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 used 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 and ASTM E 1165 for focal spot measurements as well as to harmonise standards and measurement methods between CEN, ISO, and ASTM. All objectives were achieved. Beyond the planed work, a new NanoXSpot (NxS) gauge has been designed, tested, and will be commercially available in 2023. Additionally, 2 standard drafts and 4 standard revisions have been submitted to 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 standardised methods for characterising X-ray tubes are 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 developed the first traceable evaluation methods for focal spot sizes below 5 µm. The shape of the focal spot profile was 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 were also explored, such as hole gauges.

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

New traceable methods for the characterisation of micrometre-scale line pattern and hole gauges were developed. These methods 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) were 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:

Two new standard parts were developed. It was 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 provides a simple and fast measurement method based on line group gauges without the additional need for special equipment. This is suitable for in-service validation and monitoring of the nano-focus X-ray tube performance. Furthermore, the standard part describes a more sophisticated and accurate method for the characterisation of nano- and microfocus tubes for manufactures. The newly developed standard prEN 12543-7 describes a focal spot reconstruction method based on hole-type gauges.

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 developed in the project.

For the measurements six 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. Ultimately, linear parallel line groups and circular disks or holes were selected for use with the developed methods.

Common requirements for the application of nanometre gauges with respect to image quality (CNR, SNR) and spatial resolution of imaging systems were identified. A catalogue of criteria such as minimum required contrast-to-noise ratio was compiled to compare the available gauges. None of these gauges fulfilled the requirements. Therefore, a new NanoXSpot (NxS) gauge was designed. This gauge has structures accessible for reference calibrations and allows metrological characterisation.

The redesigned NxS gauge consists of four quadrants with line group 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 or disc exposure

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.

All objectives were not only achieved but also exceeded (design and test of the new NxS gauge).

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, traceable AFM and radiographic methods were applied to newly developed uncovered gauges and combined SEM and X-ray methods were applied traceable AFM and radiographic methods were applied to embedded 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 for the 3 µm, 4 µm and 8 µm line groups in 0° (horizontal) and 90° (vertical) orientations. 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 JIMA target was FIB prepared. The quality of the gauge didn't allow proper characterisation of the line widths, but the measurements explained earlier XCT measurement findings.
- 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.

All objectives were achieved.

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 Comet 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 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 negligible.

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.

A stand-alone software called NxS Tool including graphical user interface was implemented by CEA. A dedicated website was created to distribute the software tool, the user manual, and sets of reference images (https://nanoxspot-project.cea.fr/). A secondary channel of distribution is a Zenodo repository: DOI:10.5281/zenodo.7625671.

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. A validation study was carried out on images obtained with line pattern and hole type gauges, with different structure sizes, for a variety of combinations of source spot size, magnification, transversal misalignment, rotation and vertical tilt. More than 150 test images were generated for source spot sizes from 0.3 µm to 30 µm and the results were consistent with the input parameters. The large majority of images generated with the recommended parameters showed results with a relative deviation inferior to 5%.

All objectives were achieved.

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. As a result, it was found, that 100% of the focal spot reconstruction results and 95% of fit procedure results fulfil the required measurement uncertainty below 10% as stated in Objective 1. The results of the round robin test were used to revise and finalise the two draft standard drafts for submission to CEN TC 138 WG 1. Additionally, four standard revisions have been submitted to CEN, ISO, and ASTM.

All objectives were not only achieved but also exceeded (two standard drafts and four revisions).

Impact

The stakeholder committee was extended to 18 companies/institutes/committees and 28 persons are listed in the committee, partly from management and partly from research and application. At the end of the project a NanoXSpot seminar and training workshop on measuring focal spot sizes based on the submitted two standard drafts was held online with more than 50 participants (Dec. 14^{th} – 15^{th}). The goal of the "Seminar and Training-Workshop" was the information on the new procedures for spot sizes <5 µm. All documents on the workshop are freely available at https://my.hidrive.com/share/v4b8.ohntc. The developed software was provided as free "open public" software and could be downloaded for training and test together with the reference images and the user manual (https://nanoxspot-project.cea.fr/) and are also available at the Zenodo repository (DOIs: 10.5281/zenodo.7806038, 10.5281/zenodo.7625671, and 10.5281/zenodo.7806096). The developed NanoXSpot (NxS) gauge will be commercially available in 2023. During the reporting period the results of the project were presented as two oral presentations and two posters at several conferences.

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 methods for focal spot sizes below 5 µm will enable reliable specification and comparison of these values.

Impact on the metrology and scientific communities

The key impact to metrological and scientific communities is the traceable determination of focal spot size and shape, which enables NMIs to offer metrological services and consulting to industry. One of the most important benefits of the project for the scientific communities is the availability of a dedicated software tool that follows the proposed procedures for the measurement of the focal spot size and free reference images for comparison to other software developments, which will be expected after the publication of the new standards.

Impact on relevant standards

The project provides methods for two new draft standards part 6 “Measurement of the effective focal spot size of micro- and nano-focus X-ray tubes below 100 µm”, part 7 “Focal spot reconstruction technique from hole images”, and revision of standard part 4 “Edge method with hole type gauges” and part 5 “Measurement of the effective focal spot size of mini and micro focus X-ray tubes” 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 evaluation software including reference images for focal spot measurements from line and hole pattern gauge images are made available as open access software.

Longer-term economic, social, and environmental impacts

The competitiveness of the European X-ray system manufacturing industry will be supported based on the new standard drafts 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. 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. 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

1. Benjamin A Bircher, Felix Meli, Alain Küng, and Andrii Sofiienko: Traceable x-ray focal spot reconstruction by circular edge analysis: from sub-microfocus to mesofocus. Meas. Sci. Technol. 33 (2022) 074005 (10pp). DOI: https://doi.org/10.1088/1361-6501/ac6225

2. David Schumacher, Gerd-Rüdiger Jaenisch, Uwe Ewert, Benjamin Bircher, Felix Meli, Andrii Sofiienko, Jens Peter Steffen, and Andreas Deresch: EMPIR-Projekt NanoXSpot: Ringversuch zur Untersuchung derneu entwickelten Methoden für die Brennfleckmessung an Röntgenröhren im Mikro- und Nanometerbereich. Proceedings of the DGZfP Jahrestagung 2022, Kassel, Germany, 23.-25.05.2022, https://jt2022.dgzfp.de/Portals/jt2022/bb/P10.pdf

3. Uwe Ewert, Gerd-Rüdiger Jaenisch, David Schumacher, Benjamin Bircher, Felix Meli, and Andreas Deresch: EMPIR-Projekt NanoXSpot: Neue Normentwürfe für die Brennfleckmessung an Röntgenröhren im Makro-, Mikro- und Nanometerbereich für Hersteller und Anwender. Proceedings of the DGZfP Jahrestagung 2022, Kassel, Germany, 23.-25.05.2022, https://jt2022.dgzfp.de/Portals/jt2022/bb/Mi.2.B.2.pdf

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

Project start date and duration:	01 July 2019, 42 Months	
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Internal Funded Partners: 1. BAM, Germany 2. METAS, Switzerland 3. VTT, Finland	External Funded Partners: 4. CEA, France 5. Excillum, Sweden 6. KOWOTEST, Germany 7. X-RAY WorX, Germany	Unfunded Partners: 8. PTB, Germany 9. Comet Yxlon, Germany 10. Zeiss US, United States
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