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Tritium production resulting from deuteration of different metals and alloys

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Abstract

Previous experiments have shown that tritium is produced in deuterated titanium. To define better the phenomenon a series of tests have been performed using various metals and alloys and different deuterating conditions. Sheets and shavings of titanium, zirconium, hafnium, tantalum, Zircaloy 2 and Ti-Zr 50% alloy have been tested.

A statistical analysis of the tritium production shows that significant differences are obtained varying the type of metal used. Using pure metals the tritium production increases with the increase of the atomic number of the metal. Moreover higher productions of tritium have been obtained using materials of technical purity as tantalum, Zircaloy 2 and Ti-Zr alloy.

1. Introduction

A possibility of obtaining a cold fusion phenomena was suggested by Scaramuzzi (1) which detected, during a low temperature treatment of gas deuterated titanium shavings, emission of neutrons in bursts of high intensity. A successive analysis performed on the deuterated titanium shavings (2) has allowed to detect in some cases a production of significant amounts of tritium. Also in a study conducted at BARC (3) tritium production has been detected after deuteration of titanium sheets and chips.

In fact it appears that (4) tritium production represents today the strongest evidence of cold fusion events even if reproducibility is always lacking. In order to try to understand the causes of non-reproducibility of the tests we have performed a systematic analysis

of tritium production varying the type of metals and the deuteration conditions.

Up to now tests have been performed only on palladium and titanium. Titanium belongs to the group IVb which comprises also zirconium and hafnium. All the three metals forms hydrides having the same crystallographic habit. In view of the similarity existing between these three metals it appears reasonable to extend the investigations also to zirconium and hafnium. Titanium and zirconium having a purity 99.99% has been used while hafnium has a purity of 97% containing also 2.7% of zirconium. In addition it was decided to test also tantalum which presents a large deuterium solubility (5) and do not form hydrides at temperatures higher than 42°C. The tantalum was of technical purity. Finally some tests on Ti-Zr 50 at.% alloy and on Zircaloy 2 were also performed.

A parameter which appears to be important is the deuterium stoichiometry. For the tests performed using heavy water electrolysis it is usually assumed that a high deuterium content is needed to obtain positive results. The influence of stoichiometry has been investigated on zirconium and tantalum in order to cover the case of deuterides formation and pure solubility.

Finally another parameters that will be taken into consideration is the isotopic enrichment of the deuterium gas.

2. Experimental

To verify if in an experiment tritium has been really produced it is necessary to analyse not only the deuterated metal samples but also the starting deuterium gas and possibly the residual gas which is present in the autoclave after the deuteration. A system has been set up which allows to determine the tritium content in the gas and in the deuterated metals (6).

The analysis of deuterium in the metals is performed degassing the hydrides at 1000°C for six hours in a flow of pure helium. The gas mixture is sent through a tube filled with CuO held at 420°C where the hydrogen species are oxidized to water vapor. The helium, acting as a carrier, transports the water vapor to a trap, cooled by liquid nitrogen. The collected water is then counted in a liquid scintillation spectrometer. The liquid scintillator used is the Tricarb 1400 produced by Packard Instrument Co. The cocktail Pico Fluor LLT, specific for

measurement of low levels of tritium in pure water, has been used. The counting efficiency has been obtained using a tritium standard. The analysis of the tritium contained in the deuterium gas is conducted in a similar way.

The tests were conducted using a selected deuterium having a mean tritium content of 9.5 Bq/g.

An attempt has been made to measure also the possible neutrons emission. The detection system was composed by 16 detectors 1 meter long filled with ^{3}He acting as proportional counters. The detectors were situated in six polyethylene slabs 10 cm thick which act as a neutrons moderators. Only the pulses corresponding to the peak around 764 Kev has been counted in order to minimize the influence of the electrical disturbances.

The six slabs are surrounding the deuteration furnace. In this configuration, using a ^{252}Cf source, the total efficiency measured is 13%.

The deuteration system consists of an autoclave which is connected to one side to a vacuum system and to the other to the deuterium gas bottle and to a calibrated gas reservoir. A tritium analysis on the deuterium collected in the gas reservoir was performed every deuteration in order to compare directly the tritium contained in the feed gas to that contained in the metal. For each type of metals or alloy a blank test using hydrogen was performed. The details of the deuteration procedure are given in ref. (7).

The influence of isotopic composition was examined performing some additional tests with a series of deuterium bottles, produced by C.I.L., having a deuterium enrichment of 99.8, 99.9 and 99.98%. Their tritium content was also around 10 Bq/g. Some tests were performed using deuterium-hydrogen mixture containing 5 and 10 vol.% of hydrogen.

3. Results and discussion

The blank tests performed using hydrogen to impregnate the different metals and alloys above mentioned did not show any tritium production. In the test performed using deuterium the amount of tritium produced was low and the scattering of the data rather high.

Of the different variables investigated the nature of the metal matrix seems to be predominant. A statistical analysis of the tritium produced in the different matrix has been performed. We will consider the tritium production indicative when it is higher than three times the

corresponding standard deviation (σ) and significant when it is higher than five times the σ . In table I are given, for the different metals, the number of tests which comply to these criteria together with the total number of tests.

A further analysis has been performed evaluating the mean value and the standard deviation of the tritium produced in all the tests performed using the same metal or alloy. In table II are shown the obtained values. On the same table is also given a significance factor corresponding to the ratio of the mean value to the standard deviation.

Observing the data of the two tables it appears that both comparisons give the same indications which is a signal that possible accidental errors do not play an important role.

TABLE I: Tests showing indicative and significant tritium production

Material	$T > 3\sigma$	$T > 5\sigma$	N° of tests
Titanium	2 (29%)	-	7
Zirconium	3 (27%)	1 (9%)	11
Hafnium	4 (44%)	1 (11%)	9
Tantalum	9 (75%)	4 (37%)	12
Ti-Zr 50 at%	11 (79%)	4 (29%)	14
Zircaloy 2	3 (100%)	1 (33%)	3

TABLE II: Tritium content: statistical evaluation

System	mean value Bq/g	sign. fact.	N° of tests
Titanium	0.26 + 0.161	1.61	7
Zirconium	0.55 + 0.125	4.40	11
Hafnium	0.72 + 0.147	4.90	9
Tantalum	2.13 + 0.154	13.83	12
Ti-Zr 50 at%	1.64 + 0.105	15.61	14
Zircaloy 2	1.78 + 0.263	6.77	3

The comparison between pure metals show that the tritium production increases with the increase of the atomic number. This is valid also for hafnium even if the metal used contained 2.7% of zirconium.

The materials of technical purity, tantalum and the two zirconium alloys, show a tritium production which is significantly higher than that obtained using pure metals. If we compare the tritium produced in our tests using pure titanium with those reported by Scaramuzzi (9) which uses a titanium alloy, the same effect appears. It has to be noted however that even for technical metal the data are largely scattered. It cannot be excluded that such a high scatter is due to the different distribution of impurities in the different metal sheets.

The samples treated with deuterium of high isotopic purity did not show a significant difference in the tritium production. On the contrary tests performed with 5 and 10% hydrogen-deuterium mixture showed no tritium production.

Stoichiometry did not appear to have any influence.

Neutron counts, during and after deuteration, did not show any difference in respect to background. Assuming that the possible neutron production is equal or lower than the standard deviation of the background and that tritium production arrives during deuteration we obtain a detection limit for the neutron to tritium ratio which is $3*10^{-6}$.

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