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AUTORADIOGRAPHY OF DEUTERATED Ti AND Pd TARGETS FOR SPATIALLY RESOLVED DETECTION OF TRITIUM PRODUCED BY COLD FUSION

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Introduction

For the last few months, hectic activity has been underway in various laboratories to study the Cold Fusion phenomenon. De Ninno et al.[1] reported emission of neutrons from titanium metal loaded with deuterium gas under pressure. Similar experiments have been conducted at Trombay. We report here evidence of cold fusion in D_2 gas loaded Ti and Pd targets through the use of autoradiography for spatially resolved detection of tritium. Our study employed three different techniques to observe tritium:

(i) Autoradiography using X-ray films.

(ii) Characteristic X-ray measurement of titanium, excited by the tritium β.

(iii) Liquid scintillation method for tritium β counting.

Loading of Deuterium

Titanium and palladium metal samples of various shapes and sizes were loaded with deuterium by two different ways. In the first method[2] an individual titanium target was heated by R F heating up to a maximum temperature of $\approx$ 900°C in vacuum and then in deuterium gas atmosphere to absorb deuterium. In the second method[3] the foils of palladium (Pd-Ag alloy) were heated (by ohmic heating) up to a temperature of 600°C in vacuum (10^{-5} mm of Hg) and then in D_2 gas. The deuterium gas used for loading had a tritium content of $\leq 5.5\times10^{-4}$ Bq/ml of gas, corresponding to a T/D ratio of ~ 4×10^{-14}.

Autoradiography

Autoradiography is a simple and elegant technique of detecting the presence of radiation emitting zones. This technique has the advantage of being free from any electromagnetic interference (pick ups, discharge pulses etc), has relatively high sensitivity as it can integrate over long exposure times and can give very useful information in the form of space resolved images. In order to achieve good resolution of the image, the sample was kept very close to the X-ray film. Standard medical X-ray film of medium grain size (10 to 15 μm in diameter) on cellulose triacetate base was used for this purpose. The exposure time used for the deuterated samples varied from 18 hours to a few days. At times a stack of several films was used. In some cases films were placed on both sides of the sample. For latent image formation we used IPC (India Photographic Company Ltd.) made 19B developer and IPC made fixer. The developing

time was typically 4 to 5 minutes. Out of many samples which had absorbed D_2 gas, only a few showed a latent image. The results are tabulated in Table I.

The radiograph (Fig. 1) of some of the deuterated titanium disc targets showed several spots randomly distributed within the sample boundary. The occurrence of spots all along the rim of the machined target is very intriguing. Repeated measurements over a period of one month, with the same sample with varying exposure times gave almost identical pattern and relative intensity distribution of spots, indicating that the radiation emitting regions were well entrenched in the face of the titanium lattice. The fact that the second film of a stack of films exposed to the target also indicates similar though less intense spots, rules out the possibility of any kind of chemical reduction reaction caused by the deuterium or hydrogen in the target being responsible for causing the spots. The X-ray image (Fig. 2) of a conical target showed a diffused projection of the cone.

The image of Pd-Ag foils (Fig. 3) however exhibited a more uniform image. The images however indicated variation in intensity and some spots but on the whole the fogging was more or less uniform. Unlike deuterated titanium targets, the intensity of fogging of deuterated Pd foils reduced very rapidly i.e. within a couple of days the activity reduced below measurement level.

Measurements of X-ray Emission

The characteristic X-rays emitted from the deuterated metals (Ti and Pd) were studied with the help of a Si (-Li) (Silicon - Lithium drifted) detector by the Nuclear Physics Division. The detector had a beryllium window of 75 μm thickness. The X-rays of Ti (Kα = 4.5 keV, Kβ= 4.9 keV) were observed in case of conical (Fig. 4) and disc (Fig. 5) samples. The count rate of the conical sample was much more than that of the disc sample. Some of the deuterated Pd-Ag foils indicated the X-ray peaks (Fig. 6) corresponding to titanium presumably because of a small amount of titanium impurity picked up by the foils from the D_2 loading chamber which had earlier been used for loading of Ti samples. We did not observe the L X-rays of palladium or silver.

Liquid Scintillation Counting

This was carried out at the Health Physics Division using the facilities described in Ref. 4. The sample was simply dropped into a vial containing liquid scintillator cocktail and the tritium activity was counted by two photomultiplier tubes in coincidence. The typical activities were 50 to 1000 Bq as compared to a background of less than 0.2 Bq. No correction was applied for possible quenching/ shadowing effects.

Results and Discussion

The fogging observed in autoradiographs (Figs. 1, 2 & 3) is the combined effect of tritium betas and characteristic X-rays of the host material. The radiograph of the disc sample (Fig. 1) indicates evidence of tritium localized in the form of microstructures. These spots are unevenly distributed on the face of the titanium, there being about 60 to 70 spots in all. On correlation with the X-ray counts under the peak (K X-ray peak) and liquid scintillation counting results it was found that the each emitting spot corresponds roughly to 10^9 to 10^{10} atoms of tritium, In comparison the total number of deuterium atoms loaded in the disc sample was 10^{19} to 10^{20}. The X-ray images (Fig. 3) in case of Pd-Ag foils were uniformly fogged and intensity of fogging reduced very rapidly with time unlike with titanium. This type of loss in image may be attributed

to the high mobility of tritium in palladium as compared to that in titanium. Observation of K X-ray peaks of titanium (Figs. 4, 5 & 6) by Si(-Li) detector was the result of excitation of K-shell by tritium β. L X-ray of palladium ($\approx$ 3.6 Kev) or silver ($\approx$ 3.8 Kev) was not observed because of low fluorescent yield for L X-ray and the detector window being too thick (75 μm) to allow observable quantity of L X-rays. Liquid scintillation counting further confirms the presence of tritium in the samples. It may also be pointed out that the quantity of tritium observed even on the surface of the samples exceeded the total quantity of tritium initially contained in the deuterium gas used to load the samples and hence the gaseous tritium, even if preferentially absorbed by the samples cannot explain this phenomena. Undeuterated metallic targets machined out of the same titanium rods did not indicate any detectable tritium, ruling out any contamination pick up during target fabrication/ handling.

Summary and Conclusions

The evidence presented in the paper seems to be indicative of cold fusion reactions occurring in some of the deuterium loaded titanium and palladium targets. It has not been possible to conclusively establish whether the fusion reactions occur during the deuteration process or subsequently. Also it is not clear whether the reactions occur in sporadic bursts or continuously. However one of the disc targets, which gave impressive spotty radiograph did give rise to a significant neutron burst which produced 10^6 neutrons[5] over a period of 85 minutes.

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References

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2 K C Mittal and V K Srikhande, This Report, Paper B2 (1989).
3 M S Krishnan et al, ibid, Paper B4 (1989).
4 T S Murthy et al, ibid, Paper A9 (1989).
5 A Shyam et al, ibid, Paper A4 (1989).

TABLE I

Sample No.	DS001	CS003	PS001
Material	Ti	Ti	Pd-Ag
Shape of Sample	Disc	Cone	Foil
Sample Mass (mg)	980	206	110
D_2 Absorbed (mg)	0.42	0.07	0.73
D_2 Loading process	Ref. 2	Ref. 2	Ref. 3
Date of D_2 Loading	14-6-89	9-6-89	22-8-89
Date of Exposure	23-6-89	14-6-89	24-8-89
Exposure Duration	66 Hr	24 Hr	88 Hr
Figure No.	1	2	3
No. of Times Repeated	9	3	4
Si(-Li) Result (Bq)	290	1300	2960*
Date of Measurement	16-6-89	16-6-89	24-8-89
Total Tritium Atoms	$\approx 1.5\ 10^{11}$	$\approx 6.5\ 10^{11}$	$\approx 1.5\ 10^{12}$
T/D Ratio	$\approx 1.2\ 10^{-9}$	$\approx 3.2\ 10^{-8}$	$\approx 7\ 10^{-9}$
Liq-Scin. Result (Bq)	28	850	xx
Date of Measurement	26-6-89	15-6-89	xx

* Estimated from titanium (present as impurity) X-rays; can be inaccurate.

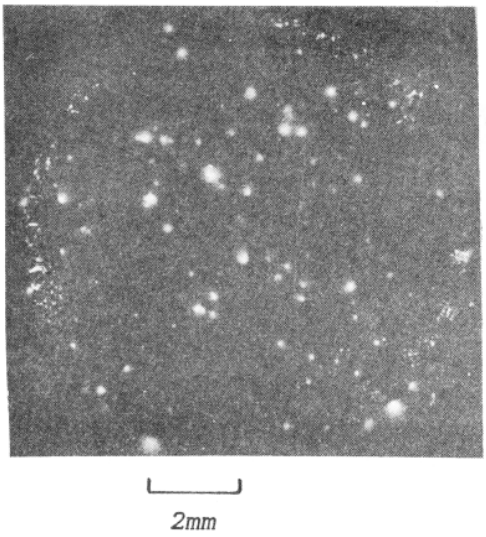

Fig. 1.

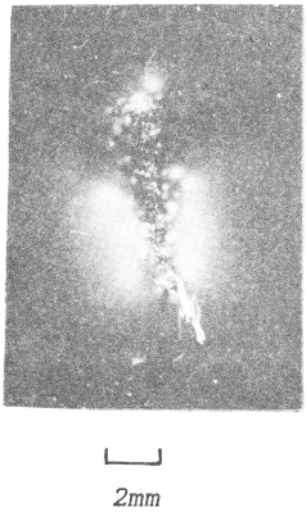

Fig. 2.

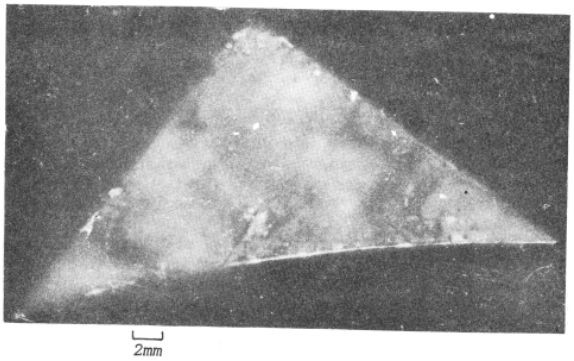

Fig. 3.

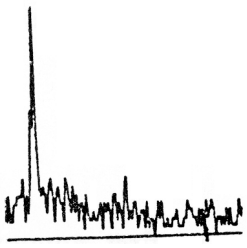

Fig. 4.

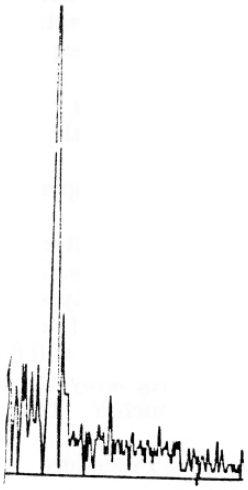

Fig. 5.

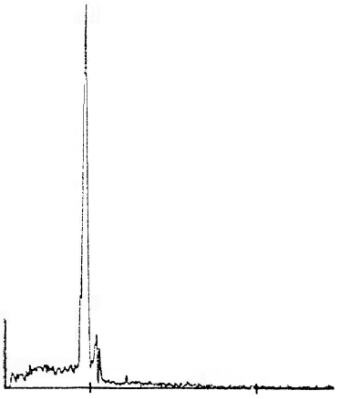

Fig. 6.