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Preliminary Results on the Variation of Electrical Resistance of a TiD_x Wire With Deuterium Concentration

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Abstract

Experiments have been carried out to study the variation and reproducibility of electrical resistance as a function of the deuterium concentration (D/Ti) in titanium wires. Deuterium loading is carried out in a series of steps by passing a D.C. current to ohmically heat the sample for some time in D_2 gas until a measurable quantity is absorbed. After every loading, the wire resistance and decrease in the gas pressure are measured *at room temperature* using a four probe resistance meter (±0.2% accuracy) and an oil manometer respectively.

Significantly, it is observed that an apparently simple property like electrical resistance is not easily reproducible. The preloading heat treatment and residual gases in high vacuum appear to play an important role on the behaviour of the resistance in TiD_x. The preliminary results also suggest that this property may not be useful in estimating the deuterium content in titanium.

1. Introduction

Titanium has been one of the materials in which occurrence of anomalous nuclear reactions have been reported after being loaded with deuterium either electrolytically[1-2] or otherwise[3-5] Exact origin of products such as neutrons and tritium which typically signify these reactions is not understood due to the fact that such experiments are still not reproducible even if materials are taken from the same stock and other conditions are apparently similar. It is therefore imperative to look for characteristics that distinguish such "identical" samples and affect the results.

Physical properties of titanium-hydrogen (deuterium) system are known to be highly sensitive to presence of oxygen[6]. Surprisingly there is no data (at least to our knowledge) on the variation of its electrical resistivity with ratio D/Ti at room temperature, although at high temperature (beta phase) such measurements have been reported earlier[6-8]. In the experiments reported here, attempts have been made to obtain such data and identify factors which may influence its behaviour. This type of data could possibly also yield a simple parameter which may be used to characterize the loading[9]. The experimental set up and procedure are described in the next section followed by the results, their implications and conclusions in subsequent sections.

2. Experimental Set up

The experimental set up used for deuterium loading is schematically shown in Fig.1. It consists of a glass reaction chamber (cell) having a provision for inserting the mounted sample on one side and connected to a silicon oil filled U-tube differential manometer on the other side. The system is connected to a high vacuum system and gas reservoir etc. through an isolation valve. Volume of cell as well as that of the remaining system are calibrated using a standard flask of known capacity. At times, to produce ultrahigh vacuum (UHV), a glass bulb containing Cesium getter is fused to the cell (and pinched off prior to filling of deuterium gas) as indicated in the figure. Two tungsten feedthroughs are used to mount the sample inside the cell. A four probe resistance meter, having a built in 7.8 mA constant current power supply, is used to measure the electrical resistance with an accuracy of ± 0.2%. The low current ensures that during the resistance measurement temperature rise in the sample is not more than 2^0C.

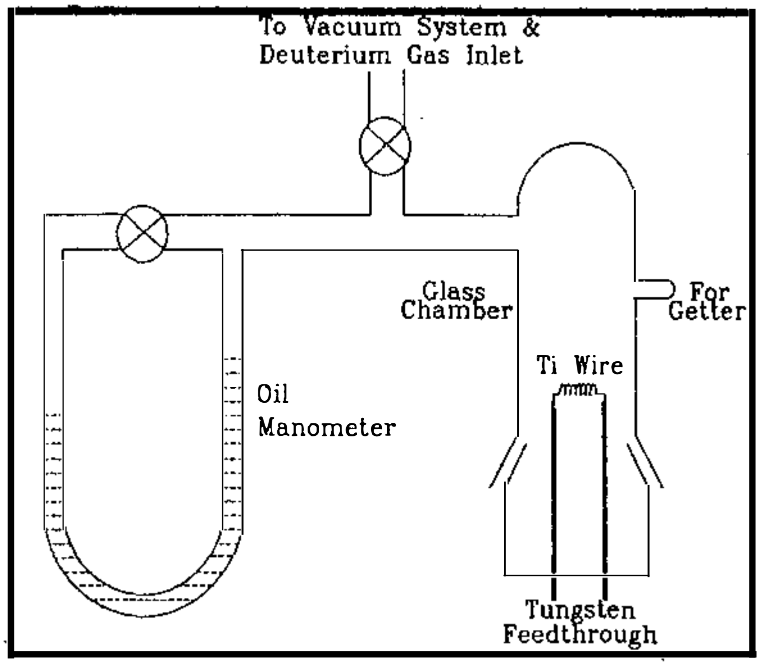

Fig.1. Schematic of The Gas Loading Set-up.

The titanium samples are taken in form of wires (Goodfellow Metals, 99.6% purity). They are cleaned in an acid mixture, weighed and spot welded to the tungsten feedthroughs. Initial resistance (R_{00}) of the free length of wire as well as that of joints and feedthroughs are then measured separately before the whole assembly is inserted into the cell.

The chamber is evacuated to a pressure of 10^{-6} mbar (high vacuum) or better (ultrahigh vacuum produced by the Cesium getter) and a D.C. current is passed through the wire for four minutes to activate it by resistive heating. After the current is switched off and wire cools down to room temperature, its preloading resistance (R_0) is measured. In a few samples, where heating and cooling cycles have been repeated a few times, R_0 has been defined as the value of resistance at the end of last cycle.

Deuterium gas (generated using a commercial Milton Roy cell) is filled into the chamber typically to a pressure of 250 mbars which is sufficient to ensure that the pressure drop on account of absorption remains insignificant. Deuterium loading versus resistance measurements are then conducted in a series of steps.

In every step, the wire is ohmically heated by passing a D.C. current for some time to allow an appreciable absorption of gas to occur and the current is then switched off. After the wire attains room temperature, its resistance (R_1) is measured. The corresponding loading ratio (D/Ti) is determined from the decrease in the chamber pressure (Δp) observed from the oil manometer. These steps are repeated until further absorption of gas becomes practically negligible.

3. Results and Discussion

The initial results obtained from the present series of experiments, which are still in progress, indicate several interesting aspects. During the initial heat treatment of samples in vacuum, the resistance at room temperature is found to increase after every cycle. A similar observation has been earlier reported by Powell and Tye[8], who have attributed it to possible oxygen pick up by titanium from the residual gases in vacuum. Such an effect can also arise due to generation of defects during rapid cooling of a material to room temperature. Since the number of defects should reach a limiting value, a few experiments have been carried out to check if the value of resistance stabilizes after a few cycles of heating and cooling in vacuum. The results are shown in Fig.2. where a trend to stabilize after about 10 cycles can be observed. The actual number of required cycles appears to vary from sample to sample.

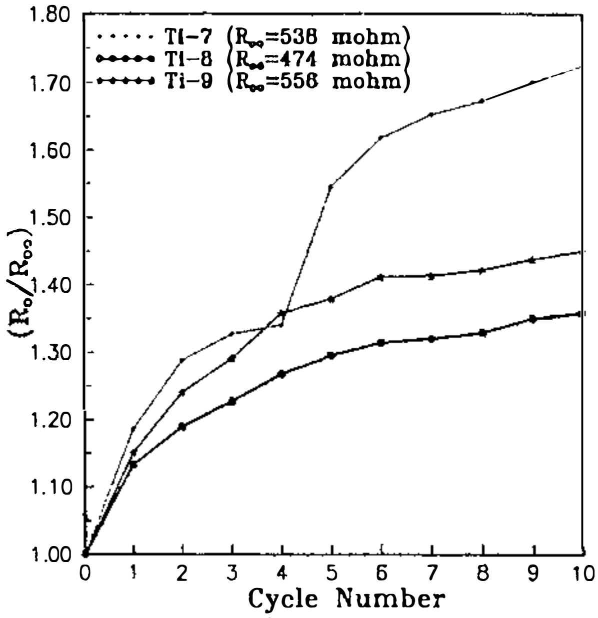

Fig. 2. Resistance Ratio Versus Heat Cycle For A Few Samples.

For most of the samples, the activation cycle has been restricted to just one. Variation of resistance with change in loading ratio (D/Ti) for some of the typical cases is displayed in Fig. 3 and Fig. 4. The samples, according to their experimental conditions may broadly be classified into three categories as noted below.

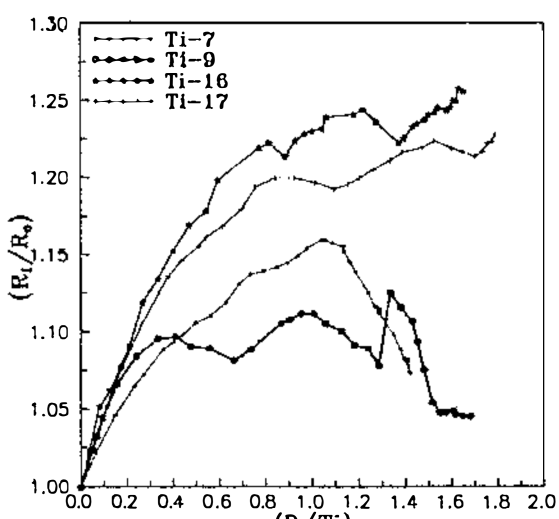

Fig. 3. Resistance Ratio Versus Deuterium Loading. (Heat Treatment in 10^{-4} mbar Vacuum)

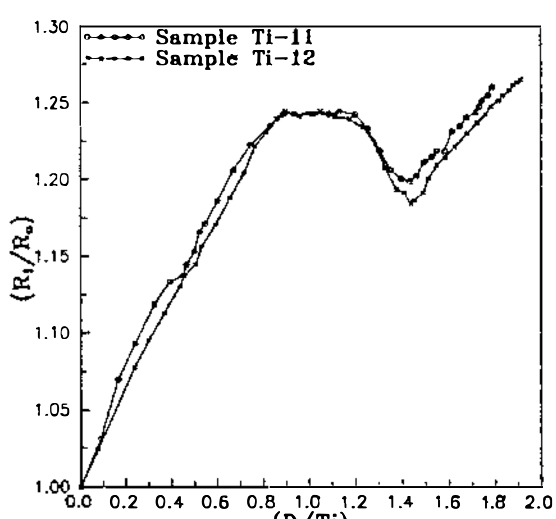

Fig. 4. Resistance Ratio Versus Deuterium Loading (Heat Treatment in UHV).

I. (Sample Ti-7 and Ti-9) Preloading treatment by repeated heating and cooling in high vacuum.

II. (Sample Ti-11 and Ti-12) Heat treatment in UHV for one cycle only.

III (Sample Ti-16 and Ti-17) Similar to Ti-11 and Ti-12 but in high vacuum.

From the results shown here, it is evident that only in case of samples Ti-11 and Ti-12, a reproducible behaviour of the resistance is obtained while an unpredictable trend is seen for other samples. This further supports the earlier indications of the samples being sensitive to the effect of residual background gases during initial heat treatment.

A difference in resistance variation is observed for samples heat treated only once (Ti-16, Ti-17) or more (Ti-7, Ti-9) in similar (ungettered) vacuum. While the resistance ratio (R_0/R_{00}) for former type of samples (Ti-16 & Ti-17) continues to increase beyond the D/Ti loading of 1.4, this ratio is found to decrease in case of samples like Ti-7, Ti-9 which have been heat treated more than once.

4. Conclusions

The results obtained so far in the present experiments imply that apparently similar samples may not yield identical results even for a well known property like electrical resistance. The residual gases and heat treatment conditions definitely appear to play an important role. The data is however to be considered preliminary in nature as various elaborate measurements are underway to cross-check it. Similar further investigations could help to identify many other factors that may affect such physical properties and possible occurrence of nuclear effects.

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