

# Program on Technology Innovation: Assessment of Novel Energy Production Mechanisms in a Nanoscale Metal Lattice

2012 TECHNICAL UPDATE



# Program on Technology Innovation: Assessment of Novel Energy Production Mechanisms in a Nanoscale Metal Lattice

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## Abstract

In 1989, Martin Fleischmann and Stanley Pons made an announcement of energy release from a palladium electrode that was infused with deuterium nuclei. Many technical groups around the world attempted to verify their claims with little or no success. The reproducibility was less than 3%, and the rate of excess energy release was sporadic and unremarkable at levels less than several watts. During the past 20 years, research in this area has continued around the world much in “stealth mode”; however, recent claims by several researchers warranted an independent investigation to experimentally replicate the findings, assess their claims, and evaluate the prospects for commercial viability. In this 2011 Electric Power Research Institute (EPRI) Technology Innovation–sponsored research effort, experiments were undertaken to investigate and attempt to replicate these recent international research claims.

Nanocomposite materials were produced, placed inside a stainless steel Dewar flask, and evacuated at 200°C. Hydrogen gas was added to the Dewar flask, and resistance thermal devices recorded the temperature rise during the exothermic reaction of nickel hydride formation. While several research reports from Europe indicated significant thermal energy output from nanotextured nickel in the presence of hydrogen gas, tests of similar materials conducted under this EPRI research grant produced only milliwatt-scale thermal power releases, and in one experiment, a 21-watt release was observed but not replicated.

While interesting results were observed, the research was not able to yield repeatable experiments, given the scope and budget for this effort. However, continued independent experimental work is recommended in this area. The exact physical mechanisms are still unknown, and a reliable and robust experimental system test is warranted to gain further understanding of the commercial viability of this possibly new energy production mechanism.

### **Keywords**

Condensed matter nuclear science  
Energy production  
Nanoscale metal lattice



## Executive Summary

In 1989, Martin Fleischmann and Stanley Pons made an announcement of energy release from a palladium electrode that was infused with deuterium nuclei [1]. Many technical groups around the world attempted to verify these claims with little or no success. The reproducibility was less than 3%, and the rate of excess energy release was sporadic and less than several watts. From 1989 to 1994, the Electric Power Research Institute (EPRI) sponsored a research program at SRI International under the direction of Michael McKubre [2] that produced several high-quality replications of the Fleischmann-Pons energy-releasing effect and tracked reproducibility to deuterium loading levels.

The field of condensed matter nuclear science needs a simple “watershed” experiment that is easy to replicate and unambiguous in the reaction byproducts. Researchers at Vibronic Energy Technologies Corporation suggested that the reactions may emanate from the unusually large vibrational modes [14, 31] of the deuterium sublattice, and three U.S. patents were issued on this topic in the 1990s [14]. In those documents, it was proposed that partitioning the metal lattice into nanoscale dimensions would increase the amplitude of the deuterium oscillations and amplify the energy output.

Researchers suspect that there is an optimal range of dimensions—between 3–12 nanometers (nm)—where the metal lattice itself undergoes large anharmonic modes that were previously observed only in the dissolved deuterium sublattice. They hypothesize that the anharmonic oscillations of the nano-palladium clusters amplify the oscillations of the deuterium nuclei to a level where there is meaningful overlap of their nuclear wave functions and where unspecified nuclear reactions result. As such, the size of the nanoparticles is the principal driving force for the energy production.

In 2006, Hernando reported anomalously large magnetic fields in 4-nm palladium clusters [15]. These reported fields may be large enough to enable neutron stripping reactions when the nuclei come into close proximity. In August 2008, Professor Yoshiaki Arata [3,4] of Osaka University made an announcement in Washington, DC that thermal energy was released from nanoscale palladium clusters (5–10 nm) infused with deuterium gas. There was no energy input, and a continuous output of thermal energy at the 100-milliwatt power level

was reported and was claimed to have endured for weeks. The thermal energy output exceeded that from any known chemical reaction by a factor of 30 before the experiment was terminated to examine the nuclear residue. Anomalous concentrations of helium-4 gas were reported with high-resolution mass spectrometry.

Arata's work with nickel and hydrogen had many similarities to work reported by Piantelli and Focardi in 1994 [16]. Piantelli and Focardi reported 40 watts of thermal excess power using nickel rods with textured surfaces and hydrogen. Andrea Rossi visited with Piantelli and Focardi in 2007 to form a collaboration and extend the work into using nickel nanopowder. Rossi and Focardi began making claims of kilowatt levels of thermal power beginning in 2009 [5]. In 2010, Defkalion Green Energy Corp also made claims of kilowatt output levels with nickel nanopowder and hydrogen [6].

### **Research Objectives**

Claims of continuous energy production with no energy input that were made by Yoshiaki Arata and Y. Zhang [3, 4] warranted the experimental investigation of the work documented in this report. The goals of this research effort were to technically replicate, via a transparent experimental approach, the experimental energy production claims by Arata and later by Rossi. Specific objectives were to find answers to the following research questions using a two-phase experimental effort:

- Is excess heat observed in a nanoscale lattice of palladium infused with hydrogen gas?
- Is excess heat observed with nano-palladium and deuterium gas?
- Are nanoscale nickel and hydrogen a better choice?
- Are there any preferred alloys of nickel that enhance the heat output?
- Are there any triggering processes to enhance energy or heat output?
- Are there any preferred particle sizes?
- Will high-voltage pulses increase the energy or heat output?

Researchers also set out to try to answer these key research questions:

- What is the physical mechanism behind the heat release?
- What is the best test vehicle for discovering the underlying process?

- What diagnostic tools are important to this understanding?
- What are the preferred triggering mechanisms?

### **Research Results**

The energy production from nanoscale metal powder was attempted to be measured in a series of small-scale experiments using palladium-nickel alloy nanopowder formulations. The general reproducibility of the phenomenon was found to depend on a triggering mechanism. The excess heating rate output did not exceed 200 milliwatts. The triggering mechanism employed was simply raising the temperature above 360°C.

While several research reports from Europe by Piantelli et al. [16] had indicated significant thermal energy output from nanotextured nickel in the presence of hydrogen gas, similar tests conducted under this EPRI research project produced only milliwatt-scale thermal power release. Based on experimental calorimetric calibrations, the amount of thermal power being produced was estimated to be about 100 milliwatts per degree C of elevation above the value of the outer resistance thermal device (RTD).

In one experiment, researchers used 10-nm nickel powder from Quantum Sphere Corp. The inner RTD was 208°C hotter than the outer RTD (533°C versus 325°C) and represents roughly ~ 21 watts from 5 grams of nanopowder, based on the calibration. The powder maintained this rate of thermal power output for a period of five days when it was terminated for evaluation. There was no sign of degradation of the power output. Researchers, however, were not able to replicate this final experiment due to limited project funding.

Radiation accompanying the excess heat was sought with four separate meters. They are

- Gamma Scout for  $\alpha$ ,  $\beta$ , and Gamma
- Ludlum Model 3 Survey Meter x-rays > 10 keV
- Nuclear Research Corp model NG-2 Neutron Gamma survey meter
- BTI Corp Bubble detector for neutrons
- No neutron radiation above background was observed in any of the experiments. Gamma rays and alpha particles were also absent. The lack of signals is not surprising as the powder is held inside a thick walled, stainless steel, high pressure dewar. This dewar had a wall thickness of 5mm so gamma rays below 100keV

and alpha particles below 10MeV would not pass through the thick wall.

- There is a possibility that nuclear processes with emission below these threshold values may account for the observed heat output.

#### Discussion and Conclusions

- Nanopowder in the size range of 5–10 nm (verified by transmission electron microscope) was found to cause small amounts of excess power (in the 100-milliwatt level) for Ni-Pd alloys in the Arata format.
- Three experiments were performed which illustrated that reproducible quantities of (several watts of) excess thermal power production can be obtained with nanotextured nickel and hydrogen. However, these samples needed to be heated above 360°C to observe the excess.
- The uncertainties in the calorimetric measurements are significant because this study was aimed at measuring the temperature progressions reported by Arata. Calibration using alumina powder and a heating coil in the Dewar flask showed that a 1°C rise in temperature was equivalent to 100 milliwatts  $\pm$  30 milliwatts from room temperature calibration.

The underlying mechanisms have not been established in this research effort.

Regardless of the underlying process, the excess thermal power output observed in this study, coupled with reports of European efforts, suggests that a new energy production concept maybe present and warrants further investigation and continued attempts for replication.

#### **Recommendations**

In view of the experimental findings of “watt-scale” thermal power production, continued experimental work is warranted in this area.

The specific recommended work is discussed below:

- The nanopowder without the zirconium oxide composite structures showed promise because the 2-nm oxide coating on the particles appears to be sufficient to prevent agglomeration of the nickel spheres up to at least 535°C. Further work with these materials is recommended.
- Continued tests with multiple thermal cycles are indicated. The triggering effects of thermal cycling have been observed, but are

not yet understood. Piantelli [16], for example, employs multiple thermal cycles over days and weeks to initiate heat release. It is believed that these cycles introduce nanoscale surface features that have an increased activity.

- Such cycling procedures may be required to achieve commercially useful thermal power levels. Cycling up and down with temperature and pressure changes has been seen as a triggering mechanism. Hydrogen embrittlement may contribute to grain-size reductions at the surfaces of nickel and accelerate the rate of heat release. Additionally, supplying electromagnetic pulses of as-yet-undetermined range of frequencies may result in additional triggering mechanisms.

The addition of electromagnetic energy at some as-yet-undetermined resonant frequency is expected to provide insight to the detailed mechanism of the energy production.

- New tests should be conducted to verify that fluctuating magnetic domains are causing the energy release. The system could be expected to respond to external stimuli at these driving frequencies. This is an experimental objective that is reasonably tightly bounded and could have enormous scientific and practical value.
- Future tests using nanopowder pressed into solid billets and applying resonant input are recommended to determine if the energy output is amplified. For example, the titanium-alloy nanopowder billets are especially interesting and are recommended. They are interesting because four consecutive experiments with titanium substituting on the zirconia sublattice showed an endothermic response. However, the mechanism that results in sub-ambient cooling is not understood.

Finally, the last experiment reported in this report, yielding 21watts of excess power (~50x more power than any of the other experimental run) should be replicated by an independent laboratory.



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# Section 1: Experimental Apparatus

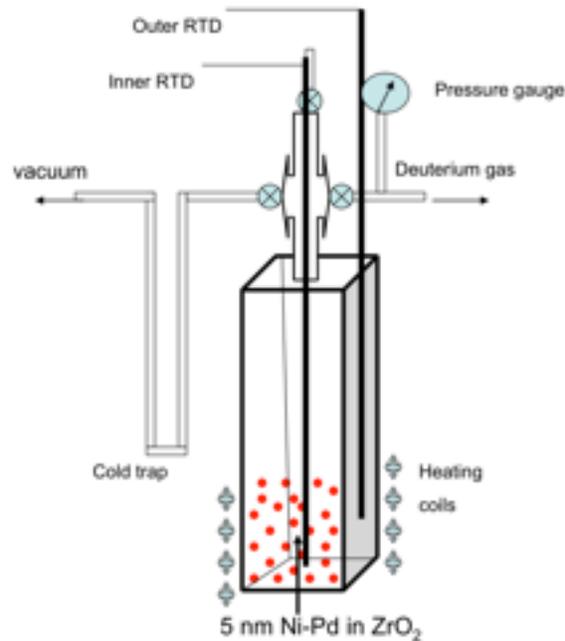


Figure 1-1  
Arata System Schematic

## Research Approach and Scope

The first important step in this project was to replicate the experiment reported by Yoshiaki Arata of Osaka University in Washington DC in August, 2008. The most important aspect of his experiment is that he used 5-10 nanometer palladium powders. This is a very important size regime that is little known to practitioners of nanoscale science. Nanoscale metals in the 3-12 nanometer size regime develop anharmonic oscillation modes in response to a feedback condition known as Energy Localization. Replication of the Arata experiment is straightforward once the proper nanoscale metal processing methods are conducted. A key feature of the effort is to prepare the nanopowders and prevent their degradation by environmental conditions.

Nanopowders are notoriously prone to absorbing contaminants and poisoning their catalytic activity. Therefore, procedures were first developed to handle the materials at every step of their processing and operation.

The powders were prepared by arc melting zirconium with nickel at 1,300°C and then cooling them rapidly in a melt spinning sequence. These procedures were conducted at Ames National Laboratory and they delivered 30 gram samples of shiny ribbons. The alloy ribbons were subsequently baked in air at 440°C for 28 hours to convert zirconium into zirconium oxide. The zirconium oxide isolated nickel into 10 nanometer nickel islands as observed in TEM micrographs as shown in Figure 1-3.

The brittle ribbons were finally ball milled in a black powder with an average grain size of 25 microns. Each micron-scale grain contained about 1 million nickel nano-islands.

Figure 1-1 is a schematic diagram of the Arata system employed in the EPRI study. A stainless steel dewar rated for 100 atmospheres with 50ml volume was connected to a vacuum system and a high pressure deuterium gas source. The components were connected by VCR fittings to limit leakage of the deuterium gas.

Back streaming of oil from the vacuum system was prevented by a dry ice cold trap. A 5-micron filter was placed in the vacuum line to prevent nanopowder entrainment into the valves. Heating tape was wrapped around the dewar to allow for vacuum bake out of the nanopowder and subsequent thermal triggering of the deuterated powder.

Three terminal RTDs with a temperature sensitivity of 0.03°C were positioned inside the powder and outside the dewar. The RTD output is recorded every 30 seconds. Figure 1-2 illustrates a set of sample traces. The top curve is short term data representing 90 minutes of data logging. The red trace is the RTD inside the powder showing an excess of 0.5°C over the RTD mounted externally (blue trace).

The lower set of traces is long term data with each vertical line representing 2 hours. Clearly, the red trace remains 0.5 degrees above the RTD mounted external to the dewar (blue trace). This data shows that the hydrated nanopowder remains above room temperature indefinitely after absorbing hydrogen gas.

During hydrogen gas addition the red trace goes off scale as the powder heats up to over 50°C. The blue trace only rises by several degrees as it is outside of the dewar. This graph is representative of the thermal history of the nanopowdered samples with power output levels never exceeding 200 milliwatts.

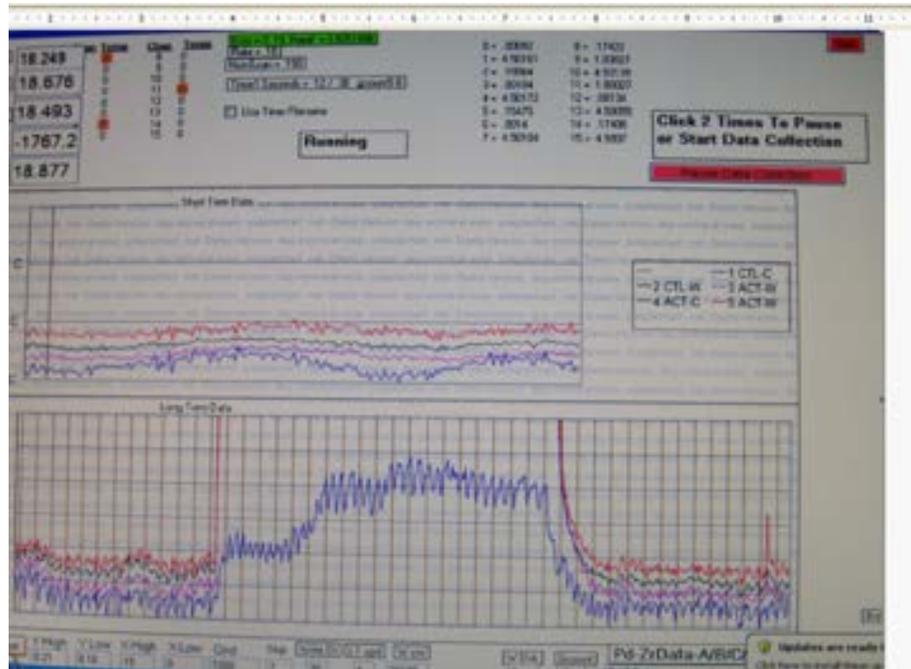


Figure 1-2  
RTD Traces for Sample

A TEM Image of a Pd<sub>35</sub>Zr<sub>65</sub> sample made by melt-spinning

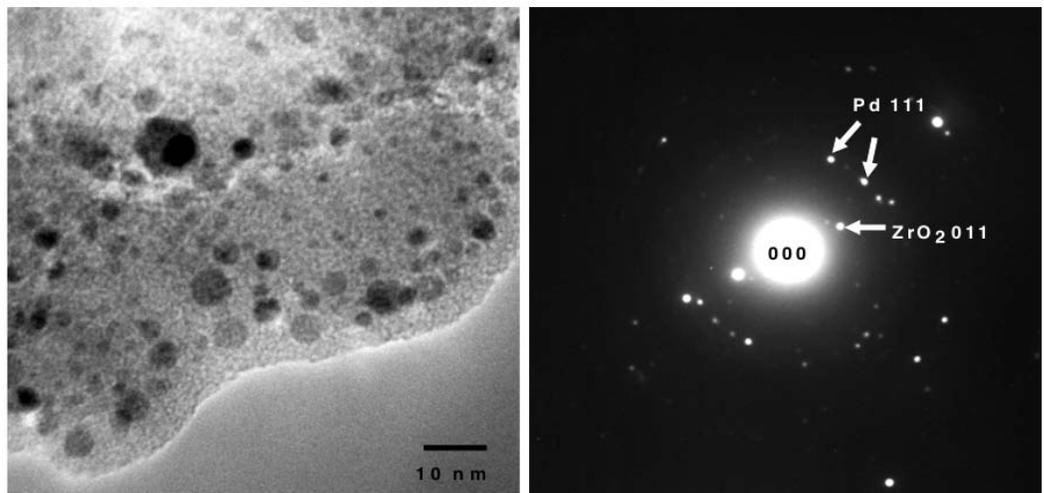


Figure 1-3  
TEM Micrograph- Ni islands in ZrO<sub>2</sub>



## Section 2: Material Processing

The details of the nanopowder processing are central to achieving a specific size regime. The researchers hypothesize that processing the powders into a specific size regime between 3–12 nanometers is a minimum condition for observing excess energy production. This theory for the size of the particles was shared with the Japanese group who reported a null result in 2008. [1, 3 ,4]

Takahashi and Kitamura revised their processing to achieve dimensions below 12 nanometers and subsequently reported excess energy production. [17]

There are various methods for achieving the stated size regime. A straightforward method that was employed in this EPRI work was developed by Yamaura and Inuo[18,19] beginning in 1992. The technique consists of arc melting an alloy of zirconium and various compositions of nickel and palladium. The arc melted alloys are melt spun by passing the hot liquid alloy between two water-cooled copper wheels rotating with an angular velocity of 25 meters/sec. The molten alloys cool from 1,350°C at a rate exceeding 1,000°C/second, resulting in a continuous ribbon alloy with the local composition similar to the liquid state. The metallic ribbon is shown in Figure 2-1.



*Figure 2-1  
Metallic Ribbon*

The nickel-palladium-zirconium alloy ribbons are then baked in air at 440°C for 28 hours. This process converts the zirconium metal into zirconia ( $ZrO_2$ ) that isolates and encapsulates the remaining metal into nanoscale islands. Without the interleaving zirconia the metal islands would coalesce into macroscopic dimension where the excess energy production is predicted to be quenched.

The metal ribbons are conducting and non-ferromagnetic before baking. After baking the brittle ribbons are semi-insulating and ferromagnetic. These two properties confirm the segregation of zirconium away from the nickel. Nickel metal is ferromagnetic, but nickel zirconium alloys are not ferromagnetic.

The dielectric properties of the zirconia may provide more than physical and electrical isolation. Lawandy [20] has argued that the dielectric layers at the metal surface can accumulate deuterium ions at densities much higher than solid deuterium in its metallic form (solid deuterium). The increased density has been reported by several other authors [21, 22, 23] recently and has been described as Rydberg Matter.

The baked ribbons are subsequently ground in a ball mill to an average particle size of approximately 15-50 microns. They become ferromagnetic as the nickel metal atoms segregate into nickel islands. The ribbons become brittle and can be ground in a mortar and pestle or ground in a tumble mill using steel ball bearings. The particle size for the powder ground for six days in the tumbler was 15 microns (Figure 2-2) as compared to 75 microns (Figure 2-3) for the mortar and pestle processing.

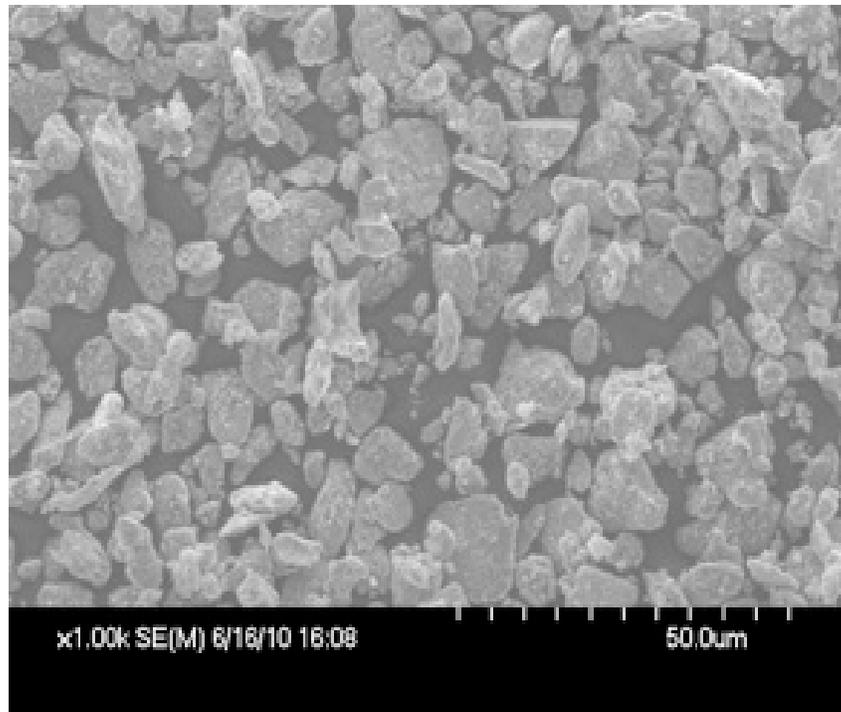


Figure 2-2  
Particle Size 15 microns

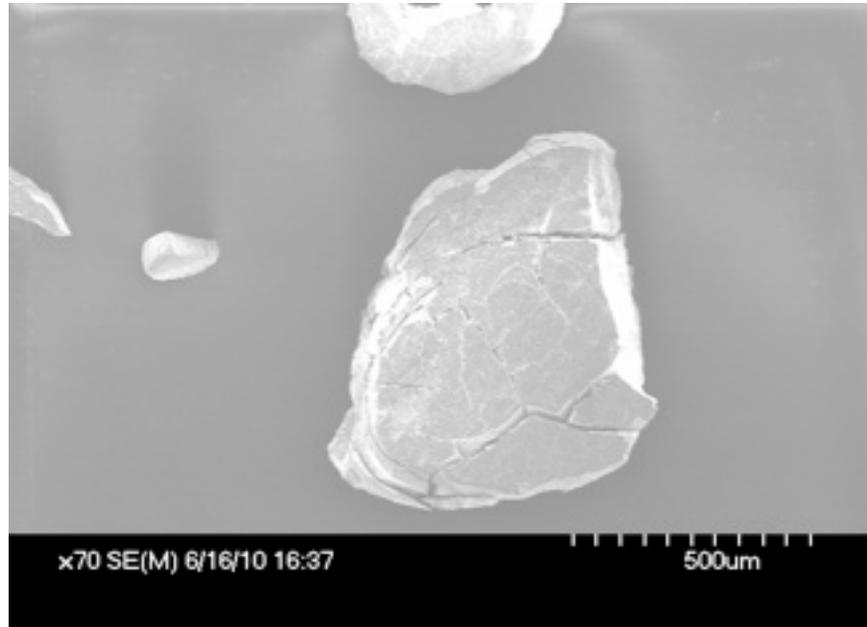
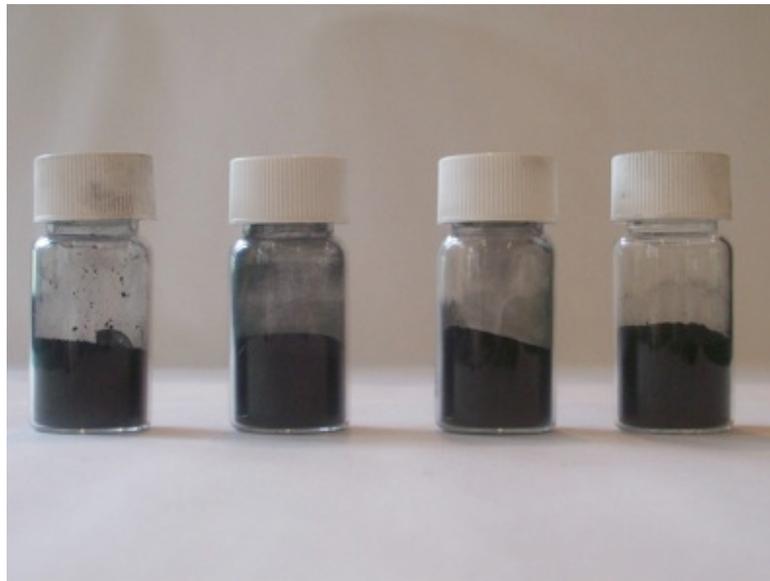


Figure 2-3  
Particle Size 75 microns



## Section 3: Experimental Procedures

The ground powder samples were typically 25-35 grams (Figure 3-1) and filled the dewar to a depth of 3cm. The powders are vacuum baked for 24 hours at 200°C to remove residual impurities and water vapor.



*Figure 3-1  
Ground Powder 15-50 microns*

The powders are placed inside a stainless steel dewar as shown in Figure 1-1. The system is evacuated and baked to remove trace water vapor (repeated sentences). The high purity hydrogen gas is added and the temperature rise is measured with sensitive RTDs to an accuracy of 0.03°C.

In the experimental set up, deuterium and/or hydrogen gas is added to the powder in batches that result in a temperature rise from the heat released by the exothermic process of deuteride/hydride production and the anticipated reaction energies were observed. The deuterium/hydrogen to metal ratio, D/H/M, is calculated by the number of batch filling operations at a given pressure and volume. The exothermic trace for deuterium filling operation is shown in Figure 3-2. Loading the H<sub>2</sub> or D<sub>2</sub> gas was accomplished by flowing gas in up to a pressure of 200psi and then watching to see the rate of pressure drop as the nanoparticles fill with hydrogen isotopes. The number of filling operations

combined with the internal volume allows for a calculation of the number of moles of H<sub>2</sub> gas loaded into the metal alloys.

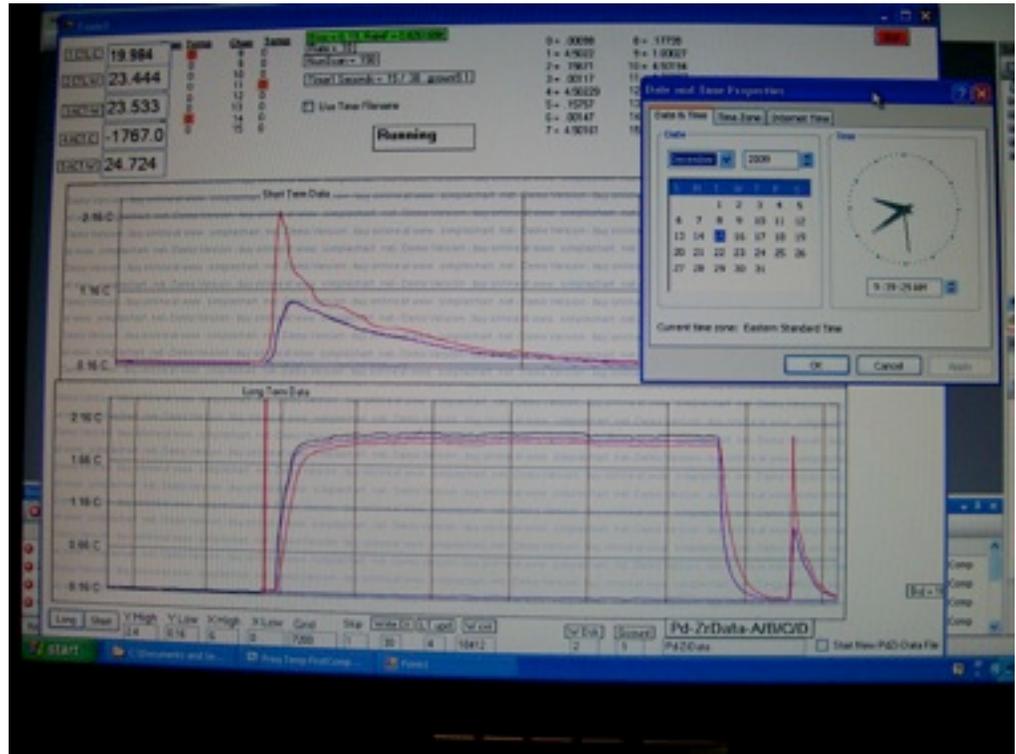


Figure 3-2  
Exothermic trace for deuterium filling operation. The upper red trace is the interior RTD short time data

The D/M or H/M ratios observed were much higher for the nickel-palladium alloys than for pure palladium or pure nickel samples. The D/M ratios for all of the samples can be found in Table 3-1.

Table 3-1  
Loading Rates and Temperature Results

Sample Date	Composition	Bake Schedule	Loading Ratio d/M	" T	Comments
4/22/2009	Ni30%-Pd5%	600C 24 hours	<4	0.0C	Overbaked ribbons
4/17/2009	Ni30%-Pd5%	450C 24 hours	??	0.6C 10 hours	Good sample rebake
4/22/2009	repeat Apr 17	regrind	1.86	1.0C 22 hours	Regrinding & reheating
5/1/2009 - 5/15/2009	raney nickel	300C 24 hours	<0.1	0.0C	No excess heat
5/31/2009	Pd 35%	325C 24 hours	1.84	0.0C	Overbaked ribbons
6/5/2009	Pd 35%	280C 30 hours	2.6	0.3C 16 hours	Fine grain powder
8/11/2009	Ni 30% - Pd5%	500C 36 hours	1.25	0.4C 4 days	Good sample
8/17/2009	Ni 30% - Pd5%	525C 24 hours	1.7	0.5C 1.5 days	Good sample
9/10/2009	Ni 30% - Pd5%	>600C 20 hours	<0.4	0.0C	Inadvertent baking
10/22/2009	Ni 30% - Pd4%	450C 48 hours	3.8	0.3C 8 days	Stopped sampling
11/9/2009	Ni 30% - Pd5%	430C 70 hours	0.4	0.0C	Overbaked ribbons
12/3/2009	Ni 30% - Pd4%	425C 24 hours	4.2	0.0C	Ideal sample
12/15/2009 - 01/12/2010	Ni 30% - Pd5%	400C 40 hours	3.7	0.5C 28 days	Re-heated
5/16/2010	Zr66Ni30Pd4%	430C for 28 hours	2.4	0.5C	explosion
3/19/2010	Zr67Ni29Pd4%	430C for 28 hours	3.7	0.4C 4 days	triggered
4/27/2010	Zr67Ni29Pd4%	420C for 28 hours	3.7	0.5C	triggered
5/11/2010	Zr66Ni30Pd4%	430C for 28 hours	4.8	0.3C	triggered
6/22/2010	Zr65Pd35%	280C for 24 hours	0.9	2.2C for 22 hours	triggered
7/1/2010	Zr65Ni35%	430C for 28 hours	<0.2	0.0C	no absorption
8/4/2010	Zr65Ni35%	450C for 48 hours	<0.2	0.0C	No H absorption
9/17/2010	Zr66Ni30Pd4%	440C for 28 hours	6.5	1.2C	Deuterium absorption high
11/12/2010	Zr67Ni32Pd1%	440C for 48 hours	leaks prevent measure	0.2C	Leaking fitting
11/26/2010	Zr65Ni35%	440C for 28 hours	<0.1	0.0C	no loading without Pd
12/8/2010	Zr67Ni32.5Pd1.5%		<0.1	0.0C	No Loading at .5% Pd
12/17/2010	raney nickel	no baking	0.2	0.0C	
12/19/2010	Zr67Ni32Pd1%	440C for 96 hours	2.6	1.7C	good loading at 1%Pd
1/19/2011	Zr67Ni31Pd2%440C for 48hours		3.4	0.3C	good loading at 1%Pd
2/15/2011	Zr42Ti15Ni30Pd3%	440C for 28 hours	Negative 1.0C endothermic		
3/10/2011	Ti67Ni30Pd3%	475 for 48 hours	H/M 2.0	neg 0.2 endothermic	
3/31/2011	Zr52Ti15Ni30Pd3%	475C for 96 hours	H/M <0.5	Negative 1.0C endothermic	
5/12/2011	Zr66Ni21Cu13%	445C for 24 hours	<0.5 0.0	0	exotherms above 360C
6/23/2011	Zr65Ni25Cu5Fe5%	440C for 28 hours	2.5	1.1C	Exotherms at high temp
*7/8/2011	Zr65Ni25Cu5Co5%	440C for 28 hours	<0.5	0.9C	exotherms at high temp

High loading ratios with H/M > 1.0 were anticipated from data generated by Arata in 2003 [1, 10]. The deuterium loading phase diagram is shown in Figure 3-3. The peak loading composition in the vicinity of Zr65%Ni30%Pd5% was the focus of this replication study, because higher deuterium densities were believed to correspond to higher nuclear reaction rates. That was the working hypothesis, but it was not supported by the test data. Excess energy was found in samples with low loading rates. The relationship between loading and excess heat does not appear to hold for nickel nanopowders. The high loading may be due to absorption by the zirconium metal that is not fully oxidized but this was not verified in any experiments.

This high loading should be considered as 'apparent loading'. The apparently high loading rates may be attributed to Spillover hydrogen. Spillover hydrogen refers to a process where the surrounding zirconium oxide may absorb the hydrogen atoms that are produced by bond cleavage of the H<sub>2</sub> molecules by the PdNi islands.

However the high power output from the final sample from QSI argues against any heat coming from the oxide sublattice. The loading for the QSI sample was only a few percent of the composite powders, yet it showed a hundred-fold increase in output power.

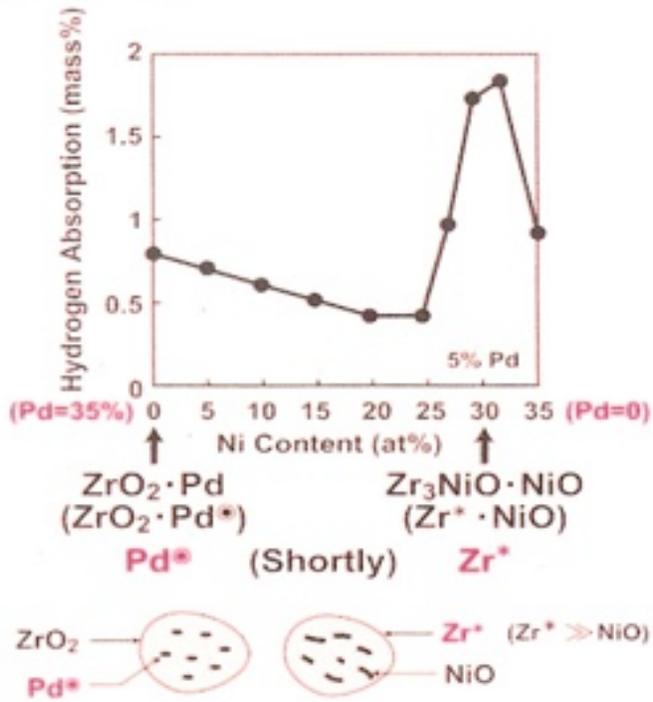


Fig. 3 Relation between sample powders of  $ZrO_2 \cdot Pd^*$  and  $Zr^* \cdot NiO$

Note: These sample are Oxidized powders of amorphous  $Zr_{63}Pd_{33}$  and  $Zr_{63}Pd_{33}Ni_{36}$ , respectively.  
Deuterium atoms are absorbed into only  $Pd^* / Zr^*$  ( $Pd^*$ : nano-Pd,  $Zr^*$ : bulk- $Zr_3NiO$ )

Figure 3-3  
Pd-Ni Deuterium loading phase diagram

After the exothermic reactions are completed the system cools down over a period of about 5 hours. In the first half of Phase I, 50% of the samples showed an excess energy trace that endured well beyond the 5 hours normally observed for a return to thermal equilibrium. At the end of this first phase of experiments a process of thermal triggering was proposed by EPRI's peer review committee. In subsequent tests, the hydrated powders were heated to 440°C at hydrogen over pressure of 15 atmospheres. The nanopowders cooling from this condition resulted in measurement of excess energy production in six consecutive samples. Sample A-12 showed a  $\Delta T$  value of 0.5°C for 38 days.

Calibration of the heat release corresponding to the continuous measurement of excess temperature of 0.5°C was found to equal 18 eV/metal atom. This Arata system output power is low but estimation of the integrated energy (over 38 days) is over four times as much energy as any known chemical bond.

This finding supports the theory that the energy production cannot arise from any known chemical process. However, there is no evidence from this study to support the nuclear origin view as no radiation was measured above background with four detectors operating.

The last powder sample in table one is Zr65%Ni35%. It was processed in the same manner as the Ni-Pd alloys, but the deuterium fill ratio was very low at 0.2 D/M. This low fill rate showed no excess energy even after thermal triggering. This loading level was low and did not provide excess energy.

The highest  $\Delta T$  observed was 2.2°C in Sample B-7. This is a higher excess energy level than reported in any other known Arata replication report to date. This finding is interpreted by CMNS supporters as evidence of energy output that is scientifically interesting, but the passive energy release is not commercially useful or practical.

### **Discussion of Phase I Tests**

Nano powders intentionally processed into in the size range of 5-10 nanometers were found to cause small amounts of excess energy for Ni-Pd alloys in the Arata format. The measurements were found to depend on a thermal triggering mechanism with over pressures of deuterium gas. The uncertainties in the calorimetric measurements are significant as this study was aimed at measuring the temperature progressions reported by Arata. The calibration using alumina powders with an overpressure of one atmosphere of hydrogen gas and a heating coil in the dewar showed that a one degree Celsius rise in temperature was equivalent to 100 milliwatts  $\pm$  30 milliwatts. This study, however, was not designed for rigorous Calorimetry Measurements, so the error bars are at least on the order of 30%.

High voltage applied across the Arata composite materials result in sporadic breakdown processes. The resistance of the sample is over a billion Ohms. However, once the dielectric strength of the composite is exceeded the resistance drops abruptly during dielectric breakdown. This occurred around 600 volts DC for an electrode separation of 6mm.

The metal nanoislands embedded in the zirconia dielectric focused the discharge energy into the 5nm particles and acted as short circuit pathways. High voltage discharges through liquid dielectrics with embedded nanopowders was first discovered at NIST in 1994 [24]. The focusing aspect of such discharges has never been reported outside of the NIST effort. These discharges were hypothesized to amplify the output from the Arata nanopowders. Unfortunately, the power supply was not robust enough to allow for studying the response of the nanopowders to pulsed energy input.





## Section 4: Phase II Testing

Continued experimental testing was conducted in a phase II effort with the objective of conducting a repeatable experiment to verify and quantify excess energy production mechanisms.

The objectives of Phase II were to replicate the procedures from Phase I to examine alloys that have catalytic potential and to find new triggering mechanisms to amplify the energy output. The early excess energy production from nickel alloys suspended in a zirconium oxide matrix occurred at a time when several European groups were reporting much larger excess energy output. Their alloys and triggering mechanisms have not been identified.

In 2010 and 2011 Rossi, Defkalion Green Technologies Corp and Piantelli [5, 6, 16] were all reporting kilowatt levels of energy production involving nanodimensioned nickel infused with hydrogen gas. These reports prompted the research team to search for a triggering mechanism that could increase the energy output in the nickel hydride system. The addition of heating the system was first examined in Phase I. The simple addition of heating the system above 360 degrees C increased the energy output from 200 milliwatts to 5 watts at the end of Phase I.

Adding catalytic metals to the nickel was next examined. Strontium, copper, iron, cobalt and titanium were added at various concentrations. None of these alloys showed significant improvement in energy output. In fact, four consecutive titanium alloy samples showed a net absorption of energy. The hydrated titanium alloy samples all showed a net reduction in temperature that was 1°C below ambient. This result suggests a new mechanism unrelated to nuclear processes. The calibration of the RTDs has a sensitivity of + 0.03 degrees C, so the effect appears to be real, but unexplained.

Another approach to energy amplification was the application of high voltage pulses directly through the composite powders. This was a complex task since the powders had very high impedance at low voltage and low impedance at high voltage. The current – voltage response was highly nonlinear and caused the power supply to fail repeatedly. The power supply was fabricated by Ivan Kruglak of Boulder Colorado and is shown in Figure 4-1.



Figure 4-1  
*Pulsed Power supply*

### **High voltage pulsed power supply**

The high voltage dewar with the electrically isolated center electrode is shown in Figure 4-2. The value of triggering with electromagnetic pulses was not established in this study, but it is strongly believed to be a fruitful avenue to pursue in future work.



Figure 4-2  
High voltage dewar with heating tape

### **Nanocomposite powders versus nickel nanopowders**

The release of small amounts of excess energy was measured in over a dozen consecutive experiments in Phase I. In Phase II the first experiment with 10 nanometer nickel powders showed no excess energy. That sample was part of a strontium alloy run where the nickel and strontium metal were brought to a temperature of 830°C to melt the strontium (m.p. 777°C) over a period of eight hours.

This sample was exposed to high pressure hydrogen gas and no excess energy was observed. Upon opening the dewar it was visually observed that the nanopowders had sintered into a dense solid block with macroscopic grain dimensions. No excess energy was found with the sintered material. This result coincides with the hypothesis that 5-10nm particles are a prerequisite for excess energy production.

In a final experiment researchers used 10nm nickel powders from Quantum Sphere Corp that were vacuum baked at low temperature (150°C) to remove water vapor. The powders were exposed to high pressure hydrogen gas at room temperature. The exothermic reaction of hydrogen absorption was minimal. The hydrogen pressure was reduced to sub atmospheric conditions.

A heating tape wrapped around the dewar was energized to heat the nanopowders. The outer RTD is mounted just inside the heating tape and its temperature is generally about 20 degrees C hotter than the inner RTD. When the temperature reached about 280°C the inner RTD showed a discontinuity and it became significantly hotter than the outer RTD. This heat flow reversal indicates energy being produced by the hydrated powder itself. There was a second discontinuity at 450°C.

From earlier calibrations the amount of thermal power being produced was estimated to be about 100 milliwatts per degree C of elevation above the value of the outer RTD. In the last experiment the inner RTD was 208°C hotter than the outer RTD (533°C versus 325°C) as shown in Figure 4-3. That represents roughly 21 watts from 5 grams of nanopowder. This value is based upon the calibration from Phase I. This is roughly equivalent to four kilowatts per kilogram. This result is approaching the level reported by the three independent European efforts [5, 6, 16]. The powder maintained this rate of thermal power output for a period of five days when it was terminated for evaluation. There was no sign of degradation of the energy output. Unfortunately the test data for this run was lost and this test run is recommended to be duplicated in any follow-on effort.

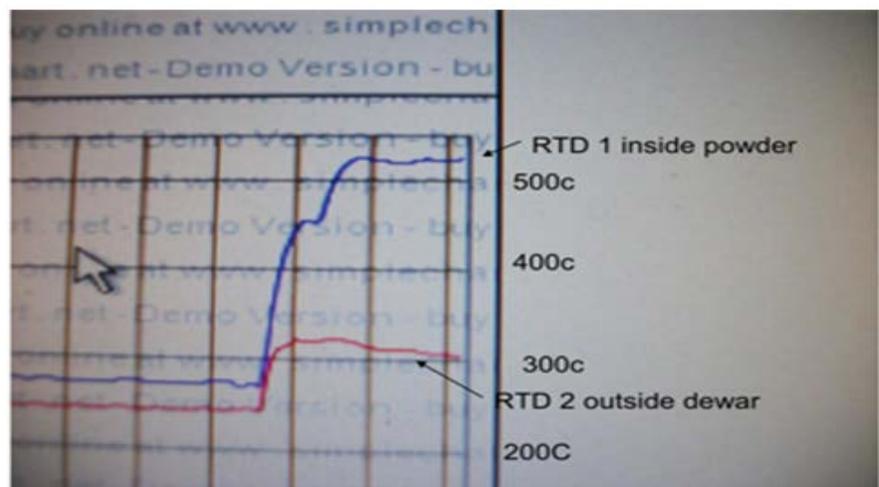


Figure 4-3  
QSI RTD Trace from final experiment

The Inner RTD is over 200° hotter than the RTD inside the heating tape on the outside of the dewar. Each vertical line is one hour. There was a discontinuity at about 280°C and 450°C where the powder began self heating.

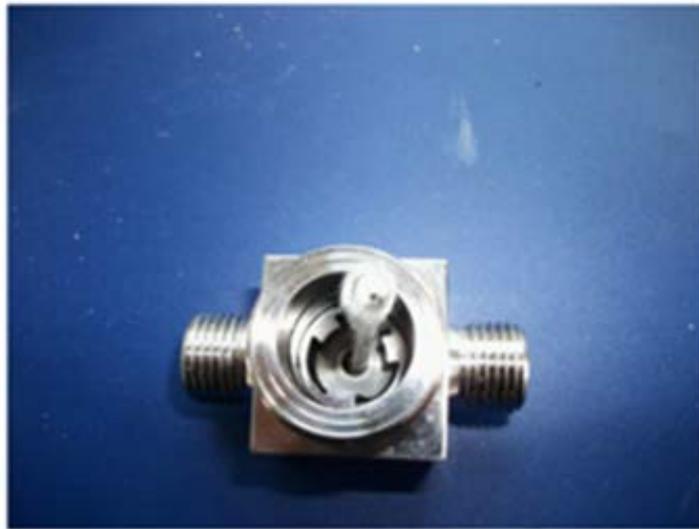
## Experimental Discussion

### ***Fundamental problems with nanopowders and vacuum systems***

Nanopowders were composites materials with an average grain size of 30–50 microns. They were easily trapped with 2 micron in-line filters. However, the 10nm powders obtained from Quantum Sphere Corp, Santa Ana, CA were challenging to work within the experimental apparatus. With 0.5 micron in-line filters (Swagelok brand) and very slow evacuation rates, the nanopowders were drawn into the evacuation path and deposited on the valve seats.

This made the system impossible to hold H<sub>2</sub> gas pressure as the hydrogen would pass through the openings in the valves littered with nanoparticles. An over pressure of 200 psi of H<sub>2</sub> gas was reduced to ambient pressure in less than 15 minutes. The valves were cleaned and hermetic before they were placed in with the nanopowders, but the extremely low settling rate of such small particles demands that they move along the streamlines of the evacuating gases. The nature of the vacuum leaks is internal. That is, the hydrogen gas leaks into the vacuum system rather than escaping through openings in any of the VCR fittings.

The black residue on the bellows from the valve is shown in Figure 4-4. This was clean and hermetic until it was placed in line with the nanopowders.



*Figure 4-4  
Black residue observed on the bellows from the valve*

Valve body with a Q-tip used to wipe the valve seats. Nanopowders blacken the Q-tip and prevent sealing.

This sealing problem did not exist with the composite nanopowders and there are no such complaints coming from Defkalion or Piantelli. It is believed that their macroscopic metals have nanoscale surface features. Andrea Rossi has stated that his mean particle diameter is on the order of one micron. [5, 3] Defkalion [6] reports their mean powder diameter is 4 microns.

However, those particles may have surface features in the nanoscale range when properly processed.

## **Conclusions for Phase II Tests**

Three experiments were performed illustrating modest quantities (several watts) of excess energy. These experiments operated above 360<sup>o</sup> C with nanograined nickel and hydrogen. The experimental set up was limited by leaks in the valves due to nanopowders slipping by the metal valve seats. Replacing the metal valve seats with soft Teflon was recently found to solve this problem. Other research efforts by Piantelli and Focardi [12, 16] have also reported to have produced much higher thermal power levels. The underlying mechanisms, however, have not been established in this research effort. Experimental findings suggest that that these measured thermal effects have an origin exceeding any known chemical processes, but a specific nuclear process has not been established at this time.

Irrespective of the underlying process, the excess thermal power output found in this study coupled with reports of European efforts strongly suggests that a new energy production concept is present. The data from Figure 6-3 indicating a thermal power equivalent to 4 kilowatts per kilogram is potentially exciting. The fact that it is a gas phase system operating at 525<sup>o</sup> C makes it a potential candidate for high pressure steam turbine applications as an eventual potential application.

## **Recommendations**

This experimental work should continue and the last experiment conducted in this project should be replicated by an independent lab such as SRI International.

The nanopowders without the zirconium oxide composite structures showed promise, because the 2 nanometer oxide coating on the particles appears to be sufficient to prevent agglomeration of the nickel spheres up to at least 535<sup>o</sup> C. As the oxide layers are removed by hydrogen at elevated temperature the nanopowders were expected to experience significant grain growth to a point where they would become inactive. The five day test at 22 watts of output power has allayed these fears to some extent.

Piantelli reports improved thermal power production with multiple cycles in temperature and H<sub>2</sub> gas pressure. This may be required to achieve commercially useful thermal power levels [5 ,6, 16].

The nanopowdered nickel from Quantum Sphere Corp worked well, but this material is currently expensive at \$18/gram or \$500/ounce. It is expected that this cost will be greatly reduced if the heat output is increased. The cost of the nickel is not expected to be a limiting factor.

More rigorous testing and measurements should be performed in future work to support measurements of the claims of excess heat measured.

The addition of electromagnetic energy at some as yet undetermined range of frequencies is expected to provide insight to the detailed mechanism of the energy production. The nickel nanopowders are magnetic and they may represent a new form of super-ferromagnetism as detailed by Van Waeyenberge[13] in a 2006 article in Nature. Separately, Liu et al [25] report localized magnetic features in a barium ferrite sample. They found that magnetic vortices arise in magnetic materials when the grain size is around 10 nanometers. Future experiments should address these mechanisms.

Bellaiche [26] has reported that these magnetic vortex structures interact when external electromagnetic fields are applied. The interactions of the vortices release large amplitude electrical pulses. These magnetic vortices or magnetic storms can interact with external fields in novel ways.

A series of recent articles by Sundaresen [27] suggest that all metal oxides in the 10 nanometer range are ferromagnetic. This would serve to explain how palladium can give off heat even when its macroscopic form is not ferromagnetic. This research opinion is shared by the observations of Chaulesu [28] where NiPd alloys remained ferromagnetic when the fraction of nickel was reduced to low concentrations.

Titanium alloy nanopowdered billets are especially interesting and should be investigated.

The research work by Dr. Mark Snoswell from Adelaide Australia [30] should be examined and replicated in future work if possible as he has reported over 100 watts of continuous and long lasting power output from the same nano-nickel powders ordered from Quantum Sphere Corp.

EPRI support for this research effort was valuable in establishing the relationship between particle dimensions and excess heat production. Future work will examine means for amplifying the heat output.



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