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EXPERIMENTAL EVIDENCE OF NUCLEAR REACTIONS IN DEUTERATED TITANIUM SAMPLES UNDER NON-EQUILIBRIUM CONDITIONS INDUCED BY TEMPERATURE VARIATION

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ABSTRACT

Several experiments of loading Titanium samples with Deuterium from the gas phase, of changing the temperature of the samples over a wide range and of monitoring the neutron emission were done. Neutron emissions in very low intensity bursts, still significantly above the background were recorded, revealing that low energy nuclear reactions in condensed matter can be produced with a very low rate, which occasionally can be high enough to become detectable.

I. INTRODUCTION

Since the first announcement [1], which stated that nuclear fusion of Deuterium nuclei occurred at low temperature without any external acceleration, experimental work to verify the statement and to investigate the processes has been done all over the world [2] - [5] in sophisticated and in poorly equipped laboratories as well.

In order to verify that nuclear reactions occurred in condensed matter, nuclear radiation should be present; i.e. γ rays, charged particles or neutrons should be detected to a significantly high level during the experiment. In the experiments described hereafter, the neutrons, which are assumed to be produced in the nuclear reaction:

$$d + d = {}^{3}He + n + 3.27\ MeV \quad (1)$$

were searched for. In Eq. (1) the energy of 3.27 MeV resulting from the nuclear reaction is divided between the neutron and the ^{3}He nucleus; the neutron will have 2.45 MeV [6]. The experiment was focused on searching for neutrons and not the γ radiation or charged particles because the neutrons are less attenuated by the 2 cm thick stainless steel wall of the reactor used for loading the samples.

II. EXPERIMENTAL SETUP

A non-spectrometric system, consisting of a BF_3 proportional counter (Nuclear Enterprise), an electronic counter and a printer was used for the fast neutron detection. In order to reduce the background produced by the natural 1.46 MeV γ line of the ^{40}K contained in concrete, a 5-cm thick Lead slab served as a ground shield for the experiments performed in the Nuclear Facility of I.T.I.M. Cluj - Napoca. The BF_3 detector, with a

cathode of 2 cm diameter, has a pressure of 400 Torr and an active zone 20 cm in length is placed (by producer) in a 30 cm diameter polyethylene moderator. The sensitivity is 4 (imp/sec)/(n/cm^2 sec) for thermal neutrons, provided by producer and consistent with the results of the check performed on a regular basis.

III. THE NEUTRON BACKGROUND

At first the neutron background was monitored to determine the occurrence of the counting rates above the background with more than 3 standard deviations (3σ). It is a standard practice to define a neutron emission from a sample, or event, to be a counting rate higher than the background with more than 3σ [7]. Several simple experiments were performed to assess the natural occurrence of such high-count rates.

The counter worked with counting cycles of 10 minutes, printing each result. The counting time was not changed during the experiments, therefore all the results refer to 10 minutes. Three background monitoring runs, each lasting more than the experiments that were performed. We define the e/t value as the ratio of the high count rates to the total number of records for the time span of the experiment. The highest e/t ratio was 0.7%, therefore it might be considered that a rate higher then 1 % in an experiment can be associated with a neutron emission.

Another method employed to test if a sample emitted neutrons was to analyze the distribution of the recorded values. A Poisson or a Gaussian distribution indicates that the neutrons are produced by one source. Distortion of a normal distribution indicates a perturbation produced by additional source [7]. For a Gaussian distribution, the theoretical standard deviation can be computed as:

$$s_t = \sqrt{< N >} \quad (2)$$

where <N> is the average of the recorded values. The standard deviation can be computed using the experimental recorded values as:

$$s_e = \sqrt{\frac{\sum_{i=1}^{n} (N_i - < N >)^2}{n-1}} \quad (3)$$

where n is the total number of recordings. If the relative difference, $(\sigma_e - \sigma_t)/\sigma_t$ is large then the distribution has a distortion. The highest relative difference for the neutron background is 8%. Next section presents the procedure employed for preparing the samples and for loading them with Deuterium.

IV. SAMPLE PREPARATION AND LOADING

A Titanium surface requires a special activation treatment for the Deuterium to be absorbed in the bulk. Special caution must be taken that the sample surface does not come in contact with organic substances for that location would not absorb Deuterium.

After surface activation samples are placed in a stainless steel reactor that can withstand a 9 MPa pressure from Hydrogen isotopes and temperatures ranging from –250 ^{o}C to 900 ^{o}C. The gas is removed from the reactor using a vacuum pump and the samples are maintained at 10^{-1} Torr and 500 ^{o}C for three hours to ensure a complete sample degazing. All four samples were activated in this manner, but the loading process was conducted differently for each of them as will be discussed. The samples consisted of Titanium small slabs, 1.5 mm x 8 mm x 9 mm.

The absorbed Deuterium mass was calculated using data from pressure and temperature monitoring in a known volume for the first three samples and by weighing the sample precisely for the fourth of them.

After the first sample was activated, the Deuterium gas at a pressure of 7.15 MPa filled the reactor containing the sample at a temperature of 500 ^{o}C and then the temperature decreased slowly as the electric heater was disconnected. Subsequently the reactor valve was closed. The pressure decreased rapidly as Deuterium was absorbed into the sample.

The same procedure was used to load the fourth sample while the second sample after activation was maintained at a temperature of 820 ^{o}C for 10 minutes with the Deuterium at an initial pressure of 8.25 MPa and then cooled at 13 ^{o}C during 3 minutes. These samples presented loading ratios between 0.573 and 1.93. The third sample was activated and then maintained at 680 ^{o}C with the initial Deuterium gas pressure of 6 MPa; the reactor with the sample was maintained at this temperature for 5 minutes and then cooled at 13 ^{o}C during 3 minutes. The reactor was closed as soon as the Deuterium reached the pressure of 6 MPa. This different treatment was applied to establish a lower value for the loading ratio and a higher pressure of the Deuterium gas in the cold reactor. After that more temperature variation cycles were done with the sample inside, allowing it to absorb Deuterium several more times.

Loading pressure and temperature, sample mass and loading ratio X defined as the number of D over the number of Titanium host lattice ions, for the four samples are presented in Table I.

Sample	Initial Pressure, MPa	Initial Temperature, ^{o}C	M_{Ti}, g	M_D, g	$X=N_D/N_{Ti}$
1	7.15	500	9.70	0.2322	0.573
2	8.25	820	6.90	0.556	1.93
3	6	680	3.90	0.308	0.31
4	7.15	500	4.9350	0.2771	1.345

Table I. Loading conditions, mass and loading ratios for the four samples.

V. NEUTRON EMISSION AFTER LOADING TI WITH DEUTERIUM

Neutron emissions from samples 2, 3 and 4 were monitored beginning with the moment the Deuterium reached the Titanium surface for samples 2 and 4, and starting 5 hours after that for the sample 3. The temperatures of the samples decreased as the reactor was losing heat in air. The samples were not subject to any other temperature variation during this type of experiment. Results are presented in Table II, where No. n emissions is the number of records that are above the background with more than 3 σ, e/t is the ratio of the total emission time to the total experiment time, R is the average counting rate and B is the average background, where both R and B are in counts for 10 minutes.

Sample	No. of records	No. n. emissions	e/t, %	$(R - B)/\sigma$
2	102	3	2,9	0,2
3	110	3	2,7	1,1
4	121	16	13	1,64

Table II. Results of the post-Deuterium loading neutron emission.

The most relevant, in respect to neutron emission, was the run conducted with sample 4 and the variation of the neutron counts during the experiment is presented in Figure 1. An interesting feature of the neutron emission is that it occurred randomly and in bursts throughout the experiment.

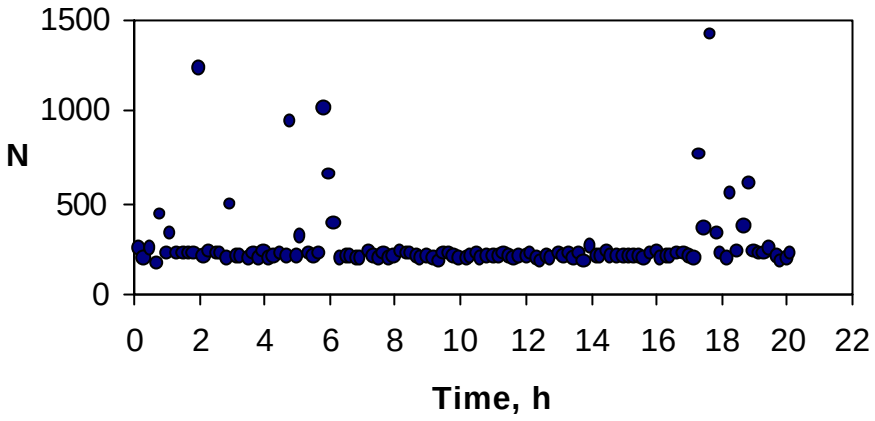

Figure 1. Neutron counts (for 10 minutes) after loading Titanium with Deuterium.

The average background was 225.8 neutrons for 10 minutes, with σ=15. The high values appeared both isolated and as groups over time intervals of 30 to 40 minutes. Another interesting feature of the results is that the average counting rate is higher than the background and for the last experiment with as much as 1.64σ.

VI. NEUTRON EMISSION DURING TEMPERATURE VARIATION BETWEEN 20 ^{O}C AND 700 ^{O}C

One run was conducted for samples 1 and 2 and three runs for the sample 3. Sample 3 was the sample that was loaded at a lower loading ratio to enable it to absorb Deuterium during these experiments. The results of the five runs are presented in Table III, the header being the same that of Table II. It shows that e/t has values considerably above 0.7%, which was the highest value found during the background monitoring.

Sample	No. of records	No. n. emissions	e/t, %	(R – B)/ σ
1	110	2	1.8	0.83
2	25	8	32	2
3	100	26	26	2.5
3	127	13	10.2	162
3	162	16	3.7	0.12

Table III. Neutron emission as a consequence of temperature variation between 20 ^{o}C – 600 ^{o}C.

The first sample was heated and maintained at 700 ^{o}C for 2 hours and cooled slowly to 20 ^{o}C. The pressure inside the reactor was 1 MPa. The second sample was heated and maintained at 700 ^{o}C for 3 hours and cooled rapidly to 20 ^{o}C in 3 minutes; the Deuterium pressure in the reactor was 1 MPa. The third sample was heated and maintained at 600 ^{o}C for 6 hours and cooled slowly to 20 ^{o}C; the initial pressure was 2 MPa and was

not monitored during the other runs. The fourth sample was heated and maintained at 670 ^{o}C for 3.5 hours and cooled slowly to 20 ^{o}C.

The results presented in Table III show a high e/t ratio, close to 1/3 for sample 2 and an average counting rate higher than the background, especially for sample 3, which are a good indication of neutron emission during the temperature variation experiments.

VII. NEUTRON EMISSION DURING TEMPERATURE VARIATION BETWEEN 293 AND 77 K

This type of experiment consisted of monitoring the neutron emission from Deuterided titanium samples during rapid temperature decrease produced by immersing the samples in liquid Nitrogen.

The first experiment involved sample 1 inside the closed reactor. The reactor was placed near the detector and liquid Nitrogen was poured over it. After the temperature decreased, the reactor was completely covered with liquid Nitrogen and it was maintained at 77 K for 2 hours, then it was extracted and heated slowly to 20 ^{o}C. Then immersing was repeated and the Nitrogen evaporated slowly. Neutron emission was monitored for 24 hours.

During the second run sample 1 alone was cooled very rapidly to 77 K by simply dropping it in liquid Nitrogen near the neutron detector. The same procedure was used for sample 2. The results of the data analysis for these three experiments are presented in Table IV, header having the same meaning as in Tables II and III.

Sample	No. of records	No. of emissions	e/t, %	$(R - B)/\sigma$
1	140	2	1.4	0.014
1	22	7	31.8	3.6
2	146	30	21	3

Table IV. Neutron emission during rapid temperature decrease.

During the experiment performed with sample 2 the average background was 206. A sequence of neutron emissions occurred when the liquid Nitrogen evaporated and the temperature of the sample was rising slowly. These sequences, located after the burst of 4821 recorded neutrons and detected 18 hours after the immersion, are presented in Figure 2.

VIII. DISCUSSION

Data from experiments described in this paper was analyzed to check if their distribution is a Gaussian one, as described in Section III. The theoretical and experimental standard deviation was computed for all the experiments performed with samples 2,3 and 4 and results are 289%, 33%, 407%. These values are considerably larger then 8%, which was the highest value found for the background. Therefore it can be concluded [7] that the Gaussian distribution distortion is caused by the neutron emission of the sample.

These experiments performed on TiD_x samples under non-equilibrium conditions confirm the results reported in many other experiments of examining neutron emissions from certain metals highly loaded with Deuterium, [8] - [10]. No other experimental variable related to the processes that accompany the loading of certain metal samples with hydrogen isotopes, like power excess, reported in many experimental papers [1] - [5], was monitored during these experiments.

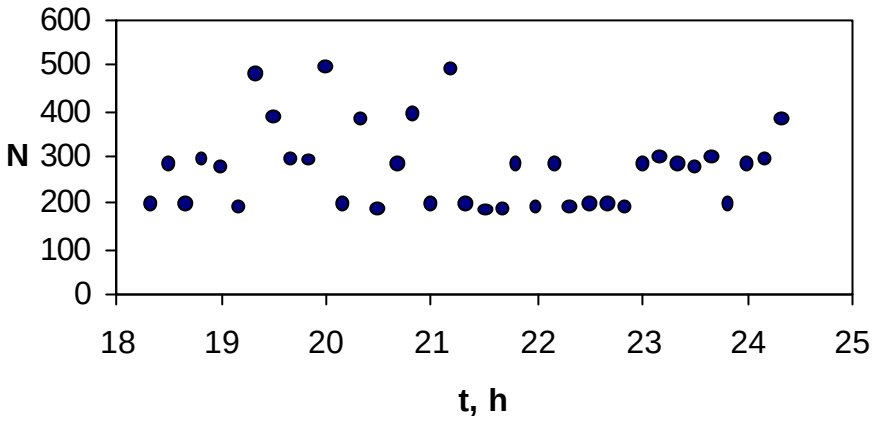

Figure 3. A sequence of neutron bursts during slow temperature increase from 77 K

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