ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Rapp. Naz.: Francesco CELANI		

Rappresentante nazionale: Francesco CELANI
Struttura di appartenenza: LNF
Posizione nell'I.N.F.N.:

INFORMAZIONI GENERALI	
Linea di ricerca	Reazioni nucleari a bassissima energia in matrici metalliche con composti iper−deuterati. Effetto citotossico D2O normale ed irradiata.
Laboratorio ove si raccolgono i dati	L.N.F.; Pirelli Labs. (Milano); ENEA Casaccia e Frascati; CSM (Castel Romano); MHI (Yokohama, Giappone), Univ. /INFN Lecce; Univ. /INFN Perugia;
Sigla dello esperimento assegnata dal laboratorio	FREETHAI
Acceleratore usato	
Fascio (sigla e caratteristiche)	
Processo fisico studiato	Reazioni fusione D−D in Pd, struttura multilayer di Sr −Hg e/o Th−Hg con tecniche elettrolitiche non convenzionali (LNF). Eccitazione laser di film nanometrici di Pd −H o D (LE). Effetto citotossico D2O, normale/ irradiata (LNF), identificazione/misura ROS radioindotti (PG).
Apparato strumentale utilizzato	Celle elettrolitiche vetro−PTFE; calorimetri a flusso; misuratori di T con LSC (ENEA Frascati); ICP−MS e OES (CSM); SEM (PIRELLI); minicrogiuoli (ORIM); laser pulsato (LE); apparati microbiologia (ENEA Casaccia); spettroscopia ottica nel IR−VIS−UV (PG), IR da DAFNE (LNF).
Sezioni partecipanti all'esperimento	L.N.F., Lecce, Perugia.
Istituzioni esterne all'Ente partecipanti	Pirelli Labs(*); ENEA Frascati e Casaccia; ORIM(*); EURESYS(*); STMicroelectronics(*), MHI. N.B. (*) = industria

Durata esperimento	3 anni

Mod EC. 1 (a cura del responsabile nazionale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Vincenzo Nassisi		

Struttura
LE

PREVENTIVO LOCALE DI SPESA PER L'ANNO 2005

In KEuro

VOCI DI SPESA	***DESCRIZIONE DELLA SPESA***	***IMPORTI*** ***Parziali*** ***SJ***		***Totale Compet.***	***di cui SJ***	A cura della Comm.ne Scientifica Nazionale
Viaggi e missioni – Interno	Collaborazione con i LNF. Collaborazione con l'Università di Bologna, Perugia e Siena. Collaborazione con l'ENEA Frascati.	6,0		***6,0***		
Viaggi e missioni – Estero	Collaborazione con il CERN. Collaborazione Laboratori giapponesi, prof. Arata, Osaka	6,0		***6,0***		
Materiale Consumo	Spese per funzionamento laser. Materiale elettrico ed ottico. Spese per analisi SEM, Microanalisi e spettrometria di massa.	7,0		***7,0***		
Trasp. e facch.						
Spese Calcolo	Consorzio: / Ore CPU: / Spazio Disco: / Cassette: / Altro:					
Affitti e manutenz. apparecchiat.						
Materiale inventariabile	Laser in CW da 100 mW operante nel visibile o IR.	4,0		***4,0***		
Costruzione Apparati						
	Totale			***23,0***	***di cui SJ 0,0***	

Sono previsti interventi e/o impiantistica che ricadono sotto la disciplina della legge Merloni ? ☐

Breve descrizione dell'intervento:

Mod EC./EN. 2 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Francesco CELANI		

Struttura
LNF

PREVENTIVO LOCALE DI SPESA PER L'ANNO 2005

In KEuro

VOCI DI SPESA		***DESCRIZIONE DELLA SPESA***	***IMPORTI*** ***Parziali*** ***SJ***		***Totale Compet.***	***di cui SJ***	A cura della Comm.ne Scientifica Nazionale
Viaggi e missioni	Interno	Spese missioni interno (Lecce, Milano, Perugia), Misure presso Casaccia, CSM, congressi e workshop.	5,0		***5,0***		
	Estero	JCF6 (Sapporo − Giappone) ICCF12 (Yokohama − Giappone) (due persone)	4,0 6,0		***10,0***		
Materiale Consumo		Reagenti chimici deuterati ed ultrapuri (>99.99%) Vetreria di laboratorio (alcune su nostre specifiche) e reagenti per microbiologia Contributo spese misure ICP−MS e OES presso CSM Parti di ricambio per distillatore ROTAVAPOR Costruzione elettronica made−home	10,0 6,0 4,0 2,0 2,0		***24,0***		
Trasp. e facch.							
Spese Calcolo		Consorzio / Ore CPU / Spazio Disco / Cassette / Altro					
Affitti e manutenz. apparecchiat.							
Materiale inventariabile		BOP (KEPCO) da 200V, 1A BOP (KEPCO) 100V, 2A Generatore di funzioni arbitrarie 0,1 mHz 16 MHz modello TGA 1244	5,0 5,0 6,0		***16,0***		
Costruzione Apparati		Costruzione di nuovo calorimetro, in quarzo e compositi, per misure IR	20,0		***20,0***		
				Totale	***75,0***	***di cui SJ 0,0***	

Sono previsti interventi e/o impiantistica che ricadono sotto la disciplina della legge Merloni ? ☐

Breve descrizione dell'intervento:

Mod EC./EN. 2 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Onori		

Struttura
PG

PREVENTIVO LOCALE DI SPESA PER L'ANNO 2005

In KEuro

VOCI DI SPESA		***DESCRIZIONE DELLA SPESA***	***IMPORTI*** ***Parziali*** ***SJ***		***Totale Compet.***	***di cui SJ***	A cura della Comm.ne Scientifica Nazionale
Viaggi e missioni	Interno	Partecipazione a congressi, misure a Frascati con luce IR da DAPHNE, riunioni della collaborazione.	5,0		***5,0***		
	Estero						
Materiale Consumo		Prodotti biochimici, acqua deuterata, spese di esercizio della strumentazione (azoto, lampade, spettrofotometro, cuvette, etc.), vetreria.	8,0		***8,0***		
Trasp. e facch.							
Spese Calcolo		Consorzio / Ore CPU / Spazio Disco / Cassette / Altro					
Affitti e manutenz. apparecchiat.							
Materiale inventariabile		Termocriostato a circolazione.	4,0		***4,0***		
Costruzione Apparati							
		Totale			***17,0***	***di cui SJ 0,0***	

Sono previsti interventi e/o impiantistica che ricadono sotto la disciplina della legge Merloni ? ☐

Breve descrizione dell'intervento:

Mod EC./EN. 2 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Vincenzo Nassisi		

Struttura
LE

ALLEGATO MODELLO EC2

Attività svolta:
Le proprietà di molti metalli di transizione idrogenati o deuterati, sono le variazioni morfologiche e la formazione di agglomerati contenenti differenti elementi.
Per verificare sperimentalmente questo risultato si è controllato l'andamento temporale della pressione pressione del gas utilizzando una minicamera dal volume di circa 8 cm3.
All'interno della minicamera si è posto, inizialmente, un disco di Pd di spessore pari a 0.5 mm, diametro 8 mm e purezza pari a 99.99% ad una pressione iniziale di H2 di 1.17±0.05 bar. Si è rilevata nel tempo il valore della pressione, per mezzo di un manometro a membrana, e la temperatura per mezzo di una sonda Pt100. La pressione della camera diminuisce drasticamente nelle prime 20 ore di assorbimento, indicando un repentino assorbimento del gas: questo è dovuto probabilmente al fatto che, inizialmente, la diffusività del gas può essere considerata approssimativamente costante in tutto il suo volume. Successivamente vi è una decrescita più moderata dipesa dal fatto che la diffusività è legata, in questo caso, alla concentrazione del gas all'interno del bulk, come pure al volume del campione. per campioni più sottili, il tempo di assorbimento è più piccolo.
Il comportamento della pressione è stato fittato con un funzione somma di due esponenziali:Aexp(−t/tau)+Bexp(−t/beta)
Attività in corso:
Attualmente sono in corso misure su film preparati dalla STMicroelectoncs

Attività prevista:
Stimolazione dei campioni idrogenati e deuterati mediante fasci laser impulsati e continui (100mW). In questo modo si incrementano gli effetti dei processi collettivi, possibili responsabili dei fenomeni.
Analisi SEM,SIMS e ICP.

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Vincenzo Nassisi		

Struttura
LE

ALLEGATO MODELLO EC2

Measurements of new elements in Pd-H_2 thin films

A. Lorusso, V. Nassisi, E. Filippo*, M. Di Giulio*, D. Manno*, G. Buccolieri, F. Celani**

Laboratory of Applied Electronics, Department of Physics, University of LecceI.N.F.N. Lecce, C.P. 193, 73100 Lecce-I.
**Department of Material Science, University of Lecce*
I.N.F.N. Lecce
*** I.N.F.N.-LNF, Frascati (Roma)*
Tel. 0039 832 297495 Fax. 0039 832 297482 E-Mail: Nassisi@le.infn.it

Abstract

The effects of H_2 absorption in Pd thin films are investigated. 10 nm thickness Pd layers have been deposited by thermal evaporation on Si substrates. Pairs of Pd/Si samples have been loaded in a 250 cm^3 stainless steel cylindrical chambers, which were evacuated down to a 10^{-5} mbar background pressure and then filled with high purity H_2 at a pressure of 5 bar. Only one sample of each chamber has been irradiated for one day before the opening by an excimer laser (?=308 nm, t_{FWHM}=20 ns, 500 shots) at a fluence of 20 mJ/cm^2, low enough to avoid film ablation. The chamber have been opened after 1, 2, 4, 8, 16, 32, 93, 109 days. The samples have been analysed by Scanning Electron Microscopy (SEM), Energy Dispersive Spectrometry (EDS) and Inductive Coupling Plasma-Optical Emission Spectroscopy (ICP-OES). Several important effects have been noticed; 1) the film surface exhibited cracks just after 4 days of soaking in H_2 atmosphere, irrespective of laser treatment; 2) after 8 days polycrystalline grains with dimensions of 1-2 µm were formed; 3) chemical composition analysis of grains exhibited elements as Al, Ca, S, Fe, Ni; 4) laser irradiation induced the increasing of grain concentration and grain dimensions.

Introduction

The controversial phenomenon called “Cold Fusion” (CF), “Low Energy Nuclear Reactions” (LERN) or “Chemically Assisted Nuclear Reactions” (CANR) started in 1989 with the famous discovery of Pons and Fleishmann(1).

Since this work was published, a lot of experiments were performed in many laboratories: the first method to load palladium with deuterium, used electrolysis cells(1). Next, the gas loading method became very utilised in many laboratories due to its low grade of contamination(2, 3). The first experiments used deuterium as load gas because of its large probability to get nuclear reaction but in same time loading by hydrogen gas were performed obtaining again interesting results.

Recently, Iwamura et al. at Mitsubishi Heavy Industry in Japan have evidenced the presence of new elements due to transmutation processes in particular systems. They deposited a thin layer of palladium (40 nm) on a layer of CaO, which had been deposited on a thick palladium substrate. To load the system deuterium gas was utilised. When the gas molecules diffused through the sandwich, several nuclear reaction effects were observed, including excess energy(4).

In this work we have utilized the gas loading method in Pd/Si systems and we have also observed the morphological modification of the samples on different days of treatment and the presence of different new elements from the Pd which they could be generated by the probable nuclear reactions that are primed through a strong loading of H_2 in the lattice of the samples. Moreover applying a UV laser light, the effects of gas loading marked the experimental results particularly in of grain concentration and grain dimensions(5).

Experimental Set- up

We have realized, by thermal evaporation technique, Pd thin films deposited on Si (100) wafers of about 1 cm^2 in surface. We chose films of 110 nm thick because they demonstrated to be very efficient in new elements production(6). They were placed in cylindrical stainless steel chambers of about 250 cm^3 in volume. Fig. 1 shows the experimental apparatus and Fig.2 shows a photo of a chamber utilized for the experiment.

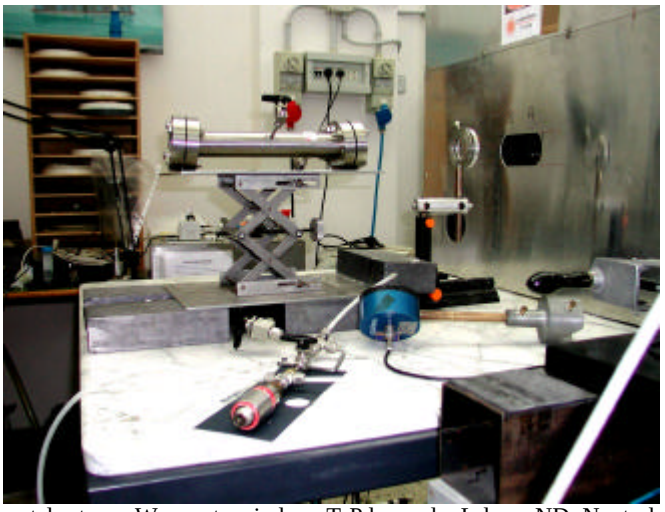


Fig.1- Experimental set-up- W: quartz window; T-Pd sample; L-lens; ND: Neutral density filters-

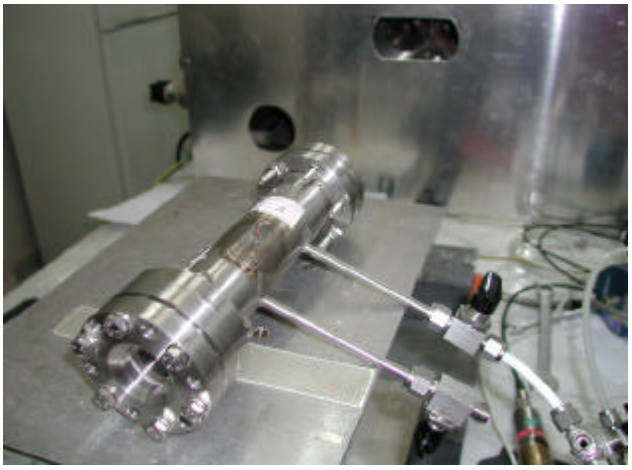


Fig.2- The chamber processing.

The chambers were equipped by two quartz windows in order to allow the laser beam to go inside until the samples. To avoid contaminations, the chambers were carefully cleaned with acetone and dried in nitrogen flux before the experiment.

Subsequently a pairs of Pd/Si samples have been placed to the inside the chambers and these filled with H_2 gas to maximum pressure of the exercise of 5 bar. The samples were loaded for 1, 2, 4, 8, 16, 32, 93, 109 days and only one sample from each chamber was irradiated by an XeCl home-made excimer laser (?=308 nm, 20 ns) one day before opening the chamber, with 500 shots.

After to have ended the treatment, the samples were analysed by a Scanning Electron Microscopy (SEM) and an EDX analyser. Some of these samples were analysed by ICP-OES technique too.

Results and Discussion

The chamber have been opened after to have executed the above protocol and the samples observed by the SEM. Different behaviours were observed for samples kept in air, laser treated and no-treated: thus, for samples kept in air, the film surface was smooth, with mirror like aspect. Instead, the samples treated and no-treated by laser showed morphological modification of the films just after 4 processing days. The morphological modifications consisted in formation of cracks just after 4 days of

soaking in H_2 atmosphere and in polycrystalline grains with dimension of 1-2 μm, after 8 treatment days. Fig.3 showes an example of grains after 10 days of gas loading.

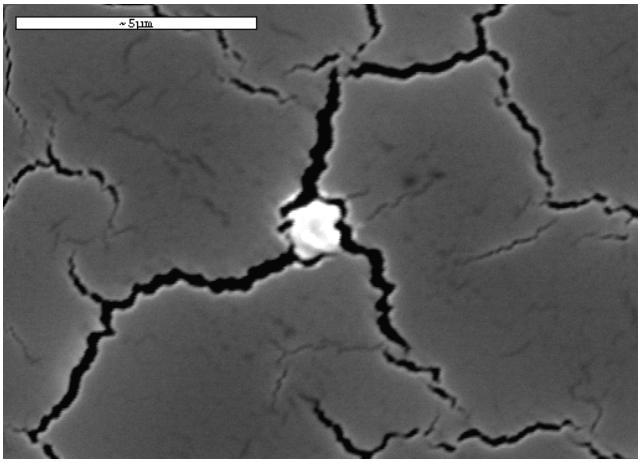


Fig.3-Cracks and grains are evident after 10 days of treatment.

By EDX analyser, we have investigated that inside the grains, for the samples treated with more than 8 days, there is the presence of new elements such as S, Ca, Fe. After 32 treatment days the number of cracks and grains increase and the number of different new elements increased with the time (see Fig. 4). So we can say that, for the H_2 loading experiments, the time duration of treatment is very important for the formation of elements different from Pd.

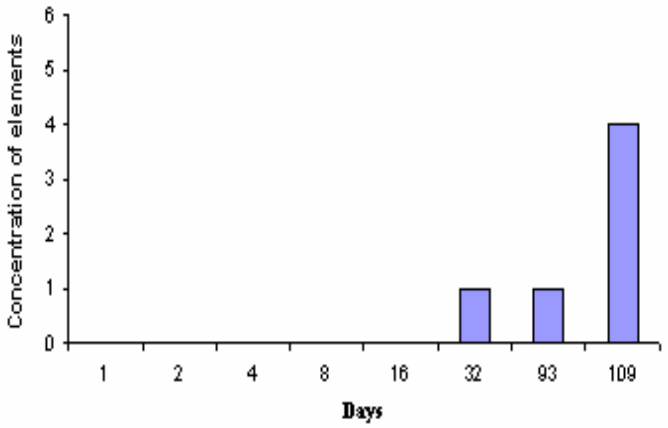
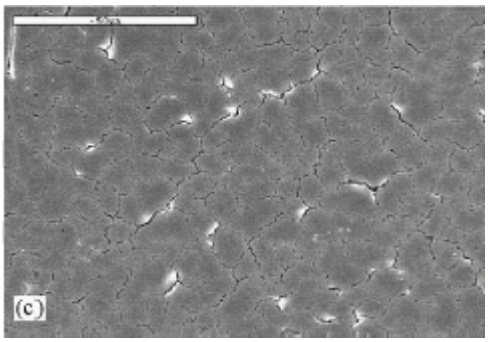


Fig. 4- Number of new elements in relation to the number of the days: no new elements before 32 days of loading in hydrogen senza laser.

Over a 109 days of treatment we have that the samples were very damaged because of the high gas loading.
Analysing by SEM the samples treated both gas loading and UV radiation we have observed that the laser effect increased the density and dimension of cracks and grains (see Fig. 5).



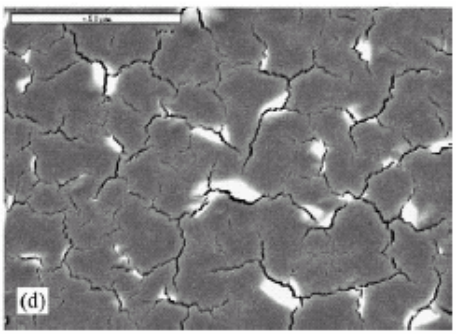


Fig. 5 –Laser effect: surface without laser (c) and with laser (d)

The Fig. 6 shows that the grain density of samples treated with different days and with the laser radiation is advanced to that one of the samples treated only for different days.

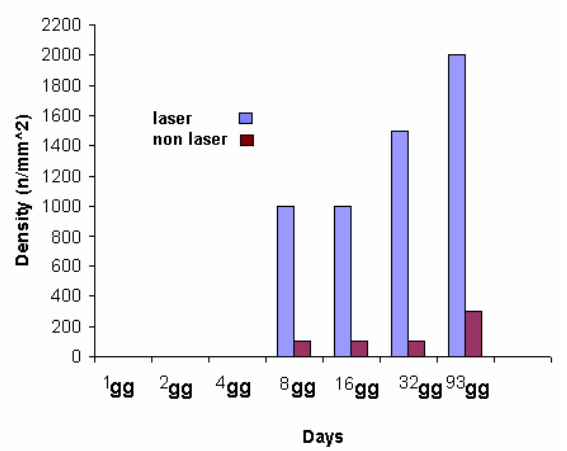


Fig. 6-Laser effect: the grain density increases stronger respect to non-laser experiment

Finally we have analysed some of treated samples by ICP-OES technique for having one quantitative measure of the elements produced from the loading of the gas and UV radiation treatments. In Table 1 the results are reported: Al amount increases with increasing number of days, while the Fe amount changes in an irregular way.

Samples	Al (µg/g)	Fe (µg/g)
32 days no laser	3.2	2.3
16 days laser	1.8	21.1
16 days no laser	0.0	4.3
Virgin	0.0	2.1

Table1- Results of ICP-OES technique

References

1 M. Fleischmann, S. Pons and M. Hawkins, *Electrochemically induced nuclear fusion of deuterium*,J. Electroanal. Chem., **263**, p. 301 (1989).

2 F. Scaramuzzi, *Survey of Gas Loading Experiments*, in Second Annual Conference on Cold Fusion, "The Science of Cold Fusion", Como, Italy (1991)

3 Y. Arata and Y.C. Zhang, *A new energy caused by "Spillover-deuterium"*, Proc. Jpn. Acad., Ser. B, **70 ser. B**, p.106 (1994).

4 Y. Iwamura et al. Detection of Anomalous Elememnts, *X-Ray and Excess Heat Induced by Continous Diffusion of Deuterium Through Multi-layer Cathode (Pd/CaO/Pd)*, in The Seventh International Conference on Cold Fusion, Vancouver, Canada (1998)

5 V. Nassisi, *Transmutation of elements in saturated palladium hydrides by an XeCl excimer laser*, Fusion Technol., **33**, p.468 (1998)

6 A. Castellano et al., *Nuclear Transmutation in Deutered Pd Films Irradiated by an UV Laser*, in 8th International Conference on Cold Fusion, Lerici, Italy (2000).

Analisi di bulk di D-Pd e H-Pd trattati con laser ad eccimeri

D. Doria, A. Lorusso, V. Nassisi, A. Pedone
Laboratorio di Elettronica Applicata, Dipartimento di Fisica, Università di Lecce,
I.N.F.N., C.P. 193, 73100 Lecce
Vincenzo.Nassisi@le.infn.it

SOMMARIO

Sono qui di seguito descritti risultati riguardanti elementi di "trasmutazione" osservati in bulk di Pd caricati con gas di deuterio e idrogeno e trattati con laser ad eccimeri. I campioni erano dischi di Pd puro di diametro pari a 8 mm e di spessore pari a 0.5 mm. Per realizzare gli esperimenti si sono utilizzate due camere di acciaio inox. Ciascuna camera conteneva due campioni. Un campione per ogni camera veniva irradiato per mezzo di un laser UV. Una camera è stata riempita con D_2 e l'altra con H_2, entrambe ad una pressione totale di 6 bar. Alla fine del trattamento, i campioni sono stati analizzati attraverso un Microscopio Elettrico a Scansione (SEM) e attraverso una sonda elettronica per effettuare la microanalisi (EDX). I campioni mostravano modificazioni morfologiche dovute alla presenza di buchi simili a piccoli crateri. All'interno di essi si sono trovati prodotti di trasmutazione indipendentemente dal gas caricato. La densità degli elementi trovati era più alta nei campioni caricati con H_2 rispetto a quelli caricati con D_2. Inoltre anche il trattamento laser ha contribuito ad aumentare la densità delle deformazioni trovate sui campioni. Come verifica, si sono tenuti due campioni di controllo in aria e sono stati analizzati insieme agli altri campioni trattati.

Si è inoltre andati a studiare l'andamento dell'assorbimento di pressione di dischi di Pd con diverso spessore valutando in che modo il volume del campione possa influire su di esso. Successivamente si è cercato di vedere sperimentalmente se l'assorbimento potesse essere considerato di tipo superficiale o di tipo bulk, andando ad unire due dischi di spessore pari a 0,5 mm ed andando ad effettuare, dopo un certo periodo di trattamento, l'analisi mediante il SEM e l'EDX sia delle superficie esterne all'unione dei due campioni e sia delle superfici interne valutandone eventuali differenze.

In ultima analisi si è cercato di schermare la camera a nostra disposizione con all'interno un dischetto, da eventuali influenze della radiazione cosmica, avvalendoci di un pozzetto di piombo.

INTRODUZIONE

In seguito alla scoperta di Fleischmann e Pons [1] negli anni ottanta, sono stati effettuati numerosi studi allo scopo di conoscere l'esatto processo che avviene negli esperimenti della fusione fredda.

In questo lavoro è stata posta particolare attenzione ai campioni trattati attraverso la tecnica a raggi X a dispersione di energia (EDAX) .

Le trasmutazioni nucleari sono state osservate sugli elettrodi utilizzati nelle celle elettrochimiche [2] e in campioni allo stato di film e di bulk [3-4]. Gli autori hanno anche misurato l'emissione di radioattività nei sistemi di PdD e NiH [5, 6].

In questi esperimenti la temperatura globale e l'energia termica in gioco erano molto basse e confermavano che i risultati sperimentali ottenuti sono dovuti alla formazione di processi non ancora conosciuti. Sperimentalmente le reazioni di trasmutazione sono addensate soltanto in piccoli spot somiglianti a grani nei film e a buchi nei bulk. Per mezzo di queste considerazioni, si suppone che soltanto un fenomeno collettivo possa giustificare i risultati sperimentali, inducendo un'alta probabilità d'interazione fra particelle.

Le interazioni fra particelle in materia condensata potrebbe essere dovuto a oscillazioni coerenti di elettroni metallici (plasmoni) [7]. Considerando l'idrogeno e il deuterio all'interno del metallo in uno stato elettronico, le oscillazioni degli elettroni possono generare un intenso campo elettrico oscillante che, successivamente, influisce sugli ioni accelerandoli. In questo modo due ioni risultano molto vicini al di sotto di 0.1 Å ed aumenta la probabilità di possibili reazioni nucleari.

La probabilità potrebbe anche aumentare a causa della distribuzione fononica, che separando le cariche positive da quelle negative potrebbe fare in modo di generare liberi elettroni in condizione di poter subire accelerazione. In questo modo i nuclei possono guadagnare energia cinetica superando, così, la barriera Coulombiana e conseguentemente generare fusioni nucleari [8].

Un'altra spiegazione potrebbe essere data dalla deformazione del reticolo cristallino di Pd-D o Pd-H che aumenta la probabilità di almeno 2-3 ordini di grandezza rispetto alla probabilità di fusione sulla superficie del reticolo [9].

In un articolo precedente le misure sono state effettuate utilizzando film di Pd depositati su substrati di Si [10]. Questo sistema permetteva di conoscere lo spessore limite del film, raggiunto il quale si ottenevano fenomeni di trasmutazione, ma non era possibile effettuare misure di concentrazione assoluta di elementi di trasmutazione. In questo lavoro si sono utilizzati bulk di Pd sotto forma di dischi, che successivamente sono stati sottoposti ad analisi mediante SEM e EDAX.

APPARATO SPERIMENTALE

I campioni di palladio usati in questo esperimento (puri al 99.95%) sono caratterizzati da un diametro di 8 mm, dallo spessore di 0.5 mm e dal peso di 0.3 gr. Per effettuare gli esperimenti sono state utilizzate due camere di acciaio inox inossidabile, di forma cilindrica aventi un diametro interno di 4 cm e lunghezza di 20 cm. Alle estremità sono state chiuse attraverso due finestre di quarzo che permettevano l'illuminazione laser dei campioni.

Ogni camera ha due saldature ϕ=6 mm di acciaio inox connesse al sistema da vuoto e al sistema di immissione del gas. La figura 1 mostra un'immagine della camera.

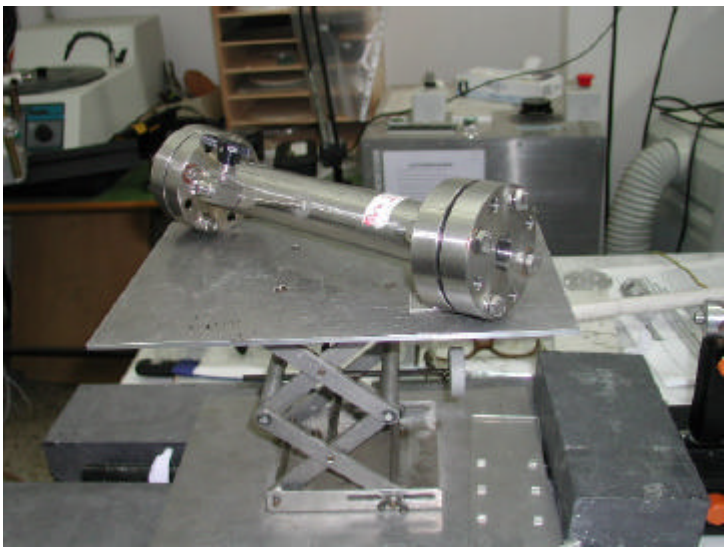


Figura 1: immagine di una camera di reazione

Sono stati posti due campioni all'interno di ogni camera e successivamente si è riempita una con D_2 e un'altra con H_2 entrambe ad una pressione totale superiore a 6 bar. I campioni erano fissati all'interno della camera attraverso un supporto di alluminio e fissati attraverso una molla di acciaio inox, fig.2.

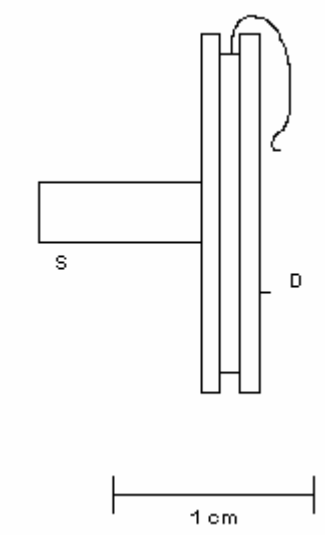


Figura 2a: disegno del supporto per i campioni

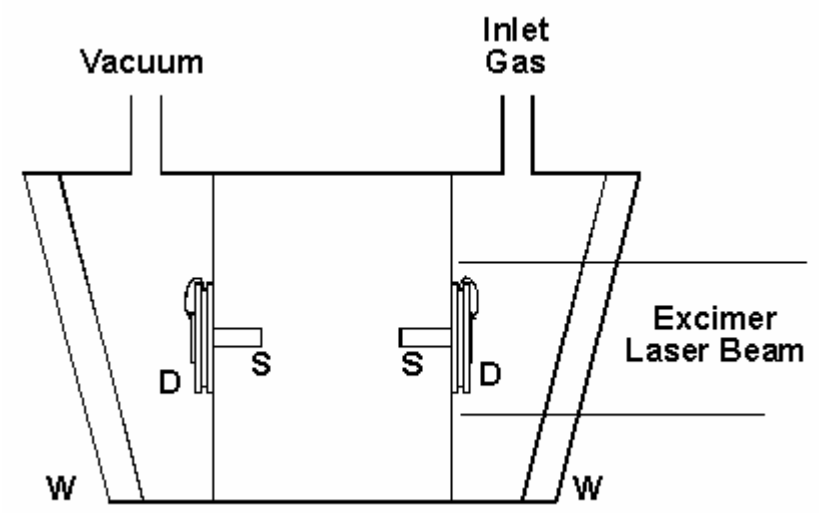


Figura 2b: disegno schematico della camera con all'interno i campioni da trattare.

Prima di effettuare gli esperimenti, le camere sono state accuratamente lavate con acetone e asciugate con azoto. All'interno delle camere si è innanzitutto effettuato il vuoto per mezzo di una pompa rotativa in serie con una turbomolecolare; una camera è stata riempita con deuterio e l'altra con idrogeno. In questo modo si sono effettuate tutte le possibili combinazioni riferite al caricamento di D_2 o H_2 e al trattamento con laser e senza laser. La tipica concentrazione d'impurità nell'idrogeno è tabulata come 1 ppm per N_2 e vapore acqueo, e livelli più bassi di altre specie gassose. Tipiche concentrazioni d'impurità nel deuterio sono <30 ppm per N_2, <15 ppm per il vapore acqueo e valori più bassi per altre specie gassose.

Il trattamento laser era effettuato dopo l'inserimento del gas all'interno delle camere ogni 15 giorni. La quantità totale dei colpi laser era di 500 colpi e una densità di energia approssimativamente di 100 mJ/cm^2. I campioni sono stati analizzati successivamente a 3500 irraggiamenti laser.

Il laser ad eccimeri lavora ad una lunghezza l'onda dell'UV vale a dire a 308 nm e con un impulso di 20 ns. Il fascio UV illumina i campioni in atmosfera di H_2 or D_2 favorendo il processo di assorbimento multifotonico che localizza gli atomi di gas all'interno del metallo ospitante[11].

Il microscopio elettronico a scansione è un Philips XL 20 equipaggiato con un microanalizzatore EDAX DX-4.

Risultati sperimentali

Dopo 113 giorni le camere sono state aperte e i campioni trattati, insieme a due campioni tenuti in aria come controllo, sono stati analizzati attraverso il SEM e l'EDAX per lo studio dell'aspetto morfologico della superficie e per la composizione chimica.

Campioni caricati con deuterio:

L'analisi morfologica mostra la formazione di piccoli crateri e caverne. La loro concentrazione era di circa 1200 e 1600 per mm^2 rispettivamente per il campione non irradiato e per quello irradiato. In tutte le misure fra gli elementi rilevati, si sono trovati Pd e O. In particolare il campione è soggetto a naturale ossidazione della superficie del palladio. Si sono analizzati approssimativamente 20 spot distribuiti su tutto il campione, trovando i seguenti elementi: Mg, Al, Si e Ti per il campione non trattato con radiazione laser mentre Al, Si, S, Cr, Fe, e Pt per i campioni irraggiati. Le figure 3a e 3b mostrano il numero di volte che un elemento è stato trovato nei 20 spot analizzati.

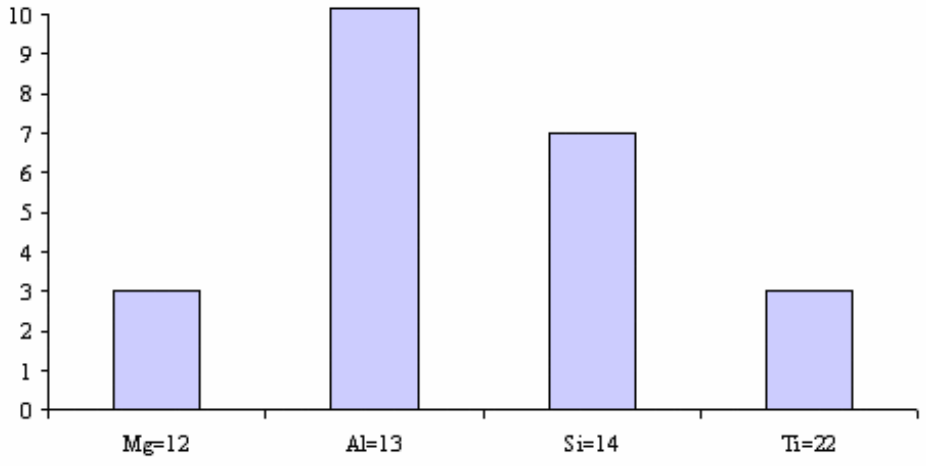


Figura *3a: elementi trovati sul campione non irraggiato*

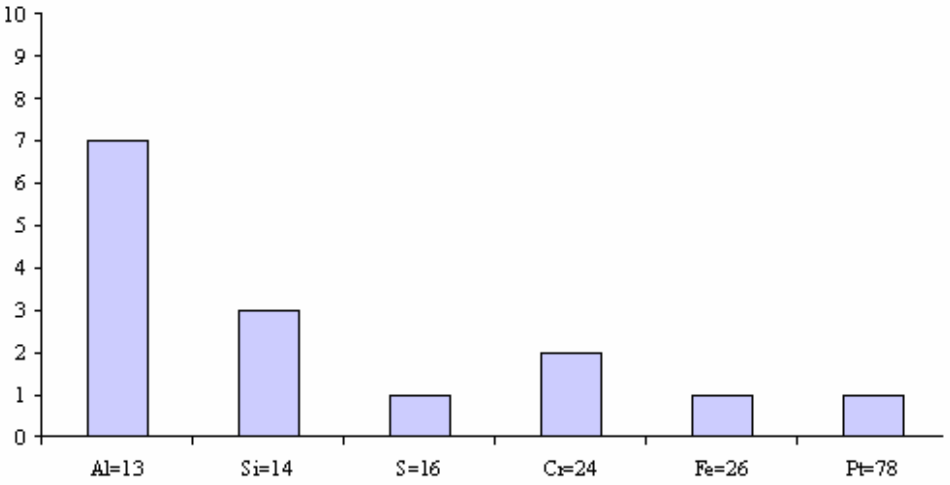


Figura 3b: elementi trovati sul campione irraggiato

Campioni caricati con idrogeno:

Anche in questo caso la concentrazione dei crateri e dei pit è di 1200 e 1600 spot per mm^2 rispettivamente per quelli non trattati e per quelli trattati. La microanalisi

effettuata all'interno delle caverne rileva in molti casi la presenza di altri elementi rispetto al Pd.

Esaminando nuovamente 20 spot distribuiti sulla superficie dei campioni si sono trovati i seguenti elementi: Na, Mg, Al, Si, S, Fe e Zn per i campioni non trattati con luce laser, mentre Al, Si, S, Cl, Fe, Cd e Pt per quelli trattati con laser. Le figure 4a e 4b mostrano il numero di volte che gli elementi sono stati trovati.

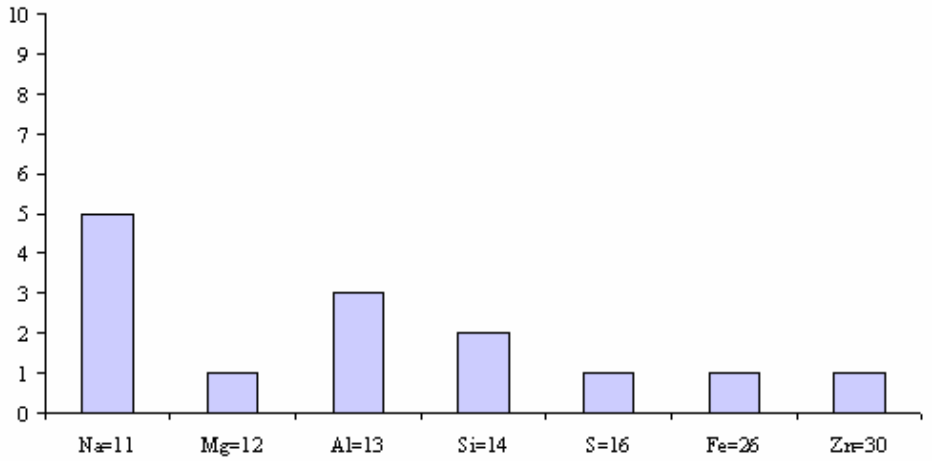


Figura 4a: elementi trovati sul campione non irraggiato

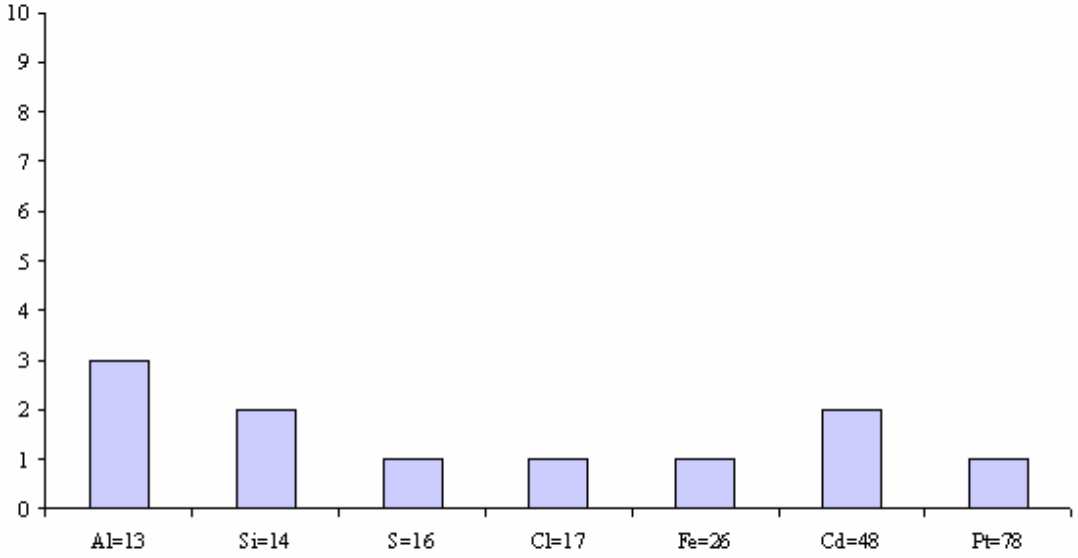


Figura 4b: elementi trovati sul campione irraggiato

Qui di seguito è riportata una tabella riassuntiva degli elementi trovati sulla superficie dei campioni trattati

Dischi di palladio caricati con gas diverso			
Tipo di gas	**Irradiato**	**Elementi trovati**	**Deformazioni/mm^2**
H_2	*No*	*Al, Si, S, Cl, Fe, Cd, Pt*	*1200*
H_2	*Si*	*Na, Mg, Al, Si, S, Fe, Zn*	*1600*
D_2	*Si*	*Al, Si,S, Cr, Fe, Pt*	*1600*
D_2	*No*	*Mg, Al, Si, Ti*	*1200*

Tabella 1- Risultati sperimentali ottenuti con campioni aventi diversi parametri di trattamento

Il fatto di aver trovato per la maggior parte elementi con numero atomico inferiore rispetto al Pd, suggerisce che i processi alla base di questo fenomeno potrebbero anche essere processi di fissioni come afferma la teoria RIFEX [9]. Secondo questa teoria, infatti, si verrebbero a generare atomi instabili durante reazioni multiple di fusione nel metallo in grado di formare un nucleo complesso che successivamente si scinde producendo la serie di prodotti osservati. La piccola quantità di energia in eccesso ottenuta dalla reazione di fissione permetterebbe la formazione di nuclei stabili con bassa emissione di radiazione molto energetica.

Differenze fra i campioni tenuti in aria e quelli trattati

I campioni tenuti in aria non presentano evidenti cambiamenti morfologici, ma soltanto delle intrinseche deformazioni di circa 400 per mm^2. Le figure 5 e 6 mostrano la sostanziale differenza fra un campione non trattato e uno trattato con idrogeno e irradiato: la densità di deformazioni è evidentemente maggiore.

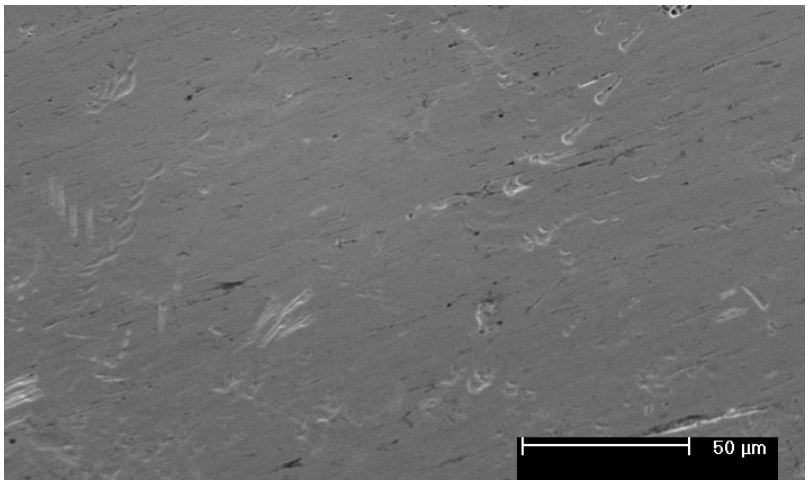


Figura 5: immagine SEM a 500X d'ingrandimento di un campione non trattato

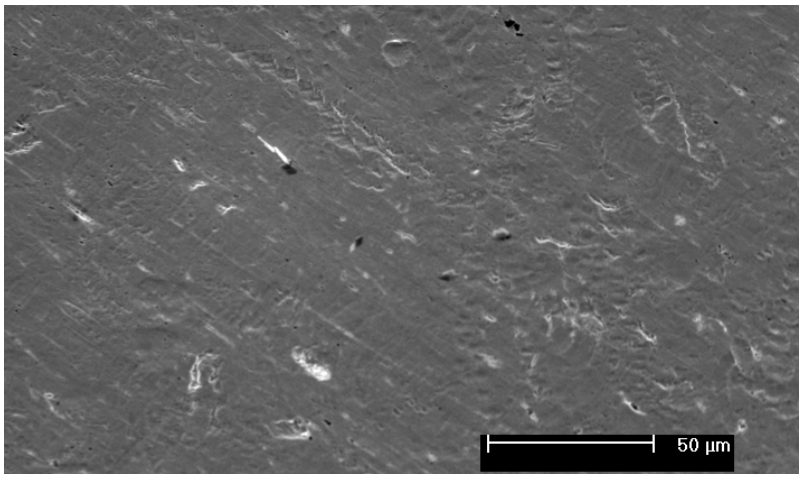


Figura 6: immagine SEM a 500X d'ingrandimento di un campione trattato con idrogeno e irradiato

Gli spettri EDX non rilevano altri elementi che Pd e O e in alcuni casi la presenza di C come dimostra lo spettro di figura 7. Sono stati scelti a caso alcuni punti per eseguire le suddette misure.

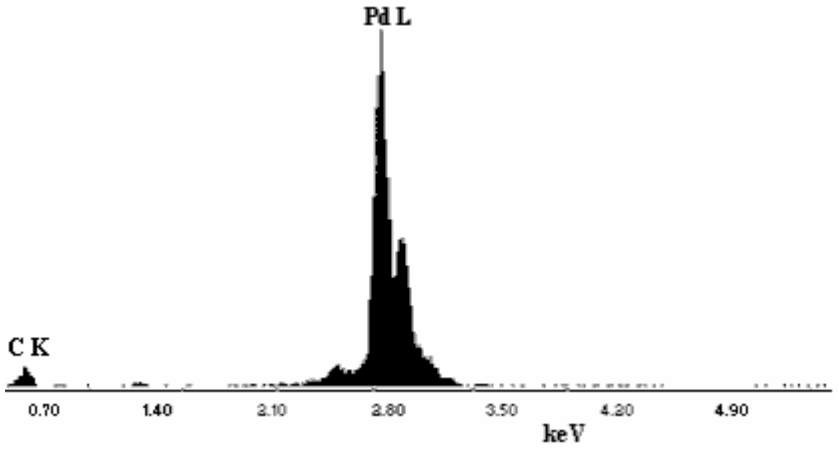


Figura 7: spettro rilevato dal campione non sottoposto a trattamento

Gli spettri rilevati sui campioni trattati (figura 8) presentano, invece, picchi appartenenti ai diversi elementi chimici trovati.

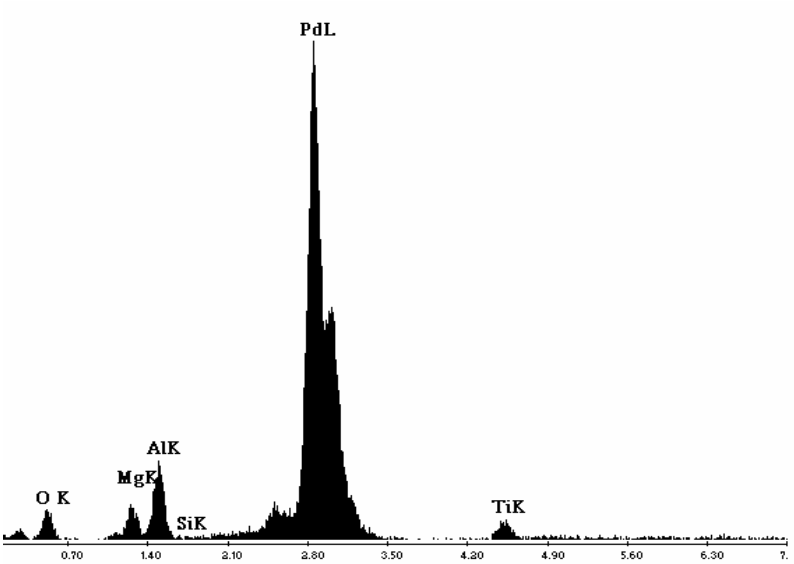


Figura 8: spettro rilevato da un campione trattato con D_2 e non irradiato

Questi elementi sono stati sempre trovati all'interno di strutture simili a quella raffigurata nella figura 9:

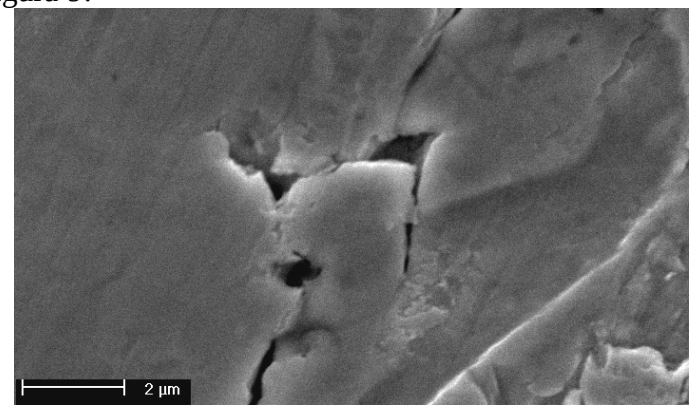


Figura 9: immagine della struttura su cui si è effettuata la microanalisi precedente.

Gli elementi trovati non sono attribuibili ad impurezze presenti sul campione al momento del trattamento in quanto dovrebbero trovarsi distribuiti su diversi punti della superficie del campione, invece, tali elementi si trovano solo all'interno di strutture somiglianti a profonde cavità come quella rappresentata nella figura 9.

Misure sull'andamento temporale della pressione

La trasmutazione del Pd in elementi diversi e gli altri effetti dovuti alla "fusione fredda", sono conseguenza del fatto che il metallo abbia un alto potere assorbente nei confronti dell'idrogeno e del suo isotopo, il deuterio: il Pd raggiunge, infatti, un rapporto assorbente totale pari a H/Pd=0.77 (D/Pd=0.77) [1.16].

Per verificare sperimentalmente questo risultato e l'andamento nel tempo dell'assorbimento della pressione, si è utilizzata una minicamera di cui se ne riporta un'immagine ed un disegno delle sue dimensioni nelle figg. 10 e 11.

Figura 10: immagine di una minicamera per la misura dell'assorbimento

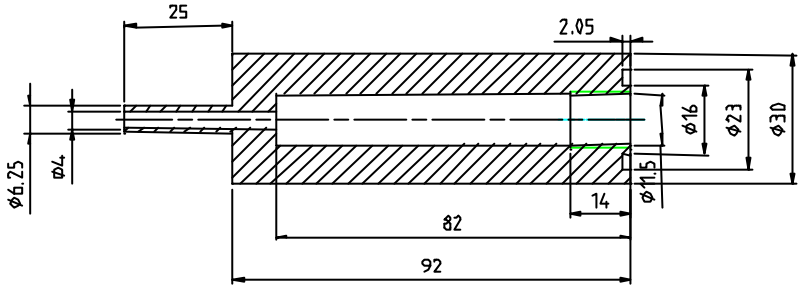


Figura 11: schema della minicamera sezionata

All'interno della minicamera si è posto, inizialmente, un disco di Pd di spessore pari a 0.5 mm, diametro 8 mm e purezza pari a 99.99% ed è stata inserita una pressione di H_2 ad un valore iniziale di

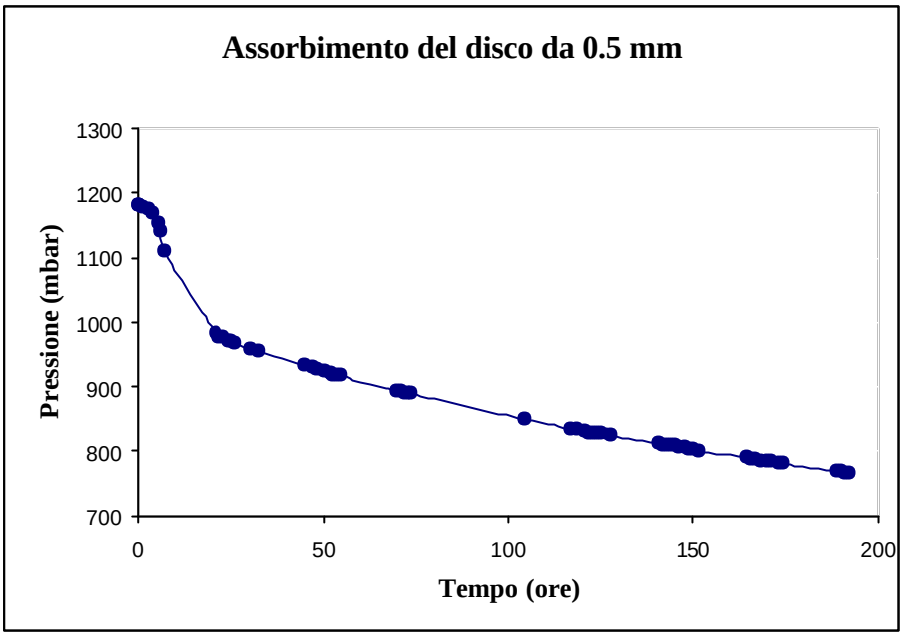


Figura 12: andamento della pressione in funzione del tempo, con un disco di spessore di 0.5 mm.

(1.17±0.05) bar, rilevando nel tempo il valore della pressione per mezzo di un manometro a membrana. L'andamento della pressione è riportato in figura 12.

Come si può osservare dalla figura , la pressione della camera diminuisce nelle prime 20 ore di assorbimento, indicando un repentino assorbimento del gas: questo è dovuto probabilmente al fatto che, inizialmente, la diffusività del gas può essere considerata approssimativamente costante. Successivamente vi è una decrescita della pressione più graduale, dipesa dal fatto che la diffusività è legata, in questo caso, alla concentrazione del gas all'interno del bulk, come pure al volume del campione.

Una volta raggiunto un certo valore della pressione del gas all'interno della minicamera il campione raggiunge uno stato quasi stazionario.

Nel momento in cui il suddetto equilibrio viene alterato, andando a ripristinare la pressione del gas, il campione di Pd continua ad assorbire come mostrato nella seguente figura:

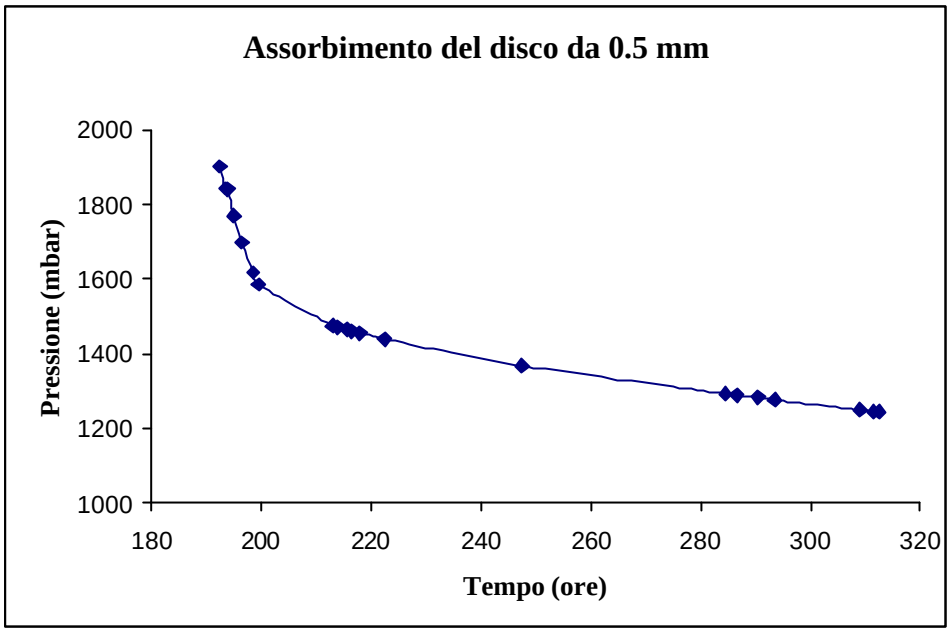


Figura 13: andamento della pressione nel tempo dopo aver effettuato un secondo caricamento del gas.

Anche nella curva rappresentata in figura 13 si nota un "caratteristico" cambio di pendenza.

Andando a studiare il comportamento dell'assorbimento utilizzando un disco di Pd di spessore pari a 0.125 mm, vale a dire ¼ dello spessore di quello precedente, si osserva (vedi fig.14) che partendo da una pressione iniziale di (1.09±0.01) bar, il cambio di pendenza è presente dopo circa 5 ore.

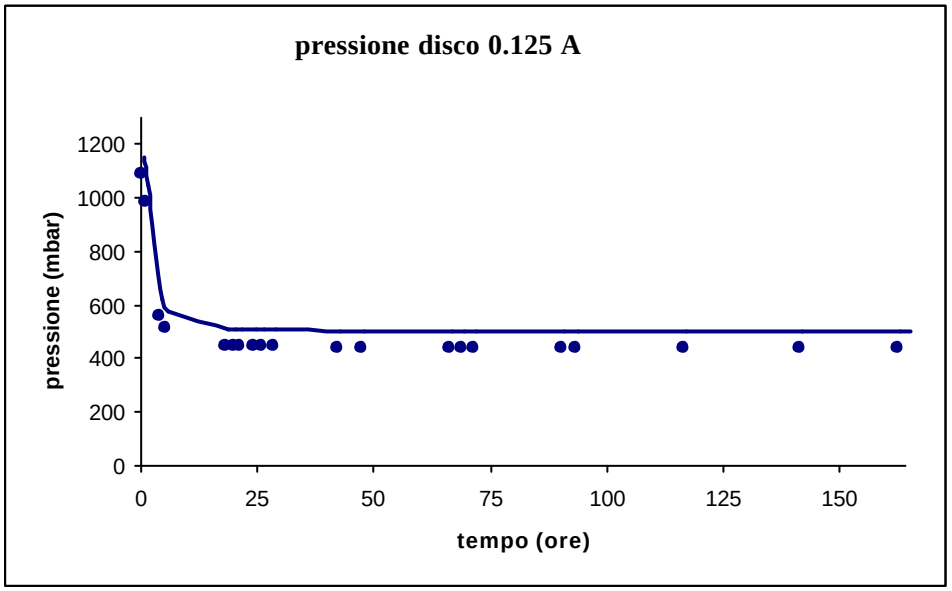


Figura 14: andamento della pressione del disco di spessore da 0.125mm, nel primo caricamento.

Questo è probabilmente dovuto al fatto che, essendo il disco più sottile, il gas sia stato assorbito immediatamente sia dagli atomi di Pd in superficie sia da quelli del bulk, arrivando allo stato di quasi equilibrio.

Inoltre il rapporto di assorbimento per il disco più spesso raggiunge il valore noto in letteratura a seguito di svariati caricamenti mentre per il disco più sottile lo si raggiunge già con il primo caricamento.

Si può allora concludere che il rapporto di assorbimento e il suo andamento nel tempo dipende dallo spessore del campione utilizzato.

Osservando gli andamenti dell'assorbimento in funzione del tempo, si è cercato di individuare una curva che meglio approssimasse i dati a nostra disposizione.

Essendo che gli assorbimenti in natura sono caratterizzati da leggi esponenziali, si è proceduto nell'adattamento dei dati sperimentali riportati in figura 4.36, per mezzo della somma di due esponenziali:

$$A\exp(-t/t) + B\exp(-t/b)$$

dove A e B sono due costanti arbitrarie, mentre t e b sono costanti del tempo di decadimento di valori pari a:

$A = 235 mbar$

$t = 20 ore$

$B = 940 mbar$

$b = 960 ore$

Il primo esponenziale tiene conto dell'andamento dell'assorbimento nelle prime 20 ore di trattamento, mentre il secondo, rappresenta l'andamento nel restante periodo di tempo ed ha, infatti, una costante di decadimento più grande.

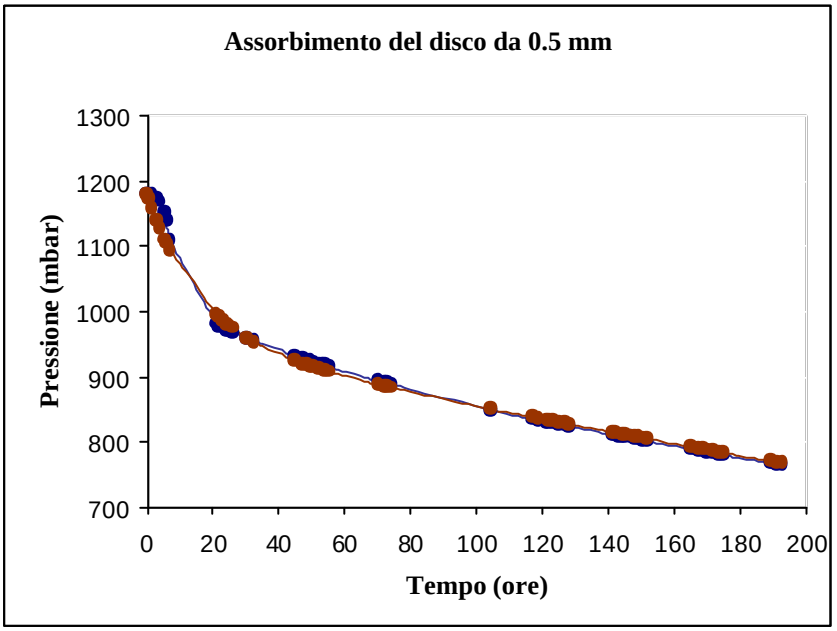


Figura 15: andamento della funzione teorica (rossa) sovrapposta all'andamento di quella sperimentale (blu).

Come si può osservare dalla figura 15, vi è un buon adattamento della curva teorica ai dati sperimentali.

Si è proceduto allo stesso modo con l'assorbimento del disco più sottile:

$C\exp(-t/g)+D\exp(-t/d)$

dove C e D sono costanti arbitrarie e g ed sono le costanti di decadimento di valore pari a:

$C = 600 mbar$

$g = 5 ore$

$D = 440 mbar$

$d = 10000 ore$

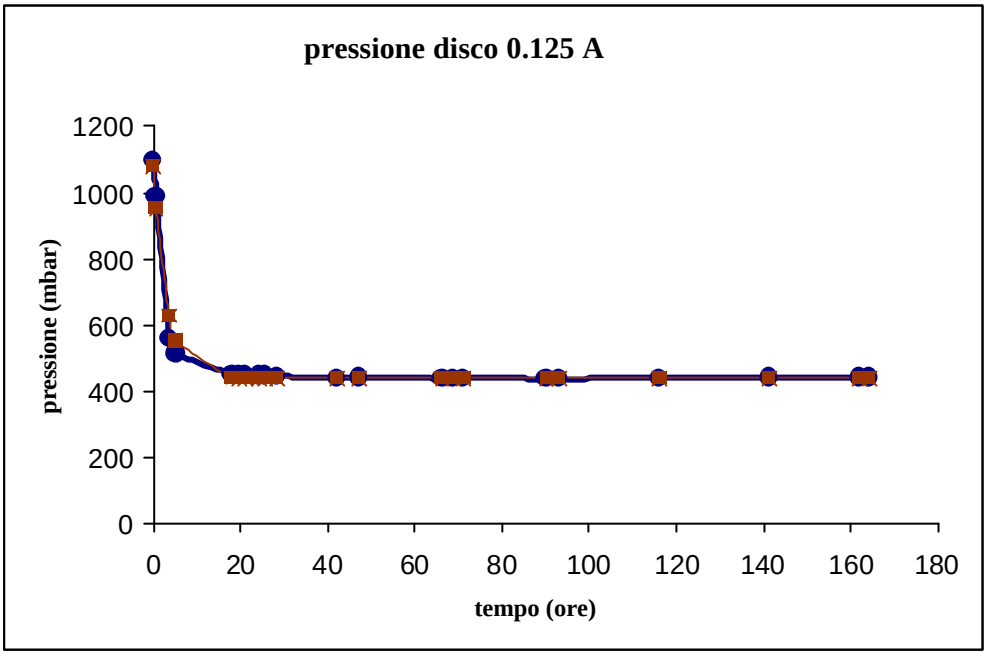


Figura 16: sovrapposizione della curva sperimentale (rossa) con quella teorica (blu) per il disco di spessore 0.125 mm.

Anche in questo caso si può notare, dalla figura 16, un buon adattamento della curva teorica ai dati sperimentali a nostra disposizione.

Schermaggio di campioni per mezzo di piombo e assorbimento di tipo bulk dei campioni

Per evitare che eventuali influenze, dovute alle componenti dei raggi cosmici, potessero alterare le reazioni che avvengono in seguito all'assorbimento di idrogeno da parte del Pd, si è posta la minicamera, con all'interno un disco di spessore di 0.5 mm, in una scatola di piombo di lunghezza pari a 32 cm e spessore di circa 4 cm.

La scatola di piombo, a nostra disposizione, è in grado di schermare la componente elettromagnetica dei raggi cosmici, ma non è in grado di fare lo stesso per la componente dei neutroni che, passando inalterati, arrivano al campione di Pd che sta reagendo con il gas presente all'interno della minicamera.

Anche in questo caso il campione è stato trattato con idrogeno ad una pressione iniziale di circa 1 bar per 40 giorni ed anche in questo caso, andando ad analizzare il campione al SEM e all'EDX, si sono riscontrati numerose deformazioni morfologiche e all'interno di esse si è rilevata la presenza di "nuovi" elementi come Si, Al, Ti, Cr.

Per cercare in qualche modo di ostacolare il passaggio della componente neutronica dei raggi cosmici si è proceduto alla realizzazione di un esperimento consistente in due dischi di Pd dallo spessore pari a 0.5 mm uniti insieme per mezzo di una molla di acciaio.

Dato che il palladio ha, fra le sue caratteristiche, quella di avere un'alta sezione d'urto di assorbimento dei neutroni termici, si sono inseriti i due dischi di Pd all'interno di una minicamera ad una pressione iniziale di circa 2 bar per un periodo di trattamento di 45 giorni.

L'analisi SEM e EDX non hanno rilevato un'eclatante differenza fra la parte interna dei due dischi e la parte esterna esposta all'assorbimento del gas. Questo ci fa intuire come l'assorbimento del gas possa avvenire non in maniera superficiale ma coinvolgerebbe l'intero bulk del disco.

CONCLUSIONI

Dagli esperimenti effettuati con questa serie di campioni si è potuto valutare che il trattamento dei dischi con idrogeno porta alla formazione di un maggior numero di elementi "nuovi" rispetto a quelli trattati con deuterio; l'azione laser, inoltre, contribuisce ad aumentare la densità delle deformazioni che si vengono a formare, a seguito di trattamento, sulla superficie dei dischi, ma non influisce nella formazione di elementi nuovi.
Infine, dalle misure effettuate sull'assorbimento dell'idrogeno da parte dei campioni, si è potuto studiare l'andamento dell'assorbimento nel tempo per dischi di diverso spessore e valutare come l'andamento dipenda dal volume del campione utilizzato.

RINGRAZIAMENTI

Gli autori hanno il piacere di ringraziare l'eccellente supporto tecnico di AR. De Bartolomeo, M. D'Elia, F. De Donno, L. Monteduro e V. Nicolardi.

RIFERIMENTI

1. M. Fleischman and S. Pons, "*Electrochemically Inducted Nuclear Fusion of Deuterium*", J. Electroanal. Chem. **261**, 301 (1989)

2. V. Nassisi and M.L. Longo, "*Experimental results of transmutation of elements observed in etched palladium samples by an excimer laserTransmutation of elements in saturated palladium hydrides by*

an XeCl excimer laser", Fusion Technol. **33**, 468 (1998).

3. G. Miley, "*Product characteristics and energetics in thin-film electrolysis experiments*", Proceedings of VII International Conference on Cold Fusion, 241 (1998)

4. IM. Chapnik, "*Possibility of electrochemically induced transmutation in PdD*", Phy. Lett. A, **161**, 111 (1991).

5. H. Kozima and K. Arai, "Localized nuclear transmutation in PdHx observed by Bockris and Minevski revealed a characteristic of CF phenomenon", Int. J. Hydrogen Energy, **25**, 513 (2000)

6. Battaglia, L. Daddi, Focardi S., V. Gabbani, V. Montalbano, F. Piantelli, PG,. Sona S. Veronesi, "*Neutron emission in Ni-H systems*", Nuovo Cimento-A, **11**, 921 (1999)

7. V. Violante, "*Lattice ion trap, confinement for deuterons and protons: possible interaction in condensed matter*", Fusion Technol., **35**, 361 (1999).
8. F. Frisone, "*Probability of deuteron-plasmon fusion at room temperature within microcracks of crystalline lattices with deuterium loading*", Proceedings of VIII International Conference on Cold Fusion, ICCF-8, **Lerici-I**, Ed. SIF (2000).

9. G. Stoppini, "*Nuclear processes in hydrogen–loaded metals"* Fusion technol. **34**, 81 (1998)

10. M. Di Giulio, E. Filippo, D. Manno and V. Nassisi, "*Analysis of nuclear transmutations observed in D-and H-loaded Pd films*", Intern. J. Hydr. Energy, (2002)
11. T.Sugii, T. Ito and H. Ishikawa, "*Excimer laser enhanced nidridation of silicon substrates*", Appl. Phys. Lett., **45**, 966 (1984).

D. Doria, E. Filippo, M. Di Giulio, A. Lorusso, D. Manno, V. Nassisi and A. Pedone
"Characterization of Pd-H_2 thin films irradiated by UV laser**"**
Technical Digest Cleo/Europe **(2003)**

A. Lorusso, V. Nassisi, E. Filippo*, M. Di Giulio*, D. Manno*, G. Buccolieri, F. Celani
Measurements of new elements in Pd-H2 thin films
J. CF5 Giappopne 2004.

A. Lorusso, V. Nassisi, E. Filippo*, M. Di Giulio*, D. Manno*, G. Buccolieri
Modification of Pd-H2 films processed by UV laser
5th Asti Workshop on Cold fusion may 2004

LASER-SOURCE S.R.L.

FORNITURE DI DISPOSITIVI ELETTRONICI E OTTICI

Via N. Tommaseo, 3
00060 CAMPAGNANO - ROMA
Tel. 06 90 41 786 - Fax 06 90 41 470
[illegible]
www.laseroptron.it

Spett.le
UNIVERSITA' DI LECCE
DIP. DI FISICA
Att.ne Prof. Nassisi
Via per Arnesano
73100 LECCE

Campagnano, 12/07/04

In riferimento alla Sua gentile richiesta siamo lieti di inviarLe ns offerta n. 4.290/SDP/gm per materiale ns rappresentata Melles Griot. e Suwtech.

Pos.	Qta'	Descrizione	Prezzo Unit. in Euro
01	01	LASER HE-NE MOD. 05 LHP 927 potenza : 17 mW completo di alimentatore	4.300,00
02	01	TEMPERATURE STABILIZED GREEN LASER MOD. DPGL 2100 potenza : 100 mW completo di alimentatore	4.000,00

Condizioni di fornitura

In caso di ordine si prega di citare per esteso le condizioni di fornitura sotto menzionate al fine di facilitarci l'evasione dell'ordine.

Pagamento :30gg. d.f. B.B.
Resa : F.co Vs magazzini
Consegna: 60 gg dal ricevimento ordine
Validità offerta :60 gg
<u>IVA : inclusa</u>
Legame valuta: 1 USD = € uro 0,825+/-1 %.
Ogni variazione del cambio si rifletterà nella stessa misura percentuale, sul valore dell'ordine in sede di fatturazione. (Non si procederà a rettifiche per variazioni contenute nel +/-1 %; nel caso di variazioni superiori al +/-1 % si applicherà l'intera variazione intervenuta).Cambio pubblicato su "IlSole 24 ORE" riferito al giorno antecedente la data del ns. D.D.T.

Distinti saluti
Per DOTT. SILVANO DE PASCALIS
Giuseppina Micozzi
LASER SOURCE SRL

LASER-SOURCE S.R.L.

FORNITURE DI DISPOSITIVI ELETTRONICI E OTTICI

Via N. Tommaseo, 3
00060 CAMPAGNANO - ROMA
Tel. 06 90 41 786 - Fax 06 90 41 470
www.laseroptron.it

Spett.le
UNIVERSITA' DI LECCE
DIP. DI FISICA
Att.ne Prof. Nassisi
Via per Arnesano
73100 LECCE

Campagnano, 12/07/04

In riferimento alla Sua gentile richiesta siamo lieti di inviarLe ns offerta n. 4.290/SDP/gm per materiale ns rappresentata Melles Griot. e Suwtech.

Pos.	Qta'	Descrizione	Prezzo Unit. in Euro
01	01	LASER HE-NE MOD. 05 LHP 927 potenza : 17 mW completo di alimentatore	4.300,00
02	01	TEMPERATURE STABILIZED GREEN LASER MOD. DPGL 2100 potenza : 100 mW completo di alimentatore	4.000,00

Condizioni di fornitura

In caso di ordine si prega di citare per esteso le condizioni di fornitura sotto menzionate al fine di facilitarci l'evasione dell'ordine.

Pagamento :30gg. d.f. B.B.
Resa : F.co Vs magazzini
Consegna: 60 gg dal ricevimento ordine
Validitá offerta :60 gg
IVA : inclusa
Legame valuta: 1 USD = € uro 0,825+/-1 %.
Ogni variazione del cambio si rifletterà nella stessa misura percentuale, sul valore dell'ordine in sede di fatturazione. (Non si procederá a rettifiche per variazioni contenute nel +/-1 %; nel caso di variazioni superiori al +/-1 % si applicherá l'intera variazione intervenuta).Cambio pubblicato su "IlSole 24 ORE" riferito al giorno antecedente la data del ns. D.D.T.

Distinti saluti
Per DOTT. SILVANO DE PASCALIS
Giuseppina Micozzi
LASER SOURCE SRL

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Francesco CELANI		

Struttura
LNF

ALLEGATO MODELLO EC2

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Francesco CELANI		

Struttura
LNF

ALLEGATO MODELLO EC2

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Onori		

Struttura
PG

ALLEGATO MODELLO EC2

Attività svolta

Abbiamo messo a punto un sistema spettrofotometrico per la stima di piccole quantità di acqua ossigenata in soluzioni acquose. Tale sistema si basa sulla analisi delle variazioni nello spettro di assorbimento del citocromo−c in seguito all'ossidazione dell'atomo di ferro legato al gruppo eme da parte dell'acqua ossigenata. Tale approccio, che appare innovativo rispetto a sistemi tradizionali utilizzati a questo scopo, permette da un lato di stimare la concentrazione di specie ossidanti in acqua con una precisione di ±10−6moli/litro e dall'altro di mettere in evidenza un effetto di interesse biologico sulla biomolecola.

Attività prevista

Il sistema messo a punto nel primo anno di ricerca verrà utilizzato per: a) l'analisi comparativa dei radicali liberi radioindotti in H2O e D2O, b) analisi della correlazione tra l'entità degli effetti biologici di H2O e D2O irradiata e presenza di specie ossidanti. Si farà uso, a questo scopo, di tecniche di spettroscopia ottica nel IR−VIS−UV con strumentazione presente nel laboratorio di Biofisica del Dipartimento di Fisica dell'Università di Perugia oltre che di misure IR con DAFNE Luce presso i Laboratori Nazionali di Frascati.

Risultati attesi

Lo studio quantitativo delle specie ossidanti presenti in H2O e D2O irradiata e l'analisi della correlazione con gli effetti biologici osservati, permetterà di comprendere meglio i meccanismi responsabili degli effetti citotossici dell'acqua irradiata e delle differenze riscontrate tra H2O e D2O.

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Onori		

Struttura
PG

ALLEGATO MODELLO EC2

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Rapp. Naz.: Francesco CELANI		

PREVENTIVO GLOBALE DI SPESA PER L'ANNO 2005

In KEuro

Struttura	**A CARICO DELL' I.N.F.N.**																				A carico di altri Enti
	Missioni interne	SJ	Missioni estere	SJ	Materiale di consumo	SJ	Trasporti e facchinaggi	SJ	Spese di calcolo	SJ	Affitti e manutenzione	SJ	Materiale inventariabile	SJ	Costruzione apparati	SJ	TOTALE Compet.	SJ			
LE	6,0		6,0		7,0								4,0				23,0				0,0
LNF	5,0		10,0		24,0								16,0		20,0		75,0				0,0
PG	5,0				8,0								4,0				17,0				0,0
TOTALI	16,0		16,0		39,0								24,0		20,0		**115,0**				

NB. La colonna A carico di altri enti deve essere compilata obbligatoriamente

Mod EC./EN. 4 (a cura del responsabile nazionale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Rapp. Naz.: Francesco CELANI		

A) ATTIVITA' SVOLTA FINO A GIUGNO 2004

Riguardo FREETHAI−LNF, (Fusione Fredda), sinteticamente, l'attivita' svolta nel periodo gennaio− giugno 2004 e' stata:
1) Sviluppata procedura innovativa per misura del coefficente termico resistivo dei composti PdH_x e PdD_y al variare delle concentrazioni x e y di H e D, "IN SITU" durante il loading/deloading. Tale tipo di misura e' stata effettuata con successo nonostante difficolta' di principio, apparentemente insormontabili, dovute a problemi multiparametrici. Risultati presentati a Conferenza Internazionale (marzo 2004): PRIMA MISURA AL MONDO.
2) Effettuate misure, dal punto di vista calorimetrico e analisi ICP−MS, con elettrolita "ibrido", cioe' alcool etilico leggero in H2O (95/5), 750 cc, a cui sono stati aggiunti solo 10 cc di D2O. Le anomalie termiche e composizionali, precedentemente riscontrate con C2H5OD e D2O, sono praticamente svanite. Le analisi ICP−MS hanno mostrato una drastica riduzione (circa un fattore 100) delle anomalie composizionali ed isotopiche precedentemente riscontrate con soluzione "pesante": risultati particolarmente confortanti, poiche' giustificano anomalie termiche/nucleari in soluzioni deuterate.
3) Costruzione di una nuova cella elettrolitica con contenitore in quarzo, in sostituzione della precedente cella in vetro borosilicato 3.3. Motivi dettati dalla necessita' di "vedere" hot spot su filo di palladio usando termocamera IR. Contenitore in quarzo utilizzato, a parete sottile, e' risultato troppo delicato: da modificare sostanzialmente la struttura costruttiva.

Riguardo la citotossicita' con D2O, (ex DEUTER) ricordiamo che l'utilizzo dell'ossido di Deuterio, come possibile agente citotossico risale al 1936 in Germania, i primi esperimenti italiani risalgono al 1938. In tutto il mondo a tutt'oggi sono stati pubblicati circa 1500 lavori. Non si e' mai passati dalla sperimentazione su cavie a quella umana: risultati sperimentali altamente contraddittori. Una possibile causa della NON RIPRODUCIBILITA' e' risultata essere la presenza anche di funghi e batteri, alcuni dei quali con dimensioni inferiori al valore di 220 nm: vanificavano la prevista sterilizzazione effettuata con le procedure universalmente adottate (filtri di sterilizzazione della Millipore a 220 nm). Con la scoperta di tali specifiche nuove specie batteriche, da noi effettuata nel 2000 e registrata a livello internazionale presso le istituzioni all'uopo deputate (Gene Bank in Usa e DDBJ in Giappone), abbiamo profondamente modificato le apparecchiature utilizzate per la distillazione, aggiugendo in situ una sezione di ultradistillazione a 100 nm, in PTFE. Il primo risultato significativo ottenuto e' stato la completa riproducibilita' degli esperimenti: abbiamo verificato che per avere una significativa azione citotossica era necessario utilizzare una soluzione con almeno il 30% di D2O. Tale valore elevato risulta essere, purtroppo, difficilmente utilizzabile in situazioni in vivo (valore limite 10%): abbiamo esplorato l'ipotesi di ottenere un effetto sinergico dell'acqua pesante "di per se" con l'acqua "ossigenata". A tale scopo abbiamo irradiato con dosi massicce di radiazioni gamma (17kGy) l'acqua pesante da noi precedentemente purificata. Ovviamente le radiazioni ci assicurano anche l'eliminazione di batteri fortuitamente ancora presenti nella soluzione. Dopo tale procedura abbiamo verificato che e' sufficiente una concentrazione solo del 3% per avere effetti citotossici significativi. (Allegati inviati in Commissione 5)

B) ATTIVITA' PREVISTA PER L'ANNO 2005

Per quanto riguarda la attivita' cosi' detta di "fusione fredda" si prevedono i seguenti tasks:
1) serie di misure sistematiche con termocamera IR (con costruzione di nuova cella elettrolitica in quarzo/compositi);
2) studio effetto di nanostrutture, opportunamente prodotte in situ e depositate sul filo di Palladio, dal punto di vista della generazione di fenomeni anomali termici e/o nucleari. Allo scopo, verranno estensivamente utilizzate anche tecniche di elettrolisi AC ad alta frequenza e potenza di picco, di cui siamo stati pionieri nello sviluppo dal 1994 (svariati articoli pubblicati su Physics Letters A) a cui verranno sovraimposte modulazioni a frequenza sia ultrasonica che dell'ordine delle centinaia di Hz;
3) Sviluppo di un anodo meno soggetto ai fenomeni di corrosione riscontrati con quelli attualmente utilizzati (Pt al 99.99% di purezza).

Attivita' prevista per ex−DEUTER:
1) Prima identificazione e quantificazione delle sostanze che aumentano in modo cosi' marcato l'effetto citotossico della D2O irraggiata (collaborazione con il Gruppo di Perugia) (Allegati inviati in Commissione 5).
2) Primi test dell'effetto citotossico "macroscopico" su cavie da laboratorio (collaborazioni, da perfezionare, con Centro Studi e Ricerche di Sanita' dell'Esercito e Facolta' di Medicina Veterinaria dell'Universita' di Pisa).
3) Collaborazione con l'Ospedale Pediatrico del Bambino Gesu', Reparto di ematologia, per test specifici.

C) FINANZIAMENTI GLOBALI AVUTI NEGLI ANNI PRECEDENTI *In kEuro*

Anno finanziario	Missioni interne	Missioni estere	Materiale di consumo	Trasporti e facchinaggi	Spese di calcolo	Affitti e manutenzione	Materiale inventariabile	Costruzione apparati	***TOTALE***
2003	**3,0**	**9,0**	**16,0**				**9,0**	**5,0**	**42,0**
2004	**12,0**	**9,0**	**30,0**				**7,0**	**6,0**	**64,0**
TOTALE	**15,0**	**18,0**	**46,0**				**16,0**	**11,0**	**106,0**

Mod EC. 5 (a cura del rappresentante nazionale)

Proceedings of the 5th Meeting of Japan CF Research Society

Edited by Hiroshi YAMADA

December 15-16, 2003

Kobe University, Japan

FURTHER TESTS ON COMPOSITION AND ISOTOPIC ANOMALIES WHEN PD THIN CATHODES ARE ELECTROLYZED IN ACIDIC C_2H_5OD/D_2O MIXTURES ADDED WITH TH-HG SALTS AT MICROMOLAR CONCENTRATION.

Francesco Celani[(1)], A.Spallone[(1)], P.Marini[(2)], V.di Stefano[(2)], M.Nakamura[(2)], A.Mancini[(3)], P.G.Sona[(4)], E.Righi[(1)], G.Trenta[(1)], C.Catena[(1)], G.D'Agostaro[(1)], P.Quercia[(1)], V.Andreassi[(1)], F.Fontana[(5)], L.Gamberale[(5)], D.Garbelli[(5)], E.Celia[(6)], F.Falcioni[(6)], M.Marchesini[(6)], E.Novaro[(6)], U. Mastromatteo[(7)].

INFN-LNF Via E.Fermi 40, 00044 Frascati (Rome)- Italy; (2) EURESYS, Via Lero 30, 00129, Rome-Italy; ORIM SrL, Via Concordia 65, 62100 Piediripa (Macerata)- Italy ; (4) Via S. Carlo 12, 20090 Segrate (Milan) Italy; (5) Pirelli Labs SpA, Viale Sarca 222, 20126 Milan-Italy; (6) Centro Sviluppo Materiali SpA, Via di Castel Romano 100, 00129 Rome-Italy; (7) STMicroelectronics, Via Tolomeo 1, 20010 Cornaredo (Milan)- Italy.

Abstract: Following the recent ICCF10 Conference held at Boston (USA) on August 2003, we made a new experiment aimed to confirm our previous data[1, 2)] showing production of new elements sometimes with non-natural isotopic composition. During the experiment a long (~60 cm) and thin (diameter 0.05mm) Pd wire is (over)loaded and unloaded several times with Deuterium (D). The materials of cell (i.e. Pd and Pt wires, solution and salts) were analysed by a high resolution ICP-MS. As already detailed in the http://www.iccf10.org report by the same Authors, the composition of electrolyte (C_2H_5OD/D_2O slightly acidic mixtures with addition of Th-Hg salts at micromolar concentration) and electrolytic current density ($10mA/cm^2$) were quite unusual.
Moreover, the D/Pd-loading ratio is estimated by measuring the Pd wire resistance. In order to measure the resistance, an AC current flows through the Pd wire (square wave at 10KHz, of quite large intensity ~ $800A/cm^2$ and, for about 30minutes every day, up to $5000A/cm^2$).
We realised that such large electromigration-like current can be efficient also to increase the D flux through the surface, similarly (and perhaps in a quite "stronger" way in some very successful experiments) to gas loading methods, at least from the point of view of "transmutation". In our "best experiment", following the calculation procedure introduced by Iwamura[3)], we calculated the "equivalent cross section" of $^{232}Th \rightarrow {}^{208}Pb$ as large as 600barn in Th-Hg experiment. Obviously, we had to adapt such calculation procedure to our experimental set-up conditions: we suppose that all D, produced by electrolysis, cross the Pd wire surface, electro-migrate through the whole wire and is "expelled" at wires end.
A completely new, transparent borosilikat (3.3) glass cell thermal insulated by a vacuum chamber was built. The most recent experimental results, after one month of operations, were reported and the general description of our procedure is discussed.
Our studies by ICP-MS analysis followed both the experimental results of Y. Iwamura[3)] (Mitsubishi Heavy Industries, Yokohama) -with replication[4)] at Osaka University- and the theoretical model developed from A. Takahashi (University of Osaka[5)]) of a "multi-body resonance fusion of deuterons" related to specific element "transmutation" in a proper multi-layers of Pd-CaO-Pd-Sr (or Cs) subjected to prolonged deuterium gas flow (according to Y. Iwamura procedures).

Keywords: PdDx, electrolytic hydro-alcoholic solution, Pd thin-long wire, Sr-Hg and Th-Hg "transmutation".

1. INTRODUCTION

During the overloading experiments (Pd-Deuterium) we changed from usual water based electrolyte to hydro-alcoholic solution since 1999. The scientific explanation for the use of such an unconventional electrolyte are stated in detail in our previous Reports at JCF (3, 4) and ICCF(8, 9, 10) series and related papers.
We used a (*very unconventional in all their aspects*) main solution made of heavy ethyl alcohol (C_2H_5OD) and heavy water (D_2O) with a ratio 90—95% to 10—5%. The main dissolved cations were Strontium (as $SrCl_2$) and Mercury (as $HgCl_2$), at a typical concentration as low as 10 micromolar and 1 micromolar respectively. The pH was kept around 4.
For the sake of comparison, most of the electrolytic "Cold Fusion" experiments (several thousands) performed in the world use pure D_2O and LiOD at a concentration of 0.1÷1 molar, i.e. giving strong basic solutions (pH $\simeq$ 13 ÷ 14). This kind of solutions follow the "teachings" of Martin Fleischmann and Stanley Pons (Utah University-USA) who in 1989 first showed the possible "nuclear" origin of some anomalous heat in their electrolytic cell.
Moreover, we use as cathode Pd electrodes consisting of wires 50 to 100 cm long with diameter as thin as 50μm instead of the most diffused rods (according to M. Fleischmann and S. Pons) or plates (according to Akito Takahashi, Osaka University-Japan, 1992). We experienced that some suggestions from Giuliano Preparata and Emilio Del Giudice (University and INFN Milan, 1994) about the peculiarity of long-thin wires are substantially effective.
As referred in detail in the papers quoted, we found excess heat (see ref. 22 in Ref. 1) and tritium well above background (see ref. 24 in Ref.1) in a series of experiments with hydro-alcoholic solutions

containing small amounts of Sr salts and Hg ions. Moreover, we found that in the hydro-alcoholic environment, during the anodic phases (that is for some hours every 1-3 days) of our loading cycles, the Pd electrode is eroded. Significant amounts of very fine Pd particles are found at the bottom of the cell at the end of the experiments. ICP-MS analysis after electrolysis showed the presence of Pd in this black powder (see ref. 26 in Ref.1).

Moreover, after several electrolytic loading-deloading cycles, the Pd wire eroded surface could absorb the deuterium dissolved in the solution (whose maximum overpressure is only 50mbar in our experimental apparatus), quickly and with no electrolysis current so as to yield a mean D/Pd ratio up to 0.75. Such a behaviour could result from an increase of the catalytic activity of the Pd surface. We would like to point out that Yoshiaki Arata[6] (University of Osaka- Japan) has stressed the importance of *nano-structures* in order to get loading of Deuterium in Pd (even over 1/1 loading ratio). Accordingly, very large amounts of "anomalous excess heat" and ^{4}He were observed in some of his experiments when a proper "trigger" was applied. It is reasonable to suggest that the increased activity exhibited by our Pd wires is caused by the formation of *nano-structures* on its surface, similar to those obtained by Y. Arata in a more elaborated way.

We recall that Y. Iwamura, at Mitsubishi Heavy Industries in Yokohama-Japan (see ref. 25 in Ref. 1) **first showed that Sr is apparently transmuted into Mo (molybdenum),** or Cesium (Cs) into the rare heart Praseodymium (Pr) when a proper multilayer of Pd/Pd-CaO/**Sr** or **Pr** is forced to be flown by deuterium gas for enough long time (several hundred of hours). We tried to check whether such a "transmutation" could occur also after the repeated D-Pd loading/deloading/loading cycles in our experimental set-up. In July 2002 we were ready to perform an independent variant of the Iwamura experiment.

Before starting, we analysed by ICP-MS all the components present in the cell (C_2H_5OD, D_2O, $SrCl_2$, DCl, $HgCl_2$), and pieces of both the Pd cathode and the 2 Pt wires (used as anode and reference electrodes).

At the end of the repeated D-Pd loading/deloading experiment, the electrolytic solution was vacuum dried, the residue was collected and again analysed by ICP-MS together with the Pd. Molybdenum was found in excess of any conceivable contamination, the isotopic composition of the Mo was different from the natural one (see ref. 26 in Ref. 1).

It appears that the phenomenon first discovered by Y. Iwamura in a flowing deuterium gas system also occurs in our electrolytic cell when it is operated according to loading-deloading-loading procedure for a time length of the order of 500-1000 hours.

The main improvement performed during the 2003 was the use of Thorium (Th) as main electrolyte instead of Strontium, again in a very low concentration range (of the order of a few tenths of micromoles/liter).

1.1. Experiment: Thorium salts as electrolyte

In January 2003, we substituted Th salts for the Sr salts used previously.

The reason for such kind of experiments were mainly two:

1) based on some results (published also from our group) on 1997-1998, indicating possible Th "transmutations" during high-electric power (and high-temperature, high-pressure) AC (50Hz) electrolysis with massive zirconium electrodes (both anode and cathode, see ref. 28 in Ref.1), we decided to test whether something similar could happen in our new experimental apparatus based on thin Pd wires and a very strict control of impurities;
2) like Sr, Th ions can form an inorganic precipitate on the cathode surface as $Th(OH)_4$ (solubility product $Ks = 10^{-50}$), by action of the current density. Another similarity to Sr^{+2} is that Th^{+4} ions cannot be galvanically deposited because of the highly negative value of their standard potential ($E_0 = -1.899V$). Moreover, even if Th is co-deposited on Pd surface through some unknown process, it can form a stable hydride-deuteride compound with supposedly no incompatibility with the aim of our specific experiment. According to such chemical-physical characteristics, it is possible to deposit the proper $Th(OD)_4$ and/or ThD_x layer(s) on the Pd cathode surface at a value of the current density lower than that required for Sr salts: the input power to the cell is reduced and anomalous excess heat, if any, can be more easily detected.

Accordingly, with the present work we intended to essay the following:

a) the occurrence of excess heat,
b) the presence, if any, of foreign elements in both the cathode and the cell after electrolytic loading.

The operations were performed with electrolytes containing small amounts of Th and Hg salts.

1.2. Experimental set- up: electrolytic cell and flow calorimeter

The cell configuration is shown in Fig. 1 of Ref.1. The sample holder, a PTFE tube, is placed in a 1000ml borosilikat glass (type 3.3) cylinder (diameter 67mm, height 460mm). The cathode and anode are both "U" shaped and are located on the opposite walls of the holder, facing each other. The cathode is a thin (diameter 0.050mm) long (60cm) Pd wire (total surface about 1cm^2; resistance about 30 Ohm with no Deuterium inside). In the lower part of the "U", at its center, a small weight (6g PTFE cylinder) keeps the wire tense during the Pd loading so as to compensate its 4--6% elongation. The anode is a Pt wire (diameter 0.250mm, length 60 cm, purity 99.99%). A

third Pt wire (diameter 0.250mm, length 30cm) is put exactly in the middle of the "U" shaped cathode for reference purposes.
To measure the cathode resistance, an AC current (16mA, 10KHz, square wave: equivalent to a current density along the wire as high as 800A/cm^2) is superimposed to the (low intensity) electrolysis DC current (2—20mA): the resistance value is continuously monitored using the resulting AC voltage drop.
A high quality LM135H thermometer (sensitivity 0.05°C), inserted in a PTFE tube, is placed in the middle of the cell. A Joule heater (max. 20W) is used to calibrate the calorimeter and is located between the electrodes in a peripheral position. It consists of a chain of 20 resistors inserted in a PTFE tube (diameter 8mm, length 30cm) filled with thermal conducting grease.
The cell is pressurised (50mbar) and thermally insulated. The electrolysis gases and vapours flow through a twin cold-traps and a silicon oil bubblier before reaching the atmosphere. Corrections for these losses of energy are not yet applied, consequently all the data about excess heat are under-estimated.
The heat exchanger within the cell consists of a 500cm long PTFE pipe, outer/inner diameter 4/2mm, wound around the PTFE holder through which distilled water flows. Temperature of the distilled water flowing in the pipe is continuously measured at the inlet and outlet of the heat exchanger with two LM135H thermometers. A computerised peristaltic pump (Masterflex 7550-62) provides a constant flow of distilled water (0.200ml/sec, with day-to-day stability of ±1%, routinely measured every 12 hours). Water is picked up by the pump from a 2-liter reservoir to which it returns from the cell. The cell, water reservoir, and pump are placed in a container held at a constant temperature of 24°C. In other words, we continue (since 1992) to use the, very reliable, flow calorimetric measurement method.

2. COMPOSITION OF THE ELECTROLYTE, CLEANING PROCEDURE

A 93 to 7 % by volume mixture of ethyl alcohol (C_2H_5OD) and heavy water (D_2O) respectively was used as the electrolyte, having a total volume of 750 ml.

- The ethyl alcohol was previously vacuum distilled at 35°C (by a vacuum distillation system Buchi 134) in order to eliminate mainly sodium and iron and ultra-filtered on-line using a 100nm, MILLIPORE PTFE filter: distillation system deeply modified in our Frascati laboratory. The density was routinely measured (Mettler Toledo DA-110M) before and after distillation, to confirm that no significant H_2O contamination occurred during the operations.
- The heavy water, 99.97% isotopic purity (reactor grade from Ontario Hydro, Canada), was distilled at 45°C under vacuum and ultra-filtered before use, similarly to alcohol. Density was measured before and after distillation.
- $Th(NO_3)_4$, (5 ÷ 15mg) was added to the electrolyte and the pH of the resulting hydro-alcoholic solution was adjusted to a value of about 3 by adding few drops of concentrated HNO_3, in order to avoid uncontrolled precipitation of $Th(OD)_4$.
- The cell was cleaned after each experiment using repeated cycles of water/organic solvents/water/nitric acid/water in an ultrasonic warm bath. After Experiment #2 (Feb. 14, 2003; see Table 1a), we increased the duration of the immersion in concentrated (65%) warm (60°C) HNO_3 from 2 minutes to 14 hours because we suspected that a residual amount of Cs might be hidden somewhere in the cell. As a consequence, the final washing cycles with distilled water were increased from 4 to over 10 because we experienced that warm, long time lasting, HNO_3 is absorbed by the non-glass parts of the cell.

3. EXPERIMENTAL RESULTS

We got both excess heat (best result was an energy gain of about 10 for several days) and several strong indications of the "production" of new elements, some of these with an isotopic composition different from the natural one.
Some of the new elements "produced" (Cu, Zn) are the same as those obtained by using Sr as the main electrolyte, others are peculiar respectively of Strontium (i.e. Mo) or of Thorium (i.e. Pb)
Surprisingly, we found that the electrolyte consumption was much larger than could be expected according to the Faraday's law. We suspect that this loss is caused by the heat being generated in a few hot spots at wire surface, where the temperature might raise to rather high values. This local over-heating could evaporate the solution so that gaseous D_2O and C_2H_5OD are lost in addition to D_2 and O_2.
We plan to build a new cell made out of PTFE, quartz, HDPE, and mylar, and use a thermo-camera in order to detect the IR radiation that might issue from the deuterated Pd wire surface.

3.1. ICP-MS Measurements.

ICP-MS measurements were performed in a chemistry laboratory operating under the ISO 9001 quality control protocol. The laboratory is located in the Interdisciplinary Research Area of Castel Romano (Rome), in the "Centro Sviluppo Materiali" building. The ICP-MS used is HP & YOKOGAWA Analytical Systems, model 4500. It has been in operation since 1996. Calibrations with Atomic Absorption Standards are made every day before starting analysis. Sensitivity is about $6{\cdot}10^{10}$ Atoms/count. Typical background is 10 - 80 counts, depending both on the mass analyzed and the "overall condition" of the instrument (mainly high voltage

setting and the Ar carrier gas flow intensity). Background is higher for well known sources of interference, due to Ar, Cl, O, N, C, H, and impurities dissolved in the deionised "Milli-Q" water and reagents used. Because of our specific experimental constraints, we almost always use hot, concentrated Aqua Regia (4cc used for about 10-20mg of material) to dissolve the materials in glass beakers (borosilikat 3.3). A nominal concentrations of 2% and 0.2% (to avoid instrumental saturation) is submitted to the instrument for analysis of the main elements in the electrolyte (Pd, Sr, Th, Hg). In other words, we have about 10--20mg of unknown elements diluted in 50cc (or 500cc) of acidic water. We make 2 runs, from mass 2 to 260 with a resolution of 0.2 AMU. Each run takes about 200 seconds and sample uptake is 0.5ml/min. Blank and mass tuning runs (^{7}Li, ^{89}Y, ^{206}Tl) are made at the beginning of the analysis, after 3 hours of operation, and at the end of the study. Calibration is done more frequently if an element is at a particularly high concentration. After each measurement there is a washing cycle of about 10 minutes using HNO_3 at 5% concentration or Aqua Regia if needed. A blank measurement is always performed.
Data are analyzed according to the general recommendation of ICP-MS manufacturer applied to our specific operating conditions. We especially want to avoid spurious results caused by double mass and half-mass signals adding to the masses of interest. To solve such problems, we performed a large number of analyses using standard solutions of the elements of interest to calibrate the apparatus. Obviously, D_2O and C_2H_5OD were also carefully characterized.

3.2. Comments on ICP-MS experimental result

The ICP-MS results are shown in Tab.1 and Tab 2.
We studied 3 main reactions: Sr-->Mo, Cs-->Pr, Th-->Pb (+other).

Sr-->Mo

Results using Sr within the electrolyte were reported in ref.26 of Ref.1. We observed that some of the Sr was transmuted to elements with mass 94 and 96, qualitatively in agreement with the Y. Iwamura results. Moreover, the total amount of Mo atoms we found ($1\text{-}2\cdot10^{15}$), normalized to Pd electrode surface (about $1cm^2$), is very similar to the Iwamura gas experiments.

Cs-->Pr

Transmutation of Cs to Pr (according to Y. Iwamura) was not observed in our experiments when we used concentrated Cs solutions, because in the ensuing conditions we were not able to achieve a sufficiently high deuterium concentrations within the cathode. Much to our surprise, results were better when a Th salt, at very low concentration, was used with a "proper mixture" of Ca and Sr salts. Using these mixtures, we apparently transmuted Cs and Pr, although in smaller amounts in respect to Iwamura procedure. We noticed that the Th salt did not contain measurable impurities of Cs or Pr, according to both the assay from the chemical company (Aldrich) and our analysis by ICP-MS. In the best experiment in which apparent transmutations have been detected, the signal is nine times the background.
Later (in experiments #5 and #6) we used only Th and Hg salts to increase the deuterium concentration in Pd. Surprisingly in experiment #6 we observed for Pr a signal to background ratio as large as 21. Experiment #4 did not achieve the necessary deuterium concentration, making it a very useful blank for ICP-MS analysis.
In other words, apparent transmutations of Th to Cs (like "fission") and later Cs to Pr (like "fusion" of $Cs+2^4He$) occurred in an electrochemical environment, similar to those reported by Y. Iwamura in a gaseous environment[3] (see also ref. 25 in Ref.1) and recently confirmed by A. Takahashi[4] *et. al.* about the speculated reaction $Cs+2^4He$-->Pr. Such kind of, very surprising, 2 step reactions need to be confirmed by more experiments and cross-checked with other kind of analysis.

Th-->Pb(+other)

We performed a total of 4 experiments with Th salts, in amounts ranging from $8x10^{-6}$ to $6.5x10^{-5}$ moles in the total volume of electrolyte. The pH for the Th solution was $\simeq$ 3 compared to $\simeq$ 4 for the Sr containing solution.

3.2.1. Details on ICP-MS results shown in Tab.1 and 2.

A list of masses relatively easy to identify is reported in Tab. 1 and Tab. 2. For this masses reproducible results were obtained. We observed several other anomalies not yet fully understood. We anticipate that most of such anomalies are in the range of masses 46 - 60, 107 - 116, along with some isotopic anomaly for Pd.

4. Conclusions

After a large number of experiments performed during 14 years of research work aimed at finding anomalous effects in systems forced to a high concentration of deuterium, we are confident that most of the observed effects occur at the interface between the solution and the Pd bulk. ***A properly formed layer of a third element is necessary. Non-equilibrium situations are also necessary to trigger the effects***. Recently, we found that deuterated hydro-alcoholic, slightly acidic solutions, work very well in producing the so called "anomalous effects". Additions of Th and Hg salts within the micromolar concentration range improve the effects even at very low electrolytic current density ($<10mA/cm^2$).

The so-called "transmutations" seem real and not an artifact. Moreover, the amount and type of new elements seem related to:

a) value of overloading and its time duration;
b) intensity of D "flux" at its interface (in our case improved because "bulk" electromigration along wire and repeated cycles of cathodic-anodic stripping);
c) the type of main electrolyte (e.g. Sr, Th);
d) a proper "third" element at Pd surface , i.e. CaO according to Ywamura, Hg in our experiment.

We think that the model developed by Akito Takahashi[5] about multi-body resonance fusion of deuterons can explain most of the thermal and isotopic anomalies, including foreign elements that we have recently observed.
Further work is necessary to fully characterize the system and increase the magnitude of the effects.

Table 1: Results of ICP-MS analysis. Please note that overloading means D/Pd>0.9.

Exp	Date: begin→ end [dd/mm/yy]	Electrolyte composition: [moles]	Comments: OVL=Overloading S/N=Signal/Noise Pr=1count=$6\cdot10^{10}$ Atoms;
#1	20/12/02 → 16/01/03	$CsNO_3[5\cdot10^{-5}]$ - $SrCl_2[1\cdot10^{-5}]$ LiOD $[1.5\cdot10^{-5}]$ - $H_2SO_4[5\cdot10^{-6}]$ NH_3OH $[1\cdot10^{-4}]$	Almost No OVL ; Anode=Pd wire 250□m Pr=80=>S/N=2
#2	17/01/03 → 14/02/03	$CaCl_2$ $[21\cdot10^{-5}]$ - $SrCl_2$ $[1\cdot10^{-4}]$ $HgCl_2$ $[2\cdot10^{4}]$ – H_2SO_4 $[1\cdot10^{-5}]$	2 times OVL, - Residual Cs?? Pr=170=>S/N=4
#3	18/02/03 → 05/03/03	$CaCl_2$ $[1\cdot10^{-5}]$ - $SrCl_2$ $[1\cdot10^{-4}]$ $HgCl_2$ $[2\cdot10^{-4}]$ -H_2SO_4 $[1\cdot10^{-5}]$ $Th(NO_3)_4$ $[8\cdot10^{-6}]$	3 times OVL R/Ro=1.706 Pr=370=> S/N=9
#4	04/04/03 → 14/04/03	$HgCl_2$ $[1\cdot10^{-5}]$ -Hg_2SO_4 $[1\cdot10^{-4}]$ $Th(NO_3)_4$ $[2.1\cdot10^{-5}]$	No OVL
#5	15/04/03 → 19/05/03	$Th(NO_3)_4$ $[2\cdot10^{-6}]$ Hg_2SO_4 $[2\cdot10^{-6}]$	Several times OVL; Excess heat; Pr=300=>S/N=6
#6	24/05/03 → 14/07/03	$Th(NO_3)_4$ $[2\cdot10^{-6}]$ Hg_2SO_4 $[5\cdot10^{-6}]$	Several times OVL - Excess heat. Pr=1.4E3=>S/N=21 - 208Pb anomaly
#7	31/10/03 → 08/12:03	$Th(NO_3)_4$ $[21\cdot10^{-6}]$ Hg_2SO_4 $[3\cdot10^{-6}]$	Few times OVL (contamination Na)

Table 2: The counts of main mass found are shown. In parenthesis the natural abundance. See Ref. 26 for composition of solution.

Exp	^{31}P(100)	^{39}K(93.3)	^{63}Cu(69.2) ^{65}Cu(30.8) 63/65=2.25	^{64}Zn(48.6)^{66}Zn(27.9) ^{67}Zn(4.1)^{68}Zn(18.8)	^{133}Cs(100)	^{206}Pb(24.1) ^{207}Pb(22.1) ^{208}Pb(52.4) 206/208=.46
#1	$7\cdot10^{3}$	$2.2\cdot10^{6}$	$2.4\cdot10^{6}$	$2.7\cdot10^{6}$	$>3\cdot10^{8}$	$1\cdot10^{4}$
#2	0	0	$2.1\cdot10^{6}$ 63/65=1.94	$1.8\cdot10^{6}$	$6\cdot10^{5}$	$4\cdot10^{5}$
#3	$4\cdot10^{5}$	$2.7\cdot10^{6}$	$6.1\cdot10^{6}$ 63/65=1.93	$1.7\cdot10^{7}$	$1\cdot10^{6}$	$9.5\cdot10^{5}$
#4	$2.5\cdot10^{4}$	$1.4\cdot10^{6}$	$2.9\cdot10^{6}$ 63/65=2.05	$7.5\cdot10^{3}$	$1.94\cdot10^{5}$	$6.9\cdot10^{5}$
#5	$1.8\cdot10^{6}$	$2.3\cdot10^{6}$	$2.4\cdot10^{7}$ 63/65=2.07	$1.2\cdot10^{7}$	$9\cdot10^{4}$	$1.5\cdot10^{6}$
#6	$2.72\cdot10^{7}$	$6.4\cdot10^{6}$	$9.3\cdot10^{8}$ 63/65=2.08	$5.1\cdot10^{8}$	$2.2\cdot10^{5}$	$2\cdot10^{8}$ 206/208=.39
#7	0	0	$3.9\cdot10^{6}$ 63/65=2.0	$3.6\cdot10^{6}$	$2\cdot10^{4}$	$1.25\cdot10^{6}$

References

1) F. Celani, et al. Proceeding - JCF4, October 17-18, 2002, pp.17-21. Iwate Univ. (Japan).
2) F. Celani, et al. Proceeding - ICCF10, Cambridge (USA), August 23-29, 2003; publishing.
3) Y. Iwamura, et al. Proceeding - ICCF10, Cambridge (USA), August 23-29, 2003; publishing.
4) T. Higashiyama,...A. Takahashi, Proceeding - ICCF10, Cambridge (USA), August 23-29, 2003; publishing
5) A. Takahashi, Proceeding - ICCF10, Cambridge (USA), August 23-29, 2003; publishing.
6) Y. Arata, Y. Chang Zhang, Proceeding - ICCF10, Cambridge (USA), August 23-29, 2003; publishing.

Iwamura, Y., et al. *Low Energy Nuclear Transmutation In Condensed Matter Induced By D2 Gas Permeation Through Pd Complexes: Correlation Between Deuterium Flux And Nuclear Products*. in *Tenth International Conference on Cold Fusion*. 2003. Cambridge, MA: LENR-CANR.org. This paper was presented at the 10th International Conference on Cold Fusion. It may be different from the version published by World Scientific, Inc (2003) in the official Proceedings of the conference.

Low Energy Nuclear Transmutation In Condensed Matter Induced By D_2 Gas Permeation Through Pd Complexes: Correlation Between Deuterium Flux And Nuclear Products

Y. IWAMURA, T. ITOH, M. SAKANO, S. SAKAI, S. KURIBAYASHI

Advanced Technology Research Center, Mitsubishi Heavy Industries, Ltd.
1-8-1, Sachiura, Kanazawa-ku, Yokohama, 236-8515, Japan
iwamura@atrc.mhi.co.jp

Observations of low energy nuclear reactions induced by D_2 gas permeation through Pd complexes (Pd/CaO/Pd) were presented at ICCF-9[1] and in a paper[2] published in the Japanese Journal of Applied Physics (JJAP). When Cs was added on the surface of a Pd complex, Pr emerged on the surface while Cs decreased after the Pd complex was subjected to D_2 gas permeation. When Sr was added to the surface, Mo emerged while the Sr decreased after D_2 gas permeation. The isotopic composition of the detected Mo was different from the natural abundance.
In this paper, recent progress of our research is described. The detected Pr was confirmed by various methods such as TOF-SIMS, XANES, X-ray Fluorescence Spectrometry and ICP-MS. Analysis of the depth profile of Pr indicated that a very thin surface region up to 100 angstroms was the active transmutation zone. Many experimental results showed that the quantity of Pr was proportional to the deuterium flux through Pd complex. The cross section of transmutation of Cs into Pr can be roughly estimated at 1 barn if we consider the deuterium flux as an ultra low energy deuteron beam.

1 Introduction

Anomalous elemental changes have been observed on the Pd complexes, which consist of a thin Pd layer, alternating CaO and Pd layers and bulk Pd, after subjecting the Pd complexes to D_2 gas permeation as we reported at ICCF-9[1] and in the paper[2] in the Japanese Journal of Applied Physics (JJAP).

In this paper, we describe recent progress. The following points have been improved or changed.

1) Pr was identified by XPS, TOF-SIMS, XANES, X-ray Fluorescence and ICP-MS.
2) A quantitative analysis of Pr has became possible using ICP-MS.
3) The correlation between deuterium flux and Pr was investigated.
4) Cs ion injection into Pd complexes (instead of the electrochemical method) was performed.
5) Depth profile and surface distribution of Cs and Pr was obtained by TOF-SIMS (Time of Flight Secondary Ion Mass Spectrometry).

Our experimental method can be characterized by the following two main features. The first is the permeation of D_2 gas through the Pd complex, as shown in Fig. 1(a). Permeation of deuterium is attained by exposing one side of the Pd complex to D_2 gas while maintaining the other side under vacuum conditions. On the D_2 gas side of the Pd complex, dissociative absorption causes the D_2 molecules to separate into D atoms, which diffuse through the metal toward the vacuum side, where they emerge from the metal, combine and are released as D_2 gas.

The second feature is the addition of an element that is specifically targeted to be transmuted. Our sample is a Pd complex composed of bulk Pd on the bottom, alternating CaO and Pd layers, and a Pd thin film on top. After fabricating a Pd complex, Cs or Sr is deposited on the surface of the top thin Pd layer, as shown in Fig. 1(b). This Cs or Sr is transmuted. In other words, with this composition, we can provide a deuterium flux through the Pd complex on which a target element is placed as a target to be transmuted. We perform elemental analyses of the given elements after D_2 gas permeation by exhausting the D_2 chamber (by making it into a vacuum chamber). Our experimental method is superior in that it clearly discriminates transmutation products from

contamination because we analyze the products by XPS (X-ray Photoelectron Spectroscopy) in vacuum, in situ during the experiment, without moving or the sample or opening the chamber.

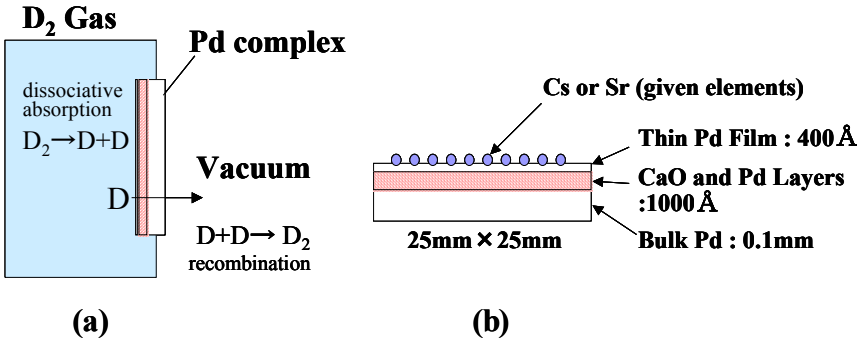


Figure 1. Schematic of the present method: (a)D2 gas permeation of the Pd complex, (b)Structure of the Pd complex deposited with Cs or Sr

2 Experimental

The experimental method and setup are basically the same as before[1,2]. Therefore we shall omit a detailed description, and describe only the changed and improved aspects of the experiment.

Cs is now added to the surface by the ion injection method, in addition to the electrochemical method, for exact depth profile analysis.

Figure 2 shows the experimental apparatus. The D_2 gas flow rate was estimated by measuring the pressure of the chamber B. (Chamber B is evacuated, but the vacuum is gradually filled with the gas that permeates through the Pd complex.) The calibration curve for pressure versus the D_2 gas flow rate was obtained in advance by letting D_2 gas into the vacuum chamber through a precision gas flow meter.

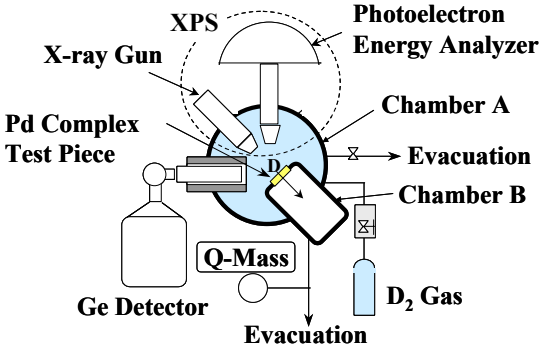


Figure 2. Experimental Setup.

3 Results and Discussion

Let us briefly describe the experimental results presented[2] at ICCF-9. A transmutation reaction converting Cs into Pr is shown in Fig. 3. Results for two runs are shown as examples. The number of Cs atoms decreased while the number of Pr atoms increased over time. No Pr was detected at the beginning of the experiments. At 120 h, the number of Pr atoms exceeded that of Cs atoms.

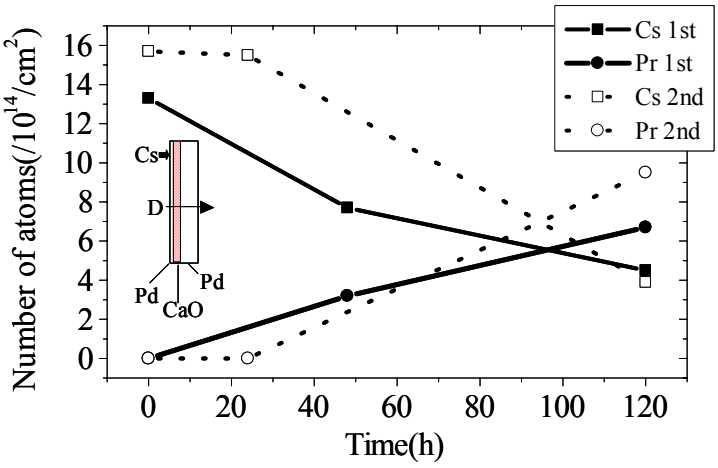


Figure 3. Time variation in the number of Cs and Pr atoms during D_2 gas permeation through Pd complex (Pd/CaO/Pd) deposited with Cs

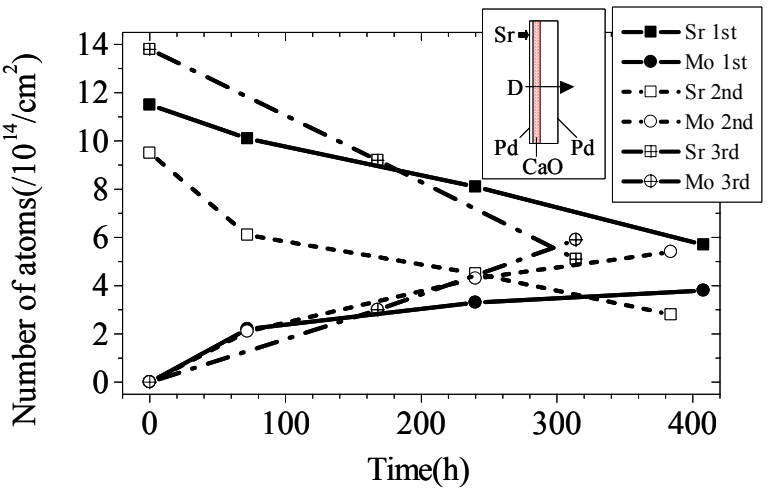


Figure 4. Time variation in number of Sr and Mo atoms induced by D_2 gas permeation through Pd complex(Pd/CaO/Pd) deposited with Sr

The experimental results for Pd complex test pieces with added Sr are shown in Fig. 4. We observed that Sr decreased while Mo increased over time. Experiments were performed three times and all data are plotted here. At the beginning of the experiments, no Mo atoms were detected. However, Mo atoms increased gradually while Sr decreased correspondingly. It should be noted that runs with Sr take longer to convert a given mass of Sr into Mo than it takes to convert that mass of Cs into Pr.

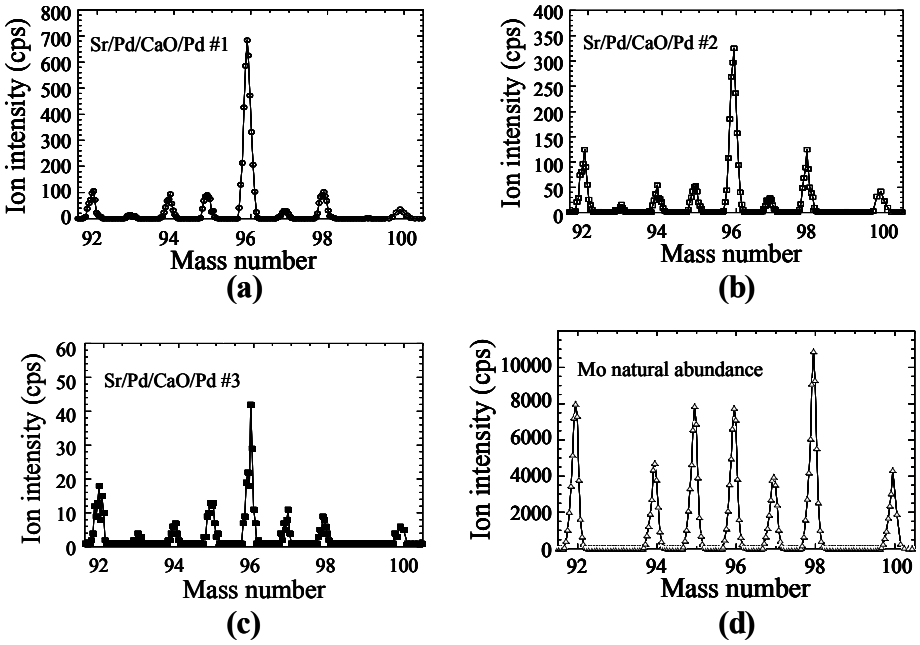


Figure 5. Anomalous isotopic composition of detected Mo: (a) Isotopic composition of detected Mo for run #1, (b)Isotopic composition of detected Mo for run #2, (c)Isotopic composition of detected Mo for run #3, (d) Natural abundance of Mo analyzed by SIMS.

Figures 5(a)-(c) show the results of SIMS analysis for the three samples. The intensities of mass number 96 were the largest for each sample, although the intensities were different. The SIMS mass spectrum for a Mo layer (400 Å thickness) that was deposited on a Pd disk is shown in Fig. 5(d). This spectrum reveals the natural abundance of Mo. Comparing these figures, we can easily recognize that the isotopic compositions of the detected Mo are different from the natural isotopic abundance of Mo.

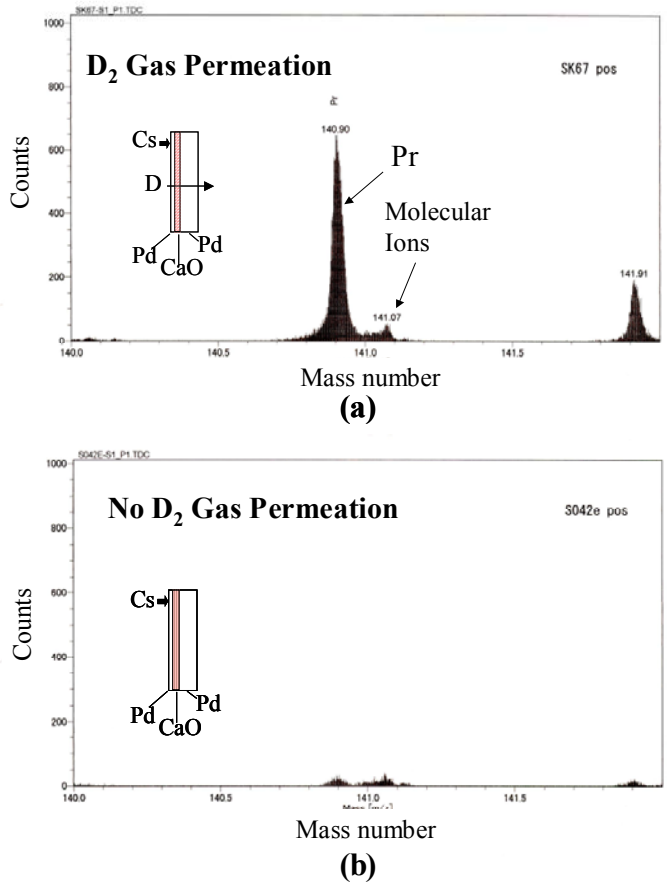


Figure 6. Identification of Pr by TOF-SIMS: (a) Mass number distribution of the sample after D_2 gas permeation, (b) Mass number distribution of the sample without D2 gas permeation

Let us move on to new experimental results. Pr, the transmuted product from Cs, was confirmed by many element analysis methods. The first example is the identification of Pr by TOF-SIMS (Time of Flight Secondary Ion Mass Spectrometry) shown in Fig. 6. The TOF-SIMS device is a model TRIFTTM II made by ULVAC-PHI. The upper figure shows the mass number distribution of the Pd complex (Pd/CaO/Pd) after D_2 gas permeation, and the lower figure is for the Pd complex without D_2 gas permeation. The TOF-SIMS can distinguish small mass difference so that Pr and molecular ions can be clearly separated, as shown in the upper figure. It is confirmed that Pr is detected only for the foreground sample.

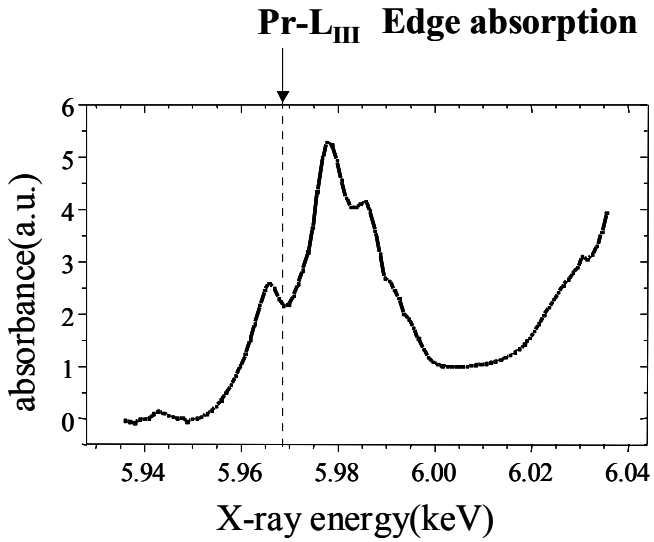


Figure 7. Identification of Pr by XANES (X-ray Absorption Near Edge Structure) .

Confirmation of Pr by XANES (X-ray Absorption Near Edge Structure) is shown in Fig. 7. This spectrum was obtained at the BL-9A Line at the High Energy Accelerator Research Organization (KEK), located in Tsukuba, Japan (www.kek.jp). A Pd complex sample after D_2 gas permeation, on which Pr was detected by XPS, was examined by XANES. Pr L_{III} Edge absorption was clearly recognized in Fig. 7

Furthermore, Pd complex samples after D_2 gas permeation were examined by X-ray fluorescence spectrometry and ICP-MS (Inductively Coupled Plasma Mass Spectrometry). Although the X-ray fluorescence spectrometry is a bulk analysis method, Pr was detected using strong SOR X-rays. The sensitivity of ICP-MS is so high that quantitative analysis of Pr is performed for all the experiments.

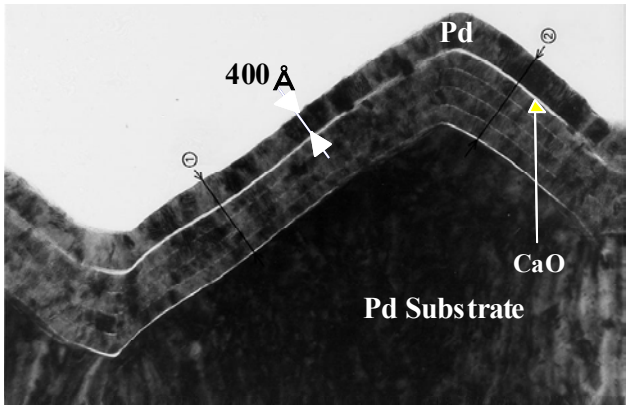


Figure 8. Cross section of Pd complex (Pd/CaO/Pd) observed by TEM (Transmission Electron Microscopy)

Figure 8 shows the cross sectional view of the Pd complex (Pd/CaO/Pd). This image was taken by TEM (Transmission Electron Microprobe). During the process of Pd complex fabrication, the Pd substrate is etched with aqua regia[1,2]. The wave-like shape of the Pd substrate is formed by the etching process. On the Pd

substrate, Pd and CaO complex layer are formed by Ar ion beam sputtering. The white lines correspond to CaO and the black parts to Pd. The 400-angstrom Pd thin film is located on the Pd and CaO complex layer.

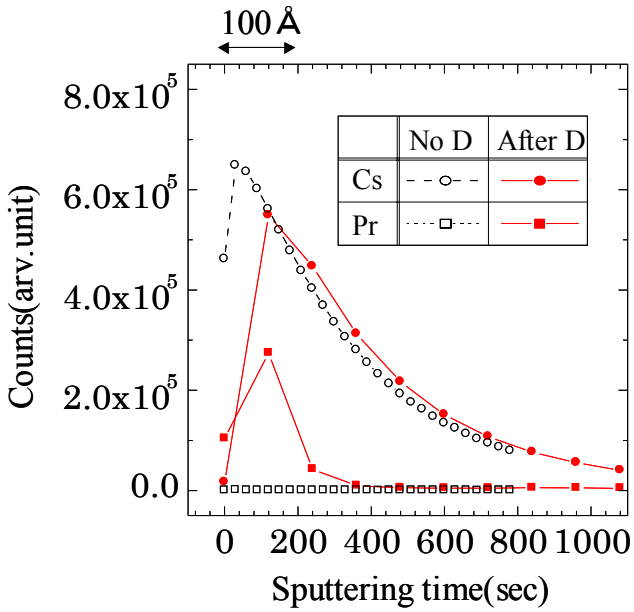


Figure 9. Depth Profiles of Cs and Pr for a Pd complex (Pd /CaO/Pd) sample after D2 gas permeation and a Pd complex (Pd /CaO/Pd) sample without D2 gas permeation

Depth profiles of Cs and Pr were plotted in Fig. 9. Two Pd complex samples were prepared and Cs was injected into them by the ion implantation method. Acceleration voltage and Cs fluence for the ion implantation were the same for the two samples, 18keV and 10^{15} ions/cm^2, respectively. The depth profiles were estimated by TOF-SIMS analysis. Physical Electronics TRIFT II was applied for the analysis and the condition of Ga^+ ion was 15keV-600pA. The relation between the sputtering time and the real depth was estimated in advance using a Pd thin film on Si substrate; thickness of the Pd thin film is known. This measurement shows that a 200 sec sputtering time corresponds to 100 angstroms.

Cs and Pr depth profiles for the Pd complex without permeation show normal results in Fig. 9. Cs decreases continuously from the surface and there is no Pr in the sample.

On the other hand, Cs and Pr depth profiles for the Pd complex after D_2 gas permeation exhibit interesting results. Cs depth profiles for the foreground and background samples agree in the deep area. However, Cs decreases near the surface after D_2 gas permeation. We can see that there is Pr, which is the same order as given Cs, in the near surface area. This experimental fact suggests that Cs transmutation reaction into Pr occurs in the near surface region up to 100 angstrom. This transmutation active zone might be correlated with the D/Pd ratio. Further investigation of the surface region is important. Figure 9 also shows that Cs atoms do not diffuse and migrate with D_2 gas permeation under our experimental conditions. Therefore it is very difficult to imagine that the detected Pr was a concentrated impurity, and not a transmutation product.

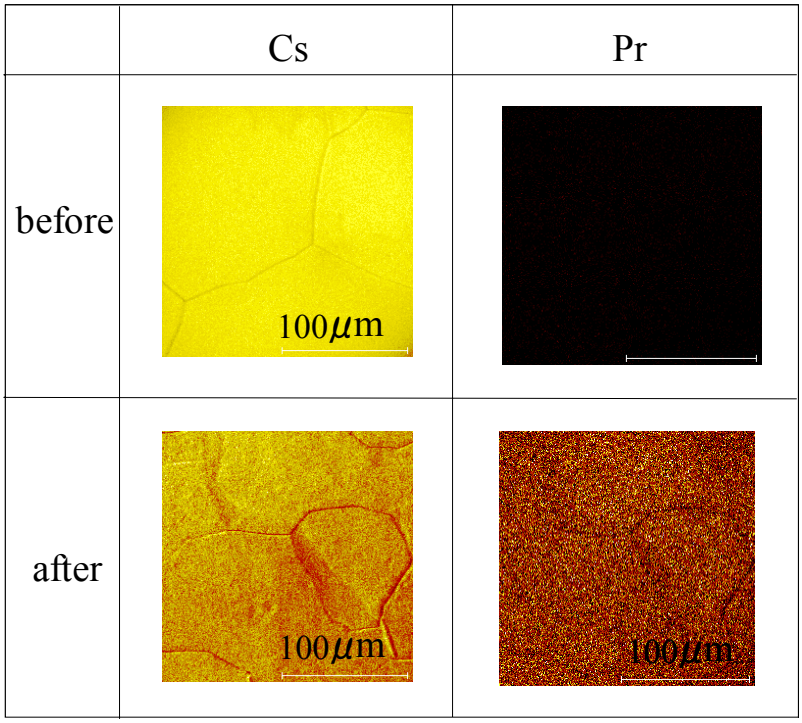


Figure 10. Surface Distributions of Cs and Pr for a Pd complex (Pd /CaO/Pd) sample after D_2 gas permeation and a Pd complex (Pd /CaO/Pd) sample without (before) D2 gas permeation

Figure 10 shows surface distributions of Cs and Pr for the two samples discussed above. Space resolving power is 1 micron. Grain boundaries can be seen in each image. These images show that the surface distribution of Pr basically seems to be uniform and has no correlation with the grain boundaries.

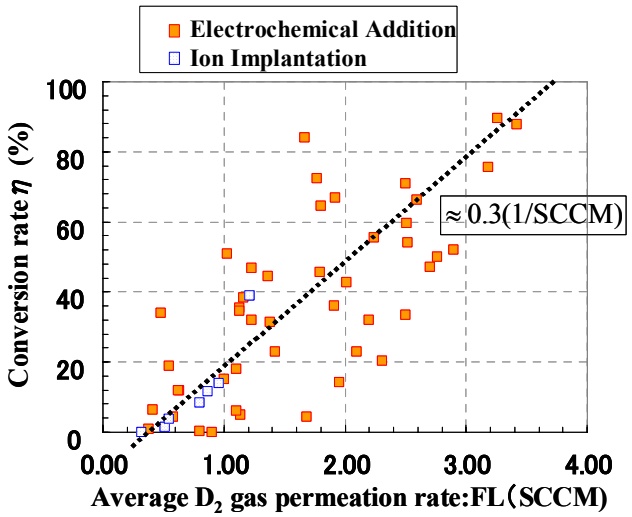


Figure 11. Correlation between D_2 permeation rate and conversion rate.

Using ICP-MS analysis, a quantitative estimate of the mass of Pr has been performed. The correlation between D_2 gas permeation rate and conversion rate is shown in Fig. 11.

The conversion rate is defined as

$$\eta = \frac{N_{\mathrm{Pr}}}{N_{Cs}} \times 100\% = \frac{N_{\mathrm{Pr}}}{N'_{Cs} + N_{\mathrm{Pr}}} \times 100\% ,$$

η : conversion rate(%), N_{Pr} : detected Pr(ng), N_{Cs} : given Cs(ng), N'_{Cs} : detected Cs after an experiment(ng). (1)

Since ICP-MS analysis is a destructive analysis method, we cannot measure the starting mass of Cs directly. Assuming that a Cs atom is transmuted into a Pr atom, the sum of the detected Pr and the detected Cs after permeation should be equal to the starting Cs.

Figure 11 suggests that the conversion rate defined as above is proportional to the average D_2 gas permeation rate. Experimental results for both the electrochemical addition and the ion implantation of Cs are plotted in the figure. It seems that they have linear correlation for the both cases.

Let us consider on the situation that D beam irradiates the Pd complex with Cs. The reaction rate is expressed as the following equation.

$$R = \sigma \cdot N_{Cs} \cdot \phi$$

R : reaction rate(event/cm^3/sec), σ : cross section(cm^2), N_{Cs} : number of Cs(1/cm^3), ϕ : deuteron beam flux (1/cm^2/sec). (2)

If we regard D_2 gas permeation as a kind of deuteron beam, the following relation is obtained by equation (2).

$$\eta = \int_t (R / N_{Cs})dt = \int_t (\sigma \cdot \phi)dt = \sigma \int_t \phi dt \approx \sigma \cdot f \cdot FL \cdot T_{\exp} / S \qquad (3)$$

FL : flow rate(sccm), $T_{\exp}$: reactiontion time(sec), S : permeation surface area(cm^2).

$$\therefore \eta \propto FL$$

This equation agrees with the experimental results shown in Fig. 11. Therefore we can roughly estimate the cross section using the obtained experimental results. If we input experimental parameters into equation (3), we obtain

$$\begin{aligned} \eta &= \sigma \cdot f \cdot FL \cdot T_{\exp} / S \\ &= \sigma \cdot \frac{2 \times 6 \times 10^{23}}{22.4 \times 10^3 \times 60} \cdot FL \cdot 100 \times 3600 / 1.0 \\ &= \sigma \left[cm^2\right] \cdot FL[sccm] \cdot 3 \times 10^{23} \left[1/cm^2/sccm\right] \end{aligned} \qquad (4)$$

The experimental results show the gradient between FL and conversion rate is about 0.3(1/sccm). (The term sccm means standard cubic centimeter per minute.) Therefore the following result is obtained.

$$0.3 \approx \sigma \cdot 3 \times 10^{23} \qquad \therefore \sigma \approx 1 \times 10^{-24} \left[cm^2\right] = 1[barn]$$

This cross section seems to be extremely large if we take it into consideration that the transmutation reaction belongs to multi-body reactions. And we should notice that we regard the deuterium permeation velocity as deuteron velocity, and the deuteron flux is estimated relatively low, leading to very large cross section. If the deuteron behavior on the microscopic level in the Pd thin film could be clarified, a more precise physical model would be developed. In any case, on the macroscopic level deuterium permeation through Pd complex can be regarded similar to an ultra low deuteron beam, and the cross section of transmutation of Cs into Pr is estimated at 1 barn according to our experimental results.

We would like briefly touch on a few points, starting with the problem of discriminating contamination and transmutation products. Since the detected material, Pr, is a rare earth element, it is difficult to imagine that Pr accumulated on the Pd complex test samples by any ordinary process. As mentioned in Fig. 9, Cs atoms do not diffuse and migrate by D_2 gas permeation. Therefore it can be postulated that Pr atoms also do not migrate. The purity of our D_2 gas is over 99.6% and the most of the impurity in it is H_2. Other impurities detected by a mass spectrometer are N_2, D_2O, O_2, CO_2, CO and hydrocarbons; they are all under 10ppm. We analyzed Pd complex test pieces deposited with Cs by ICP-MS mass spectrometry and confirmed that Pr in the test samples was below the detection limit (0.1ng). On the other hand, the detected Pr ranges from 1ng to 100ng. The amount of the detected Pr exceeds the maximum possible contamination of Pr. Therefore we conclude the detected Pr was transmuted from Cs.

Our next point is that the isotope ratio of the synthesized elements is anomalous. The isotopic anomaly of the Mo is particularly strong evidence that this Mo was produced by some nuclear processes. Some might speculate that the anomalous isotope ratios were caused by Mo contamination undergoing some sort of isotopic separation process, leaving only ^{96}Mo to be detected. (See Fig. 5). However, such efficient isotope separation would not be possible.

We noticed that a certain rule exists between starting and produced elements[1,2]. The increase in mass number is 8, and the increase in atomic number is 4 in the case of Cs and Sr. It appears that 4d addition reactions occur. We also observed 2d and 6d addition transmutation reactions[3].

At present, we do not have a complete theory that can explain the experimental results without a few assumptions. The EQPET model [4,5] proposed by Prof. A. Takahashi can basically explain our experimental results, by assuming that a short lived, quasi-particle electron pair like Cooper-pair can be generated. The observed transmutation processes must belong to a new category of nuclear reactions in condensed matter. Therefore much more theoretical investigation is necessary.

4 Concluding Remarks

Nuclear transmutation of Cs into Pr and Sr into Mo can be observed during D_2 gas permeation through Pd Complexes. Pr was identified by various methods such as XPS, TOF-SIMS, XANES, X-ray fluorescence spectrometry and ICP-MS. A very thin surface region up to 100 angstroms was the active transmutation area, as determined by the analysis of depth profile of Pr. The quantity of Pr was proportional to deuterium flux through the Pd complex. The cross section of transmutation of Cs into Pr can be roughly estimated at 1 barn if we regard the deuterium flux as an ultra low energy deuteron beam.

Some replication experiments producing transmutation reactions of Cs into Pr or Sr into Mo were planning or presented for the ICCF10 conference[6,7]. Positive results were obtained not only in a gaseous environment[6] presented by Prof. A. Takahashi *et al.*, but also in an electrochemical environment[7] performed Dr. F. Celani's team.

Acknowledgments

The authors would like to acknowledge Prof. A. Takahashi, Dr. F. Clelani, Dr. I. Tanihata, Dr. T. Ishikawa, Dr. Y. Terada, Dr. K.S. Grabowski, Dr. G.K. Hubler, Prof. T. Okano, Dr. K. Fukutani, Prof. S. Tanaka, Prof. K. Okuno and Prof. J. Kasagi for their valuable discussions.

References

1. Y. Iwamura, M. Sakano and T. Itoh, *Elemental Analysis of Pd Complexes: Effects of D_2 gas permeation.* Jpn. J. Appl. Phys. **41** (2002), pp. 4642-4648.
2. Y. Iwamura, T. Itoh, M. Sakano and S. Sakai, *Observation of Low Energy Nuclear Reactions induced by D_2 gas permeation through Pd Complexes*. Proc. of ICCF9 19-24 May 2002, Beijing (China); pp.141-146.
3. Y. Iwamura, T. Itoh and M. Sakano, *Nuclear Products and Their Time Dependence Induced by Continuous Diffusion of Deuterium through Multi-layer Palladium Containing Low Work Function*. Proc. of ICCF8, 21-26 May 200 Lerici (Italy), SIF Conf. Proc.Vol.70, pp.141-146.
4. A. Takahashi, *Tetrahedral and octahedral resonance fusion under transient condensation of deuterons at lattice focal points*. Proc. of ICCF9 19-24 May 2002, Beijing (China); pp.343-348.
5. A. Takahashi. *Mechanism of Deuteron Cluster Fusion by EQPET model.* Proc. of ICCF10, in press.
6. T. Higashiyama et al., *Replication of MHI transmutation experiment by D_2 gas permeation through Pd complex.* Proc. of ICCF10, in press.
7. F. Celani et al., *Thermal and Isotopic Anomalies when Pd Cathodes are Electrolysed in Electrolytes Containing Th-Hg Salts Dissolved at Micromolar Concentration in C_2H_5OD/D_2O Mixtures*. Proc. of ICCF10, in press.

Celani, F., et al. *Thermal and Isotopic Anomalies when Pd Cathodes are Electrolysed in Electrolytes Containing Th-Hg Salts Dissolved at Micromolar Concentration in C2H5OD/D2O Mixtures*. in *Tenth International Conference on Cold Fusion*. 2003. Cambridge, MA: LENR-CANR.org. This paper was presented at the 10th International Conference on Cold Fusion. It may be different from the version published by World Scientific, Inc (2003) in the official Proceedings of the conference.

Thermal and Isotopic Anomalies when Pd Cathodes are Electrolyzed in Electrolytes Containing Th-Hg Salts Dissolved at Micromolar Concentration in C_2H_5OD/D_2O Mixtures.

F. CELANI, A. SPALLONE, E. RIGHI, G. TRENTA, C. CATENA,
G. D'AGOSTARO, P. QUERCIA, V. ANDREASSI
INFN-LNF, Via E.Fermi 40, 00044 Frascati (Rome)-Italy, e-mail: francesco.celani@lnf.infn.it

P. MARINI, V. DI STEFANO, M. NAKAMURA
EURESYS, Via Lero 40, 00144, Rome-Italy

A. MANCINI
ORIM SrL, Via Concordia 65, 62100 Piediripa (Macerata)- Italy

P.G. SONA
Via S. Carlo 12, 20090 Segrate (Milan)-Italy

F. FONTANA, L. GAMBERALE, D. GARBELLI
Pirelli Labs SpA, Viale Sarca 222, 20126 Milan-Italy

F. FALCIONI, M. MARCHESINI, E. NOVARO
Centro Sviluppo Materiali SpA, Via di Castel Romano 100, 00129 Rome-Italy

U. MASTROMATTEO
STMicroelectronics, Via Tolomeo 1, 20010 Cornaredo (Milan)-Italy

Abstract

Discussed in this paper is the evolution of work that started by using the M. Fleischmann and S. Pons method and ended by using thin palladium wires electrolyzed in an electrolyte consisting of slightly acidic heavy alcohol-water solution containing thorium (Th) and mercury (Hg) salts at micromolar concentrations. The resulting large and dynamic loading of the Pd wires was studied. The recent use of thorium instead of strontium resulted in thermal anomalies and detection of new elements in larger amounts. The results with Sr are qualitatively in agreement with what was found by Y. Iwamura (Mitsubishi Heavy Industries) using multilayers of Pd-CaO-Pd-Sr in flowing deuterium gas. Most results seem to be in agreement with a "multi-body resonance fusion of deuterons" model recently developed by A.Takahashi (Osaka University).

1 Introduction.

1.1 From heavy water D_20-LiOD to hydro-alcoholic Th-Hg electrolytes .

Since 2000, we have employed a very particular electrolyte for loading Pd with D. This electrolyte is an acidic hydro-alcoholic mixture with small additions of uncommon ions like strontium (Sr) at very low concentrations (10^{-5} -10^{-4}M) and mercury (Hg) at even lower concentrations (10^{-6} - 10^{-5}). Recently (January 2003) we replaced the strontium with thorium at a concentration near 10^{-5}M.

To explain why we used such a "strange" electrolyte, let us begin with a short history of our experimental approaches, with both key results and turning points.

1.2. Deep underground measurements and fracto-emission phenomena.

Just three days after Martin Fleischmann[1] and Stanley Pons[1] (Univ. of Utah, Salt Lake City, USA) announced their discovery, we constructed our first electrolytic cells based on the Fleischmann-Pons description.

Since April 15, 1989, studies were made in the deep-underground "Gran Sasso Laboratory" of INFN-Italy. The Pd rods were sometimes replaced by thin-walled Pd tubes in order to reduce the deuterium loading time and to induce the deuterons to flow from the outer surface in contact with the electrolyte to the empty inner surface. In addition, we tested Ti bars as described by S. E. Jones[2]. We[3, 4] were looking mainly for neutrons from D-D fusion using a very low background in the Gran Sasso, typically near 10^{-6}n*cm^{-2}*s^{-1}, which is about 1000 times lower than at sea level. We detected neutrons at 10^{-23} - 10^{-20} n/s, the so called "Jones level", with up to 10^{-17} n/s during some burst emissions. These effects were found when the electrolytic cell was operating in a variety of nonequilibrium conditions. A procedures consisting of first loading (about one hour after the start-up), rapid on-off cycles of the electrolysis current, AC electrolysis using high frequencies, cell irradiated by intense pulsed light sources, and fast cooling of the electrolyte by immersion of the cell into a vessel containing liquid nitrogen were used.

We also found neutron emission from Portland cement cured with heavy water and during the superconducting transitions of deuterated high temperature, YBCO type, superconductors[5]. S.E. Jones[6] also reported similar results. We tried to stimulate "extra neutrons" from deuterated YBCO using a weak (2200 n/s) Am-Be neutron source. This produced "extra neutrons" at high multiplicity (3) mainly during the superconductivity transitions[7], as in previous experiments[5].

All these experiences convinced us that most of the neutron emission results could be produced by the fracto-emission phenomena[4], well known since 1938 (with pioneering work in Germany), which is of high scientific interest but of no practical use because most of the positive results are associated with irreversible phenomena.

After 2 years of experiments in the Gran Sasso Laboratories, we realized that the D-Pd system could be thought of as "working" just like an Otto-Benz engine cycle. First, the Pd lattice is filled with the proper fuel (deuterium). Second, the deuterium is stuffed into the lattice, for example through electrolytic loading. Third, a triggering spark must occur, induced by transient phenomena, which will provide for the nuclear "firing". Finally, nuclear ashes are produced. Unfortunately, at that time (1991), both the necessary D-loading threshold value and the nature of a "really effective trigger" were unknowns that require a direct determination of the actual D/Pd loading ratio.

1.3. Search for excess heat: the A. Takahashi L-H electrolysis procedure.

Beginning in 1992, we started a search for anomalous heat generation by adopting the so called "saw-tooth, low-high current" electrolytic loading cycles as developed by Akito Takahashi[8]. We used LiOD 0.3M in D_2O, Pd plates of 25mm x 25mm x 1mm, and a maximum current density of about 0.5A/cm^2. During the experimental work with this technique, and after extensive discussions with A. Takahashi, we concluded that excess heat was related to mechanical hardening and to the type of surface texture present on the Pd plates. The experiments were performed in a "normal" (not underground) laboratory where we measured[9] by flow calorimetry excess heat up to 5-10% of applied power. We propose that "anomalous excess heat"is "triggered"

by applying to the cell a low-high electrolysis current. We believe that the effectiveness of Takahashi procedure is caused by non-equilibrium conditions even during weeks of operation.

1.4. μs pulsed electrolysis (INFN-LNF method) and use of thin Pd wires.

After these pioneer activities (1993), we came to the conclusion that the necessary and sufficient conditions for the Cold Fusion phenomena to occur can be expressed as follows:

a) Loading of D in the Pd lattice to give a D/Pd ratio >0.9.
b) "Motion" of deuterons at the cathode surface and/or into the Pd lattice without deloading.

Regarding the condition a), we set-up and tested (1993) a very interesting and original method: the "High Power, High Frequency, Pulsing Electrolysis" (HPPE), which could operate at a very high current density up to 12A/cm^2 using "Takahashi type" plates and several hundred A/cm^2 when applied to thin wires. This current produced a high cathodic overvoltage because, according to the Tafel law, the overvoltage is proportional to the logarithm of current density.

The home made pulsed power supply could deliver to the cathode high voltage and high current pulses up to 250V and 150A. In addition, these pulses had a rise-time of <100ns at a peak current of less than 40A, a duration of 500--5000ns, and a repetition rate of 100-30000Hz. The overall maximum duty-cycle was 5% and the power was 80W.

The first tests using this technique were applied to cathode plates[10, 11, 12]. Unfortunately, continuous monitoring of the D loading could not be obtained using the electrical resistance of the cathode (see below, Ref. 13) because the resistence of the plates was too low. On the other hand, Coulomb-metric measurements[9, 10, 11, 12] give accurate results only at the beginning of the loading, when the deuterium absorbed is relatively large with respect to the total discharged D. When the loading ratio is high and the loading rate is low, i.e. when it is most interesting to know exactly what is happening, the accuracy becomes unacceptably low.

Later on, considering the capital importance of a direct and continuous evaluation of D/Pd ratio, and after profitable discussions with Giuliano Preparata[16] and Emilio del Giudice (University and INFN at Milan-Italy), we used thin cathodes (50-100μm thick) and long Pd wires because of their sufficiently high resistance.

Thin wires give a number of advantages.

- Continuous monitoring of the actual loading ratio in the Pd cathodes is possible. In fact, it is known that a precise relationship exists between the Pd electrical resistivity and the loading ratio x (at fixed temperature), the so called "Baranowsky curve". The Pd electrical resistivity is usually expressed as R/R_0, where R is the resistivity at a H/Pd ratio and R_0 is the initial resistivity. Therefore, it is possible to follow the progress of the loading process by measuring the cathode resistance .
- A flux of deuterons can be created. When a dc or ac current flows through a thin Pd cathode, the voltage drop forces the deuterons to move inside the Pd lattice (ref. 13, 14, 15). Because of the skin effect, this variation on the Cohen effect is magnified when a square wave, i.e. one having a fast rise time, is used instead of the sinusoidal current pulse.

By operating with thin Pd wires, both of the above conditions could be met.. The typical energy gain obtained with this technique[13, 14] ranged from 5 to 40% of applied power. Our best result was an energy gain of the order of 200% lasting over 12 hours, with an input power of about 50W[15]. The HPPE technique also allowed for better reproducibility. Occurrence of excess heat was found less dependent on the purity and metallurgy of the Pd cathodes and had a success between 20% and 30%.

Besides these interesting results, a serious drawback appeared when we started to use long thin Pd wires. The self-inductance of the wires could produce sudden undershoot and overshoot voltage spikes that were difficult to manage even by using ultra-fast power diodes. In many cases, such spikes caused catastrophic failure of the data acquisition system in addition to generating high-power and high-frequency "noise". The time and expense of repairing the measuring equipment forced us to suspend most of the research program using this powerful method (1998). We now use the method only for some explorative tests. Meanwhile, the use of thin Pd wires as cathodes gave us the idea to develop, in parallel with the HPPE technique, a completely new approach to meet the above mentioned requirements.

1.5. The impurities problem.

When thin wires are used as cathodes, it is necessary to use very dilute electrolytes, i.e. around 10^{-4}M. At such low concentrations, the purity of D_2O becomes crucial. We found that certain metallic impurities, usually dissolved in D_2O, are galvanically deposited on the cathode and formed an impervious layer, which could seriously hinder the loading process. Such problems were known since 1994 after several Pd plates used during the HPPE technique were examined by SEM. Some our unpublished results were shown, discussed, and video-recorded at the Minsk Symposium[11].

1.6. Proper poisoning of Pd surface.

High deuterium loading of Pd can be achieved by altering the physical-chemical properties of the cathode surface instead of using HPPE. The following main processes occur at the cathode surface:

a) $2H_2O + 2e^- \rightarrow 2H_{(adsorb.)} + 2OH^-$ local alkalization in proximity of the cathode

b) $H_{(adsorb.)} \rightarrow [H]_{Pd}$ loading reaction

c) $2H_{(adsorb.)} \rightarrow H_2$ parasitic reaction (bubbling out of H_2 gas)

First, reaction c) should be inhibited as much as possible. This can be done by reducing the catalytic properties of the surface by forming a thin, continuous, but not impervious layer of some appropriate substance. Our first idea was to add to the electrolyte small amounts of metallic ions capable of precipitating insoluble hydroxides on the cathode surface using the local alkalinity to initiate the process. The pH values at the cathode surface can be controlled by changing the current density. Unfortunately, most of the metallic ions (i.e. Fe^{+3}, Cu^{+2}, Ni^{+2} etc.) capable of precipitating as insoluble hydroxides tend to form compact and almost impervious metal layers on the cathode surface.

1.6.1. Searching for new electrolytes, acid environment.

It is known that an alkaline environment promotes the precipitation of alkaline-earth carbonates. It is also known that alkaline-earth ions, because of the strongly negative value of their standard potentials E_0, cannot be galvanically deposited as metals. Accordingly, we found[17, 18] that small amounts of $SrCl_2$ (10^{-5}--10^{-4}M) added to an acid (pH=4) electrolyte, produce a thin and porous layer of $SrCO_3$ on the surface of the cathode, when, and only when, the electrolysis current density exceeds a certain threshold value. The small amount of CO_3^{2-} ions normally present in water are largely sufficient to insure precipitation of $SrCO_3$.

Operating with light water and with this type of electrolyte, H/Pd ratios greater than 0.97 (R/Ro<1.4) were achieved[17].

1.6.2. The micromolar Hg addition and discovery of new "heavy water" bacteria.

It is known that Hg exhibits a very high cathodic over-voltage of 1.21V in 1N HCl at 10^{-2}A/cm^2, which is thought to reduce H+ loss and H-H recombination at the electrode surface. Accordingly, we decided to test whether the addition of Hg ions could increase the Pd overloading. It was found that in the presence of even very small amounts (10^{-6}-10^{-5}M) of mercury ions (Hg^+, Hg^{2+}), the loading ratio (H/Pd) was strongly increased to give our best result of R/Ro = 1.15![17].

The required amount of Hg ions is very critical. In fact Hg ions are reduced to metallic Hg on the cathode surface, thereby forming an amalgam, which beyond a certain concentration of Hg becomes impervious and stops the loading and deloading processes.

Once the correct amount of Hg salt was established, the loading ratios of H/Pd were in most cases excellent and reproducible with R/Ro<<1.3 being currently achieved[20]. Our results were reproduced by Pirelli Labs[21] (Milan-Italy) and SRI- International[23] (Stanford-USA). All things considered, it seemed we were dealing with a pretty good loading protocol in 1999.

When we tried to apply such a protocol for D loading using D_2O, the results were consistently worse[18] and less reproducible. The reasons for such failures were:

- The impuritie concentration usually present in the "as received" heavy water is too large. Double distillation of D_2O is a necessary but not a sufficient pre-treatment.
- Heavy water contains bacteria belonging to two new species. One of these, *Ralstonia* type[19], was found to form colonies on the cathode surface, which hinder the proper formation of the Sr carbonate layer. Moreover, such bacteria can metabolize almost any kind of heavy metals, including Hg and uranium, thereby removing them from the electrolyte. Finally, they can change the pH of the solution up to about 9. A bulk precipitation occurs at such a pH, thus preventing formation of a very thin precipitate on the cathode surface. Our loading procedure works properly only when the mean pH value of the solution is about 4 and the pH is expected to rise up to 10 only in the proximity of the cathode as a consequence of the applied current density[22].

This bacteria has been submitted for registration in June 2000 (DDBJ –Japan and NCBI-USA) with the name of *Ralstonia detusculanense* and accepted on October 2001. The potential for bio-remediation has yet to be fully explored.

We found that a particular and very complex procedure was required for satisfactory D_2O sterilization[18]. This includes double distillation in presence of $KMnO_4$, firstly in acid and then in alkaline, while operating in dry nitrogen at 95°C.

We tried to eliminate at least part of the time consuming and troublesome pre-treatments needed not only to "kill" the bacteria but also to free the heavy water of inorganic and organic pollutants.

1.7. Use of alcohol-water solution and Sr-Hg salts. Possible "intrinsic" production of Pd nanoparticles. Replication Iwamura experiment.

The main controlling parameter of the D-Pd loading is the ratio between the total amount (not the concentration) of the impurities present in the electrolyte and the surface area of the cathode. Consequently, we propose two simple ways to solve the problem.

a) A small cell can be used containing only 50 ml of electrolyte.
b) The cell currently used in our experiments, whose volume is about 1000ml, can be filled with a new electrolyte that is prepared by diluting the impure heavy water with a suitable organic solvent.

The reduction to 50ml is equivalent to a 20 times reduction of the impurities. However, the second approach is more attractive because it does not require construction of a new cell and the ratio between organic solvent and heavy water can be varied over a wide range.

As far as the choice of the organic solvent is concerned, the following requirements should be satisfied:

- large miscibility with water;
- very small amount of H_2O present as residual impurity (to avoid isotopic contamination in D loading);
- negligible acid properties with no isotopic contamination for partial dissociation in H^+ ions;
- boiling point not far from 100°C.

Alcohols, ketons and esters seemed to be the most promising solvents.

After several experiments we found that the partially D substituted ethyl alcohol (C_2H_5OD) was the best choice. It does not exhibit significant isotopic contamination and it is available at a reasonable price of about 1100Euro/l. Nevertheless, for our purposes distillation was necessary before use (see below). Moreover, distillation of the D_2O was also very effective in improving reproducibility and eliminating unexpected impurities. This requirement became clear when the cathode was subjected to ICP-MS analysis after the study.[26]

We performed a series of experiments with hydro-alcoholic solutions containing small amounts of Sr salts and Hg ions during which we found excess heat[22] and tritium[24] well above background. We found that in the hydro-alcoholic environment, during the anodic phase of our loading cycles, the Pd electrode is eroded. Significant amounts of very fine Pd particles are found at the bottom of the cell at the end of the experiments. ICP-MS analysis after electrolysis showed the presence of Pd in this black powder[26]. After several loading-deloading cycles, the Pd wire could absorb deuterium quickly and without applied current to give D/Pd up to 0.75. Such a behavior appears to result from increased activity of the Pd surface. We would like to point out that Yoshiaki Arata (University of Osaka, Japan) has stressed the importance of *nano-structures* in order to get loading of Deuterium in Pd (even over 1/1). As consequence, very large amounts of "anomalous excess heat" and ^4He[27] are observed. It is possible to suggest that the increased activity exhibited by our Pd wires is caused by formation of *nano-structures* on its surface, similar to those obtained by Yoshiaki Arata.

Y. Iwamura [25] showed that Sr is apparently transmuted into Mo (molybdenum) in a gas loaded system. We tried to check whether such a transmutation could occur also after the repeated D-Pd loading/deloading cycles in our experimental set-up. In July 2002 we were ready to perform an independent variant of the Iwamura experiment. Before starting, we analyzed by ICP-MS all the components present in the cell including the electrolyte (C_2H_5OD, D_2O, $SrCl_2$, DCl, $HgCl_2$), and pieces of the Pd cathode and Pt anode. At the end of the repeated D-Pd loading/deloading experiment, the electrolytic solution was vacuum dried. The residue was collected and again analyzed by ICP-MS together with the Pd cathode after it was dissolved in aqua regia. Mo was found in excess of any possible contamination, and the isotopic composition of the Mo was different from the natural one[26]. It appears that the phenomenon first discovered by Y. Iwamura in a flowing deuterium gas system also occurs in our electrolytic cell when operated using our loading-deloading procedure.

1.8. Thorium salts as electrolyte.

In January, 2003, we substituted Th salts for the Sr salts used previously. We decided to test whether something similar could happen in our experiments, based on some 1998 results indicating possible Th transmutations during high-power AC (50Hz) electrolysis with zirconium electrodes[28]. Like Sr, Th ions can form an inorganic precipitate on the cathode surface as $Th(OH)_4$ (solubility product $Ks = 10^{-50}$), by action of the

current density. Another similarity to Sr^{+2} is that Th^{+4} ions cannot be galvanically deposited because of the highly negative value of their standard potential (E_0 = -1.899V).

Accordingly, as objectives of the present work, the following were sought:

a) occurrence of excess heat,

b) presence of foreign elements in both the cathode and the cell after electrolytic loading.

The operations were performed with electrolytes containing small amounts of Th and Hg salts.

2 Experimental set- up

2.1 Electrolytic cell and flow calorimeter

The cell configuration is shown in Fig. 1. The sample holder, a PTFE tube, is placed in a 1000 ml borosilicate glass (type 3.3) cylinder (∅ 67mm, height 460mm). The cathode and anode are both "U" shaped and are located on the opposite walls of the holder, facing each other. The cathode is a thin (∅ 50μm) long (60cm) Pd wire (total surface ≈ 1cm^2). In the lower part of the "U", at its center, a small weight (6g PTFE cylinder) keeps the wire tense during the Pd loading so as to compensate its 4 - 6% elongation. The anode is a Pt wire (∅ 250μm, length 60 cm, purity 99.99%). A third Pt wire (∅ 250μm, 30 cm long) is put exactly in the middle of the "U" shaped cathode for reference purposes.

To measure cathode resistance, an AC current (16mA, 10KHz, square wave) is superimposed on the electrolysis DC current, and the resistance value is continuously monitored using the resulting AC voltage drop.

A high quality LM135H thermometer (sensitivity 0.05°C), inserted in a PTFE tube, is placed in the middle of the cell, perpendicular to the cathode and anode. A Joule heater (max. 20W) is used to calibrate the calorimeter and is located between the electrodes in a peripheral position. It consists of a chain of 20 resistors inserted in a PTFE tube (∅ 8mm, length 30cm) filled with thermal conducting grease.

The cell is pressurized (50mbar) and thermally insulated. The electrolysis gases and vapors are made to flow through a silicon oil bubbler and through a twin cold trap before reaching the atmosphere. The correction for this loss of energy is not yet applied, consequently all the data about excess heat are understimated.

The heat exchanger within the cell consists of a 500cm long PTFE pipe, outer/inner diameter 4/2mm, wound around the PTFE holder through which water flows.Temperature of the distilled water flowing in the pipe is continuously measured at the inlet and outlet of the heat exchanger with two LM135H thermometers. A computerized peristaltic pump (Masterflex 7550-62) provides a constant flow of distilled water (0.200ml/sec, with day-to-day stability of ±1%, routinely measured every 12 hours). Water is picked up by the pump from a 2-liter reservoir to which it returns from the cell. The cell, water reservoir, and pump, are placed in a container held constant at 24°C.

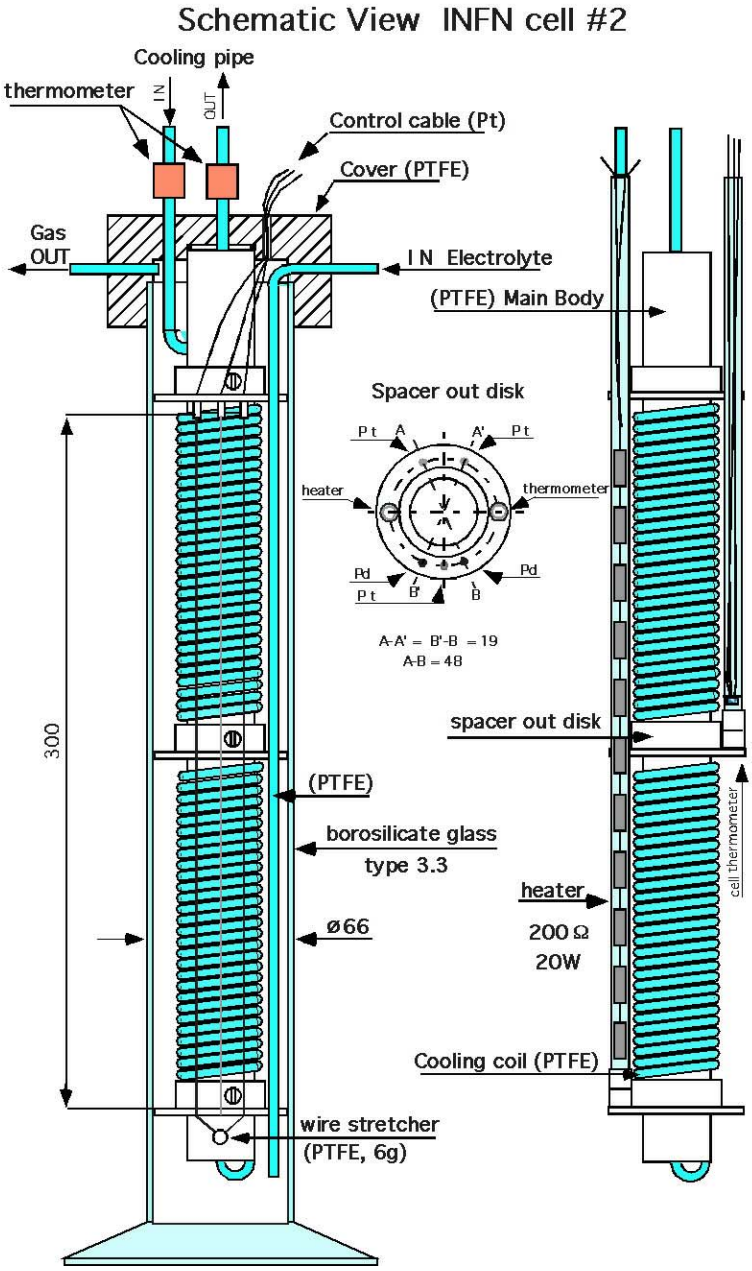


Figure 1 – Schematic view of the electrolytic cell.

2.2 Composition of the electrolyte

A 93 to 7% by volume mixture of ethyl alcohol (C_2H_5OD) and heavy water (D_2O) was used as the electrolyte, having a total volume of 750 ml.

The ethyl alcohol was previously vacuum distilled at 35° C in order to eliminate mainly sodium and iron and ultra-filtered using a 100nm, MILLIPORE PTFE filter. The density was routinely measured (Mettler Toledo

DA-110M) before and after distillation, to confirm that no significant H_2O contamination occurred during the operations.

The heavy water, 99.97% isotopic purity reactor grade (Ontario Hydro), was distilled at 45°C under vacuum and ultra-filtered before use. Density was measured before and after distillation. $Th(NO_3)_4$, (5--15mg) was added to the electrolyte and the pH of the resulting hydro-alcoholic solution was adjusted to about 3 by adding a few drops of HNO_3, in order to avoid precipitation of $Th(OD)_4$.

3. Experimental results

3.1 Excess heat generation and abnormal resistance behavior.

Under "normal" conditions when electrolysis current is switched off, the cathode resistance gradually increases toward its maximum because of D deloading. In contrast, when the electrolyte contains thorium, stopping the current causes the cathode resistance to decrease toward a miminum value (Fig. 2) where it remains stable for a few minutes. Finally, it starts to rise, moving gradually towards its maximum as expected.

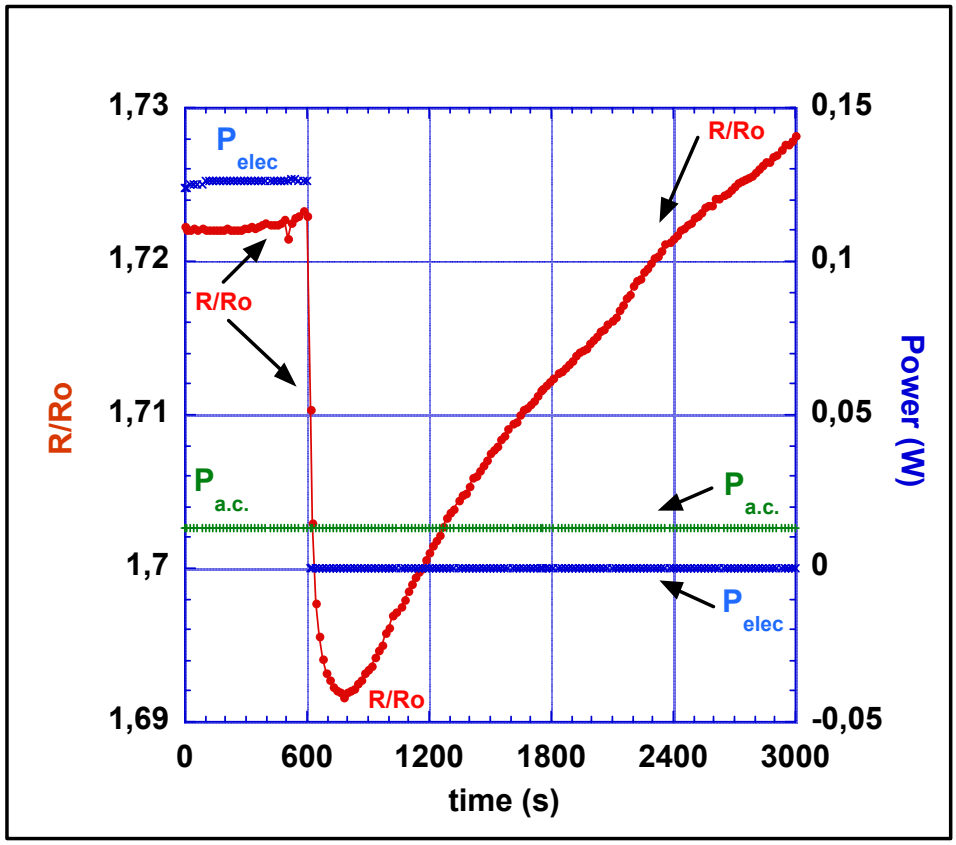


Figure 2 – The fall in cathode resistance when the electrolysis is swithched off. R/Ro is the ratio between the actual resistance R and resistance at D/Pd=0. P_{elec} is the power input due to the electrolysis current. $\mathbf{P_{a.c.}}$ is the power input due to the a. c. current used to measure the cathode resistance.

We propose that before interruption of electrolysis, the wire was at a temperature higher than that of the solution, and the observed decrease in resistence was caused by cooling of the wire.

It can be easily demonstrated that the observed phenomenon cannot be attributed to Joule heating induced by the electrolysis current flowing into the cathode. In our loading cycles we kept the electrolysis current constant, typically at 10mA. The cathode resistance is about 60 ohm. Power input into the cathode is given by the formula:

$$W=i^2 \cdot R$$

where R is the cathode resistance, and i is the current flowing into the cathode averaged along the wire. By roughly assuming that this current is equal to the electrolysis current, power experienced by the cathode cannot exceed 6mW. This amount of Joule heating has a negligible effect on the resistence. In order to achieve a quantitative evaluation of the observed phenomenon, we performed the tests summerized in Fig. 3.

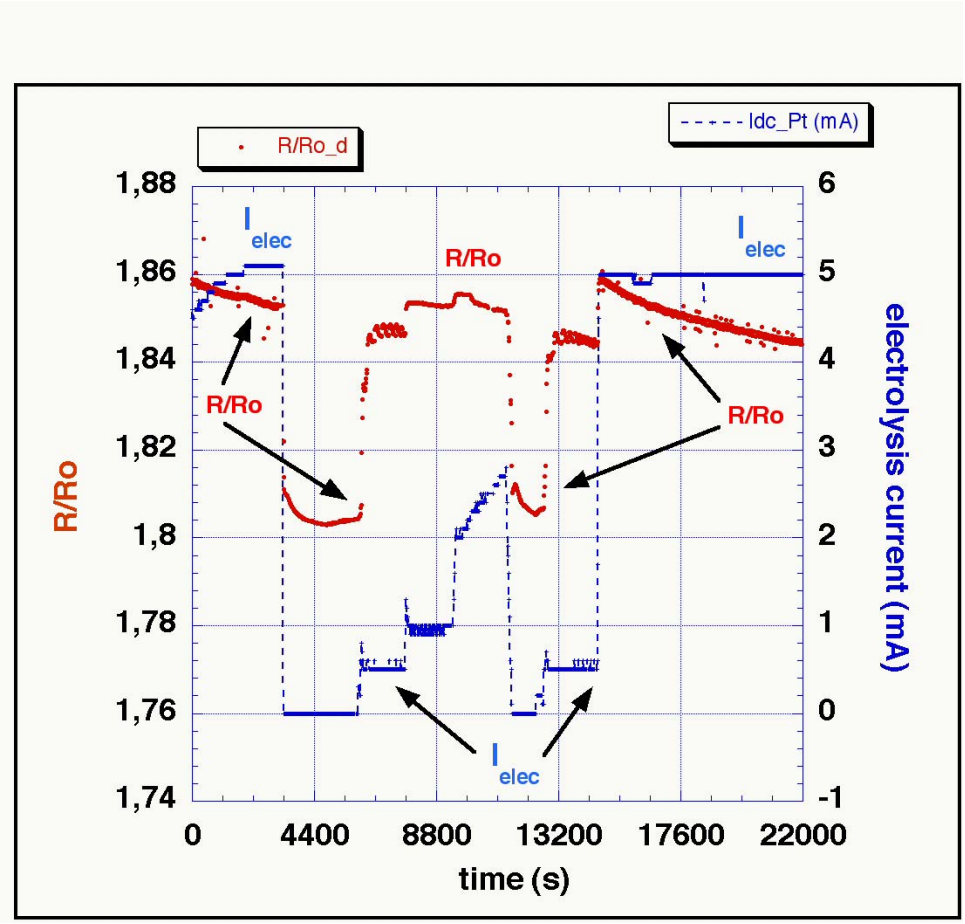


Figure 3 – Evidence of the phenomenon of restoration of the R/Ro value with very low electrolysis currents and consequent demonstration that the drop down of R/Ro is not due to the shut down of the Joule effect. I_{elec} is the electrolysis current.

After power was turned off and cathode resistance decreased, an electrolysis current of 0.5mA was applied again in the circuit. This supplied 15 microwatt of Joule heating to the Pd wire. No resistance rise can be expected from such a small amount of power. Yet, the resistance of the wire jumped up to 90% of the level it had reached before shut-down. Further incremental increases in the electrolysis current up to 3 mA produced progressively smaller increases in resistance. The test were repeated several times and the results were substantially the same.

It appears that the observed fall in cathode resistance was not due to the Joule heating induced by electrolysis current flowing into the cathode. In fact, the only plausible explanation we can find for the observed phenomenonis is that the Pd wire generates excess heat once a small electrolysis current is applied. The phenomenon, whatever it is, appears to occur at the cathode surface.

We previously recorded this kind of anomalous decrease in resistance several times during some previous loading experiments with alcohol-free electrolytes containing Sr-Hg. It was observed only once with as great an intensity when the hydroalcohlic Th electrolytes were used. The largest event with non-alcohol electrolyte was reported in Ref. 18, pg. 189, Fig. 8 where we cautiously proposed that the cell was producing excess heat.

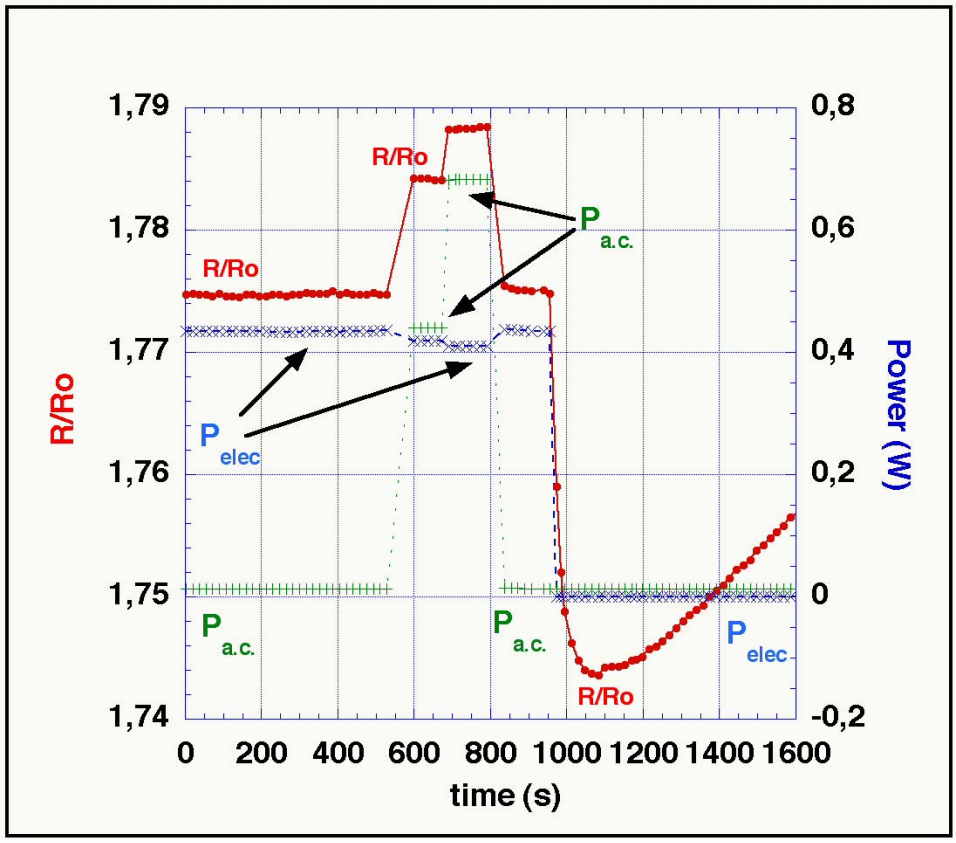


Figure 4 – Self calibration procedure. On the left side it is shown the increase of R/Ro due to the Joule effect produced by increasing the a. c. power input (P_{ac}). On the right side it is shown the R/Ro drop down when the electrolysis power (P_{elec}) is reduced.

3.2 Self calibration procedure.

As we performed the tests described here, we realized we had on hand a simple method to assess and evaluate the occurrence of excess heat in the cell: the cathode itself can act as an "in situ" micro-calorimeter. Fig. 4 shows how this calibration can be achieved and how the excess heat can be rapidly and precisely estimated.

While DC current is supplied to the cathode, an AC current (about 100mA) is superimposed, which generates additional power of about 680mW. This additional power causes cathode resistance to increase from the initial R/Ro value of 1.775 to the final one 1.788, caused by the resulting temperature change. When the AC current is turned off, resistance returns to its initial value. In other words, a variation of 0.013 in cathode resistance ratio corresponds to an input power of 680mW. After this "calibration" was done, electrolysis current was switched off. We observed that the R/Ro ratio dropped from 1.775 to 1.744. Based on the previous "calibration", this change in ratio indicates that the cathode was generating 31/13 x 680mW = 1621mW.

To evaluate the total power balance we have to consider total external power fed into the cell. About 150mW resulted from the DC electrolysis current. The net gain of ~1440mW means the output/input ratio is ~9.5. Because this auto-calibration method may be considered unconventional, we decided to confirm the excess heat using flow calorimetry.

3.3 Flow calorimeter measurement.

During the tests shown in Fig. 4, the cell was not thermally insulated because we had to visually observe the inside of the cell during the test to be sure the cylindrical weight kept the Pd wire under enough tension. As soon as we realized that the cathode was producing excess heat, we applied makeshift thermal insulation to the cell, to improve calorimetry. Nevertheless, diurnal temperature fluctuations (± 3°C) caused the inlet water temperature to vary over a range ±0.5°C. This change caused the calorimetric to oscillate by ± 500 mW during the day/night cycle. Despite this variation, measurement of heat generated within the calorimeter is still valid

because the calorimeter was used to make absolute measurements. In fact, the measured values are underestimated because of heat loss through the imperfect thermal insulation of the cell.

The absolute measurement of the heat flow out of the cell is shown Fig. 5. While the power input was maintained constant at ~150mW, the measured power output oscillated around an average value of 1400mW ±400mW, with a tendency to rise while the R/R_0 ratio slowly decreased, which indicated that the D/Pd ratio had increased during this time. Apparently, the flow calorimeter measurements are in substantial agreement with the "in situ" micro-calorimetric estimations. Further experiments are in progress to optimize electrolyte composition and heat recovery.

Surprisingly, we found that electrolyte consumption was over 5 times larger than predicted by Faraday's law. We suspect that this loss is caused by heat being generated in a few hot spots where temperature is very large. This local heating evaporates the solution so that gaseous D_2O and C_2H_5OD ~~is~~ are lost in addition to D_2 and O_2. We plan to build a new cell made from PTFE, quartz, HDPE, and mylar in order to detect IR radiation from the deuterated Pd wires using a thermo-camera.

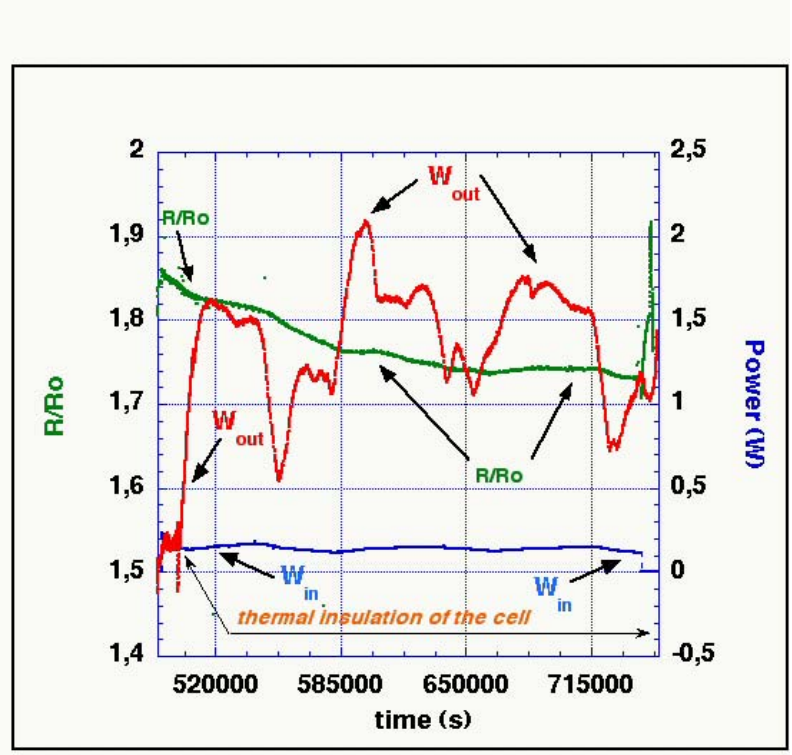


Figure 5 – Flow calorimeter measurements. W_{in} is the total power input. W_{out} is the power flowing out of the calorimeter with the outgoing water flow.

4. ICP-MS Measurements.

4.1 Procedure

ICP-MS measurements were performed in a chemistry laboratory operating under the ISO 9001 quality control protocol. The laboratory is located in the Interdisciplinary Research Area of Castel Romano (Rome), in the "Centro Sviluppo Materiali" building. The ICP-MS used is from HP & YOKOGAWA Analytical Systems, model 4500. It has been in operation since 1996. Calibrations with Atomic Absorption Standards are made every day before starting analysis. Sensitivity is about $6*10^{10}$ Atoms/counts. Typical background is 10 - 80 counts, depending both on the mass analyzed and overall condition of the instrument. Background is higher for two well known sources of interference, due to Ar, Cl, O, N, C, H, and impurities dissolved in the deionised "Milli-Q" water and reagents used. The carrier gas is Ar. Because of our specific experimental constrains, we almost always use hot, concentrated Aqua Regia to dissolve the materials in glass beakers (borosilicate 3.3). A concentration of 0.25% is submitted to the instrument for analysis of the main elements in the electrolyte (Pd, Sr, Th, Hg). We make 2 runs, from mass 2 to 260 with a resolution of 0.5 AMU. Each run takes about 200

seconds and sample uptake is 0.5ml/min. Blank and mass tuning (Li, Y, Tl) runs are made at the beginning of analysis, after 3 hours of operations, and at the end of the study. Calibration is done more frequently if an element is at a particularly high concentration. Between each measurement there is a washing cycle of about 10 minutes using HNO_3 at 5% concentration or Aqua Regia if needed. A blank measurement is always performed.

Data are analyzed according to the general recommendation of ICP-MS manufacturer and to our specific operating conditions. We especially want to avoid spurious results caused by double mass and half-mass signals adding to the masses of interest. To solve such problems, we performed a large number of analysis using standard solutions of elements of interest to calibrate the machine. Obviously, D_2O and C_2H_5OD were also carefully characterized.

The cell was cleaned after each experiment using repeated cycles of water and organic solvents in an ultrasonic hot bath. After Experiment #2 (Feb. 14, 2003; see Table 1a), we increased the duration of immersion in HNO_3 (65%, 60°C) from 2 minutes to 14 hours because we suspected that a residual amount of Cs might be be hidden somewhere in the cell.

4.2 Experimental results

4.2.1 Sr→Mo

Results using Sr within the electrolyte were reported in Ref.26. We observed that some of the Sr was transmuted to elements with mass 94 and 96, qualitatively in agreement with the Y. Iwamura results. Moreover, the total amount of Mo atoms we found ($1\text{-}2\cdot10^{15}$), normalized to Pd electrode surface (about $1cm^2$), is very similar to the Iwamura gas experiments.

4.2.2 Cs→Pr

Transmutation of Cs to Pr (according to Y. Iwamura) was not observed when we used concentrated Cs because we were not able to achieve a sufficiently high deuterium concentration in the cathode. Much to our surprise, results were better when a Th salt was used with a "proper mixture" of Ca and Sr salts. Using these mixtures, we apparently transmuted Cs and, in smaller amounts, Pr. We note that the thorium salt did not contain measurable impurities of Cs or Pr, according to both the assay from the chemical company (Aldrich) and our routine analysis by ICP-MS. In the best experiment in which apparent transmutations have been detected, the signal is nine times background.

Later (in experiments #5 and #6) we refined the method using only Th and Hg salts to increase the deuterium concentration in Pd. In experiment #6 we observed a signal to background ratio as large as 21. Experiment #4 did not achieve the necessary deuterium concentration, making it a very useful blank for ICP-MS analysis.

In other words, apparent transmutations of Cs into Pr occurred in an electrochemical environment, similar to those reported by Y. Iwamura in a gaseous environment[25, 29] and recently confirmed by A. Takahashi *et. al.*[30]

4.2.3 Thorium results

We performed a total of 4 experiments with Th salts, in amounts ranging from $8\text{x}10^{-6}$ to $6.5\text{x}10^{-5}$ moles in the total volume of electrolyte. The pH for the Th solution was 3 compared to 4 for the Sr solution.

4.2.4 ICP-MS: results

Some results are summarized in Tables 1 and 2. We list masses that are relatively easy to interpret and for which reproducible results were obtained. We observed several other anomalies that are not yet fully understood. We anticipate that most of such anomalies are in the range of masses 46 - 60, 107 - 116, along with some isotopic anomaly for Pd.

Table 1: Results of ICP-Ms analysis. Please note that "overloading" is defined here as D/Pd>0.9.

Exp	**Date: begin→ end [dd/mm/yy]**	**Electrolyte composition: [moles]**	**Comments on results OVL=Overloading S/N=Signal/Noise Pr=1count=6·10^{10} Atoms;**
#1	20/12/02 → 16/01/03	$CsNO_3$[$5\cdot10^{-5}$] - $SrCl_2$[$1\cdot10^{-5}$] LiOD [$1.5\cdot10^{-5}$] - H_2SO_4[$5\cdot10^{-6}$] NH_3OH [$1\cdot10^{-4}$]	Almost No OVL ; Anode=Pd wire 250μm Pr=80=>S/N=2
#2	17/01/03 → 14/02/03	$CaCl_2$ [$21\cdot10^{-5}$] - $SrCl_2$ [$1\cdot10^{-4}$] $HgCl_2$ [$2\cdot10^{4}$] – H_2SO_4 [$1\cdot10^{-5}$]	2 times OVL, Residual Cs?? Pr=170=>S/N=4
#3	18/02/03 → 05/03/03	$CaCl_2$ [$1\cdot10^{-5}$] - $SrCl_2$ [$1\cdot10^{-4}$] $HgCl_2$ [$2\cdot10^{-4}$] -H_2SO_4 [$1\cdot10^{-5}$] $Th(NO_3)_4$ [$8\cdot10^{-6}$]	3 times OVL R/Ro=1.706 Pr=370=> S/N=9
#4	04/04/03 → 14/04/03	$HgCl_2$ [$1\cdot10^{-5}$] -Hg_2SO_4 [$1\cdot10^{-4}$] $Th(NO_3)_4$ [$2.1\cdot10^{-5}$]	No OVL
#5	15/04/03 → 19/05/03	$Th(NO_3)_4$ [$2\cdot10^{-6}$] Hg_2SO_4 [$2\cdot10^{-6}$]	Several times OVL; Excess heat; Pr=300=>S/N=6
#6	24/05/03 → 14/07/03	$Th(NO_3)_4$ [$2\cdot10^{-6}$] Hg_2SO_4 [$5\cdot10^{-6}$]	Several times OVL; Excess heat. Pr=1.4E3=>S/N=21 *^{208}Pb anomaly*

Table 2: shown here are the counts of the main mass found; the natural abundance is in paranthesis. The composition of the solution is described in Ref. 26.

Exp	**^{31}P(100)**	**^{39}K(93.3)**	**^{63}Cu(69.2) ^{65}Cu(30.8) 63/65=2.25**	**^{64}Zn(48.6) ^{66}Zn(27.9) ^{67}Zn(4.1) ^{68}Zn(18.8)**	**^{133}Cs(100)**	**^{206}Pb(24.1) ^{207}Pb(22.1) ^{208}Pb(52.4) 206/208=.46**
#1	$7\cdot10^{3}$	$2.2\cdot10^{6}$	$2.4\cdot10^{6}$	$2.7\cdot10^{6}$	$>3\cdot10^{8}$	$1\cdot10^{4}$
#2	0	0	$2.1\cdot10^{6}$ 63/65=1.94	$1.8\cdot10^{6}$	$6\cdot10^{5}$	$4\cdot10^{5}$
#3	$4\cdot10^{5}$	$2.7\cdot10^{6}$	$6.1\cdot10^{6}$ 63/65=1.93	$1.7\cdot10^{7}$	$1\cdot10^{6}$	$9.5\cdot10^{5}$
#4	$2.5\cdot10^{4}$	$1.4\cdot10^{6}$	$2.9\cdot10^{6}$ 63/65=2.05	$7.5\cdot10^{3}$	$1.94\cdot10^{5}$	$6.9\cdot10^{5}$
#5	$1.8\cdot10^{6}$	$2.3\cdot10^{6}$	$2.4\cdot10^{7}$ 63/65=2.07	$1.2\cdot10^{7}$	$9\cdot10^{4}$	$1.5\cdot10^{6}$
#6	$2.72\cdot10^{7}$	$6.4\cdot10^{6}$	$9.3\cdot10^{8}$ 63/65=2.08	$5.1\cdot10^{8}$	$2.2\cdot10^{5}$	$2\cdot10^{8}$ 206/208=.39

3 Conclusions

After a large number of experiments during 14 years of research aimed at finding anomalous effects in systems forced to a high concentration of deuterium, we are confident that most of the observed effects occur at the interface between the solution and the Pd bulk. A properly formed layer of a third element is necessary.

Nonequilibrium is also necessary to trigger the effects. Recently, we found that using a deuterated hydro-alcoholic solution made slightly acid works very well. Addition of Th and Hg salts at micromolar concentrations improve the effects even at very low electrolytic current density (<10mA/cm^2).

We think that the model developed by Akito Takahashi about multi-body resonance fusion of deuterons[31] can explain most of the thermal and isotopic anomalies, including foreign elements, that we have recently observed.

Further work is necessary to fully characterise the system and increase the magnitude of the effects.

4 Acknowledgements

We would like to thank Jed Rothwell and Edmund Storms for their critical reading of our paper and useful suggestions.

References

1. M. Fleischmann, S. Pons, M.Hawkins - Journal of Electroanalytical Chemistry 261, 301-308, (April 1989).
2. S.E. Jones, E. P. Palmer et al. *Observation of Cold Nuclear Fusion in Condensed Matter*, Nature 338: 737-740 (April 1989).
3. F. Celani,…A. Spallone, V. di Stefano, P. Marini et al.. *Results of 1st generation experiments, at Gran Sasso underground laboratory, on nuclear cold fusion*, <<Understanding Cold Fusion Phenomena>>, 15-16 Settembre 1989 Varenna (Italy), SIF Conf. Proc., Vol. 24, pp.257-266; ISBN 88-7794-031-X.
4. F. Celani, A. Spallone...V. di Stefano, P. Marini. *Further measurements on electrolytic cold fusion with D_2O and palladium at Gran Sasso Laboratory*, Fusion Technology, Vol.17, pp.718-724, (1990). ISSN: 0748-1896.
5. F. Celani, A. Spallone,….P. Marini et al. *Measurements in the Gran Sasso Laboratory: evidence for nuclear effects in electrolysis with Pd/Ti and in different tests with deuterated high temperature superconductors.* <<Anomalous Nuclear effects in deuterium/solid systems>> Provo, UT 1990 (USA) American Institute of Physics: Conference Proceedings Vol. 228, pp. 62-100. ISBN 0-88318-833-3.
6. S. E. Jones, D. Bennion et al. *In quest of a trigger mechanism for neutron emissions from deuterium/solid system.* <<Anomalous Nuclear effects in deuterium/solid systems>> Provo, UT 1990 (USA) American Institute of Physics: Conference Proceedings Vol. 228, pp. 206-235. ISBN 0-88318-833-3.
7. F. Celani, A. Spallone,…P. Marini et al. *Search for neutron emission from deuterided high temperature superconductors in a very low background environment* <<The Science of Cold Fusion>> ICCF2, 29 June-4 July 1991 Como (Italy) Conference Proceedings Vol.33 SIF, pp.113-121 ISBN 88-7794-045-X.
8. A. Takahashi, A. Mega et al. *Anomalous Excess Heat by D2O/Pd Cell under L-H Mode Electrolysis* <<Frontiers of Cold Fusion>>, October 1992, Nagoya (Japan), Edited by H. Ikegami. Frontier Science Series No. 4. Univ. Acad. Press Tokyo (Japan). pp.79-91; ISBN 4-946443-12-6.
9. F. Celani, A. Spallone et al. *Measurements of Excess Heat and Tritium during Self-Biased Pulsed Electrolysis of Pd-D_2O.* <<Frontiers of Cold Fusion>> Oct. 1992, Nagoya (Japan), Edited by H. Ikegami; Frontier Science Series No. 4. Univ. Acad. Press Tokyo (Japan). pp.93-105; ISBN 4-946443-12-6.
10. F. Celani, A. Spallone,….P. Marini, V. di Stefano. *High power μs pulsed electrolysis for high deuterium loading in Pd plates.* Transaction of Fusion Technology Vol.26, No. 4T, December 1994, pp.127-137. ISSN: 0748-1896. Proceedings of the ICCF4, Lahaina, Maui, Hawaii, December 6-9, 1993.
11. F. Celani, A. Spallone ,…..P. Marini, V. di Stefano. *D/Pd loading ratio up to 1.2:1 by High Power μs Pulsed Electrolysis in Pd Plates.* Proc. of the "International Symposium on Cold Fusion and Advanced Energy Sources", Belarusian State University, Minsk, Belarus, May 24-26, 1994. Edited by Hal Fox. Published by "FUSION INFORMATION CENTER", Salt Lake City, Utah-USA.
12. F. Celani, A. Spallone,….P. Marini, V. di Stefano et al. *Reproducible D/Pd ratio>1 and excess heat correlation by 1μs pulse, high current electrolysis.* Fusion Technology, Vol.29, pp.398-404, 1996. ISSN: 0748-1896.
13. F. Celani, A. Spallone,…P.Marini, V. di Stefano,..A. Mancini. *High power μ s pulsed electrolysis using palladium wires: evidence for a possible "phase" transition under deuterium overloaded conditions and related excess heat*. ICCF5, April 9-13, 1995 Monte-Carlo; pp.57-68.
14. F. Celani, A. Spallone,…P. Marini, V. di Stefano,..A. Mancini. *Deuterium overloading of palladium wires by means of high power μs pulsed electrolysis and electromigration: suggestion of a "phase transition" and related excess heat.* Physics Letters A 214 (1996), pp.1-13.

15. F. Celani, A. Spallone, … P. Marini, V. di Stefano, A. Mancini et al. *New kinds of Electrolytic regimes and Geometrical Configurations to obtain Anomalous Results in Pd(M)-D Systems.* ICCF6 <<Progress in new hydrogen energy>> October 13-18 1996 Toya-lake (Japan). Vol.1 pp.93-104. Edited by Makoto Okamoto. Published by NEDO and IAE , Tokyo (Japan) on December 11, 1996.
16. Giuliano Preparata *QED COHERENCE IN MATTER,* World Scientific, 1995, ISBN: 981-02-2249-1.
17. A. Spallone, F. Celani, P. Marini, V. di Stefano. *New electrolytic procedure for the obtainment of very high H/Pd loading ratios. Preliminary attempts for its application to the D-Pd system*. ICCF8, 21-26 May 2000, Lerici (Italy). SIF Conf. Proc. Vol.70, pp.191-198. ISBN 88-7794-256-8.
18. F. Celani, A. Spallone, P. Marini, V.di Stefano, M. Nakamura, A.Mancini,…C. Catena, G. D'Agostaro, … P.Quercia, E. Righi, G. Trenta. *High hydrogen loading into palladium wires through precipitate of alkaline-earth carbonate on the surface of the cathode: evidence for new phases in the Pd-H system and unexpected problems due to bacteria contamination in the heavy water.* ICCF8, 21-26 May 200 Lerici (Italy), SIF Conf. Proc.Vol.70, pp.181-190. ISBN 88-7794-256-8.
19. G. D'Agostaro, F. Celani et al. *Ralstonia detusculanense,* National Centre for Biology Information—GenBank (Bethesda-USA); Locus:AF280433, Taxonomy ID:148618, October 24, 2001; http://www.ncbi.nlm.nih.gov/
20. A.Spallone, F. Celani, P. Marini, V. di Stefano. *Experimental studies to achieve H/Pd loading ratio close to 1 in thin wires, using different electrolytic solutions.* <<Condensed Matter Nuclear Science>> ICCF9 19-25 May 2002, Beijing (China); pp.319-322. ISBN 7-302-06489-X.
21. D. Garbelli, D. Azzarone, F. Fontana. *Hydrogen/deuterium loading in thin palladium wires*. ICCF8, 21-26 May 2000, Lerici (Italy). SIF Conf. Proc. Vol.70, pp.199-204. ISBN 88-7794-256-8.
22. F. Celani, A. Spallone, P. Marini, V.di Stefano, M. Nakamura, A. Mancini, G. D'Agostaro, E. Righi, G. Trenta, P. Quercia, , C. Catena. *Electrochemical D loading of Palladium wires by heavy ethyl-alcohol and water electrolytes related to Ralstonia bacteria problematics*. <<Condensed Matter Nuclear Science>> ICCF9 19-25 May 2002, Beijing (China); pp.29-35. ISBN 7-302-06489-X.
23. P. Tripodi,… F. Celani, V. Violante. *Temperature coefficient of resistivity at compositions approaching PdH.* Phys. Lett. A 276 (2000), pp.122-126.
24. F. Celani, A. Spallone, P. Marini, V.di Stefano, M. Nakamura, E. Righi, G. Trenta, P. Quercia, , A. Mancini, G. D'Agostaro, C. Catena, …V. Andreassi. *Evidence of anomalous tritium excess in D/Pd overloading experiments.* <<Condensed Matter Nuclear Science>> ICCF9 19-25 May 2002, Beijing (China); pp.36-41. ISBN 7-302-06489-X.
25. Y.Iwamura et al. Jpn. J. Appl. Phys. **41** (2002), pp. 4642-4648.
26. F. Celani, A. Spallone, P. Marini, V. di Stefano, M. Nakamura, A. Mancini, G. D'Agostaro, E. Righi, G. Trenta, P. Quercia, C. Catena, V. Andreassi, F. Fontana, D. Garbelli, L. Gamberale, D. Azzarone, E.Celia, F.Falcioni, M. Marchesini, E. Novaro. *Unexpected detection of new elements in electrolytic experiments with deuterated ethyl-alcohol, Pd wire, Sr and Hg salts*. "The 4th Meeting of Japan CF-Research Society" October 17-18,2002, pp.17-21. Iwate Univ. (Japan). http://www.eng.osaka-u.ac.jp/nuc/03/nuc03web/JCF/.
27. Y. Arata, Yue Chang Zhang. *Characteristics of compact and practical "Solid Deuterium Nuclear Fusion Reactor"*. ICCF10, in press.
28. F.Celani, …P.G. Sona, A.Mancini. *Preliminary results with "Cincinnati Group Cell" on thorium "transmutation" under 50Hz ac excitation* ICCF7. April 19-24, 1998 Vancouver (Canada) pp.56-61. Edited by ENECO Inc. (Salt Lake City, USA).
29. Y. Iwamura et al. *Low energy nuclear transmutation in condensed matter induced by D_2 gas permeation through Pd complexes: correlation between deuterium flux and nuclear products*. ICCF10, in press.
30. T. Higashiyama,…A. Takahashi *Replication of MHI transmutation experiment by D_2 gas permeation through Pd complex.* ICCF10, in press.
31. A. Takahashi. *Tetrahedral and octahedral resonance fusion under transient condensation of deuterons at lattice focal points*. <<Condensed Matter Nuclear Science>> ICCF9 19-25 May 2002, Beijing (China); pp.343-348. ISBN 7-302-06489-X.

ISTITUTO NAZIONALE DI FISICA NUCLEARE
Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Rapp. Naz.: Francesco CELANI		

PREVISIONE DI SPESA

Piano finanziario globale di spesa

In KEuro

ANNI FINANZIARI	Missioni interne	Missioni estere	Materiale di consumo	Trasporti e facchinaggi	Spese di calcolo	Affitti e manutenzione	Materiale inventariabile	Costruzione apparati	TOTALE Compet.
2005	16,0	16,0	39,0				24,0	20,0	115,0
TOTALI	***16,0***	***16,0***	***39,0***	***0,0***	***0,0***	***0,0***	***24,0***	***20,0***	***115,0***

Mod EC./EN. 6 (a cura del responsabile nazionale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Vincenzo Nassisi		

Struttura
LE

COMPOSIZIONE DEL GRUPPO DI RICERCA

N	RICERCATORE Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ricerca	Qualifica: Incarichi Assoc.	Affer. al gruppo	%
1	Buccolieri Giovanni				R.U.	5	40
2	Di Giulio Massimo				P.A.	5	30
3	Filippo Emanuela				Dott.	5	100
4	Lorusso Antonella				Dott.	5	100
5	Manno Daniela				P.A.	5	20
6	Nassisi Vincenzo			P.O.		5	30
Numero totale dei ricercatori							**6**
Ricercatori Full Time Equivalent							**3.2**

N	TECNOLOGI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ass. Tecnol.	%
Numero totale dei Tecnologi					**0**
Tecnologi Full Time Equivalent					**0**

N	TECNICI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 15	Qualifica: Incarichi Collab. tecnica	Qualifica: Incarichi Assoc. tecnica	%
1	De Donno Francesco				Univ.	20
2	Miccoli Crocifisso				Univ.	30
3	Monteduro Luigi				Univ.	30
4	Nicolardi Vincenzo				Univ.	20
Numero totale dei Tecnici						**4**
Tecnici Full Time Equivalent						**1**

SERVIZI TECNICI

Denominazione	mesi−uomo

Annotazioni:

Osservazioni del direttore della struttura in merito alla disponibilità di personale e attrezzature

Mod EC./EN. 7 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Francesco CELANI		

Struttura
LNF

COMPOSIZIONE DEL GRUPPO DI RICERCA

N	RICERCATORE Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ricerca	Qualifica: Incarichi Assoc.	Affer. al gruppo	%
1	CATENA Carlo				E.N.E.A	5	100
2	CELANI Francesco	I Ric				5	100
3	D'AGOSTARO Giacomo				E.N.E.A	5	100
4	MARCELLI Augusto	I Ric				5	20
5	RIGHI Enzo			D.R.		5	100
6	SPALLONE Antonio				P.S.	5	100
7	TRENTA Giorgio				D.R.	5	100
	Numero totale dei ricercatori						**7**
	Ricercatori Full Time Equivalent						**6.2**

N	TECNOLOGI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ass. Tecnol.	%
	Numero totale dei Tecnologi				**0**
	Tecnologi Full Time Equivalent				**0**

N	TECNICI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 15	Qualifica: Incarichi Collab. tecnica	Qualifica: Incarichi Assoc. tecnica	%
1	ANDREASSI Vincenzo	O.T.				100
	Numero totale dei Tecnici					**1**
	Tecnici Full Time Equivalent					**1**

SERVIZI TECNICI

	Denominazione	mesi–uomo

Annotazioni:

Osservazioni del direttore della struttura in merito alla disponibilità di personale e attrezzature

Mod EC./EN. 7 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Resp. loc.: Onori		

Struttura
PG

COMPOSIZIONE DEL GRUPPO DI RICERCA

N	RICERCATORE Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ricerca	Qualifica: Incarichi Assoc.	Affer. al gruppo	%
Numero totale dei ricercatori							**0**
Ricercatori Full Time Equivalent							**0**

N	TECNOLOGI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 23	Qualifica: Incarichi Ass. Tecnol.	%
Numero totale dei Tecnologi					**0**
Tecnologi Full Time Equivalent					**0**

N	TECNICI Cognome e Nome	Qualifica: Dipendenti Ruolo	Qualifica: Dipendenti Art. 15	Qualifica: Incarichi Collab. tecnica	Qualifica: Incarichi Assoc. tecnica	%
Numero totale dei Tecnici						**0**
Tecnici Full Time Equivalent						**0**

	SERVIZI TECNICI	
	Denominazione	**mesi−uomo**

Annotazioni:

Osservazioni del direttore della struttura in merito alla disponibilità di personale e attrezzature
La previsione di spesa e l'attività sono congrue con le disponibilità di personale e attrezzature.

Mod EC./EN. 7 (a cura del responsabile locale)

ISTITUTO NAZIONALE DI FISICA NUCLEARE

Preventivo per l'anno ***2005***

Codice	Esperimento	Gruppo
	FREETHAI	5
Rapp. Naz.: Francesco CELANI		

MILESTONES PROPOSTE PER IL 2005

Data completamento	Descrizione
31/12//2005	Misure comparative (soluzioni idrogenate e deuterate) di hot spot, con termocamera IR, proveniente da filo di palladio immerso in soluzione elttrolitica.
31/12/2005	Valutazione effetto nanoparticelle di palladio dal punto di vista della presenza di nuovi elementi nella cella elettrolitica.
31/12/2005	Veriazione dell'effetto citotossico della acqua pesante irradiata rispetto al tipo di cellule utilizzate.
31/12//2005	Primi risultati dell'effetto citotossico, indotto dall'acqua pesante irradiata, su forme neoplastiche in piccoli animali di laboratorio.
31/12//2005	Completamento analisi di eventuali nuovi elementi, presenti su campioni di film di palladio idrogenati o deuterati, dopo trattamento con laser in continua o pulsati.
31/12/2005	Identificazione e quantificazione ROS presenti in H2O e D2O. Correlazione con effetti macroscopici citotossici.

Mod EC./EN. 8 (a cura del responsabile nazionale)