientation of <100> also favored power production. The <100> face in Pd is the most open crystallographically, which might help with loading and the occurrence of LENR.

The second materials paper by Sarto *et al* provided details of the characterization of Pd foil surfaces. The data were obtained by use of an Atomic Force Microscope (AFM). This is one of the very few instances when that central tool of nano-science and –technology has been use for materials in LENR experiments. The data obtained by 2-dimensional square scans of the foil surfaces, generally 24 micrometers on a side, was used to compute PSD. The foils were then used in electrochemical calorimetric experiments to test their ability to produce excess heat. The results were stated in the last paragraph above.

The third paper on materials from the tri-laboratory collaboration by Castagna *et al*, gave details of the metallurgical treatment of the Pd foils and the measurements of their surface texture, that is, the orientation of surface grains. Correlations between the ability to produce excess heat and several materials parameters were obtained. They include mean grain size, grain orientation, grain boundaries, hardness, and deuterium loading. This is the work that showed <100> crystallite orientations favored production of power.

Miley and his colleagues presented a materials paper on the evidence for the formation of clusters of deuterons at dislocations within Pd cathodes in heavy-water electrochemical experiments. Such nanometer-scale features are thought to be the locations at which LENR occur. Hence, their increase is desirable. These authors discuss ways in which the density of deuteron clusters can be increased, which they call a “Roadmap to Power Cells”.

Swartz and Verner provided a paper on the spiral cathode (called a Phusor™) used in their electrochemical experiments. The paper makes the case that the spiral structure is a metamaterial. Such a material gets its electromagnetic properties from the structure (shape) of the material, rather than from its chemical composition. The authors report that their cathode shape is the best among many designs and variations for reproducible behavior with good power gains, and that such is the case for several Group VII metals. The electric fields associated with the spiral cathode were computed. The authors focus on the intra-electrode flux of deuterons, which is due to the electrical field distributions that cause asymmetric deuteron entry into the cathode. The authors believe that such deuteron fluxes are necessary to produce LENR.

The papers on materials presented at ICCF-14 illustrate the complexity of the study of materials for LENR experiments. There are many options for acquisition of the base materials, processing prior to experiments and conduct of the experiments. Numerous experimental tools are available for the characterization of the materials at any step in the overall process, and after experiments. Sophisticated and expensive equipments, which generally require skilled scientists for their operation and interpretation of the data obtained, are needed. The work is slow and requires great attention to details. Hence, the study of materials for LENR experiments is costly. Few research efforts in LENR have the funds to carry out these desirable characterizations of materials.