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 and 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 EN 12543 series 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, which 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 committee 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 will be investigated. 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 will be developed for the spot parameter measurements (i.e. size, position, shape). The evaluation of line pattern gauges will be 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 will also be developed.

Inter- and intra-laboratory comparisons and validation of the methods, and incorporation into draft standard:
The new work item standard proposal CEN EN 12543 will have two parts. The first part will provide industrial users with a simple and fast measurement method without the need for specialist equipment, for the regular validation of the nano-focus X-ray tube performance. The second part will provide manufacturers with a more sophisticated and accurate method for the characterisation of their devices.

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 develop.

The European standard is based on a geometrical technique of calculating the focal spot size from the edge unsharpness in high-resolution radiographs. It exploits the relationship between focal spot size, geometric unsharpness, and magnification.
The American standard is based on the same relationship between focal spot size, geometric unsharpness, and magnification as in EN 12543-5. The optimal magnification and the precision of the method are derived from the achieved Signal to Noise Ratio (SNR) and Contrast to Noise ratio (CNR) in the image. The maximum tube voltage for spot size measurements is regulated.

For the measurements six possible types of gauges have been identified, namely Siemens star, linear line group patterns, pinhole, wire (single and duplex wire made of tungsten or platinum), disk with hole (hole diameter 0.5 mm, 10 mm disk diameter, 100 μm thickness), tungsten conical tip. Alternatively, Fresnel zone plates and metal or polymer spheres / glass spheres may be tested.

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 is compiled to compare the available gauges.

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 are defined and compared (for example range of focal spot sizes, X-ray, 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
  - FprEN 12543-2: 2020
- ASTM International E07.01 committee:
  - E1165-17
  - E2903-18
- IEC (International Electrotechnical Commission):
  - IEC 60336:2006
  - IEC 62976:2017

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. The conclusion of this survey is that most users prefer easy to use methods for focal spot characterization. The selected gauges are line group patterns, Siemens star and hole structures. Unfilled structures were considered challenging, because the direct characterization methods are destructive. Therefore, combining traceable AFM/SEM methods and X-ray methods have been discussed. Calibration methods using μ-CMM and Photomask MM for the size and shape of hole gauges and apertures were tested.

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

The selection of CT Systems for simulation studies is completed. Three CT-systems have been selected:

- Excillum – Nanotube Metrology Setup,
- METAS – Metrology CT System
- YXLON FF20CT System

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:

- Xradia 620 versa
- Excillum – Nanotube Metrology Setup
- Zeiss Metrotom 1500
Impact

A stakeholder committee was founded. 13 companies join the stakeholder committee and 15 persons are listed in the committee, partly from management and partly from research and application. The official NanoXSpot website hosted by PTB is online. It consists of a public area and a member’s area accessible only for consortium members and a special part for stakeholders (https://www.ptb.de/empir2019/nanoxspot/home/). Several partners also host informative websites with general information about the project.

So far 3 training courses have been organized for the stakeholders. Different software tools were trained to conduct focal spot measurements based on active CEN and ASTM standards as well as the new “focal spot CT”. One poster to introduce the NanoXSport project and two lectures have been presented on two European and on international conference. Additionally, also tweets on Twitter have been submitted to introduce the project.

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 dependable specification and comparison of these values.

The competitiveness of the European X-ray system manufacturers will be strengthened based on the new draft standard 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.

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 procedure for the measurement of the focal spot size.

Impact on relevant standards

The project will provide methods for a new draft standard part 6 in the standard series of CEN EN 12543: “Measurement of the effective focal spot size of micro- and nano-focus X-ray tubes below 5 µm”. New alternative measurement procedures and structured test gauges will be developed, evaluated and
standardised. The new draft standard will be the basis for international harmonisation with ASTM E07.01 (CT for metrology), 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 pattern or hole gauge images will be made available as public domain 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 standard 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 125 SC 5 and ASTM E07.01 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 standard(s) 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. metal, 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’s 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 society.
<table>
<thead>
<tr>
<th><strong>Project start date and duration:</strong></th>
<th>01 July 2019, 36 Months</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Coordinator:</strong> Gerd-Rüdiger Jaenisch, BAM Tel: +49 30 8104 3659 E-mail: <a href="mailto:Gerd-Ruediger.Jaenisch@bam.de">Gerd-Ruediger.Jaenisch@bam.de</a></td>
<td></td>
</tr>
<tr>
<td><strong>Project website address:</strong> <a href="https://www.ptb.de/empir2019/nanoxspot/project/">https://www.ptb.de/empir2019/nanoxspot/project/</a></td>
<td></td>
</tr>
<tr>
<td><strong>Chief Stakeholder Organisation:</strong> GE Sensing &amp; Inspection Technologies GmbH</td>
<td><strong>Chief Stakeholder Contact:</strong> Eberhard Neuser</td>
</tr>
<tr>
<td><strong>Internal Funded Partners:</strong></td>
<td><strong>External Funded Partners:</strong></td>
</tr>
<tr>
<td>1. BAM, Germany</td>
<td>4. CEA, France</td>
</tr>
<tr>
<td>2. METAS, Switzerland</td>
<td>5. Excillum, Sweden</td>
</tr>
<tr>
<td>3. VTT, Finland</td>
<td>6. KOWOTEST, Germany</td>
</tr>
<tr>
<td>7. X-RAY WorX, Germany</td>
<td></td>
</tr>
<tr>
<td><strong>RMG:</strong></td>
<td></td>
</tr>
</tbody>
</table>