REPORT ON ELECTROLYSIS EXPERIMENTS AT ENERGETICS TECHNOLOGIES

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Introduction

The primary objectives of the Energetics Technologies experimental program are:

(1) Improvement of reproducibility of excess heat generation
(2) Amplification of power and energy gain

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The program focuses on electrolytic cells driven by I. Dardik's SuperWaves

Several experimental approaches are being pursued:

- Optimization of Super Wave
- Modify cathode surfaces by special annealing-etching procedures jointly developed with ENEA (Frascati) and the University of Rome
- Modify cathode surfaces by SuperWaves glow discharge etching
- Develop new cathodes
- Electrolysis with ultrasonic wave excitation

Experimental results in each of these research areas are reported along with results from metallurgical and the X-ray analysis of the cathodes.

Successful replications of Energetics heat production was accomplished at SRI and at ENEA (Frascati); results will be presented separately.

ET ELECTROLYTIC CELL



EG is inside a Teflon beaker that is placed inside an isoperibolic calorimeter that is placed inside a thermo-stated water bath (3 cells per bath)

Typical EC cathode



Pd foil -- 50 µm

Annealed at 870°C in vacuum for 1h **Etched:**

- 1) in Nitric Acid 65-67% 1 min
- 2) in Aqua Regia 1:1 water solution 1 min**Rinsed:**
- 1) D_2O four times
- 2) Ethanol 95% twice
- 3) Ethanol Absolute once

Dried:

in vacuum at ambient temperature for 24 h

Electrolyte:

0.1M LiOD in D_2O of low tritium content; 230 ml

SuperWaves formation principles

 $\mathbf{F}_0(t) = A_0 \sin^2(\omega_0 \mathbf{t})$



 $F_1(t) = A_0 \sin^2(\omega_0 t)(1 + A_1 \sin^2(\omega_1 t))$







 $F_{3}(t) = A_{0} \sin^{2}(\omega_{0} t) [1 + A_{1} \sin^{2}(\omega_{1} t)(1 + A_{2} \sin^{2}(\omega_{2} t)(1 + A_{3} \sin^{2}(\omega_{3} t)))]$

Typical SuperWave



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Reproducibility

		Excess Heat	
Number of experiments by category	Nº of exp.	Nº of exp.	% of giving EH
50 microns Pd foil - regular*	98	16	16
50 microns Pd foil* - optically tested**	20	3	15
Glow discharge etching	21	10	50
PdNi alloy	16	3	19
Multilayer ^{***} Pd/Me(MeO)/Pd [Me = Metal]	25	6	24
Single Wall Carbon Nano Tubes Pd composite***	59	11	19
Total	253	49	19
US excited electrolysis	29	17	59

*samples prepared by Dr. V.Violante et al

**samples tested by Prof. C.Cibilia et al

***samples prepared by Dr. A.Lipson et al.

Target - Excess power like in Exp. # 64a



Excess Power of up to 34 watts; Average ~20 watts for 17 h

Power

SEM-EDS analysis of foil # 64 vs. 63

Surface of Pd foil after rolling and annealing at 870°C

foil #64 (EH) many "black spots" foil #63 (no EH)





View of typical black spot on # 64 and its composition



SEM-EDS

Elemen	t Wt %	At %
C	35.77	52.48
0	26.19	28.84
Na	4.92	3.77
Al	0.43	0.28
Si	1.05	0.66
Pt	0.39	0.04
S	1.44	0.79
Cl	10.68	5.31
Pd	2.55	0.42
K	11.07	4.99
Ca	5.52	2.43
Total	100.00	100.00



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Cathode surface modification

An attempt was made to create cathode surface similar to that of foil #64:

- embed "black spots" on the surface using special rolling procedure
- anneal under similar condition
- find the right etching procedure
- specify the surface properties by an optical method

Foil type	Nº of experiments	№ of giving EH	% of giving EH
50 microns Pd foil - regular	98	16	16
50 microns Pd foil - optically screened	20	3	15

• Unfortunately, no difference has been found!

• Power gain is relatively low (10-15%)

Glow Discharge etching – Statistics

First Series

Gas	<pre># of significant* experiments</pre>	# giving EH	% of Power gain	R/R ₀
Argon	1	1	10-20	peak
Argon-Oxygen	0	0	n/a	n/a
Deuterium- Helium	5	2	17-20	1.93-1.32
Deuterium	5	1	25-75	1.87-1.26
Helium	2	1	8-10	1.88-1.62

Total

13

5

38%

Recent Series

Gas	# of significant [*] experiments	# giving EH	% of Power gain	R/R ₀
Deuterium	8	5	10-25	1.95-1.22
Total	8	5	62.5 %	

Total

*Experiments without Glow Discharge induced defects.

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Glow Discharge etching – tremendous surface restructuring



Exp.#141 after deuterium GD etching



Commercial alloy 5Ni95Pd



High loading for NiPd alloy



*) F.A. Lewis, I.Lewis and S.G.McKee "Correlation of the relationships between hydrogen content, hydrogen chemical potential and electrical resistivity for palladium alloy-hydrogen systems: possible catastrophe theory representation of the relationships". Journal of the Less-Common Metals, 101(1984) 503-521

Kinetics of deloading outside cell



Estimated D/Pd atom ratio as a function of time after shut down of the electrolysis experiment: 1-Pd-5Ni alloy (Exp. #449), 2-Pd.

Exp.#449 – 5NiPd alloy



Time

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Multilayer structure

20 Pd substrate 50 μ m foil coated with 5-7 layers of PdO_x. Ni or Re alternating with 6-8 layers of Pd. Top layer is always Pd.

Exp.#375 – multilayer with Rhenium



Composite Pd/Carbon Nano Tubes (SWCNT)/Pd



Single wall Carbon Nano tubes (SWCNT)

Series #	1	2	3	4	6
Manufacturer	CNI (old)	CNI (old)	Helix	Alfa Aesar	CNI (new)
Diameter, nm	1.1-1.2	1.1-1.2	1.1-1.2	1.1-1.2	1.1-1.2
Length, µm	1-2	1-2	0.5-40	10-20	1-2
Purity, %	95	95	90	>96	95
Adhesion	bad	good	very bad	excellent	excellent
Number of experiments	4	11	13	16(tested 12)	16(tested 8)
EH giving experiments	3	4	3	0	0
% giving EH	75	36	23	0	0

SWCNT opened by special etching procedure

Exp.#357 – Carbon nano tubes from 1st series



Electrolysis with ultra-sound excitation



Schematic view

Electrolytic cell for ultra-sound excitation setup A & B



Electrolysis with ultra-sound excitation - Statistics

Setup type	Year	Calori meter	Thermo- stated	# of exp.	# giving EH	% giving EH
Α	2006	no	no	6	4	67
	2006	yes	no	13	6	46
В	2007	yes	no	5	5	100
	2006	yes	yes	2	0	0
C	2007	yes	yes	3	2	67
		Total		29	17	59

Observations:

- extremely high loadings ($R/R_0 = 1.47-1.43$) applying very low current densities (not higher than 10 mA/cm2).
- ability to stay loaded in open air during 1-12 weeks (stable β phase)

Setup with flow calorimeter (Setup B)



Exp.#ETUS1-15; calorimeter not thermo-stated (setup B)



26 days of run

Thermo-stated E-US cell with flow calorimeter (Setup C)



Exp.#ETUS3-5 in Setup C – "heat after death"



Experiment ETUS3-05



Loading Path

The most recent experiment ETUS3-6; Setup C



32 days of run

Exp.#ETUS1-17; calorimeter not thermo-stated (setup B)



"Heat after death" – heat generation during 11 h after termination of loading

ETUS3-6 surface after experiment SEM image magnification x1000



Non-metallic contaminations on the cathode surface

Comparison of ETUS3-6 and ETE-64 (no US) surface after experiment – both gave EH SEM image magnification x4000 and x8000



³Strongly pitted surface

Plastic deformation

Conclusions

- Highest reproducibility is obtained with ultra-sound excitation. Need to optimize application of US.
- First series of Pd/SWCNT/Pd sandwich targets had high reproducibility. Need to find comparable SWCNT.
- Also promising are Pd foils that underwent etching using glow-discharge with deuterium
- Significant "Heat-after-Death" was obtained in 2 experiments with ultra-sound excitation and with Pd/SWCNT/Pd sandwich targets. <u>It is suggested that</u> <u>SuperWaves exposure could create a long-term effect</u> (memory) in the cathode in the form of spontaneous lattice oscillations that result in the heat release.

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