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E-PLATE: Electrostatic Powder pLating for Accelerator TargEts

Project financed as Grant for young researchers CSN5 2018-2019

Report of the activities

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E-PLATE

Electrostatic Powder pLating for Accelerator TargEts

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Detailed description of activities realized

WP1. AUTOMATED DEPOSITION SYSTEM CONSTRUCTION

WP1.1. Choice of the automatable power supply.

Preliminary search of the power supply has included the EPS series of ISEG Spezialelektronik GmbH, MK, FJ and KT series of Glassman High Voltage Inc., BT-GP Series of Advanced Energy, SL and SLM series of Spellman High Voltage Electronics Corp. For the further evaluation MK60N1.2 and FJ60R2 of Glassman and SL60N60/230 and SLM60N300 of Spellman were chosen. According to an evaluation based on technical details, convenience of automation and final cost of the unit, the Spellman SL60N60/230 with eSL Ethernet option was chosen. The order of purchase was realized.

WP1.2. Design of power supply control system.

The PC-based automation system based on LabVIEW was chosen in order to have maximum flexibility for any further changes in control system. The PC should be dedicated only to this experimental set-up.

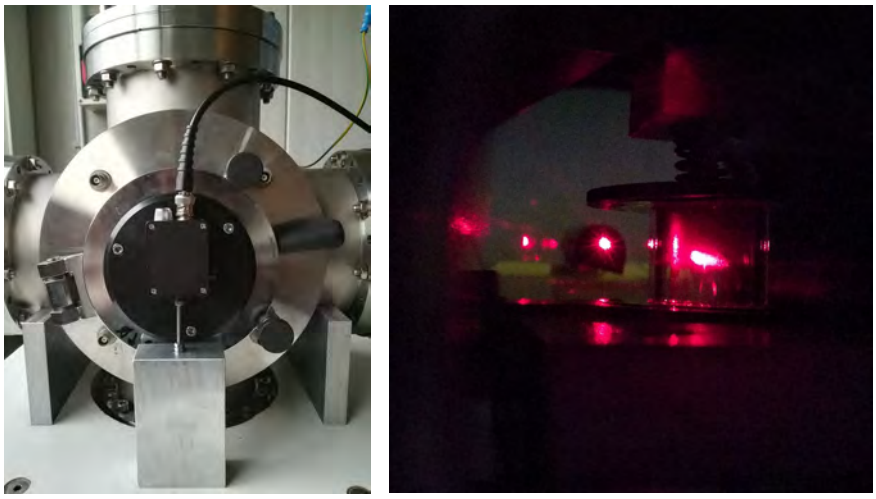

FIGURE 1. SETUP FOR LASER TEST OF POWDERS MOTION CONTROL

For the remote control of the process the laser-based approach was tested in February 2018. It should be mentioned that before we have used only visual observation to control the process, that is far from the strategy of complete process automation. We have tested as the light source the simple laser pointer and then laser diode powered with lab variated range HP E3612A DC power supply (0-60V, 0-0.5A) and photoresitance and then DET100A Thorlabs photodiode as a receiver. To observe the photodiode signal Tektoniks MDO3024 oscilloscope was used. The back blind flange of the system was changed to a viewport. All components were borrowed all around the LNL lab for the trial.

The laser and photodiode were placed outside the vacuum chamber in a way that a laser beam was passing the quartz cylinder of the deposition system (Figure 1). According to the set of tests with the type of the signal it seems possible to distinguish the presence (Figure 3) or absence (Figure 2) of powders movement and sparks. In a case of no movement and metallization of quartz cylinder the signal is supposed to be a line similar to Figure 2, but lower level. If any particular behavior of the powders not described by this signals will occur, visual cross-check is required to understand it.

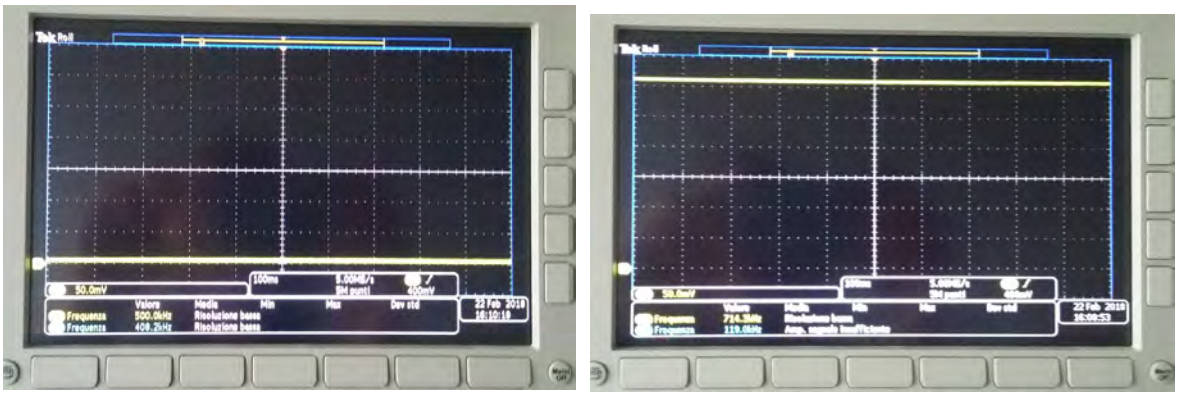

FIGURE 2. OSCILLOSCOPE SIGNAL NO POWDERS MOVEMENT LASER OFF (LEFT) AND ON (RIGHT)

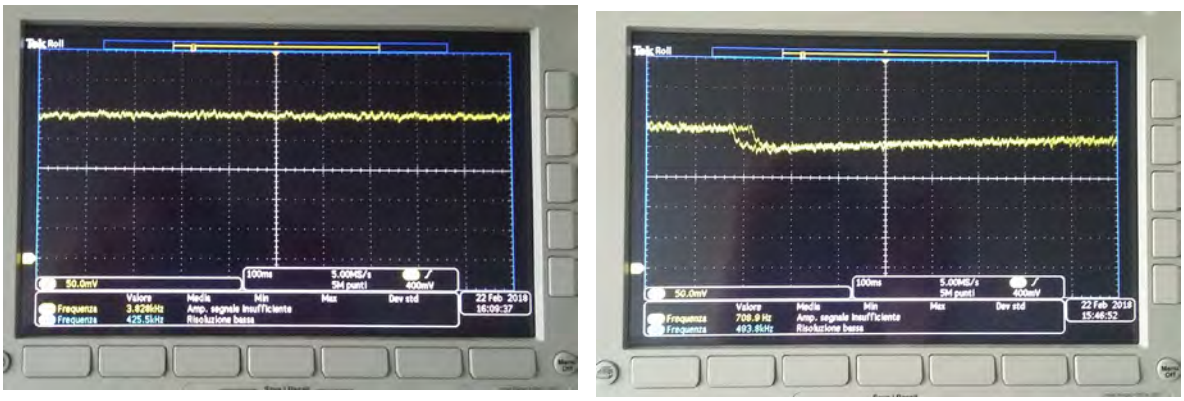

FIGURE 3. OSCILLOSCOPE SIGNAL POWDERS MOVEMENT NORMAL (LEFT) AND SPARK (RIGHT)

Conclusion: control of powders motion in HIVIPP (HIgh energy VIbrational Powders Plating) process with the laser in transmission is possible. **The next components are required for system realization: Laser diode with power supply, photodiode, additional view-port, LabVIEW-Icompatible board to read the signal directly inside LabVIEW program.** For the moment we propose such method for on-line monitoring of the process and post-data treatment. In future the opportunity to make the interlock for HV power supply switching off, when no powder motion occurs because of cylinder metallization is also taking into account.

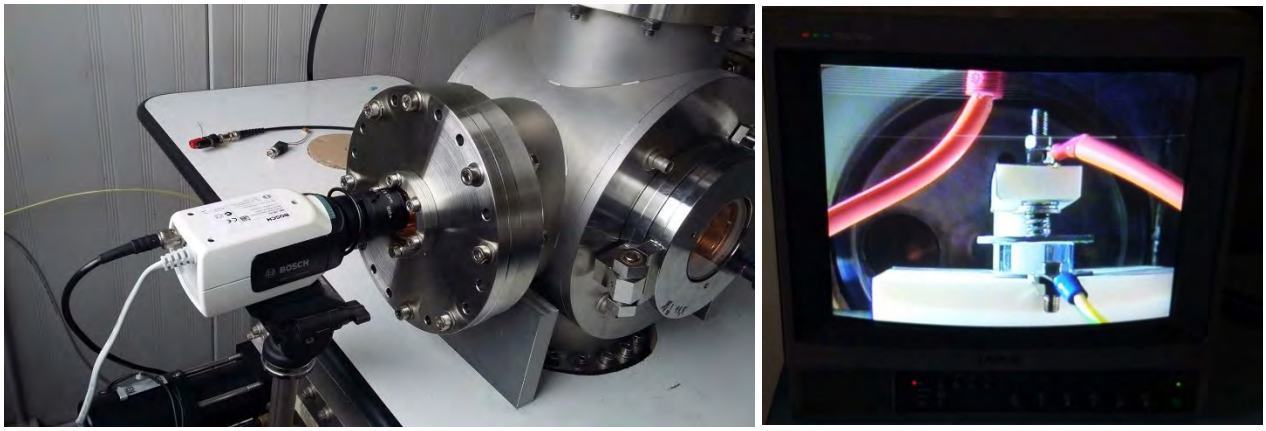

FIGURE 4. CCD CAMERA SETUP FOR POWDER MOTION OBSERVATION

Also an alternative distant on-line method of the process observation was evaluated in February 2018: the BOSCH VBC-255-51 color CCD camera with a monitor (all pieces borrowed). Unfortunately, either due to low level of light, or due to low sensitivity the powders movement is not seen well, but we plan to retry inserting a chain of LEDs inside the chamber to increase the illumination.

WP1.3. Purchase of the components.

The definition of the suitable HV power supply was realized in advance. The Spellman production is realized in USA and the delivery terms are long enough (>3 month). In order to help us to realize system automation in time terms proposed (within June 2018), the necessary funds were anticipated with the agreement to return back in 2018. The order of purchase SL60N60/230 with eSL Ethernet option been realized with the final cost is 5kE.

The list of chosen electronic components required for automation is in stage of preparation. It is comprising power supplies for gate-valve and valve for turbo pump safety, components for powders motion monitor, described in previous paragraph, board for LabVIEW for reading analogue signals, possible CCD camera (if we reach good sensitivity with increased light intensity and if still the laser signal will be possible to distinguish) Ethernet hub to connect to Ethernet vacuum controller and HV power supply.

WP1.4. System installation.

The HV power supply has just arrived in March 2018. New system installation will start after the purchase of missing electronic components. The tests of installation laser-based powders motion monitor are described in previous paragraphs. **It should be said that in order to guarantee to the laboratory the safety of operation of new 60kV power supply, the system should be equipped by 60kV high vacuum feedthrough.** Thus, we ask to move the funds for the feedthrough from the year 2019 to 2018.

The construction of vacuum system for the HIVIPP deposition was complete in June 2017 (at the moment that E-PLATE project was proposed to CSN5) at “zero-cost” mainly using recovered units. Pumping group included Alcatel 5400 turbomolecular pump with controller and Varian SH-100 scroll pump.

FIGURE 5. MODIFICATION OF THE PUMPING GROUP

Working in the second part of 2017 and beginning of 2018 we have met a number of problems of recovered components. The scroll-pump of that type should provide $5x10^{-2}$ mbar ultimate pressure, but due to a defect of the scroll its best performance is $1x10^{-1}$ mbar, characterized also by elevated thermal load to the ambient.

The Alcatel 5400 turbomolecular pump is an old machine (‘90th) with a number of problems: the inlet pressure should be no worse than $1x10^{-2}$ mbar, it can be fixed only in vertical position (and in our system in this case only below the chamber, because of its water cooling for the safety reasons). The controller of the turbomolecular pump has its own problem – it has bad control of the frequency of the pump, which results in not so stable pump operation and high level of noise. If the turbo is forced to start at $1x10^{-1}$ mbar instead of $1x10^{-2}$ mbar, more rotation frequency variation appears. Due to this turbo frequency problem the initial start of turbo up to more-or-less stable frequency requires more than a working day.

Trying to preserve turbo in safe mode operation an additional pump Alcatel Drytel 30 borrowed was added to a system, using for pre-vacuum (Figure 5).

It should be mentioned that both pumps have no opportunities for interlock automation. We are searching for a solution for more safe system operation inside LNL lab.

WP1.5. Programming automation of PS for full process with the arc control.

The main logic of programming of the power supply automation was developed in January 2018 (see Figures 7), comprising HV ramp and discharge alarm (Figure 6).

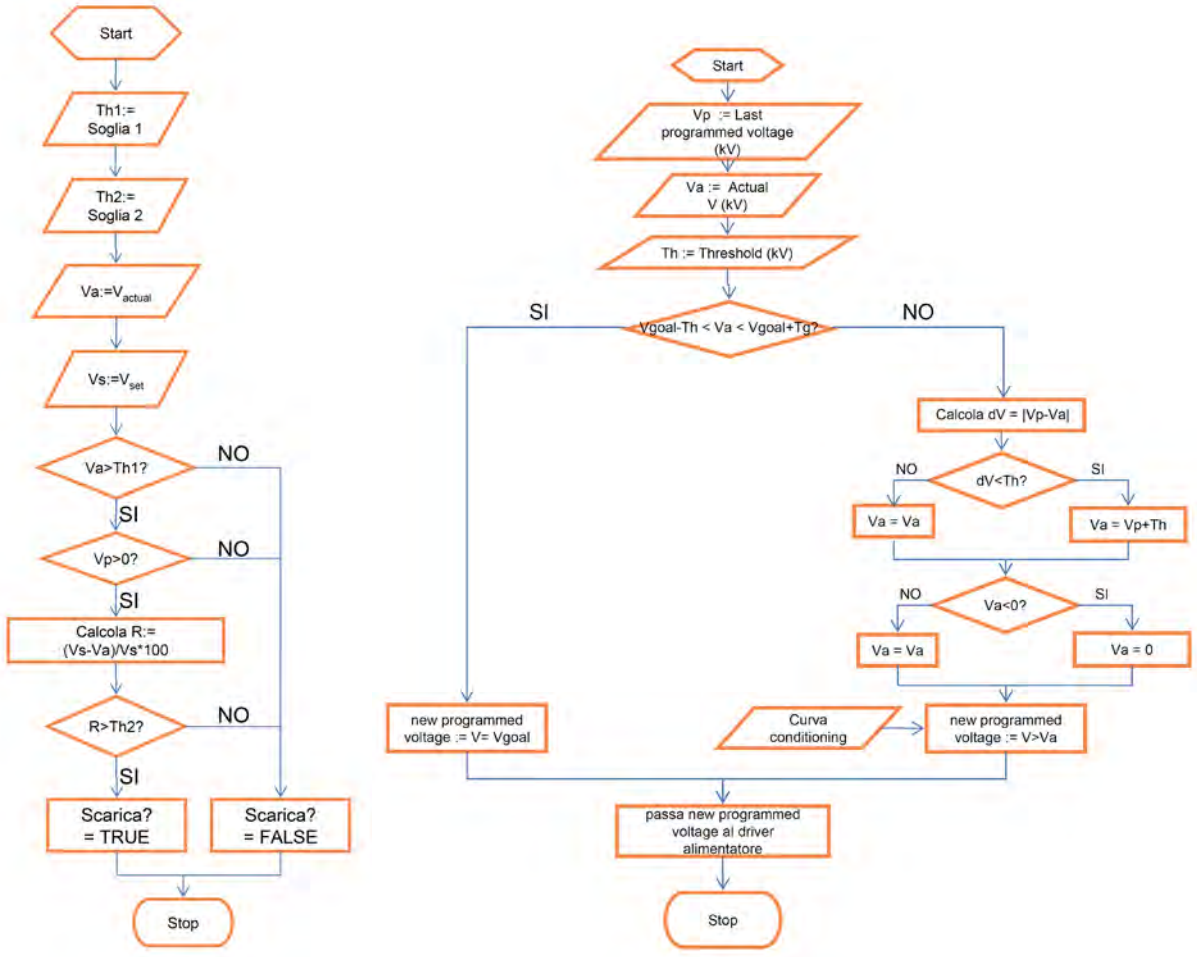

FIGURE 6. DISCHARGE ALARM (LEFT) AND VOLTAGE RAMP (RIGHT) PROGRAMMING LOGIC

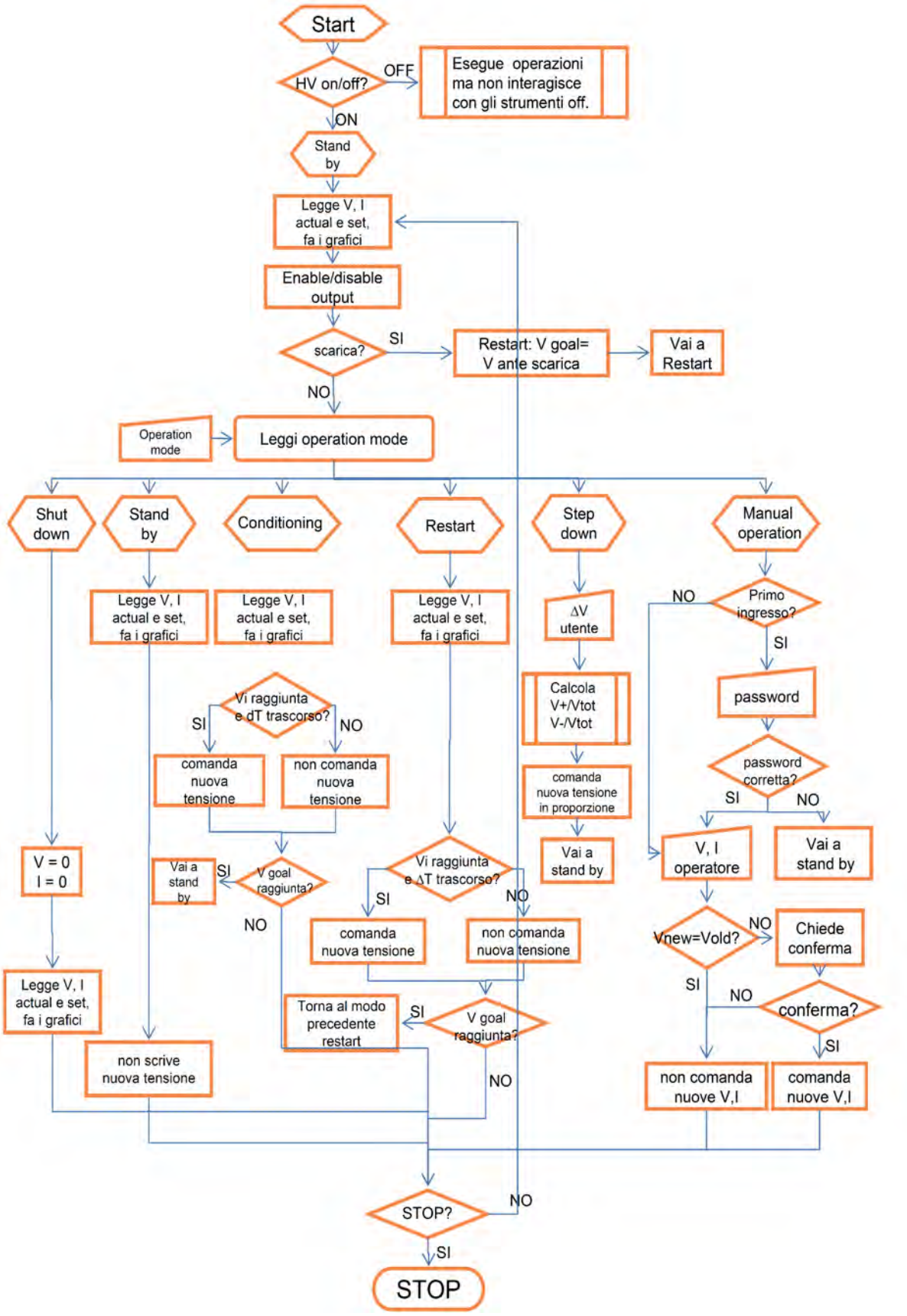

FIGURE 7. GENERAL HV POWER SUPPLY PROGRAMMING LOGIC

WP1.6. Automation of safety interlocks.

The level of vacuum based on the signal of the vacuum meter controller (Ethernet compatible) is decided to be used as an interlock for the HV power supply. Other interlocks are under evaluation.

WP2. DEPOSITION OF TARGETS FOR CROSS-SECTION MEASUREMENT EXPERIMENTS

WP2.1. Ti for PASTA project.

In E-PLATE project we proposed to provide Ti-48 and Ti-50 enriched targets to PASTA project as a deliverable. For the moment the list of Ti isotopes of interest of PASTA was enlarged including also Ti-49. The WP2 is a kind of implementation of HIVIPP technique in the field of cross-section measurements. By the logic the implementation should start after the study of the process, but we proposed to do it in parallel to cover the needs of the project in collaboration.

A number of experiments on Ti-nat (September-October 2017) and Ti-48 enriched (October 2017-January 2018) was realized. The Table 1 presents the results, the Figure 8 shows an example of Ti-nat and Ti-48 targets on different thickness Al substrate deposited with HIVIPP technique. The deposition parameters are not presented here, because with the current set-up and manual variation of the voltage provided by the old power supply the process is uncontrollable with the set of the parameters non-reliable. Even if we were able to realize a set of depositions and provide Ti-48 targets for PASTA (just 50% of enriched targets were finally used for irradiation experiment), we are not able to explain the difference in deposits and to reproduce the process with reliability. Indeed, the automation of the process is one of the objectives of E-PLATE project and it is necessary to understand the HIVIPP technique.

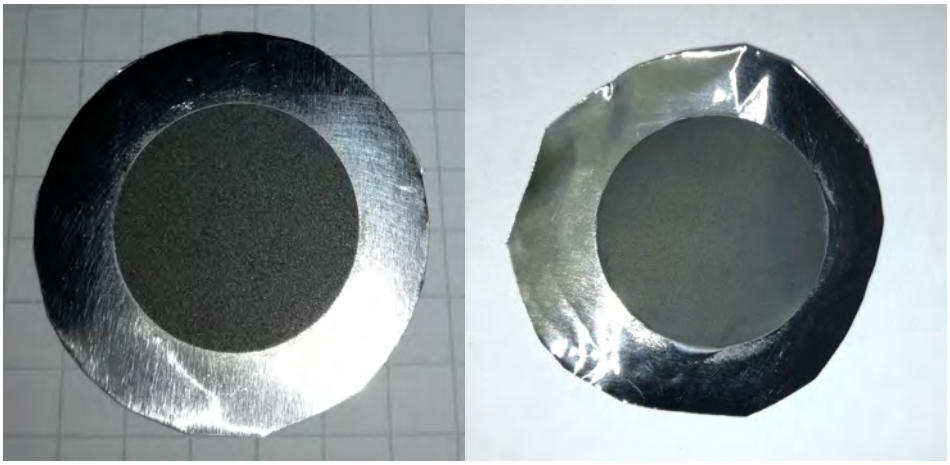

FIGURE 8. TI-NAT TARGET ON AL 100UM (LEFT) AND TI-48 TARGET ON AL 25UM (RIGHT)

The order to purchase Ti-49 and Ti-50 isotopic powders from Oak Ridge National Laboratory is already in process. The deposition and analysis is expected to be done in May-June 2018.

WP2.2. Cr for METRICS project.

We are at the stage of choice of Cr natural powders for the first tests.

WP3. INFLUENCE OF NEW FACTORS ON THE DEPOSITION PROCESS

WP3 will be realized after the automation of the system is complete. Some observations on WP3.2 are already available.

TABLE1: RESULTS DEPOSITION TI-NAT AND TI-48 ENRICHED

N	Sample description			Ti deposited density		Ti bulk thickness	Ti deposited density RBS (preliminary data)	
	Element	Powders	Substrate	mg/cm^2	10^{18}At/cm^2	10^{18}At/cm^2	10^{18}At/cm^2	µm
14l	Ti-nat	5-50µm	Al 100µm	4,5	57	10	no RBS measurement was available	
14u	Ti-nat	5-50µm	Al 100µm	4,8	60	11		
15l	Ti-nat	5-50µm	Al 100µm	2,9	36	6		
15u	Ti-nat	5-50µm	Al 100µm	3,2	40	7		
16l	Ti-nat	5-50µm	Al 100µm	3,8	47	8		
16u	Ti-nat	5-50µm	Al 100µm	3,8	48	9		
17l	Ti-nat	5-50µm	Al 25µm	2,9	36	6		
17u	Ti-nat	5-50µm	Al 25µm	2,6	33	6		
18l	Ti-nat	5-50µm	Al 100µm	4,7	60	11		
18u	Ti-nat	5-50µm	Al 100µm	3,8	47	8		
20l	Ti-nat	5-50µm	Al 25µm	3,4	43	8		
20u	Ti-nat	5-50µm	Al 25µm	3,0	38	7		
21l	Ti-48	0.1-5µm	Al 25µm	1,2	14	3		
21u	Ti-48	0.1-5µm	Al 25µm	0,4	5	1		
22l	Ti-48	0.1-5µm	Al 25µm	0,2	2	0,4		
22u	Ti-48	0.1-5µm	Al 25µm	0,2	2	0,4		
23l	Ti-48	0.1-5µm	Al 25µm	0,3	4	0,7		
23u	Ti-48	0.1-5µm	Al 25µm	0,4	5	0,8		
24l	Ti-48	0.1-5µm	Al 25µm	1,4	18	3	Non-uniform	
25l	Ti-48	0.1-5µm	Al 25µm	1,0	12	2	9,0	1,6
25u	Ti-48	0.1-5µm	Al 25µm	0,7	9	2	8,9	1,6
26l	Ti-48	0.1-5µm	Al 25µm	0,3	3	0,6	3,0	0,5
26u	Ti-48	0.1-5µm	Al 25µm	0,4	5	0,9	3,6	0,6
27l	Ti-48	0.1-5µm	Al 25µm	0,6	7	1	7,1	1,3
27up	Ti-48	0.1-5µm	Al 25µm	0,6	7	1	7,1	1,3
28l,u	TiO_2		Al 100µm	Not uniform detached easily				
29l	Ti-48	0.1-5µm	Al 25µm	2,1	27	5	Non-uniform	
29u	Ti-48	0.1-5µm	Al 25µm	4,0	50	9		
30l	Ti-48	0.1-5µm	Al 25µm	1,1	13	2	8,6	1,5
30u	Ti-48	0.1-5µm	Al 25µm	2,2	28	5	7,8	1,4
31l	Ti-48	used in exp 30 + mortar	Al 25µm	0,4	5	0,9	2,1	0,4
31u	Ti-48		Al 25µm	0,3	4	0,7	1,5	0,3
32l	Ti-48	0.1-5µm	Al 25µm	0,6	8	1	3,4	0,6
32u	Ti-48	0.1-5µm	Al 25µm	0,5	7	1	3,2	0,6
33l	Ti-48	0.1-5µm	Al 25µm	0,7	8	1	4,0	0,7
33u	Ti-48	0.1-5µm	Al 25µm	0,4	5	0,9	2,6	0,5

WP3.2. Powders properties.

The test with TiO_2 natural powders (exp. 28) realized in January 2018 with the current experimental set-up has resulted in very bad uniformity of the deposit, and after the process it was observed that attachment of oxide powders on Al substrate is only electrostatic, without bonding.

During deposition of Ti-48 at the beginning of the set of experiments (October-December 2017), when the box with Ti-48 powders was just opened (exp. 25-27), the results of measurement of weight and RBS are enough similar, instead, following tests made in January 2018 (exp. 30-33) showed higher discrepancy between weight measurement and RBS data, attributed possibly by superficial oxidation of powders during storage.

Using the powders of previous experiment (exp. 31) much lower quantity deposited was observed. The reason also could be the oxidation.

Thus, is evident that oxidation of the powders is very important parameter for the process of HIVIPP deposition. **In order to realize the experiments in conditions "non-disturbed by oxygen" we would like to perform all the manipulation of Ti and Mo powders in an inert atmosphere. For this we propose to use gloves-box with Ar atmosphere. As a low-cost initial trial the use of a glove-bag with Ar flow is planned.**

If the oxidation is so important is needed to return back also to the aspect of improvement of the level of vacuum inside the quartz cylinder. When the internal system is closed, Al foil is providing a kind of sealing, and pumping inside cylinder is very complicated. **The internal system of electrodes and cylinder is to be redesigned in next month. And the design will vary in respect to the decision to make cylinder opening or not.**

For the evaluation of the influence of powder size, the use of sieves to separate the powder is decided. A market research was done in order to find the best solution responding to our requirements, which are:

- Very low amount of powder (tens of mg) to be sieved means very small sieves;
- Small size of the net opening (5, 10, 30 µm);
- Metallic nets of the sieve in order to avoid electrostatic attachment of the powder;
- No losses of the sieved powders.

Several companies suggested such types of machines based on different principles:

- Air jet sizer from EN.CO. S.r.l or VERDER scientific (This type of sieving machine is effective for small size powders, but the system is used to separate large amounts of powders in respect to our request and some powders with size less than um can be lost, machine has much higher cost than mechanical sieving)
- Analytical vibratory sieve shakers from Retsch® (AS 200 basic) or FRITSCH (Analysette 3 pro) and others. (This type is the most standard and economic, but the external size of the sieve is starting from Ø10cm, thus they are too large for our amount of powder. Besides that, no guarantee efficient the sieving of micron-size powders)

As a solution we have found the Wet Sieving machines that can be used also for dry sieving. They allow to treat lower amount of material thanks to smaller external diameter of the sieve and availability of grids with smaller opening. This seems the best choice for our purpose but

the machine cost 3.5k€, and the sieves, cover, bottom collector must be taken separately (~200€ each).

For the first trial, the manual use of the sieves without machine (Stainless steel sieve) has been chosen. Different companies provide the sieves of different sizes and measuring range (HAVER & BOECKER, Giuliani Tecnologie, Vetrotecnica). At the end the purchase was done from the cheapest company, Vetrotecnica. The purchase order is in process. If the manual sieving is not efficient, purchase of sieving machine will be done (the missing 1.5kE should be taken from preserved part of the purchase of HV power supply). It should be noted, that sieving in not oxygen-free atmosphere will cause oxidation of powders.

WP4. FINAL UPGRADED TECHNIQUE

WP4 will be realized after the WP3 is complete (in 2019).

WP5. ANALYSIS

WP5.1. SEM, EDS, optical microscopy

SEM was used to understand the difference of the microstructure of the targets deposited with different powders of Ti (Figure 9, 10) and for quality control.

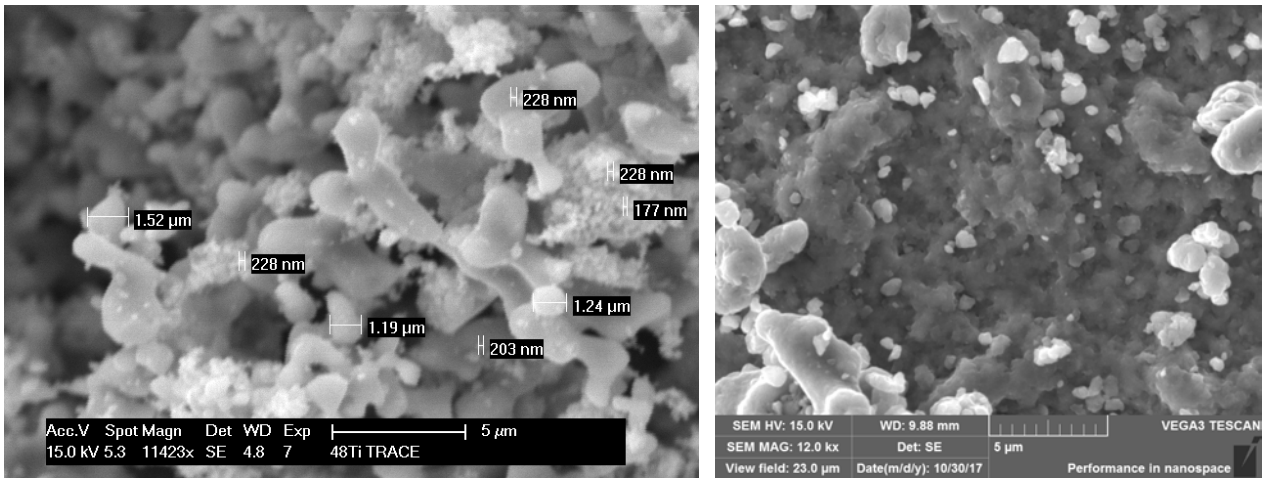

FIGURE 9. SEM OF TI-48 FROM TRACE (LEFT) POWDERS AND TARGET PREPARED BY HIVIPP DEPOSITION ON AL 25UM SUBSTRATE (RIGHT)

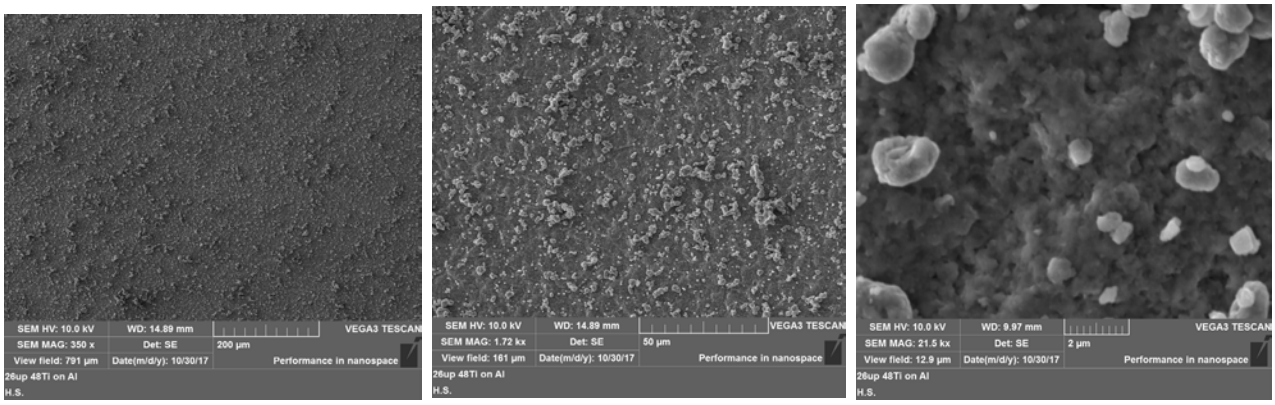

FIGURE 10. SEM ANALYSIS OF 26UP TI-48 TARGET ON AL 25UM SUBSTRATE AT DIFFERENT MAGNIFICATION

The SEM images at high magnification (like the one for Ti-48 deposit on Figure 10 right) have bad quality, they are blurred. **This is related to a distortion by the external vibration sources.**

To improve the performance of the SEM is required maintenance including the control of the sources of vibration.

WP 5.2. Irradiation tests.

The irradiation tests of 21_L, 23_L, 23_{UP}, 25_L, 25_{UP}, 27_L, 27_{UP} Ti-48 targets has been realized with C70 cyclotron in ARRONAX, France in the framework of PASTA project. The two irradiations were done at 34 MeV and 40MeV and 115nA proton current. No any visual modification or damage of the targets occurred during irradiation. The results of gamma spectroscopy of the products of the nuclear reaction is under treatment by our collaborators of PASTA. It should be mentioned that the gamma-spectroscopy showed presence of desired Sc-47 in the target after irradiation, and also that the choice of 25um Al substrate has permitted to get the gamma-spectra with the Sc-47 enough good quality signals in respect to the Al monitor (no signal oversaturation because of too thick Al). This means that the targets produced by HIVIPP are fitting the requirements of cross-section measurement. The samples $30_{L,UP}$-$33_{L,UP}$ are ready for the next irradiation at ARRONAX in April 2018 in the framework of PASTA to complete the measurement on Ti-48 cross-section.

WP5.3. RBS analysis for target.

Three AN2000 turns of RBS measurement of Ti-48 enriched targets on Al substrate were realized on 24.10.17, 06.12.17 and 01.02.18 with 1.8 MeV 5-30 nA proton beam. The experimental set-up is presented in Figure 11. The data treatment was realized with free XRump software.

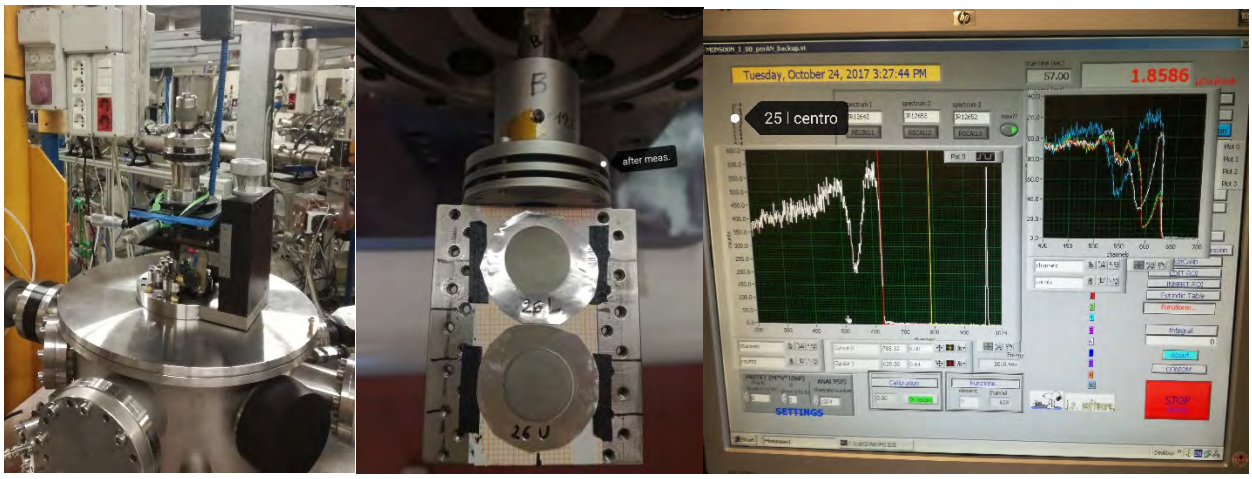

FIGURE 11. EXPERIMENTAL SET-UP FOR RBS MEASUREMENT: CHAMBER WITH PRECISE POSITIONING SAMPLE-HOLDER ON THE TOP, SAMPLE-HOLDER, SOFTWARE FOR DATA ACQUISITION

Treatment of RBS data appeared to become a real challenge! First, because at 1.8MeV proton irradiation Al (substrate) has non-Rutherford cross-section with a number of resonances, that finally are distorting the spectrum of Ti on Al targets (Figure 12). Besides that, the Ti peak appeared to have a modified shape, that can be explained by extremely high roughness of the deposit and the intermixing of Ti and Al substrate (Figure 13).

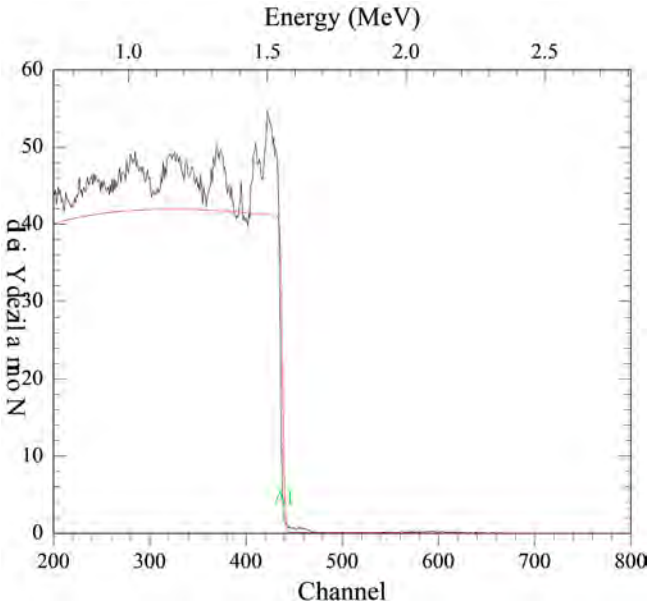

FIGURE 12. REAL SPECTRUM OF AL (BLACK) VS RUTHERFORD SPECTRUM OF AL (RED) AT 1.8MEV PROTON INCIDENT BEAM

The preliminary data presented in Table 1 were obtained simply as Ti-48 peak integral. It has shown enough difference from the thickness expected from the weight analysis (Table1). The reason could be the presence of oxygen in deposit that is evident by RBS.

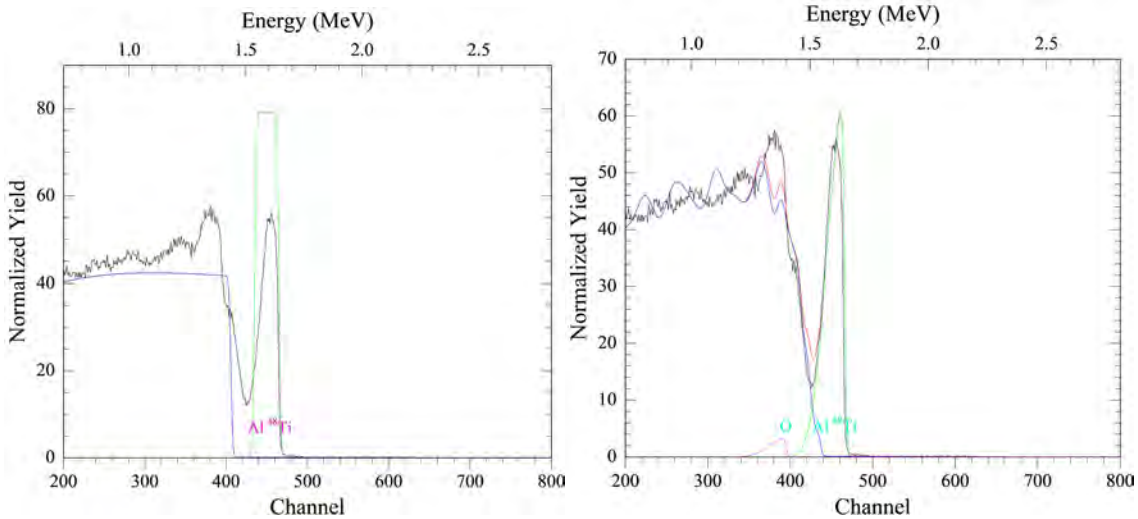

FIGURE 13. SIMULATION SIMPLE TI LAYER ON AL (LEFT) AND WITH AL NON-RUTHERFORD CROSS-SECTION, INTERMIXING LAYERS, ENERGY STRAGGLING, OXIDATION TI (RIGHT)

More reliable data analysis is in work. Several tricks for the modelling with XRump are under consideration: addition into database of XRump of Al non-Rutherford cross-section data, intermixing of layers, correction of energy straggling due to thickness, modelling oxygen content in Ti. Figure 13 shows some trials. **Creation of the model able to describe well the RBS experimental data and real properties of the samples, can require additional measurements: like profile measurement in order to use the roughness characterization as a parameter of the model, and also probably additional measurements with deuteron beam (CN of LNL is proposed) to measure quantitatively oxygen,** since the background in this reaction is low, and oxygen can be observed better.

It is evident that only sample weight measurement analysis is not sufficient. Adding the RBS analysis is still not providing complete characterisation. Thus, we have evaluated also the opportunity to measure the roughness and profile of the deposit with a portable profiler.

The market study in order to find a portable profiler able to provide variable controlled low level stylus force (in order to don't damage the samples) was realized. Several companies were contacted: OGP HOMMEL Italia S.r.l., Mahr U.K. Plc, Carl Zeiss S.p.A, Schaefer South-East Europe Srl., Mitutoyo Italiana S.r.l., G.Gambetti Kenologia Srl. No-one can propose portable contact profilers (roughness measuring device) with variable stylus force (for all is fixed at 0.75mN). Even non-portable benchtop profile stages have the lower limit force of about the same 1mN. The only solution about contact profilers is the laboratory benchtop devices like DektakXT® of Bruker Nano Surface Division or Alfa-Step® of KLA-Tencor, that give stylus force of 0.03-15mg (more or less corresponding to 0.3-150 μN). Alternative solutions assuring no damage of samples are optical profile devices, like SmartScope FlashCNC 200 of OGP, Zeta 20 of Zeta Instruments Inc., Sensofar S of Sensofar, ST500 of Nanovea. All these devices are requesting significant financement, exceeding the current project budget, thus, we are searching for the alternatives to control thickness and roughness of the deposit.

WP6. PRESENTING RESULTS

There were some discussions with the other groups interested in HIVIPP technique for target preparation. Particularly, there is the interest of target preparation group (Torino) of the INFN project NUMEN to test the opportunity to deposit Sn-onto pyrolytic graphite foil.

MILESTONES AND DELIVERABLES CONTROL

TABLE2: MILESTONES, DELIVERABLES 2018

Name	Description	Starting time	Deadline 2018	Realized	
M1	Realization of automated deposition system	01.01.18	**30.06.18**	30%	
M2	First batch of experiments with different Mo deposition parameters	01.08.18	**31.12.18**	0%	
D1	New system installation (no automation)	01.04.18	31.05.18	0%	
D2	Ti-nat targets on AL foil for PASTA	01.09.17	01.11.17	100%	Overall ~40%
	Ti-48 targets on AL foil for PASTA	01.10.17	01.04.18	100%	
	Ti-49 targets on AL foil for PASTA	16.04.18	08.06.18	0%	
	Ti-50 targets on AL foil for PASTA	16.04.18	08.06.18	0%	
D3	Completely automated deposition system	01.01.18	31.07.18	30%	
D4	Cr-nat targets on AL foil for METRICS	13.11.18	11.12.18	0%	

*The deadlines for the milestones realization defined by referees are **in bold**.*