Executive summary:

The first X-rays imaging systems were developed more than 100 years ago, but the technology used to produce X-rays has changed very little since then; despite being a mature technology whose technological advancements are currently limited by its physical features to upgrades especially in software components, design and form factors, X-rays instrumentation has experienced a lot of research and developmental activity. Research is pushed forward by manufacturers and final end-users, yearning to overcome the huge downsides carried by conventional technologies, mainlydue to their most critical component: the electron source.

Medical diagnostics devices, archaeometric characterizations, fluorescence spectrometry, security, quality control in various industrial fields (electronics, aerospace, mechanics, food, etc), are only some of the main sectors demanding a breakthrough in X-Ray devices, in terms of reliability, cost effectiveness and performances. Heat generation, power consumption, out-gassing, thermal inertia, thermal drift, slow response times and above all high maintenance costs due to relatively short life cycles of the electronic sources (heated filaments) are major drawbacks in the state of the art of X-Rays tubes. That's why some of the mostly renowned research centres in Nanotechnology and Field Emission properties of nanomaterials collected the requirements pointed out by the SMEs proposers of this project and envisaged a solution that represents a potential breakthrough for this sector: this project,indeed, will realise an innovative and well beyond the state of the art portable X-Ray generator based on carbon nanotubes (CNT) field emitters as a source of electrons. NANORAY is an innovative device capable to generate X-rays by means of a novel concept of cold cathode, based on carbon nanotubes selectively grown upon ad-hoc synthesized nanostructures (such as vertically oriented nanotubes on metallic tips, selected deposition of CNTs on predetermined areas and array of oriented bundles).

Its performances can be wrapped up by mentioning some of the most relevant features:
1)A reduced focal spot (down to 0.1 mm, well below the actual state of art for commercial X-Ray tubes;
2)A very low power consumption (due to the use of a field-emission based cathode);
3)A pulsed x-ray radiation with programmable width and repetition;
4)A long life-time

Moreover, the system is portable, with an overall weight of less than 5 kg (including power supply), allowing the use of X-rays in places like ambulances or in field security surveys. In addition to portability and easiness to use, the system provides higher image resolution with respect to the state of the art of X-rays devices thanks to the smaller focal spot, and it represents a cost effective solution for everyone is facing the economic issues related to the maintenance of thermionic cathodes. Therefore, instead of using thermionic emission from a hot filament to eject the required electrons, NANORAY proposes the implementation of field emission based cold cathode, using as electron source suitable architectures/array of carbon nanotubes.

The use of nanotube field-emitters in X-ray source design not only prolongs the lifetime of such sources, but also -by reducing the size of the source tube and the power supply required to drive it- lead to portable X-ray diagnostic systems for in the field use. Furthermore,

since electrons can be easily produced at room temperature by quantum mechanical field emission phenomena it does not require much electrical energy as the conventional thermionic tungsten filament tube does.
Another important feature of field emission based cathodes that has been exploited by NANORAY is the focalization both of the electron beam and of the X-Ray beam. An innovative solution has been achieved through the use of polycapillary optics, allowing a wider freedom of parameters in the design of the X-Ray source for specific applications.

Project Context and Objectives:

Two levels of quantifiable targets can be considered for NANORAY:
1) Technical and
2) Economic:
- Technical targets: NANORAY system will be basically a CNT based microfocus x-ray tube, using a field emission cold cathode with one focusing electrode. For x-ray generation, field-emitted electrons will be accelerated by the anode voltage to bombard on the target (Cu, Al will be in particular investigated as possible materials). The cold cathode will be closed in a glass device, with suitable feed trough pins, under medium-high vacuum conditions (10-5 - 10-6 Torr).

NANORAY will be mainly based on the following systems/components with high and innovative technological content:
-Cold cathode for electronic beam generation based on the Field Emission (FE) properties of CNTs. Although the concept of cold-cathode x-ray tubes has been investigated in the past using materials including diamond and carbon nanotubes CNTs, development has been hindered by the lack of cathodes that can deliver stable currents comparable to the values used in conventional x-ray tubes (approximately 10-50 mA for fixed anode and 50- 500 mA for rotating anode tubes). By optimization of the CNT films morphology and their adhesion to the substrate, the possibility of large and stable emission current from CNT cathodes has been recently demonstrated and two of the RTD performers (UNITOR and ENE) already realized and tested successfully proof-of-concepts of X-Ray tubes based on a simple diode configuration. The CVD deposition of CNTs will be optimized in order to achieve:
- An adequate adhesion of CNTs on substrate
- Accuracy and reliability in the deposition running with the aim to achieve a stable emitted current (with density values comparable or better than of conventional thermionic X-Ray tubes) and a longer life-time.
-Triode configuration with a focusing electrode able to reduced and control the X-Ray spot

NANORAY will be based on a cathode using a triode-type configuration that, on the base of the preliminary simulation carried out by the proposers and of literature data, could guarantee the achievement of:
-X-Ray flux comparable to that from the conventional x-ray tubes
-A focal spot of 0.3 mm
-The tuning of X-Ray intensity trough the control of the anodic current and anodic voltage
-The possibility to achieve very high frequency pulsed x-ray radiation with programmable width and repetition.

The design will be defined on the base of simulated data, taking into account some different and possible geometries for the electron emitting sources (CNT deposited on metallic tips, CNT deposited on array, uniform layers of CNT-containing composite, etc). A key step for the achievement of the expected values will be the optimization of the grid between anode and cathode, acting as focusing electrode.

-Focusing X-Ray system based on polycapillary lens:
Overall, focalization stages based on the use of polycapillary lenses will be designed and tested in order to achieve a further reduction of the spot size and a better uniformity of the intensity across the spot

area. The design will be made by means of "PolyCAD", a CAD tool specifically realized by the RTD performer ENE for X-ray photon tracing in polycapillary optics. In our opinion, the use of polycapillary lens placed in front of a nanotube X-ray source is not only recommendable but it should represent a "must" for future X-Ray system. Micro-polycapillary lenses, now at the 5th generation, may improve the efficiency of the system by increasing the X-ray spot density on the sample while polycapillary semi-lens allows to obtain parallel X-ray spot with uniform intensity on all its area. The target of NANORAY project is to achieve a focal spot size of 0.3 mm with uniform intensity.

-The electronic control system,
which will be able to control in real time the grid voltage in order to maximize both the emitted current and the beam focalization. It will be based on a feedback controlled system, which gauges the emitted current and adjust the value of the grid voltage accordingly, to keep the generated current as high and steady as possible.
-The portable high voltage generator, integrated in the tube's chassis and able to supply the required accelerating voltage, to deliver to the electrons the necessary energy to hit the target and generate the X-ray radiation.

This generator will be designed by Labor with the following requirements:
- Power supply: 220 V/50 Hz power mains and 9V rechargeable battery
- Dimensions less than: 50 mm/50mm/50mm (Width, Length, Depth)
- Voltage: 10-50 kVp
- Current: 0.1-1 mA (depending on the geometry of the triode configuration).

In summary, the main expected results that will be pursued by the project are:
- Portable X-rays generator, based on FE cold cathode, electronically controlled by the management unit in order to optimize its performances.
- Application of the research results obtained on CNTs production (CNTs selectively grown upon suitable nanomaterials) to the realization of a stable and reliable cathode with an industrial process.
- Definition of the process for the realization of the cathode: synthesis of CNTs by means of Microwave plasma assisted chemical vapor deposition (MWPCVD), Hot Filament Chemical Vapor Deposition (HFCVD), Thermal Chemical Vapor Deposition greater than Deployment of a portable power supply, for the generation of the high voltage necessary for the electronic beam acceleration greater than Deployment of the electronic control system of the grid greater than Deployment of the X-Ray focusing system based on polycapillary lenses greater than Electromagnetic simulation for the optimization of both the grid design and the overall arrangement of the cathode - anode - grid in the tube's structure greater than Implementation of the technical solutions for the process steps needed for product finishing, sealing, electrical connections, electrical safety and radiation shielding greater than Complete pre-industrial production process definition (month 18) greater than Realization of prototypes, forced ageing tests and validation in real conditions (month 24) X-rays generation with at least 90 % efficiency greater than Configurations amenable for mass production greater than Product specifications consistent with the application to medical imaging, archaeometric surveys, integrated circuit inspection, security checks and so on.

In order to decrease the focal spot size, increase and make more uniform the X radiation density in the focal spot position, NANORAY tubes will be equipped with additional focalization systems based on the use of ad-hoc designed polycapillary lens based systems. Polycapillary lenses allow the increase the X radiation density in the focal spot position. Moreover, polycapillary semi-lenses allow to obtain quasi parallel X-ray beams from divergent sources or convergent beams from quasi-parallel sources. Focalization stages based on the use of polycapillary lenses will be designed and tested in order to achieve a further reduction of the spot size and a better uniformity of the intensity across the spot area. The design will be made by means of "PolyCAD", a CAD tool specifically realized by the RTD performer ENE, in collaboration with LNF- (National Laboratory of Frascati - Italy's National Institute of Nuclear Physics) for X-ray photon tracing in polycapillary optics. The project target is to achieve a focal spot of 0.3 mm in diameter, with an uniform distribution of X-Ray intensity, that would represent an exceptional value for a commercial product. Overall, it appears reasonable the possibility to decrease such dimension down to 0.1 mm. With respect to the existing commercial X-Ray systems based on thermionic emission, and compared to the state of the art of field emission based devices, NANORAY claims the following innovations:
greater than An electron source with improved characteristics (in particular a much lower energy spread of the emitted electrons, as well as the possibility of working at room temperature without any cooling requirement);
greater than A focal spot decreased down to 0.3 mm or less;
greater than A very low power consumption, thus allowing one to realize a 'true' portable system for measurements on field ;
greater than A very high beam stability;
greater than The possibility to have an output current voltage controllable;
greater than Generation of pulsed X-ray radiation at higher frequencies (1 kHz) with programmable width and repetition rate;
greater than An increased life-time (especially in case of working in pulsed conditions).

In the framework of field-emission based systems, the main forecasted advancements of NANORAY, both in the knowledge and in technology, can be summarized as follows:
- Improve the adhesion of CNT emitters on the substrate, by exploiting different methodology of substrate surface treatments and catalysts preparation, on the basis of already acquired wide experience by some of the RTD;
- Improve the possibility of achieving selected growth of CNT on patterned substrate, by exploiting different 3D morphologies (edge shape, thickness, etc.) of the selected and pre-treated areas where the selected grown is desired;
- Optimize and control the morphology and assembling of CNT emitters, as a function of the specific requirements of the project and on the basis of skills and know-hows of RTD performers involved in the project;
- Use of an innovative focalization methodology, based on the use of polycapillary lenses, that will can easily address specific technological requirements: in particular, in order to evaluate the focusing/homogenizing system parameters, a specific modelling software (PolyCAD) will be used.

This original X-Ray optics CAD has been recently developed by one of the RTD performers (ENE).

Finally, we can notice that further advantages in the application of this new technology can be summarised as follows, when exploring for comparison the traditional techniques for X-rays generation:

- fast response time (exposition to X-rays): 25-50% quicker
- drastic reduction in size and weight of X-ray device: 80% thinner and 75% lighter
- a drastic reduction of maintenance costs, due to the lower costs for the substitution of the cathode with respect to thermionic technology.
- improvement of the focal spot characteristics (highly fine spot), that turns into a better definition of the generated image.

Project Results:

The main scientific and technological results expected to be produced by the project are the following, as described in the DoW.
- CNTs synthesis process
- Focalization system
- Mechanical and electronic design
- NANORAY prototype

The process for substrate preparation and selective growth of CNTs on specific surfaces has been studied and defined by UNITOR and IOFFE, who owns pre-existing knowledge in the field. The focalization system has been designed and implemented by ENE and, up to date, constitutes one of the main innovations introduced by NANORAY. The design of the device, including the dimensioning of each subsystem, the choice of the components and accessories have been produced with the contribution of the RTD performers LABOR and IOFFE. The design guidelines and component selection for the power supply system (light, portable and powered by rechargeable batteries) as well as for the electronic control system for the management of the grid voltage, will be acquired by the Russian company NTI. The prototype of NANORAY device has been realised and assembled with the contribution of all the RTD performers.

We must remember that the main scientific and technological achievements of NANORAY project can be identified to be the following:
- CATHODE MANUFACTURING AND TESTING: The optimisation of the CNTs synthesis process by Tor Vergata University led to the possibility of performing several tests on variously shaped cathodes: among them, planar cathodes, both patterned and unpatterned, and tip cathodes, namely cathodes realised by direct deposition of the Carbon nanotubes on a filament.
- FIELD TESTING WITH REAL APPLICATIONS: Several different tests sessions with NANORAY device have been performed from month 16 on, in order to observe and deduce the sources' performances and the field emission properties of the cathodes, also assessing the tube's best configuration for maximising the X-ray flux and achieving the target focal spot (0.3 mm).
- PERFORMANCE ASSESSMENT AND VALIDATION OF THE TECHNOLOGY: The tests performed with the support of the X-ray Lab at INFN led to the realisation of a deep performance assessment by which the Consortium determined not only the best cathode for the X-rays generation, but also outlined the tube's best configuration for realising a portable device which is also compact and easily adaptable to specific sectors' instruments for X-ray applications.
- FINAL DESIGN OF THE TUBE: The organisation of the laboratory tests on NANORAY tube was planned as the crucial activity for the period; this justified the extensive laboratory sessions performed in the second year of the project.

The external apparatus to be coupled to NANORAY prototype presented, at first, several difficulties in setting up the correct configuration for the voltage generators in order to reduce noise and any potentially occurring short-circuit among the components. In this sense, the experimental setup implied a series of working activities aimed at eliminating any potential problem affecting the X-ray generation and detection.

As foreseen by the project workplan, the final system was tested until the end of the project. However, the SMEs decided that some more efforts should be put in the technology validation phase, consisting in the validation of the cold cathodes' emission and X-rays focalisation, in parallel with the design of the best internal configuration allowing for competitive performances of the tube respect to traditional ones. The deviation from the workplan then led to the realisation of a valve-like tube which is optimised, has been estimated under production costs, and is duly working since it fulfils the requirements on the spot and on the portability features representing the main achievements of the project. No pre-industrial NANORAY device has been realised, but the CAD designs and performances have been assessed, showing that, with a due time for refinement, the device can be industrialised and commercialised in the next future.

The deviation from the workplan then led to the realisation of a valve-like tube which is optimised, has been estimated under production costs, and is duly working since it fulfils the requirements on the spot and on the portability features representing the main achievements of the project.

OPTIMISATION OF THE SYNTHESIS PROCESS

The first step of the work was dedicated to the investigation of the performance of different carbon nanotubes (CNTs) based cathodes. Starting from the consolidated experience in growing CNTs on selected-area substrates, initially a large number of experiments were carried out in order to optimize the best growth conditions of CNTs on differently patterned substrates. At this aim three different apparatuses were tested: the Hot Filament Chemical Vapour Deposition (HF-CVD) chamber, the Micro Wave Plasma Enhanced Chemical Vapour Deposition (MW-PECVD) chamber and the Thermal Chemical Vapour Deposition (T-CVD) chamber. The CNTs synthesis procedure on shaped cathodes were preliminarily investigated by performing CNT growth on substrates with different kinds of catalyst patterns. Highly doped silicon slides were used as substrates for CNTs deposition. We can observe the homogeneity and density of the carbon nanotubes deposits which are both characterized by a good vertical alignment of the tubes with respect to the substrate. Additional investigations about synthesis experimental conditions were performed aiming to produce homogeneous and well-adherent CNTs deposits on the micrometric patterns.

The optimized process for the CNTs' synthesis can be hereby reassumed: we have optimized the CNTs' growth on a Silicon substrate, but we have found that this cannot represent the most suitable cathode for a future automation of the process, which is one of the desired features for NANORAY system. Even if the study on the deposition and the growth of nanotubes on a substrate stands at a research level still, we believe that CNTs deposition on tip-cathodes stands as the most suitable technique realized for NANORAY project, which is, furthermore, the easiest method to be made an automatic and low-cost process.

It would be necessary, in alternative, to research and develop other deposition techniques, using more stable resins to both vacuum and heat, and to further develop the attachment operation of CNTs to the filaments; this is obviously not a simple task, which exactly goes in the direction of the actual research performed by the scientific community on CNTs. From an experimental point of view, and especially in the case of a device that will be brought to the market holding well-determined technical features, it is far more crucial to evaluate the tube's emitted current respect to the current density.

However, since the question of measuring densities of current values still proves ambiguous, we can notice that there exists a very famous article - among the field emission researching community- that clearly explains which is the basic error in estimating the current density that may occur to those performing the research. To the aim of NANORAY project, thus, we have agreed on taking into account the observed current values, especially when using tip-cathodes, whose emitting surface value would be of difficult, and surely imprecise, estimation.

FIELD EMISSION

The field emission functional properties were studied by means of a custom-designed apparatus. The characterization set-up is constituted of a plan to plan geometry in which the anode-cathode distance can be changed during the measurements. All the cathodes have been investigated in a diode configuration and it has been demonstrated that the reduction of the pattern dimensions and the optimization of the growth conditions allow to increase the maximum emitted current. Starting from these preliminary results, we have performed field emission measurements on other patterned CNTs cathodes, in particular by using square lithography of the substrate in which the dimensions and number of the emission areas has been varied.

Usually, the parameter of merit for device applications is the integral current density, i. e., the total current emitted divided by the entire cathode area, but the integral current density depends upon cathode area. As a consequence, a very high current density is obtained only from a very small cathode area. At the same time, the potential for high emission currents has always been an attractive feature of field emitters, although the evaluation of the emission sites is still a widely discussed problem. For this reason, with the aim of obtaining realistic experimental parameters useful for the application of the CNTs cathodes as electron sources in the X-ray tube prototype, for all the samples we have reported and discussed the behaviour of current intensity respect to the applied voltage. The second step of the work was focused on the study and characterizations of cathodes made of commercial CNTs. The sample was prepared by drop casting a dispersion of the tubes in CH3OH. The deposit is characterized by entangled bundles of CNTs horizontally aligned on the substrate.

The field emission behaviour of the pristine deposit is evidenced. It showed a good relation between the applied voltage and the current recorded as evaluated in the Fowler-Nordheim theory. Currently we are investigating protocols to functionalize the commercial CNTs by decorating them with nanometric crystals of diamond. For what concerns the cathodes' life cycle it is necessary to distinguish two cases:
1) CNT-based cathodes (both Single-wall and Multi-wall nanotubes) which have been fully produced in our laboratories, and
2) tip-cathodes which have been assembled in laboratory using Nanocyl's Carbon nanotubes.

For the first type of cathodes, it is technically very difficult to provide an estimation of their duration in time and resistance to stress conditions, for the synthesis process proves different at any cathode production; since this process is not performed at an industrial scale and allows no reproducibility of the experimental conditions under which the cathode is realized, each single cathode holds different life cycles and emission properties. Furthermore, the experimental conditions for the activation of the deposited CNTs are extremely different in voltage and vacuum respect to the ones setup for their bare production in laboratory. For safety purposes, the tests on these cathodes will be performed for 6-8 hours continuously in order to obtain the required measurements of current and X-ray flux. However (as will be evident from D9 reporting the tests details), different tests sessions on the same cathodes have been performed for more than a month with periodic on-off modality, and this grants that the laboratory produced cathodes can sustain the vacuum and thermal conditions inside the tube for at least 1 month without damage.

In the second case - cathodes realized by Nanocyl - the same estimation must be related to the CNTs' emission properties and to the technique used for their synthesis, which strongly affects the quality of performances. When tested in NANORAY tube, these cathodes have been observed to behave better than the previous ones produced in lab for current stability conditions and number of emitted X-rays. We can then estimate that these cathodes can resist twice the time of the previous ones without appreciable modifications of their performances in field emission.

CATHODE MANUFACTURING AND TESTING

The first step of the work was dedicated to the investigation of the performance of different carbon nanotubes (CNTs) based cathodes. Starting from the consolidated experience in growing CNTs on selected-area substrates, initially a large number of experiments were carried out in order to optimize the best growth conditions of CNTs on differently patterned substrates. At this aim three different apparatuses were tested: the Hot Filament Chemical Vapour Deposition (HF-CVD) chamber, the Micro Wave Plasma Enhanced Chemical Vapour Deposition (MW-PECVD) chamber and the Thermal Chemical Vapour Deposition (T-CVD) chamber. The CNTs synthesis procedure on shaped cathodes were preliminarily investigated by performing CNT growth on substrates with different kinds of catalyst patterns. Highly doped silicon slides were used as substrates for CNTs deposition.

CNTs deposition by combined CVD techniques was also carried out on triode configuration substrate: in this case we observed CNTs homogeneously cover the micrometric patterns. All deposits are characterized by dense and well packed CNTs bundles. In particular in the case of nanometric patterns, the CNTs growth takes place in all the catalyst spots. Moreover the deposits are characterized by a good alignment of the nanotubes. The optimisation of the CNTs synthesis process by Tor Vergata University led to the possibility of performing several tests on variously shaped cathodes: among them, planar cathodes, both patterned and unpatterned, and tip cathodes, namely cathodes realised by direct deposition of the Carbon nanotubes on a filament.

MECHANICAL SPECIFICATIONS

The main objective of this task was to provide a framework for discussing the requirements of the different materials to be used in the mechanical design of the NANORAY prototype.

The main topics discussed are herewith summarized:
- Normative framework
- X-Ray shielding: High-energy-electromagnetic radiation poses a great health hazard to those who must work around scanners, test and measurement instruments, fluoroscopes and X-ray machines, collimators, and other equipment. The recommended quantity for shielding design calculations for x rays is air kerma (K), defined as the sum of the initial kinetic energies of all the charged particles liberated by uncharged particles per unit mass of air, measured at a point in air (ICRU, 1998). The unit of air kenna isjoule per kilogram, with the special name gray (Gy). However, many radiation instruments in Europe are currently designed and calibrated to measure the quantity exposure (ICRU1998), using the previous special name roentgen (R). The recommended radiation protection quantity for the limitation of exposure to people from sources of ionizing radiation is effective dose (E), defined as the sum of the weighted equivalent doses to specific organs or tissues (i.e., each equivalent dose is weighted by the corresponding tissue weighting factor for the organ or tissue wt (NCRP, 1993).
- Vacuum compatibility: Materials used inside a vacuum process have to comply with the following technical requirements:
- Mechanical Properties
- The material must be capable of being machined and fabricated.

- It must have adequate strength at maximum and minimum temperatures, and must retain properties over the expected temperature range.
- Thermal Properties
- The material's vapour pressure must remain low at the highest temperature.
- Thermal expansion of adjacent materials must be taken into account, especially at joints.
- Gas Loading
- Materials must not be porous
- Materials must be free of cracks to avoid entrapment of cleaning solvents which can become a source of virtual leaks.
- Surface and bulk desorption rates must be acceptable at extremes of temperature and radiation.
- High Voltage insulation

For each of these topics a brief overview of requirements, advantages and drawbacks, is presented in the delivered documents. Further, following these general guidelines, several materials have been chosen for both metal and non-metal components.

ELECTRONICS SPECIFICATIONS
This task has been devoted to the identification of the preliminary configuration of the power supply unit and of the electronic control system of the grid, able to real time adjust the extracting voltage in order to keep at a constant level the electronic flux from the cathode to the transmission target.

The initial specifications from the power supply unit are:
- Anode - Cathode Voltage (in order to accelerate electrons emitted by CNTs): 30 kV.
- It must be integrated in the system's chassis.
- It must be powered both by standard 9V rechargeable batteries and by 220/50 Hz power mains.

For the Alpha prototype, an external power supply unit will be used, while the final prototype will have it integrated in the tube casing. As for the electronic control system design, the preliminary sketch of the architecture has been proposed (D5), amenable to adjustments following to the results of the first battery of tests with the Alpha Prototype. In the following we describe how the design of the power supply units has been performed according to the tests on the alpha prototype mainly performed during the second year of NANORAY.

POWER SUPPLY SYSTEM DESIGN

Since NANORAY system is a portable device, the power supply unit is based on a battery. Thanks to a preliminary session of experimental tests, it was possible to define some details about the power supply unit of NANORAY system. Just to summarize some of specifications about the tube, the figure below shows the basic concept of the preliminary x-ray tube, based on the triode configuration: it has a cathode composed by carbon nanotubes, electron extraction metal grid, and anode made of copper. Empirical rules told us that working in an efficient way at 8keV needs a 30KV differential voltage between cathode and anode. X-ray generation mainly depends on the spot size and intensity of the electron beam that impact on active anode, and so is also directly correlated with the power supply unit: the electronic current that flows in the anode must be sustained by the DC/HVDC converter. So the maximum intensity of the electron current flowing inside the tube is directly correlated to the current characteristics of the DC-HVDC converter unit. This is the reason because the final choice about which is the correct DC/HVDC converter for the project depends on the preliminary tests. From preliminary experimental tests (D9), we know that is possible to obtain a good x-ray emission with a value of cathode current around 300uA for planar cathodes (and lower for tip cathodes), with some unstable peaks around 1mA: so the choice should go to a DC/DC converter that has at least 30KV × 300uA = 9W. We observed also good x-ray emission at lower voltages. So, choosing a 10W DC/HVDC converter we can have a power supply system (correctly) oversized. Moreover, preliminary tests demonstrated that was necessary to have more than an electric lens inside the x-ray tube, in order to focalise the electron bean over the anode and so have more efficient x-ray emission.

For this reason, the final architecture of the tube has four DC-HVDC converters, able to give to the x-ray tube the necessary voltages to work properly. In the first version of the tube the cathode voltage is zero, and the anode is at positive voltage less than 30kV, and is also isolated from the metallic enclosure. Grid voltages and focalisers voltages are in the middle between cathode and anode. The new and final architecture maintains the same absolute voltage differences, changing the point connected to ground. The new ground point is the anode, and the cathode is connected to a very high negative voltage source, in a way that allows to have the same voltage difference of the first tube. This choice allows to start the market phase with an architecture that respect laws about the work safety, since in this case the anode could be placed in contact with the metallic enclosure placed at zero potential, and the metallic enclosure is always in contact with a human being. Probably the best choice is to cover the x-ray tube with a plastic, or make the tube with glass lead doped and then cover it with a plastic. What changes from an electronic point of view in the power supply unit is the model of DC/HVDC: the EMCO 4200N is suitable for the "complementary" architecture, and is able to give -20kV from the same battery input of 24V and it can be used to obtain the voltages for cathode, grid and focalisers. In an experimental scenario in which the cathode is at zero, the grid is at 1600V, the first focaliser at 960V, the second focaliser is at 1300V, and the anode at 10KV, the new "complementary" configuration should have: Cathode at -10kV, Grid at -8.4kV, focaliser 1 at -9.04kV, focaliser 2 at -8.7kV and anode at 0V. In D8 we report the design of the final x-ray tube, obtained summarizing all the experience done in last two years and the collaboration between partners. It's a very integrated tube respect to the alpha prototype: it is just long 7cm and it has 3cm diameter. Screenshots show the tube working with planar CNTs cathodes. The idea is to have all parts (cathode, focalisers, and anode) integrated in a small x-ray tube structure similar to an electric valve.

The internal structure of the small valve is the result of the best experimental configuration found during the tests. The dimensions of the focaliser, the distances between them, the distance between the cathode and the grid, and also between the anode and the electric lens, are factors found thanks to the experimental tests done at INFN using the preliminary prototype. Chosen materials are stainless steel for contacts, glass for supports and enclosure, copper for anode, and of course carbon nanotubes for the cathode.

ELECTRONIC CONTROL SYSTEM DESIGN

The working principle of the control unit is a typical application of negative feedback to control the stability of x-rays emission, acting on the grid voltage. During the impact of electrons on the anode material, most of the electron energy is lost as heat and only a small part (around 1%) produces x-ray radiation. The smaller the charge on the grid, the smaller the current to the anode, and so the x-ray intensity produced. The anode current is read by a microammeter, and then elaborated by the MCU using an isolated analog digital converter. The small keyboard and the display represent the user interface and show the status of the portable x-ray tube, for i.e. measured anode current and chosen grid voltage. Grid voltage is limited by the electric field we want to apply in order to extract electrons from the nanotubes. Since nanotubes stretches when a very strong electric field is applied, distance between cathode and grid must be limited and also grid voltage must be limited to 2kV otherwise there could be a short circuit between them. This is one of the most important reasons that convinced us to design a preliminary x-ray tube: this was very useful to adjust the best value of electric field applied to nanotubes, and this means also the best values of grid voltage and grid-nanotubes distance. NANORAY prototype was realized using commercial components: we have presented a summary of the main electronics components involved in this design, with some information about their consumption (that is important since the final product will be a portable product) and the dimensions. NANORAY Control unit is a very compact board, without the addon board dedicated to user interface. It's designed to work with more complex x-ray tubes, with more than one voltage and current to monitor.

Experimental tests on the x-ray tube (see D4) have defined some details about the feedback software routine that is going to be implemented inside the microcontroller. For example how much the electron emission by the CNT cathodes remains stable when the voltage sources are fixed and stabilized. User interface buttons and display are external because they are placed in the front panel of the instrument, and they are connected to the board using the connectors of the printed circuit board. Microcontroller unit is programmed through an USB port, that is a separate small module.

FOCALISATION SYSTEM

NANORAY tube is equipped with additional focalization systems, based on the use of ad-hoc designed polycapillary optics device, in order to decrease the focal spot size and to increase the X radiation density in the focal spot position. Focalization stages based on the use of polycapillary lenses will be designed and tested to achieve a further spot size reduction and a better intensity uniformity across the spot area. Interaction of a cold cathode tube (NANORAY tube), portable in situ, and the high brightness micro spot X-ray beams, obtained through polycapillary optics, can generate a portable instrument of a great interest in the industrial, cultural heritage, medicine and multidisciplinary fields. The optics that has been used in NANORAY tests for further reducing the X-ray beams' spot was a commercially available set of 4th generation lenses produced by Unisantis company (more information at: http://www.unisantis.com/index.php). Unisantis Europe GmbH is a designer, developer and manufacturer company for proprietary Kumakhov X-ray polycapillary optics (X-ray lenses). Polycapillary optics control X-ray and neutron beams and are based upon the principle of multiple external reflections. This field of optics was conceived by

Professor M.A. Kumakhov in the early 1980's. This polycapillary X-ray focusing and collimating optics:
- Shows high flux density on the sample under investigation that provides an increase of 2-3 orders of magnitude over traditional optics
- Eliminates the need for monochromators, collimators and slits
- Requires low power X-ray sources making them compact and portable
- Analysis time comes to be significantly reduced due to beam collimating efficiency
- Increased and diversified analysis range - parallel, convergent and divergent high-intensive beams are available for multiple X-ray application and tasks

When providing the results of Polycad simulations, the focal spot dimensions obtained with poly-capillary lenses need to be further reduced to reach the requested (and desirable) values for real X-ray tubes applications, that is accurately indicated and justified in the project's results. In this sense, the project target is to achieve a focal spot of 0.3 mm in diameter, with an uniform distribution of X-Ray intensity, that would represent an exceptional value for a commercial product. Overall, it appears reasonable the possibility to decrease such dimension down to 0.1 mm. This is the reason why we have considered a further focalizing system, internal to the tube, necessary. What we intend to obtain with this additional focalization system is the reduction of the electron beam spot reaching the copper surface of the anode. This will, as a consequence, result in a more 'dot-like' feature of the emitted X-ray beam.

As indicated in D7 (electromagnetic analysis), all the technical information for the design of the final tube derived from an accurate discussion among the partners, that led to the focalization design and implementation, documented with CAD designs. Starting from theoretical considerations, and supported by the following simulations carried out by IOFFE, we concluded that an internal focalization system was needed for NANORAY. The simulations about the focusing potential demonstrate the possibility for the system to reach electron focalization with one focusing electrode. The conclusion of the technical discussions that followed this phase demonstrate that the maximum focalizing efficiency can be obtained by inserting two cylindrical focalizers between grid and anode.

We have finally chosen for NANORAY tube some optimised parameters, like the internal distances between the cathode and the first focalizer, between the first and the second focalizer, and between the last and the anode surface. We can state that the scientific objectives expected for the WP have been fully achieved by means of an iterative procedure, for which the experimental tests suggested the suitable process for CNTs synthesis and field emission enhancement, and the design of the electrodes was accordingly performed, by subsequent integrations. The focalisation system, in agreement with the results of the electromagnetic analysis, has been upgraded until the end of the project.

TEST PROTOCOLS

Tests have been performed by University of Rome 'La Sapienza', at LNF-INFN. In the following, we point out a tentative test protocol, in order to foster the discussion amongst the partners and therefore to define the best experimental procedures to follow to optimize all the parameters. The intended optimisation phase can be divided into two steps.
STEP 1: Optimisation of the emission characteristics of the cathode

This phase will include the tests on the different types on cathodes, ranging from the un-patterned to the tip ones. Purpose of this set of measurements is to provide a final representation of the current absorbed by the grid with respect to the other interested parameters, namely the cathode-grid distance and the voltage applied to it. This result can be achieved in the following way:
- The anode is constantly kept at zero voltage for this set of measures, so that X-rays are not generated during the process;
- For each of the provided cathodes, the grid current is investigated as a function of the distance from the cathode itself, and of the grid voltage (varying in the range 0-2 kV). This set of measurements will be plotted on a graph;
- The most suitable operating region for the generation of X-rays will be considered at this point by observing the behaviour of the grid current respect to the generated electric field.

STEP2: X-rays measurements
Once this first step has been carried out, and the best 'operating region' has been evaluated from the results, the anode can be given a variable tension. So, reassuming, for every different cathode shape, for every 'work region' individuated, thus having fixed the distance d and the voltage of the grid, the emission of X-rays is observed and studied.

EM ANALYSIS -
The EM analysis performed in WP4 presents:
1) the simulation hypotheses on the CNTs cathodes structure and field emission, on the grid shape and mesh, and on the anode electrode
2) the experimental parameters relative to the internal structure of the tube, with the introduction of a focusing system aiming at reducing the out coming x-ray beam's focal spot
3) the values of these parameters which are necessary for the achievement of the optimal performance in terms of current and x-ray emission intensity

The following issues were assessed with the support of Ioffe Institute, where the electromagnetic simulations have been performed, according to the steps :
1. to analyze calculations and experimental data on field electron emission from different carbon nanostructures,
2. to analyze of applicability of carbon nanotubes for cold cathode in X-ray tube, and make the suggestions on the technical specifications for the prospective tube,
3. to study experimental features at characterization of field electron emissions from carbon nanostructures,
4. to suggest an optimal structure of focalizing electrode system of X-ray tube based on carbon nanotubes (CNT) and calculate the optimal working potentials for the system

In the design of a triode-type X-ray source like the one foreseen for NANORAY tube, the challenge is to ensure that fine beam focus and efficient electron emission can be simultaneously obtained. This led to an accurate analysis of the tube's geometrical parameters, such as the electrodes' relative distances and the grid mesh design must be duly optimized. The goal of the calculations for the single focusing electrode configuration was to obtain the preliminary estimations for focalizing the electron beam with a flat cold cathode. The steps of the procedure included:

-Determination of the distribution of the potential between grid and anode
-Determination of the distribution of the electric field under grid using EH for lacing
-Determination of the electron tracks under and on the top surface of the grid
-Determination of the electron tracks between the grid and anode using the speed v

- Single focusing electrode configuration

Let us start from the description of the field simulations for the one-electrode focalizing system, placed within NANORAY tube.
The calculated electron tracks in the space adjacent to the grid and cathode demonstrate the very small value of the radial speed component of the overall speed of the electrons beyond the grid. So all the trajectories could be regarded as normal to the plate of the grid. This gives the good possibility to obtain the small focalized spot on the surface of the cathode.

- Double focusing electrode configuration

In order to increase the value of the anode current we have to enlarge the square of the flat cathode and to accordingly increase the radius R2 of the tube. The increase of the radius has required enhancing the focalizing system to keep the radius of focal spot obtained in the previous configuration. According to the calculation results, the potential distribution in the grid cell is similar to the case of single focusing electrode system. Two sequential cylindrical electrodes form two good independent electrostatic lenses. The results of calculations of the electron tracks demonstrate the good efficiency of not just a single focusing electrode, but that of a double electrode focalizing system. The fine structure of the focal spot allows to determine the criteria for the optimization of the values of voltages applied to the focalizing electrodes for the smallest focal spot at different anode potentials.

The results of the calculations of the optimal focusing potentials for the tube with flat cathode and double electrode focalizing system allow to make the following conclusions:

- The electrode configuration of the prototype with flat CNT cathode allows to obtain the minimal focalization spot up to 0.1 mm at 10kV on anode and up to 0.24 mm at 30 kV
- The increase of the grid potential makes the spot smaller mostly at high anode voltages
- The increase of the grid potential up to 2.3 kV can allow to obtain the 0.2 mm spot at 30 kV of anode voltage

In order to reduce the cathode current losses and to obtain the maximal anode current we replaced the grid with cylindrical cells for the wire grid with rectangular cells. For this reason we were obliged to reject the simplifications concerned to the cylindrical geometry and turned to apply the rectangular coordinate system for the calculations. Applying the idea of the additional acceleration of the electrons in the tube with tip cathode we have determined the electrode configurations and working potentials for the structures of x-ray tubes with tip field emission CNT cathodes with expected critical parameters (focalizing spot size and current efficiency). The main distinguishing feature of the configuration examined above is the presence of the accelerating grid and the intermediate focalization cylinder 3 between the main and accelerating grid. The additional energy of the electrons after the acceleration grid allows avoiding the collisions of the electrons with the focalizing cylinders and obtaining a better focalization. The anode current of this configuration is 96% (due to the collisions only with grid wires) of the cathode current and the minimal available size of focalizing spot is about 0.5 mm.

The further investigation of the potential distribution in the electrode configuration with the described improvements has shown that its parameters could be enhanced more by the reducing the number of the electrodes and simplifying the configuration. We report below the schematics of such configuration with calculated values for working potentials almost optimal for the best focalization. It differs from the previous configuration by the presence of only one main focalizing cylinder and by the 7 mm smaller overall length. The focalized spot on the anode could be decreased smaller than 0.25 mm.

The results of simulations on the structure with double focusing electrode configuration allow us to make the following conclusions:
1. A flat cathode is more preferable for x-tubes due to simple focalizing and high current level. The optimal values of the potentials on the focalization system at different anode voltages have been calculated
2. Using the tip cathode is less preferable due to the spreading of the electron trajectories under the grid and high current density on the cathode

However:
1. Focalizing the electron trajectories in the tube with tip cathode is possible using an additional element (cathode ring). The size of the ring and the corresponding focalization potentials have been determined from calculations
2. The focalization and the current efficiency can be further enhanced by applying the accelerating potential in the space between the grid and the focalization system.

DESIGN OF THE TUBE
According to the first tests performed and to the simulations carried out, also in literature articles, it is experimentally accepted that to have a consistent and reliable X-ray flux it is necessary to have at least 30kV of acceleration between cathode and anode.

The chosen configuration, as pointed out in D1 - NANORAY Data Sheet, is the transmission target, in which the target consists of two different layers of material. Most of these transmission targets are made from a thick layer of backing material, such as Beryllium or Aluminium, with a low density and a low atomic number and weight.

The purpose of the backing material is to close the tube and maintain a vacuum. It also forms the backing, which provides mechanical strength for the target layer, in which x-rays are generated. Target layers usually consist of a thin, 5-µm layer of metal such as Tungsten, Molybdenum or Copper, with a high density and high atomic number and weight. The target layer is sputtered onto the backing layer.

With transmission tubes, as opposed to directional tubes from which the X-ray beam is issued at a 30, 60 or 90 degree angle, the design features forward-beam geometry. Electrons enter the back of the transmission target, and x-rays radiate from the front. NANORAY will be designed for very close anode-to-sample coupling for compact portable XRF instruments.

Since the first version of the tube follows also the "transmission" way, the x-rays are generated by copper and go out from the tube following the direction identified by the tube axis. Summarizing the main characteristics of the preliminary tube until now:
- Triode architecture: cathode, modulation grid, anode
- Cathode: planar and linear carbon nanotubes
- Grid: metal net to modulate the electron current
- Anode: copper
- Output window: placed in line to cathode-grid-anode

The identified structure of the preliminary tube consists in a six flanges stainless steel cross, with the following components included:
1) A cathode holder flange with UHV micrometer drive (50 mm length)
2) A flange for the connection to the vacuum pump (DN40 CF-KF adaptor)
3) A flange for visual inspection
4) A flange for High Voltage connection cable (anode)
5) A flange with 3 connectors for cathode and grid voltage setting
6) A flange for the output of X rays, with a beryllium disc window

We hereby present the core of the beta prototype, the miniaturized x-ray tube. In our design it measures only 7 cm in length and 3 cm of diameter, very much smaller than the previous. The internal structure is analogue to that of the alpha prototype, and it re-organizes all the active elements in a special glass valve that acts as x-ray tube for the NANORAY device. In this configuration there is no possibility to change distances by the user: the user can change only the electric parameters via touch display of the entire device. Distances and configuration derives directly from the experimental tests and experience made during this project. As reported in Deliverable 7 (Electromagnetic Analysis), using a tip-shaped cathode which provides the best focalising effect on the X-ray beams' focal spot, we need a redefinition of the electrodes' disposition within the tube, also foreseeing a third accelerating grid.

We report, in D8 and in the attached pdf., the designs of all the components of the valve-like structure that will be easily adapted to different applications and instrumentation. This easy-to-handle and independent part of X-ray-based instruments will allow for the substitution of the cathodes, in case the CNTs deposited on them were no more able to emit electrons. We report, in the figure below, the scheme already discussed in D7 for comparison with the CAD images of the tube to be commercialised. Further comparisons with the already existing micro-focus X-ray tubes' features will be discussed in detail in D11, which will also describe how this configuration of NANORAY tube will be adapted for realising a commercial tool.

The parallel activity of electromagnetic simulations performed at IOFFE Institute proved of great support in understanding the dynamics of the electrons generated by the cold cathode source; in this way we could carry out a continuous upgrade of the tube's structure which resulted in the final revised prototype showing the expected performances in terms of field emission properties.

As already introduced, there was a deviation from the project workplan in the mechanical realisation of the final (beta) prototype; since we considered as a far more crucial task that of validating the field emission properties of nanotubes, we concentrated on the laboratory tests instead of developing a final tube including a non complete technology, in order to have a result which could be real and tangible, since this phase proved to need more time that the expected. The performances of the tube clearly show that all the scientific objectives of the project have been achieved, and this is the solid base on which a concrete exploitation of the device will be able to occur after the end of the project itself.

TESTS ON THE PROTOTYPE

Several different tests sessions with NANORAY device have been performed from month 16 on, in order to observe and deduce the sources' performances and the field emission properties of the cathodes, also assessing the tube's best configuration for maximising the X-ray flux and achieving the target focal spot (0.3 mm). In order to verify the effective functioning of NANORAY system, aware of the intrinsic experimental difficulty in the CCD sensors positioning, some Polaroid plates were placed on the output flange, in correspondence of the Berillium window of the system itself. The test consisted of 3 trials.

- The first trial has been carried out to verify the X-rays system emission.
- Since the X-rays generation proved positive during this test, a second trial has been made with defined and monitored experimental conditions
- The third trial was obtained by applying a zero grid voltage to the tube, to avoid the arising of a difference in voltage between grid and cathode: this test proved that the X-rays emission was exclusively due to the field emission phenomenon that characterizes the CNTs deposited on the cathode.

According to the simulations carried out at IOFFE Institute the optimal values for the two internal cylinders constituting the focalizing system can be considered to be 960 Volts and 1300 Volts, for the first and the second cylinder respectively. While, in absence of focalizing lenses the number of counted photons was about 100, the operation of turning on the lenses gave such a high counting number that it was not even detectable, because it exceeded the scintillator's linearity region. What is important to remark here is the phenomenon that was observed under the experimental conditions of a tip-like cathode and focalizing lenses turned on, namely the relevant increase in the cathode's current which was found to be about 3 times higher respect to the initial value, when the focalisers were turned off and the current itself had a very stable behavior (few uA to about 10 mA).

They are:
- distance planar CNTs cathode - grid: about 100um
- grid - focaliser 1: 10mm
- focaliser 1 - focaliser 2: 2mm
- focaliser 2 - anode: 20mm
- length focalisers: 10mm
- diameter cathode and anode: 20mm

Materials chosen are copper over beryllium for the anode, stainless steel for focalisers, grid and electric cables, glass for internal supports, and of course, carbon nanotubes for the cathode.

JJ Electronics, leader in production of audio valve has estimated that the price in production (only NANORAY tube) should be around 40 EUROS each one. This is good since the cathode needs to be changed after a long, but limited, period of work time: the tube will work as a consumable.

The last step of the session was the use of the knife-edge technique, in order to obtain an indirect evaluation of the focal spot dimensions. This technique is intended for determining the profile of the spatial intensity distribution of radiation beams. The problem of measuring the intensity profile in such beams can be overcome, for example, using an X-ray film as a recorder; however, the film has a number of obvious disadvantages, such as the strong dependence of the results on the film's "age", the development time and temperature. These hard-to-control factors can cause one to make a number of extra operations, like i.e. fixing several images on a single film, performing control exposures, etc.

Moreover, high-resolution x-ray films are not frequently commercially produced. Such photographic materials have a low sensitivity, and their processing takes a long time.

In the world practice, a modern and fast recording technique is the so-called "x-ray knife method". It consists in the following procedure: an opaque screen for the measured radiation (that is, opaque to x-rays) is gradually introduced into the beam under study. The detector thus records the portion of radiation scattered by this screen.

The derivative of the measured scattering intensity curve yields a one-dimensional beam profile. In practice, we performed a counting of the outcoming photons in relation to how much the knife is covering the beam's exit cone. Ideally, if we had a point-like source, and performing this operation right on the source, we would get a step function progressively diverging and losing its settlement on the edges.

(Alternatively, if our source does not have a point-like shape, like it is in a real case, then what we have is a cone gasket). Knowing the aperture of the cone from the different measurements, and also keeping in mind the tube's parameters, we made a projection of the cone on the anode, thus obtaining the dimension of the anodic spot.

We applied the parameters reported above and used an Aluminium sheet as a filter - and a lead plate for the X-rays attenuation - so as to obtain the counting of X photons respect to the plate's height (performed 2 times).

The distance between the outer flange where photons come out and the lead plate being acknowledged, we performed the fit of the acquired data, and by merely geometrical considerations we obtained the value of the angle subtending the outcoming cone, which resulted to be about 9°.

This gives us a spot diameter of about 5.2 mm. By simple geometrical considerations we get the value of the angle subtended to the exit cone, from which the estimation of the spot on the source, which is found to be of about 3.28mm. This is the calculated spot of the electrons impacting on the anode; now, the final step that we can perform to reduce the beam's spot is to add the polycapillary optics to the prototype. The focal spot, in this situation, was found to be 300 µm.

As a conclusion to the validation phase of NANORAY device, we hereby report the observed performances of the tube, that will be a useful set of information when we will outline the future exploitation of the device and define an appropriate business plan for it.

What we have observed in these tests sessions, and especially in the case of our last configuration using tip-cathodes, is the following:

- Electron beam focal spot size on the anode (estimated): 3.0 mm
- X-ray beam diameter (with polycapillary optics) ~ 300um
- Operating Voltage: anode 12kV (tunable), focalisers around 1kV (tunable), extraction grid around 1.5kV (tunable), cathode @ ground
- Anodic Current: 0.1 - 1.0 mA, best results with 100uA
- Power consumption: max 15 W of the final tube prototype
- Cooling: NOT REQUIRED
- Length estimated: less than7 cm for the final tube prototype
- Diameter estimated: less than3 cm for the final tube prototype
- Weight: less than1kg for the final tube prototype

The future commercialisation of the tube will be therefore based on these technical features and will take into account some crucial quantities listed below.
1) X-Ray flux: the actual photon counting obtained with a scintillator counter gives values of about 5000 ph/s, with 32 Aluminium plates (total thickness of 1 mm) and considering the surface of the external sensor
2) Focus distance: 21mm considering the anode, on which the electron beams are focalised, and the plate which is placed outside the tube.
3) Focus size: we have estimated the focus size on the anode in 3.0 mm, with reverse measurements (knife method)
4) Time/position stability: using Nanocyl carbon nanotubes we obtained a high stability in current for each experimental test (duration 6-8 hours)
5) Photon energy: copper 8keV
6) Life time: from commercially available nanotubes it seems that the cathodes built up with CNTs can work properly for about 1 month continuously without being damaged and stop their emission.

ASSESSMENT OF RESULTS

The tests performed with the support of the X-ray Lab at INFN led to the realisation of a deep performance assessment by which the Consortium determined not only the best cathode for the X-rays generation, but also outlined the tube's best configuration for realising a portable device which is also compact and easily adaptable to specific sectors' instruments for X-ray applications.

NANORAY market study is intended to provide a general overview of the market segments which are directly related to the X-ray application fields and an introduction to the already commercialized traditional X-ray tubes, in order to better assess the market potential of NANORAY system and related products; this report consists of:
- An overview of the market potential of the achieved project objectives:
1) Cold cathodes based on CNTs for field emission
2) X-ray tube for specific spectroscopy applications
- A study of the demand and the segmentation of the market
As accurately described in the Technical Annex of NANORAY, the project's objective is to develop a X-ray tube which makes use of a carbon nanotube-based cold cathode (CNT): this could represent a significant advance in X-ray technology development and could lead to portable and miniaturized X-ray sources for both medical and industrial applications. Some modifications have been made to the initially delivered document; in

particular, we have added some information regarding what, during the validation phase of NANORAY technology, seemed to be the closest sectors in which to exploit the project's technological results.

In this sense, we have thought about XRF technique (X-ray Fluorescence), here described and detailed in the optics of becoming the exploitation sector of NANORAY tube. X-Ray fluorescence is used in a wide range of applications, including:
-research in igneous, sedimentary, and metamorphic petrology
-soil surveys
-mining (e.g., measuring the grade of ore)
-cement production
-ceramic and glass manufacturing
-metallurgy (e.g., quality control)
-environmental studies (e.g., analyses of particulate matter on air filters)
-petroleum industry (e.g., sulfur content of crude oils and petroleum products)
-field analysis in geological and environmental studies (using portable, hand-held XRF spectrometers)

The direct observation of NANORAY tube performances during the test sessions reported in D9 (field tests and performance assessment) showed how the applications on the market that NANORAY could reasonably support with its innovative principles are based on the combination of AFM (atomic force microscopy) and Micro X-ray fluorescence spectrometers.

This combined system could, in fact, enhance AFM capability with an additional elemental analysis. The system would consist of the following set of instruments:

- Small size- High Voltage Power supply with X-ray tube
- Dead time and automatic signal processing digital multichannel processor
- Silicon Drift Detector (SDD) with resolution less than 140 eV

We have already described - when talking about XRF techniques and commercially available products - the system realized by NT-MDT. After long evaluation, within the Consortium, about the potential of NANORAY system, and in accordance with the R&D Department of this company supporting NANORAY developments, we have agreed on considering the CNT-based cathodes tested during the project as the perfect sources for X-ray emission in systems like the ones implemented at NT-MDT on an industrial scale. We will thus develop an Exploitation plan (D11) leading to the introduction of this novel technology on the market, with a deep study of XRF techniques and already commercialized products in the spectroscopy and materials analysis sector.

In summary, the Consortium will synergically work with the Research and Development Department at NTMDT in order to integrate NANORAY solution within the already available tubes. A detailed exploitation plan will be developed with a commercial strategy in mind, and will be illustrated in D11.

FINAL DESIGN AND RE-ENGINEERING
The organisation of the laboratory tests on NANORAY tube was planned as the crucial activity for the period; this justified the extensive laboratory sessions performed in the second year of the project.

The external apparatus to be coupled to NANORAY prototype presented, at first, several difficulties in setting up the correct configuration for the voltage generators in order to reduce noise and any potentially occurring short-circuit among the components. In this sense, the experimental setup implied a series of working activities aimed at eliminating any potential problem affecting the X-ray generation and detection. As foreseen by the project workplan, the final system was tested until the end of the project.

However, the RTD performers' efforts have been put in the tests sessions that provided the best internal dimensions and parameters for the design of the final tube. The deviation from the workplan then led to the realisation of a valve-like tube which is optimised, has been estimated under production costs, and is duly working since it fulfils the requirements on the spot and on the portability features representing the main achievements of the project. No pre-industrial NANORAY device has been realised, but the CAD designs and performances have been assessed and are ready to be industrialised, as described in the mini-Business Plan developed in Deliverable 10 (Market Study).

The activities performed by the consortium for dissemination and exploitation purposes will be reported in the following.

MARKET STUDY

In spite of being traditional equipment, X-ray instruments are finding renewed demand from several commercial and industrial application. Some of the current market features are:
- Market is mature with stable and steady growth rate;
- The expected recovery of the semiconductor industry will probably boost demand for X-ray instrumentation;
- Advancements in technology are mostly based on improved automation and software inclusions;
- Specialized applications, high average price, and cost of manufacture have tended to heighten the barriers to entry into this market;

We have spotted two potential market applications for NANORAY technology, where a compact, portable and with ultra fine focal spot system can be implemented:
- Micro-nano focus tomography
- XRD

Proposed values for NANORAY project:
1. Focal spot size is 10 times smaller in general. But some special models, such as UltraBright, may offer a similar functionality.
2. Power: Seems to be a main advantage over all models. Only PN90502 may compete in this area.
3. Current: Relatively small values for the industrial group.
4. Size: Another advantage, but not in the case of another tube with a cold cathode.
5. No power needed for cathode heating.
6. Quick on/off, short pulses, high repetition rate.
7. No thermal noise of electrons.
8. Longer life time of the cathode.
9. No need of electromagnetic focalization

What is important is to figure out how these innovative features can be successfully translated into a marketable product: in general, NANORAY is somewhere between the microfocus and the industrial segments. One possibility is to go towards the industrial one, claiming high stability, a small device size and very low energy consumption; in this case, anodic current value might not be as high as required. Or we might decide to aim to the microfocus segment, lowering focal spot size.

DISSEMINATION OF RESULTS

The philosophy of the NANORAY dissemination actions is based on the following key dissemination objectives:
1. To raise awareness on the features of the new NANORAY devices into the target markets
2. To show the results of the project and to foster new application of cold cathodes technologies

The above-mentioned strategy is currently being made operational in the NANORAY plan for using and disseminating knowledge, that provides an outline of the proposed activities. The guidelines of NANORAY dissemination activities are based on the following principles:
- Conceive dissemination as "knowledge sharing" and bi-directional;
- Perform cross fertilization and liaison with industrial, research, and standardisation communities;
- Involve to NANORAY also external organisations and experts;
- Involve independent experts to validate NANORAY results;
- Establish close collaboration with related projects;
- Publish NANORAY results in relevant international scientific journals;
- Organize seminars and workshops within relevant conferences in the area, producing ad hoc brochures and posters;
- Have a web site dedicated to the project, containing both a public area and a restricted area for use only by the project partners;

The University of Tor Vergata was invited and participated with a presentation on NANORAY project, with the support of INFN (National Institute of Nuclear Physics), which also organized and chaired the Conference, Channelling 2010, held in Ferrara (Italy) from 3rd- 8th October 2010.

More information can be found at http://www.lnf.infn.it/conference/channeling2010/home.html .
Prof. Sultan Dabagov, responsible for the LNF-INFN Laboratory which hosted the validation tests of the x-ray tube developed during the project, took part to the Conference in the role of Chairman. IOFFE Institute participated to the conference with the main purpose of getting updates on the technological achievements in the field of carbon nanotubes and graphene applications.

Event website:
http://www.diamond-conference.elsevier.com/conference-speakers.html
During the last months of the project, the partners found the opportunity to take advantage of an important dissemination tool for NANORAY results' spreading. In December 2010 an interview was finally fixed with the International channel Euronews (see http://it.euronews.net/ online), which was highly interested in the project's scientific and technological challenges.

On December 23rd, then, Labor in the role of the project Coordinator and ENE with the support of INFN- where the validation tests have been performed in the last year of the project- met the television channel for a brief interview reporting: 1) a summary of the project's intents and 2) a demonstration of the working prototype.

During this one-day interview, which will be broadcast in February 2011, the generation of X-ray beams from the CNTs deposited on the cathodes' substrates was presented and shown, as performed during the project's test sessions. Several actions have been undertaken from the beginning of the project for disseminating the project's technological achievements, some of them have already been attended by the project Consortium, a list of other events like seminars/conferences has been agreed with the partners for future participation.

Furthermore, a timetable with training activities of RTD performers towards the SMEs will be agreed after the end of the project for sharing the acquired knowledge and thus become the beneficiary of the technology.

EXPLOITATION PLAN
Several applications have been taken into consideration in Deliverable 10 on the study of x-ray applications market; together with NT-MDT, the Russian company represented within the project Consortium, we have evaluated the NANORAY performances and consequently its potential application field. XRF technique proved to be the most suitable activity on which to concentrate our effort for the NANORAY device engineering, for several reasons that have been described in both D9 and D10. This conclusion also comes from the analysis performed in D10 on the European and global spectroscopy market (which goes into detail in the XRF segment), which suggested that NANORAY device could properly fit in this sector. In order to support this consideration we have carried our a parallel anteriority analysis for exploring how many and which patents have been realised in the past years for protecting the technological results related to both CNTs synthesis process and X-ray tubes implementation as instruments embodying cold cathode sources. A brief description of the international patent depositions is thus reported.

The results are listed according to the Transaction Table included in the Annex 1 - Description of Work; nonetheless it is important to understand how the commercially exploitable knowledge arising from the project can be summarised as:
- The Cold Cathode, exploitable in several industrial applications (Result 1)
- The NANORAY X-Ray device, which is obtained through the integration of Results 2 to 4;

According to what has been stated in the project Description of Work, the partners evaluated the possibility to protect these four results with patents/licenses. To do this, an accurate anteriority research has been performed in the last months of the project, in order to have clear ideas on which is the state of the art of the research on these issues, and if other companies, throughout Europe and in other continents, have already patented them. As a consequence, an exploitation strategy will be decided and pursued.

-CNTs synthesis process patents

A list of the existing patents is reported below, for completeness of analysis. We have here selected some of the existing patents, mainly concerning the process of carbon nanotubes synthesis by CVD (which is the one used for NANORAY cathodes realization).

- US patent 7504570: Method of manufacturing carbon nanotubes (Publication Date:17/03/2009)
- US patent 7794797: Synthesis of carbon nanotubes by selectively heating catalyst (Publication Date:

14/09/2010)
- US patent 7214361: Method for synthesis of carbon nanotubes (Publication Date:08/05/2007)

Other registered patents, like the European one (published on 1st September 2010), mostly refer to carbon nanotubes/nanofibers production and recovery process or to their manufacturing using innovative techniques (EP1980530). When referring to field emission properties of carbon nanotubes for the production of cathodes, we can notice one very aged European patent (EP 0 801 805 - extended to DE, FR, GB, AT, CH/LI- published 23/02/1994) and the relative American one (US patent 5773921), in which "the use of CNTs field emission cathodes" is claimed. The exclusive license has returned to Till Keesmann in Heidelberg. Till Keesmann is registrant, owner of all rights and licensor. ARGUS Holding® GmbH concluded an exclusive license agreement with the US company SI Diamond Technology, Inc. in 2000 to further market the technology.

- CNTs-based X-ray tubes patents
We are dedicating this section of the deliverable for describing an important patent registered some months before the formal end of NANORAY project, because this seems to be the most relevant one for the future actions that the Consortium or some of the participant SMEs will decide to undertake for engineering and exploiting the project prototype. The mentioned patent is:
-US patent 7778391 - Published on 17/08/2010
The invention concerns a field emission cathode as well as an X-ray tube with such a field emission cathode. The cathode has a field emitter and an extraction grid, which are moveable respect to each other. In this sense, the internal configuration of the suggested tube differs from NANORAY internal electrodes configuration since our tube has a fixed distance between the electrodes and takes advantages of a multiple accelerating electrode system, coupled with a series of focalizing cylinders.

However, the purpose of the tube described in this patent by Siemens seems to be close to NANORAY's purpose, namely the intention of the developers to provide highly durable and long-service-time cathodes and X-ray tubes.

greater than NANORAY Exploitable results
We have developed, together with a description of the technological content of each exploitable result, its current state of development according to NANORAY project timetable and research, and we have further indicated which could be the next steps for the SMEs of the Consortium for planning the specific result's commercialization or additional improvements needed for approaching the market.

As already introduced, the main results coming from NANORAY project will be the cold cathode and the total device. In fact, several patents have been registered, from the invention of prof. Kumakhov on, concerning the polycapillary optics able to focalise an X-ray beam (just as an example we can mention the US patent 5497008).

Therefore, since for the purpose of NANORAY system we have used some commercial 4th generation policapillary optics, this result will not be considered as specifically applicable for patenting. In this deliverable the state of development of the two individuated results is also described.

For a more quantitative plan of the production phases of NANORAY device as intended in the delivered designs of the final tube, we have reported some additional information about the estimated costs associated to both the cathode production and the internal 'valve' realization.

The proposed actions for the realization of a commercially interesting device to be used as a concrete alternative to traditional sources have been identified to be the following:
1) deep analysis of the cathodes' costs, considering that an automated process will be necessary for large scale productions.
2) 6 months research - mainly constituted by validation tests - to be carried out for further improving the tubes' performances (with the objective of reaching 0.1mm focal spots and 1 mA stable current)
3) Identification, or confirmation, of the application field of NANORAY device: up to now, the tubes' performances can appreciably fit for an XRF application, but the specific use of this technique must be chosen, this implying a further customization of the device for specific purposes.
4) The intention of the project SMEs is to patent the fourth result (NANORAY prototype) in one of their countries first (Russia or Italy have been the proposed ones at the time we are writing). The next step, after engineering and producing a finite product for a specific market application, will be to extend the patent to other European countries after considerations on its validity and interest to the chosen market segment

THE STATE OF THE ART:
Although at the time we have been working at NANORAY prototyping phase there were no evidences of the presence, on the market, of a commercial product based on CNTs technology for X-ray tubes by Oxford Instruments, some very recent trials have been made in this direction to reach this goal.

One of the most important, that must be mentioned, is the news - dated the end of 2007 - reporting about Oxford Instruments, in collaboration with several other companies for realizing a space application with carbon nanotube-based tubes. Ames Research Center awarded inXitu Inc. (formerly Microwave Power Technology), of Mountain View, California, a Small Business Innovation Research (SBIR) contract to develop a new design of electron optics for forming and focusing electron beams that is applicable to a broad class of vacuum electron devices.

This project resulted in a compact and rugged X-ray tube with a carbon nanotube (CNT) cold cathode with a circular electron beam that is focused to a diameter of less than 80 microns. Oxford's X-ray Technology Group provides laboratory space and production support for continuing development and commercialization of advanced CNT-based vacuum sources. The company produced Eclipse 1 and Eclipse 2 X-ray sources from inXitu's prototype that was used in hand-held and portable fluorescence spectrometers for in situ analysis of materials and surfaces. However, no evidence can be found on the Web regarding the possibility to buy such a device at commercial conditions and prices, since it has been explicitly developed for NASA applications and again with research purposes. Even if the research on CNTs' field emission properties is rapidly growing and reaching extraordinary results that prove the feasibility of their use in several different fields, up to now we have no knowledge of actually commercialized X-ray tubes in Europe based on this technology.

Potential Impact:
The objective of the intended NANORAY project is to realize an innovative X-Rays tube based on the field emission properties of CNTs; the possible applications of such a device are manifold and comprise:
- Quality control applications such as validation and verification of incoming raw materials or product composition compliance monitoring, especially in food industry.
- Archaeometric surveys, in order to establish the dating of art and analyzing issues related to the materials used to make the objects, their degradation and ageing. These questions must be answered to ensure the proper conservation of the objects, whether it is preventive or involves intervention on the objects and X-ray diffraction has always played an important role in non destructive study of works of art and museum objects. Forensic applications such as forgery identification in art museums or precious metals content in jewelry.
-Medical diagnostic systems: society is deriving immense benefits from the use of X-rays for detection of a variety of diseases, bone fractures and deformations and this technology is widely used in clinics and hospitals. Ambulance personnel could use portable X-ray equipment to assess injuries at an accident scene, for instance and that would a potential stunning breakthrough. Furthermore, a coherent X rays beam such the one produced by the intended NANORAY project could be adopted in dentistry, where there is the need of small, portable device, with low radiation doses for the patient.
-Security applications: Airport X-rays aren't just for baggage anymore. Radiation-emitting devices now are screening people and vehicles at terminals, border crossings, and other checkpoints where security officials want to look for weapons or contraband. More such use is likely after officials from the Food and Drug Administration and other agencies described how lower-level radiation machines could be used without special precautions. That's why the introduction of small, portable reliable and harmless X rays generator will find a fruitful market.
-Aerospace applications: Remote spectral analysis techniques have been applied to many objects within our solar system, with the result that in general terms, we know the compositions of the solid surfaces that we can Image. The elemental / chemical information obtained by in situ X-Ray spectroscopy, while informative, has spawned a cottage industry of studies of analog materials and a myriad of interpretations. Remote sensing is principally useful in the formulation of hypotheses that can later be investigated by in-situ analysis techniques. There is a wide variety of objects in our solar system to which in-situ instrument packages could be profitably deployed.
-Environmental applications such as metals in soils and air particulate filters, or accurate alloy identification for scrap metal recycling
-Mining and mineralogy applications such as exploration, core sampling, tailings analysis, and product control

Both the NANORAY system itself and the production process of CNTs selectively grown on special materials in order to be suitable emitters for cold cathodes, are results that can easily and rapidly break into the market and greatly improve the competitiveness and profits of SMEs involved in the project.

It is worth to picture the target market in terms of revenues and expansion forecasts: concerning only security applications, for instance (banks, airports, etc.), market rolled by from hardly consistent to very substantial, with more than a billion dollar of turnover only in 2005 and it is steadily growing up, especially due to the persistent terrorist threat.

Researches show that final end users push towards a radical product innovation to improve the performances of the current state of the art X Rays tube, above all in terms of spatial resolution and tube's life cycle; increasing spatial resolution would indeed improve the effectiveness of the analyses performed in quality control in food industry and above all in medical diagnosis, allowing to discriminate smaller defects, invisible to current systems.

End users interested in this new technology range from:
- Universities and Research Centers, who have always represented important customers for X-Rays machineries
- Pharmaceutical and biotechnological companies, whose commitment toward quality control has been hugely increased due to new FDA's regulations.
- Semiconductor industry, represented in the Consortium by multinational company Selex Sistemi Integrati, for the control of integrated systems whose market has exponentially grown up since '90s.
- Chemical and petrochemical industries, traditionally interested in determination of the concentration of additives and polluters in oil's derivates in order to assess their environmental impact
- Hospitals and Medical centers, interested in applying X rays on the field.
- Aerospace industries, for advanced materials characterization.

Finally, there is an increase in the request of small and portable X-Rays devices in sectors not interested in this technology before, due to new European regulations such as "Restriction of Hazardous Substances" (RoHS), "Waste Electronic and Electrical Equipment" (WEEE) and "End of Life Vehicle" (ELV). The group of SMEs will exploit the Intellectual Property Rights gained in the project, taking advantage of the cooperation with the RTD performers in working out different parts of the system. The following table indicates the result gained by each SME proposer from the project.

The hypothesis made in the assessment of the potential market and the economical viability of the project are the following:
1. timing and growth rate: assumption of 1 year for engineering and marketing after the end of the project, before starting the production. Only 500 units produced in the first year, an high sales growth rate in the first 3 years, gradually reduced to 5% in the long term.
2. cost of NANORAY units: estimated to 4000 EUROS in the first year, gradually reducing to a more competitive value, 3000 EUROS;

SME Competitiveness

First of all we will consider the relationship between SMEs and nanotechnology: the role of SMEs in the field of nanotechnology (Nanomaterials, nanoparticles, etc,) will be of paramount importance in the very next future. Large and transnational enterprises have led the research and the resources investment over last decades opening the road to new industrial products and technologies. Two of the major companies involved in this sector, CNP Scientific and Fuji-Kerzai, has recently fixes to bring their investment for nanotubes to more than 400 Ml of Euros before year 2007 and to more than 2000 Ml of Euros (up to 10000 Ml of Euros at most) by year 2010.

In these last years the industrial and scientific panorama has shifted the market references from these huge companies to small and flexible companies. The market demand changes quickly and requires a continuous production process and products updating.

The larger the company is the higher economical resources are needed to conform products and production processes. In fact this scenario obliges Large companies to modify production structures and technologies to stay competitive on the market. The SMEs indeed, thanks to their organization and market arrangement capability, are more easily ready to face the European and international market changes and needs. As a consequence the role of SMEs in the development of very innovative products, like carbon nanotubes based devices, has become more and more fundamental.Then it is important to evaluate the strategic advantage for companies like TECNA and NTI in acquiring the know how to exploit and push to the market an innovative device such as NANORAY, that could pave the way to a new way to look at radiography.

Project objectives are very relevant for the group of proposers, 4 companies active in the field of industry equipment, nanotubes, X rays manufacturing and advanced characterization systems, coming from 4 member states. The specificity of the nanotubes, which require to develop knowledge and skills in a wide range of scientific disciplines, will therefore have an impact on the development of nanosciences and nanotechnologies, generally speaking, more important that any other topic.

Patent protection will be certainly sought, and will constitute a joint patrimony of the group of SMEs.

All these companies are highly motivated by the perspectives of the new application, and will strongly benefit from mutual collaboration and cooperation with renowned European research centres. The participation of 4 companies allows to dilute individual development risks and create a wide basis for exploitation.

Societal implications of the proposed work
The research will have important societal implications concerning new possible application of nanotubes based systems which can have an enormous impact on quality and health of European citizens, considering for instance the reduced dose necessary to get a diagnostic radiological image with this new concept of X-ray device. The diffusion of a cheap and high quality portable X ray device based nanotubes could be speed up the development of new devices with great implications on everyday life of European citizens.

Synergies with education at all levels
Participants to the project know the importance of the communication in the field of materials technologies and their application for mass consumers. So the attention to disseminate clear and scientific information into schools and at different educational levels will be considered by the partners of NANORAY very important.

To take up of nanotube-based products and nanotechnology in general requires training of researchers and industrialists, which will be promoted by organising workshops and training courses. RTD performers could adopt the knowledge acquired in the project to update the content of university courses and laboratory classes by organizing short seminars for students and providing them with scientific data and monographies reviewing the project results and potentialities in nanotechnologies potential. These visits could be also organized during and after the project accordingly to other partners in each country represented in the consortium. The SMEs are interested in organising periodical visits of students particularly addressed to the local colleges dealing with Nanomaterials and industrial equipment topics. Special attention will be also dedicated by these partners to explain the new equipments developed and methodologies while focusing on the best practices suggested by tests.

List of Websites:

http://www.nanoray-project.