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HYDROGEN LOADING EQUIPMENT SIEVERTS' TYPE

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Internal Note No 200/00

This internal note was written in order to serve as a guide line receipt to experimentalists willing to undertake experiments of hydrogenation of titanium alloys using the CRPP's experimental device.

A general description of the experiments and of the apparatus is given. A systematic method of proceeding with the instruments will provide the experimentalists with a necessary " know how " to handle and tackle the problems related with such experiments.

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The present study is part of the "Titanium alloy irradiation testing" task⁽¹⁾ (ITER Task No: T429-1). The objective of this task is to investigate the effects of irradiation on tensile, fatigue, and fracture toughness properties of the Ti-6Al-4V and Ti-5Al-2.5Sn alloys, which include the preparation of hydrogenated Charpy samples to be neutron irradiated and tested. The neutron irradiation will be carried out in the DR-3 reactor at Risø National Laboratory to a dose of 0.3 dpa at 50 and 350°C.

1. Introduction

Titanium alloys appear to offer advantages for use as first wall and blanket structure in a fusion reactor based on a combination of factors which include strength, cost, corrosion resistance and a mature industry⁽²⁾.

One of the problems regarding the use of titanium in a fusion reactor is the compatibility with the fusion reactor environment, particularly with respect to hydrogen⁽³⁾. The presence of quite small amounts of hydrogen in titanium alloys (often < 200 ppm) can result in serious loss of ductility⁽⁴⁾. This is referred to as hydrogen embrittlement, and can be separated into two distinct types based upon rate of load application and designated: impact embrittlement and low strain rate embrittlement. The first produces a gross reduction in ductility, as for example, measured by a fracture toughness parameter, during a rapid strain rate tensile test. This is a form of classical embrittlement due to the presence of a uniformly distributed brittle precipitate (hydride-phase). The second type occurs during slow strain rate conditions, or in long-term static load situations. The time-dependent failure is a consequence of the diffusive mobility of hydrogen at normal working temperatures, causing local concentrations of hydrides precipitates, allowing progressive failure through such embrittled zones. The localised nature of the process allows embrittlement to occur at generally low bulk hydrogen concentrations⁽⁵⁾. Then, hydrogen embrittlement can occur at high or low strain rates, but both types of failure are attributed to hydride-phase precipitation.

Mechanical properties of a titanium alpha alloy (Ti-Al-Zr-Nb-V) with various hydrogen content up to 180 ppm was studied by Kozhevnikov et al⁽⁶⁾. The investigated material represents an alloy in which each alloying element does not exceed its solubility limit in alpha titanium and the whole amount of beta stabiliser is less than 2% (2-3% of residual beta phase).

Impact bending tests have shown that at hydrogen content more than 40 ppm a sharp decrease on impact toughness 8 to 2 happens due to hydrides precipitation after this hydrogen concentration (Figure 1).

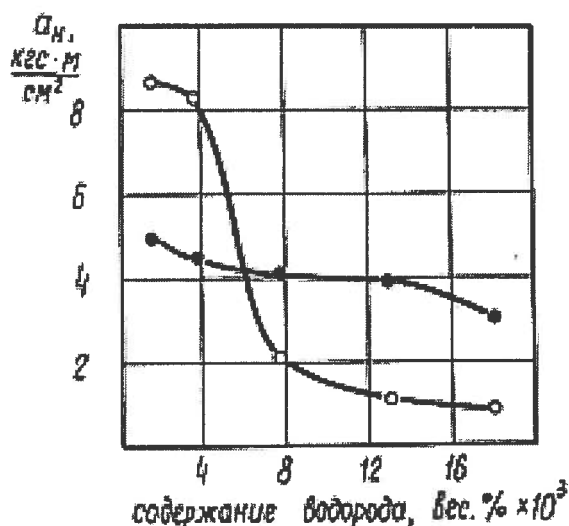


Figure 1: Hydrogen concentration dependence of impact toughness in Ti-Al-Zr-Nb-V alloy before (o) and after irradiation at 10^{20} cm^{-2} (•)⁽⁶⁾. The numbers in the abscissa axis must be multiply by 10 to obtain the hydrogen concentration in ppm units.

After neutron irradiation at a fluence of 10^{20} cm^{-2} (irradiation temperature: 350°C, test temperature: 20°C), the magnitude of the impact toughness reduces from 8 to 4 at hydrogen concentrations lower than 60 ppm. The most probable reason for this last behaviour is that dissolved hydrogen forms hydrides on radiation defects that intensifies hydrogen embrittlement. At hydrogen concentrations higher than 60 ppm, that is to say, at hydrogen concentrations higher than the hydrogen solubility limit in the alloy, irradiation leads to some impact toughness increase and

consequently decrease the susceptibility to hydrogen embrittlement with respect to the same experiment without irradiation (Figure 1).

In order to explain this last behaviour, Kozhevnikov et al studied the hydrogen distribution in the material by means of the autoradiography technique. This technique permits an identification of localised tritium in the metal and an analysis of its distribution in the structure. Autoradiography established that before irradiation the larger content of hydrogen is found under beta phase and hydrides, which arranges along the alpha-beta boundaries.

Neutron irradiation stimulates residual beta phase decay already at 350°C and a large amount of fine dispersion hydrides ($\leq 1000 \text{ \AA}$) of complicated globule form ("wicker ball"). This fact could be explained by neutron influence: hydrogen atoms, changing to alpha phase as a result of radiation stimulated beta phase decay and hydrides dissolution, are collected in depletion zones of radiation cascades. So, neutron irradiation does not amplify the hydrogen embrittlement at higher hydrogen concentration and leads to some increase in impact toughness at hydrogen content more than 60 ppm. This phenomenon is caused by redistribution of hydrogen in the alloy: a large amount of fine dispersed globular hydrides decreasing the magnitude of the plate hydrides.

The objective of this work is the production of a large number of Charpy samples of Ti-6Al-4V and Ti-5Al-2.5Sn at different hydrogen concentrations that simulate the behaviour of Ti alloys candidates in the future fusion reactors.

To choose the hydrogen concentrations it is necessary to take into account that the severity and extent of the hydrogen interaction with titanium can be modified significantly by alloy additions. The most common alloying element is aluminium that significantly alters the hydrogen solubility. The terminal hydrogen solubility has been observed to increase with increasing aluminium content ⁽⁷⁾, as shown in Figure 2. Hydrogen concentrations chosen to verify the effect referred by Kozhevnikov et al is observed in Ti-6Al-4V and Ti-5Al-2.5Sn alloys, were around 150 ppm and 400 ppm, that is to say before and after the hydrogen solubility limit, respectively. Hydrogen charging was performed at high temperatures (see below) which could modify the material properties. Then, reference samples were annealed in vacuum at the same temperature.

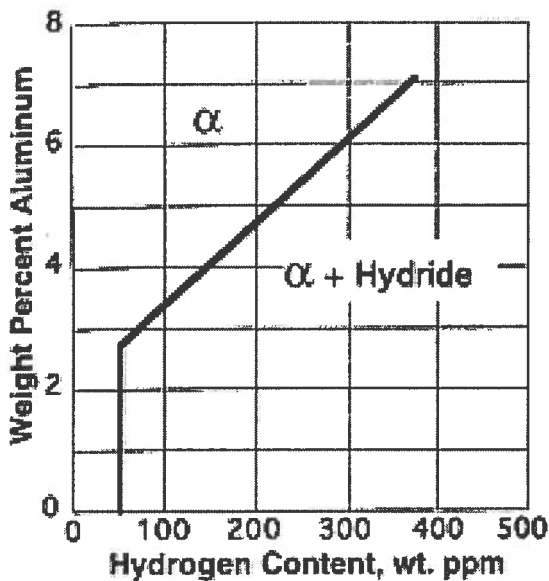


Figure 2: Room temperature terminal hydrogen solubility in titanium as a function of aluminium content.

2. Hydrogen charging

2.1. Materials and heat treatments

Table 1 shows the chemical composition of the Ti-6Al-4V alloy. The material was received with a mill annealing treatment (730°C, 1.5 h, air-cooled) ⁽⁸⁾.

Table 1: Chemical analysis of the Ti-6Al-4V alloy, wt%

Al	C	H	N	O	Fe	V
6.08	0.0056	<0.006	0.0065	0.176	0.14	3.95

The microstructure of the Ti-6Al-4V alloy consists in equiaxed primary α (grain size: 15 - 20 μm), acicular secondary α (grain size: 1 -10 μm) and intergranular β phase (grain size: 0.2 - 2 μm).

The chemical composition of the Ti-5Al-2.5Sn α alloy is given in Table 2. The material received a β - solution treatment (1 h, 1055°C, air-cooled) and then was annealed for 1 h at 815°C and air-cooled.

Table 2: Chemical analysis of the Ti-5Al-2.5Sn alloy, wt%

Al	Sn	H	N	O	Fe
5.0	2.4	0.0036	0.010	0.179	0.36

The microstructure of the Ti-5Al-2.4V alloy consists in equiaxed α grains (grain size: 15 - 25 μm) and 1% of residual β -phase, anisotropically distributed.

Charpy subsized (3 x 4 x 27 mm) specimens of both alloys were machined from the original rod, prior to hydrogen charging.

2.2 Hydrogenation

There are two ways to hydrogenate materials: electrochemical hydrogen loading or gaseous hydrogen charging. Electrochemical charging to introduce hydrogen into materials has several drawbacks. It produces a steep hydrogen concentration gradient from the highly hydrogen-charged outer skin to the core of the sample, the charging process itself may introduce significant damage into the material; and most importantly, it is difficult to relate to in-service exposure to hydrogen containing environments ⁽⁹⁾. For these reasons, gaseous hydrogen charging was chosen. Absorption of hydrogen by titanium alloys in gaseous hydrogen is highly dependent on temperature, gas pressure, gas moisture and oxygen content, metal surface condition, alloy composition and the nature of the surface oxide formed on the metal ⁽¹⁰⁾.

Hydrogen absorption by titanium alloys is usually performed in a so-called Sieverts' apparatus ⁽¹¹⁾⁽¹²⁾⁽¹³⁾⁽¹⁴⁾⁽¹⁵⁾⁽¹⁶⁾. It consists in a quartz tube, where the samples to be charged are introduced, one end of the tube being closed and the other end connected, by means of valves, to a vacuum and a gas input systems, where it is possible to measure pressures and temperatures (Figure 3).

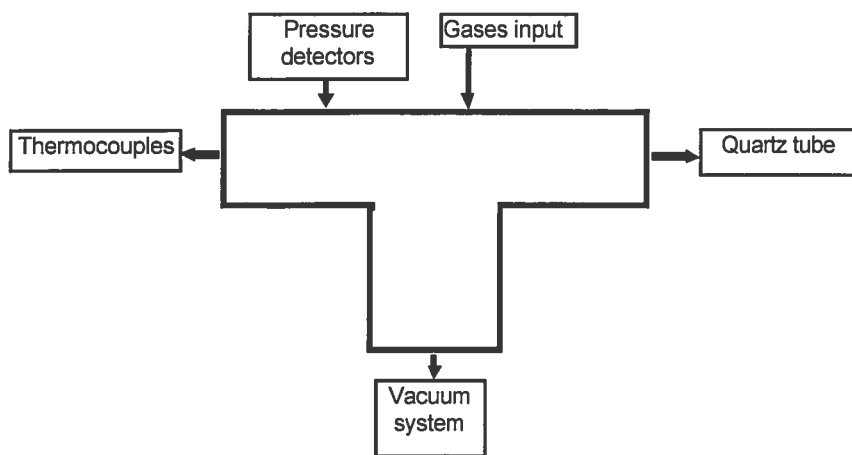


Figure 3: Sketch of the Sieverts' apparatus

On the basis of the apparatus previously described, the hydrogenation equipment shown (Figure 4), was designed and constructed, at CRPP, EPFL.

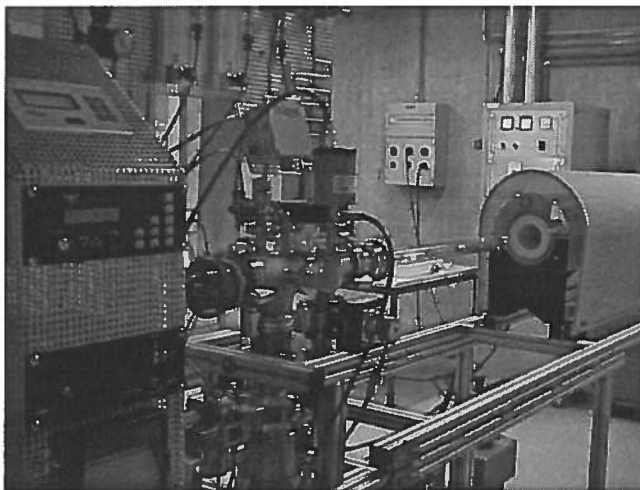


Figure 4: Hydrogenation equipment at CRPP, EPFL.

The work tube is a close one-end quartz tube of 40 mm in diameter and 750 mm long and fits with the main pumping Tee via an aluminium flange. Specimens container, made in 304 SS, is shown in Figure 5. The specimens container is placed in the work zone at the centre of the furnace. The temperature is measured using S-type thermocouples (platinum, platinum-rhodium 10%: $\pm 0.35\%$ T of accuracy when $600^{\circ}\text{C} < T < 1600^{\circ}\text{C}$) and plotted with a Tracecord E100 recorder. The tips of the thermocouples were located in three positions, close to the first, the middle and the last titanium samples surfaces, respectively.



Figure 5: Specimens container in the quartz tube.

The furnace system consists in a SFL tube furnace ⁽¹⁷⁾ with a three-zone capability and a console where is mounted the control. This late is based on an Eurotherm 902P as master controller to programme the rate of temperature rise and the length of time at constant temperature. Two Eurotherm 902S control the border zones, acting as slaves. An independent Eurotherm 92 over temperature alarm ensure the safety of heating elements or furnace contents.

The vacuum system is a combination of a primary rotary pump and a turbo pump capable of reaching 2×10^{-7} torr and can be isolated from the main Tee-quartz tube assembly by a pneumatically operated valve.

The pressure detectors are: 1) a Baratron capacitance manometer, for hydrogen pressure measurement, and 2) a ion gauge, for high vacuum monitoring down to 10^{-2} mbar in the absence of hydrogen. They are directly connected with the main T-tube (Figure 6). The MKS Baratron 627A-12MDC⁽¹⁸⁾ provides a pressure measurement (by means of a MKS Type 651C pressure controller) from 100 mbar to 5×10^{-2} mbar.

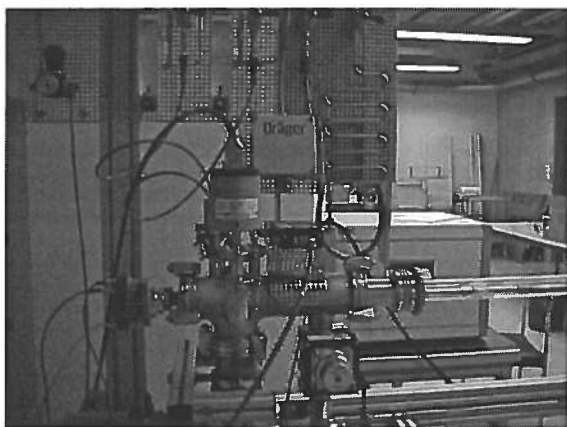


Figure 6: Direct and by-pass pumping unit, with vacuum gauge and hydrogen detector

This manometer can be changed for another MKS Baratron 627A-11MDC that provides a pressure measurement from 10 mbar to 5×10^{-3} mbar.

The MKS Type 1179A mass-flow controller measures and controls the mass flow rates of hydrogen, directly at the main Tee. The full-scale range (nitrogen equivalent) is 10 sccm and it is controlled by a MKS Type LAF 232 to display the reading and to provide the power and set points commands.

It is possible to inject to the system other gases as nitrogen, helium or argon by means of needle valve, directly connected with the main T-tube.

For hydrogen safety, two sensors, connected to a main control unit, are able to detect hydrogen leak at very low level. One is positioned above the main T-tube, near the mass-flow controller and the quartz tube, and the other above the H-bottle, in the storage gas cabinet. The control unit turns on an alarm light when the sensor above the equipment reaches 10% of the LFL* and turns off the alarm when it reaches 8% of the LFL. The sensor above the H-bottle turn on when it reaches 20% of the LFL and turn off when it reaches 15% of the LFL. The control unit turns on a sound horn and closes down the hydrogen supply at the H-bottle closing a valve, when one of the sensor reaches 40% of the LFL. The horn is turned off when the sensor reaches the 35% of the LFL. Moreover, it is possible to do the last action manually pushing an urgency switch placed at the wall. The control unit turns on the alarm light when defects in the H line and safety equipment are detected. In order to reduce the hydrogen concentration in the vacuum pumping system, it is recommended to add N₂ directly to the primary pump by opening a safety valve to allow inlet of nitrogen during hydrogen pumping.

Until now, hydrogenation experiments are performed at a fixed volume of hydrogen, but the equipment has the possibility to make hydrogen charging with a constant flux by means a butterfly valve connected like a bypass between the main T-tube and the turbo pump (MKS 253B throttle valve). When this valve works, the equipment has a pressure control system that works as

* The lowest concentration of the combustible gas capable of leading to ignition is called the lower flammability limit (LFL) of the given gas in air. For H₂, the flammability in air at 20°C and 1 atm is 4%.

follows: 1) the Baratron capacitance manometer senses pressure in the vacuum chamber; 2) this pressure is then compared to the desired set point pressure in the pressure controller; and 3) the pressure controller commands the control valve opening, adapting the chamber pressure to the desired process pressure set point. In this case, the hydrogen content of the specimens would be controlled by the time of hydrogenation and the partial pressure of hydrogen in the hydrogenated gas (H + Ar, for instance ⁽¹⁹⁾⁽²⁰⁾).

The following points must be considered in selecting the conditions for the hydrogen loading:

1. Surface oxides must be completely dissolved. Since surface barriers become less effective at elevated temperatures, hydrogen can be rapidly absorbed and can penetrate deeply into a titanium structure. The natural oxide film on titanium begins to break down at temperatures above about 650°C, hence temperatures below 700°C, at least for initial absorption, are risky. The hydrogenation temperatures found in the literature referring to titanium are between 650 and 850°C for Ti-6Al-4V⁽²¹⁾⁽²²⁾.
2. If the surface is dirty or has a lot of gases absorbed before the hydrogen sorption, the sample will not absorb hydrogen until those gases or contaminant layers are removed. Thus, before each sorption experiment the sample must be degassed by heating at 750°C and 10⁻⁶ mbar for several minutes to reduce the impurities in the body of the samples increasing the free available volume to be occupied by hydrogen atoms and to activate the surface for hydrogenation ⁽²³⁾.
3. The initial absorption time is not thickness dependent, but the achievement of a uniform hydrogen distribution depends on diffusion, and diffusion times are roughly proportional to the square of the thickness for a given temperature and inversely proportional to hydrogen diffusion coefficient ($t=x^2/D$). Hirohata et al.⁽²⁴⁾ measured the diffusion coefficient of Ti-6Al-4V alloy at constant temperatures (700, 750, 800 and 830°C) and obtained

$$D_{\text{Ti-6Al-4V}} = 6.6 \times 10^{-3} \text{ cm}^2 \text{ s}^{-1} \exp \frac{-65438 \text{ J}}{RT} \quad (1)$$

According to the points mentioned in the precedent paragraph and analysing several hydrogen loading found in the literature (for instance, references (25) (26), etc.) the hydrogen loading was carried out following the next steps:

1. Samples degassing at 750°C for 1 hour under dynamic vacuum of 4 x 10⁻⁷ torr.
2. Stabilisation of pressure to room temperature.
3. Vacuum system isolation. Close the main valve.
4. Injection of hydrogen to the main T-tube at room temperature at room temperature.
5. Samples heating in the furnace at 750°C after hydrogen input.
6. Sorption of hydrogen.
7. Homogenisation of the hydrogen in the specimens inside the furnace during 4 hours.
8. Cooling at room temperature extracting the quartz tube for the furnace, injection of nitrogen to primary pump inlet, injection argon in the quartz tube to accelerate the cooling, remaining hydrogen and argon scavenging. This process was mandated because leaving the hydrogen in the tube during cool-down resulted in much higher hydrogen contents because the solubility of hydrogen is greater at lower temperatures ⁽²⁷⁾.

The check list to accomplish the experiences is shown in Appendix I.

Before to hydrogen loading the samples are cleaned subsequently in acetone, trichlorethylene and propilic acid.

To calibrate the equipment, i.e. to obtain $p(C_H)$, the hydrogen concentration in the titanium alloys was estimated using the ideal gas equation (see Appendix II):

$$p(\text{torr}) = 0.03118 \frac{C_H(\text{ppm})m_{\text{Ti}}(\text{g}) I(\text{K})}{V(\text{cm}^3)} \quad (2)$$

where p is the pressure, C_H is the hydrogen concentration, m_{Ti} is the specimens weight, T is the temperature and V is the equipment volume. Appendix III shows how to calculate the pressure in order to obtain a determined value of ppm of hydrogen inside the specimen.

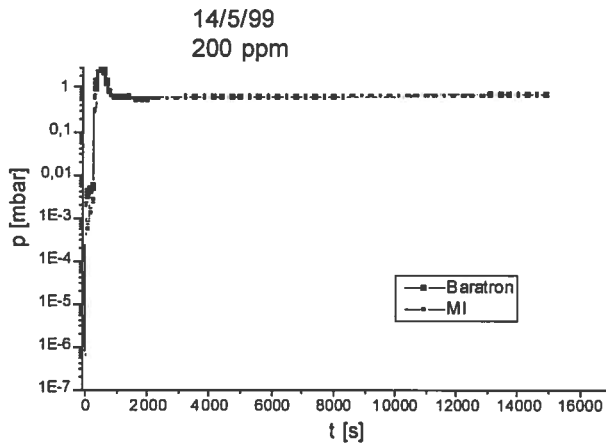


Figure 7: Pressure evolution during the hydrogen loading (MI: ion gauge).

During the hydrogen loading the pressure undergoes the following changes (see Figure 7 above):

1. an increase due to the vacuum system isolation (-when closing the - main valve.),
2. a second increase due to the hydrogen injection in the system (at room temperature),
3. a third increase due to the system heating,
4. a decrease that indicates the sorption (the temperature for this process was around 600°C),

5. a little increase and decrease due to the fluctuations in the temperature to introduce the furnace on the quartz tube (the same behaviour is observed without hydrogen a slow increase during the hydrogen homogenisation process.

Finally, the heating is interrupted and the system goes back to room temperature.

To obtain the calibration, $p(C_H)$, three different values of pressure were chosen to charge the samples and the vacuum-fusion method (LECO, Hot Lab, PSI) was chosen for the determination of hydrogen content of Ti alloys. Figure 8 shows the calibration obtained.

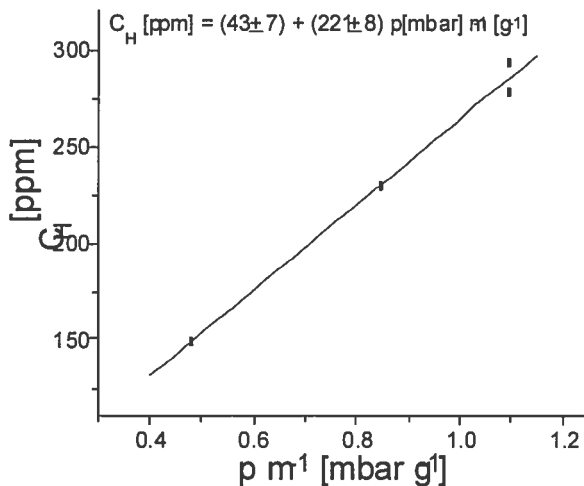


Figure 8: Sieverts' apparatus calibration

Therefore, the pressure necessary to put in the equipment to obtain the hydrogen concentration required in the samples:

$$p(\text{mbar}) = \frac{(C_H(\text{ppm}) - 43)m_{\text{Ti}}(\text{g})}{221}$$

The flow rate in sccm units, F (sccm), must be calculated as is described in Appendix II. We prepared 48 samples to irradiate in DR3 (RISØ) and 48 samples to test without irradiation (Appendix IV).

The specimens to be analysed were selected at random from each test, cleaned of any oil, grease and foreign matter weighed and inserted in a LECO.

The results are shown in Table 3, below.

Table 3: H content in the samples

Samples	Hydrogen [ppm] (aim)	Hydrogen [ppm] (obtained)
Ti-5Al-2.4Sn (annealed)	reference	5
Ti-5Al-2.4Sn	150	152
Ti-5Al-2.4Sn	400	435
Ti-6Al-4V (annealed)	reference	5
Ti-6Al-4V	150	167
Ti-6Al-4V	400	436

CONCLUSION

The device, which has been presented in the present publication, is well adapted for hydrogenation of material samples that could be potential candidates for fusion device as for example for internal components. Moreover, this equipment could be used to do heat treatments in materials.

APPENDIX I

Check list for using the LAF 232 (mass-flow controller)

1. Turn on the LAF 232 (switch on the left).
 2. Allow 2 minutes for warm-up time (try to leave the power to the instrument on at all times, for optimal performance).
 3. Check the flow controller zero. If it is necessary, adjust the "ZERO" pot until the readout displays zero, using a small screwdriver.
 4. Set the "FLOW DISPLAY" switch to the fugitive position "GAS CORR." to display the gas correction factor (Ar: 14.5; He: 14.5; H₂: 10.1; N₂: 10.0). This value can be changed using a small screwdriver.
 5. Set the "FLOW DISPLAY" switch to the fugitive position "SET PT." to display the flow rate in sccm units. This value can be changed using a small screwdriver.
 6. Check the "SET PT." switch is in the "INT" position.
 7. Set the "ON/OFF/REM" switch to the "ON" position to establish the flow rate (the green led will turn on) during the desired time. Then, switch to the "OFF" position (the green led will turn off).
- NOTE: the pressure value on the mass-flow controller (MKS type 1179) must be less than 0.8 bar.

Check list to begin the vacuum

1. Turn on the primary pump (switch: position 1).
2. Open the "Speedivalve" (valve between the turbo and the primary pump).
3. Turn on the turbo pump (press the "Pumping Unit" key in TCP 300).
4. Turn on the pneumatically operated valve (valve between the main T-tube and the turbo pump).
5. When "Rotation" on TCP 300 reaches the 60% of the green zone, press the "PWR" key in APC 2000, to turn on the filament of the ion gauge.

Check list to open the equipment

1. Turn off the Baratron.
2. Turn off the mass-flow controller.
3. Close the "Speedivalve".
4. Turn off the turbo pump (press the "Pumping Unit" key in TCP 300). The air will enter to the equipment.

Check list to cool the samples

1. Open the N₂ valve to the primary pump (open carefully! You have not hear a primary pump noise)
2. Turn on and take off the furnace of the quartz tube.
3. Open the needle valve to send Ar until the Baratron shows around the full-scale range.
4. Turn on the pneumatically operated valve. When the Baratron displays ~ 0 (seconds) turn off the pneumatically operated valve.
5. When the samples temperature reaches a value less than 100 °C open the equipment.

Check list for using the TRACECOR E100 recorder

- Turn on the TRACECOR E100: The message 8.8. 8.8.8.8.8.8. will be displayed. The, the message MESURE and finally the temperatures measured by the thermocouples: 1 value, 2 value, and 3 value.
- To stop the impression: Press the < key until the message IM INH is displayed. Press the ENTER key.
- To modify a parameter:
 1. Press the SET UP key: the message CONFIG will be displayed, then the message LE ANALOG.
 2. Press the < or > key to choice the sub-menus: ANALOG, ALARME, DIGITA, MESSAG, DIAG, DIVERS, IMPRIM, EVNMNT, CLVIER.
 3. Once chosen the sub-menu, for instance ANALOG, press the ENTER key. The message A1 CPTEUR will be displayed
 - The Δ and ∇ keys let change A1 for A2, A3, etc.
 - The < and > keys let change the sub-menus chosen parameters.
- Once chosen the parameter to modify, for instance A1 CPTEUR, press the ENTER key. The current value will flash.

- Press the Δ and ∇ keys to modify it.
- Press the ENTER key to confirm the modification or press the SET UP key to return to parameters selection menu.



Check list to using the furnace

1. If your need to programme the experience:



Press the MAINS ON switch. LOW WATER FLOW will turn on.

EUROTHERM 92 (OVER TEMP. ALARM):

The display is flashing:

1. Press the  key: the alarm unity will be displayed ($^{\circ}\text{C}$).
2. Press the  key: the message AL1 will be displayed.
3. Press the Δ or ∇ to display the alarm value (1050°C).
4. Kept depressed (a few seconds) the Δ and ∇ until the Clr message is shown.
5. Press the ∇ key: the alarm will be illuminated.
6. The temperature measured in the central part of the furnace will be displayed if over a period of 10 seconds no key is depressed.

To change the alarm value:

1. Kept depressed (a few seconds) the Δ or ∇ key until the Clr message is shown.
2. Press the  key: the alarm unit will be displayed.
3. Press the alarm unity ("secret key") to access to the protected list.
4. Press the  key (the parameters of the protected list are displayed) until the AL1 message appears.
5. Press the Δ or ∇ key to modify the alarm value.
6. The temperature measured in the central part of the furnace will be displayed if over a period of 10 seconds no key is depressed.

EUROTHERM (CENTRE ZONE CONTROLLER):

1. Kept depressed the number 6 key over a period of 5 or 10 seconds until the "OPER" message ("Operator Menu") is displayed.
2. Press the number 6 key: the "PrOG" message ("Programme Entry Menu") will be displayed.
3. Press the number 1 key: the Pr1 message ("Programme Ramp 1") will be displayed to set the ramp value.
4. Press the number 4 or number 5 key to set the ramp value (for instance, $5^{\circ}\text{C}/\text{min}$). Indication of access to alter values is given by a dot at the top of the lower display. This dot will flash if the value can be adjusted.
5. Press the number 6 key: the "PL1" message ("Programme Level 1") will be displayed to set the maximum temperature.
6. Press the number 4 or number 5 key to set the maximum temperature value (for instance, 750°C).
7. Press the number 6 key: the "Pd1" message ("Programme Dwell 1") will be displayed to set the period of time the maximum temperature will remain.
8. Press the number 4 or number 5 key to set "END" to maintain the maximum temperature until the end of the experience.
9. Press the number 6 key: the "PLc" message ("Programme Loop Count") will be displayed to set the number of times that the programme is repeated.
10. Press the number 4 or number 5 key to set the value of the parameter "PLc" (if is not desired to repeat a programme a value of "1" must be entered).
11. Wait 20-30 seconds to leave the programme mode and return to the normal mode.
12. Press the number 4 and number 5 keys together to reset the programme.
13. The message "MAIN" must not be displayed. If this message appears, press the number 3 key to return to the automatic mode.
14. The message "SP" ("Set Point") must be displayed. If this message does not appear, press the number 6 key until "SP" appears.
15. The lower half of the display will now read the room temperature in $^{\circ}\text{C}$ as 20. If this message does not appear, press the number 4 or 5 key to set it.
16. Press the "FURNACE ON/RESET" button. LOWER WATER FLOW will turn off.



17. Press the number 2 key to begin the ramp. Indication of the enabling of this feature is the illumination of the RAMP beacon in the lower right hand corner of the display. When the ramp reaches the local set point (the maximum temperature), the RAMP beacon will be replaced by an E, illuminated just to the right of the ramp.

2. If the programme is ready:

Press the MAINS ON switch. LOW WATER FLOW will turn on.

EUROTHERM 92 (OVER TEMP. ALARM):

The display is flashing:

1. Press the  key: the alarm unity will be displayed (°C).
2. Press the  key: the message AL1 will be displayed.
3. Press the Δ or ∇ key to display the alarm value (1050°C).
4. Kept depressed (a few seconds) the Δ or ∇ key until the Clr message is shown.
5. Press the ∇ key: the alarm will be illuminated.
6. The temperature measured in the central part of the furnace will be displayed if over a period of 10 seconds no key is depressed.

EUROTHERM (CENTRE ZONE CONTROLLER):

1. Press the number 4 and number 5 keys together to reset the programme.
2. Press the "FURNACE ON/RESET" button. LOWER WATER FLOW will turn off.
3. Press the number 2 key to begin the ramp. Indication of the enabling of this feature is the illumination of the RAMP beacon in the lower right hand corner of the display. When the ramp reaches the local set point (the maximum temperature), the RAMP beacon will be replaced by an E, illuminated just to the right of the ramp.

APPENDIX II

Assuming ideal gas behaviour:

$$pV = nRT \quad (1.1)$$

where $R = 6.236 \times 10^4 \text{ torr cm}^3 \text{ K}^{-1} \text{ mol}^{-1}$. As the hydrogen concentration is

$$C_H(\text{ppm}) = \frac{m_H(\mu\text{g})}{m_{Tl}(\text{g})} = \frac{m_H(\text{g}) \times 10^6}{m_{Tl}(\text{g})} \quad (1.2)$$

and the moles number is

$$n = \frac{m_H(\text{g})}{MW(\text{g})} = \frac{m_H(\text{g})}{2} \quad (1.3)$$

Combining equation (1.2) with equation (1.3)

$$n = \frac{C_H(\text{ppm})m_{Tl}(\text{g}) \times 10^{-6}}{2} \quad (1.4)$$

Then, substituting equation (1.4) into equation (1.1) we get

$$p(\text{torr}) = 0.03118 \frac{C_H(\text{ppm})m_{Tl}(\text{g})T(\text{K})}{V(\text{cm}^3)} \quad (1.5)$$

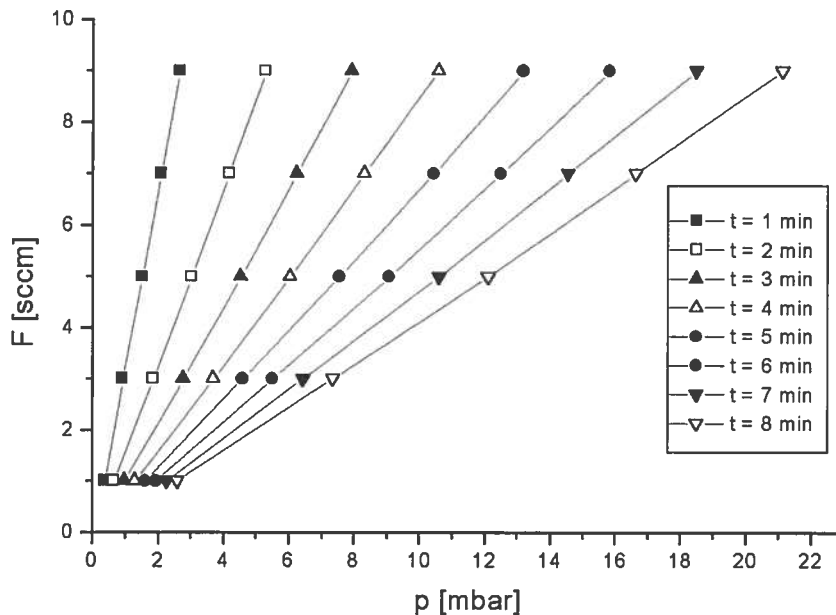
APPENDIX III:

It is possible to obtain the value of the equipment volume (V) from the equation

$$F(\text{sccm}) = \frac{V(l)\Delta p(\text{mbar})}{t(\text{s})} \quad (\text{II.1})$$

where F is the flow rate in sccm units (1 sccm = 1.69 x 10⁻² mbar l/s), t is the time we need to inject the hydrogen using the LAF 232 (switch to the "ON" position) and Δp is the difference between the pressure required to charge the samples (p) and the initial pressure (the pressure in the equipment before to open the mass-flow controller, negligible in front of p). Figure I.1 shows the value obtained for p for each for nine flow rates values, during a desired time.

Figure I.1: Mass-flow controller calibration. The pressure is measured by means the MKS Baratron 627A-12MDC.



The equations obtained are $F(p) = A + B p$, where "A" and "B" are:

T[min]	A	B
1	-0.25	3.5
2	-0.16	1.73
3	-0.17	1.15
4	-0.16	0.86
5	-0.17	0.69
6	-0.16	0.58
7	-0.16	0.49
8	-0.16	0.43

The average equipment volume (V) obtained from these equations is $V = 3.5\text{l}$. Substituting this value in the equation (2):

$$p(\text{torr}) = 0.03118 \frac{C_H(\text{ppm})m_T(\text{g})I(\text{K})}{3500} \quad (\text{II.2})$$

where T is the room temperature. Then, with the value of pressure obtained, we choose the $F(p)$ equation for the minimum loading time (t).

APPENDIX IV

Material prepared in Lausanne to irradiate in DR3 (RISØ):

Material	$T_{\text{irradiation}} = 50^{\circ}\text{C}$	$T_{\text{irradiation}} = 350^{\circ}\text{C}$
Ti-5Al-2.4Sn (annealed)	14C9	14C13
	14C10	14C14
	14C11	14C15
	14C12	14C16
Ti-5Al-2.4Sn ($C_{\text{H}} \approx 150$ ppm)	14C17	14C21
	14C18	14C22
	14C19	14C23
	14C20	14C24
Ti-5Al-2.4Sn ($C_{\text{H}} \approx 400$ ppm)	14C25	14C29
	14C26	14C30
	14C27	14C31
	14C28	14C32
Ti-6Al-4V (annealed)	25C1	25C5
	25C2	25C6
	25C3	25C7
	25C4	25C8
Ti-6Al-4V ($C_{\text{H}} \approx 150$ ppm)	25C9	25C13
	25C10	25C14
	25C11	25C15
	25C12	25C16
Ti-6Al-4V ($C_{\text{H}} \approx 400$ ppm)	25C17	25C21
	25C18	25C22
	25C19	25C23
	25C20	25C24

Material prepared in Lausanne to test without a preview irradiation:

Material	$T_{\text{test}} = 50^{\circ}\text{C}$	$T_{\text{test}} = 350^{\circ}\text{C}$
Ti-5Al-2.4Sn (annealed)	14C1	14C5
	14C2	14C6
	14C3	14C7
	14C4	14C8
Ti-5Al-2.4Sn ($C_{\text{H}} \approx 150$ ppm)	14C33	14C37
	14C34	14C38
	14C35	14C39
	14C36	14C40
Ti-5Al-2.4Sn ($C_{\text{H}} \approx 400$ ppm)	14C41	14C45
	14C42	14C46
	14C43	14C47
	14C44	14C48
Ti-6Al-4V (annealed)	25C25	25C29
	25C26	25C30
	25C27	25C31
	25C28	25C32
Ti-6Al-4V ($C_{\text{H}} \approx 150$ ppm)	25C33	25C37
	25C34	25C38
	25C35	25C39
	25C36	25C40
Ti-6Al-4V ($C_{\text{H}} \approx 400$ ppm)	25C41	25C45
	25C42	25C46
	25C43	25C47
	25C44	25C48

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