## PROGRESS REPORT #3 INSIGHTS OBTAINED DURING THE INITIAL STUDY

Subject: Part 3. Insights obtained during the initial study Date: 8/21/15 Written by: Edmund Storms, Kiva Labs

## INTRODUCTION

This study involves creation of various alloys, their activation for reaction with deuterium in an electrolyte cell, and measurement of any energy resulting from LENR. The process involves variables important to success, which are described in this series of progress reports. Copies of Reports #1 and #2 can be accessed at <u>www.LENRexplained.com</u>, where subsequent reports will be found.

Progress report #1 describes the construction of the calorimeter being used in this study and the approach used to find nuclear active material. Progress report #2 describes the initial calibration and the expected errors. This report summarizes some problems and solutions discovered during the initial tests.

**1. Calorimeter drift:** The calibration of the calorimeter has been found to have changed, probably because the epoxy used to attach the TEC to the aluminum box has cured and now has a slightly different thermal conductivity. This change resulted in what appeared to be excess power being generated by the samples being studied. A routine test of the calorimeter using an inert platinum cathode revealed this change and the resulting error.

This test also revealed an error caused by the rapid variations in cell voltage caused by bubble formation that is not present when a resistor is used to apply energy to the calorimeter. This error was eliminated by inserting a 10,000 mfd capacitor in the voltage circuit to smooth the variations and by increasing the number of measurements that are averaged. These changes produce agreement between the power applied to the electrolytic cell and power applied to a resistor to within 0.02 watt over the range of applied power (0-34 watts) used in this study.

The resulting calibration values are plotted in Fig. 1, to which a quadratic equation is fit.



FIGURE 1. Summary of data on which the present calibration is based.

**2. Preparation of samples:** The samples are prepared by melting together Pd and Ag using a flame. The initial flame used LP gas and oxygen, which placed significant carbon in the material and caused many blisters to form on the surface after reaction with deuterium. These blisters interfere with making an accurate measurement of thickness. Fig. 2 shows a large blister on a typical sample. Many of the blisters were too small to detect by eye.

In addition, the flame was not hot enough to fully melt the entire sample, leaving an unmelted region where the sample contacted the graphite sheet on which it rested. Consequently, a uniform composition of silver was difficult to achieve.



FIGURE 2. Example of a typical blister on the surface of a sample of Pd+Ag after reaction with  $D_2$ .

A solution to both problems was obtained by using a flame produced by the  $H_2+O_2$  reaction. This flame fully melted the metals without addition of significant carbon.

**3. Activation of samples:** In order for hydrogen to react effectively with Pd, the surface of the material must be clean and activated. Cleaning is frequently reported using Aqua Regia. While this mixture of  $HNO_3$ +HCl cleans effectively, it can introduce chlorine, which is a known poison for the reaction with hydrogen. In this work, the surface is cleaned using fuming nitric acid after rolling the previously melted button to the required thickness.

After cleaning, the sample is heated to 900° in air, held at this temperature for several hours, and then slowly cooled to room temperature after the furnace is turned off. This slow cooling in air allows a thin layer of oxide to form on the surface. This oxide layer is known to activate the surface and allow rapid reaction with hydrogen. In addition, an uneven color, which is normally blue to gray, reveals any non-uniformity in silver content.

4. **Measurement of surface composition using EDX:** All the X-ray lines in an EDX spectrum for Pd nearly overlay those produced by Ag. Consequently, identification of Ag in Pd is subject to a large error and is difficult to determine simply by looking at the spectrum, as can be seen in Fig. 3. The relationship between the amount of Pd and Ag mixed to create the sample and the resulting ratio provided by the EDX software is shown in Fig. 4. This figure can be used to calibrate a measurement of the EDX composition observed during subsequent studies.



FIGURE 3. EDX spectrum of Pd+Ag.



FIGURE 4. Comparison between measured Ag/Pd ratio and the amount of these elements mixed before melting.

A number of errors and problems have been found and solved. This experience will be applied to future studies of the Pd-Ag system, as described in future Progress Reports.