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DEUTERATION OF MACHINED TITANIUM TARGETS FOR COLD FUSION EXPERIMENTS

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Introduction

Cold fusion experiments were initiated with solid targets made from titanium loaded with deuterium gas on receipt of reports of the successful Frascati experiments[1]. The absorption of deuterium by Ti is a reversible process and when titanium is heated in a deuterium atmosphere, the reaction will continue until the concentration of deuterium in the metal attains an equilibrium value[2]. This equilibrium value depends on the specimen temperature and the pressure of the surrounding deuterium atmosphere. Any imposed temperature or pressure change causes rejection or absorption of deuterium until a new equilibrium state is achieved. If the surface of titanium is clean, the rate of absorption increases rapidly with temperature. At temperatures above 500°C, the equilibrium is achieved in a matter of a few seconds. However deuterium absorption is considerably reduced if the surface of Ti is contaminated with oxygen. Keeping in view these facts, a procedure was evolved for titanium target preparation and subsequent deuteration. The following sections describe the details of preparation of the targets, their chemical cleaning and degassing followed by deuteration process.

Preparation of the Targets

Titanium targets of different sizes and shapes (planer, conical etc) were prepared. Targets were typically a fraction of a gram in mass and were machined out of a Ti rod using tungsten carbide tools with continuous cooling arrangement. Care was taken to avoid overheating during machining because any overheating could harden titanium and thereby inhibit its capacity to absorb H_2/D_2.

The machined targets were first degreased ultrasonically in trichloroethylene. Then the oxide layer if any was removed by immersing the targets in a 1:1:1 mixture of water, nitric acid and sulphuric acid. They were then rinsed in water and dried in acetone. This was followed by HCl treatment to form an adherent hydride layer on the surface. Targets thus prepared were preserved in a moisture free environment prior to deuterium absorption.

Degassing and Deuteration of Targets

The chemically cleaned targets were first degassed by heating to ~900°C in a glass vacuum chamber using a 3 kW, 2 MHz induction heater. Degassing was continued till a vacuum of less than 10^{-5} Torr was achieved. Targets were then heated to ~600°C in H_2 atmosphere at a few Torr pressure and allowed to cool. H_2 was absorbed in the targets while cooling. Absorbed H_2 was released again by heating to 900°C. At least three cycles of H_2 absorption/ desorption were given to create active sites for D_2 absorption.

After release of all H_2, the targets were heated to ~600°C in D_2 atmosphere at few torr pressure and allowed to cool by switching off the induction heater. D_2 gas was absorbed while cooling. At least three cycles of D_2 absorption/ desorption were given, similar to H_2 absorption/ desorption. The fall in pressure recorded by an oil manometer is a measure of the quantity of D_2 absorbed. It was found that the quantity of gas absorbed increased in each new cycle and tended to saturate in the 3rd or 4th cycle. Table I illustrates the maximum absorption of hydrogen and deuterium in different Ti targets.

It was noticed that targets could typically absorb $\approx 10^{19}$ molecules of D_2. Considering that the mass of the Ti is a few hundred milligrams, this corresponds to an overall D/Ti ratio of $\approx 10^{-3}$ only. However, if most of the absorption is restricted to the surface, as we suspect, it is likely that the D/Ti ratio is higher than 0.001 in the near surface region.

While preparing the targets, we found that successful deuteration depends on various experimental factors as listed below:

(i) Initial sandblasting of the targets for cleaning and roughening of the surface leads to better absorption of D_2.
(ii) Impurity content (such as O_2, N_2 etc) in D_2 should be <0.1%.
(iii) Since the glass vacuum chamber is isolated from the pumping system during D_2 absorption, it is important that the vacuum chamber be leak tight. Small air leaks may contaminate the D_2.

The deuterated targets were sent to the Neutron Physics Division for analysis in quest of evidence for cold fusion. Ref. 3 and 4 describe the autoradiography and neutron counting results.

Postscript

As mentioned earlier the deuteration of the titanium targets was carried out using a 3 kW induction heater operating at 2 MHz frequency. The power supply of this heater became defective in July 89 following failure of the main driver tube,. Since then gas loading of targets could not be carried out in this division. Similar experiments were thereafter commenced at the Heavy Water Division using a resistance furnace as described in Ref. 5. However although the loading procedure adopted there was such that very large quantities of D_2 gas (≈6 litres at 1 kg/cm^2) could be successfully absorbed in titanium pieces (mass ~5 grams), none of the Ti samples have shown any evidence of tritium so far. It is possible that use of high frequency (2 MHz) induction heating may have had some role in causing the detectable levels of cold fusion.

When a metallic object is heated by induction heating, the current distribution within the object is non-uniform with the current density decreasing exponentially from the surface to the centre of the metallic work load[6]. The characteristic penetration or skin depth δ is defined as that distance over which the current density is reduced to 1/e times the surface value and is given by

$$\delta = (\rho/f\pi\mu)^{1/2}$$

where ρ is the resistivity, μ, the permeability of the workload and f is the frequency of the applied alternating magnetic field. For a 2 MHz induction heater the skin depth in titanium works out to be ≈0.1 mm. It is believed that most of the absorbed D_2 gas is accumulated in the near surface region even though the entire sample would have reached high temperatures due to conduction. Hence it is likely that D_2 density is very much higher in the near surface region though the gross D/Ti ratio is hardly 0.001.

Further investigations to confirm these conjectures are underway.

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TABLE I. Maximum Absorption of H_2/D_2 in Different Titanium Targets

Sr.No	Target Shape	Mass (g)	H_2 Absorption		D_2 Absorption	
			mm of oil	mm of Hg	mm of oil	mm of Hg
1	Disc	0.980	35	2.8	22.5	1.8
2	Cone	0.198	16	1.28	5	0.4
3	Cone	0.206	16	1.28	5	0.4
4	Kite	0.610	36	2.88	34	2.72
5	Cone	0.200	18	1.44	15	1.20
6	Disc	0.875	54	4.32	46	3.68
7	Cone	0.460	41	3.28	34	2.72
8	Disc	0.875	46	3.68	33.5	2.68
9	Sponge	0.350	108.5	8.68	85	6.80
10	Pellet	1.025	1000	80.0	955	76.40
11	Cone	0.200	10.5	0.84	11	0.88
12	Cone	0.203	23.5	1.88	29.5	2.36
13	Disc	2.837	208	16.64	372	29.76
14	Cone	0.460	53	4.24	35	2.80
15	Cone	0.203	14	1.12	7	0.56
16	Pellet	1.02	1010	80.8	970	77.60
17	Disc	0.860	57	4.56	40	3.20
18	Cone	0.190	22	1.76	15	1.20
19	Kite	0.605	60	4.80	45.5	3.64