

Research Article

# Investigation of Possible Neutron Production by D/Ti Systems under High Rates of Temperature Change

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## Abstract

This paper discusses recent attempts to reproduce thermal shock experiments from 1991 using additional diagnostics. This was motivated by an apparent series of neutron bursts which were observed during the deuterium loading of titanium powder at cryogenic temperatures. The neutron count rate and temperature measurements are presented for control and experimental trials. Some neutrons were observed during trials, but the magnitude of the burst which motivated this work could not be replicated. Control trials with inert gas, which should produce no reaction, also demonstrated neutron signal. Future experimental directions are also discussed.

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*Keywords:* Gas loading, Neutron production, Temperature shock, Titanium

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## 1. Introduction

Since the announcement by Fleischmann and Pons in 1989, a number of low energy nuclear reaction (LENR) experiments have been enacted; some claimed to observe neutrons [1–3] while a majority claimed a null result [4]. Of the experiments which recorded evidence of neutrons, significant inconsistency existed among energy and emission frequency [5]. One series of experiments were carried out at the University of Missouri in 1991 using titanium powder which had been saturated with deuterium at cryogenic temperatures; the recently published results indicated that in excess of two million neutrons were produced within a very short time when the system was rapidly heated [6]. There was also a simultaneous effort by the community to develop the theoretical mechanism for this neutron production and determine whether it was hot fusion, a variant of hot fusion called fractofusion [7], or an entirely new process.

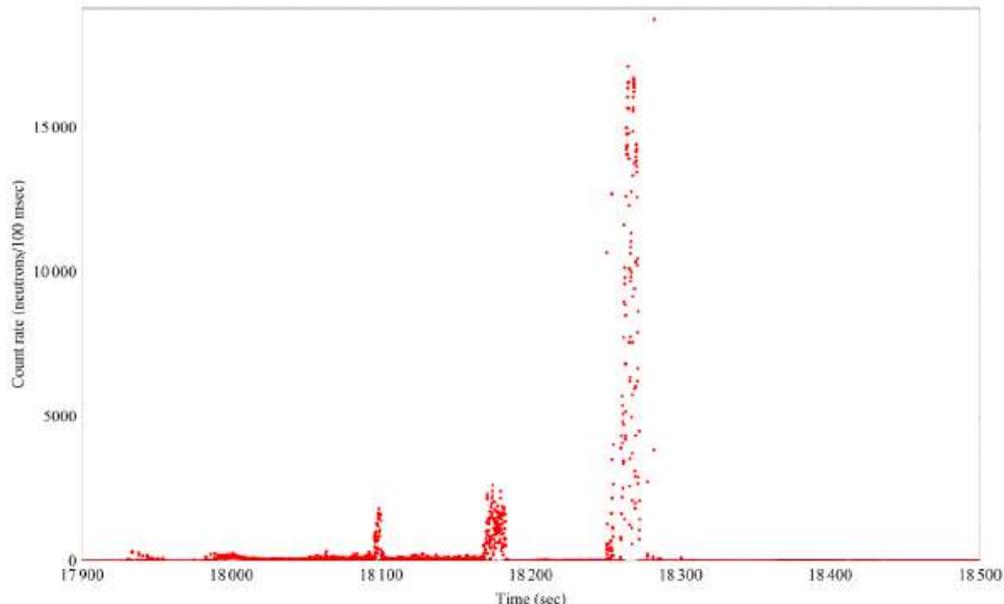
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It was decided in 2012 to perform gas loading experiments similar to those which were completed in 1991. Preliminary results of this recent series were obtained using functionally equivalent apparatus were presented at ICCF-17; they showed consistent results with the 1991 experiments and the literature, namely uncommon temporary increases in neutron production rate during a thermal shock event. Shortly after ICCF-17, the series of significant bursts shown in Fig. 1 were recorded by the helium-3 counter during the cryogenic loading of titanium with deuterium. These observed bursts were similar in duration and magnitude to those observed in 1991. The burst lasted for 10 min and produced 1.5 million total raw neutron counts. After this series of bursts, additional software-controlled diagnostic systems were incorporated to the setup and additional trials were performed. The primary systematic improvements which have been incorporated within the last year are: software-controlled data-logging which now allows for continuous observation of several system parameters of interest; the use of standardized titanium powders whose relatively high surface area should allow for improved deuterium absorption; and the inclusion of argon trials as a control to determine whether or not the apparent neutron signals recorded during experiment are gas-dependent. If the neutron signal only appears during deuterium trials, it would reinforce the claim that neutrons are actually being observed. If the experiment can demonstrate neutron production in a way which is reproducible, deuterium-dependent, and statistically-relevant, then neutron spectrometry will be added to the observation suite and pulse shape discrimination will be used to distinguish the signal from sporadic low-energy gamma rays in the background.

## 2. Theory

The theory which governs the behavior of these experiments relies on fractofusion, whereby large potential gradients are produced along grain boundaries and within cracks that form during the deuterium loading of the metal under investigation, in this case titanium [7,8]. Once the titanium sample is sufficiently loaded with deuterium at  $-196^{\circ}\text{C}$ , both within individual grains and along the boundaries, a rapid temperature increase may drive a cluster of deuterons



**Figure 1.** Anomalous neutron signal during cryogenic deuterium loading.

close enough together for some to overcome the Coulomb barrier and fuse [2,9]. In this case, the process reduces to very abrupt periods of hot fusion, which should consist of roughly even branching ratios for tritium and neutron emission seen below. In other types of LENR experiments, namely electrolysis and co-deposition experiments [10], this was not the case; neutrons were measured with an energy range spanning 3–10 MeV [11] instead of the monoenergetic 2.45 MeV neutrons which would be produced in (2).



### 3. Experimental, Apparatus and Method

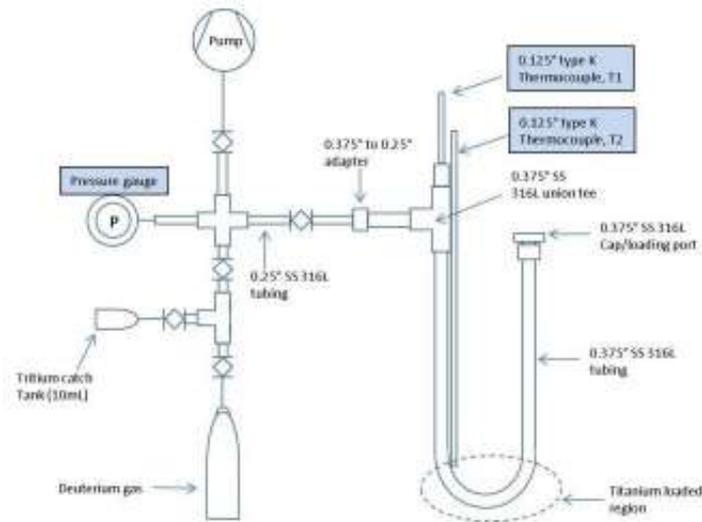
The apparatus used in these experiments is divided into two categories: sample preparation and thermal shock. Common to both categories was the u-tube; a 3/8" 316 L stainless steel tube bent to a 180° “U” shape which contained the titanium powder in use. One end of the u-tube terminated in a fitting; the other end terminated in a cap and was the loading port for titanium powder into the tube. As shown in Fig. 2, a 1/8" × 20" type K thermocouple was connected to one end of the tee and fed into the u-tube through a 1/8" adapter fitting. The thermocouple probe tip was placed in the middle of the bend so that it would be in contact with the titanium sample. The other tee connected to the rest of the experimental apparatus described below.

The materials used in the experiments were titanium and deuterium gas. The titanium consisted of 99.99% pure, –325 mesh, dehydrided, titanium powder purchased from Alfa Aesar. The powdered form of titanium provided the maximum surface area and highest potential loading of commercially available stock. The methods used to prohibit oxidation of the titanium powder are detailed below.

#### 3.1. Sample preparation

The sample of titanium powder was prepared in an inert argon atmosphere to minimize the possibility for a reaction between the titanium and either oxygen or hydrogen in the air before deuterium loading. The apparatus used to accomplish was: the u-tube, a portable glove box, a diaphragm pump, a bottle containing the dehydrided titanium to be loaded into the u-tube, two wrenches, and a reducing fitting. The parts were first cleaned using alcohol and acetone (if appropriate) to minimize glove box contamination and tear hazards. They were then placed within a portable glove box (PGB) and the glove box was sealed shut. The air was then vacuumed out of the glove box using a diaphragm pump rated for 100 μTorr. The pump valve was then closed and a purge was conducted by partially filling the PGB with argon. Three purges were conducted. The PGB was then refilled with argon, leaving enough room to manipulate the tools and materials, and the argon tank valve closed. The cap was then removed from the u-tube and the 1/2–3/8" reducer fitting attached. The titanium bottle was then opened and approximately half of the dehydrided titanium was poured into the tube.

The titanium bottle was then closed and resealed, the valves on the u-tube were closed, and the PGB contents removed. The u-tube was reconnected to the experimental setup immediately after removal from the PGB. The rest of the experimental apparatus was then evacuated using a Pfeiffer HiCube 80 turbomolecular pump station. The rest of the apparatus was then purged using argon in the same manner as the portable glove box. Once the air removal procedure



**Figure 2.** Experimental apparatus for thermal shock experiment.

was complete, the valve separating the u-tube was slowly opened and the entire assembly was then evacuated overnight so deuterium loading could proceed in the morning.

Before deuterium was introduced to the u-tube, the valve on the catch tank was closed to isolate the catch tank from the rest of the experimental system. The valve on the pump was then closed to isolate the vacuum from the rest of the system. The valve to the pressure regulator of the deuterium tank was then opened, allowing deuterium gas to flow into the u-tube up to a pressure of 100 Psi g. After several minutes the deuterium loading was complete, the valve was closed to the deuterium tank, the deuterium tank was disconnected, and the titanium-containing portion of the tube was lowered into a LN<sub>2</sub> bath. The thermal shock procedures were initiated after the bend of the u-tube, which contains the titanium, was exposed to LN<sub>2</sub> for several hours.

### 3.2. Thermal shock

The thermal shock apparatus used in these experiments consisted of: a cryogenic bath filled with liquid nitrogen (LN<sub>2</sub>); a heated water bath; a polyethylene-moderated 252257 helium-3 counter from LND, Inc., which included a Tennelec Tc 952 high voltage power supply, Orten 142PC preamplifier, Ortec 673 amplifier and gated integrator, and Ortec 550 single channel analyzer; a computer capable of running the LabView data-logging program; a National Instruments SCB-68 Connector Block; and the experimental apparatus shown in Fig. 2. The instrumentation attached to the experimental apparatus consisted of a Druck DP104 pressure gauge capable of measuring 68.9 MPa (10,000 Psi g), a type K thermocouple mechanically bound to the u-tube exterior; and a catch tank to capture deuterium gas and other products produced during the experiment for future RGA analysis. Before thermal shock trials were enacted the helium-3 counter was allowed to run uninterrupted to observe any potential defects in device function.

The helium-3 counter output, pressure gauge output, and both thermocouples were connected to the SCB-68 for data logging before an experiment; data were monitored by LabView program developed for this experiment. The signal from the helium-3 counter was fed into an edge-counting virtual instrument. The potential difference produced by the thermocouple was converted into a temperature value using virtual instruments which had been previously verified to

report accurate results. The pressure gauge was set to output a 0–5 V signal proportional to a pressure scale between 0 and 10,000 Psi g. The pressure results were proportional to the temperature results and are not presented due to space. All data were continuously recorded by the LabView program with a period of 10 Hz over a period of days.

After each reloading of the u-tube with new titanium, the experimental apparatus was placed in the insulated LN bath for a period of at least six hours to achieve maximum deuterium loading, with LN<sub>2</sub> replenished as necessary. Once sufficient time had passed and the LabView software was recording, the lid of the cryogenic bath was removed, and the u-tube end of the experimental apparatus was submerged up to the union tee within the heated water bath without touching the bottom. Once the thermal shock was complete, the heater was turned off and the system was allowed to return to room temperature. Once the apparatus had reached room temperature, it was reinserted in the LN bath and the process was restarted a number of times for each titanium powder trial.

Control trials were enacted using argon gas after the deuterium trials were completed. At that time a .01–20 Hz software bandpass filter was incorporated into the thermocouple input channels within the LabView program to reduce the effect of high-frequency noise.

#### 4. Results

A summary of peak neutron count rate and trial duration is provided in Table 1. Characteristic results for the trials are available in Figs. 3–5. Count data from the helium-3 detector were converted into millisecond-interval count rates. The thermal shock of deuterium-loaded titanium appeared to produce a small numbers of neutrons; however no burst was as large as the one observed during the series of 1991 experiments. The largest single burst observed in this series of experimental trials appears to have occurred during the argon control trial; as this behavior is unexpected, it was assumed that all of the bursts may be the result of a false signal occurring within the measurement electronics. However the error could not be consistently reproduced in trials.

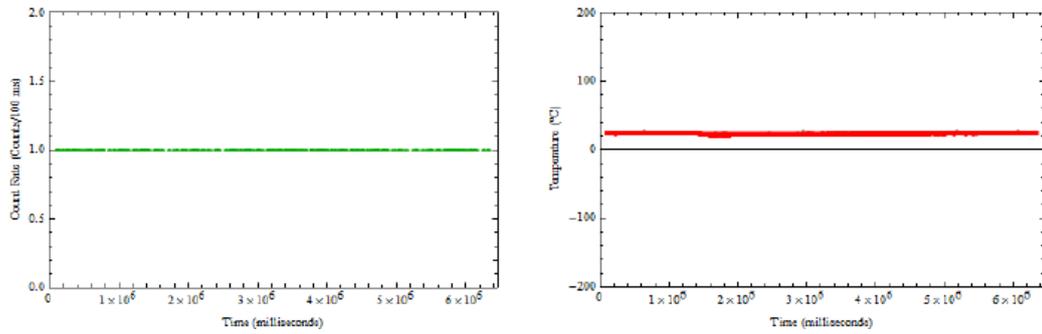
#### 5. Conclusions

The neutron burst from run 4 was of similar duration and magnitude to the burst observed in 1991. Although the results from run 4 were much larger than subsequent runs, some neutrons appear to have been detected during these additional thermal shock trials. More trials will be performed to try to consistently reproduce the large magnitude bursts. Once that is complete, neutron spectroscopy will be implemented.

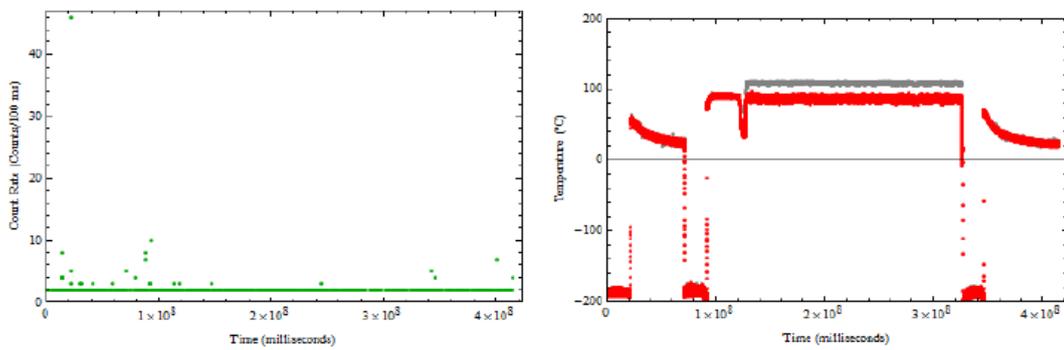
Two future research avenues are available. The first is to continue the series of experiments detailed here using a higher rate of temperature increase, which will require a redesign of the heating mechanism. The second option is to conduct additional experimental trials on deuterium loading of dehydrided titanium powders at cryogenic temperatures in order to replicate the burst shown in Figs. 3–5. Once the cryogenic neutron phenomenon is sufficiently replicated, attention could then return to thermal shock experiments.

**Table 1.** Summary of results using second experimental setup.

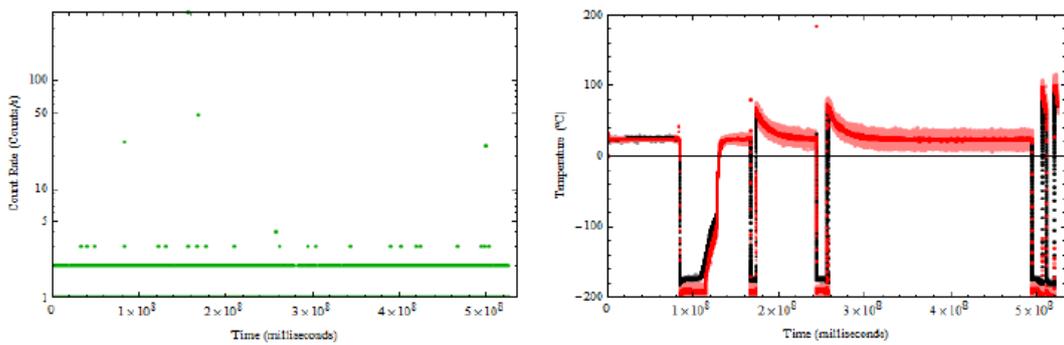
Trial	$\int_0^1 n_{\max}$ (cts/100 $\mu$ s)	$\Delta t_{\text{trial}}$ (days)
He-3 check	3	1.86
1	14	4.07
2	46	4.80
3	9	6.92
4	28	1.79
Argon check	427	6.074



**Figure 3.** Typical background results from the helium-3 counter (*left*) and temperature gauge (*right*) during trials which show no count rates greater than one neutron per 100 ms when the system is at room temperature.



**Figure 4.** Data showing neutron count rate greater than 1 neutron per 100 ms (*left*) and temperature (*right*) during thermal shock trial 2. Interior u-tube temperature is in red, exterior temperature is in gray.



**Figure 5.** Count rate (*left*) and temperature (*right*) results of thermal shock control trials using argon gas which includes the following temperature data: u-tube exterior (*pink*), noise-filtered u-tube exterior (*red*), actual u-tube interior (*gray*), noise-filtered interior temperature (*black*). Actual interior temperature curve (*gray*) is obscured by noise in the external temperature channel (*pink*).

## Acknowledgements

The authors would like to thank the research, administrative, and support personnel at Sidney Kimmel Institute for Nuclear Renaissance. We would also like to thank Dr. Jaewoo Kim of KAERI for his assistance and advice.

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