MEASUREMENT OF NEUTRON BURST PRODUCTION IN THERMAL CYCLE OF D_2 ABSORBED TITANIUM CHIPS

COLD FUSION

TECHNICAL NOTE

KEYWORDS: *neutron burst, deuterium-absorbed titanium, thermal cycle experiment*

RONGBAO ZHU, XIAOZHONG WANG, FENG LU, DAZHAO DING,
JIANYU HE, HENGJUN LIU, JINCAI JIANG, GUOAN CHEN,
YUAN YUAN, LIUCHENG YANG, and ZHONGLIN CHEN
China Institute of Atomic Energy, P.O. Box 275-26
Beijing, People's Republic of China

HOWARD O. MENLOVE *Los Alamos National Laboratory*
Los Alamos, New Mexico 87545

Received February 14, 1991
Accepted for Publication May 2, 1991

A high-level neutron coincidence counter equipped with 18 ^{3}He tubes and a JSR-11 shift register unit with a detection limit of 0.20 n/s for a 2-h run is used to study the neutron signals in D_2 gas experiments. Different material pretreatments are selected to review the changes in frequency and size of the neutron burst production. Experimental sequence is deliberately designed to distinguish the neutron burst from fake signals, e.g., electronic noise pickup, cosmic rays, and other sources of environmental background. Ten batches of dry fusion samples are tested, among them, seven batches with neutron burst signals that occur roughly from −100°C to near room temperature. In the first four runs of a typical sample batch, seven neutron bursts are observed with neutron numbers from 15 to 482, which are 3 and 75 times, respectively, higher than the uncertainty of the background. The samples seem to be inactive after four or five temperature cycles, and the inactive samples could be reactivated by degassing and recharging of deuterium. The same anomalous phenomena were observed in the Mentou Valley Underground Laboratory situated 580 m below ground.

I. INTRODUCTION

Neutron generation was declared as one of the evidences for the occurrence of cold fusion in deuterium-charged titanium or in a palladium cathode in both the Fleischmann and Pons[1] electrochemical cells (category I) and the Jones et al.[2] type cells (category II). De Ninno et al.[3] reported neutron production in the processes of thermal cycle of deuterium-absorbed titanium materials (category III). Both random and burst neutron emissions were observed in the warmup period in a deuterium/titanium system by Menlove et al.[4] of Los Alamos National Laboratory (LANL), using several ^{3}He systems coupled with fast preamplifier, discriminator, and a shift-register coincidence counter. A series of experiments was initiated in October 1989 at the China Institute of Atomic Energy to search for neutron burst production by applying high-pressure deuterium to titanium metal chips and putting the system into thermal cycles from liquid nitrogen to room temperature.

Because the detection of neutron burst events with the characteristics of low intensity and frequency would most likely be challenged by fake signals from cosmic-ray spallation and electronic noise, especially for those events that do not occur in regular time, attention was paid to experimental design incorporating procedures to avoid confusion in explaining the results. Recently obtained results strongly support the evidence for neutron emission in the thermal cycle of deuterium-absorbed materials found at LANL (see Ref. 4).

II. THERMAL CYCLE EXPERIMENTS

This phase of experiments began in October 1989 for the purpose of duplicating category III experiments, especially searching for the random and burst neutron generation observed by Menlove et al. The titanium chips were prepared for nine sample batches by drilling, lathing, or shaving a 12-mm-thick TA1 plate (of pure titanium) and for one last batch by lathing a titanium alloy rod (6% aluminum, 6% vanadium, 2% tin). The thickness of chips is in the range of 0.2 to 1.0 mm, with widths of 1.5 to 4.0 mm. The chips underwent the following pretreatment procedure:

1. Procedure I: The sample was cleaned with chloroform and methyl alcohol or etched with 5% hydrofluoric acid for a few seconds and washed promptly with a large volume of distilled water and dried with acetone. (When etched for a little bit longer, the sample was found to have zero effect in the fourth batch of samples.)

2. Procedure II: The sample was degassed in a vacuum by heating to ~200°C for ~2 h and filled with 2 atm D_2 gas several times to clean the system.

3. Procedure III: The sample was heated to ~400 to 600°C in a sealed sample bottle with 5 to 8 atm D_2 gas, which was absorbed promptly, and a layer of TiD_2 was formed at the surface of the titanium chips. After the D_2 gas pressure dropped to 0.1 atm, the titanium chips were allowed to cool down to room temperature.

4. Procedure IV: The sample was filled with D_2 gas to a pressure of 40 to 90 atm when the temperature of the titanium chips balanced with room temperature.

When liquid nitrogen was removed, the sample bottles were put into the well of the HLNCC-II neutron counter, which was coupled with a JSR-11 shift register coincidence electronic package and an HP-97 computer. The main characteristics of the HLNCC-II are listed in Table I. Using six AMPTEK fast preamplifiers and a derandomizing buffer storage at the input to the JSR-11 greatly enhanced the capability of the system to detect the neutron burst events. The experimental setup is illustrated in Fig. 1. Two main sources of interference to neutron burst determination are being considered, and the precautions being taken are listed in Secs. II.A and II.B.

TABLE I

Detector Parameter for HLNCC-II

Item	Parameter
Cavity diameter	14.5 cm
Cavity height	41.0 cm
Outside diameter	34.0 cm
System weight	43 kg
Helium-3 tubes	
Number	18
Active length	50.8 cm
Diameter	2.5 cm
Gas fill	4 atm
Gas quench	Ar + CH_4
Efficiency	20.0%
Die-away time	~45 μs

II.A. Cosmic-Ray Spallation

An ~30-cm paraffin shielding for the HLNCC-II was built to reduce the coincident background counting rate from 7.08/1000 s to 4.96/1000 s. The HLNCC-II and paraffin shielding were moved to the Mentou Valley underground laboratory, situated ~580 m below ground (Fig. 2), which make the interference of cosmic-ray spallation with burst determination negligible.

II.B. Electronic Noise

During the operation of the HLNCC-II system, it was noticed that a fake signal similar to detection of a neutron burst event could not always be avoided if special precautions were not taken. The fake signals came mainly from the high humidity of either the air in the high-voltage distribution box or in surrounding space, which most likely caused the high-voltage spike. However, a simulation test indicated that vibration such as tapping the high-voltage distribution box or the SR11 package did not cause a burst in the coincident channel.

The following precautions and procedures were taken to minimize the interference of electronic noise:

1. The silicon basket was dried in an oven for 3 to 4 h and was put back into the high-voltage distribution box to absorb the moisture for 2 days before the system was put into operation; this procedure was repeated every 3 to 4 weeks or whenever there was an anomalous increase in the random background count rate.

2. The room temperature was maintained at ~18 to 20°C and humidity at <40 to 70% by air conditioning.

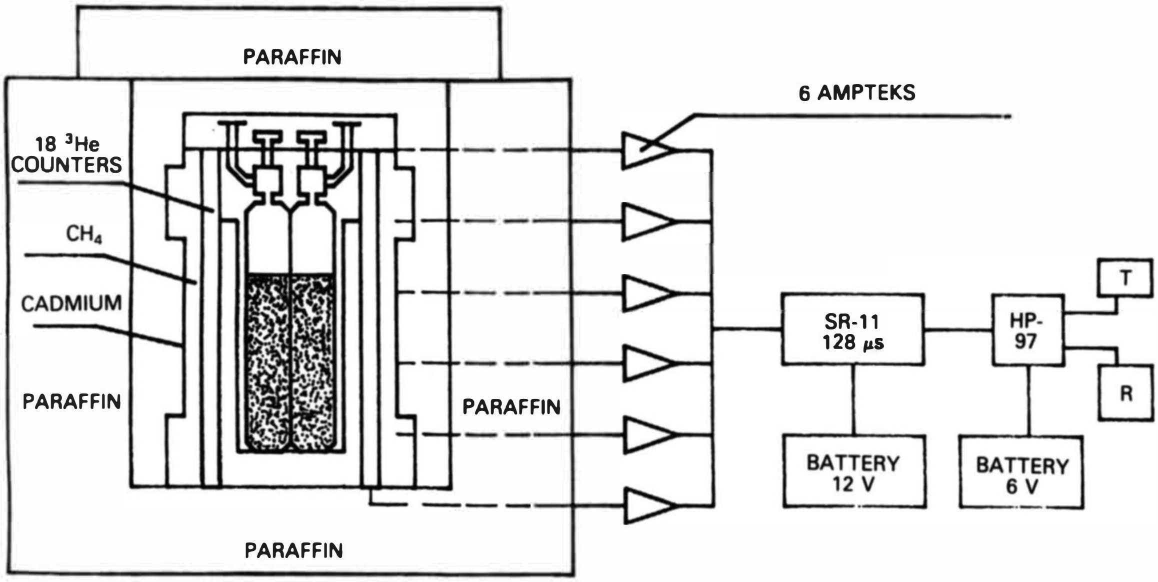

Fig. 1. Schematic diagram of experimental setup for thermal cycle experiments.

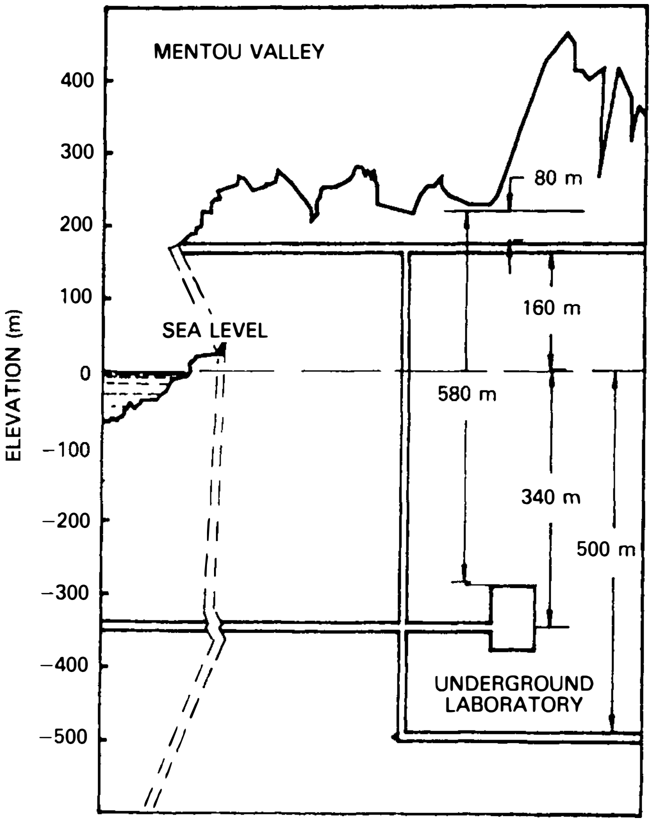

Fig. 2. Diagram of the Mentou Valley Underground Laboratory.

3. Background testing was run 2 or more days before a new batch of sample was put into operation.

4. The batteries were used to power both the HLNCC-II electronics and the HP-97 computer to avoid interference signals through the power line.

5. The H_2 dummy batch of samples was used between two temperature cycles of real samples to check whether the burst signals come from samples or result from misuse of the system. The summary of the experimental results obtained in thermal cycles of deuterium-absorbed titanium chips is listed in Table II.

During the warmup processes of all ten batches of sample, strong effects with relatively high frequency and intensity for burst signals occurred in four of them, along with at least three dominant events containing neutron numbers from 15 to 535 for each signal. Medium effects were found in operation in the eighth batch of sample, which had two medium-sized events. Only three of the batches had no effect. The titanium chips of the fourth sample batch were etched too long (for 30 s) with 5% hydrofluoric acid and that made the surface of the chips covered, most likely, with a black layer of fluoride. During the preparation of the fifth batch of sample chips, the TA1 plate was shaved to chips with a thickness <0.2 mm. Although all the experimental conditions and procedures were controlled carefully, no single burst event was found. The data of neutron generation in operations of some typical sample batches with strong or median effects are listed in Tables III and IV and Figs. 3, 4, and 5, respectively. Careful examination of the results obtained suggests the following:

TABLE II

Summary of Experimental Results from Thermal Cycling D_2-Absorbed Titanium Chips

Batch Number	Rough Material	Pretreatment (Procedure I)	Burst Signal	Underground Laboratory
1	TA1 t,s,d[a]	De-oil	Strong	No
2	TA1 t,s,d	Etch for a few seconds	Strong	No
3	TA1 t,s,d	Etch for a few seconds	Strong	No
4	TA1 t,s,d	Etch for 30 s	No	No
5	TA1 s(<0.2 mm)	Etch for a few seconds	No	No
6	TA1 d	Etch for a few seconds	Weak	No
7	TA1 d	Etch for a few seconds	Weak	No
8	TA1 d	Etch for a few seconds	Medium	Yes
9	TA1 d	Etch for a few seconds	No	Yes
10	Titanium alloy (6,6,2) t	De-oil	Strong	Yes

[a] Here, t = turning, s = shaving, and d = drilling.

TABLE III

Burst Production of Second Batch Sample

Number of Bottles: 3
Material: TAl Turning
Total Amount: 270 g
Pressure: 90 atm

Beginning Date (month/day/year)	Time	Number of Cycles	Warmup Time (h)	$R + A$[a]	Number of[b] Neutrons (n)	$(n + b)/b$	n/σ_b
4/4/90 to 4/10/90		Background		No burst			
4/10/90	19:54	1	1.81	49	34	2.82	6.71
			2.36	1123	221	12.96	44.13
4/11/90	9:10	2	0.14	503	144	9.68	24.88
			3.47	3191	385	24.15	66.36
4/11/90	18:04	H_2 dummy		No burst			
4/12/90	9:30	H_2 dummy		No burst			
4/12/90	18:00	3	4.58	18	15	1.84	3.11
4/13/90	9:30	4	1.53	91	51	3.78	8.00
			2.92	4968	482	27.04	74.96
4/13/90	18:00	5		No burst			
4/14/90	8:50	H_2 dummy		No burst			
4/14/90	15:40	H_2 dummy		No burst			
4/15/90	9:40	6		No burst			
4/15/90	18:00	7		No burst			
4/16/90	9:40	8		No burst			
4/16/90	18:00	H_2 dummy		No burst			
4/17/90	9:15	H_2 dummy		No burst			

[a] Real + accidental counts.
[b] $R = n\epsilon(n\epsilon - 1)/2$; ϵ = absolute detection efficiency.

TABLE IV

Burst Production of Tenth Batch Sample

Number of Bottles: 4
Material: 662 Alloy (6% aluminum, 6% vanadium, 2% tin) Turning
Total Amount: 270 g
Pressure: 60 atm

Beginning Date (month/day/year)	Time	Number of Cycles	Warmup Time (h)	$R + A$[a]	Number of[b] Neutrons (n)	$(n + b)/b$	n/σ_b
10/6/90 to 10/10/90		Background		No burst			
10/10/90	14:45	1	3.75	231	105	308.82	82.08
			4.58	101	68	201.79	53.88
10/11/90	8:30	2	2.08	1376	260	764.15	205.06
			2.64	139	81	237.94	63.85
10/11/90	14:45	3	1.69	1534	274	807.24	216.62
			1.81	2076	320	940.26	252.32
			2.64	2451	348	1022.29	274.33
			4.58	4190	455	1338.88	359.29
			4.86	175	91	267.85	71.88
10/12/90	8:10	4		No burst			
10/12/90	14:00	5		No burst			
10/13/90	8:10	6		No burst			

[a] Real + accidental counts.
[b] $R = n\epsilon(n\epsilon - 1)/2$; ϵ = absolute detection efficiency.

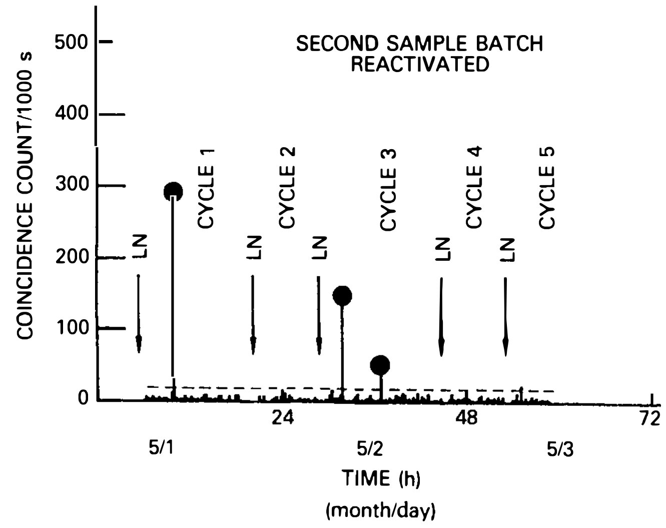

Fig. 3. Burst production of sample batch 2 reactivated.

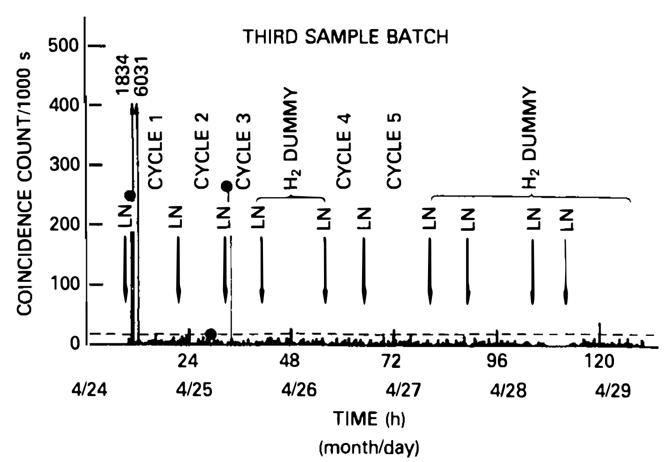

Fig. 4. Burst production of sample batch 3.

1. Neutron bursts come to the coincident channel in a time interval of <128 μs. The intensity of some neutron burst events could be one to two orders greater than the background level, which was calculated on the basis of a 1000-s count.

2. Groups of neutron bursts occur in the first few thermal cycles in the temperature range between −100°C and room temperature. After the active period, the chips seem to be inactive and did not emit neutron bursts thereafter.

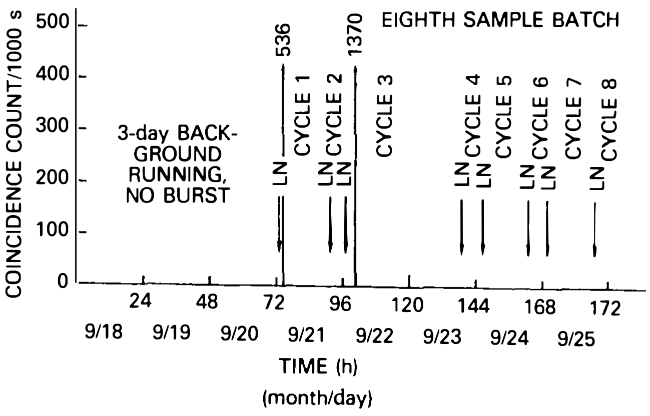

Fig. 5. Burst production of sample batch 8.

3. The inactive chips could be reactivated by degassing and could be recharged with high-pressure deuterium gas; however, the frequency and intensity of burst signals decreased significantly.

4. A great difference between the behaviors of the sample batch and the H_2 dummy batch was found to exist that could not be explained by any sources of interference, background, or system failure recently considered and did not support the conclusion that all these burst events are caused by electronic noise.

5. Fake signals from the HLNCC-II could be eliminated to the extent that they would not seriously interfere with the detection of burst events from the sample batch with the frequency and intensity that were observed in our experiments. However, a multidetector system and neutron energy measurement would be necessary to confirm such a weak anomalous nuclear effect.

REFERENCES

1. M. FLEISCHMANN and S. PONS, "Electrochemically Induced Nuclear Fusion of Deuterium," *J. Electroanal. Chem.*, **261**, 301 (1989).

2. S. E. JONES et al., "Observation of Cold Nuclear Fusion in Condensed Matter," *Nature*, **338**, 737 (1989).

3. A. De NINNO et al., "Evidence of Emission of Neutrons from a Titanium-Deuterium System," *Europhys. Lett.*, **9**, 221 (1989).

4. H. O. MENLOVE et al., "Measurement of Neutron Emission from Ti & Pd in Pressurized D_2 Gas and D_2 Electrolysis Cells," LA-UR 89-1974, Los Alamos National Laboratory (1989).