# AN INTERPRETATION OF SOME POSTELECTROLYSIS NUCLEAR EFFECTS IN DEUTERATED TITANIUM

NUCLEAR REACTIONS IN SOLIDS

**KEYWORDS:** *titanium hydride formation, deuterium diffusion, postelectrolysis effects*

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*An explanation is proposed for the time dependence of the neutron emission transient observed after interrupting the electrolysis in Fleischmann-Pons–type experiments with titanium cathodes. It is suggested that the time structure of the neutron emission is related to a reduction of active volume (i.e., the volume with a loading ratio higher than the critical value necessary for cold fusion to take place) in the deuterated titanium. This reduction occurs during the postelectrolysis time due to deuterium transport from the $TiD_x$ delta-phase layer to the undeuterated bulk of the cathode. Calculations of the active volume decrease are done by using the Wagner model.*

## INTRODUCTION

The nuclear effects related to cold fusion experiments are characterized by several peculiarities that form a complicated frame. The mechanisms underlying those peculiarities are not completely understood at present, and in several cases, a hypothesis that can be used has not yet been proposed, at least not as a working tool. One of these peculiarities appears as a consequence of the electrochemical loading of titanium with deuterium: the subsistence of neutron emissions after interrupting the cell electric current. In particular, it has been published[1,2] that during one long neutron emission, the electrolytic current was switched off, and the cell was left in open circuit conditions: The neutron emission did not disappear but perdured with an almost constant rate for ~40 min before decaying smoothly below the detector sensitivity. The whole neutron emission transient lasted for ~100 min. Although other postelectrolysis phenomena, both nuclear and related to excess heat production in palladium, have been described by other authors,[3,4] as far as we know, no explanation for them has been found up to now. We propose in this paper an interpretation of these phenomena based on the deuterium concentration decrease that takes place in the highly deuterated titanium layer due to deuterium diffusion, as described by the Wagner model.[5] The main concept of our explanation is that of the active volume, i.e., the sample volume in which the loading ratio (D/Ti) is higher than a critical value necessary for cold fusion phenomena to take place. The use of this concept is highly supported by recent experimental results obtained by several researchers[6,7] and emphasized by the Stanford Research Institute group.[8]

In the first part of the paper, the H(D) concentration profiles in titanium during electrolysis of $H(D)_2O$ are briefly described. Then, the profile evolution after interrupting the cell current is discussed, and the relation between the active volume decrease and the neutron emission transient is analyzed.

## EXPERIMENTAL DATA ON NEUTRON EMISSIONS AFTER INTERRUPTING THE ELECTROLYTIC CURRENT

The experimental results quoted in this section, corresponding to the experiment labeled ETi6, and the experimental conditions in which they were obtained have been previously reported.[1,2] The titanium cathode was a plate 15 × 15 × 1 mm, and $D_2O$ with $Li_2SO_4$ was used as the electrolyte. The potential difference between the cathode and the anode was kept during the experiment at between −4.4 and −10 V. We now describe only those aspects that are relevant to the aim of this paper—mainly those related to the prolonged neutron emission that continued for at least 100 min after switching off the cell current.

Neutrons coming from the electrolytic cell were detected with a $BF_3$ proportional counter (mod2202D from Alnor, Finland) placed ~20 cm from the titanium cathode in the ETi6 experiment. Taking into account the

detector efficiency and the experimental system geometry, we obtained a ratio of $\sim 5 \times 10^{-5}$ for the conversion of the detector counts per second to neutrons per second emitted from the titanium cathode. The signal from the detector was sent directly to a recorder and to an electronic pulse counter.

Twenty-five different neutron emissions were detected during the 800 h that the experiment lasted. Figure 1a shows a semilog plot of the average intensities of the neutron signals as a function of the time of the experiment. Each bar width represents the emission duration. More details about their characteristics can be found in Refs. 1 and 2. The neutron detector was placed 218 h after the beginning of electrolysis. Neutron emissions 24 and 25 are shown in Fig. 1b. During neutron emission 24, the electrolytic cell current was switched off, as indicated in Fig. 1b, and the neutron emission still lasted for ~100 to 120 min (neutron emission 25). The count rate given by the detector remained almost constant or slightly increased for 40 to 50 min after the current was switched off, and then it decayed up to the background level [1 to 2 counts per hour (cph)] shown in Fig. 1c for comparison purposes. The full recorder scale in Fig. 1b was 1 V, much higher than the amplitude of the signal corresponding to an isolated count (6 mV), whose decay time was 30 s.

According to the signal in the recorder and figures from the electronic pulse counter, the entire duration of neutron emission 24 was ~8 h. The total number of counts during this long neutron emission was $\sim 3 \times 10^5$, which gives an average in cph of 39 500. We assume that this average value is valid during neutron emission 25 after stopping electrolysis.

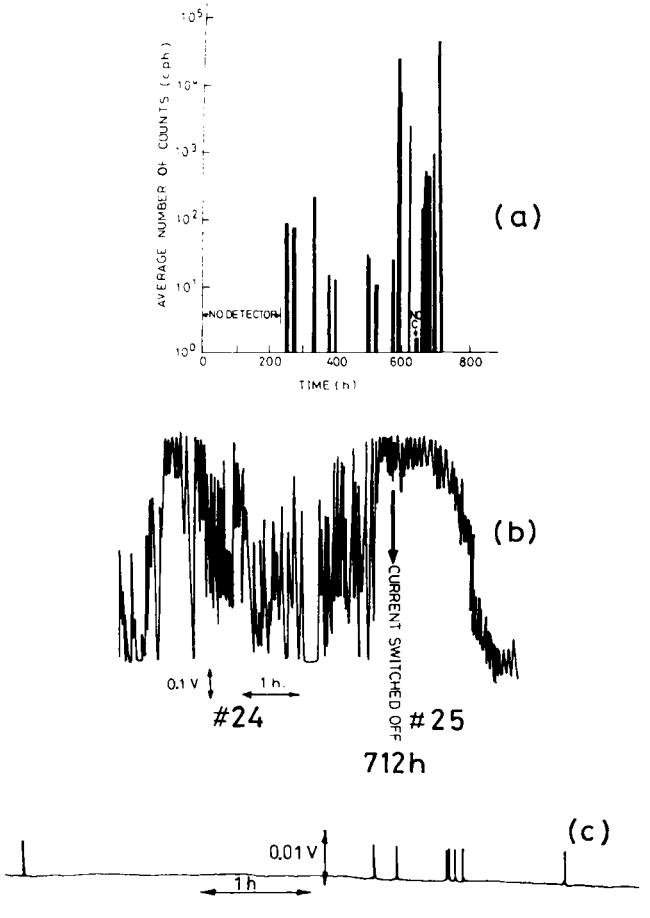


Fig. 1. (a) Semilog plot of the average intensities (in cph) of the different neutron emissions detected during the ETi6 experiment. (b) Neutron emissions 24 and 25 in ETi6 from Ref. 2. (c) Background level of the $BF_3$ proportional counter during the ETi6 experiment.

## DEUTERIUM CONCENTRATION PROFILES IN TITANIUM CATHODES DURING THE ELECTROLYSIS OF HEAVY WATER

Hydrogen and deuterium concentration profiles of electrolytically loaded titanium cathodes were recently published by Mizuno et al.[9] and Fernández et al.[10] Results from other authors are quoted in those papers. References 9 and 10 show that the experimental results are well explained by the Wagner model,[5] which involves the diffusion of one specie [H(D)] in a medium with more than one phase present [the alpha and delta phases in the Ti-H(D) system at room temperature]. The Wagner model assumes that the hydride (deuteride) phase (face-centered-cubic delta phase) nucleates fast enough so its growth is controlled by the H(D) flow through the delta-alpha interface or, in other words, by the H(D) gradient in the delta-layer edge inside the titanium cathode.

When a plane titanium cathode is placed with two platinum plates as anodes (as shown in Fig. 2a) and the electrolysis of heavy water is done to load the cathode, the formation of the $TiD_x$ delta phase takes place in both cathode faces following the equations:

$$\frac{\partial C}{\partial t} = D_\delta \frac{\partial^2 C}{\partial z^2} \; [0, \xi] \; , \tag{1}$$

$$(C_{\delta\alpha} - C_{\alpha\delta}) \frac{\partial \xi}{\partial t} = -D_\delta \frac{\partial C}{\partial z_{\xi-}} + D_\alpha \frac{\partial C}{\partial z_{\xi+}} \; , \tag{2}$$

and

$$C(0,t) = 2 \; , \tag{3}$$

where

$z$ = depths into the cathode

$C$ = deuterium concentration expressed as atomic ratio (D/Ti)

$t$ = electrolysis time

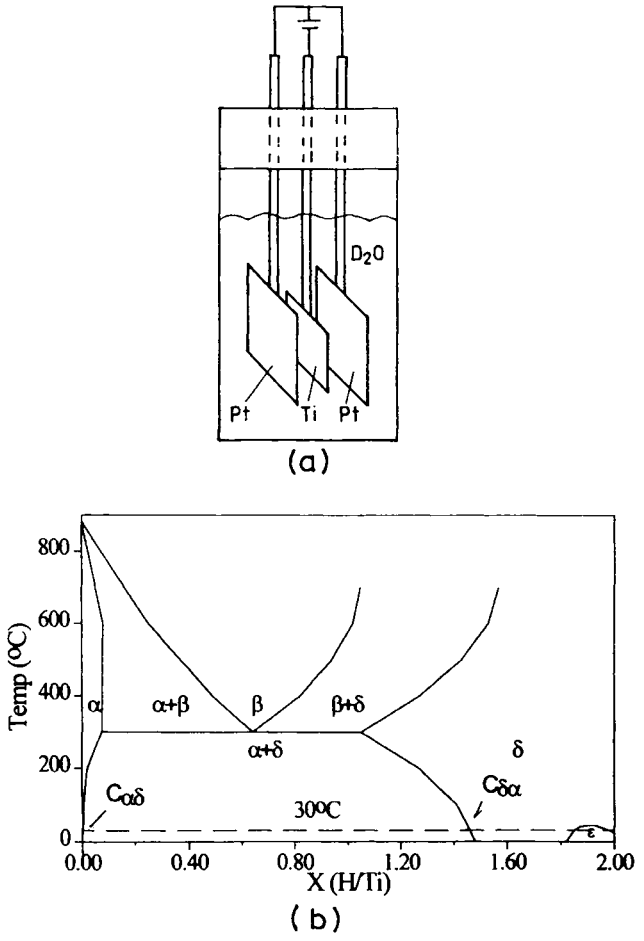


Fig. 2. (a) Electrode arrangement in the ETi6 experiment. (b) Phase diagram of the Ti-H system. Concentrations $C_{\alpha\delta}$ and $C_{\delta\alpha}$ are indicated.

$\xi$ = alpha-delta interface position in the titanium cathode as measured from its surface. The deuterium concentration at this interface is equal to $C_{\delta\alpha}$.

$D_\alpha, D_\delta$ = diffusion coefficient of deuterium in titanium and in titanium deuteride (delta phase), respectively

$C_{\alpha\delta}$ = maximum deuterium concentration in the alpha phase

$C_{\delta\alpha}$ = minimum deuterium concentration in the delta phase.

The terms $C_{\alpha\delta}$ and $C_{\delta\alpha}$ are indicated in Fig. 2b for a temperature of $\approx$303 K at which the electrolysis was done in the ETi6 experiment.

Equation (1) is the diffusion equation applied to the delta layer. Equation (2) describes how the delta layer grows into the titanium cathode, assuming that the deuteride phase nucleation is much faster than the deuterium diffusion through the titanium bulk, the basic assumption of the Wagner model. The two terms on the right side of Eq. (2) represent deuterium flows into and out of the alpha-delta interface. The second term, which involves deuterium leaving the interface because of the deuterium gradient in the alpha phase, is not considered in the calculations that follow because its contribution to the behavior of the delta phase is irrelevant. Equation (3) gives the boundary condition for the diffusion equation. The infinite deuterium source chosen implies that the deuterium flow that enters the near-surface bulk from the surface is larger than the deuterium flow that leaves the near-surface bulk by diffusion. This entry flow is determined by the kinetics of several processes: the kinetics of the Volmer and Tafel processes at the metal electrolyte interface and therefore by the general conditions of the electrolysis (polarization current, hydrogen overpotential, pH of the electrolyte, surface morphology, etc.) and the kinetics associated with the step of deuterium adsorbed at the surface to deuterium absorbed in the bulk. Phillips et al.[11] measured H/Ti in the near-surface bulk after electrolysis and showed that current densities above 5 mA/cm$^2$ ensure a H/Ti ~ 2 during the entire process. We assume the same behavior for an experiment done with $D_2O$.

Deuterium concentration profiles in one face of the titanium cathode during electrolysis are given by the analytical solution of Eqs. (1), (2), and (3), mainly,

$$C(z,t) = 2 - \mathrm{Berf}\left(\frac{z}{2\sqrt{D_\delta t}}\right) , \qquad (4)$$

$$B = \frac{2 - C_{\delta\alpha}}{\mathrm{erf}(b)} , \qquad (5)$$

and

$$\frac{2 - C_{\delta\alpha}}{C_{\delta\alpha} - C_{\alpha\delta}} = 2b^2 . \qquad (6)$$

According to Eqs. (4), (5), and (6), the delta layer in the titanium plate progresses with the electrolysis time, as shown in Fig. 3a. The deuterium profiles shown correspond to $T = 303$ K. The values for the parameters involved in Eqs. (4), (5), and (6) at 303 K are taken from Ref. 10; i.e.,

$$C_{\alpha\delta} \approx 0 \ , \quad C_{\delta\alpha} = 1.5 \ , \quad D_\delta = 2 \times 10^{-11} \ \mathrm{cm^2/s} \ .$$

The profiles from Fig. 3a are in good agreement with the experimental results reported in Ref. 9, which proves the goodness of the Wagner model when applied to the Ti-H(D) system. One can see that the alpha-delta interface progresses slowly in the titanium plate during the electrolysis time. After 1 month of electrolysis, the delta layer extends $\approx$58 $\mu$m into the cathode. The electrolysis temperature plays an important role through the titanium dependence of $D_\delta$. For the same electrolysis time, the deuterated volume is approximately double at 60°C than at 30°C. Figure 3b shows experimental

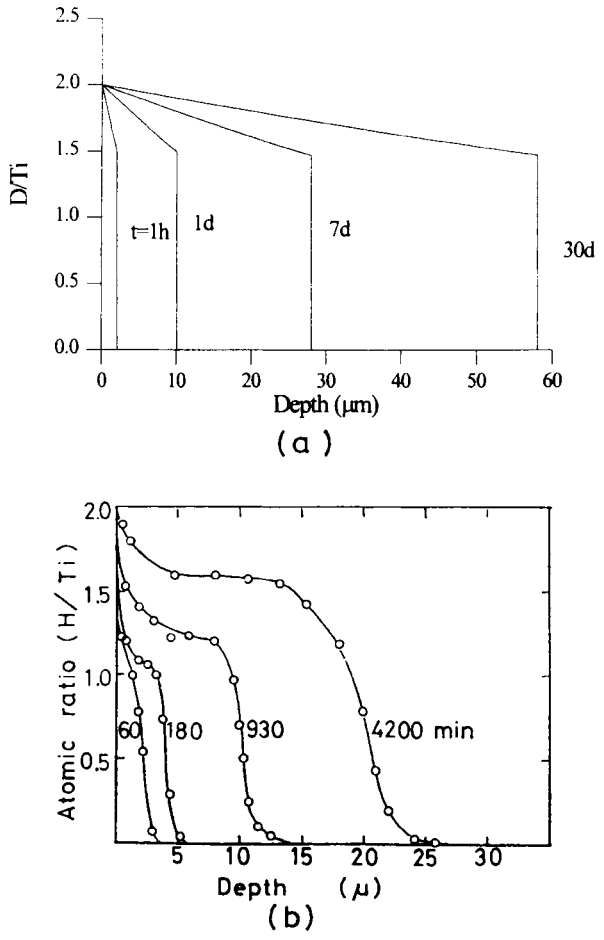


Fig. 3. (a) Deuterium concentration profile evolution in a titanium cathode during electrolysis of heavy water at 303 K according to the Wagner model. (b) Experimental hydrogen concentration profiles in cathodically polarized titanium (1 $N$ $H_2SO_4$, 303 K, 20 mA/cm$^2$) from Ref. 9.

hydrogen profiles in a titanium cathode from Ref. 9. They were measured following electrolysis of ~60, 180, 930, and 4200 min accomplished in a 1 $N$ $H_2SO_4$ aqueous electrolyte at 20 mA/cm$^2$ and 303 K. One can see from Fig. 3 that the agreement between the calculated and the experimental data is quite good both in the shape of the profile curve and in the maximum depth reached by the delta phase during a fixed electrolysis time.

## DEUTERIUM CONCENTRATION PROFILES IN TITANIUM AFTER ELECTROLYSIS

The postelectrolysis evolution of the deuterium profiles is also described by the Wagner model, and it is widely discussed in Ref. 10. Therefore, only the results related to the subject of this paper will be quoted now. The mathematical problem to be solved is the deuterium diffusion equation in the delta phase, Eq. (1), plus the alpha-delta interface progression given by Eq. (2), with adequate boundary conditions at the metal-electrolyte interface and adequate initial conditions.

The initial condition for the postelectrolysis problem is the deuterium profile given by Eq. (4) with the electrolysis time = 712 h for ETi6. However, we have observed in several of our experiments that bubbles evolve at the cathode surface for a short time (1 to 2 min) after the electrolytic current is interrupted. This gas evolution readily decreases, so deuterium losses through the titanium cathode surface are not significant after the initial violent bubbling. We have estimated that only a very thin layer close to the surface (of $10^3$-Å thickness) is involved in the deuterium evolution already described. This initial $D_2$ evolution is taken into account by correcting the initial profile for the calculus assigning D/Ti = 1.5 to the first $10^3$ Å.

The boundary condition $C(0,t) = 2$ suitable for the infinite deuterium source provided by the electrolysis is not already valid. We have accepted that deuterium losses through the titanium cathode surface, except those described in the former paragraph, are not significant during the time after electrolysis. This suggests that a barrier is formed at the cathode surface, and it avoids any further escape of deuterium. This behavior seems to be characteristic of some metals and probably is not the case of others like palladium. Therefore, we have accepted Eq. (7) as an adequate boundary condition for our postelectrolysis deuterium evolution:

$$\frac{\partial C}{\partial z}(0,t)_+ = 0 \ . \qquad (7)$$

Equations (1) and (2) have been solved by numerical methods with the ETi6 figures. The difference technique was chosen. The spatial variable has been discretized in subintervals of 0.1 μm (to take into account the initial deuterium lost, formerly quoted). The time has been subdivided into subintervals of 0.01 min to ensure the method stability.

The deuterium profiles at different times after the electrolysis is interrupted are shown in Fig. 4a. One can see that they change very slowly. The atomic ratio (D/Ti) decreases with time in the delta phase, and simultaneously, this phase progresses into the titanium piece passing from a depth of ≈58 to ≈65 μm 30 days after finishing the electrolysis. The final stable situation is that the delta phase reaches its minimum concentration at 303 K ($C_{\delta\alpha} = 1.5$), and then its progress into titanium stops because the deuterium gradient in the delta phase has disappeared. These results from the Wagner model are in good agreement with the experimental data in titanium, as was recently shown in Ref. 10. Figure 4b shows experimental deuterium profiles from Ref. 10 in two titanium cathodes labeled

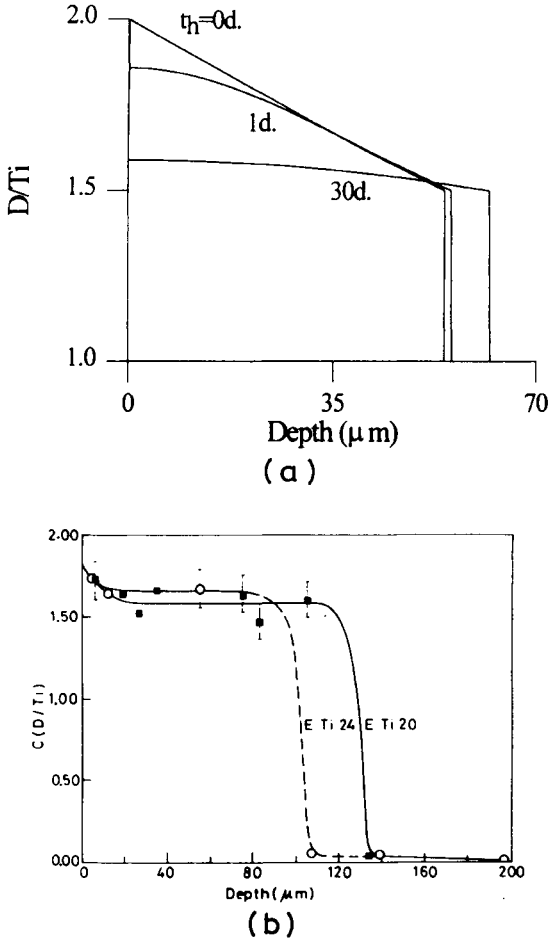


Fig. 4. (a) Deuterium concentration profile evolution in a titanium cathode after the electrolysis is interrupted, as predicted by the Wagner model. The calculations were done with numerical values corresponding to the ETi6 experiment. (b) Experimental deuterium concentration profiles in cathodically polarized titanium 600 days (ETi20) and 180 days (ETi24) after finishing the electrolysis from Ref. 10.

ETi20 and ETi24. ETi20 was used as a cathode in electrolysis that lasted 768 h, done with electrolytic currents between 0.5 and 1 A in a basic medium (0.1 *M* LiOH, $D_2O$). The temperature was not controlled during the electrolysis, and it reached a maximum value of 60°C. ETi24 was loaded under cathodic polarization in an acid medium (0.08 *M* $D_2SO_4$, $D_2O$) at current densities up to 460 mA/cm$^2$. The temperature was controlled at 25 to 30°C during the 1550 h of the experiment. The deuterium profiles were measured 600 and 180 days after the end of the electrolysis, respectively. Therefore, both the experimental results and the calculations show that the main process that takes place after switching off the current is homogenization of the deuterium atomic ratio in the delta phase. For this reason, we call the postelectrolysis time the homogenization time $t_h$.

Figure 5 shows the deuterium profile evolution in a thin layer near the surface ($\approx 10$ $\mu$m) during the first 60 min after switching off the current. Figure 5 clearly indicates that the correction done to the initial deuterium profile, to take into account the initial bubbling, causes a deuterium flow entering the first 1000 Å of the cathode from the rest of the bulk. This deuterium flow to the near-surface region does not last for more than 15 min.

## INTERPRETATION OF THE AFTER-ELECTROLYSIS NEUTRON EMISSION: THE HYPOTHESIS OF AN ACTIVE VOLUME IN $TiD_x$

It is well accepted now that a high loading ratio (D/Pd $\approx$ 1) is needed to favor the appearance of cold fusion–related phenomena in palladium cathodes.[6,7] Therefore, we assume that reaching such a high deuterium concentration in titanium (D/Ti $\approx$ 2) is also a necessary condition for cold fusion phenomena to take place in titanium cathodes. To get D/Ti $\approx$ 2 with electrochemical techniques presents several difficulties, as has been emphasized by different authors.[12,13] It is clear now[9,10] that only a thin layer close to the titanium surface is loaded with a high enough deuterium concentration and that this concentration decreases with time once the electrolysis is interrupted. Therefore, the cathode volume with a high deuterium atomic ratio diminishes with time after electrolysis.

Based on those experimental facts, one can explain the time dependence of the postelectrolysis neutron emission as follows. We accept that a high D/Ti ratio [higher than a certain critical value, $(D/Ti)_c$, unknown

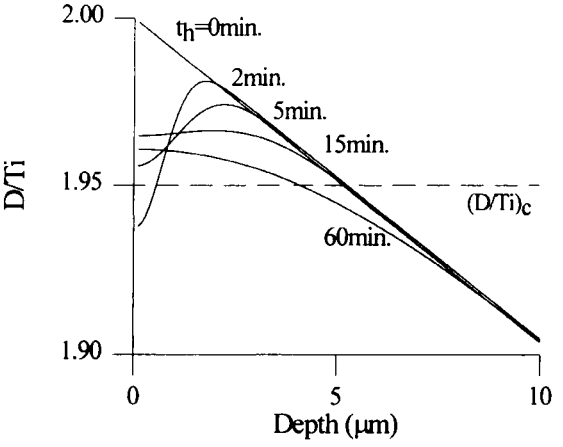


Fig. 5. Deuterium concentration profile evolution in a titanium cathode during the first hour after switching off the current and in a thin layer near the cathode surface. The critical concentration $(D/Ti)_c$ (see text) is shown by the dashed line.

for the moment] is a necessary but not sufficient condition for nuclear reactions to take place: Some triggering effect must also exist. We also assume that whenever such an unidentified effect triggers the mechanisms that produce nuclear reactions in the cathode, their number per unit time, and therefore the number of neutrons emitted, is proportional to the cathode volume loaded with an atomic ratio $\geq (D/Ti)_c$. Such a volume is called active volume $V_a$. It is clear now that $V_a$ [whatever $(D/Ti)_c$ is] decreases during the time following electrolysis and that this decrease could explain the postelectrolysis neutron emission transient observed in the ETi6 experiment and shown in Fig. 1b.

To check the foregoing panorama, we have calculated the evolution of $V_a$ with $t_h$ (the time following electrolysis) from the profiles in Figs. 4a and 5. The results corresponding to several possible values of $(D/Ti)_c$ are shown in Fig. 6. One can see that the $V_a$ decrease with $t_h$ resembles quite well the time structure of the neutron emission transient in Fig. 1b. Two facts must be emphasized. First, $V_a$ diminishes readily, to increase again, during a time that is a function of the loading ratio accepted as critical. Second, $V_a$ decreases smoothly to zero following a time structure similar to that shown by the neutron emission transient.

One can deduce the most probable value of $(D/Ti)_c$ by comparing the time structure of the $V_a$ transient with the detected neutron curve. From this comparison, a critical atomic ratio $(D/Ti)_c \approx 1.95$ is deduced and indicated in Fig. 5 by a dashed line. The initial decrease and increase of the active volume (the cathode volume with deuterium concentration above 1.95) lasts 30 to 35 min; afterward, it diminishes to zero in $\approx 130$ min. Both features appear in the neutron emission detected after the electrolysis is interrupted with similar time constants.

In conclusion, we can say that the time evolution of $V_a$ [with $(D/Ti) \approx 1.95$] could explain the time evolution of the neutron emission after switching off the current. Neutrons become undetectable when $V_a$ exhausts or becomes small enough. One fact of the analyzed experimental phenomenon must be emphasized: The nuclear reaction that produces neutrons was already active when the electrolytic current was switched off.

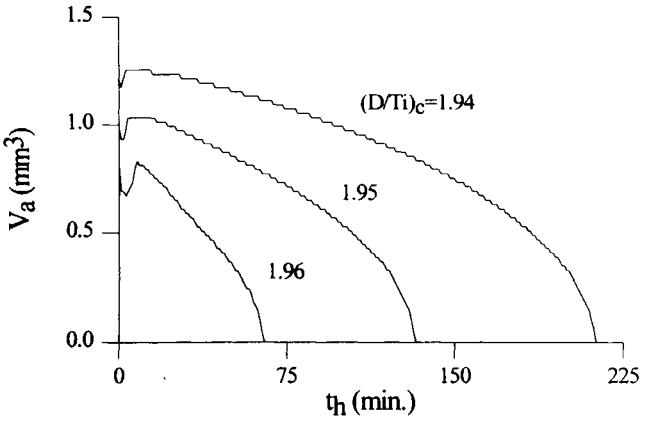


Fig. 6. Active volume $V_a$ as a function of the postelectrolysis time $t_h$ for different values of $(D/Ti)_c$ thresholds as predicted by the Wagner model.

In principle, the simple interpretation that we have proposed should be applicable to understanding neutron emissions that take place during electrolysis. The extension of the former analysis to this situation leads to some striking conclusions. As reported in Refs. 1 and 2 and shown by many other authors,[4,14] neutron emission intensity during electrolysis has transient behavior with a maximum, so its entire time pattern very oftenly resembles a bell shape. This was clearly shown in Ref. 14 and in some of the emissions detected in Refs. 1 and 2. Therefore, one should try to explain at least two fundamental facts: Why does the neutron emission start? and why does the neutron emission become exhausted?

According to the experimental results, a thin layer of highly deuterated titanium ($D/Ti \geq 1.95$) is formed in the cathode in a short time (some minutes) after starting electrolysis, and its width increases with time following a $\sqrt{t}$ law. Therefore, neutrons should be detected soon after beginning electrolysis, and the intensity of the emission should increase continuously on time. However, this is not the situation found in the experiments. For example, in the ETi6 experiment, the detector was placed 218 h after the beginning of the experiment, and the neutrons were first detected 50 h later.

These facts clearly emphasize that to detect neutrons, one must have a highly loaded cathode but that this requirement by itself is not sufficient and that some triggering mechanism of the nuclear fusion reaction must also exist. These two conditions (a very high loading ratio and the appearance of a triggering mechanism) seem to be necessary. According to most of the authors, the triggering of nuclear reactions seems to be related to the presence of some (unknown for the moment) nonequilibrium conditions in the metallic cathode. Experimental results suggest that those nonequilibrium conditions are present in a rather random and unpredictable way, although in some cases they are forced to appear by changing some experimental parameters (for example, the sample temperature[14,15]).

The second point is the transient nature of detected neutron emission. One alternative to explain this behavior is to consider that deuterium nuclei inside the titanium are consumed by fusion reactions (like the fuel in a combustion process), which leads to a decrease of the reaction rate. So, one has to continue electrolysis to refill the cathode before observing a new neutron emission. In this way, some periodicity would characterize the neutron emissions during electrolysis. However, the fusion reaction rates detected in ETi6 imply a decrease of D/Ti of the order of $\sim 7 \times 10^{-16}$/s due

to deuterium consumption in the active volume region. The deuterium transport from the cathode surface during electrolysis is efficient enough to replace the consumed deuterium, and no decrease in D/Ti would be produced. Therefore, one must conclude that the nuclear fusion reactions become nonactive because of some changes in the deuterated region induced by the electrolysis itself. This conclusion is justified by the fact that when electrolysis is not taking place and fusion reactions are already running (like in our detection No. 25), they continue until the time when $V_a$ becomes small enough by deuterium diffusion.

We therefore must accept that a loading ratio $(D/Ti)_c \geq 1.95$ is not a sufficient condition for cold fusion to occur and that a triggering mechanism must appear in the system. Some nonequilibrium situation that appears and vanishes in the cathode during electrolysis should be responsible for triggering the reaction. In absence of electrolysis (like in our detection No. 25), the cathode is not altered, and neutron emission ends only when $V_a$ becomes small enough. The agreement reached between the time dependence of the neutron emission after electrolysis was interrupted in ETi6 and the calculated evolution of the active volume shows that $(D/Ti)_c \geq 1.95$ should really be considered as a necessary condition.

## CONCLUSIONS

An explanation consistent with the experimental data has been proposed for neutron emission transients observed after the electrolysis is interrupted in Fleischmann-Pons–type experiments with titanium cathodes.[1,2] The explanation is based on the fact that the layer volume of the titanium cathode loaded with a high enough atomic ratio changes when the electrolysis current is switched off.[9,10] The Wagner model[5] allows one to calculate the evolution of this highly loaded volume with postelectrolysis time.

It has been deduced that a minimum loading ratio of $D/Ti \geq 1.95$ is necessary for cold fusion reactions to take place in deuterated titanium. It has been also shown from the extension of the analysis to the period of electrolysis that a $D/Ti \geq 1.95$ does not ensure the triggering of the fusion reactions and that some unidentified phenomenon must be responsible for triggering the nuclear reactions. On the other hand, the end of those nuclear reactions during electrolysis seems to be determined by some phenomenon that is not operative in the postelectrolysis time.

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