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DEPARTMENT OF THE NAVY

OFFICE OF NAVAL RESEARCH
875 NORTH RANDOLPH STREET
SUITE 1425
ARLINGTON, VA 22203-1995

IN REPLY REFER TO:

5720
Ser BD042/043
ONR FOIA 16-041
March 8, 2016

Mr. John Greenwald



Mr. Greenwald:

This is the final response to your Freedom of Information Act (FOIA) request received by the Office of Naval Research (ONR) on February 26, 2016. We assigned your request ONR FOIA number 16-041. You requested a copy of the October 17, 2003 Final Report entitled, "Anomalous Energy Transfer: Continuing Studies" by Tanzella, Francis, McKubre, and Michael. For your reference, I have attached a copy of your request.

Parts of the records you requested have been excluded under 5 U.S.C. § 552 (b)(6), better known as Exemption 6 of the FOIA. Exemption 6 protects information about individuals in "personnel and medical files and similar files" when the disclosure of such information "would constitute a clearly unwarranted invasion of personal privacy." Per our review standards, we are withholding one name under Exemption 6. We have attached a copy of the responsive records with the above mentioned redaction. Since you indicated that you are willing to accept clearly releasable information, we do not consider this exclusion to be a denial or partial denial of your request.

No fees have been assessed for the processing of this request. If you have questions about this letter, please feel free to contact Ms. April Harrid at (703) 696-4309 or ONRFOIA@navy.mil. Please reference ONR FOIA 16-041 when communicating with us about this case.

Sincerely,

Kathy Thompson
FOIA Officer

Attachments:
As stated

Final Report – October 17, 2003

ANAMALOUS ENERGY TRANSFER: CONTINUING STUDIES

CONTRACT N00014-02-C-0465

Prepared for:

(b) (6)
Office of Naval Research

Prepared by:

Francis Tanzella
Michael McKubre
Materials Research Laboratory

~~Oct 2003~~
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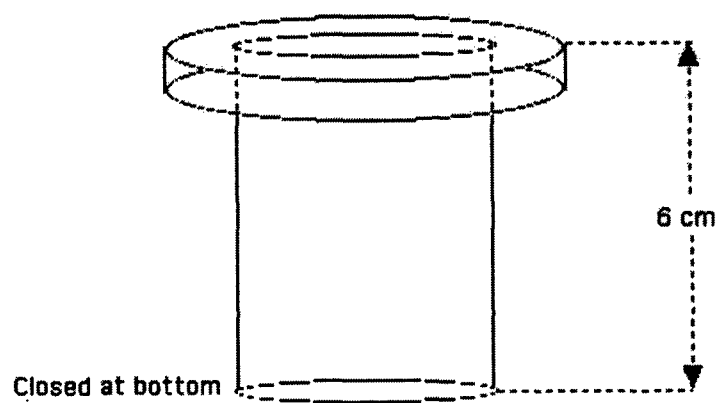
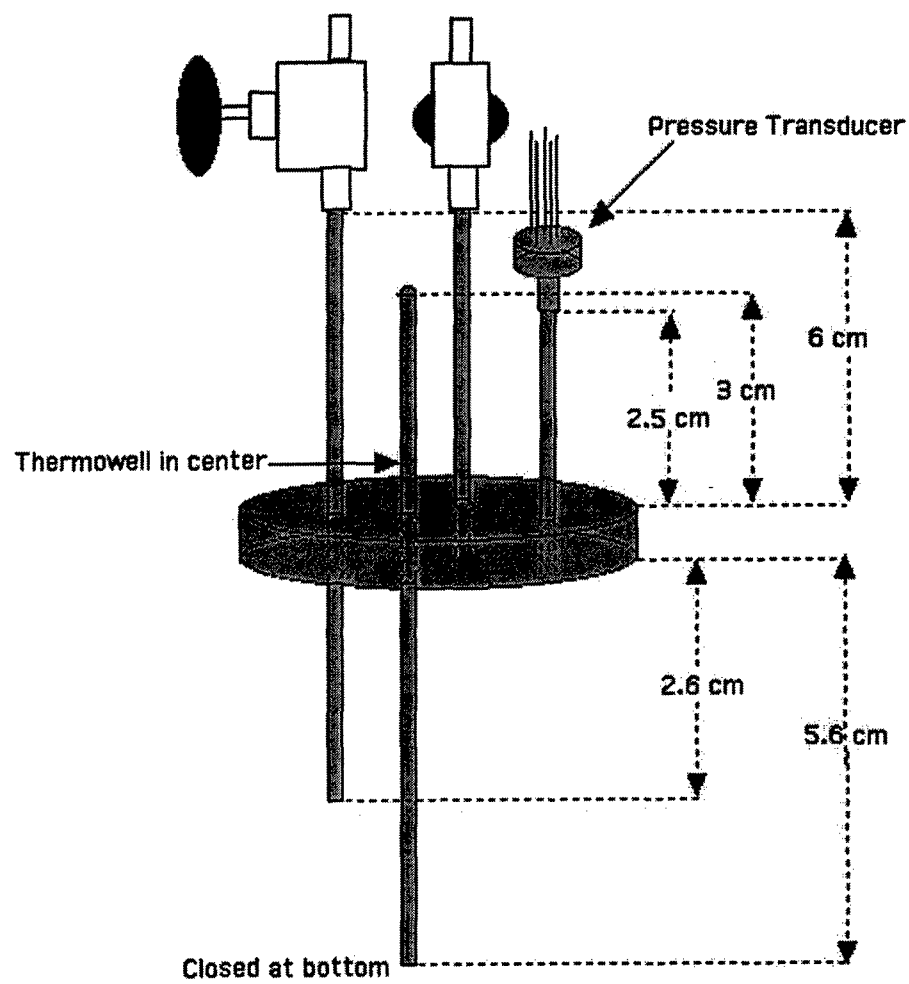
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Task 1: Gas Phase Measurements.

Twelve Case catalyst experiments were run during this period. As the major emphasis of these studies was on the correlation of ^3He concentration with ^4He concentration, no calorimetry is reported. Precise modeling of the thermal losses of these cells under operation has been carried out. This modeling will facilitate future correlation of excess power with helium generation. The results of ^3He and ^4He measurements for these twelve experiments are presented.

We performed twelve case catalyst experiments in what we referred to as the Case matrix vessels, consisting of 4 cells at a time in one heater block. Figure 1 shows the design of the cells. Gas is withdrawn either for pumping or sampling through the tube ending in the middle of the gas headspace. Gas is introduced through the tube terminating at the top of the vessel. All permanent seals are welded, and the two vessel halves, based on ConFlat[®] high vacuum seals, are closed using a copper gasket. We have shown all connections to be helium leak tight.



A small manifold is connected to each vessel for the introduction and sampling of gas. A 1/8" diameter stainless steel tubing, connected to a liquid nitrogen trapped rotary pump, is used to evacuate the vessel. A capillary tubing is connected to the inlet of the mass spectrometer's sample preparation chamber.

The first stage of the sample preparation system consists of 4.0 g of saturated charcoal in a steel U-tube immersed in liquid nitrogen, comprising a carbon cryotrap. A valve separates the cryotrap from the one-liter expansion chamber with an ion gauge. This chamber can be isolated from a 150 l/min turbo molecular pump. A getter (SAES # 700) is used to getter any non-noble gases that transit the cryotrap. The exit of the expansion chamber is connected to the inlet of the mass spectrometer via a flexible stainless steel bellows line. The Operating Procedure for the spectrometer and a ^3He spectrum are shown in Appendix A.

The four cells run simultaneously in the first group were designated SC19 through SC22. Each was charged with 10.0 g of G-75E 0.5% Pd on coconut charcoal catalysts (United Catalyst, Lexington KY).

Each cell was evacuated and filled with 1.5 atm D_2 (Isotec 99.99%, 100% Isotopic) and heated to $\sim 100^\circ\text{C}$. After 210 minutes the vessels were topped up to ~ 1.9 atm. Each cell was vented three times in the next 48 hours to release most of the D_2O formed from the reduction of oxide on the catalyst. On the third release, little or no D_2O steam was seen.

Initial ^4He measurements were made soon after the last venting. The ^4He concentration in the gas was measured regularly for the next 8 weeks. Sample SC20 never showed any ^4He concentration over 0.05 ppm. Gases were archived from the other three experiments. As shown in Table 1, the ^4He concentration in the other three cells reached a maximum of 4.2 to 9.1 ppm. Approximately 24 weeks after the experiment terminated, the new Micromass VG 5400 high resolution, high sensitivity, noble gas mass spectrometer was ready for use. Results measured from the three archived gases are shown in Table 1. The results from SC19 and SC22 measurements approximately one year after the experiments' termination are also shown.

Table 1 He Concentration from Experiments SC19 through SC22

Sample	He-4	He-3	Ratio	Sampled
SC19	6.5	8.8	1.35E-06	3/29/2002
SC21				3/29/2002
SC22	0.46	0.65	1.41E-06	

SC19 old	6.03	8.11	1.34E-06	9/11/2001
SC21 old	5.36	8.18	1.53E-06	9/12/2001
SC22 old	0.4	0.58	1.45E-06	9/12/2001

Experiment ended 3/26/01

SC19	9.1
SC20	5.6
SC22	4.2

Final Cold

SC19	6.0
SC21	3.8
SC22	4.2

SC20 = 0 at all times

The persistence of a ^4He concentration above lab ambient (5.22 ppm) in SC19 argues against in-leakage from air. However, since the $[^3\text{He}]/[^4\text{He}]$ ratio does not significantly differ from natural isotopic ratio, we cannot invoke the formation of a single isotope.

Four more experiments, SC23 through SC26, were each charged with 10g of the same catalysts used in SC19 through SC22. These were pre-treated as above but after the final steam venting, each vessel was pressurized to ~ 3.0 atm (abs) D_2 . Again, ^4He concentrations in the cells' headspace were measured regularly over the following eight weeks. Approximately eight weeks after completion, ^4He and ^3He concentration measurements were made.

Results of all helium measurements for experiments SC23 – SC26 are shown in Table 2. All cells showed non-zero ^4He concentration but only SC 23 showed a concentration significantly larger than that for air ambient. Interestingly the $[^4\text{He}]/[^3\text{He}]$ ratio was identical to natural for SC23 but 10 to 15% above natural for the other three cells. The results of all four experiments suggest that the He present did not come from air but cannot conclusively suggest a reaction mechanism.

Table 2. He Concentration Measured from SC23-SC26

Sample	He-4/ppm	He-3/pptr	He-3/He-4 Ratio	Note
SC23	3.8			Maximum
SC24	1			Maximum
SC25	7.3			Maximum
SC26	0.5			Maximum
SC23	3.8			Experiment Termination
SC24	1			Experiment Termination
SC25	5.4			Experiment Termination
SC26	0.4			Experiment Termination
SC23	9.15	12.43	1.36E-06	8 weeks after experiment
SC24	2.98	4.57	1.53E-06	
SC25	5.81	8.78	1.51E-06	
SC26	2.61	4.07	1.56E-06	

Experiments SC27-SC30 were performed to compare the different lots and Pd concentration of Pd on C catalysts. SC27 and 28 had 10 g. of a different lot of G-75E from that used in SC19-SC26. SC29 used the same catalysts lot used in the above experiments. SC30 was charged with 10 g of G75F (0.6 to Pd on coconut shell carbon). At the suggestion of Dr. L. Case, the gas pretreatment and venting was carried out using H₂ gas. After final venting, SC28-SC30 were evacuated quickly and filled with ~ 3.0 atm (of D₂. SC27 was topped with ~ 3.0 atm H₂. For this experiment each cell was sampled regularly, measuring both ³He and ⁴He concentrations in the reactors' headspace. The results of these measurements are shown in Table 3. The results from SC28 and SC29 are very similar to those measured for SC27, the H₂ experiment. SC30 shows a ⁴He concentration 30% above that for air ambient with a [³He]/[⁴He] ratio not significantly different from natural. Again, this yields inconclusive evidence of helium formation.

Table 3. He Concentration Measurements for Experiments SC27-SC30

Time/hr	Sample	Pressure/atm	[He-4]/ppm	[He-3]/pptr	Ratio	Atoms He-4	Atoms He-3	9/20/2001 8:00
3	SC27	3.89	2.82	0.5	1.88E-07	1.7E+16	3.2E+09	9/20/2001 11:00
4	SC28	3.83	0.08	0.2	2.13E-06 *	4.8E+14	1.0E+09	9/20/2001 12:00
6	SC29	0.56	0.2	0.3	1.40E-06 *	1.7E+14	2.4E+08	9/20/2001 14:00
8	SC30	2.53	0.06	0.2	3.00E-06 *	2.4E+14	7.1E+08	9/20/2001 16:00
197	SC27	3.63	1.67	0.3	1.80E-07	9.4E+15	1.7E+09	9/28/2001 13:00
271	SC28	3.14	0.1	0.2	1.50E-06 *	4.9E+14	7.3E+08	10/1/2001 15:00
272	SC29	2.01	0.23	0.0	0.00E+00 *	7.2E+14	0.0E+00	10/1/2001 16:00
288	SC30	0.82	5.14	7.4	1.44E-06	6.6E+15	9.4E+09	10/2/2001 8:00
439	SC27	3.71	0.93	0.3	3.33E-07	5.4E+15	1.8E+09	10/8/2001 15:00
441	SC28	2.46	0.07	0.1	1.14E-06 *	2.7E+14	3.1E+08	10/8/2001 17:00
458	SC29	1.6	0.31	0.5	1.55E-06 *	7.7E+14	1.2E+09	10/9/2001 10:00
462	SC30	0.95	6.51	8.8	1.35E-06	9.6E+15	1.3E+10	10/9/2001 14:00
675	SC27	3.1	0.47	0.1	2.98E-07	2.3E+15	6.7E+08	10/18/2001 11:00
676	SC28	2.22	0.05	0.1	1.80E-06 *	1.7E+14	3.1E+08	10/18/2001 12:00
678	SC29	1.38	0.34	0.5	1.56E-06 *	7.3E+14	1.1E+09	10/18/2001 14:00
681	SC30	0.97	6.81	9.5	1.40E-06	1.0E+16	1.4E+10	10/18/2001 17:00
819	SC27	2.13	0.19	0.1	5.26E-07	6.3E+14	3.3E+08	10/24/2001 11:00
820	SC28	1.98	0.06	0.1	1.67E-06 *	1.8E+14	3.1E+08	10/24/2001 12:00
822	SC29	1.2	0.4	0.6	1.53E-06 *	7.5E+14	1.1E+09	10/24/2001 14:00
825	SC30	0.95	6.45	8.9	1.38E-06	9.5E+15	1.3E+10	10/24/2001 17:00
1131	SC27	1.1	1.02	0.7	6.86E-07	1.7E+15	1.2E+09	11/6/2001 11:00
1132	SC28	1.57	0.42	0.5	1.29E-06 *	1.0E+15	1.3E+09	11/6/2001 12:00
1134	SC29	1.2	1.1	1.6	1.42E-06 *	2.1E+15	2.9E+09	11/6/2001 14:00
1137	SC30	0.95	6.31	8.5	1.35E-06	9.3E+15	1.3E+10	11/6/2001 17:00
1323	SC27	1.1	0.25	0.3	1.32E-06	4.3E+14	5.6E+08	11/14/2001 11:00
1324	SC28	1.57	0.78	1.1	1.45E-06 *	1.9E+15	2.8E+09	11/14/2001 12:00
1326	SC29	1.2	0.93	1.4	1.49E-06 *	1.7E+15	2.6E+09	11/14/2001 14:00
1329	SC30	0.95	6.69	9.5	1.42E-06	9.9E+15	1.4E+10	11/14/2001 17:00

SC29 topped up to 3.89 atm at 7 hours

* large uncertainty

** H2 gas

In sum, suggestive but not conclusive, evidence of helium formation has been seen in at least half of the twelve Case Catalyst experiments performed during this phase of the project. Technical problems associated with the mass spectrometer have prevented further measurements along these experimental lines.

Task 2: -Double Structured Cathodes.

The double-structured (DS) cathode prepared by ENEA Laboratories for this experiment, is very similar to that used by Arata. The cathode has only one lead wire since we did not measure bulk resistance because such measurements do not reveal the loading of the Pd powder deuterium loading. The cell has a large Pt anode and operates with 500 ml of 0.1 M LiOD/D₂O. A large surface area (40 cm²) Pt on carbon cloth catalyst is used because of the high currents employed in this experiment. The cell is mounted in a $\pm 4\%$ reproducibility mass flow calorimeter. This calorimetry was substantially less accurate and precise than our earlier reported results. As the cells are not watertight and cannot be completely immersed.

Three DS cathode experiments were performed; one using a stainless steel cathode in LiOH/H₂O and the other two using Pd DS cathodes in LiOD/D₂O. The operation of the cell and calorimeter are described elsewhere. These experiments require approximately three months of cathodic electrolysis. At the conclusion of the cathodic experiment an extended period of anodic electrolysis was performed to reduce the internal pressure of the DS cathode. This was expected to increase the helium concentration, drive some tritium into the electrolyte, and make for safer cathode puncturing.

At the end of the current protocol the cathodes were separately punctured using the apparatus shown in Figure 2. Figure 2 also shows the cathode from EA-Pd1 with the puncturing tool inserted. The bellows chamber is He leak-tight and connected to a manifold with a pressure transducer. The cathode is inserted and the puncturing tool centered. After sealing and evacuation, the bellows chamber is compressed in a hydraulic press while monitoring the internal pressure on the transducer. When the cathode has been successfully punctured, the internal pressure increases rapidly. At this point, the gas is sampled and ⁴He and ³He concentration determined. The chamber is then heated to 250°C and another gas sample is collected and measured. Finally, an evacuated collection vessel is connected, through the evacuated manifold, to the chamber and cooled in liquid nitrogen. The gases evolved/released from puncturing/heating are collected, as much as possible, in this collection vessel. This allows for subsequent ³He concentration measurements to determine if ³He is being produced from ³H decay. The electrolyte is collected and a portion is distilled and its ³He content measured using liquid scintillation methods. Mass flow calorimetry was performed on all cells. No excess power, greater than the limits of error, was measured.

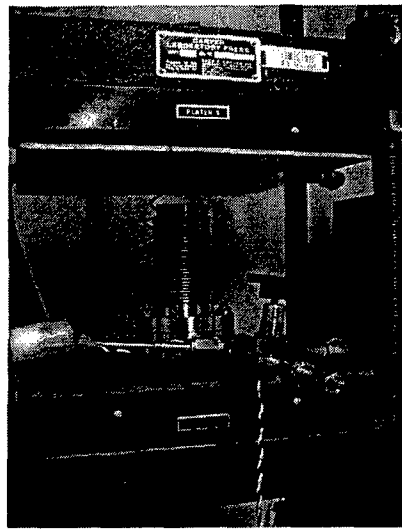


Figure 2. DS Cathode and Puncturing Device

Table 4 shows the current protocol followed for both Pd cathode experiments. At the termination of experiment EA-Pd1 the cathode was removed and punctured in the apparatus described above and the gases collected and analyzed for He concentration. The results presented in Table 5, obviously show that no significant amount of He was produced. The tritium present in the electrolyte is shown in Table 6. Again, no tritium enrichment was seen.

Table 4. Current Protocol for DS Cathode Experiments

EAPd #1		EAPd #2	
Time/hr	Current/A	Time/hr	Current/A
1031	5	338	5
216	5.5	216	5.5
241	6	241	6
100.5	6.5	100.5	6.5
503.5	1	503.5	1
66	6	66	6
27.5	6.5	27.5	6.5
503	7	503	7
26	-0.01	26	-0.01
813.5	-0.03	813.5	-0.03

Table 5. EAPd1 He Gas Phase Concentration

Description	He-4/ppm	He-3/pptr	ratio (*10^6)	error
First Puncture, room temp	0.36	0.43	1.19	0.20
First puncture, sample heated to 250 C	0.14	0.03	0.21	2.00
2nd puncture, room temperature	0	0		
2nd puncture, repeat	0	0		
2nd Puncture, repeat	0.19	0.22	1.16	0.20
2nd Puncture, repeat	0.02	0.05	2.50	2.00

Table 6. ENEA - Arata Pd1 Electrolyte Tritium Results

Sample	CH 1/cpm	Ch 2/cpm	
H2O	22	17	
D2O	112	74	
Pd 1	109	68	
			ch2/ch1
D2O*	90	57	0.63
Pd1*	87	51	0.59

Experiment EA-Pd2 was treated and analyzed similarly to EA-Pd1. The helium gas phase concentration results are enumerated in Table 7. No significant He concentration or tritium enrichment was seen.

Table 7. EAPd2 He Gas Phase Concentration

Description	He-4	He-3	ratio (*10^6)	error
First Puncture, room temp	0.08	0.22	2.75	1.20
First puncture, repeat	0.14	0.03	0.21	1.50
First puncture, repeat	0.15	0.29	1.93	0.60
First puncture, high pressure aliquot	0.02	0.02	1.00	2.00

Figure 3 is a photograph of the surface of the EA-Pd1 cathode. The large crack in the picture extends into the inner, Pd powder chamber. This would have released any He generated in this inner chamber. Arata claims that the inner Pd powder is the source of both the He and the excess power.³ As such, if this crack grew in the first few weeks of the experiment, before any excess power would be expected, no excess power would be produced due to direct access of the electrolyte of the palladium black without benefit of applied electrochemical potential. Since the tritium seen in a previous DS cathode experiment (see Appendix B) came from the decay of ³He in the inner chamber, no tritium enrichment would be expected as well.

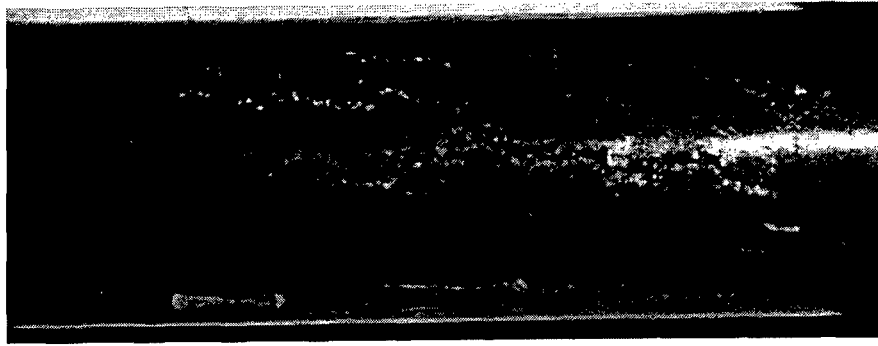


Figure 3. Photograph of Crack in EA-Pd1 Cathode

The lack of significant anomalous results in experiment EA-Pd2 is less easily accounted for. The DS cathode produced by ENEA is similar enough to the original Arata cathode to allow proper loading of the powder in the inner chamber. However, we cannot be sure that the metallurgy of the Pd powder is identical to that used by Arata. Arata claims that the fundamental particle size of his Pd powder is less than 100nm. The Alfa # 00776 Pd powder, used in our experiments has a particle size of 400 ± 150 nm. This may not be small enough to participate in the spillover deuterium loading mechanism reported by Fujita and Arata.

Task 3: -Metal Phase He Studies.

Figure 4 is a photograph of the solid sample furnace for the Micromass VG5400. The large copper feed-throughs allow for the high current necessary to melt the Pd samples. The tungsten spiral wire holds the sample (powders are placed in a tungsten foil) when it is heated to 1500°C by passing >12A through the Tungsten. The temperature vs. current curve for the furnace is shown in Figure 5.

No mass spectrometric data has been collected from this furnace due to technical failures associated with the spectrometers' electronics. We intend to collect spectroscopic data immediately upon effecting repairs on the instrument, which we expect later this year.

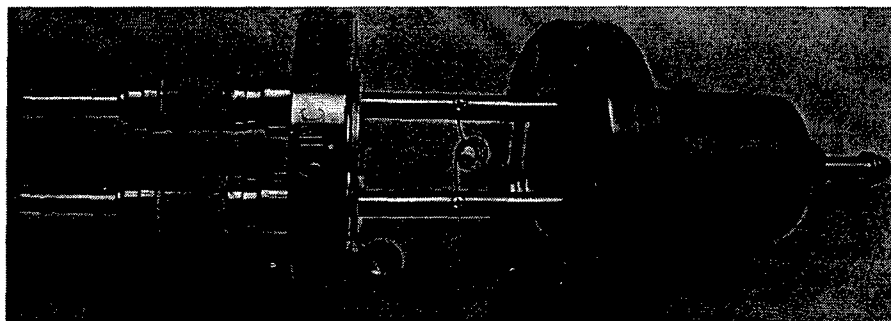
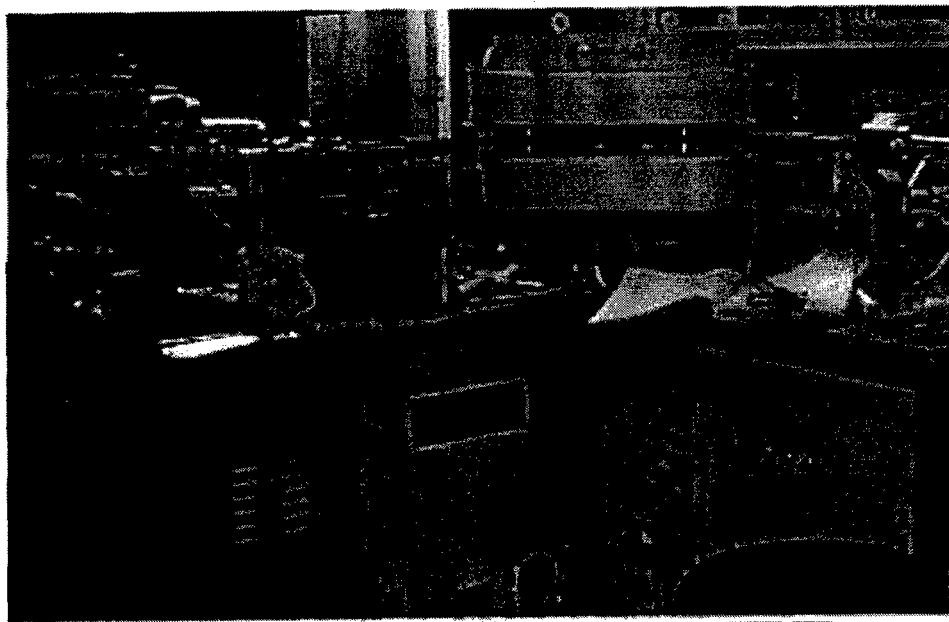


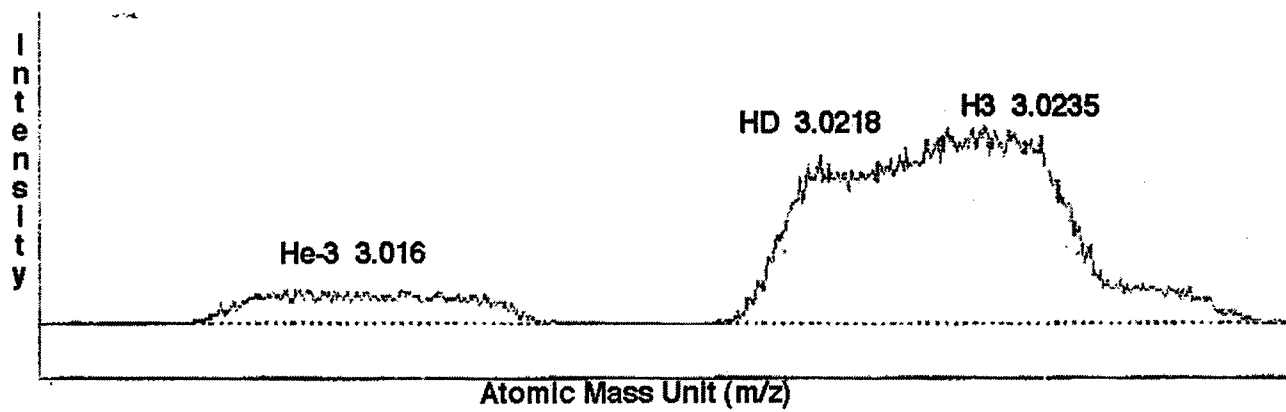
Figure 4. Photograph of the Solid Sample Furnace

Table 7. EAPd2 He Gas Phase Concentration

Description	He-4	He-3	ratio (*10 ⁶)	error
First Puncture, room temp	0.08	0.22	2.75	1.20
First puncture, repeat	0.14	0.03	0.21	1.50
First puncture, repeat	0.15	0.29	1.93	0.60
First puncture, high pressure aliquot	0.02	0.02	1.00	2.00

APPENDIX A
Micromass VG 5400 Magnetic Sector Mass Spectrometer
Operating Procedure





Mass 3 Spectrum from Micromass VG 5400

BAKING

General:

- Turn off Output and main switches on PPS2A, and Emission on console.
- Turn off WARP + EM.
- Vent Faraday Head and Remove
- Disconnect all EM cables, etc. including grounded Bias (shorted BNC).
- Remove preamp and cables.
- Pull out plug; unscrew acrylic shield, remove board screws & barrels carefully
- Push magnet back. Put insulating flaps on Floor
- Clean surface with acetone
- Remove crank handles

Into ion pump:

- Close turbo valve
- Close Getter valves – Back off 1 turn
- Ion pump open and on (usually)
- Isolation valve open
- Protect both getter valves with Al foil loosely
- Prep System valve closed

Into Turbo:

- Ion pump off – valve 1/2 turn open
- Isolation valve open
- Turbo valve open
- Getters - 1 turn open
- Prep Sys. valve closed

Installing Ovens:

- Rear Middle first then two side rear ovens
- Front Middle, Front Right, then Front left - tight tolerance near ion pump
- Ion gage on
- Heater tapes – full bore
- Start tapes same as 1st heaters (or 1 hour later)
Stagger next heaters ½ hour and 1 hour later
1st Center, 2nd Left, 3rd Right
- Control switch on top (bottom is always on) Temperature set to 320X or 250 C
- Ovens off 4a.m. Tapes off at least 1 hour later

Cooling:

During cooling degas getters during turbo bake outs (Ion pump closed).

At 60 C ramp up to 90V carefully. Leave for 20 minutes.

- Leave tapes on (keep $< 1 \times 10^{-5}$ torr)
- To degas ion gauge, close analyzer and ion pump valves, turbo open. Try to do with tapes on and system still at 100C. Turns off after 20 min.
- Don't degas anything into ion pump
- Remove ovens at 50 C up to room temperature.
- Attach and reconnect preamp. Recable detector, etc. (Connect EM + Faraday, Lens, Retard, "C" signal out collector to PreAmp box, "B" (Bias) shorted BNC, "F" High Voltage Cable marked "Elect Multiplier")
- Collimator suppressor cable is directly below faraday
- Faraday Cable Twist Plug.e.fo
- Check source continuity and shorts. Connect source cables. Connect interlock plug.

Starting up:

WARP on, EM on, (trip on)

Emission control power on, press Trip Reset.

PPS on (turns on source). Output on, (takes 45 seconds to warm up.) Bring up to 4.5 kv slowly.

OPENING SPECTROMETER

Venting flight tube

- Turn off filament 10 minutes before venting. Close turbo valve. Vent through prep system.
- Turn off ion gauge if pressure rises.

Removing Ion Source

- To replace filament or work on source
- Remove at connection flange (Very Heavy)
Use 4 bolts to jack off the flange
Keep from hitting ceramic with wrench (VERY IMPORTANT)
Use the 4 bolts as stand and/or handle.

Removing Faraday Cup

- Vent Faraday Cup using knurled nut over potentiometer.
- Pump for 5 minutes. To vent, close "AMP housing ISO" valve, open vent
- Wait 10-15 minutes to vent

Venting turbo pump and ion gauge

- Isolate turbo from flight tube
- Turn off rot. pump,
- Turn off turbo pump
- Open turbo vent valve when $< \frac{1}{2}$ speed (2 bars)
- At 0 bars on turbo comptroller, open AMP housing ISO" valve

When Done

- Bake @ 250 C into turbo before turning on source.
- Check for contaminants and rate of growth. If o.k. then continue to tuning.
- If H₂O etc. are present, bake again @ 350 C into ion pump or turbo.

Operational Reference

Oven - Oven resistance should be 33 ohms

Cu gasket valves have marks for tightening

AQS UNIT

MAN ALLOWS UP/DOWN TO CHOOSE CHANNEL

RUN/HOLD TOGGLES DISPLAY

CH 9 IS HV (9.00=4500V)

CH 3 IS FARADAY OUTPUT

Hi Res Magnet control

Digital is Computer Control, Man is Manual Control

Keep Field/Current control on Field

Emission Control: For He set to 800 mA. For others use 200ma

$E_{\text{ionization}}$ usually has 2 maxima in each mass range. (for He use ~92 eV, for Ar use ~83 eV)

WAIT FOR ION GAGE TO STABILIZE BEFORE TURNING ON FILAMENT

Programmed Row sys. Acc. @ all measure at 4500V

Source Control A Fill current ~ 2A of 200 μ A temp and ~2.8 A at 800 μ A
Rep. -14V moxxxx 4 (not likely to change for more 3)

E.V Ip
Emission Current < 1 mA (0.4 mA min)

Repeller Focus and beam center settings should be set n knobs not meter.

To adjust Fil. limit SA

Pull out emission control box

Adjust trimpot that is on left most forward

When switch is on Fil Limit, Fil is not getting current

When Replacing Fil

Bake system several times then bake system with Fil on

Overnight

Fil. on down to 10 μ A temperature

Can turn off HiV P.S. (but takes ≥ 1 hour to warm up).

Software

- F1 Configuration #'s – doesn't control anything
- F2 Calibration mass Cal (Back ID)
- F3 A Same
- F4 Peak flat mass with a peak of isotope → 200 Kcps
Turning on lap peak (Ar 10V)
Scan using High Far. – Pause (F5 in center of peak)

Tuning

Tuning Peak Height

- Tune with ≤ 0.2 amu on screen display
- Sit on top of peak. Repeller Offset is most sensitive
- Iterate Repeller, fine focus, repeller beam center
- Read peak height on analog or digital meter
- Calculate resolution – one peak on screen – press Alt R

Tuning Peak Flatness

USE F4 PROGRAM – TUNE AS ABOVE

Tuning Magnet

- To tune from ^{40}Ar to mass 4 must tighten CW ~ 1.0 turns in front and loosen xxreap (CCW)
- Turn flat (1/6 turn) at a time
- Set up ^4He on High Faraday with air sample
- Check resolution (Alt R) after every screen. Adjust magnet and look for better (higher) resolution number (use 5% resolution).

Tuning WARP Control

- Retardation Potential: adjust E_{retard} until total CPS starts to come down and then back off.
- Adjust lens for best peak shape and iterate with E_{retard} .

Setting Multiplier Gain

- Adjust EM voltage and Dsc for best long flat peak. (Plot Int.vs Des. settings at different EM V).
- Calibrate using High Faraday. Measure baseline voltage
- Use Peak intensity $< 3\text{mV}$ over baseline. Subtract baseline voltage and multiply by $10^{11} \Omega$ to calculate I (A)
- Convert I to coulombs/s
- Multiply charge/s x (0.85) → CPS
Adjust EM HiV to get same CPS
Baseline = 5.6 mV, Peak (28) = 7.5 mV :

$$\frac{(1.9 \times 10^{-3} \text{ V})}{10^9 \Omega} = 1.9 \times 10^{-14} \text{ A}$$

$$(0.85 \text{ count/charge}) \times (1.9 \times 10^{-14} \text{ coulomb/s}) / (1.6 \times 10^{-19} \text{ coulombs/charge}) = \sim 100,000 \text{ count/s}$$

APPENDIX B
PROCEEDINGS OF 8TH INTERNATIONAL CONFERENCE ON COLD
FUSION - MAY 2000
LERICI, ITALY

The Emergence of a Coherent Explanation for Anomalies Observed in D/Pd and H/Pd Systems; Evidence for ^4He and ^3He Production

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Introduction

Numerous observations have been made of apparent anomalies in carefully performed experimental studies of D/Pd and H/Pd systems. Such anomalies include: prompt emission of electrons and charged particles; unexplained heat in excess of known input sources; the residual presence of light elements (notably ^3H , ^3He and ^4He); the possible occurrence of isotope anomalies in higher mass elements (including the host metal); unusual electrical conductance effects both stable and transient (not discussed in this paper). The features that unify these apparently disparate observations are the common elements of the needed experimental stimuli, and the requirement for extended lattice coherent processes in any obvious explanation.

Presented in this paper are the results of four experiments conducted using palladium structures loaded with hydrogen or deuterium by gas and electrolytic means. The purpose was to evaluate possible evidence for d-d fusion reactions in lattice structures, in the temperature range 25-250°C. Primary evidence sought and evaluated were the appearance of anomalous excess heat accompanied by the production of light isotopes: ^3He and ^4He .

Description

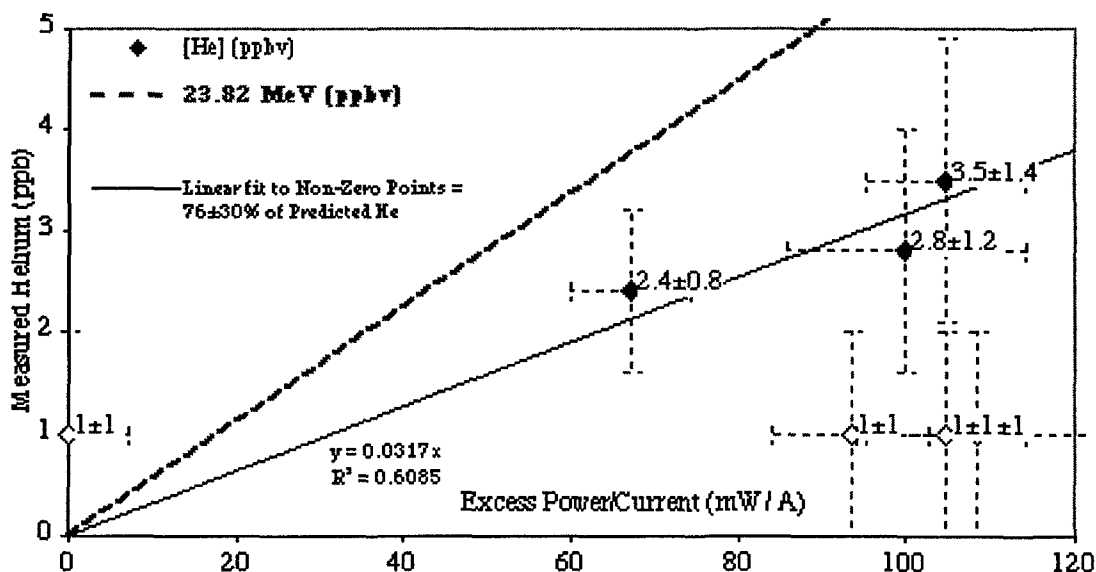
Experiments were performed in four basic geometries:

- 1) Open cell electrolysis of D_2O at Pd and Pd-alloy wire cathodes using an accurate integral boundary Seebeck calorimetry.¹ These experiments were designed to replicate earlier observations of Miles et. al.² of the correlated appearance of excess heat and helium in glass cells. The experiments at SRI were performed in rigorously metal sealed cells incorporating active exclusion of ^4He to increase the measurement sensitivity and accuracy.
- 2) Loading of D_2 and H_2 into Pd on carbon supported catalyst using modest gas pressures (1-3 Atm.) and temperatures (170°–250°C). These experiments were designed to test the claim by Case³ to observe excess temperature and increasing ^4He levels under similar conditions. Experiments at SRI were performed in sealed Nupro[®] 50 cc stainless steel vessels connected to a steel manifold. Periodic measurements of ^4He were made by direct connection to an Extrel[®] mass spectrometer capable of resolving the mass-4 peaks of D_2 and ^4He . Using information recorded from temperature sensors placed inside and outside active and reference gas cells it was possible to obtain heat-flow calorimetric information at times when the catalyst bed temperature rise was significant.

- 3) Closed cell electrolytic loading of D into Pd wire cathodes in a rigorously metal-sealed apparatus using highly accurate mass flow calorimetry.¹ These experiments were intended to replicate earlier results of excess heat measurement at SRI in thermodynamically closed electrolytic loading cells, for which ambient exclusion of ⁴He was not possible.^{4,5}
- 4) Closed cell electrolytic loading of D (and H) into hollow Pd cathodes sealed to contain small dimension Pd-black powders. These experiments were performed to replicate published results by Arata and Zhang in which excess heat, ⁴He and ³He, were found to be associated with the electrolysis of such "double structured" cathodes⁶ in D₂O, but not in H₂O. In experiments performed at SRI accurate mass flow calorimetry⁴ was used to evaluate and compare the heat production of double structured cathodes electrolyzed in D₂O and H₂O in otherwise identical cells. The contents of the cathode void volumes were subjected to retrospective analysis for light isotopes.

Results

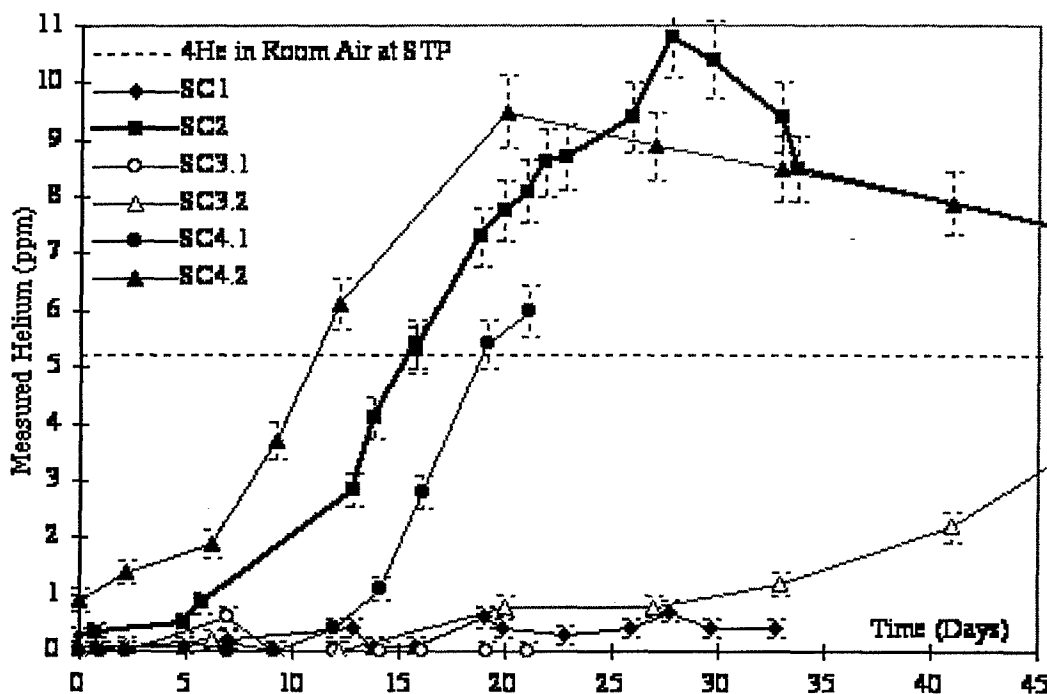
Figure 1 presents the results of concurrent excess power and helium measurements performed during open cell electrolysis using two different Pd and Pd-alloy cathodes. In three instances where excess power was measured at statistically significant levels, ⁴He also was found to be conveyed out of the cell in the electrolysis gases (D₂ + O₂). The solid line in Figure 1 plots the regression fit of these data to a line passing through the origin; the dashed line is that expected for ⁴He generation according to the reaction;



It is clear from the slopes of these two lines that the observed ⁴He constitutes only 76 ± 30% of the ⁴He predicted by equation [1]. A more significant problem in Figure 1 is

that three further ^4He samples, taken at times of non-zero excess power (open diamonds), exhibited helium concentrations only at the level of the analytical uncertainty, as did numerous samples taken in the apparent absence of excess power production. Clearly if ^4He is produced in association with excess power, it is not released to the gas phase immediately, or completely.

Experiments in category "2" in which Pd on C catalyst materials were exposed to D_2 and H_2 gases for prolonged periods, exhibited a range of behaviors. Figure 2 summarizes 6 of 16 results obtained in paired cells.



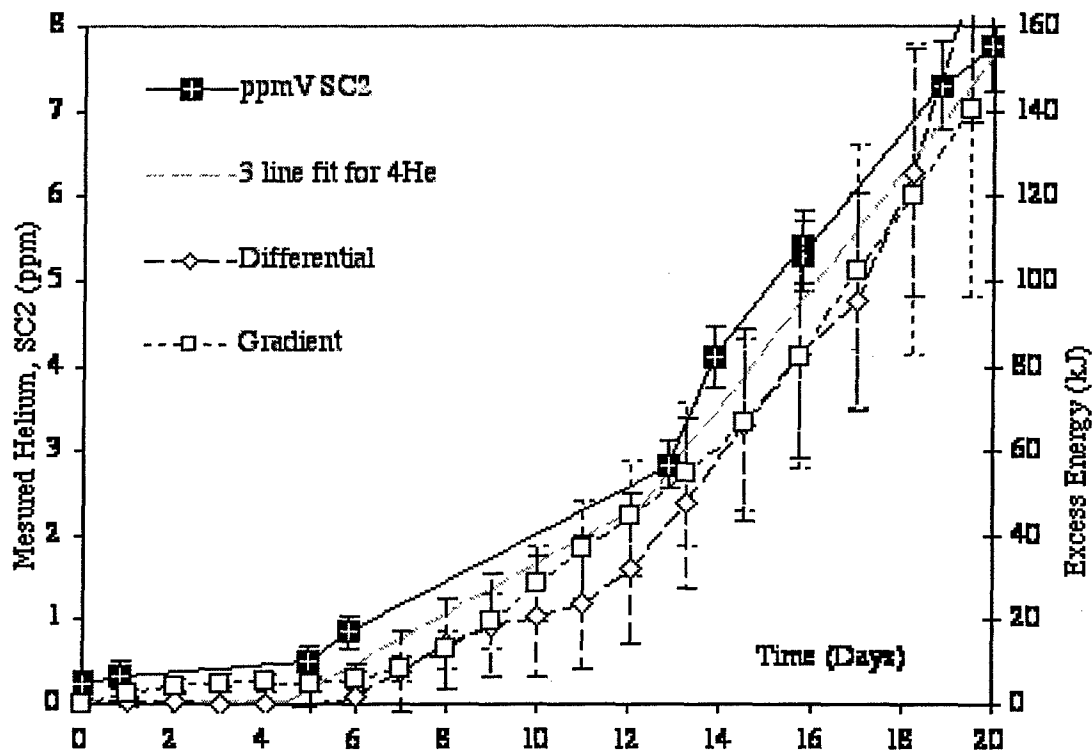
Using direct, on-line, high-resolution mass spectrometric measurement of $[\text{}^4\text{He}]$ we observed the following behaviors:

- (i) cells that show no increase of ^4He over long periods of time (including all cells operated with H_2 instead of D_2);
- (ii) cells that exhibit a slow, approximately exponential increase in $[\text{}^4\text{He}]$ with time;
- (iii) cells that display no measurable increase in $[\text{}^4\text{He}]$ for a period of several days, followed by a rapid, approximately linear rise in $[\text{}^4\text{He}]$ to levels sometimes exceeding that of the ambient background.

Using data from temperature sensors placed in the catalyst and gas phases, and situated in the room, we are able to make heat flow estimates in one of two ways:

- a) a Gradient method, based on the relationship between the temperature difference between catalyst bed and confined gas, and the heater input power;
- b) a Differential method, based on the temperature differences between active and reference catalyst bed sensors, and room temperature, as a function of the relative input heater powers.

The energy estimated in excess of that provided by the heater for these two calorimetric methods is plotted in Figure 3, together with the measured helium concentration during the time of greatest derivative, $\partial[{}^4\text{He}]/\partial t$ in experiment SC2. It is clear that the appearance of excess heat and the apparent increase in $[{}^4\text{He}]$ are temporally correlated.



In an attempt to establish a quantitative correlation, Figure 4 plots the two calorimetric estimates of excess heat production interpolated from Figure 3, versus the measured increase in $[{}^4\text{He}]$ (the value plotted in Figure 3 minus the ${}^4\text{He}$ initially present in the D_2 gas). Regression lines through these data incorporating the origin have slopes: $Q=31 \pm 13$ and 32 ± 13 MeV per ${}^4\text{He}$ atom, respectively, for the gradient and differential calorimetric methods. Although these Q values include that of reaction [1] within their assigned uncertainties, the mean values for the ${}^4\text{He}$ presented directly to the gas phase for analysis is only $\sim 75\%$ of that predicted by equation [1].

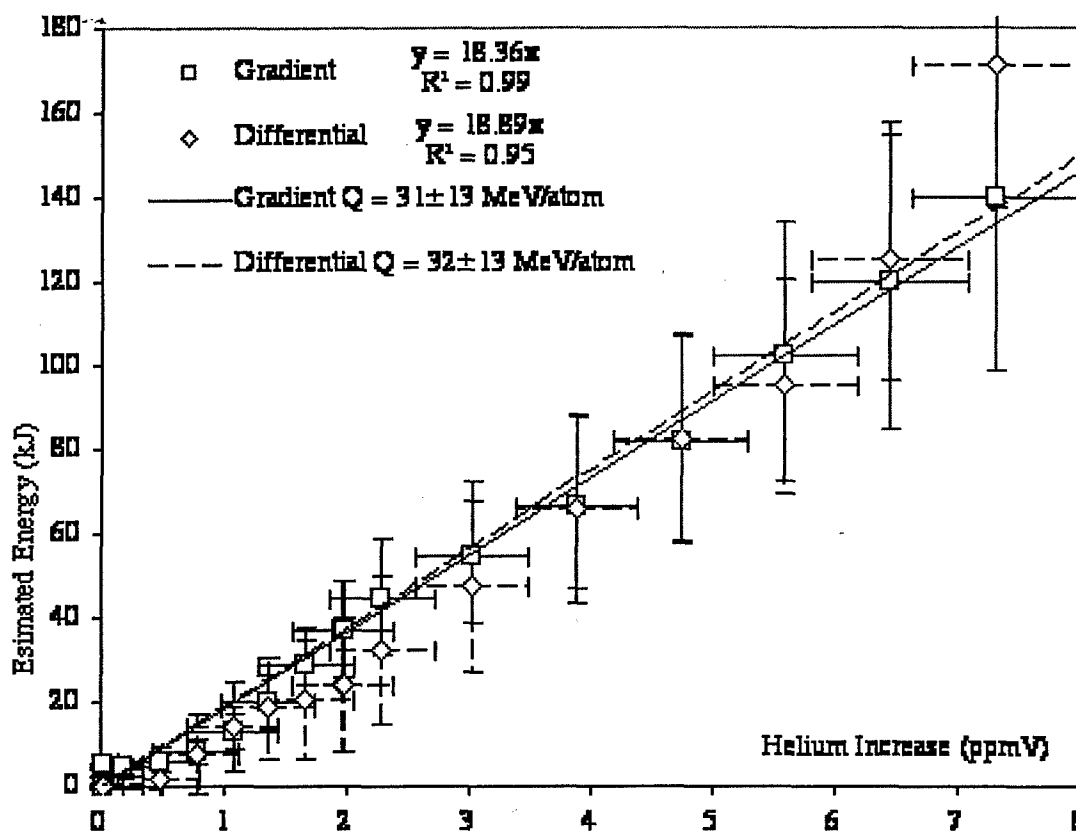


Figure 4 Estimated Energy versus Helium Increase.

Clarification of a possible origin for the apparent ^4He deficit in experiments "1" and "2" can be obtained from the results of experiment "3". Approximately 82 kJ of excess heat was measured in the electrolysis of a 100 mm x 1mm Pd wire cathode in D_2O . This experiment was performed in a rigorously metal sealed and helium leak-tested cell and apparatus provided with the facility to sample the gas in the headspace. When initially analyzed following a period of excess power production, the gas phase contained only 62% of the ^4He expected if reaction [1] were the source of the excess heat. A second sample showed an increase in [^4He] despite the fact that the helium content of the vessel had been diluted with D_2 containing low levels of ^4He , in order to make up the initial gas volume after the first gas sample. Taking these increases as evidence of sequestered ^4He , the cathode was subjected to an extended period (~200 hours) of compositional and temperature cycling by varying the current density in both anodic and cathodic directions.

A mass balance of ^4He was calculated based on two further gas samples: one to determine the helium content of the D_2 gas used initially to fill and refill the sealed metal cell ($0.34 \pm 0.007 \text{ ppmV}$); the other to measure the final helium concentration in the gas phase after exercising the cathode to release trapped gases ($2.08 \pm 0.01 \text{ ppmV}$). Taking into account the amounts lost by sampling, and introduced with make-up D_2 , a calculated mass balance for ^4He in the gas phase after compositional and thermal cycling of the cathode results in a number that is $104 \pm 10\%$ of the number of atoms quantitatively correlated with the observed heat via reaction [1].

The results of experiment "4" generally confirm those published earlier by Arata and Zhang of the appearance of excess heat in the electrolysis of "double structured" palladium cathodes⁶ in D_2O , but not in H_2O . Figure 5 plots the calorimetric results of cathodically exercising two nominally identical cathodes in intentionally similar cells, one in 0.1M LiOD and the other in 0.1 M LiOH. In the same range of input powers, the heavy water cells yields clearly more output heat (plotted as excess power) than does the light water cell.

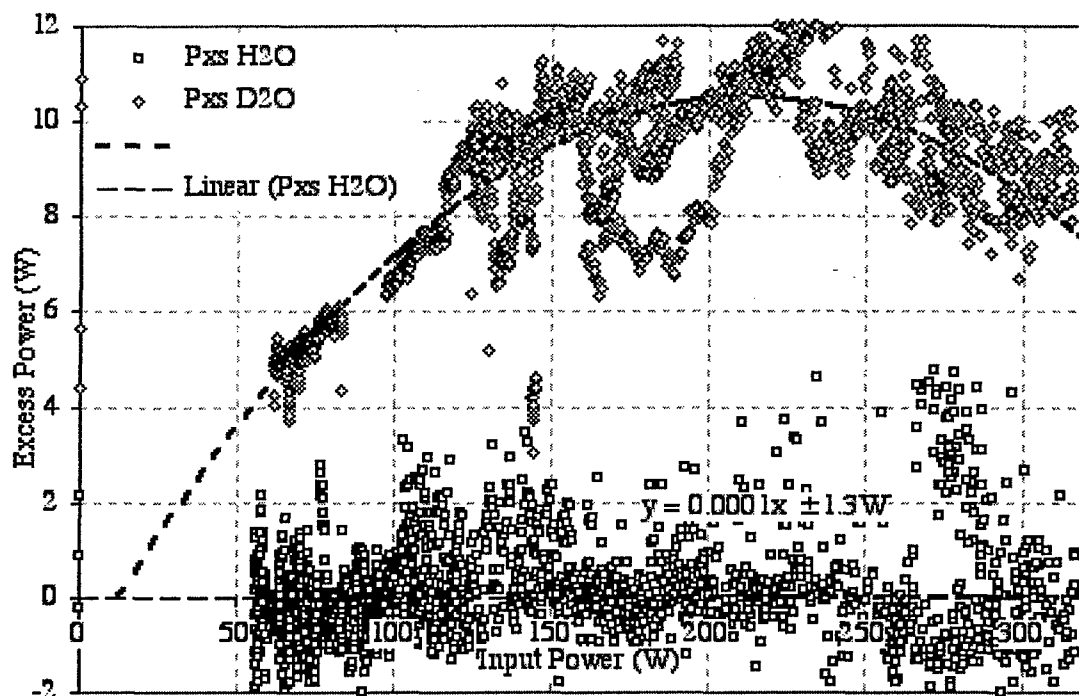


Figure 5 Excess Power versus Input Power measured for Arata-Zhang Double Structured Cathodes using Mass Flow Calorimetry.

The maximum excess power observed in D_2O was $9.9 \pm 1.3\%$ of the measured power input, with the average value being approximately half the maximum. The measured power excess exceeded the experimental uncertainty (1-2%) for a period of ~86 days to produce an integrated energy excess of 64 ± 6 MJ for the D_2O cell. For the H_2O cell in the same period of time the measured energy excess was -1 ± 6 MJ.

At the conclusion of the experiment, both cathodes were removed and placed successively in a sealed chamber where they were punctured mechanically, and the gas contents of the cathode void volumes were extracted for analysis. The Pd black powders also were removed and the Pd metal walls of the hollow cathode were sectioned for analysis. Measurements of 3He and 4He made by volatilizing samples of the metal phases in an ultra high resolution helium isotope mass spectrometer⁷ revealed the presence⁸ of small amounts of 4He , but very large amounts of 3He relative to the natural isotopic abundance ratio (1.38×10^{-6}). This result was observed for the D_2O cathode but not for the

H₂O cathode. One set of such results is summarized in Table 1.

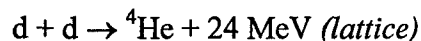
Table 1 Measurements of ³He and ⁴He.

Isotope Error	Sample ⁴ He	Palladium - Bulk ³ He	⁴ He	Palladium - Black ³ He	
	(± 10%)	(± 1%)	(± 10%)	(± 1%)	
Measured	7 x 10 ⁷	1.06 x 10 ⁶	8 x 10 ⁷	4.57 x 10 ⁶	Atoms/mg
Total	7.3 x 10 ¹²	1.1 x 10 ⁹	9.9 x 10 ¹¹	5.5 x 10 ¹⁰	Atoms
⁴ He/ ³ He	67	1.2 x 10⁴	18	4.0 x 10⁴	R

The numbers in bold represent the factor by which the ³He/⁴He ratio is enhanced over the natural abundance, and indicate unambiguously a non-natural source for ³He. The radial distribution of this ³He (not shown here) clearly demonstrates that a source of this isotope was established within the cathode void volume, and that this source was not present at the outset of the experiment, or in the light water blank.

Conclusions

There exists a strong time correlation between the rates of heat and helium production measured using on-line high-resolution mass spectrometry. In experiments performed using three different metal sealed cells, three different calorimetric methods, by electrochemical and gas loading means and with ⁴He analyses made at three different institutional laboratories, there is observed a quantitative or near-quantitative correlation between heat and ⁴He production consistent with the reaction:



Evidence for near-surface retention of ⁴He in the lattice can be used to accommodate the discrepancy between measured and expected yields of ⁴He.

Evidence was obtained for excess heat production in the electrolysis of D₂O using "double structured" cathodes, supporting original claims by Arata and Zhang⁶. Associated with this result, although not quantitatively correlated to it, is the production of a significant amount of ³He within the cathode void volume, suggesting the presence of a second nuclear reaction. The origins of this ³He and its theoretical implications are being explored further.

Acknowledgment

We gratefully acknowledge the crucial support and helpful guidance of a large number of contributors to the findings presented here. In particular we would like to recognize the technical contributions of Prof. Y. Arata, Dr. B. Bush, Dr. L. Case, Dr. S. Crouch-Baker, Prof. B. Clarke, Dr. D. Di Gioacchino, Mr. R. George, Dr. B. Oliver, Dr. T. Passell, Dr. V. Violante and Prof. Y. Zhang. Without the sterling contributions of each of these individuals we could not have accomplished this work.

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APPENDIX C
PROCEEDINGS OF 9TH INTERNATIONAL CONFERENCE ON COLD
FUSION - MAY 2002
BEIJING, CHINA

PROGRESS TOWARDS REPLICATION

M. McKubre¹, F. Tanzella¹, P. Tripodi² and V. Violante²

Abstract

A collaborative effort has been established formally between SRI International in Menlo Park, California, and ENEA (Frascati) in Italy. In addition to providing a framework for an International replication effort, this collaboration is intended to focus the complementary skills of the two laboratories on carefully selected problems of Pd/D studies.

Researchers are attempting to test and demonstrate the cross-laboratory replicability of gas phase Pd/D₂ excess heat, helium and tritium observations. Similar facilities are being established in both countries to allow on-line determination of heat effects correlated with ⁴He, and ultimately ³He measurements from so called "Case" experiment involving the application of modest temperatures and D₂ gas pressures to a packed bed of palladium on carbon catalyst and other finely divided metallic catalyst and conductive support materials.

A second facet of this collaboration is the joint attempt to replicate the production of tritium in "Arata/Zhang" double-structured cathodes. Massive hollow palladium electrodes were manufactured at ENEA and sealed to contain palladium black within the enclosed void. Two electrodes were operated at SRI as electrolytic cathodes in LiOD electrolyte. On experiment termination these electrodes were vented and their contents examined for ⁴He, ³He and evidence of tritium. Results of initial gas analyses are presented here. Further analyses are underway and planned.

Experiments also are underway to study the metallurgical aspects controlling the loading of D into Pd, and to measure anomalous electrical characteristics and emissions from highly loaded specimens. The results of these scientific endeavors are reported in accompanying papers [1,2].

Introduction

A formal collaboration has been established between SRI International and ENEA (Frascati) with the primary purpose of demonstrating experiment portability and replicability in the emerging field of Condensed Matter Nuclear Science (CMNS). Experiments are being performed to:

- develop and understand the means to obtain, control and sustain high H/Pd and D/Pd stoichiometric ratio loading in small dimension Pd wires, foils and coatings,
- study, thin film electrolysis cells for possible evidence of x-ray generation, tritium evolution, ³He, ⁴He and higher mass isotope production,
- replicate observations of excess heat and ⁴He generation in Pd/C gas phase loading experiments first reported by Case [3],
- further replicate experiments of Professors Arata and Zhang [4,5] with particular attention to replicating tritium measurements in the void space of double structured cathodes by Clarke *et al* [6].

The evidence of solid state nuclear effects has been amply demonstrated in a number of observations initially considered to be "anomalous". The purpose of the discussion here is not to review the numerous published observations (but see, *e.g.*, [7]). Instead we simply identify the phenomena observed with high degree of certainty that are not subject to conventional (non-nuclear) interpretation.

The existence of an unaccounted heat source in addition to known electrical and mechanical input has been demonstrated in the Pd/D system, the Ni/H(D) system and possibly others. The extent of this excess heat exceeds conventional chemical energy by at least 2-3 orders of magnitude. As such, nuclear energy production is implicated. Numerous experiments have yielded ⁴He as a near-quantitative companion of this "excess" heat. Tritium has been measured, both directly and via its decay product ³He, as a substantial but sub-quantitative product. Higher mass isotopes have been generated in some instances at isotopic ratios differing significantly from natural. Energetic particles and radiation have been observed in the form of lattice gamma activation, X-ray production, charged particle and neutron emission [7].

There has been clear exposition of many of these phenomena at this conference, and these observations are not isolated or incoherent. Evolution of theory in the last 13 years has identified basic physical concepts and models to explain the set of observations without violating fundamental principals of physics. That is, we have exposed clearly nuclear solid state effects and developed rational bases to

understand the mechanisms and rates of the underlying nuclear physics processes. At this point it is appropriate to ask: what more might we do?

The principal issue remaining is one of reproducibility, or, more specifically, it's lack. We need to establish necessary and sufficient conditions to expose nuclear effects in condensed matter and it appears unlikely that we will establish this understanding by an empirical sifting of experimental conditions. Our collective intuition has taken us well beyond the boundaries of what initially was known, but the parameter space as yet unstudied is daunting. To establish reproducible experiments we require a fundamental understanding of the physical processes of condensed matter nuclear science.

We need a comprehensive and correct theory to assert applied physics, valid and simple diagnostics, and engineering control over key system parameters including the following:

- 1) Preparation of the host lattice and control of minor constituents. Some emphasis has been placed on reproducible preparation of the metallic host (e.g. Pd, Ni, Ti) for absorption of hydrogen isotope (D, H). It has long been recognized that minor alloying or surface adsorbed elements can critically impair the host's ability to absorb or retain hydrogen [8]. More recently recognized is the role of lattice defects [9] and of synergistic minority elements [10] in establishing conditions necessary for solid state nuclear effects.
- 2) Establishment of high loading, chemical potential or "fuel" activity. For the loading of deuterium into bulk, macroscopic palladium it was very quickly recognized that "heroic" loadings to very high deuterium chemical potentials were necessary to stimulate nuclear processes. Early work at IMRA (Japan) [11] and SRI [12] identified a threshold loading for deuterium in palladium wires.
- 3) Suitable system stimulation. At ICCF5 we presented [13] an empirical function to describe excess heat production in 1 mm Pd wires loaded electrochemically with D. In addition to the expected loading threshold dependence [11, 12] and previously observed threshold dependence on current [13], a term was included to account for the flux of D through the wire surface. Although subject to previous theoretical speculation [9], this was the first clear experimental indication that excess heat, and potentially other condensed matter nuclear effects, require stimulation or perturbation from an extreme thermodynamic equilibrium or steady state. Flux of material (or electrons) across a boundary appears to provide suitable stimulation and various other means have been attempted (acoustic, magnetic, thermal flux and other) with varying results. The effect of D flux in inducing nuclear effects has been amply demonstrated here [10].

Experimental

Our effort to establish reproducible experiments that can be used to prove the existence of a new phenomenon is based on: (i) fundamental theoretical understanding; (ii) clear and simple experiments; and (iii) reliable diagnostic measurements of unarguably nuclear effects.

As a suitable diagnostic we have initially focused attention on the measurement of light isotopes: ^3H , ^3He and ^4He . Considerable effort has been spent in establishing a mass spectrometric capability for this purpose. With contract support [14] we have purchased and installed (effective September 2001) a Micro-Mass 5400 noble gas mass spectrometer. This instrument has a magnetic sector mass analyzer with 90° extended geometry ion optics giving a dispersion length of 54 cm, allowing mass resolution of 0.001 dalton. Two detectors are employed to allow different ranges of sensitivity. A Faraday cup has sensitivity of ~ 3 ppb (parts per billion) for both ^3He and ^4He . The channeltron detector has sensitivity of ~ 2 pptr (parts per trillion) for ^4He and ~ 0.05 pptr for ^3He , allowing absolute resolution for ^4He of $\sim 1 \times 10^7$ atoms, and ^3He of $\sim 2.5 \times 10^5$ atoms. In general these atom sensitivities are limited by the instrument background and may be reduced by using different analysis methods.

We are interested in accurately quantifying [^4He] in the presence of D_2 and [^3He] in the presence of D_2 and H_2 . High resolution is required to resolve the $^4\text{He}^+$ mass peak from D_2^+ (0.025 dalton) and "ultra-high" resolution is required to separate $^3\text{He}^+$ from H^3+ and HD^+ (0.0?? dalton), to ensure that measured masses are not contributed by the hydrogen sample or background.

Sensitive measurement of ^3He is required for two reasons: (i) to determine the $^3\text{He}/^4\text{He}$ isotope ratio and thus ensure that one or other of these isotopes is produced in the experiment and does not derive from air ambient contamination; (ii) as a means of measuring [^3He] from $\partial[{}^3\text{He}]/\partial t$. The air ambient $^3\text{He}/^4\text{He}$ isotope ratio is 1.38×10^{-6} [15] thus ppb measurements of ^4He require pptr measurements of ^3He if the latter is not significantly enhanced. Similarly, with half-life over 12 years, the rate of production of ^3He on experimental time-scales is sufficiently small from modest amounts of tritium to require high sensitivity.

An initial phase of experiments was performed using double structured (DS) cathodes prepared at ENEA (Frascati). A previous replication attempt at SRI resulting in copious tritium production (measured

as ^3He by Clarke, Oliver *et. al.*, [6]), was performed with cathodes prepared by Professors Arata and Zhang. An attempted replication without direct benefit of their specialized knowledge and skill, while ambitious, was felt to be a better test of experiment transferability. This effort was also designed to teach us about metallurgical, machining and welding issues related to the preparation of Arata/Zhang DS cathodes:

Two cathodes designated EAP1 and EPA2 were prepared with the following dimensions:

EAP1: 60 mm long x 12.5 mm diameter; mass = 78g; internal cavity; 45 mm long x 4.4 mm diameter.

Pd powder mass = 0.5 g.

EAP2: 70 mm long x 13.5 mm diameter, mass = ?? g; internal cavity 45mm long x 4.4 mm diameter.

Pd powder mass = 0.6 g.

The Pd powder used to fill the internal cavity was 0.25-0.55 μm , 99.95% (metals xxxx), from Alfa Aesar, Ward Hill, MA. The palladium vessels were filled with Pd black inside an argon dry box, and then enclosed within a sealed vessel (under argon) for transfer to the electron beam welder. The Pd black filled cathodes were evacuated in the welding device at $\sim 5 \times 10^{-3}$ torr for one hour before welding closed. Ultrasound analysis revealed that the weld thickness was not less than 4mm.

The two cathodes were electrolyzed at SRI in 0.1 M LiOD roughly following Arata/Zhang current protocols [4,5]. Table 1 lists the current-time history of electrolysis. The final 35 days of anodic treatment was intended to reduce the D_2 gas pressure in the internal cavity allowing for subsequent safe handling of the electrodes.

Table 1 Current Protocol for DS Cathodes

Current	5.0	5.5	6.0	6.5	1.0	6.0	6.5	7.0	-0.01	-0.03	Amps
EAP1	1031	216	241	100	504	66	28	503	26	814	Hours
EAP2	338	216	241	100	504	66	28	503	26	814	Hours

Following anodic stripping the electrodes were transferred to a device constructed at ENEA, capable of puncturing the cathode internal cavity and releasing whatever gas remained into an enclosing, evacuated chamber. By means of an attached pressure transducer it was possible to observe and quantify this gas release. Two gas samples were obtained from each electrode: the first at ambient temperature (25°C) immediately following void puncture intended to reflect the equilibrium condition of gas in the internal cavity; the second after heating of the extraction vessel to 125°C for 15 hours and 2 hours at 250°C, to sample hydrogen isotopes absorbed in the palladium metal bulk and black.

Vittorio – do we have a good enough picture of the extractor to insert as Figure 1?

Mike - (series of pictures showing puncturing hardware?)

Results

Figure 3? shows cathode EAP1 after removal from the electrochemical cell and puncturing. The surface of the electrode exhibited considerable metallurgical effect and damage resulting from deuterium loading. At least one axial crack appears to penetrate to the internal cavity effectively preventing detection of any novel gases in the vessel space. The second cathode, EAP2, showed evidence of the surface expression of a much smaller axial crack (Figure 4?). Metallurgical examination of both cathodes is planned to measure the depth of crack penetration.

Table 2 presents the results of gas sampling and initial analyses. Samples were collected in the 250 cc volume of the pre-evacuated bellows puncturing device. Sample EAP1(A) thus was $\sim 1/4\text{atm}$ primarily of adsorbed D_2O and absorbed D_2 . As indicated in the last column of Table 2, the helium in this sample analyzes at the air ambient isotope ratio presumably from air-derived helium adsorbed on the high surface area Pd black.

Heating EAP1 to yield sample (B) resulted in a substantial de-sorption of D_2 from the Pd bulk and black, an associated pressure increase, and decrease in helium concentration. Because of the measurement uncertainty in $[\text{He}]$, this sample also displays no anomaly in helium isotope ratio.

Table 2
Preliminary Gas Determinations

Sample	Temperature	Volume cc atm	³ He pptr	⁴ He ppb	³ He/ ⁴ He	R _A
			±0.05	±1.0		
EAP1 A	25°C	63	0.18	138	1.3 E-06	1.0
EAP1 B	250°C	275	0.05	25	2.0 E-06	1.5
EAP2 A	25°C	16	0.22	76	2.9 E-0	2.2
EAP2 B	250°C	1000	<0.05	3		

$$R_A = 1.38 \times 10^{-6} [15]$$

Sample EAP2(A) yielded a potentially more interesting result. The initial sample while only ~1/4 of the volume of EAP1(A), shows a higher concentration of ³He and a lower concentration of ⁴He. The helium isotope composition of this sample is significantly different from the ambient air background, suggesting that at least some of the ³He was produced during the course of the experiment.

Heating EAP2 to 250°C stimulated a very significant desorption of D₂ and a pressure rise to more than 4 atm. At this dilution the uncertainties in measurement made it not possible for us to calculate the helium isotope ratio.

Discussion

The results described above are both preliminary and incomplete; they are discussed here as the results of a "work in progress". Within the next few months we plan to make a thorough analysis of the gas contents of the four samples, and repeated analyses of ³He (and ⁴He) in an attempt to quantify any ³He and potentially ³H present in these samples.

A metallurgical examination is already underway at ENEA to determine the reasons for, and consequences of, electrode mechanical failure. These electrodes will also be sectioned and analyzed to determine the ³He (and ⁴He) content of the bulk metal phase and of the Pd black. Since the diffusion range of helium in Pd is very limited while that of hydrogen is long, careful measurement of ³He provides a very sensitive test of the past presence of tritium. The capacity to measure helium in metal samples will be established in the next phase of collaborative activity.

A new series of experiments also is planned. The ³He/⁴He isotope ratio measured at 2.2 times the air ambient background for sample EAP2(A) in the present series must be taken as significant and encouraging. This number is, however, substantially less than was measured [6] in our previous attempt to replicate the results of Professors Arata and Zhang. With improved measurement capability, fabrication experience gained, and expanded collaborative support, we anticipate making substantial further progress towards replication.

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