Celani-Type Ni + H₂ Replication Jeff C. Morriss jeff.c.morriss@gmail.com

Experimental procedure

Approximately 8 feet of 28 ga Ni wire was wound around a 3/32" steel rod and then removed. The tightly wound wire was then stretched to a length of approximately 3 feet, resulting in an expanded helical form that was wound around a 20 mm OD quartz tube as shown below. Each end of the Ni wire was then secured to electrodes that connected to a Conflat feedthrough. The assembly shown below fits into a quartz tube that is sealed on each end by machined aluminum end caps. Pressure and vacuum seal are maintained by radially loaded O-rings and vacuum grease.



Figure 1: Oxidized Ni Wire Wound Around Quartz Tube

<u>Oxidization of the Ni wire in air</u>. A voltage of ~65 volts was applied to the electrodes yielding a power dissipation of ~300 watts. The Ni wire was heated to a bright orange color, and heating was maintained for approx. 2 minutes.

The Ni wire and tube assembly were placed in a 32 mm ID x 1 mm wall thickness quartz tube that is sealed on both ends as previously described. The aluminum end caps are held together by three threaded SS rods. Electrical power and gas/vacuum enter the cell through 1.33" Conflat feedthroughs on each end.

<u>Determining temperature of the Ni wire</u>. An indirect method, utilizing nickel's relatively high resistivity temperature coefficient of .006, was used to determine temperature. An initial measurement of the wire's resistance at room temperature was made using a 4-wire technique. In situ temperature measurements were then made by noting voltage and current applied to the Ni wire, where voltage was measured at the thermal boundary of the heater wires entering the calorimeter chamber. Voltage measurements were made using a Keithley 2000 multimeter.

<u>Vacuum Bakeout</u>: Simultaneous vacuum and heating to ~300C were applied to the cell to remove air, water vapor and other volatiles. Heat was furnished by Joule heating of the Ni wire. Pumping was continued until a pressure of <10 mT was attained. This process required approx. 90 minutes. At the end of this step power to the Ni wire was removed and the vacuum pump powered off. A series of Swagelok VCR valves controls entry of H₂ and application of vacuum.

All valves may be closed, resulting in a closed system, and this is the configuration maintained once radiation monitoring commences.

<u>Oxide Reduction</u>: NiO reduction dynamics is highly temperature dependent, and elevated temperatures provide the most complete reduction. For temperatures >800 °C reduction can complete in a matter of seconds, Additionally, the surface morphology of rapidly reduced Ni displays rich nanometric features which may enhance LENR activity.¹

Per the preceding reference, NiO reduction was carried out at approx. 800 °C. Initial attempts to reach that temperature with 1 atm of H_2 pressure failed due to the high thermal conductivity of H_2 and limitations on the power supply. This problem was overcome by reducing the H_2 pressure to 5 m Torr. It was then possible to heat the wire to a bright orange color for a time of 1 minute. The Ni wire surface, which had previously been turned dark grey by the oxidation step, now appeared to be silvery and shiny.

<u>Cell Evacuation</u>: Power was removed and vacuum was again applied to remove any water vapor that had formed due to the NiO reduction process. Pumping was maintained until a pressure <10 mT was reached. After reaching the desired vacuum level Joule heating was turned off.

<u>Hydrogen Loading</u>: An initial H₂ pressure of 5 Torr was introduced with the cell at room temperature. Almost immediately the pressure began to drop, indicating that the Ni was absorbing H₂. The cell was maintained at room temperature for approx. 30 minutes, at which time the H₂ pressure had stabilized after dropping ~10% from its initial value. The cell was then powered at 10.0V, 15.0V and 20.0V, corresponding to Ni wire temperatures of 312, 398, and 498 °C, respectively. Each of the three temperatures was maintained for 30 minutes. At the end of the last heating cycle power to the Ni wire was turned off.

<u>Radiation Measurement</u>: Radiation was measured using a Ludlum Model 3 survey meter connected to a 44-9 detector head. Several sheets of aluminum foil were wrapped around the detector head to shield it from heat generated by the cell. Connection to a DAC module required opening the unit and soldering a pair of wires across the meter movement.

Radiation measurements were made with the cell in the insulated airflow calorimeter chamber with the lid partially open to accommodate the cable to the detector head. No excess power measurements were attempted at this time. Constant airflow through the calorimeter guaranteed that neither the cell nor the detector head overheated. The active surface of the detector head was situated at a 45 degree angle approximately 3 inches from the outside of the quartz tube. A better orientation was not possible due to mechanical interference between the inside walls of the calorimeter and the detector head. The GM detector was set to the slow filtering position which corresponds to a time constant of ~5 seconds. Data was captured by the DAQ module at 10 sec intervals, with no averaging. Radiation was monitored before, during and after the H₂ loading procedure.

<u>Results</u>: For this experiment the only continuously recorded parameter was the voltage across the meter movement on the GM detector, where a full scale reading of 2.0V corresponds to 0.2 mrem/hr. At the start of this experiment the apparatus was in a cold state with no power having been applied for at least 12 hours. Radiation level at the start was approximately 0.03

¹ Manukyan, et al. J Phys Chem C, 119 16131-16138

mrem/hour measured next to the cell. This value is the same as measured 20+ feet away from the apparatus.

When the cell was evacuated with 20V across the Ni wire no increase over background was detected. Introducing ~7 Torr of H₂ into the likewise cold quartz cell likewise produced no radiation above background. When the Ni wire was heated to 10, 15, 20 V levels the radiation level started to increase significantly, reaching a max of 0.15 mrem/hr. Power was then removed while we continued to monitor the radiation level. Interestingly, the level did not immediately drop, even when temperature effects were taken into account. As soon as power was turned off the detector probe was temporarily removed from the vicinity of the cell to ascertain whether the probe had been neutron activated. It had not. When the probe was repositioned in a slightly different position a higher radiation level was observed. After several hours the level did drop, exhibiting the typical exponential decay profile with a time constant of ~63 minutes.

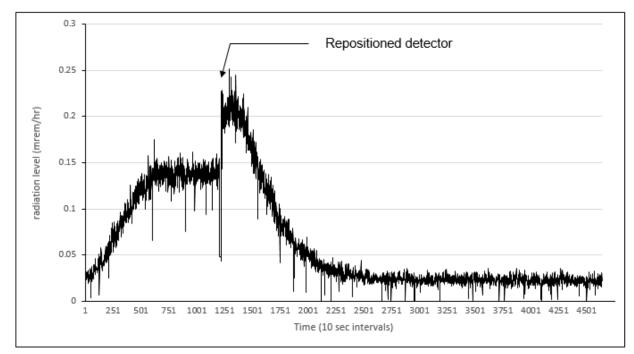


Figure 2: Geiger Counter Radiation Measurement

<u>Discussion:</u> There can be no doubt that radiation of some sort was generated when the Ni wire was Joule heated and H₂ loaded. A radiation signature similar to that shown above was observed on two consecutive experimental runs with the same piece of Ni wire. With a ~7:1 ratio over background, it is extremely unlikely that some statistical artifact occurred, especially as the elevated radiation levels changed only slowly and remained far above background levels for many hours. Furthermore, on a previous run, the detector head was held near the cell only intermittently (to avoid heating it) and similar levels of radiation were detected.

After the apparatus was powered down it was possible to probe near the different components comprising the sealed cell and attempt to locate hot spots. Interestingly, the aluminum alloy end pieces showed the highest levels of radiation even though they remained at relatively low temperatures throughout the experiment. It is therefore possible that one of the constituents of the 2024 Al alloy was activated, and that would imply that neutrons were present. To the

author's best knowledge only neutrons are capable of transmuting a non-radioactive isotope into a radioactive one.

The author is uncertain of the pathway by which neutrons could be produced by a Ni/H interaction, although the quartz tube had been used previously in experiments that included Li. Since the present instrumentation does not permit quantifying gamma energies the only data available is the radiation level decay time. A quick perusal of the CRC handbook table of isotopes did not reveal any obvious candidates with a ~63 minute half-life, although the decay profile may be the composite of decay paths of multiple isotopes.

When the detector was removed from the vicinity of the apparatus and the Al foil removed from the detector head the radiation level dropped to near background, which indicates that no material in the detector head was activated. Replacing the aluminum foil led to slight increase in radiation level, so aluminum or an alloying element in the foil is possibly a source of gamma emissions due to neutron activation.

Next Steps

The observed levels of radiation should be sufficiently large to permit more accurate characterization of energies once improved equipment becomes available. A CZT or Nal detector plus an MCA would permit characterization of gamma energies. Better yet would be a cooled Si or Ge detector. A thermalizing type or a direct energy detecting neutron detector would also be of great use, if for no other reason than safety. With the energy signatures of the radiation quantified it will be possible to determine the isotopic origins thereof and potentially the pathways by which LENR activity occurs.