

ULTRASONIC FUEL TREATMENT AS A GATEWAY TO NICKEL/HYDROGEN LENR

Hank Mills and Alan Smith (www.lookingforheat.com) OCTOBER 2016

"There are very few human beings who receive the truth, complete and staggering, by instant illumination. Most of them acquire it fragment by fragment, on a small scale, by successive developments, cellularly, like a laborious mosaic. Anais Nin"

Abstract: We describe a method of preparing nickel powder for use in 'dogbone' or 'Model T' nickel-lithium-hydrogen reactors. This method, while not new, has seemingly been overlooked by many LENR researchers and replicators. The use of ultrasonic cleaning of nickel as a slurry in hexane, magnetic separation of pure nickel from oxide fragment and subsequent measures to limit the reformation of oxides and promote the absorption of hydrogen are described in detail. Links to published research papers on sonication of nickel powders and the subsequent stimulating effect on catalytic activity are appended. Replicators should be aware that this is a time-consuming process demanding care, thought, and a fair degree of technical skill in setting up and handling the required equipment.

Purpose: To induce nuclear based exothermic reactions in an LENR experiment by utilizing meticulous cleaning methodology, including the use of ultrasound and other processes such as vacuum Ddegassing under heat to enhance the catalytic properties and hydrogen absorbing potential of nickel.

Hypothesis: Using the methods described below enhances removal of the stubborn oxide layer from nickel powder and reduces particle size. Such an effect has been widely discussed in the literature of catalysis. This cleaning process improves hydrogen absorption, thus facilitating low energy nuclear reactions inside the reactor.

Desired Outcomes: Improved success rates of anomalous heat production, possibly as good as those achieved by other replicators, hopefully comparable to the most successful tests of the Rossi Effect performed by third party replicators including "heat after death" (persistent heat production with zero input.)

Materials and Equipment

1) We chose 'Gem Grade' 20uM Nickel Powder from Archer UK. This was chosen for its low declared level of impurities, and because the stated particle size is in our opinion a good starting point for those using this method. We hope our choice reduces the chance that contaminants or impurities remain in the Nickel powder that are "catalyst poisons."

2) Alfa Aesar 97% LiAlH₄ -- Chosen for reasonably high purity and small particle size of

around 10 microns vs. the much larger particle size for other brands (e.g. Sigma Aldrich 50 -150 microns). This small size helps to improve surface area and thus maximize hydrogen release at the lowest temperatures possible.

3) Looking for Heat "Model-T" Unit. We actually used 2, all heaters coupled in series and with all ports calibrated to achieve equal temperatures with empty 'control' tubes.

4) Packard 7525 High Purity Hydrogen Generator. (99.999% pure H₂ @ 300ml minute/max delivery pressure 6 Bar). Other types are available.

5) Ultrasonic Generator (28khz & 100w). Ex Ebay

6) Anhydrous Hexane (Assay: >99% Molecular Formula: CH₃(CH₂)₄CH₃:Molecular Weight: 86.18:Boiling Point: 69 deg C supplied by APC Pure Ltd UK.

7) Vacuum Pump. Single stage oil-filled - theoretically good down to 0.5Pa

9) Glove Box -- Argon Filled.

10) Fume hood (Optional but very desirable.)

Protocols

0- HIGH TEMP ANNEALING - Pre-heating in ordinary atmosphere to 700C for one hour. The growth of nickel oxide due to this process may alter surface features. Additionally, the high temperature annealing of nickel films has been reported to alter the internal texture of grains to allow for fast hydrogen absorption and transport.

1 - IRRADIATION TO REMOVE OXIDES - In argon filled glove box, place nickel powder (5 grams each) and hexane (21milliliters each) into two glass vacuum flasks. Stir briefly to create slurries. Attach ultrasonic generators and electrical leads. Seal each port before removing from argon filled glove box. Place flasks into ice bath to prevent heating during sonification. In a manner that allows zero atmosphere to enter flasks, attach vacuum line(s). Perform ultrasound irradiation of slurries for five hours while maintaining temperature below a maximum of 25C to prevent unwanted bulk chemical reactions between nickel powder and hexane.

2 - EXTRACTION -

Remove vacuum lines from flasks containing slurries in a manner that allows zero exposure to atmosphere and re-seal. Transfer flasks to argon filled glove box. Use strong permanent magnets to extract ferromagnetic nickel from sonicated slurries, leaving broken up nickel oxide fragments behind. Wash extracted nickel twice with hexane utilizing

manual agitation and magnetic extraction.

3 - CLEANING EQUIPMENT BEFORE USE - Clean next flask to be used. Apply high temperature while passing a stream of hydrogen through it to de-oxygenate the interior surfaces as much as possible. After 30 minutes at 400C, stop the flow of hydrogen, fill the interior with argon, then allow to cool to room temperature, adding a little more argon from time to time as it cools to avoid creating a vacuum inside.

4 - HEATING AND BAKING UNDER VACUUM - Inside of argon filled glove box, transfer nickel to secondary vacuum flask and seal openings. Remove from glove box and attach vacuum line in a manner that allows zero atmosphere to enter. Switch on vacuum pump and turn on electrical resistor. To avoid sintering of oxide free nickel, heat only to 250C. Continue with heating for six hours with vacuum pump turned on for only the first thirty minutes of every hour to prevent burn out.

5 - HYDROGENATION - In a manner that allows zero atmosphere to enter, break vacuum and apply hydrogen from generator to approximately two bar or the highest pressure that flask can contain without seals being damaged. Turn on resistor and slowly bring the temperature up to 250C over a one hour time period. Keep hydrogen generator applying pressure continually if possible or intermittently to compensate for pressure reduction by absorbed hydrogen. With at least intermittent application of positive pressure to eliminate the possibility of oxygen entering flask, continue process for several hours or longer. Please note this process may also reduce any residual oxides on the nickel to at least some extent. For hydrogen reduction of nickel oxide, temperatures of 625C and higher are preferred. However, due to the nickel being in the form of powder and already clean of oxides - which promotes sintering -- a lower temperature must be utilized to avoid sintering and reduction of catalytic activity. Clean nickel has been reported to sinter at temperatures of 310C according to Bob Higgins of the MFMP. The temperature of 250C has been chosen in the hope that no sintering whatsoever will take place.

6 - COLLECTION - Allow flask to cool and remove hydrogen line in a manner that allows zero exposures to atmosphere. Place flask into argon glove box and remove nickel powder. If powder appears to have clumped or sintered to any degree, manually agitate to separate. Place finished powder into appropriate argon filled bag/bottle within another argon filled container.

ACTIVE RUN

1 - In argon glove box, fill three reactors – cleaned with hydrogen to remove oxides – with a gram of nickel and LiAlH₄ fuel mixture. Each reactor will contain a different content of LiAlH₄ which shall be 5%, 10%, and 20%. Fill control reactor with gem nickel that has not been hydrogenated or sonicated. If we use sonicated and hydrogenated gem nickel, we may see excess heat due to previous hydrogenation.

2 - Seal the reactors and take them out of the glove box. For a short period of time, minutes, apply vacuum to each reactor (if doing so won't suck out fuel) to remove argon. After vacuuming each reactor fill with hydrogen to atmospheric pressure and seal.

4 - Slowly begin heating the nickel through 200C at a VERY slow pace below 1/K per minute to 200C to allow the transition from LiAlH₄ to Li₃AlH₆ to take place without the nickel melting. This should allow for the nickel to not be wetted and for hydrogen absorption to be maximized. After 200C, heat up to 700C at a pace below 5C per minute as suggested by contacts.

5 - At 700C or a little higher, drop temperature to 500C and rapidly increase to 700C to commence triggering. If no excess heat compared to control is triggered, increase temperature slowly to 900C, lower temperature to 500C, and quickly bring temperature up to 900C. If no excess heat produced, slowly increase temperature to 1200C or maximum capable of being achieved in the Model T. Drop temperature to 900C, and push temperature to highest possible.

EXCESS HEAT PROTOCOLS

(This protocol written with the understanding that all active reactors are in series and input power cannot be individually adjusted.)

1 - If one or more reactors are producing excess heat compared to control before first triggering, continue with heat ramp up for all reactors.

2 - If one or more of the reactors begins robustly and powerfully surging in temperature beyond first triggering point of 700C, keep power to all reactors constant for a period of time to monitor and observe excess heat phenomenon. If the reactor producing high levels

of excess heat is not already at 1350C, continue with heat ramp up for all reactors at a very slow pace and proceed with additional triggering attempts. If reactor producing excess heat exceeds a temperature of 1350C, stop heat ramp up for all reactors.

3 - When triggering has been attempted for all reactors at specified temperatures or a single reactor has reached 1,350C, perform "heat after death" test by cutting off power.

4 - If no reactor begins to self sustain, do not allow the temperature of any reactor to fall below 200C.

5 - If a reactor does begin to self sustain, do not cut power back on even if other reactors fall to room temperature. Continue monitoring self sustaining reactor until its temperature begins to drop significantly.

6 - If no reactor has self sustained or the self sustaining activity of a reactor has ended, slowly ramp up the temperature of all reactors and repeat triggering efforts.

7 - Repeat above procedures.

SAFETY PROTOCOLS

1 - Place blast shield around Model T units during operation.

2 - Place blast shield around flasks during all phases when filled with pressurized hydrogen.

3 - Wear safety goggles, lab coat, gloves, mask.

4 - Keep a dry-powder fire extinguisher handy.

5 - Have radiation sensors present.

Day Two Tests

1 - If significant excess heat produced on day one, attempt replication by putting the exact same ratio of LiAlH_4 and nickel into three more reactors and repeating the day one experiment.

2 - If no significant excess heat produced on day one, attempt to change variables to produce excess heat.

a - Reactor one should utilize ultrasonically irradiated nickel and a higher percentage of

LiAlH₄: 70% nickel and 30% Alfa Aesar LiAlH₄.

b - Reactor two should be loaded with a different brand of LiAlH₄ that is not from Alfa Aesar: 90% nickel and 10% LiAlH₄.

c - Reactor three should utilize LiAlH₄ that has been ball milled at high intensity to reduce particle size and improve hydrogen desorption characteristics. 90% nickel and 10% LiAlH₄.

DISCUSSION POINTS

1 – The ultrasonic irradiation process may do more than clean the surface of nickel and copper of oxides. A thin layer of carbon has been found on the surface of both ultrasonically irradiated nickel and copper. It is speculated that the carbon may come from the carbon in the solvent being thermochemically cracked when two particles in the solvent collide at high speed producing temperatures of thousands of degrees at the contact point. Interestingly, one production method of graphene is to ultrasonically irradiate carbon that has been deposited onto a nickel surface. There is speculation that broken, imperfect sheets of graphene and similar arrangements of carbon atoms may be produced on the surface of ultrasonically irradiated nickel. The extremely high levels of catalytic activity after ultrasonic irradiation may be too high to be explained by only the removal of the oxide layer.

2 – Acids are also often used to remove oxide layers from nickel powder. One example of a commonly used chemical is hydrochloric acid. However, the chlorine in HCL points the catalytic ability of nickel to adsorb hydrogen – the first process in hydrogen absorption. This is because chlorine is an electronegative element. For this reason and the fact we do not wish to contaminate the fuel with a multitude of different chemical elements, we are forgoing the use of chemical dissolution of the oxide layer.

3 – Potassium is an alleged catalyst used by Rossi in the past. It is an electropositive element that increases the catalytic properties of nickel to adsorb hydrogen. Additionally, it was used in an experiment described by Thermacore to produce excess heat.

4 – Rossi's original Italian patent application indicated that copper could be mixed with nickel or could be used by itself. Copper is a known “spillover” catalyst that can increase the catalytic properties of nickel. When exposed to ultrasonic irradiation, copper's surface is modified and cleaned in a similar manner to nickel.

REFERENCES ON NICKEL POWDER CATALYST IMPROVEMENT TECHNIQUES

1. **ABSTRACT:** Treatment of nickel powder slurry in decane with 20 kilohertz high intensity ultra sound at 50 watts per cc. of slurry at a temperature of 293K. Starting with a particle size of 160um, sonication resulted in a particle size of approximately 80um, reduction in the thickness of the surface oxide layer and a massive increase in the catalytic properties of nickel. Improved catalyst performance (to similar levels as Raney nickel) was primarily due to the decrease in oxide level

Papers by the Suslick Research Group, University of Illinois.

(1A) <http://www.scs.illinois.edu/suslick/sonochemistry.html>

Heterogeneous Sonocatalysis with Nickel Powder. Kenneth S. Suslick* and Dominick J.Casadonte

(1B) <http://scs.illinois.edu/suslick/documents/jacs873459.pdf>

Sonocatalysis. Kenneth S. Suslick and Sara E. Skrabalak

(1C) <http://www.scs.illinois.edu/suslick/documents/sonocatalysis.pdf>

(1D) <http://www.scs.illinois.edu/suslick/documents/science901439.pdf>

The Effects of Ultrasound on Nickel and Copper Powders: Suslick, Casadonte And Doktycz. School of Chemical Sciences, University of Illinois.

(1E) <http://www.scs.illinois.edu/suslick/documents/solidstateionics.1989.pdf>

The Chemical Effects of Ultra Sound

(1F) <http://www.scs.illinois.edu/suslick/documents/sciamer8980.pdf>

(1G) <http://www.scs.illinois.edu/suslick/documents/ultrasonsonochem.2014.1908.pdf>

Ultrasonic Irradiation Of Copper Powder

ABSTRACT: Interparticle collisions still occur and surface morphology and chemical reactivity can be affected, but particle agglomeration will not occur, for example, as with 160um diameter Ni. In sufficiently viscous liquids the velocity of inter-particle collisions will probably be diminished. However we observed similar inter-particle collision in various synthetically useful liquids including several alkanes (n-octane through n-tetradecane), dimethylformade, and dioxane. Further work is in progress.

(1H) <http://www.scs.illinois.edu/suslick/documents/chemmater.1989.pdf>

PARTICLE MORPHOLOGY STUDIES.

2. **ABSTRACT:** Electron micro-photographs taken 'before and after' sonication.

The Effect Of Ultrasound Irradiation On The Morphology Of Ni Powder Synthesized By Electrodeposition Authors: Mahdiyar HOOBAKHT , Alireza ZAKERI, Alireza SHAHIDI

(2A) <http://www.ultrasonic.co.ir/files/Ni%20powder.pdf>

Larger Image of Smoothed Nickel Particles

(2B) <http://chempedia.info/pics/4121/>

Chemical Modification of Chemisorptive and Catalytic Properties of Nickel

(Chlorine, a common contaminant in LiAlH_4 , poisons the ability of nickel to adsorb hydrogen due to being electronegative. Other electro-negative elements also poison the nickel. Potassium, found in Rossi's early fuel, enhances adsorption due to being electropositive. Lithium is not discussed in this paper, but since it is electropositive, it may help hydrogen adsorption. NOTE: Adsorption (attachment to surfaces) is the first process which takes place before hydrogen absorption can happen.

Chemical Modification of Chemisorptive and Catalytic Properties of Nickel

http://www.chem.tamu.edu/rgroup/goodman/pdf%20files/40_app%20ss-19-84-1.pdf

1) https://books.google.com/books?id=osog-S8Sw1wC&pg=PA33&lpg=PA33&dq=nickel+slurry+in+hexane&source=bl&ots=UY8TsK9vwq&sig=idZ8HKMq96XCa8fjZomlVn_EWzw&hl=en&sa=X&ved=0ahUKEwj1md_Fy7HPAhXClx4KHWp0AJUQ6AEISzAI#v=onepage&q=nickel%20slurry%20in%20hexane&f=false

Nickel vapors were allowed to react with three different solvents (hexane, toluene, and THF) to give very reactive metal slurries having high surface areas. Furthermore, these metal-solvent complexes can react with triethylphosphite to afford nickel clusters. Interestingly, metal slurries can be deposited on supports such as alumina, silica, molecular sieves, or activated carbon. Additionally the metal particles formed from metal slurries on warming can serve as active catalysts. Nickel-hexane powders are more active hydrogenation catalysts than Raney nickel. In contrast, Ni-THF powder is a poor and unreacted hydrogenation catalyst, but it is efficient for alkene disproportionation.

https://books.google.com/books?id=Lz8yWgNdL7QC&pg=PA227&lpg=PA227&dq=nickel+hexane+powder&source=bl&ots=lwAEgswJly&sig=r9CW_CB3f9jTKAxMijcKhYstFu4&hl=en&sa=X&ved=0ahUKEwi9vOLL0bHPAhVHPR4KHcbPCbcQ6AEIPDAG#v=onepage&q=nickel%20hexane%20powder&f=false

New catalysts by mixing metal vapour with solvents

A new method, using solvents, for making tiny metal particles has been invented by scientists working at the University of North Dakota. They may have created a new class of industrial catalysts.

K.J. Klabunde, H. F. Efner, T. O. Murdock and R. Ropple have recently been experimenting with the technique of vacuum deposition. In this technique materials are vaporised, then deposited onto a cold finger usually a quartz probe kept at negative 196C. They began a project where a metal and a solvent were deposited at the same time. When they codeposited nickel with the solvent hexane, toluene, and tetrahydrofuran, they managed to produce nickel powders with extremely interesting properties (Journal of the American Chemical Society, vol 98, p1021).

Nickel powders of a tiny particle size were produced in a series of stages. Briefly, when the metal atoms and solvent molecules are codeposited, a weak complex forms between them. Warming slightly allows the metal to dissolve, especially if the solvent is in large excess (more than 30 parts to 1). The solutions produced contain metal atoms. Warming again causes the metal atoms to coalesce to form particles, their size and shape dependent on the solvent used. Excess solvent can be evaporated to leave a finely-divided metal powder. When hexane is used as the solvent, a black complex forms. This ultimately gives a powder which is an extremely active hydrogenation catalyst for compounds like benzene and norbornene. It has proved to be more active than Raney nickel which is presently used widely in the chemical industry. The yellow nickel-tetrahydrofuran complex forms a very finely divided powder comprised of very finely divided powder comprised tiny spheres with particle sizes of 0.5 to 1.5µm, which also compares favorably to Raney nickel.

<https://books.google.com/books?id=HL07onDn-RgC&pg=PA255&lpg=PA255&dq=nickel+hexane+powder&source=bl&ots=3rRTjYqGsv&sig=kYWp9pghfDvJUeYznf4XUZst51Y&hl=en&sa=X&ved=0ahUKEwi9vOLL0bHPAhVHpR4KHcbPCbcQ6AEIRjAJ#v=onepage&q=nickel%20hexane%20powder&f=false>

