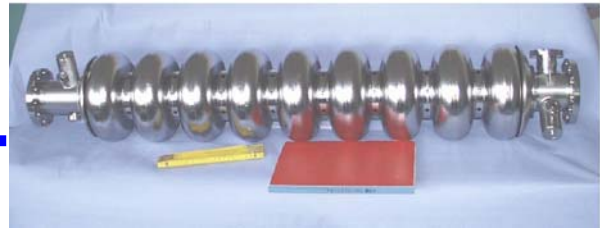**Deliverable 5.3.5.5: "Automated EP: Conclude on best electrolyte"**

Vanessa Rampazzo, G. Mondin and M. Ceccato
Superconductivity Lab of National Lab of Legnaro
National Institute of Nuclear Physics

Abstract

Electropolishing (EP) is a standard surface cleaning and processing method for the fabrication of superconducting Niobium cavity. This technology is well known since many years. In practice, however, it is not easy to establish the optimal parameters for the shape of accelerating resonators. In this work a computerized control system has been developed. It determines the plateau of the so called polarization curve and varies the voltage / current parameters in a way that the electropolishing parameters stay at the optimum value. This paper describes the hard- and software requirements as well as presents a simple handbook for the operation of the automated EP. The source code is available by the authors.
In a late stage of finalizing the automated EP with optimized standard chemistry an orthogonal electropolishing "chemistry" was detected: a special kind of ionic liquid which does not use any hazard acid like HF. First tests with samples were so promising that detailed R&D effort was carried out by two thesis investigations. Both papers are presented as appendices to this report. This complete new electropolishing liquid has the potential of totally reducing the hazard of acid thus considerably reducing risk and cost in industrial application.

Acknowledgements

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INSTRUCTION MANUAL FOR AUTOMATED ELECTROPOLISHING

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Instruction Manual for Electropolishing Automatic Program

SYSTEM REQUIREMENTS

In order to use the Automatic Program there are some hardware and software prerequisites:

Hardware:

1. Personal computer with processor Intel ® Pentium 4 or AMD ® Athlon 64 with minumum speed 3.0 Ghz, 2 GHz RAM, hard disk 80 GB, Ethernet interface 10/100 Mbps, and standard accessories.

2. Power Supply for Electropolishing process: this power supply must be controlled by a external PLC. The generator base of Field Point must be connected to the analog control interface of power supply.

3. National Instruments® Field Point® Ethernet controller module

4. National Instruments® voltage or current generator base.

5. National Instruments ® digital input/output

Software:

1. Microsoft® Windows® XP

2. National Instruments® Labview® 7.1

3. Real Time® module

See the requirement for the National Instruments® component in data sheet.

Example of configuration:

Power Supply with analog control inteface

NI FP-2010

Dual-Channel Terminal Base FP-TB-10

Dual channel Analog Output Voltage 0-10V FP-AO-V10

Dual Channel Analog Input Voltage 0-10 V FP-AI-V10

Dual Channel Digital Output DC60 3-60 Voltage DC FP-DO-DC60

Dual Channel Digital Input FP-DI-DC

The NI Field Point and the other instruments must be configured using the NI MAX® Automation utility.

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DESCRIPTION OF COMMAND

The Automatized Program for Electropolishing is written in Labview ® 7.1 RT and can work in connection with the PC or stand alone into the physical memory of NI Field Point®. When the program works in connection with the computer, all the parameters can be changed during the process and the display refreshes the polarization curve.
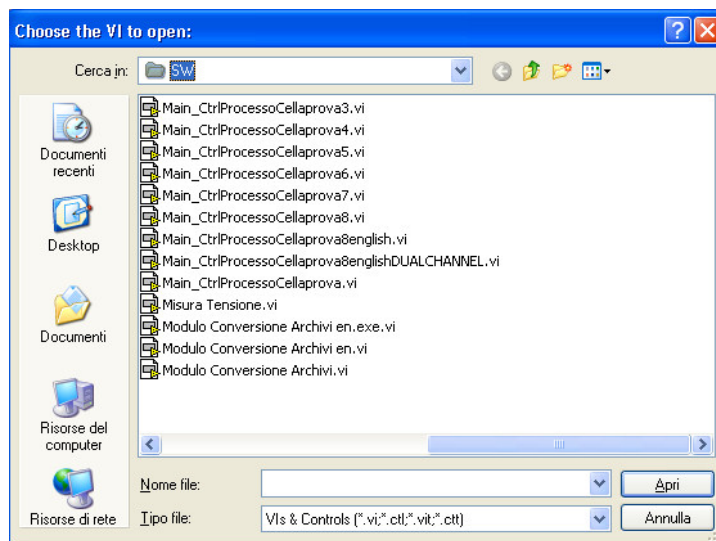
SETTING OF FIELD POINT® CHANNELS

Open the Labview® RT program and select the Field Point IP address.

Figure 1: Initial windows of Labview RT

Open the automation program.

Figure 2: Program menu

After the program opens, the panel of program appears on the screen. The panel is composed of two parts: the display of polarisation curves and the two windows of controls, that can be selected by a left click of mouse on the top.
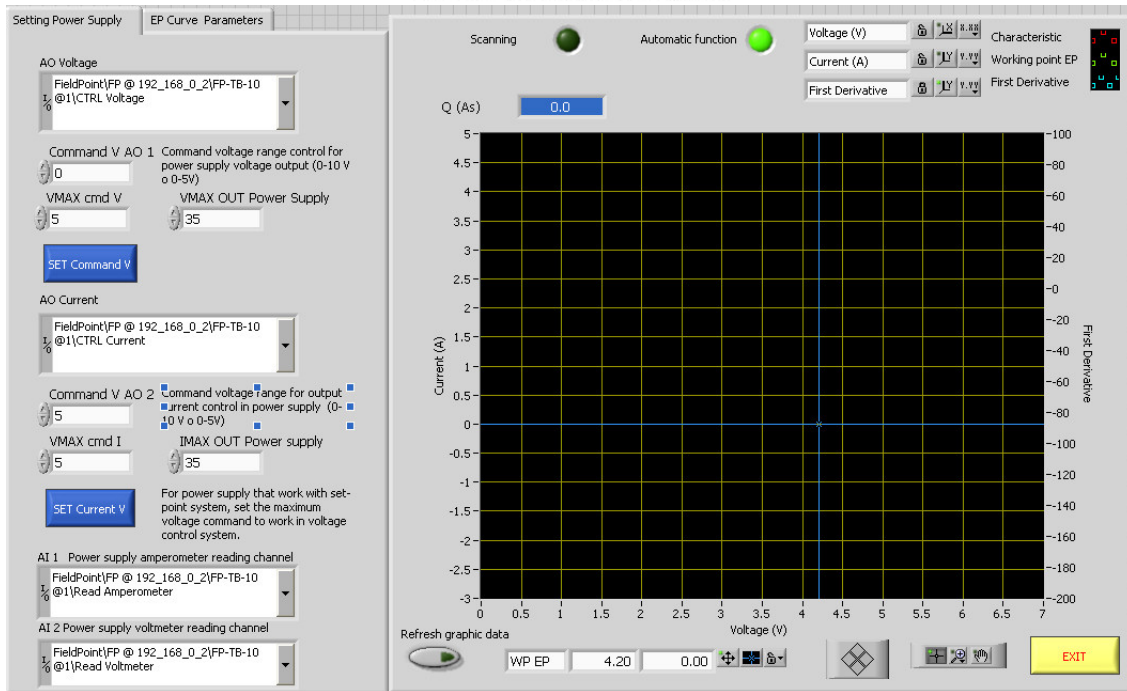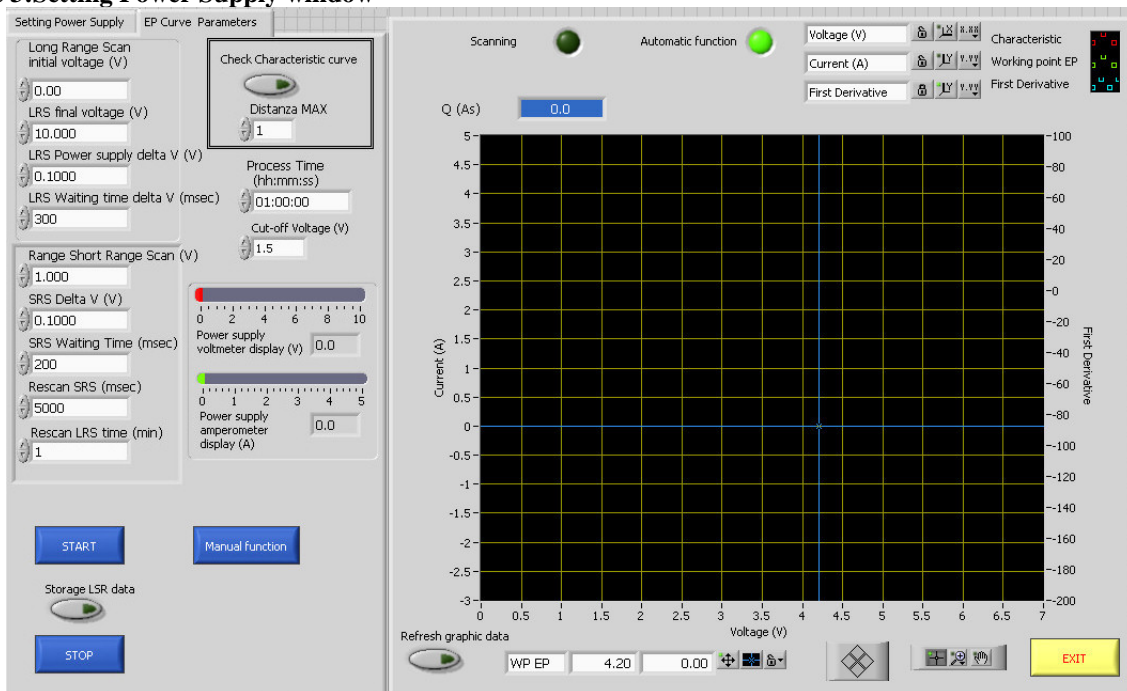
Figure 3:Setting Power Supply window

Figure 4: Setting EP Parameters

The output channels control the voltage and the current, provided by the power supply. In "Setting Parameters" there is all the information on the power supply used for electropolishing. Most of commercial power supplies work on the principle of "Set-point". If the voltage value is lower than

the current, the power supply works on voltage control and changes the voltage. On the contrary, if the voltage value goes over the voltage value, the supply changes the current.

The electropolishing program works controlling the voltage . To ensure to work in voltage control, there are two set points of voltage and current: the "Command V AO1" for voltage and the "Command V AO2" for current: put the 0 value on voltage control and the maximum value (10 or 5) on current control.

VOLTAGE CONTROL

1. Set the Field Point channel of voltage control
2. Set the set-point value for voltage on of power supply (value 0)
3. Set the maximum value of voltage control range (10V or 5V)
4. Set the maximum output voltage (V) of power supply
5. Press "Set Command V"

CURRENT CONTROL

1. Set the Field Point channel of voltage control
2. Set the set-point value for voltage on of power supply (value 10V or 5V)
3. Set the maximum value of voltage control range (10V or 5V)
4. Set the maximum output current (A) of power supply
5. Press "Set Command I"

READING VOLTMETER

1. Browse the input channel of Field Point for reading the power supply voltmeter

READING AMPEROMETER

1. Browse the input channel of Field Point for reading the power supply amperometer

Figure 5: Description of Setting Parameter window

After this preliminary setting, it's possible to set the values as a default. On Labview command,

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select "Operate", then "Make Current Value Default".

Save the program.

Now the saved program must be downloaded into the memory of Field Point®. Press the white arrow below the command "File", and the download starts.

The next section deals with the Electropolishing Parameters.

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SETTING OF ELECTROPOLISHING PARAMETER
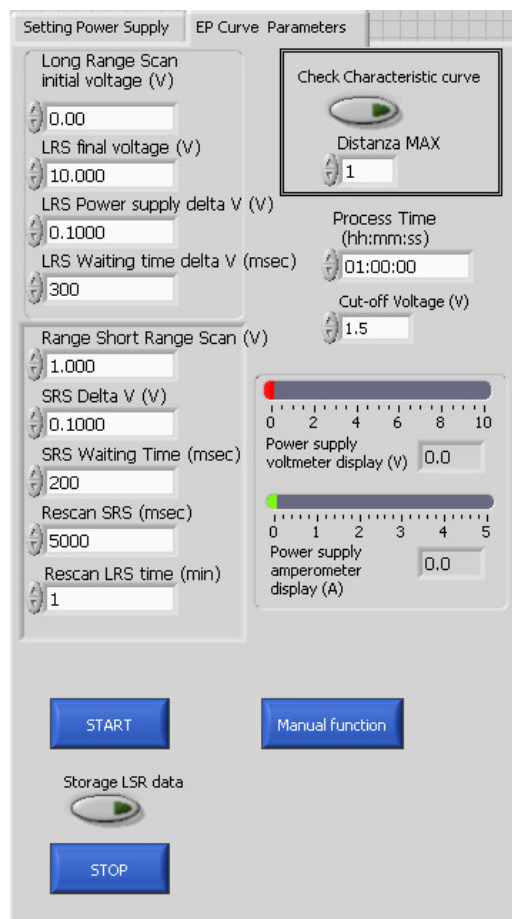
Figura 6: EP Curve Parameter menu

The EP Curve Parameters is divided in sections:

- LONG RANGE SCAN
- SHORT RANGE SCAN
- Check characteristic curve
- Instruments display
- Automatic-Manual Function

LONG RANGE SCAN (LRS) section

The first scan of program displays the characteristic curve in order to find the electropolishing voltage. The program needs the initial voltage and the final voltage of scan.

The distance and time between the points of curve can be varied using the "Power supply delta V" and the "Waiting Time delta V" fields.

![INFN logo]
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The program starts calculating the curve between initial and final voltage.

To keep the data refreshed on the display, ensure that the button "Refresh graphics data" below the display is on (green led).

After the scan, the system calculates the first derivative and selects the voltage corresponding to the derivative minumum value. Now, the program uses the data in SHORT RANGE SCAN to follow the voltage point found.

SHORT RANGE SCAN (SRS) section

After the Long Range Scan, the program starts to follow the minimum seeking around the point found in the LRS.

The "Range" value is divided in two: substracting and adding the half value to the EP voltage, the program defines the initial and final values of the short range scan. After the SRS Waiting Time, the system starts to scan.

Attention! We recommend not to exceed this value because the program doesn't refresh the new data set in the field until the SRS Waiting Time is past. However, the program accepts every time value. We recommend value not over 60000 millisecond.

After the "Rescan LRS Time" the program repeats the LSR.

CHECK CHARACTERISTIC CURVE
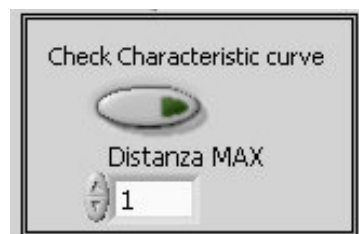
Figura 7: Check Characteristic

In particular cases, the characteristic curve changes after a very short time, so the first LRS doesn't find a good EP point. To avoid this problem, the program can repeat the LRS two times. If the absolute difference between point to point is lower than a defined value in "Maximun Distance", the program accepts the characteristic. If the difference is greater, the system repeats the characteristic until the distance condition is retained.

INSTRUMENTS DISPLAY

The power supply amperometer and voltmeter are duplicated on digital menu. Left clicking on the number bar can change the scale of instrument.
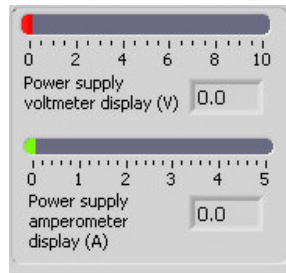
Figura 8: Amperometer and Voltmetere

PROCESS TIME AND MAX CHARGE

The program can be stopped by two events: the time and the charge.

The maximum duration of EP process can be set in PROCESS TIME field and the maximum charge in SET Q MAX.

The program measures the total charge of process and the time past, and stop when one of this two condition are reached.

Attention!: If the sistem must work only controlled by time or by charge, then set a value two or three times greater on charge or time.

CUT-OFF VOLTAGE

The Cut Off Voltage set an inferior limit to the routine of finding EP voltage, in order to ensure that the program doesn't find a false minimum due to flat current near 0 voltage, calculated by the numerical derivative dV/dI, or in special cases, as a characteristic curve made by great oscillation. It's possible to use this value to artificially limit the range of finding minumum. This value can be changed dinamically during the EP process in order to adapt this value to the various EP solution and materials.

We recommend to set this value not below 1 Volt.
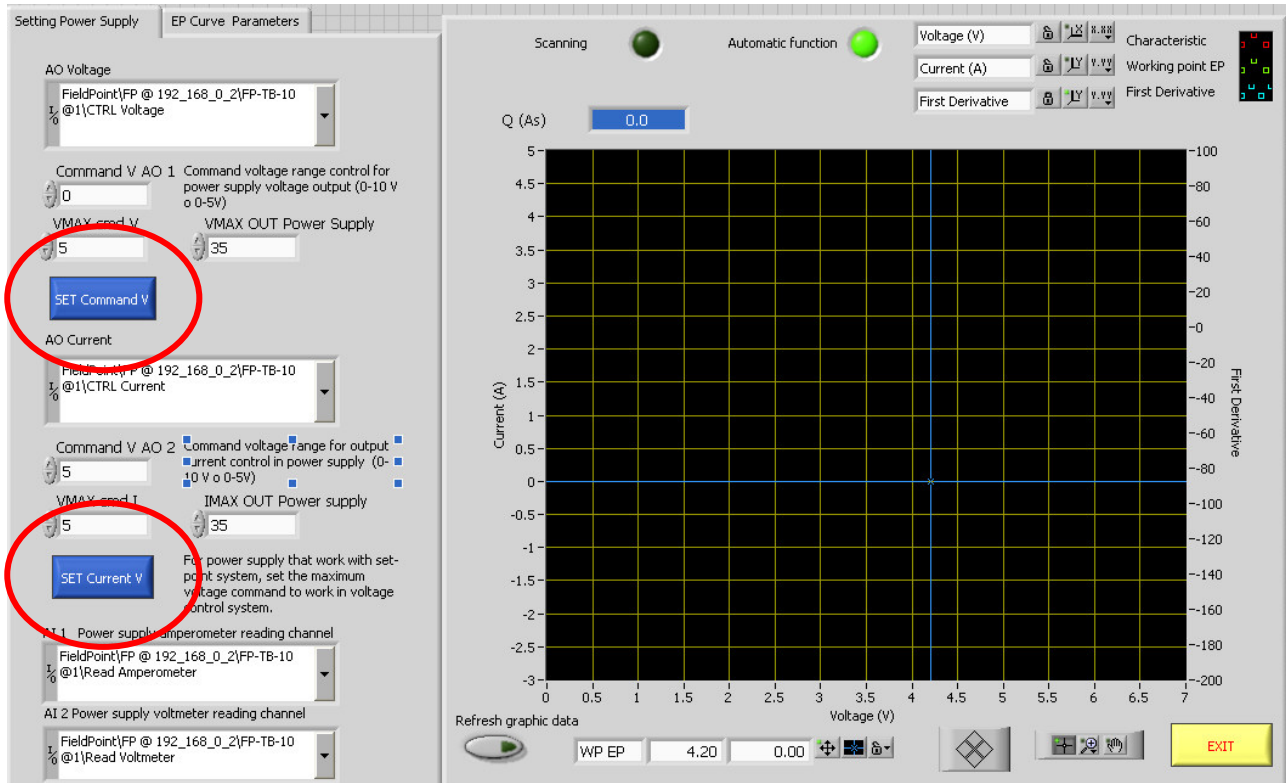
AUTOMATIC - MANUAL FUNCTION

The program can be forced to work on a different point from the voltage found in the automatic scan. Pressing the "Manual" button the program switches on the semi-automatic mode, *after the Rescan Time set in SRS*. Now it's possible to choose graphically the new EP point and confirm

Attention! When use this function we recommend to set a low time value in SRS Rescan Time, not over 5000 millisecond, to ensure that the program refreshes continuosly the new data set.

FIRST SCAN

1) Ensure that the channel of Field point in Setting Power Supply is active. Press the button "SET Voltage V" and "SET Current V"

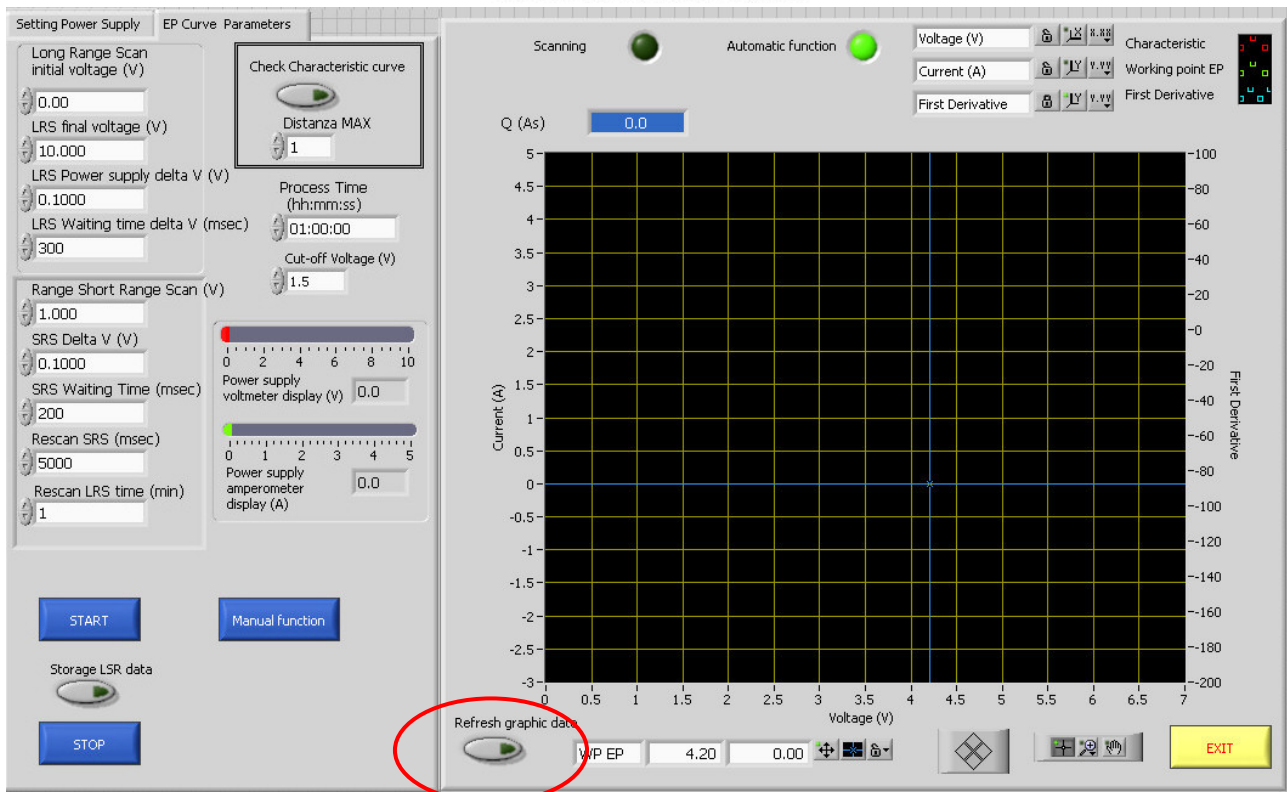2) Change the window by clicking on the "EP Curve Parameters". Set all the parameters required .

3) To refresh the graphic data on display, click on "Refresh graphic data".

4) To store the Long Range Scan data , click on "Storage LRS Data"

5) To set the duration of EP, fill the "Process Time" field. This value can be changed during the EP process.

6) The "Cut-Off Voltage set a voltage valute that limit the range of finding EP voltage. This value can be changed dinamically during the EP process.

After the preliminary operation, click on "START" button .

Attention: In the DESY version of this program, the button START and STOP are disabled. See the section DESY MODIFIES.
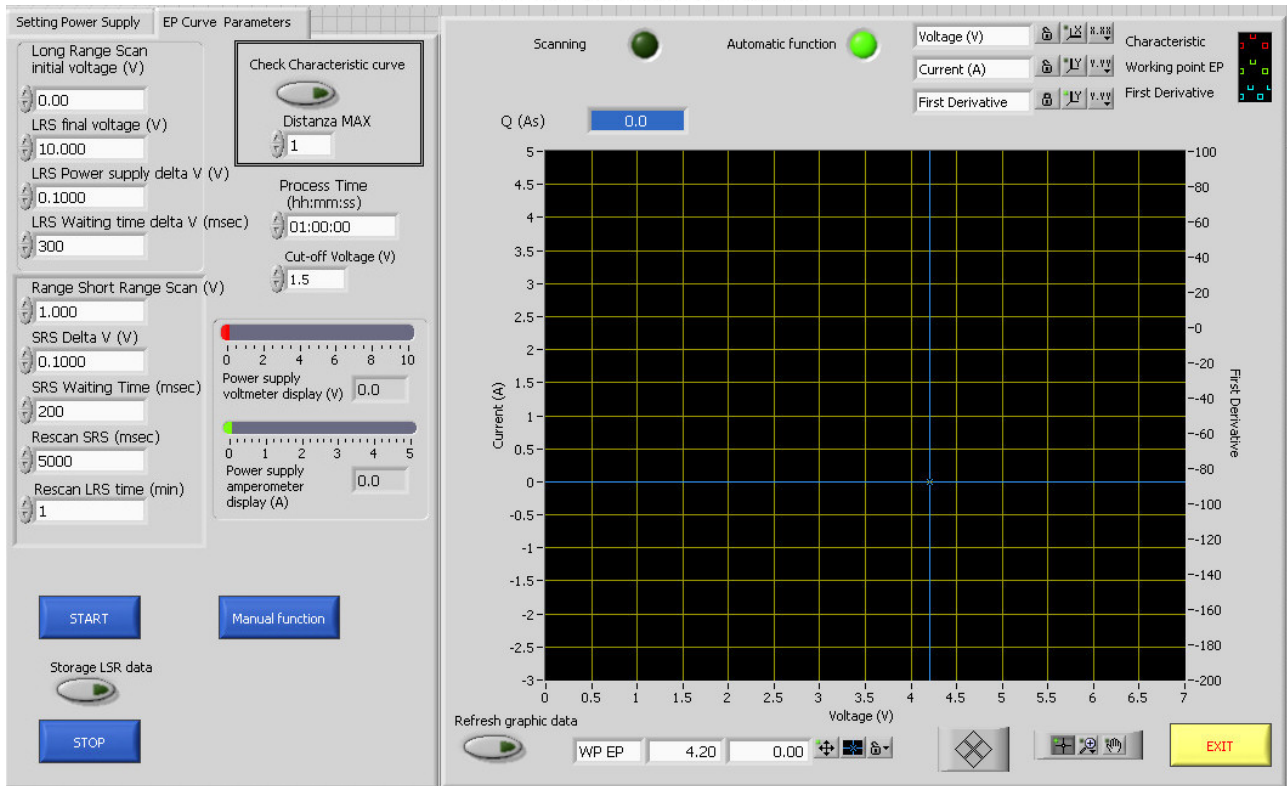
AUTOMATIC FUNCTION

The program starts to scan the range inside the LRS. After the scan, the program calculates the numerical derivative dI/dV, and finds the minumum value. The system sets the new EP value. After the SRS Waiting Time, the program re-scans the curve around the EP value found and recalculates the minimum of derivative.

After the Rescan LRS Time, the program rescans the curve using the LSR values.

All the parameters in LRS scan and SRS scan boxcan be changed during the process, but the program refreshes the set data after the SRS Waiting Time or LRS Waiting Time.

When the countdown of Process Time arrived to 0, the system stops the program and sends a digital input.

MANUAL FUNCTION

If the user wants to change the EP voltage, the automatic finding of EP voltage can be excluded by clicking on "Manual Function" button.

Attention! When using this function, we recommend to set a low time value in SRS Rescan Time, not over 5000 millisecond, to ensure that the program keeps refreshing the new data set.

The layout changes to reveal two new buttons. The voltage can be changed grafically by shifting the line on display .When the desired voltage is found, set the new volage value by click on "Confirm Voltage".

The program starts to follow the EP voltage using the data set in SRS field, but it doesn't rescan all the curves by LRS data.

To return to automatic function, click on "Automatic Function". The program retains the EP value set by the user, but now the repetition of LRS is enabled.

STORAGE LRS DATA

The polarization curve can be stored into the physical memory of NI Field Point by clicking on button (green led is on when the system save the polarisation curve). The data must be downloaded from the memory using a FTP program.

After the download, the curve are saved with a "log" extension. To display the curves and convert into a tabulation column text, use the utility "ModuleConversionArchives.exe". *See the Display and Convertion files" section.*

STOP

When the button "STOP" is pressed, the system interrupts all the processes and resets the time. To exit off the Labview® application, press the button "EXIT".

The program decrease the voltage slowy after the stop, to ensure that the power supply doesn't suffer of Eddy currents that can breack the internal circuits.

DESY MODIFIES

The buttons START and STOP are disabled. The system must receive a costant signal on STOP channel to enable the program, and waits a single digital input from external PLC to start the program . When the program finished the routine, a single digital input is send to external PLC.

DISPLAY AND CONVERTION POLARISATION CURVE

This utility displays on PC the saved LSR scan saved by the program.

The log files of polarisation curves downloaded from the FP can be display using the "ConvertionArchiveModule.exe" utility.
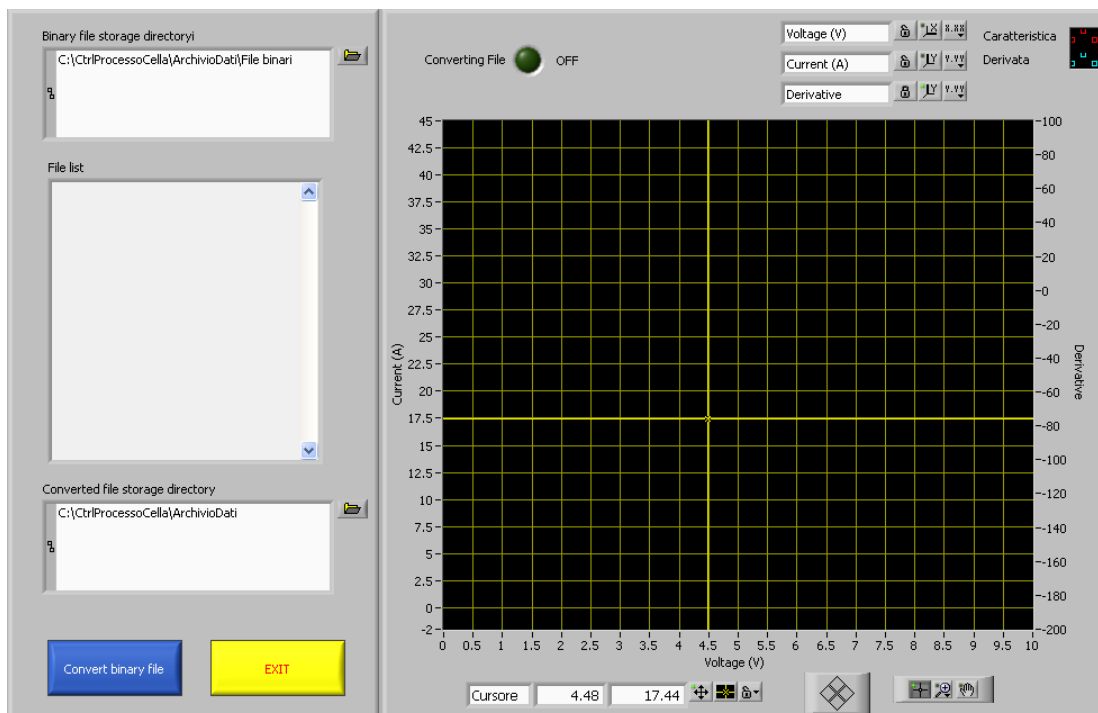
A left click on Dir Binary File button browses into the directory in order to select the log file location. When the directory is opened, click on a single log file and select "Select Cur Dir".
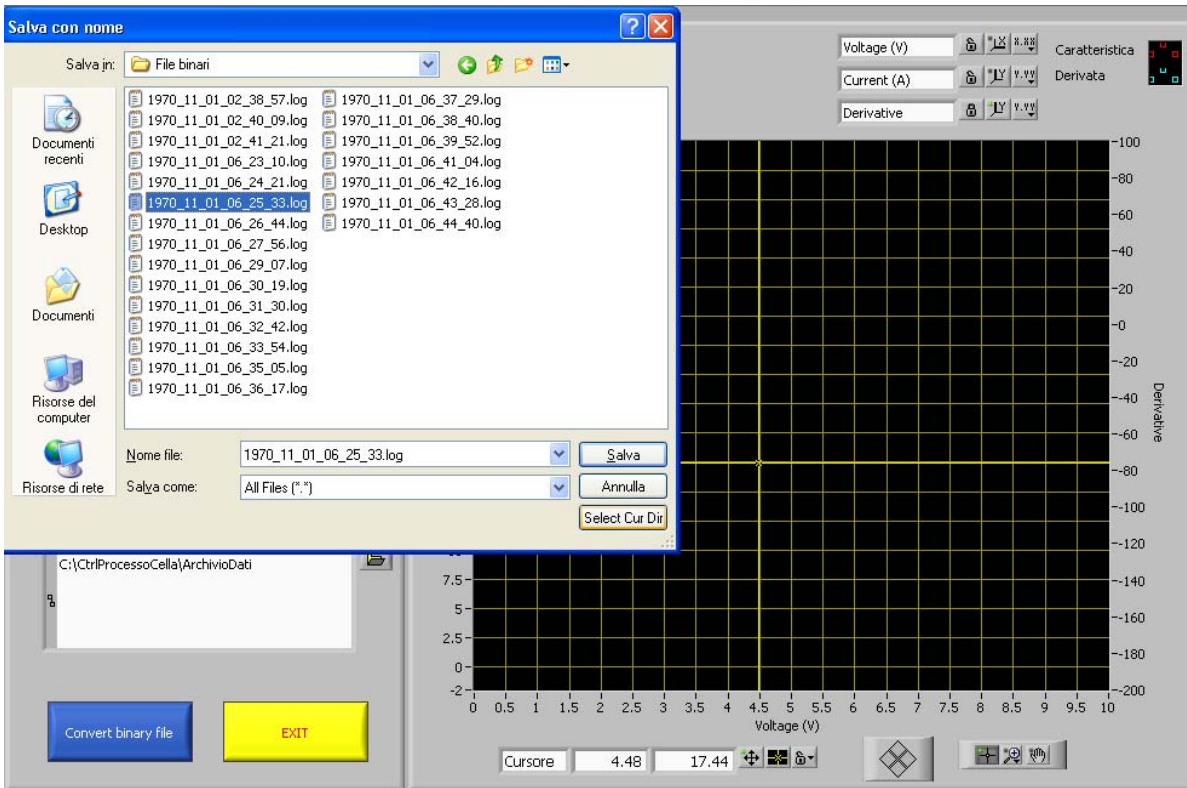
All the log file are now displayed on "List Files" box. Clicking on a file listed in "List files" box displays the curve in the lateral screen.

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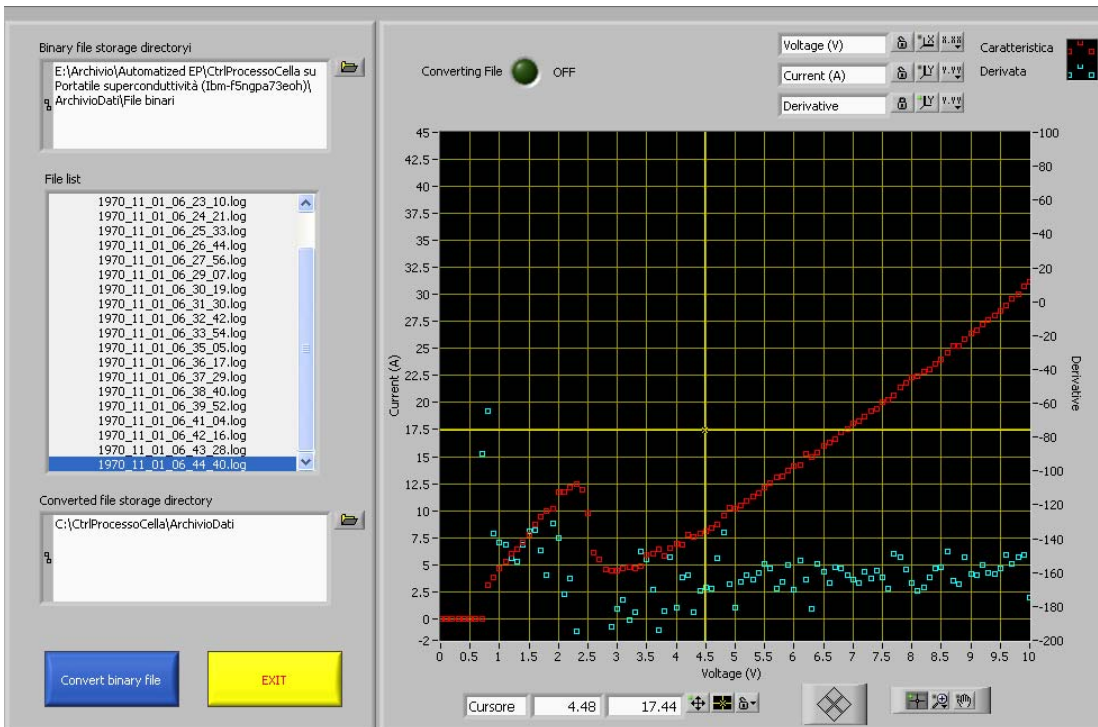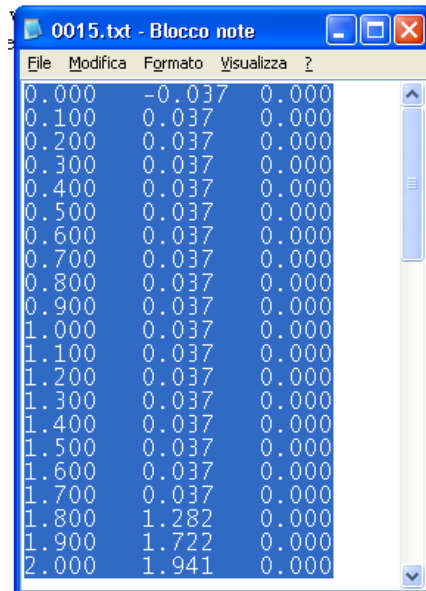
To convert the log files into a text file as tabulation separated colum, click on "Convert Binary Files" button. The program asks a directory destination, it's possible create a new directory or retain one existent. Open the directory and click "Select Cur Dir" button.
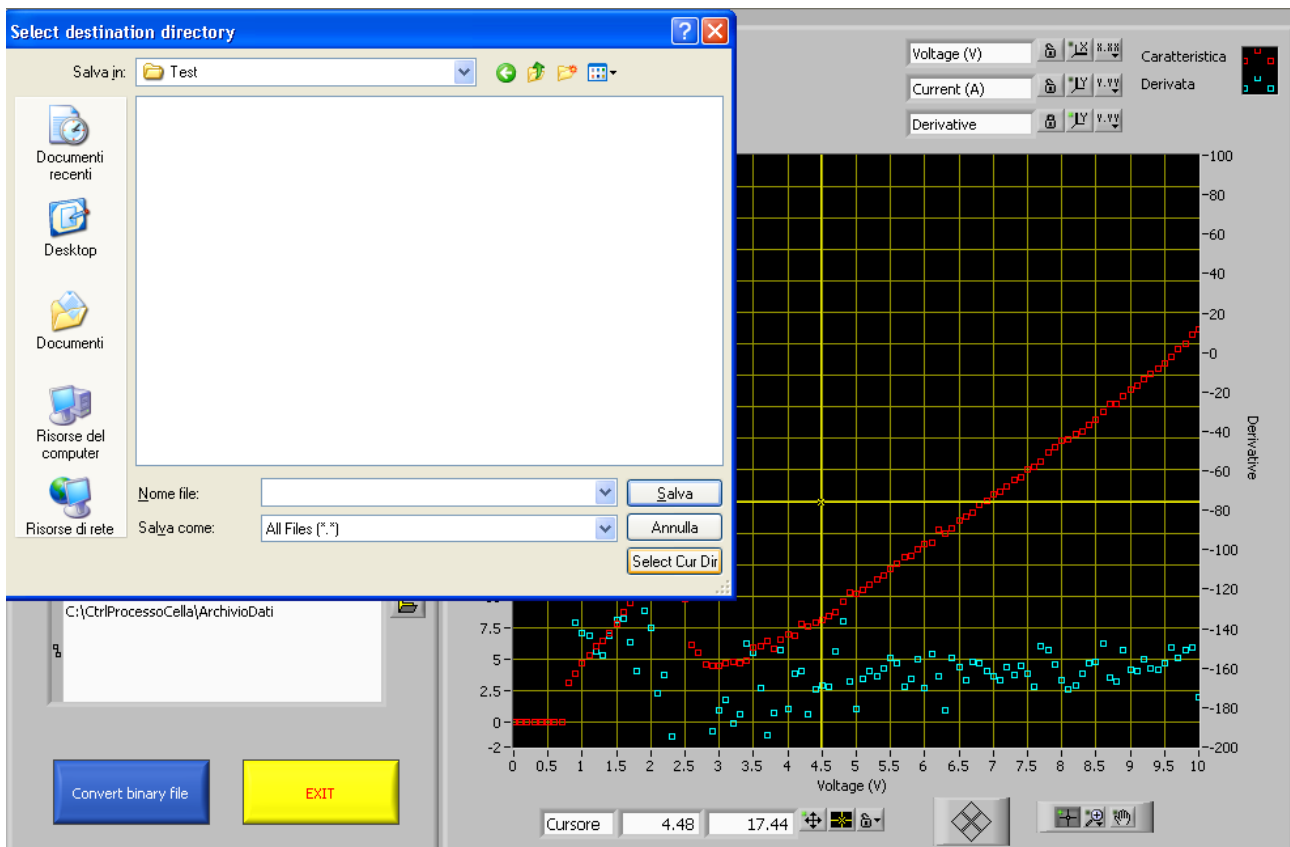
The convertion program starts and the text files are listed on the destination directory. This files can be opened by a mathematical program, as "Origin®" by OriginLab Corporation or another similar one.

The first column represents the voltage values, the second one the current obtained and the third one shows the derivative dI/dV data.

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PROBLEM

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INTRODUCTION ionic liquids

   Niobium superconducting resonant cavities are the heart of a particle accelerator: they accelerate beams of electrons to speeds close to that of light, allowing the execution of particle physics and nuclear physics experiments. In particular, collisions of great interest are the electron-positron collisions: studying the subatomic particles and phenomena that arise after the impact, physicists can understand better and better the structure of matter [1, 2].

   The efficiency of resonant cavities can be improved by acting on various parameters, one of the most influential is the surface finish: it is universally accepted and reported in the literature that a smooth, polished and free of defects surface ensures better performance of a rough one. There are various techniques to improve the surface finish: mechanical, chemical and electrochemical (electropolishing). The latter in particular has proved to be the most effective [3].

   Usually, electro polishing of niobium cavities is performed in acid electrolytes consisting of a solution of sulphuric and hydrofluoric acid, in volume ratio of 1:9 [4]. Because of the hazards related to the use of these substances, during the recent years several research teams have developed alternative electrolytic solutions, more or less effective [5, 6].

   At LNL (Legnaro National Laboratories) a new type of electrolyte for niobium electro polishing is being tested. It is an ionic liquid, i.e. a salt that is liquid at room temperature, obtained by mixing two solids: urea and choline chloride. This electrolyte was first introduced in 2003 by Abbot et al. [7]. Subsequently, they showed its potential successfully electro polishing stainless steel [8]. Recent studies at LNL have proven that electro polishing of niobium, using the above ionic liquid, is possible and the results are very promising considering the fact that this is a new process, virtually unknown, unlike the "classic" electro polishing process in acid solution, which dates back to the 70s and has been widely studied and improved [9].

   If an efficient and reproducible method for electro polishing niobium superconducting cavities with ionic liquid will be developed, it would demonstrate the existence of a viable alternative to the use of sulfuric and hydrofluoric acid in the treatment of cavities surface. In particular, it would be possible to introduce large-scale use of a chemical compound decidedly less dangerous, less polluting and less expensive than those used today.

   In this thesis the electro polishing process of small niobium samples in ionic liquid has been investigated, with the aim of providing guidance for future tests on cavities electro polishing. By exploring the basic parameters involved in the process (current density, temperature, duration, stirring), the optimum values in order to obtain the best possible polished surface have been determined. Once the best conditions have been find, the electro polishing in ionic liquid of some 6 GHz niobium superconducting resonant cavities has been tried.

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[3]   K. Saito, Proceedings of the 2003 Particle Accelerator Conference, Portland, OR, USA, 2003, p.462 – 466.
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[5]   J. R. Delayen *et al.* Proceedings of 10th SRF workshop, Tsukuba, Giappone, 2001.
[6]   V. Palmieri *et al.* Proceedings of 10th SRF workshop, Tsukuba, Giappone, 2001.
[7]   A. P. Abbott *et al.* Chem. Commun. 1 (2003) 70–71.
[8]   A. P. Abbott *et al.* Electrochim. Acta. 21 (2006) 4420–4425.
[9]   M. Ceccato, Tesi di Master, Università di Padova, Italia, 2007.

Theses

The theses documents can be found in the appendix.

TESI DI LAUREA
Giovanni Mondin
**UTILIZZO DI LIQUIDI IONICI PER
L'ELETTROPULITURA DI CAVITA'
SUPERCONDUTTIVE IN NIOBIO
CARE-Thesis-08-004-SRF**

Tesi di MASTER in "Trattamenti di Superficie per l'Industria"
Marcel Ceccato
**ELETTROPULITURA DEL NIOBIO CON ELETTROLITI
A BASE DI LIQUIDI IONICI
CARE-Thesis-07-004-SRF**

CONCLUSIONS ionic liquids

In this thesis the ability of the ionic liquid consisting of urea and choline chloride (in 3:1 molar ratio) to successfully electro polish niobium samples has been verified, demonstrating the efficiency of the process: the final result was a sample with an excellent surface finish, as proven by SEM and profilometer characterization. To achieve this result it was necessary to study the various factors affecting the process: current density, temperature, time, stirring and anode-cathode distance. The investigations carried out on these parameters showed that the ideal current density must be greater than about 0,2 A/cm^2 to avoid an uneven erosion, but less than about 0,5 A/cm^2 so that the sample does not present excessive roughness. The higher the temperature and the better the surface finishing, but it should not be exceeded the temperature at which the ionic liquid degrades, so it must stay below about 180 ° C. The higher the duration of the process and the higher the quality of the surface, although after 20 minutes the improvements seem negligible, then there is no need to prolong the electro polishing process for not erode excess material and change the structure of the object (thickness decreasing, rounding of corners, etc). Stirring has proven to be essential but difficult to control: it prevent the stationing of gas bubbles on the anode surface, which compromise a homogeneous electro polishing, but at the same time it can prevent the formation of the viscous film, necessary for the process: in short, stirring must be controlled carefully. Finally, the lower the anode-cathode distance and the better the surfaces finish.

The characterization of the best obtained sample (sample 71) showed that is possible to get results close to those achieved with the classic method, but using an electrolyte definitely safer and less expensive, just to cite two main advantages. Given the recent birth of this method is likely to assume that there will be future improvements that will be used to optimize it and achieve the same surface finish obtained with the classic method with acids. The same is true for niobium superconducting cavities: they are a far more complex system than a sample, thus requiring further study and a suitable electrochemical device. As mentioned in the introduction, there would be great benefits if a new ionic liquids-based method of electro polishing niobium superconducting cavities will be optimize and spread on large scale, such as greater safeness, lower cost, easy availability of reagents and their simple management.