





Rapporto sul collaudo finale del forno ad alta temperatura

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Report RdS/PAR2013/218

RAPPORTO SUL COLLAUDO FINALE DEL FORNO AD ALTA TEMPERATURA

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Settembre 2014

Report Ricerca di Sistema Elettrico Accordo di Programma Ministero dello Sviluppo Economico – ENEA Piano Annuale di Realizzazione 2013 Area: Produzione di Energia Elettrica e Protezione dell'Ambiente Progetto: Attività di fisica della Fusione complementari a ITER Obiettivo: Progettazione e costruzione di una camera sperimentale per prove di erosione-corrosione ad alta temperatura (1000°C) di campioni di SiC/SiC in litio-piombo Responsabile del Progetto: Aldo Pizzuto, ENEA



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Summary

The construction of the experimental apparatus consisting of a high temperature oven has been completed. After its completion, the oven has been installed at ENEA Frascati labs where start-up and preliminary tests took place.

The acceptance test was carried out at Frascati labs in presence of F4E and JAEA delegates on January 2014. Then the apparatus has been packed in agreement with the air transport regulations and needs (wooden boxes, tiltwatch sensors, shockwatch sensors, etc.).

Other activities have concerned the preparation of the Installation and Operating Manuals and the visit of ENEA delegates in Rokkasho for supporting the JAEA colleagues during the first start-up operations of the oven.



1 Introduction

SiC fiber reinforced SiC matrix composites (SiCf/SiC) are candidate materials for applications in fusion power reactor as both functional and structural materials in the designs of DEMO blankets and of test blanket modules of ITER. Among the the key aspects to be studied for these applications, the study of chemical/physical interactions with the operating environments (LiPb) is of particular importance.

According to the Broader Approach (BA) program, in the framework of the DEMO R&D on SiC/SiC Composites for the International Fusion Energy Research Centre (IFERC) Project a study of the erosion-corrosion of SiC materials in Li-Pb has been planned through the construction of an experimental apparatus for performing high temperature tests of rotating SiC materials into the liquid metal. The experimental apparatus designed and built at ENEA Frascati laboratories has been shipped to Rokkasho IRFERC site for the testing.

This report describes the activities carried out from Oct. 2013 to the first semester 2014 in the framework of the second deliverable (Annual Report 2013) of the Procurement Arrangement IFERC-T1PA02-EU.ENEA.

2 Description of the activities

2.1 PRELIMINARY ACCEPTANCE OF THE EQUIPMENT AT ENEA

After its completion the experimental apparatus has been installed at ENEA Frascati labs for carrying out the start-up operations and the preliminary tests.



Fig. 1 - Delivery at ENEA Frascati of the oven.



Fig. 2 - Installation at ENEA Frascati labs of the oven.

Particularly, the data acquisition and control system based on a dedicated software developed by Labview has been realized.

The preliminary acceptance test was carried out at Frascati labs in presence of F4E and JAEA delegates on 21-22 January 2014. The minutes of this meeting are reported in Annex A. During this meeting the status of the apparatus and of its main components has been firstly verified. Then the testing of the apparatus has been carried out by following the procedure established in the previous meeting (thermal cycling, rotation of the specimen, control and data acquisition system, intervention of alarms, etc.). After test completion the integrity of the internal components of the oven has been verified, with no any component damage.

2.2 PACKING OF THE EXPERIMENTAL APPARATUS

As agreed in the PA, ENEA was in charge for the packing of the apparatus. The oven and its components have been packed in agreement with the air transport regulations (special wooden boxes) and other needs (tiltwatch sensors, shockwatch sensors, etc.). The description of the shipment consisting of 6 wooden boxes is reported in the ANNEX B.

2.3 MANUALS

ENEA provided the documentation related to the experimental apparatus and consisting of two documents: - Operational Manual: it describes in details the apparatus, its components, the software control system and the operating procedure for executing the erosion corrosion tests,

- Installation Manual: it provides information to assembly the apparatus and to verify its functioning. These manuals are reported in the Annex C.

2.4 START-UP OF THE OVEN IN ROKKASHO

During the visit of the ENEA delegates at Rokkasho on 9-11 June 2014, the status of the oven has been verified followed by the start-up of the apparatus. Particularly, a misalignment of the crucible vs. the SiC disk has been observed: after the removal of the insulation material inside the oven chamber, the heating



resistance module has been correctly located and then the insulating material has been inserted again into the oven. Also the rotation system exhibited a malfunctioning and has been repaired.

Then, the apparatus has been tested by checking the vacuum/purging system and the heating and rotation systems.

The minutes of the event are reported in the ANNEX D.

3 Conclusions

This report describes the completion of the oven for high temperature tests, the acceptance tests carried out at Frascati and the support to the installation at Rokkasho laboratories. With these actions all the activities of the Procurement Arrangement IFERC-T1PA02-EU.ENEA have been successfully completed.

ANNEX A IFERC-T1PA02-EU.ENEA/ T1-2-5 Minutes of the meeting at ENEA Frascati 21-22 January 2014

List of attendees: JAEA: Takashi Nozawa, ToshihikoYamanishi F4E: Marco Ferrari ENEA: Silvano Tosti, Andrea Moriani, Luigi Verdini

Test of the equipment in Frascati as part of "Preliminary Acceptance Tests"

21 and 22 January 2014

The status of the equipment acceptance tests as described in report IFERC-T1PA02-EU.ENEA/ T1-2-5/RP2 is resulted to be (in bold):

1) presence of LiPb in the crucible: it has been agreed between the Parties to use Pb instead of LiPb (conservative conditions). DONE AND ACCEPTED

2) carrying out of a thermal cycle consisting of heating from room temperature to 800 °C at a ramp rate of 10 °C/min, holding at 800 °C for 60 min, increasing temperature up to 1000 °C at a ramp rate of 10 °C/min, holding at 1000 °C for 10 min. **DONE (as documented in Figures 1, 2 and 3) AND ACCEPTED.** Figure 1 includes also evolution of current, as per below pit 3)

3) rotation of specimen in order to achieve at least a peripheral sample velocity of 0.5 m/s during the holding at temperature of 800 °C. **DONE (as documented in Figure 4) AND ACCEPTED,**

4) measurement and record of the current on the electrical heater during all the thermal cycle. **DONE (as documented in Table 1 and also shown in Figure 1) AND ACCEPTED,**

5) verification of the intervention of the interlock system in case of failure of gas flow, failure of watercooling flow and failure of the electrical resistance. **DONE AND ACCEPTED**

Moreover, the correct intervention of the "emergency stop", both manual and software, has been successfully verified. DONE AND ACCEPTED

After test completion the integrity of the internal components of the oven has been verified, with no any component damage, see Figures 4 and 5.

Delivery of draft documentation to JAEA as part of "Preliminary Acceptance Tests"

4 Following agreement between the parties a draft of installation and operational manuals have been delivered for information and comments to JAEA representatives.

ENEA commits to finalize this documentation tentatively within two weeks time, including any minor comments from JAEA.

In case JAEA will provide substantial major comments the parties will agree a new delivery date of the final version.



AOB

It has been further clarified that the change of ownership from ENEA to F4E can be signed only when list of parts is provided by ENEA and the final version of the above mentioned documentation (installation and operational manuals) will be provided by ENEA, uploaded in DMS and reviewed by F4E and approved by JAEA.

This is for F4E a formal condition for launching the contract for the shipment of the test equipment in Japan.



Thermal cycle of the acceptance tests - melting lead plus rotation - 21/01/2014

Figure 1 – Full thermal cycle



Figure 2 - Zoom of 800 C-deg, 1 hr crucible temperature



Thermal cycle of the acceptance tests - melting lead plus rotation - 21/01/2014

Figure 3 - Zoom of 1000 C-deg, 20 min crucible temperature



Time	Current [A]
[minute]	
85	35
106	25
114	20
126	15
157	32
185	14
200	16
254	18
271	17
325	15
362	15





Figure 4 – Screen snapshot showing rotation speed



Figure 5 – Rotating element



Figure 6 - Crucible



ANNEX B

DESCRIPTION OF THE SHIPMENT

 Oven chamber and support frame. weight 435 kg - size (L1, L2, h) =100 x 151 x 187 cm



 Electrical transformer TR1 (15 kW) weight 125 kg - size (L1, L2, h) = 50 x 57 x 72 cm



3. Electrical transformer TR2 (6 kW) weight 80 kg - size (L1, L2, h) = 40 x 50 x 72 cm



 Vacuum pump weight 32 kg - size (L1, L2, h) = 70 x 26 x 35 cm



5. Hoist

weight 20 kg - size (L1, L2, h) = 60 x 30 x 30 cm



6. Tools and other components Box of size 80x60x50 cm, with the following parts:



• 2 crucibles and one cup in SiC (reported in the scheme and in the picture). weight 3.5 kg



• 2 sample disks in SiC/SiC composite(reported in the scheme and in the picture). weight 0.1 kg – diameter 80 mm, thickness 3mm.



1 tube in SiC and 1 flat component in SiC/SiC composite (reported in the scheme and in the picture).
weight 0.3 kg.





- 1 laptop DELL LATITUDE E5530 (LCD monitor da 15.6 ").
- Other minor items and tools (about 2 kg).



ANNEX C

OPERATIONAL AND INSTALLATION MANUALS

ENEA Unità Tecnica Fusione Frascati Research Center Andrea Moriani, Luigi Verdini

Reference: Procurement Arrangement for the DEMO R&D on SiC/SiC Composites for the IFERC Project: erosion/corrosion of SiC and SiC/SiC in liquid metal - IFERC-T1PA02-EU.ENEA



February 2014, version v1

OPERATIONAL MANUAL

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A. INTRODUCTION

This manual provides the information to execute the erosion corrosion test. This manual in first describes all the systems that constitute the apparatus and then describes the software control system. After this introduction, the detailed description of the operating procedure for execute the erosion corrosion test will follow.

B. SYSTEMS OF THE EXPERIMENTAL APPARATUS

The furnace is composed of six main systems (figure 1):

- > Argon system.
- Cooling water system.
- ➢ Heating system.
- Sample motion system.
- Safety system.
- Control system.

SYSTEMS OF EXPERIMENTAL APPARATUS



Figure 1: Systems of the erosion/corrosion apparatus

In the following paragraph we describe the purpose and the components of each system.

> Argon system

The purpose of this system is to purge the furnace from the air by establishing a flow of argon. Making reference to the diagram in Figure 2 it is possible to identify the following components of the argon system:

- 1. F1: Ball gas flow meter.
- 2. V3: Valve.
- 3. M1: Pressure meter.
- 4. V1: Valve.

- 5. VF: Valve
- 6. FL3: Flow gauge.
- 7. VM2: Motorized valve.
- 8. P1: Vacuum pump.
- 9. PR: Pressure gauge.



Figure 2: Argon System

Cooling water system

The purpose of this system is to cool the exterior of the furnace through a controlled flow of water. Making reference to the diagram in Figure 3 it is possible to identify the following components of the cooling water system:

- 1. VM1: Motorized valve.
- 2. FL1: Flow gauge.
- 3. V4: Valve.
- 4. T3: Thermocouple for measuring the water inlet temperature.
- 5. FL2: Flow gauge.
- 6. T2: Thermocouple for measuring the water outlet temperature.



COOLING WATER SYSTEM



Figure 3: Cooling water System

NOTE: The cooling water system is divided into four sub-circuits. Two circuits cool the outer side wall of the furnace, one cools the argon gas output from the furnace and one cools the closure flange in the vicinity of the translation system. The balance of the four hydraulic circuits in terms of water flowrates and heating removal has already been executed. If there was the need for a further adjustment, the operator must operate on the four inlet valves and the four outlet valves shown in figure 3a, making sure to have a proper flow of water in each of the four water circuits.



Figure 3a: The two manifold for the inlet and outlet water

Heating system

The purpose of this system is to heat the furnace through specific thermal cycles imposed by the operator (i.e. to reach and maintain the operative temperature). Making reference to the diagram in Figure 4 it is possible to identify the following components of the heating system:

- 1. R: Electrical resistance for heating the oven.
- 2. TR2: Voltage transformer.
- 3. SSR: Solid state relay.
- 4. TC: Temperature Controller.
- 5. T1: Thermocouple of the heater.
- 6. T4: Thermocouple of the crucible.

HEATING SYSTEM





Sampled motion system

The purpose of this system is to handle the specimen. There is a translational system (moving up/down the sample) that is operated manually and a rotational system that is operated through an electrical motor. Making reference to the diagram in Figure 5 it is possible to identify the following components of the rotational motion system:

- 1. MO: Electrical motor.
- 2. EPOS2 70/10: Motor control board.



Figure 5: Motion System

> Emergency system

The purpose of this system is to put in safety condition the furnace in case of an emergency situation. Making reference to the diagram in Figure 6 it is possible to identify how the control system can take the action of disconnect the electrical power in presence of an emergency situation.

EMERGENCY SYSTEM



Figure 6: Emergency System

There may be conditions in which the operator could want to stop the test for an emergency situation.

The operator can stop suddenly the electrical power supply to all systems acting on the mushroom red switch on the control panel.

After is resolved the danger situation, to restored the normal operating condition, the mushroom red switch must be reset.

The same action can be achieve pushing the "EMERGENCY" button in the Graphical User Interface (GUI) of the software of control.

Control system

The purpose of this system is to monitor the behaviours of the furnace during the test and to be able to recognize abnormal operating situation and to be able to operate to restore the furnace in safety conditions. Making reference to the diagram in Figure 7 it is possible to identify the following components of the control system:

1. NI-USB 6009: Data acquisition board.

This specific electronic acquisition board has the following digital and analog input/output:

- Digital inputs [DI] :
 - (1) DI1 = Presence of electrical power supply.
 - Digital output [DO] :
 - (1) DO1 = Emergency stop of the electrical power supply.
 - (2) DO2 = Emergency stop of the electrical motor.
 - (3) DO3 = Emergency stop of the heating of the furnace.



- (4) DO4 = Actuation command of the motorized valve VM1.
- (5) DO5 = Actuation command of the motorized valve VM2.
- Analog inputs [AI] :
 - (1) Al1 = Thermocouple for the control of the furnace temperature (T1).
 - (2) AI2 = Thermocouple for the cooling water inlet temperature (T3).
 - (3) AI3 = Thermocouple for the cooling water outlet temperature (T2).
 - (4) AI4 = Pressure of the furnace (PR).
 - (5) AI5 = Thermocouple for the monitor of the furnace temperature (T4).
 - (6) AI6 = Flow gauge for the inlet cooling water (FL1).
 - (7) AI7 = Flow gauge for the outlet cooling water (FL2).
 - (8) AI8 = Flow gauge for the argon (FL3).
- Analog output [AO] :
 - (1) AO1 = Temperature set points for the temperature control board.



Figure 7: Control System

C. CONTROL SOFTWARE

This paragraph describe the graphical user interface (GUI) used to perform the erosion corrosion test.

To run the control software, just left-double-click on the icon located on your PC desktop.



The software main window will appear:



Fig. 8 – Software Main window



Preliminary overview

Look at the Data acquisition indicators on middle-top of the main window:

If you see this:

Data acquisition			
DAQ Device type Serial Number Status			
	0	Exit	

Control board not connected: Verify USB connection cable between PC and control board

If you see this:

Data acquisition			
DAQ Device type Serial Number Status			
USB-6009	117BBF8	Running	

Control board connected and properly working.

Look at the status indicators on the left-top of the main window



They all should be green, but in case they weren't....

MAIN PWR OFF	Verify electric connection. Turn on the Main Power switch
SERVICE OFF	Push the Service button (see note below)
DAQ error	Verify USB connection between PC and acquisition board.
MAIN PWR ON	Power supply is OK
SERVICE ON	Service are correctly supplied.
DAQ device OK	Control board connected and properly working

Note:

When the Service push button is pressed, the motor control board is powered up and the pop-up window below will appear on the PC screen:

Device Name	EPOS2	
Protocol Stack Name	MAXON SERIAL V2	-
Interface Name	USB	
Port Name	USB0	
Baudrate	1000000	•
Timeout	500 ms	

Just left-click on the "Open" button on the right.

Look at the "Motor Control" panel on the Main window:

If you see this:

Motor Control		
Target Velocity Actual velocity Actual current 0 rpm 0 rpm 0.00 A		
Error START STOP		

Check the USB cable connection between PC and Motor Control Board.

If you see this:

Motor Control			
Target VelocityActual velocityActual current0rpm0rpm0.00A			
Idle	START	STOP	

Motor is ok and ready to run.

Note: START button is enabled only if no alarm condition is present.



Rotation control

Motor Control			
Target VelocityActual velocityActual current0rpm0rpm0.00A			
Idle	START	STOP	

To start the rotation just left-click on the "START" button.

IF crucible temperature is below 400 °C (Pb-Li alloy may be not melted) this warning message will appear

ATTENTION! Crucible temperature is below 400 °C Are you sure you want to start motor?
YES NO, cancel

This allows you to test the motor.

Click on YES only if you are sure the sample is not into solid alloy!!

IF you pressed YES or IF T crucible is above 400 °C a new pop-up window will appear:

12	Motion parameters
	Target Velocity 🗧 120 rpm
	Acceleration 12 rpm/s
	Deceleration 12 rpm/s
	Set values and start running

This will allow you to change motion parameters. Click on the pushbutton and start motion

After that, the motor will start running

	Motor Control	
Target Velocity	Actual velocity 65 rpm	Actual current
Running	START	STOP

(the motor is accelerating...)



(the motor has reached the target velocity)

To stop the motor, simply press the STOP button.



Gas flow and pressure control

After you have completed the "purge" phase as described in an other section, you can operate in two different modes

1. Manual mode (P auto OFF)



In manual mode you can **set the desired value of argon flow**, simply writing it into the "Set sccm" control box. The gas flow controller will adjust the flow to the desired value.

2. Automatic mode (P auto ON)



In automatic mode you can **set the desired value of pressure** writing it into the "Set mbar" control box. The desired pressure is obtained by means of a PID software that calculates the appropriate gas flow.

Temperature control



To set the desired target value and ramp rate of temperature set point of the crucible, first press the button "pwr OFF/ON" than write the values in the boxes "Targhet SP" and "Rate".

The actual set point will increase (or decrease) toward the target value with the selected rate. You can change both values at any time.



Data recording

		Recording		
File name:	C:\Documents and	Settings		
Clock (s)	# of records		Rec OFF	View
🕻 6 sec	0	Start	Stop	Recordings

To start data recording press (left-click on) the "Start" button of this section.

You will be asked to choose a file name to record data in.

The data file is in .txt spreadsheet format , so you can open and edit it with any Excel-type software.

-			Recording		
	File name:	c:\Documents and s	ettings\Test01.tx	t	
	Clock (s)	# of records	Rec ON		View
+	6 sec	1251	Start	Stop	Recordings

The recording rate can be changed at any moment simply changing the value in the "Clock (s)" box.

Pb-LI 20140206 Temperat 325 ble Temperat 300 e Press 275 s Flew iscom r Flow IN (i) w OUT (lim 225 er IN (°C) 200 Water OUT (C) 175 SP Crucible (*C) 150 SP Heater (*Q) 125 :00 -50 75 -100-15 Jan 18: 15 Jan 18:10 15 Jan 10:07 15 Jan 10:00 15 Jan 10:09 15 Jan 10:11 15 Jan 18:12 14 44 < | **>>** 1 min /div. Auto scroll ON ► Back to panel

Pushing the "View Recordings" button you will see the graphs of the acquired data..

Push the "Back to panel" button to come back to the main window.

Alarms

Cooling Max. Temp	Min.Temp.	Crucible Heater	Gas flow
EMERGENCY	Mute alarm	Reset alarms	QUIT

(No alarm/failure)

Alarm					
Cooling	Max. Temp	Min.Temp.	Crucible	Heater	Gas flow
EMERGE	NCY	Mute alarm	Reset	alarms	QUIT

(Cooling alarm)

If an alarm condition (or more than one) occurs during the test run, warning and alarm boxes will light and a buzzer will sound.

Click on the "Mute alarm" button to stop the buzzer sound.

If the alarm condition disappears before the delay+alarm duration time expired, no action is taken and both buzzer and alarm lamp will turn off.

If the alarm condition persists, actions will be taken by the software (if no action is taken by the operator).

Delay (no action, no sound, no alarm light) and duration (buzzer and alarm light on), along with alarm levels, can be set for each alarm condition by right-clicking on the lower boxes of the corresponding alarm section and selecting "Settings".

A pop-up window like the one below will appear.





Examples of alarm behaviour:



No alarm state. (In this case the cooling flow is ok.)



Warning state: cooling flow below the minimum value. Delay.

No buzzer sound and no alarm lamp on yet. If the alarm condition disappear, the system come back to the "no alarm" state.



Alarm state: delay time expired, buzzer turns on, alarm lamp turns on. No action is taken until alarm duration is expired. If the alarm condition disappear, the system come back to the "no alarm" state.



Failure state: Alarm condition still on, alarm duration expired, actions are taken by the software (see next page).



Failure state: Alarm condition actually off , actions already taken by software.

Reset alarms

In the last condition you have to push on the "Reset alarms" button to reset the system and turn off the red box and the acoustic and light alarms.

Alarm sections

There are six alarm sections. They all works as described in the example above, but alarm conditions and actions taken by software are as described below:

Cooling:

Alarm condition: cooling flow below a certain value (default value is 3.2 l/s) Action in case of failure: heating system switched off, rotation stopped.

Max. Temp.:

Alarm condition: crucible temperature over 1100 °C or furnace temperature over 1450 °C Action in case of failure: heating system switched off, rotation stopped.

Min.Temp.:

Alarm condition: crucible temperature below 400 °C while motor is running Action in case of failure: motor is switched off.

Crucible:

Alarm condition: difference between set point and real temperature of crucible is more than 100 °C Action in case of failure: heating system switched off, rotation stopped.

Heater:

Alarm condition: difference between set point and real temperature of the heater is more than 50 °C Action in case of failure: heating system switched off, rotation stopped.

Gas flow:

Alarm condition: difference between set point and real argon flow is more than 30 sccm Action in case of failure: heating system switched off, rotation stopped.

EMERGENCY

<u>EMERGENCY</u> button: Push to turn off the electrical power to the erosion/corrosion apparatus. The behaviour is the same of pushing the EMERGENCY STOP button on the main control panel.

QUIT

QUIT button: This button closes the control software. This action requests a confirmation by the operator.


Mimic Argon subpart:



Argon section of the Graphical User Interface (GUI)

In this mimic the operator oversees the scheme of the argon system.

During the purge phase two horizontal slide bars under the valves V3 (filling) and V1 (pumping) give indications about the argon flow.

The operator should operate the valves in a way that the indicators stay near the right end of the horizontal slide bar.

The controlled VM2 valve can be operated from the operator taking into account that the control system automatically open the valve if pressure arise above 1090 mbar and close it when pressure decrease below 1040 mbar.

These two levels of pressure can be varied right-clicking on the valve push button and selecting "Settings". A pop-up window will appear allowing the operator to change settings.

The "Gas Flow and Pressure" part was illustrated in a previous Section.

Mimic Cooling subpart:



Mimic Cooling subpart of the Graphical User Interface (GUI)

In this mimic the operator oversees the scheme of the water cooling system.

It is possible to monitor the in/out flow rates of cooling water and also its in/out temperature.

The instantaneous heating power removed from the cooling water is shown in the "Cooling power" indicator (value theoretically assessed).



Mimic Heating subpart:



Mimic Heating subpart of the Graphical User Interface (GUI)

In this mimic the operator oversees the scheme of the Heating system.

During the heating of the erosion/corrosion apparatus, the operator reads the indication of the crucible temperature on the indicator "T crucible", the heater temperature set point and the real heater temperature on the indicator "Heater".

The "Temperature control" part was illustrated in a previous Section.

Appendix : Control software installation

A CD containing:

- a copy of the executable control sw (folder Pb-Li yyyymmdd EXE);
- a copy of Labview Run-Time Engine (folder LVRTE2009SP1);
- a copy of the currently used device drivers (folder NI-DAQmx 911)
- a folder containing the configuration files (folder Pb-Li)

is provided along with the experimental apparatus.

<u>The control software is already installed on the PC</u>, so you don't need to install anything, but in case you need to install the software for the first time on a different PC you should follow the steps below:

- 1. Install the Labview Run Time Engine LVRTE2009SP1;
- 2. Install the NI-DAQmx 911 drivers;
- 3. Copy the **Pb-Li yyyymmdd EXE** folder into the folder C:\Programs of your PC
- 4. Open the folder C:\Programs\ Pb-Li yyyymmdd EXE and create a shortcut of the executable file Pb-Li yyyymmdd.exe on the PC desktop
- 5. Copy the **Pb-Li** folder into the folder **C:** of your PC

Now you should see this icon on your PC desktop:



To run the control software, just left-double-click on the icon.



D. APPARATUS SPARE PARTS AND SPECIAL TOOLS

The experimental apparatus is accompanied by the following components:

• Two crucibles and one crucible cup.



Figure 9: Crucible and crucible cup

• Two special wrenches for assemblying the sample handler on the steel rotation bar.



Figure 10: Special wrench

• A special tool for the insertion of the crucible in the furnace.



Figure 11: Special tool for grabbing the crucible

E. OPERATING PROCEDURE

After the description of all systems of the experimental apparatus and the description of the Graphical User Interface (GUI), we are ready to describe and understand the operating procedure for performing the erosion corrosion test of a sample disk in the molten metal (lithium lead).

The first thing to do is to install the apparatus by following the "INSTALLATION MANUAL" where it is described how to do the electrical and piping connections. The "INSTALLATION MANUAL" provides also information to check the functionality of the experimental apparatus.

The experimental apparatus is composed of a supporting structure in which is placed the furnace. On the closure flange of the furnace are mounted the two systems of handling the sample.

The translation (up/down) system is manually operated through a special hand-wheel. The rotational movement is performed through an electrical motor. The electrical motor drives a steel rotating tube. On this steel tube is mounted the sample holder. On the sample holder is mounted the specimen (SiC composite disk).

Now we start the operating procedure:

1. Turn on the "Main Power" switch on the control panel (figure 12).



Figure 12: Control panel

2. Activate the control software (figure 8) with a double click on the icon of the PC desktop that is showed in figure 13.





Figure 13: icon of the control software

- 3. Turn on the "Service" button (figure 12).
- 4. Now we mount the sample on the sample holder. In figure 14 there is the explosion view of the sample holder with the sample: (from left to right) it consists of a tension rod, a SiC disk sample, a crucible cup, a composite SiC tube, a steel support tube, a steel spring and a long screw. The assembly procedure is:

5

Figure 14: explosion view of the sample holder

• Mount the tube in silicon carbide on the steel support tube through three specific screws (figure 15).

NOTE: cover the thread of the three screws with an appropriate lubricant (i.e. boron nitride spray is suggested).



Figure 15: silicon carbide tube joined to the steel rotating tube

• Insert the crucible cup in the silicon carbide tube and fix the specimen disk at its free end (figure 16).



Figure 16: Crucible cup and specimen on the silicon carbide tube



• Insert the spring inside the steel rotating tube (figure 17).

Figure 17: Spring inside the steel tube

• Insert the tension rod inside the silicon carbide tube (figure 18a). The steel terminal of the tension rod must enter in a specific slot of the steel support only in this way the tension rod can reach the slot in the silicon carbide tube (figure 18b).



Figure 18: (a) Placing of the tension rod inside the silicon carbide, (b) Placing of the end of the tension rod in the slot of the silicon carbide tube

- Insert the long screw inside the steel tube where there is the spring and tighten the screw for reaching a compression of the spring of 2-3 millimetres (figure 19). Because the elastic constant of the spring is 10 N/mm, in this way we achieve a preload of about 20-30 N.
- NOTE: cover the thread of the three screws with an appropriate lubricant (i.e. boron nitride spray is suggested).



Figure 19: Preload of the spring

- 5. Now we mount the sample holder on the rotating steel tube of the closure flange.
 - Turn on the "Hoist" switch on the control panel (figure 12).



Moving the manual translation system to 70 mm, this value is read on the metric scale (figure 20).



Figure 20: Manual translation system at 70 mm

- Unscrew the eight bolts that connect the closure flange to the furnace (figure 20).
- Lift the closure flange by using the electric hoist (figure 21).



Figure 21: Lifting of the closure flange

• Move the closure flange on the left side of the furnace using the specific handler bar (figure 22).



Figure 22: Positioning of the closure flange in the working area (on the left side of the furnace)

• Moving the manual translation system to 175 mm, this value is read on the metric scale (figure 23).



Figure 23: Manual translation system at 175 mm

- Mount the sample holder on the steel rotation tube. This operation must be carefully done by inserting the sample holder in the hole of the polycrystalline insulator and turning gently the sample holder in clockwise direction (figure 24) in order to screw the sample holder on the steel axis. With the special wrench (Part Number T002) it is possible tight gently for using the special tool you must moving the translation system to 165 mm so is possible to see the groove in the steel part of the sample holder (remember after the job to moving the manual translation system to 175 mm). Please, consider that the rotation direction of the steel tube is such that the sample holder cannot be unscrewed.
- NOTE: cover the thread of the three screws with an appropriate lubricant (i.e. boron nitride spray is suggested).





Figure 24: Mount the sample holder on the steel rotating tube

- 6. Now we mount the crucible in the furnace:
 - Before mounting the crucible inside the furnace, it must be filled with the lithium lead alloy. In (figure 25) the crucible filled with 950 cm³ of lithium lead alloy is shown. The free height between the surface of the lithium lead alloy and the upper part of the crucible is about 140 mm: . Melting of the LiPb alloy must be done in another oven. If you perform the melting in this oven, please consider that the sample holder has not to be installed and that during this first melting of the alloy a great release of gas occurs. Therefore, it could be necessary to operate with the VF valve switched on to avoid an abnormal increase of the pressure inside the furnace.
 - NOTE: The pressure inside the furnace is regulated by the control system. There is also installed a spring loaded safety valve (figure 26a) that opens at 1350 mbar and there is also a rupture disk that breaks at 1600 mbar (figure 26b).



Figure 25: crucible with lithium lead alloy



Figure 26a spring loaded safety valve



Figure 26b: rupture diagram

- The thermocouple that measures the crucible temperature is installed at the bottom of the heater (figure 27).
- Before inserting the crucible, verify the position of the thermocouple in order to ensure its contact with the crucible. If needed, lift up the thermocouple..





Figure 27: Lift up the crucible thermocouple

• Using the specific tool (Part Number T003) is now possible mount the crucible filled with the alloy inside the furnace (figure 28). There is a specific annular ring in polycrystalline alumina that helps to centre the crucible in the heater.



Figure 28: Insertion of the crucible in the furnace

- Be sure that the crucible is stable with a good planar position of the top part.
- Lift the closure flange, on the left side of the apparatus, using the electric hoist (figure 29).



Figure 29: Lifting of the closure flange

• Positione the closure flange on the right side of the furnace using the specific handler bar (figure 30).



Figure 30: Positioning of the closure flange on the furnace

• Lower the closure flange. There are two reference pins to help the operator during its moving down (figure 31).



Figure 31: Reference pins



• Make sure that, during the moving down of the closure flange, the cap of the crucible is correctly inserted on the crucible (figure 32). If necessary, lift up the closure flange and adjusts the position of the crucible.



Figure 32: well-positioning of the cap over the crucible

• Starting with the manual translation system to 175 mm, lower slowly the closure flange through the electrical hoist. Stop the lowering at 100 mm between the closure flange and the top of the furnace (figure 33).



Figure 33: lowering of the closure flange at 100 mm

• Moving the manual translation system to 140 mm, this value is read on the metric scale (figure 34).



Figure 34: Manual translation system at 140 mm

• Lower slowly the closure flange with the electrical hoist. Stop the lowering at the measured of 70 mm between the closure flange and the top of the furnace (figure 35).



Figure 35: lowering of the closure flange at 70 mm

• Moving the manual translation system to 110 mm, this value is read on the metric scale (figure 36).





Figure 36: Manual translation system at 110 mm

• Lower slowly the closure flange through the electrical hoist. Stop the lowering at the measured of 40 mm between the closure flange and the top of the furnace (figure 37).



Figure 37: lowering of the closure flange at 40 mm

• Moving the manual translation system to 80 mm, this value is read on the metric scale (figure 38).



Figure 38: Manual translation system at 80 mm

• Lower slowly the closure flange through the electrical hoist until to touch the top of the furnace (figure 39).



Figure 39: Closure flange on the top part of the furnace

Moving the manual translation system to 70 mm, this value is read on the metric scale (figure 40).



Figure 40: Manual translation system at 70 mm



• Fix the closure flange on the furnace by using the eight bolts (figure 41).



Figure 41: Fix the closure flange

- Turn off the "Hoist" switch on the control panel (figure 12)
- 7. Now we start to remove the air from the furnace and establish an argon atmosphere:
 - NOTE: Before starting to purge the furnace the operator must clean the vacuum pump filter. The following pictures (figure 42) show the location and how to remove the filter. The filter must be clean with air pressure flux.



Figure 42: Cleaning of the vacuum pump filter

• Close the valve V1 and V3 (figure 42a).



Figure 42a: Position of the argon system valves

• Open the bottle of argon and set up a pressure of 2 bars in the argon line (figure 43).



Figure 43: Establish a pressure in the argon line

- Turn on the "Vacuum Pump" switch of the control panel (figure 12).
- Open the V1 valve slowly. There is a horizontal indication bar below the V1 valve in the control software mimic (figure 44), this indicator can reach but must not overcame its right side end.





Figure 44: control software mimic of the argon system

- When the pressure inside the furnace goes below 3 mbar, wait for 5 minutes. Then, close the V1 valve and open slowly the V3 valve. There is a horizontal indication bar below the V3 valve in the control software mimic (figure 44), this indicator can reach but must not overcame its right side end.
- When the pressure inside the furnace rise up to about 1050 mbar the operator must close the V3 valve.
- Repeat the last three steps for two times. This means that we purge the gas in the furnace three times for ensuring an argon atmosphere inside the furnace.
- Now the operator has both V1 and V3 valve close. The operator has to push the "P auto OFF" to let to the control system to maintain in an automatic way a pressure of 1100 mbar inside the furnace (figure 44).
- NOTE: During the operation of the oven, the cleaning of the manual regulator valve VF could be necessary. It is suggested (i.e. it is good practice) to open completely and then close completely the VF valve, and then open the VF valve for 2 turns (figure 45).



Figure 45: position of the manual regulator valve VF

- Turn off the "Vacuum Pump" switch on the control panel (figure 12).
- 8. Now we start to establish a flow of water in the cooling system:

NOTE: The procedures until now followed have required a lot of time. In this time the flow of water was zero and the cooling alarm could be orange or red (In fact, the control system has a part devoted to the emergency management. This part is described in the "CONTROL SOFTWARE" paragraph). After the establishment of the water flow in the cooling system, this alarm could/should be reset using the specific button.

- Open the V4 valve to establish a water flow of 4.2 L/min.
- The operator must check the indicator of the in/out flow of water and the in/out temperature of the water for their correct functioning.

NOTE: The automatic valve VM1 of the cooling system is normally open. The operation of the valve could be done by the operator by clicking on the valve on the mimic. The valve is also operated by the control system during the emergency management.

- 9. Now we are ready to start the heating of the furnace.
 - Before starting the heating is suggested (i.e. it is good practice) to record the experimental data. How illustrated in the "CONTROL SOFTWARE" paragraph there is a specific part devoted to manage the recording of the experimental data.
 - NOTE: We describe how perform the thermal cycle used in the acceptance test. This cycle is composed of a first rump up to a crucible temperature of 450 °C with a rate of 10 °C/min. After holding 30 minutes at this temperature, we insert the sample specimen in the lithium lead alloy. After 10 minutes (needed in order to reach the thermal equilibrium of the specimen) we start the rotation through the electrical motor and then we raise the temperature of the crucible to 800 °C with a rate of increment of 10 °C/min. Once reached such a temperature, we wait for 60 minutes and then we stop the motor and remove the specimen from the lithium lead alloy. Then we raise the temperature of the crucible at 1000 °C with a rate of 10 °C/min. Once reached such a temperature, we wait for 10 minutes and then we stop the motor and then we switch off the heating (the furnace will cool for 24 hours).



- NOTE: The maximum temperature of the heater is imposed through the control system to 1400 °C. This limit ensures a good reliability of the heater element but it could require long time for reaching the temperature of 1000°C with a ramp rate of 10°C/min.
- Turn on the "Heating" switch of the control panel (figure 12).
- Write in the "Temperature Control" of the mimic in the Graphical User Interface (GUI) the target set point of the crucible temperature by writing 450 in the "Target SP(°C) box and imposing a rate of 10°C/min by writing 10 in the "Rate(°C/min) box (figure 46 and see also the "CONTROL SOFTWARE" paragraph).



Figure 46: Temperature Control part of the Graphical User Interface (GUI)

- The operator must push the "pwr OFF" button to start the heating of furnace (figure 46). NOTE: Now the control system starts to increase the temperature in the indicator "Actual SP(°C)" while the indicator "T crucible(°C)" show the real temperature of the crucible. The control of the crucible temperature is done by the control system through the power of the heater. In the mimic of the GUI there is a part devoted to indicate the status of the heater and the crucible.
- When the heating system reaches the imposed crucible temperature of 450 °C, wait for 30 minutes and then operate the manual translation system in order to insert the specimen in the lithium lead alloy (the insertion has to be done step by step in order to operate in quasi-equilibrium conditions). The actual position of the manual translation system must be 70 mm. Start to operate on the manual translation system by imposing increments of about 10 mm with a time interval between two increments of 1 min. Finally, reach the translation position of 175 mm (figure 47).



Figure 47: Manual translation system at 175 mm

 After 10 minutes (needed for reaching the thermal equilibrium) of the specimen the operator can start the rotation through the electrical motor. This operation has to be done in the "Motor Control" of the Graphical User Interface (GUI) (figure 48 and see also the "CONTROL SOFTWARE" paragraph).

	Motor Co	ontrol		_
Target Velocity	Actual ve	locity	Actual cu	rrent
0 rpm	0	rpm	0.37	A
Idle	STAF	स	STOP	,

Figure 48: Motor Control part of the Graphical User Interface (GUI)

The operator must push the "START" button. After the pressing of the button the control system shows the window of figure 49.

Target Velocity	:	120	rpm
Acceleration	:	12	rpm/s
Deceleration	:	12	rpm/s

Figure 49: Window to enter the motor parameters

In this window the operator can set the specimen target velocity in turns per minutes (rpm) and also the acceleration and the deceleration. After setting the motor parameters, the



operator must push the "Set values and start running" button and then the motor starts to operate.

- The operator can now set the temperature of the crucible of 800 °C with a ramp rate of 10°C/min. This operation must be done in the "Temperature Control" of the mimic in the Graphical User Interface (GUI) the target set point of the crucible temperature by writing 800 in the "Target SP(°C) box and setting a rate of increment of 10°C/min by writing 10 in the "Rate(°C/min) box (figure 56 and see also the "CONTROL SOFTWARE" paragraph).
- When the heating system reaches the imposed crucible temperature of 800 °C, wait for 60 minutes.
- Now the operator must remove the specimen from the lithium lead alloy. The actual position of the manual translation system is 175 mm. Start to operate on the translation system by imposing decrements of 10 mm with a time interval between two increments of 1 min. Finally, reach the position of the manual translation system of 70 mm (figure 60).



Figure 50: Manual translation system at 70 mm

- The operator can now set the crucible temperature of 1000 °C with a ramp rate of 10°C/min. This operation must be done in the "Temperature Control" of the mimic in the Graphical User Interface (GUI) the target set point of the crucible temperature by writing 1000 in the "Target SP(°C) box and setting a ramp rate of 10°C/min by writing 10 in the "Rate(°C/min) box (figure 46 and see also the "CONTROL SOFTWARE" paragraph).
- NOTE: The maximum temperature of the heater is imposed through the control system to 1400 °C. This limitation ensures a good reliability of the heater, but it could require long time for reaching the temperature of 1000 °C with a ramp rate of 10 °C/min..
- When the heating system reaches the crucible imposed temperature of 1000 °C ,wait for 10 minutes.
- Now we have completed the thermal cycle (it was that of the acceptance test).

10.We have terminated the thermal cycle and now we want to end the test.

- Turn off the "Heating" switch of the control panel (figure 12).
- Wait until the temperature of the crucible is below 100 °C.

NOTE: Starting with a temperature of the crucible of 1000 °C you have to wait almost 24 hours.

• The erosion-corrosion test is terminated and the operator can view the acquired data by using the button "View Recording" in the "Recording" part of the Graphical User Interface (GUI). In figure 60a is shown the graph of the entire acceptance test.



NOTE: Remember that this function can be used at any time of the testing phase.

Figure 50a: part of the GUI dedicated to the graphic representation of the experimental data acquired

- Stop the registration of the experimental data. The operator must push the "Stop" button in in the part "Recording" of the Graphical User Interface (GUI).
- Close the flux of water in the cooling system by closing the V4 valve.
- Close the flux of argon in the furnace by closing the bottle of argon (figure 43).
- Open the motorized valve VM2 by clicking on the icon in the Graphical User Interface (GUI) in order to establish the same pressure outside and inside the furnace.
- Unscrew the eight bolts that connected the closure flange to the furnace (figure 21).
- Turn on the "Hoist" switch of the control panel (figure 12).
- Lift the closure flange using the electric hoist (figure 21).
- Move the closure flange on the left side of the furnace by using the specific handler bar (figure 22).
- Move the translation system to 165 mm, this value is read on the metric scale.
- Remove the sample holder by using the two special wrenches (Part Number T001 and T002) how shown in figure 51.





Figure 51: The two special wrenches used to disassemble the sample holder from the steel axis.

• Remove the crucible from the furnace by using the special tool (Part Number T003) (figure 52).



Figure 52: Removal of the crucible

- Lift the closure flange, on the left side of the apparatus, using the electric hoist.
- Move the closure flange on the right side of the furnace using the specific handler bar.
- Lower the closure flange on the top part of the furnace. There are two reference pins to help the operator during the lowering.
- Fix the closure flange on the furnace by using the eight bolts (figure 51).
- Turn off the "Hoist" switch on the control panel (figure 12).
- Exit from the control software.
- Turn off the "Main Power" switch of the control panel (figure 12).

INSTALLATION MANUAL

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F. INTRODUCTION

This manual provides the information to assembly the apparatus as is arrived to the Japanese laboratory. We also provide the information to verify the functioning of the systems that constitute the experimental apparatus.

G. CONTENTS OF THE BOXES

The apparatus will be shipped in six boxes, with the following content:

- 1. Main apparatus disassembled in two principal parts.
- 2. Electrical transformer TR2 for the electrical supply of the heating system.
- 3. Electrical transformer TR1 for matching the electrical Japanese standard to the European standard.
- 4. Vacuum pump.
- 5. Hoist.
- 6. Computer dedicated to the control system plus spare parts for the apparatus and other special tools.

H. PROCEDURE FOR INSTALLING THE APPARATUS

The first box to be opened is the box containing the main apparatus. The apparatus is divided in two parts. You have to install on the lower part (figure 1) the upper part (figure 2) that supports the hoist. The hoist is mounted on the handler bar trough a hook (figure 2) after the mechanical installation of the hoist connect it to the power electrical supply and the control pad through the two specific cables.



Figure 1: Lower part of the main apparatus



Figure 2: Upper part of the main apparatus

The two parts must be assembly using the fixing plate showed in figure 3.



Figure 3: Plates joining the lower part and the upper part of the main apparatus

You can now mount the vacuum pump on the side of the furnace how shown in figure 4 where it is possible to see how the specific tube is installed between the vacuum pump and the v1 valve. Also, you have to install the outlet of the vacuum pump (the yellow corrugated tube). Figure 5 shows the electrical connection of the vacuum pump.



Figure 4: Vacuum pump pipe connections



Figure 5: Vacuum pump electrical connections

You can complete the connection of the sweep gas system with the inlet and the outlet argon tube and the inlet of the air pressure on the rear part of the furnace how shown in figure 6.

Figure 6: Inlet/outlet argon and inlet air pressure

Then, you can connect the water inlet and the outlet of the cooling system situated on the back of the main structure of the furnace how shown in figure 7.

Figure 7: Inlet/outlet water

Now it is possible to install the electrical transformer TR2 for the electrical power of the heating system. How it is possible to see in figure 8, there are two electrical plugs to be fixed to the TR2 panel and two terminals (cables) to be fixed on the electrical terminal block for the electrical power of the heater.

Figure 8: Electrical connections on the transformer TR2

Now you can install the electrical transformer TR1 for the adaptation of the European standard with the Japanese standard. In figure 9 it is shown the transformer TR1 with the red electrical outlet fixed and on its left there is the electrical cable where the electrical plug compatible with the Japanese standard must be installed.

Figure 9: Electrical connections on the transformer TR1

You can terminate the installation of the furnace by making the connection on the personal computer of the two USB cables. One related to the electrical motor and the other one related to the control system board (figure 10).

Figure 10: Electrical connection of the two USB cables

I. TO VERIFY THE FUNCTIONING OF THE SYSTEMS

NOTE: Before going on, please read the paragraph "CONTROL SOFTWARE" of the "OPERATING MANUAL".

Now that you have mounted the experimental apparatus it is possible to make same functional verifications. Connect the electrical transformer TR1 at the electrical power.

Turn on the "Main Power" switch on the main panel (figure 11).

Turn on the computer and run the control software with a double click on the icon of the desktop shown in figure 12.

The "SERVICE ON" button will be red (figure 13): you have to turn on the "Service" switch on the electrical main panel (figure 11) and the "SERVICE ON" button will become green.

Figure 11: Electrical main panel

Figure 12: icon of the control software

Figure 13: Graphical user interface (GUI)

Please, check the following:

- Verify the functioning of the hoist turning on the switch "Hoist" on the electrical main panel (figure 11). Then, by using the control pad of the hoist it is possible verifying its functioning.
- Unscrew the eight bolts that connect the closure flange to the furnace (figure 14). Lift the closure flange by using the electrical hoist. Soft material could be installed for the transport between the electrical resistance and the closure flange: please, remove it, if any. Move down the closure flange through the hoist and then tight the eight bolts. Turn off the switch "Hoist" on the electrical main panel.

Figure 14: Closure flange

Verify the functioning of the electrical motor by pushing the "START" button on the Motor Control part of the Graphical user interface (GUI) (figure 15). If the temperature of the crucible is below the melting point of the lithium lead alloy (400 °C), the software will ask you if you are sure of activating the motor (figure 16). The control system shows the window in figure 17. In this window the operator can impose the target velocity of the specimen in turns per minute (rpm), the acceleration to reach this velocity set point and the deceleration to come back to zero velocity. After imposing the motor parameters, the operator must push the "Set values and start running" button and the motor starts to operate. After reaching the target velocity, you can push the "STOP" button on the Motor Control part of the Graphical User Interface (GUI) of fig. 15.

Figure 15: Motor Control part of the Graphical User Interface (GUI)

LV	23
ATTENTION!	
Are you sure you	want to start motor?
YES	NO, cancel
YES	NO, cancel

Figure 16: Warning window appearing if the electrical motor is actuated when the temperature of the crucible is below 400°C

Target Velocity	;	120	rpm
Acceleration	:	12	rpm/s
Deceleration	:	12	rpm/s

Figure 17: Window to enter the motor parameters

Open the valve (if any) of the cooling water circuit upstream the inlet of the cooling water (figure 7). Verify the indication of the IN/OUT flow and the IN/OUT temperature of the water on the Graphical user interface (GUI) (figure 18): close and open the motorized control valve VM1 by pushing the valve on the GUI (green button=valve open, orange button=valve closed).




Figure 18: Mimic Cooling subpart of the Graphical User Interface (GUI)

• Verify the indication of the crucible temperature and the heater temperature on the Graphical user interface (GUI) (figure 19). Verify the temperature on the "Heater" on the electrical main panel (figure 11); it is indicating the heater temperature.



Figure 19: Mimic Heating subpart of the Graphical User Interface (GUI)

• Verify the vacuum pump. Close the V3 valve and the V1 valve (figure 20). Turn on the switch "Vacuum Pump" on the electrical main panel (figure 11) and verify that the pump works regularly. Turn off the switch "Vacuum Pump" on the electrical panel.



Figure 20: Position of the argon system valve

Close the V3 valve and the V1 valve (figure 20). Open the valve (if any) of argon gas upstream the argon gas inlet (figure 6). Impose on the "Gas Flow and Pressure" part of the Graphical user interface (GUI) shown in figure 21a value of argon flow on the "Set sccm" box (for example 50 sccm). Verify that the indication on the "sccm" indicator is the same. Verify also the lecture of the pressure inside the furnace on the "mbar" indicator (figure 21).



Figue 21: Mimic Argon subpart of the Graphical User Interface (GUI)

• Verify the electrical continuity of the electrical resistance for the heating. Turn off the switch "Main Power" then open the door of the electrical main panel and turn off the switch "6KM02". Check the



electrical continuity of the heating resistance using an electrical tester how shown in figure 22. Turn on the switch "6KM02".



Figure 22: Check for the electrical continuity of the heating resistance

If you have completed with success the previous verifications, you can start the operating procedure to run the erosion corrosion test. To this purpose, you have to read the "OPERATING MANUAL".

ANNEX D IFERC-T1PA02-EU.ENEA/ T1-2-5 Minutes of the visit of ENEA delegates at Rokkasho – 9-11 June 2014

List of participants: JAEA: Takashi Nozawa ENEA: Silvano Tosti, Luigi Verdini

9 June

S. Tosti supplies to *T.* Nozawa the SiC composite disk missing in the shipment of the apparatus. In order to start preliminary testing, the following operations are performed:

- vacuum pumping and sweeping with Ar the oven chamber,

heating is switched off and the chamber remains under Ar atmosphere.

- insertion of the SiC crucible and assembling of the sample holder with the SiC disk.

After assembling of the sample holder, it has been verified a misalignment of the crucible vs. the SiC disk. It is supposed that during transportation the heating resistance module moved from its correct position. In fact, the crucible is hosted in a cylindrical cavity in the mid of the heating resistance. So, the insulation material inside the oven chamber is removed, the heating resistance module is correctly located and then the insulating material is inserted again into the oven.

After the insertion of the crucible, a new procedure of vacuum pumping and sweeping with Ar the oven chamber is carried out. During the night the oven remains under vacuum.

10 June

After venting with Ar, the oven is opened in order to check the correct position of the thermocouple measuring the temperature inside the heating resistance. After this positive verification, the oven chamber is vacuum pumped and swept with Ar.

During the testing of the specimen rotation a failure of the rotation system is observed. Then the coupling of the rotation motor with the rotating bar holding the specimen is disassembled by verifying that two screws are not in position. After repair of the rotation system, the chamber is vacuum pumped and swept with Ar. A heating tests is carried out by reaching the crucible temperature of about 200 °C. During the night the

11 June

The heating of oven is started by setting up the temperature of 450 °C. After about 1 h of holding the oven at that temperature, the temperature is increased at 700 °C. After verifying the stability of the temperature in this conditions, the heating resistance is switched off then starting the cooling of the oven.