

AN EXPERIMENTAL SYSTEM FOR “COLD FUSION” EXPERIMENTS WITH SELF-PRODUCED IODIDE TITANIUM FILMS

F.CUEVAS, J.F. FERNÁNDEZ, M. ALGUERÓ and C.SÁNCHEZ
Dpto. Física de Materiales C-IV. Facultad de Ciencias. Universidad Autónoma de
Madrid. Cantoblanco 28049. Madrid. Spain.

Abstract

An experimental system has been developed to grow pure titanium films on tungsten substrates. The physicochemical properties of these films have been widely studied and ad hoc samples can be used for Cold Fusion experiments avoiding their contact with atmosphere. Different Cold Fusion experiments are proposed in a new experimental setup that allows deuterium gas loading of the film while electrical current is applied through them. Thus, an experimental configuration similar to an electrochemical loading is attained.

1. Introduction

Most of the Cold Fusion experimental work has been carried out without paying much attention to the physicochemical properties of the metal matrix, as they were usually run with commercial samples. However, the purity and structural properties of the matrix sample have a strong effect on its hydrogenation/deuteration behavior^{1,2}. For example, gaseous impurities can reduce the maximum hydrogen uptake and influence the mobility of the hydrogen atoms. Therefore, we think that the lack of reproducibility in Cold Fusion experiments could be related to some non controlled characteristic of the metal matrix, as was evidenced by the dependence of reported results on the commercial batch production number³. Considering this scene, we have been working on the production of very pure titanium films of well-controlled characteristics to be used in Cold Fusion experiments.

2. Film Growth and Characterization.

Titanium films were grown by a modified Iodide Process. This technique is based on the thermal decomposition of TiI_4 vapors according to the overall reaction

$TiI_4 \leftrightarrow Ti + 4I$. Its major success comes from the fact that the obtained material is nearly free of oxygen, nitrogen and carbon, as these gases do not react with iodine.

In our experimental system, a flow of iodide vapors is passed over a tungsten ribbon filament (40x2x0.025 mm) electrically heated up to 1100-1500 °C and located in a Pyrex glass chamber. The quantity of titanium deposited mass is monitored on line by accurate measurements of the electrical resistance variations of the filament. The obtained samples have an average titanium mass of 60 mg to reach a final thickness about 200 μm. The films can be deposited at distinct filament temperatures and mass deposition rates, by suitable adjustment of the electrical current and TiI_4 vapor pressure. When the titanium deposition is finished, the filament chamber is sealed off. Thus, residual iodine and TiI_4 are taken away and the film is never exposed to the external atmosphere avoiding its superficial oxidation. A more detailed information about the sample preparation system is given elsewhere⁴.

The obtained films were analyzed by EDAX (energy-dispersive analysis of X-rays) and XRD (X-Ray diffraction), and observed by SEM (scanning electron microscopy). Its hydrogenation behavior was also studied in an external system^{5,6}. The main feature yielded by these analyses is the strong diffusion of the tungsten substrate into the deposited titanium films depending on the growth rate and temperature. Due to the tungsten diffusion the obtained films exhibit special physicochemical characteristics compared to pure titanium. It was determined by XRD that the high temperature crystallographic structure of titanium (β-titanium phase, BCC) is stabilized when tungsten exceeds a certain concentration (~8 at%) whereas the common α-phase (HCP) is presented when tungsten concentration is lower than this value. In relation to their hydrogenation behavior, a large hysteresis effect was observed in the β-δ plateau of the P-X diagram that increases as tungsten diffusion does. However, the hydrogenation kinetics of the films does not differ much from pure titanium as the diffusion coefficients of hydrogen in these films have been found to be, at most, an order of magnitude lower than that for pure titanium.

Finally, it is worthy to comment that the titanium deposition rate has also a significant influence on the morphological characteristics of the films. Slow deposition rates produce a very compact bulk structure and high crystallinity, while fast deposition rates can even develop macroscopically cavities inside the films.

3. Cold Fusion experimental setup and features.

Once the film has been grown, the filament chamber is joined to the “Cold Fusion” experimental setup, which is sketched in figure 1. Three different parts can be distinguished in this arrangement: the hydrogenation line, the neutron detection system, and the electrical control system of the sample.

The hydrogenation line, constructed in S.S (316) and Pyrex glass, behaves as a Sieverts type apparatus. The entire system can be evacuated to 10^{-6} torr by means of a turbomolecular pump and it is equipped with Pirani, Penning and Piezoresistive gauges and a quadrupole mass spectrometer (Balzers QMG 064). In the Pyrex line, a

magnetic striker is located to open the filament chamber after the system is evacuated. Several valves allow the entry of hydrogen or deuterium in the chamber by doses.

The neutron detection system comprises two liquid scintillator detectors: a BC501, properly shielded with a 10 cm thick wall of Pb, and a NE213. Far from the experiment, we have installed a BF_3 detector (2202D Alnor) to monitor possible variations of the neutron background. For the sample-detectors configuration our neutron detection efficiency is equal to 0.5%. Neutron energy and time spectra are eventually monitored in different MCA (Canberra).

The electrical control of the filament allows to pass an electrical DC current through the film while two tungsten wires ($25 \mu\text{m}$ OD), welded at its edges, serve as two probes for determining the voltage drop in the film. This configuration constitutes a four probe system. The electrical current is applied by a DC power supply (Keenwood 2036D) which is operated manually or controlled by a function generator. In addition, an electrically heated furnace or a liquid nitrogen container can be placed around the filament chamber to attain temperatures from -196 to 300°C . All the electrical and pressure parameters are finally monitored by a PC.

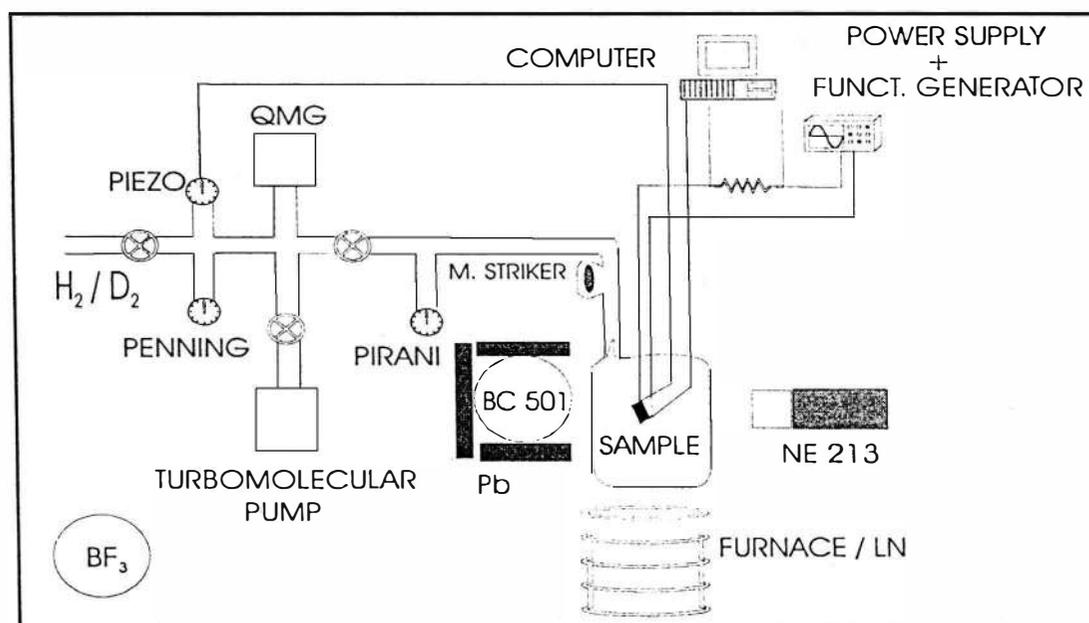


Figure 1. Sketch of the "Cold Fusion" setup.

This experimental system provides very interesting new highlights. Electrical currents up to 12 A can be passed through the sample that is heated up, in this way, to 1500°C . When using the external liquid nitrogen container, sample temperature is cooled down, at least, at -100°C . Hydrogen uptake is measured by the drop of the hydrogen pressure and is correlated with filament resistance variations. Besides, absorption and desorption cycles can be done under controlled rates by electrical heating of the samples. Nevertheless, as an electrical current can be passed through the

sample during its hydrogenation, this system resembles an electrochemical Cold Fusion experiment. This fact probably constitutes the principal highlight of this arrangement. In addition, hydrogen/deuterium diffusion coefficient can be made very similar to those attained in the Pd hydrogenation in electrochemical cells, by heating the Ti samples at 300-500°C. Furthermore, both β -Palladium hydride and δ -Titanium hydride present the same crystallographic structure (FCC) at these circumstances.

Considering the well-known physicochemical properties of the films described before it will be able to relate the possible nuclear reactions with their characteristics. In addition, it should be remarked that the presence of the tungsten substrate will proportionate good mechanical strength to the film during loading-unloading hydrogenation cycles as tungsten does not absorb appreciably hydrogen.

4. Summary

A novel experimental system for the production of titanium samples has been developed and it can be adapted to Cold Fusion experiments. This system provides a good control of the physicochemical properties of the metal matrix and produces films with low content of gaseous impurities even at a superficial level. A wide field of Cold Fusion experiments in the gas phase can be run with the described setup and, under certain circumstances, deuterium loading can be done in a very similar way to that used in electrochemical cells by applying electrical current to the films. We feel that all this scheme will help to obtain interesting and reproducible results in the very next future.

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