Evolution and Progress in Material Science for Studying the Fleischmann and Pons Effect

V. Violante\textsuperscript{1}, F. Sarto\textsuperscript{1}, E. Castagna\textsuperscript{1}, S. Lecci\textsuperscript{1}, M. Sansovini\textsuperscript{1}, A. Torre\textsuperscript{1}, G. Hubler\textsuperscript{2}, D. Knies\textsuperscript{2}, K. Grabowski\textsuperscript{2}, M. McKubre\textsuperscript{3}, F. Tanzella\textsuperscript{3}, C. Sibilia\textsuperscript{4}, Z. Del Prete\textsuperscript{4}, T. Zilov\textsuperscript{5}

(1) ENEA CR Frascati  
(2) NRL Washington DC  
(3) SRI International Menlo Park  
(4) University La Sapienza Rome  
(5) Energetics Technologies Omer

Contributors:

M. Bertolotti  
L. Bettinali  
L. Capobianco  
L. D’Aulerio  
D. Dominguez  
D. Kidwell  
D. Lecci  
R. Livoti  
M. Mastrangelo  
G. Miley  
S. Moretti

\textit{ICCF15 Rome 5-9 October 2009}
Reproducibility of Loading and of Excess of Power as a Material Science Problem

Threshold effect McKubre (SRI) e Kunimatzu (IMRA Japan) (1992)

ICCF15 Rome 5-9 October 2009
Since 1996 in ENEA material status was identified to be responsible of the loading

1) Material science study to increase both reproducibility and signals

2) Calorimetric experiments conceived to have an appropriate signal/noise ratio

3) Theoretical work to identify methods to trigger the effect
H or D entering Pd lattice produces an elongation of the lattice parameter

Loading is not homogeneous and concentration gradients produce a stress field

The stress field modify the chemical potential of hydrogen dissolving into the lattice

\[ \mu_H^* = \mu_H - V_s \sigma_h \]

(V solute molar volume, \( \sigma_h \) trace of the stress tensor)

In presence of a stress field the flux equation reads:

\[ \bar{J} = -D(\nabla c - \frac{c \nabla}{RT} \nabla \sigma) \]

A metallurgy able to minimize the concentration gradients is required
A process has been defined to have a metallurgy enhancing mass transfer to reduce D concentration gradients and stress.

Cold rolled and annealed at 850 °C Pd foil

Normalized Resistance evolution

(Please see also S1_04)
Theoretical Frame

A theoretical model was developed that predicts intense E.M. field caused by charge density oscillations at the interface leading to:

1) Deuterons interaction in Pd Lattice
2) Modified decay channel for D+D reaction


(Please see also S10_04)
Field Effect on the Decay Channel

The interaction Hamiltonian is calculated by using the same values of the field producing the close approach between deuterons

\[ \hat{H}_I = \frac{e}{m} \hat{p} \hat{A} \]

Within the single particle approximation the probability amplitude results to be

\[ |a_{fi}|^2 \approx \gamma^2 [3 - 2(\cos(\omega_0 T) + \sin(\omega_0 T))] \]

\[ \gamma = f(\langle 1|H_I|0 \rangle) \]

leading to

\[ \frac{\sigma^{*}_{(D,n)}}{\sigma_{st}} < 10^{-5} \quad (if \quad A_0 = E_0 / \Omega > 10^{-2}) \]
2002-2004 Experimental Campaign

Calorimetry

Flow Calorimeter

ICCF15 Rome 5-9 October 2009
Closed Electrochemical Cell

\[ P_{\text{IN}} = V \times I \quad P_{\text{OUT}} = W \cdot C_p \cdot (T_{\text{OUT}} - T_{\text{IN}}) \]

\[ P_{\text{EX}} = \frac{P_{\text{OUT}}}{\text{Efficiency}} - P_{\text{IN}} \]
Light water experiment (typical calibration)

Input and output power and energy with H$_2$O. Calorimeter efficiency = 97.5%.

ICCF15 Rome 5-9 October 2009
Excess of power and energy gain C3 (R/\(R_0\)=1.8, I=25 mA, V=4.1 Volt).

ICTF15 Rome 5-9 October 2009
Laser Triggering Experiment: Calorimetric Results

Excess of power and energy gain obtained by HeNe laser triggering

ICCf15 Rome 5-9 October 2009
The concentration threshold is always overcome.

Observed power excesses are 5 over a total number of 20 experiments.

Samples qualified by ENEA showed similar results at other Institutes.

The effect is observed with deuterium but not with Hydrogen.

The active electrodes showed similar characteristics.
L17 experiment. Excess of power: the output power becomes 5 times larger than the input.

2005-2007: USA-Italy Revisions Projects
ENEA-MSE and SRI Experiments

Research Projects having Government support are carried out both in Italy and US to verify the existance of the effect

(Please see also S1_03)

Electrolyte temperature

Electrolyte temperature increasing during the excess

ICCF15 Rome 5-9 October 2009
## Transferred Reproducibility Synoptic

<table>
<thead>
<tr>
<th>Electrode</th>
<th>Original Lot</th>
<th>Excess %</th>
<th>MJ/Mol</th>
</tr>
</thead>
<tbody>
<tr>
<td>ETI 17</td>
<td>L1</td>
<td>20 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 011</td>
<td>L5(2)</td>
<td>25-60 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 35 7a</td>
<td>L17</td>
<td>10 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 35 8a</td>
<td>L17</td>
<td>15 (SRI)</td>
<td>11</td>
</tr>
<tr>
<td>ETI 43 7a</td>
<td>L14(2)</td>
<td>80 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 051 7</td>
<td>L25B(1)</td>
<td>12 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 051 8</td>
<td>L25A(1)</td>
<td>NO (SRI)</td>
<td>23 (ENEA)</td>
</tr>
<tr>
<td>ETI 051 8</td>
<td>L18</td>
<td>NO (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 056 7</td>
<td>L24F</td>
<td>25 (SRI)</td>
<td>19</td>
</tr>
<tr>
<td>ETI 056 8</td>
<td>L24D</td>
<td>NO (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 056 9</td>
<td>L25B(2)</td>
<td>5 - 6 (SRI)</td>
<td>1</td>
</tr>
<tr>
<td>ETI 58 9</td>
<td>L25A</td>
<td>200 (SRI)</td>
<td></td>
</tr>
<tr>
<td>ETI 61 7</td>
<td>L25B(1)</td>
<td>50 (SRI)</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L26</td>
<td>69 ENEA</td>
<td></td>
</tr>
</tbody>
</table>

Reproducibility was 60% in ENEA and 70% at SRI

*ICCF15 Rome 5-9 October 2009*
The focus was moved on other features of the samples correlated with the occurrence of the effect.

Other conditions are required to increase the excess of power reproducibility.

A different behavior was observed with Pd cathodes loaded above the threshold D/Pd = 0.9:

1) High power gain during the excess.
2) Low power gain during excess.
3) No excess.
Differences in Two Lots from the Same Producer

Spectrum of Contaminants in the Rough Materials (qualitative study).

Both lots were 99.95%

Reproducibility reduced under 20% with Lot II

I LOT: excess of power > 100%
II LOT: excess of power < 20%

Reproducibility > 60%
Reproducibility < 20%
Contaminants Effects

Contaminants may act on:
- Grain size
- Crystal orientation
- Grain boundary

Same treatments

Lot 1
Lot 2
Crystal Orientation (NRL Study)

X Ray Diffraction : Crystals orientation after metallurgical treatment

a) **LOT 1** is well aligned $<100>$, little or no $<110>$

b) **LOT 2** samples is a mix of $<100>$ and $<110>$  (Please see also S1_04)

c) A third lot giving a different spectrum of contaminants undergone to the same treatment revealed mostly $<110>$ very little $<100>$

*No excess was produced by testing cathodes realized with this third lot.*

Crystal orientation and surface morphology are further conditions for having the excess

Power spectral density has been selected as merit figure to identify the status of the surface *(please see S10_O1)*:

*ICCF15 Rome 5-9 October 2009*
Typical Surface Morphology (after Etching) giving Excess of Heat

(Please see also P_41)
PSDF of sample #64 producing 3500% excess of heat.

PSDF of sample L25 producing up to 250% excess of heat.

PSDF of sample ET-UTS-05 producing 25% excess of heat under electrolysis.

PSDF of a sample not producing excess of heat

Surface morphology results to be a third condition.
The experimental evidences leaded to produce a material having characteristics close to the ones described.

A lot of Pd having a spectrum of contaminants approaching the lot 1 one was undergone to the treatment leading to: dominant <100> orientation and an appropriate metallurgy.
After performing chemical etching the surface achieved a status identified by the PSDF calculated from the surface image given by the Atomic Force Microscopy.

Even if of reduced amplitudes the peaks structure belonging to samples producing excess of heat is clearly recognized. A small excess is expected.
Evolution of the input and output power during experiment L-46:
Excess up to 12% was observed and the amplitude is again
proportional to the PSDF peaks amplitude.

This is giving an additional evidence that a proper surface morphology is a
third condition for having the excess of power.
Some Recent Results with "Designed Materials"

(L72) Please see also S1_05

ICCF15 Rome 5-9 October 2009
Conclusions

- Results from designed and rebuilt materials
- Important result for understanding the effect, even if the amplitude of the signals was not extraordinary
- Amplitude and shape of the PSDF peaks is proportional to the amplitude of the observed thermal signals
- The ability to produce a sample giving the excess is only at the early stage but the walk has been initiated
- Energy gains above $10^1$ eV/atom have been observed in the experiments
- A large amount of produced energy per atom it’s difficult to be interpreted as chemical effect
- A big challenge for material science and a great interdisciplinary task
“The energy is the external line of our soul”

From Marriage between Haven and Hell

William Blake, 1799