Publishable Summary for 18NRM07 NanoXSpot
Measurement of the focal spot size of X-ray tubes with spot sizes down to 100 nm

Overview

X-ray-based computed tomography (CT) systems are increasingly used in industries such as aerospace and medical devices, for non-destructive testing and for the evaluation of defects and inner structure. These industries require inspection resolutions at the nanometre scale. Therefore, the overall aim of this project is to develop traceable measurement methods for determining the focal spot size, shape, and position of nanometre resolution X-ray tubes. The project will use these methods in the preparation of pre-normative documents for submission to CEN TC 138 (Non-destructive testing) WG 1 (Radiographic testing) and to revise standards in the series EN 12543 for focal spot measurements as well as to harmonise standards and measurement methods between CEN, ISO, and ASTM.

Need

Digitalisation and advances in the manufacturing industries and the health care sectors lead to substantial progress and benefits for society. Modern cars, in particular electrical vehicles, have increasing numbers of electronic components and safety-critical electronic systems, which must be fully inspected. The increased use of lightweight compounds in the aviation industry requires new inspection capabilities for internal structures in the micro- and nanoscale. A similar trend towards micro- and nano-scale non-destructive testing is observed in many industries with ongoing miniaturisation and use of new materials or new production methods like additive manufacturing.

A new generation of X-ray tubes with nanometre capabilities exists, that enables the visualisation of nanometre structures. Metrological computed tomography (CT) is used in industry for the verification of product dimension and integrity. However, the performance of these inspection systems depends on the focal spot size, shape, and stability of the spot position. Currently no standardised measurement methods exist for spot sizes below 5 µm. X-ray equipment manufacturers apply proprietary measurement methods leading to inconsistent results.

To meet the future requirements of various industry sectors to resolve structures down to 50 nm, X-ray techniques with large magnification must be used. One essential parameter for the image quality and resolution is the achievable image unsharpness, mainly influenced by the focal spot size, shape, and position. Additionally, new nanometre X-ray techniques will only be accepted by industry if a standardised method for characterising X-ray tubes is available. Therefore, new methods based on traceably characterised nanometre gauges for the determination of the spot size, shape and position must be developed and an internationally recognised standard needs to be installed.

Objectives

The objectives of the project are to develop a traceable method for the measurement of focal spot sizes in the nanometre range and to provide a draft standard to be submitted to CEN TC 138 WG 1.

The specific objectives of the project are:

1. To develop a traceable measurement method for determining the spot size, shape, and position of X-ray tubes from 100 nm to 5 µm including the uncertainty of the measurements (precision and bias) with measurement uncertainty below 10%.

2. To develop traceable methods with uncertainty < 5% to characterise nanometre gauges used for the measurement of spot size, shape, and position taking into consideration line pattern and edge structures.
3. To develop numerical algorithms for the calculation of spot parameter measurements (i.e. size, position and shape) made using nanometre gauges, including software implementation using numerical modelling and an evaluation of other parameters affecting spot size.
4. To perform inter- and intra-laboratory comparisons of the methods developed in objectives 1-3 and from the results validate the methods. Further, to incorporate the new methods into a draft standard on the characterisation of X-ray tubes with focal spots < 5 µm.
5. To contribute to the standards development work of the technical committees CEN TC 138 WG 1, ISO TC 135 SC 5 and others, where appropriate, to ensure that the outputs of the project are aligned with their needs, communicated quickly to those developing the standards and to those who will use them, and in a form that can be incorporated into the standards at the earliest opportunity.

Progress beyond the state of the art

Traceable measurement methods for determining the spot size, shape, and position of X-ray tubes:

This project will develop the first traceable evaluation method for focal spot sizes below 5 µm. The shape of the focal spot profile will be taken into account, in order to ensure compatibility with existing standards in their application range while ensuring relevance of the determined spot size for characterising the effect on geometrical unsharpness in radiographic methods. Alternatives to line pattern gauge methods will also be explored, such as hole gauges.

Traceable methods to characterise nanometre gauges used for measurement of the spot parameters:

New traceable methods for characterisation of nanometre-scale line pattern gauges will be developed. These methods will take into consideration the cross section of attenuating structures. In order to achieve sufficient attenuation at higher photon energies, line pattern gauges with aspect ratios greater than 10:1 were investigated but ultimately deemed unsuitable for achieving the targeted measurement uncertainty. The reference methods for the determination of the manufacturing accuracy (bias) and tolerances (precision) will be developed within the project.

Numerical algorithms for the calculation of spot parameter measurements made using nanometre gauges:

Numerical algorithms were developed for the spot parameter measurements (i.e. size, position, shape). The evaluation of line pattern gauges is based on the analysis of averaged line profile functions. A complementary method based on reconstruction of the 2D intensity distribution of the focal spot from a multitude of edge profiles acquired with precision hole gauges was also developed.

Inter- and intra-laboratory comparisons and validation of the methods, and incorporation into draft standard:

New standard parts will be developed. It will be proposed to revise existing standard parts and add new standard parts for the series of CEN EN 12543 based on the project results. The newly developed standard prEN 12543-6 will provide a simple and fast measurement method without the need for special equipment or software to industrial users. This will be suitable for in-service validation and monitoring of the nano-focus X-ray tube performance. Furthermore, the standard part will describe a more sophisticated and accurate method for the characterisation of nano- and microfocus tubes for manufactures.

Results

Traceable measurement methods for determining the spot size, shape, and position of X-ray tubes:

The overview of existing standards for the measurement of microfocus spot sizes and the literature survey on alternative gauges and measurement methods were finished.

Two standards were identified, namely EN 12543-5:1999 and ASTM E 2903-18, which have an overlap range with the procedure to be develop in the project.

The European standard is based on a geometrical technique of calculating the focal spot size from the edge response function in high-resolution radiographs. It exploits the relationship between focal spot size, geometric unsharpness, and magnification.

The American standard ASTM E 2903 is based on similar relationship between focal spot size, geometric unsharpness, and magnification as described in EN 12543-5. The optimum magnification and the precision of the method are derived from the achieved Signal to Noise Ratio (SNR) in the image. The maximum tube voltage for spot size measurements is regulated.

For the measurements five possible types of gauges have been identified, namely converging line pattern (similar to a Siemens star), linear line group patterns, pinhole, wire (single and duplex wire made of tungsten or platinum), and gauges with circular disks or holes.

Common requirements for the application of nanometre gauges with respect to image quality (CNR, SNR) and spatial resolution of imaging systems were identified. The imaging requirements defined will also lead to requirements for the mechanical design of feasible nanometre gauges such as the material of line pairs or the aspect ratio between the width and the height of a pattern. A catalogue of criteria such as minimum required contrast-to-noise ratio was compiled to compare the available gauges.

To ensure the metrological characterisation of the gauge, a new NanoXSpot (NxS) gauge with structures accessible for reference calibrations has been developed. The NxS gauge consists of four quadrants with line group patterns, converging line patterns, and hole patterns. For the investigation of the different pattern types, the following techniques have been developed and implemented:

- fit procedure to investigate the line group patterns
- reconstruction techniques to reconstruct the focal spot from the hole and the converging line patterns

A specification of a radiographic setup for every type of measurement was derived, considering the requirements of current focal spot standards, to ensure the comparability of results from different partners.

Existing and published draft standards were reviewed. The areas of overlap were defined and compared (for example range of focal spot sizes, X-ray energy, imaging parameters, type of gauge).

The areas of overlap between the following standards were analysed and compared:

- CEN TC 138 WG 1:
    - EN 12543 Part 1-5:1999-2008
    - EN 12543-2:2021
- ASTM International E07.01 committee:
    - E1165-20
    - E2903-18
- IEC (International Electrotechnical Commission):
    - IEC 60336:2020
    - IEC 62976:2017

The developed focal spot reconstruction method and the new NxS gauge permit the first time the standardized measurement of focal spot sizes and shapes in the range of 200 nm to 100 µm. Such measurements are currently possible with the pin hole method for spot sizes > 100 µm only, as described in EN 12543-2 and ASTM E 1165.

Traceable methods to characterise nanometre gauges used for measurement of the spot parameters:

A survey on the requirements of the dimensional metrology of nanometre gauges was carried out. One conclusion of this survey is that most users prefer easy to use methods for focal spot characterisation. The selected gauges are line group and hole/disk patterns. Structures covered by or embedded in a protective layer were considered challenging, because the direct characterisation methods are destructive. Therefore, combined traceable AFM/SEM and X-ray methods were applied to newly developed uncovered gauges.

- AFM measurements on line group patterns (line widths 3 µm – 8 µm) were performed. Line width, groove width, line bending, line edge roughness and side wall angle were measured. Standard uncertainties of the line width measurements were 5 nm – 25 nm (PTB) and 110 nm – 190 nm (VTT), and clearly below the target uncertainty (5%). Surface roughness on top and bottom surfaces were also measured.
- Hole structures were measured with both AFM and radiographic methods and the results agree within the stated uncertainties.

- A JIMA target with golden structures embedded in silicon was selected for destructive FIB preparation to verify the X-ray measurements. The measurements are ongoing.
- The effects of different parameters in the radiographic measurements were simulated to find which parameters are important and what the required uncertainty in the calibration is.
- Accessibility of the structures for metrological characterisation was considered in the design of the NanoXSpot gauge and gauge holder.
- Calibration methods using a µ-CMM, an optical photomask measuring machine, and a radiographic calibration technique to determine the size and shape of hole gauges and apertures were tested and applied.

Numerical algorithms for the calculation of spot parameter measurements made using nanometre gauges:

Three systems were selected for simulation studies:

- Laboratory Nanotube CT Setup installed at Excillum
- Laboratory Metrology CT System installed at METAS
- Industrial CT System YXLON FF20 CT

These systems were modelled with the help of simulation tools developed by CEA, BAM, and Excillum. According to the recommendations for the experimental acquisition parameters, a variation study has been carried out in order to verify the optimal choice of parameters. The impact of photon scattering effects and phase contrast effects were also evaluated with the help of simulation tools. For typical use cases, their impact was considered to be negligible. However, in some specific configurations, phase contrast effects may degrade the results of the numerical algorithms and therefore potential approaches for reducing this contribution were discussed.

An important topic addressed was the evaluation of the impact of geometrical deviations in physical gauges. The study used CAD models which included the most common deviations from the expected shape, which may have a high impact on the final evaluation result. These results were included in the D3 deliverable report.

A stand-alone software tool including graphical user interface was implemented by CEA. A dedicated website was created to distribute the software tool and sets of reference images (https://nanoxspot-project.cea.fr/).

The robustness study was completed by using a set of images generated with simulation tools for line group and hole patterns. The analysis of the images has shown that the implemented algorithms are robust within the tested range.

Inter- and intra-laboratory comparisons and validation of the methods, and incorporation into draft standard:

The System Properties Report, describing in total 8 systems, is completed. The following systems were described:

- Zeiss Xradia 620 versa
- Excillum – Nanotube Metrology Setup
- Zeiss Metrotom 1500
- METAS – Metrology CT System
- GE phoenix v|tome|x s 240
- X-RAY WorX R&D Lab
- YXLON FF20 CT System
- YXLON FF35 CT System

Based on this information and the capabilities and requirements of the developed methods, a plan was created for the validation tests. The major goal of the validation test is to verify the applicability of the available two draft standards. The results of the round robin test will be used to revise and finalise the draft standards for submission to CEN TC 138 WG 1.

Impact

A stakeholder committee was founded. 16 companies/institutes/committees joined the stakeholder committee and 22 persons are listed in the committee, partly from management and partly from research and application. Three stakeholder meetings have been held so far. The official NanoXSpot website hosted by PTB is online (https://www.ptb.de/empir2019/nanoxspot/home/). It consists of a public area and a members' area accessible only for consortium members, as well as a dedicated area for stakeholders. Several partners also host informative websites with general information about the project. The website created by CEA to distribute the software tool and reference images is now also accessible to stakeholders.

So far three training courses have been organised for the stakeholders. Different software tools were demonstrated for conducting focal spot measurements based on active CEN and ASTM standards as well as the new “focal spot reconstruction”. One poster to introduce the NanoXSpot project and five lectures have been presented on international and European conferences. In addition, several tweets have been posted on Twitter (via @bamResearch) and a LinkedIn project profile (https://www.linkedin.com/company/nanoxspot/) was created to introduce the project to a wider audience.

Impact on industrial and other user communities

The results of this project are expected to be used by a broad range of end users, including manufacturers of nano- and microfocus X-ray tubes and systems, inspection and metrology service providers, and their respective customers, e.g. in the fields of electronics, microbiology, and additive manufacturing. The availability of a traceable measurement method for focal spot sizes below 5 µm will enable reliable specification and comparison of these values.

The competitiveness of the European X-ray system manufacturers will be strengthened based on the new draft standards and give them an advantage over low-cost, low-quality systems from non-European markets. The emerging European industry for substitution of coordinate measurement machines (CMMs) by metrology-CT systems for product dimensioning and tolerancing will be supported.

Impact on the metrology and scientific communities

The key impact to metrological and scientific communities will be the traceable determination of focal spot size and shape, which will enable NMIs to offer metrological services and consulting to industry.

The outputs from this project will also create early impact on the metrological and scientific communities by providing calibration services for reference standards and metrology for focal spot size measurement. The project results will contribute to a significant increase in credibility of high-resolution CT measurements and will support industrial advances. The new development will benefit identifying flaws as well as measuring their dimensions and the surveillance of production tolerances. Furthermore, knowledge about the focal spot distribution is indispensable to establish comprehensive *digital twins* of CT systems, i.e. virtual CTs, for measurement planning and uncertainty analysis.

Results from the project will accelerate the already ongoing tendency of NMI/DIs to invest in CT technology to be used in dimensional metrology. One of the most important benefits of the project for the scientific communities will be the availability of a dedicated software tool that will follow the proposed procedures for the measurement of the focal spot size.

Impact on relevant standards

The project will provide methods for a new draft standard part 6 “Measurement of the effective focal spot size of micro- and nano-focus X-ray tubes below 20 µm” and a new revision of standard part 4 “Edge method with hole type gauges” in the standard series of CEN EN 12543 “Non-destructive testing — Characteristics of focal spots in industrial X-ray systems for use in non-destructive testing“. New alternative measurement procedures and structured test gauges were developed, evaluated and will be standardised. The new draft standards will be the basis for international harmonisation with ASTM E07.01 (Radiology (X and Gamma) Method), ISO TC 213 (Dimensional and geometrical product specifications and verification), and ISO TC 135 SC 5 (Radiographic testing).

The evaluation software for focal spot measurements from line and hole pattern gauge images will be made available as open access software. Two methods will be proposed for standardisation: one for manufacturers with high accuracy and one for users for validation measurements.

Longer-term economic, social and environmental impacts

The competitiveness of the European X-ray system manufacturing industry will be supported based on the new draft standards avoiding competition by low-cost, low-quality systems from non-European markets. Therefore, the standard practice for measurement of nano-focus spot size after implementation to ISO TC 135 SC 5 and ASTM E 07.01 will guarantee international application and fair trade. It will allow a worldwide comparison of products.

This new standard will also be the basis for further new standard developments at ISO TC 135 SC 5, ASTM E07, and ISO TC 213.

The developed standards will be applied for system qualification and long-term stability surveillance of micro- and nanoscale radiological systems. Industrial corporations must use approved international standards for methods of radiographic testing and digital radiography, which require e.g. the documentation of the focal spot size of the X-ray tube used in testing for quality assurance.

The potential of CT permits the substitution of CMMs by metrological CT systems. Accurate CT measurements will enable the detection of small and very small flaws and therefore fewer rejects during the production process in almost all industrial sectors, thus improving the quality, lifetime, and safety of products.

The comparable and fair selection of X-ray tubes, based on the accurate spot size characterisation, will improve the ability of X-ray micro- and nano-CT in areas such as electronic devices, electronic components like surface-mounted devices (SMDs), ball grids, and packages of integrated structures, semiconductor packaging, batteries, and fuel cells, new materials (e.g. metals, plastics, carbon fibre reinforced polymers (CFRP), microsystems, micro-electro-mechanical systems (MEMS), micro-opto-electro-mechanical systems (MOEMS), medical devices like hollow needles and surgery equipment, micro-bioengineering, small functional metal parts, i.e. injection moulds, laser weldings, and small fine castings and investment castings.

With progress in nanotechnologies the requirement for high resolution microscopy increases. Especially for microbiology and materials sciences, structures in the nanometre region need to be visualised and investigated. The new measurement procedure developed in this project will also be used for the determination of the resolution of X-ray based CT microscopes, which use nano-focus X-ray tubes and focusing X-ray optics in combination with optical magnification, to increase their acceptance and application.

High precision fuel injection systems and modern catalytic converters are used e.g. in marine applications, power generation, locomotives, and cars. The application of high-resolution metrological CT systems enables the quality assurance and optimisation of new designs, which help to reduce fuel consumption considerably and therefore also the emission of pollutants.

Many industries such as pharma-biotech, semiconductor, micro- and nanotechnology, aviation, and energy production will benefit from the project output. Inspection of microstructures with metrological CT systems will enable reliable functioning of products in the face of increasing reliance on electronics and additively manufactured parts, thus increasing Europe’s innovative capacity, leading to higher employment and wealth for the society.

List of publications

No publications to date.

<table>
<tr><td colspan="2">Project start date and duration:</td><td colspan="2">01 July 2019, 42 Months</td></tr>
<tr><td colspan="4">Coordinator: Gerd-Rüdiger Jaenisch, BAM Tel: +49 30 8104 3659 E-mail: Gerd-Ruediger.Jaenisch@bam.de
Project website address: https://www.ptb.de/empir2019/nanoxspot/project/</td></tr>
<tr><td colspan="2">Chief Stakeholder Organisation:
Baker Hughes Digital Solutions GmbH</td><td colspan="2">Chief Stakeholder Contact:
Eberhard Neuser</td></tr>
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9. Yxlon, Germany
10. Zeiss US, United States</td></tr>
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