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Real-time nano-characterisation technologies

NanoQI

Multimodal X-ray and Hyperspectral Thin-Film Nano-material Evaluation and Quality Imaging

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Whitepaper and teaching material on machine and radiation safety of inline-insitu X-ray + HSI units

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Executive Summary

This public report constitutes deliverable D1.8 “Safety assessment and approval for inline units”. It concerns a safety risk assessment for integrating both the HSI camera and XRD/XRR units to the machines defining needed safety measures. This report contains widely applicable guidelines on what safety measures are required when XRD/XRR and / or HSI are integrated into coating machines or lines. It shall guide other companies to find the best suited path for their particular situation.

For both XRD / XRR operation and HSI machine integration examples of security related and operation clearance issues will be discussed

X-ray safety is thereby guaranteed through the set-up of the devices based on the “x-ray full protection device” principle. No radiation will leave the casing of the systems during operation and opening / sample exchange will be only possible while the x-ray source is securely turned off through a safety interlock switch.

HSI integration does not require specific safety measures that goes beyond already implemented machine safety operation instructions.

Table of Contents

1. Introduction

Functional performances of nano-materials and thin films with nano-scale thickness are determined not only by material selection but also by their nano-physical dimensions, nano-scale structure and their nano-scale chemical composition. Precise characterisation of these properties is critical to develop new functional nano-materials and optimise processes toward higher performance, improved reproducibility and yield and up-scaling to larger quantities.

X-ray characterisation techniques such as X-ray diffraction analysis (XRD) or X-ray reflectometry (XRR) are widely used in research laboratories for this task but are rarely used in industrial material development and assessment of production processes due to technical limitations and required high level expertise.

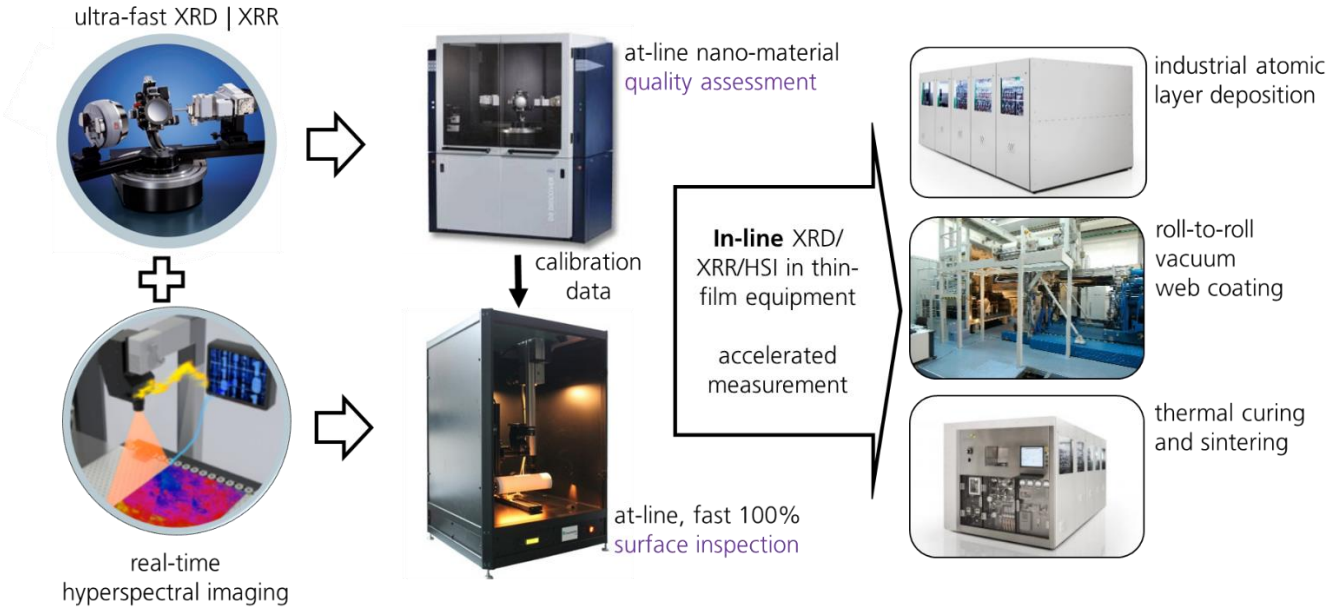

Figure 1: NanoQI Concept

The project NanoQI (see concept Figure 1) targets the development of an industry-suited, real-time and in-line capable technique to characterise nano-structure and nano-dimensions of (thin-film) nano-materials by optimisation of area-detector based XRR and XRD concepts and their multi-modal combination with a novel wide-angle hyper-spectral imaging (HSI) technique. Therewith, NanoQI will provide industry access to real time evaluation of nano-material geometry, structure and morphology and correlative imaging of deviations of these properties.

NanoQI technology will be demonstrated in three relevant industrial application scenarios: in-situ process assessment in manufacturing of perovskite solar cells; large-area vacuum roll-to-roll coating of polymer webs and industrial atomic layer deposition of dielectric and gas barrier layers.

X-ray characterisation techniques generate radiation, which are harmful to health and have to be shielded to satisfy national law regularities and to ensure safety environment for device operators. In the NanoQI project existing pilot plants are upgraded with XRR, XRD and HSI measurement equipment. Safety assessment should ensure that the modifications will not alter or reduce the safety level of the system. A full radiation safety assessment will secure that all regulations are satisfied and operation is allowed in the daily operation conditions. The results of the radiation safety evaluation is integrated to teaching material to accelerate industry uptake of the system at future customers of NanoQI Technology.

This report gives short overview about safety measures that are required for (a) running adapted X-ray metrology units *at-line* (in proximity of manufacturing machines) and (b) *in-line* integrated into a process chain in a manufacturing machine. Furthermore, machine safety considerations are given for the integration of HSI equipment as inline metrology into thin film processing machines.

The information is given as an example for countries Germany, The Netherlands and Italy which are the place of business of technical NanoQI project partners.

2. Safety Measures required for XRD integration

The operation of X-ray equipment and x-ray interference radiators can, if used improperly, cause the risk of external exposure with possible danger to the life and health of the employees involved. Therefore, specific safety regulations have to be fulfilled to avoid or to reduce the dose of radiation. In the European Union the graded approach to regulatory control is fixed in article 24 of the council directive 2013/59/EURATOM [1]. This Directive establishes uniform basic safety standards for the protection of the health of individuals subject to occupational, medical and public exposures against the dangers arising from ionising radiation. EU member states may have enacted further specific regularities. As an example of specific regularities and needed approval conditions, these have been investigated in three countries, Germany, the Netherlands and Italy.

In Germany, the operation of X-ray devices is subject to the provisions of the Radiation Protection Act '*Strahlenschutzgesetz*' (StrlSchG) [2] and in addition the Radiation Protection Ordinance '*Strahlenschutz-verordnung*' (StrlSchV) [3].

In the Netherlands, this is covered by a decree (by law) on Basic Safety Norms on Radiation Protection ('*Besluit basisveiligheidsnormen stralingsbescherming*' [4] and Regulations (by law) of the Authority Nuclear Safety and Radiation Protection on Basic Safety Norms on Radiation Protection ('*ANVS-verordening basisveiligheidsnormen stralingsbescherming*' [5]).

In Italy, the reference norm is a recent law, published on the '*Gazzetta Ufficiale della Repubblica Italiana*' on August 12th 2020 as '*Decreto Legislativo 31/7/2020 n. 201*' [6], which implements the application of the European directive 2013/59/EURATOM and replaces all laws transposing previous Euratom directives. More specific regulations for X-rays instrument for research laboratories, recommendation and operating safe practice are prepared by ANPEQ (National Association of qualified Expert professional). This guideline make reference to the law in [1] or the previous one, such as (for example) the instruction manual for research personnel '*La radioprotezione nelle attività industriali di ricerca*' (Safe measurements for ionization sources in the industrial research activities) [7]. It describes general safety norms applicable to the operation of X-rays diffraction instruments.

For approval process, the owner has to consider the specific conditions at the machine, in which the X-ray device should be integrated. One can roughly distinguish between three cases:

(a) The X-ray device should be implemented in existing equipment without special X-Ray shielding
(b) The X-ray device will operated using certified full protection devices, only.
(c) The X-ray device should be integrated by using devices, that fulfil the principles of a full protection device without having a certificate by official authorities.

In case (b) and (c) the x-ray device itself guarantees that no radiation is released outside the system casing itself. In case (a), the existing machine – to which the radiation source is integrated – must ensure radiation safety for the machine operators. Figure 2 summarizes the three cases for such upgraded machines and the needed actions for approval. Please be aware, that minor changes within the X-ray device could lead to an expiration of certificate and shifts case (b) to case (c).

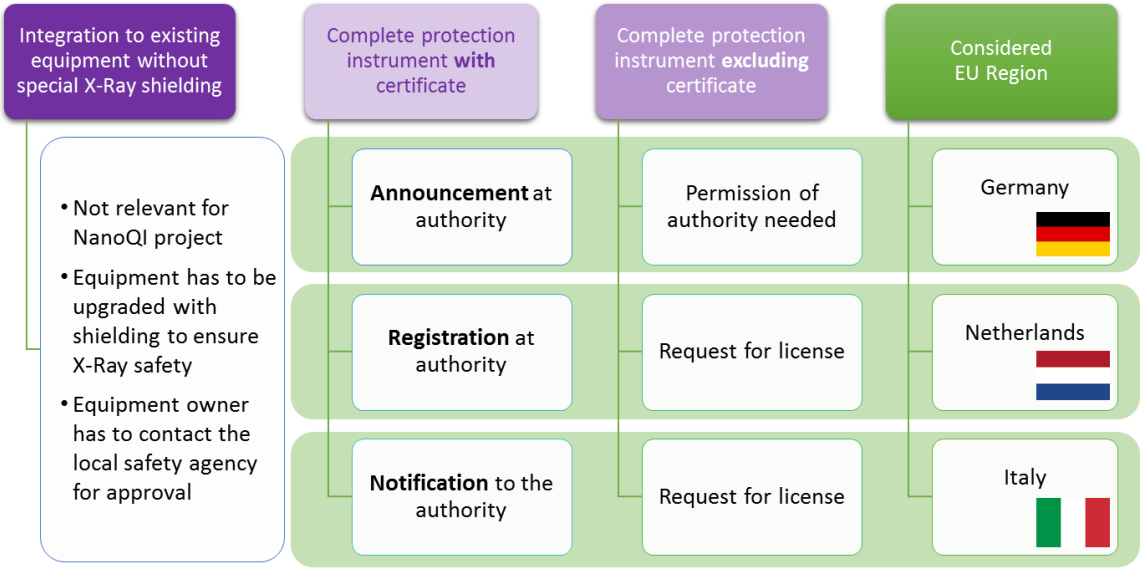

Figure 2: Decision tree for approval a XRD/XRR characterization device

2.1. Radiation safety in Germany

According to legal regulations in Germany the certain requirements have to be fulfilled by the operator of devices generating ionizing radiation. in particular, according to §§ 4, 12, 22 StrlSchG, the operator has to announce corresponding equipment to relevant German agency. As an example, the NanoQI participant FhG-FEP has enacted an addional radiation protection instruction directive, which stipulate appropriate safety activities. The essential aspects should be concluded as follows:

- Systems should emit the minimal radiation intensity. The allowed local dose rate at a distance of 10 cm from the system surface is maximum 0.2 µSv / h above the natural background radiation.
- X-ray interference radiators and X-ray devices may only be tested or operated if a responsible radiation protection officer is present in the facility of FEP.
- The daily operating time of the x-ray emitters must be recorded in system-related lists.
- Only trained and instructed persons are allowed to operate X-ray equipment and stray radiation sources.
- Changes to X-ray equipment or stray radiation sources that could impair radiation protection must be discussed in advance with the radiation protection officer.
- A functional check of the safety precautions for radiation protection must be carried out regularly. It is forbidden to manipulate or disable safety devices.

According to §5 StrlSchV the operation of X-ray equipment is free from approval under certain circumstances. In NanoQI, the designated X-ray device D8 diffractometer in its original shipped version was certified as "X-ray full protection instrument" ('*Vollschutzgerät*') according to the German X-ray safety standards called "Röntgenverordnung". That means, safety interlock switches and operation instructions will be stipulated by the manufacturer BAXS and device configuration has been approved by the responsible official German authority (here: *'Physikalisch-Technische Bundesanstalt'*) as full protection device. No further approval would be necessary if the X-ray device is installed in certificated version.

In the NanoQI specific device configuration it was necessary to increase the distance between the X-ray source and the sample for getting enough space to install all needed components on the goniometer arm and for enhancing the sensitivity. Consequently, the installed position of X-ray source was not any more within the licensed ranged and the certificate as "X-ray full protection instrument" became invalid. Therefore, safety X-ray measurements at the surrounding system surface were carried out by FhG-FEP radiation protection officer and by an external reviewer with the result that the local dose rate was not increased in comparison to natural background radiation and the radiation safety can be ensured. Nevertheless, it was necessary to ask the corresponding German authority for approval. Beside this approval aspect, no separate job safety analysis was necessary.

2.2. Radiation safety in the Netherlands

The current legal provisions regarding radiation safety are determined by a national decree (BWBR004017, [4]) from October 23rd, 2017, containing rules for the protection of persons against danger of exposure to ionizing radiation

(Besluit basisveiligheidsnormen stralingsbescherming or Bbs). The Authority for Nuclear Safety and Radiation Protection (*'Autoriteit Nucleaire Veiligheid en Stralingsbescherming'* or ANVS) supervises compliance to this ruling. According to Section 3.2 (Licenses, registration and notifications), § 3.2.3 (Registration), Article 3.8 (Categories of operations requiring a license) and Article 3.10 (Categories of operations requiring a registration), operating a device used for X-ray diffraction or spectrography, enclosed in a safety cabinet, needs a registration but not a license.

In addition, Section 4.2 (Devices and accelerators), Article 4.5 (Obligations of entrepreneurs using devices and accelerators) states that the entrepreneur responsible for devices and accelerators ensures that the rules are met as laid down by ANVS concerning:

a. testing a device or accelerator before putting it into use;
b. the shielding of a device or accelerator against ionizing radiation;
c. the arrangement of a device or accelerator and the associated aids and safety equipment;
d. the mode of operation of a device or accelerator;
e. measures to prevent unauthorized use of a device or accelerator;
f. checking the operation of a device or accelerator; and
g. the environmental dose equivalent rate that a device or accelerator is allowed to cause.

The regulation (BWBR0040581, [5]) of the ANVS of January 9th, 2018, nr. ANVS-2018/137, contains further rules for the protection of persons against danger of exposure to ionizing radiation. Of relevance for XRD devices are § 4.2 (Devices and accelerators): Article 4.5 on inherent safety (shielding) requirements, Article 4.6 on safety requirements and article 4.7 on inspection requirements).

In the case of a appropriately shielded X-ray equipment, registration of the device is sufficient for the actually installed tool. However that the conditions referred to above are met has to be checked by an independent radiation authority whose services can be called in. The check can be prepared beforehand, but will only be done when the tool is available for inspection (on site). This applies also for the mandatory Risk Inventory & Evaluation approval of the use of new equipment and/or processes in general.

3. Design and safety measured need for HSI in machines

3.1.1. Safety

Due to the fact, that neither the HSI system emits ionizing radiation nor the operation of the HSI is harmful to health of employees, no separate job safety analysis was necessary in addition to typical machine safety measures.

3.1.2. Protecting the HSI device

For operating the HSI system at low noise level a sufficient illumination is needed. Usually, an array of halogen lamps is installed nearby the sample. The lamps as well as the HSI camera may generate a major amount of heat, which has to be dissipated by sufficient cooling system. Special challenges arise, when the HSI is used in clean room conditions or in vacuum were no fan cooling is possible.

4. Conclusions

X-ray devices used for NanoQI, were constructed according to “x-ray full protection device” conditions. X-ray diffractometer devices should have undergone a thorough safety assessment according to machine safety guidelines during their development process. This also includes a standardized risk analysis and certification of various parts through specialized official external companies (like TÜV) or HALT/HAST tests. X-ray safety regulations, as shown, need to be fulfilled any time. HSI does not require any special security measures.

5. Degree of progress

The deliverable D1.8 is fulfilled for 100%.

6. Dissemination level

This Deliverable is public and will be shared on NanoQI website.

7. References

[1] https://eur-lex.europa.eu/eli/dir/2013/59/oj
[2] https://www.gesetze-im-internet.de/strlschg
[3] https://www.gesetze-im-internet.de/strlschv_2018
[4] https://wetten.overheid.nl/BWBR0040179/2018-07-01
[5] https://wetten.overheid.nl/BWBR0040581/2020-01-01
[6] https://www.gazzettaufficiale.it/eli/gu/2020/08/12/201/so/29/sg/pdf
[7] https://www.ospedalesicuro.eu/attachments/article/190/RadIoniz_2002_ManRadProd_LavInd.pdf

8. Appendix 1

No Appendix are included within this report.