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NanoXSpot

Measurement of the focal spot size of X-ray tubes with spot sizes down to 100 \mbox{nm}

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1 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.

2 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.

3 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:

- 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.

4 Results

4.1 To develop a traceable measurement method for determining the spot size

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

Literature survey of standards results in the fact that no standards exist for the measurement of spot sizes of $<5 \,\mu$ m. See Figure 1.



As a preparation for the project, an overview of the standards EN 12543-5:1999 and ASTM E2903-18 was compiled. These standards were designed to measure focal spot sizes from 5 - 300 microns. Furthermore, existing literature was surveyed for methods and gauges that might have the potential to be applied to focal spot sizes down to 0.1 μ m. Six types of gauges and corresponding methods have been evaluated. It turned out that none of these were suitable to support the objective of this project.

It was decided by the consortium to design a gauge with line pattern and holes that would fulfil the imaging requirements in terms of image contrast and possibility of metrological characterization.

Furthermore, the existing gauges and the new designed NanoXSpot (NxS) gauge, shown in Figure 2, were evaluated by BAM, X-Ray WorX, METAS, Excillum, Zeiss, and Comet Yxlon in terms of feasibility for industrial application.









Figure 2: (a) new gauge design NxS-1020, (b) redesigned gauge NxS-1022, (c) gauge holder

Three measurement procedures were developed by METAS, Comet Yxlon, BAM, and CEA. These procedures should allow the comparison of different gauges and methods.

- 1. Direct evaluation of modulation contrast ("user method") is a fast method to get an idea about the size of the focal spot. It evaluates the modulation contrast of a profile at a given spatial frequency and can be interpreted as estimating the modulation transfer function (MTF) at this distinct frequency.
- 2. The fit method is based on the idea of finding an analytical description of the focal spot intensity distribution in a distinct direction and subsequently to determine its size. By imaging a well-known structure and fitting a function to its intensity profile, the image function can be fully described analytically. From that analytical description of the profile function and from the knowledge of the real object geometry, one can derive the system's Line Spread Function (LSF) in the direction of the analysed profile (see Figure 3).
- 3. The reconstruction of the focal spots 2D intensity distribution by means of projection images uses a circular aperture (i.e., hole), which is imaged with a sufficiently high magnification, so that the unsharpness caused by the focal spot, i.e., the geometric unsharpness, dominates the total unsharpness in the image. The 2D intensity distribution, and hence the shape of the focal spot, is generated from numerous edge profiles in all angular directions and subsequent filtered back projection (see Figure 4).



Figure 3: Fit method based on an analytical description of the focal spot intensity distribution to determine the focal spot size.







Figure 4: Tool for focal spot reconstruction by filtered backprojection designed by METAS.

Radiographic setups have been defined to allow the comparison between the different gauges and evaluation methods. The specification of these radiographic setups should also be used for measurements using simulated data. The setups include requirements for magnification, contrast to noise ratio and X-ray parameters.

Based on the specified setups, BAM, Excillum, METAS, X-RAY WorX, Zeiss, and Comet Yxlon carried out a Round Robin Test (RRT) using different X-ray tubes. Using pre-defined sets of X-ray parameters, the measurements were performed by the partners for focal spots smaller than 5 μ m and focal spots larger than 5 μ m.

The goal of this first small RRT was to evaluate the given description of how to perform the test and to draw conclusions for the method for the measurement of effective focal spot dimensions for the RRT in Objective 4.





Figure 5: Absolute FWHM spot sizes across all measurements for hole and line group structures. Error bars represent the 2o interval.

The major results of the small RRT are listed below:

- 1. General measurement setup
 - a. The required magnification could not be achieved in many cases. Results of these measurements should be handled with care!
 - b. Distinct magnification requirements/modulations should be provided for the repetitive measurements.
- 2. Gauges
 - a. The 3 µm line group structures on the NxS gauge suffer from severe line bending. These structures should be excluded from subsequent measurements.
 - b. The holes on the NxS gauge cannot be easily identified at full magnification. Design changes are necessary. These were incorporated in the second design iteration of the NxS gauge.
 - c. The NxS gauge shows some influence of the baseplate on structures near edges/corners
- 3. Analysis
 - a. Detailed step-by-step instructions required.
 - b. Too many manual steps in the analysis procedures
 - c. Detailed description of analysis tools required.
 - d. Strict JIMAfit parameters need to be provided.
 - e. All tools (JIMAfit, FocalSpotReco, ImageJ plugin for EN 12543-5) were relatively slow (in the order of minutes).
 - f. FocalSpotReco and JIMAfit were not comparable (2D vs. 1D fit), so an option to analyse hole measurements as an integrated line profile (ILP) was implemented.
- 4. Reporting
 - a. Clear description of parameters to be reported in spreadsheet
 - b. A distinct folder structure should be provided, which eases automated batch processing of data without too much manual work.

The results of the RRT are summarised in Figure 5.

After the small RRT the uncertainty budgets for hole pattern gauges and for line group pattern have been evaluated by METAS, PTB, and BAM.

To evaluate the convergence properties of two of the analysed methods with the existing standard method EN 12543-5, the normalized error E_n is calculated for each pair of considered measurements as

$$E_n = \frac{X_{lab} - X_{ref}}{\sqrt{U_{lab}^2 + U_{ref}^2}}$$

 X_{lab} denotes the average focal spot size (FWHM) of a set of measurements of the hole gauge or line pair method, respectively, and X_{ref} the average focal spot size calculated by using the wire gauge method (EN12543-5) serving as a reference. The associated expanded uncertainties are U_{lab} and U_{ref} . If the normalized error is below 1, it was concluded that the compared methods show a good convergence in the overlapping area.





Except for one dataset, all normalized errors were lower than 1. It was concluded that the proposed methods show a good convergence with the existing standard EN 12543-5 in the overlapping area of application (5- $20 \mu m$).

In summary, objective 1 was achieved by specifying two methods in this work package for the measurement of focal spot sizes below 5 μ m. The imaging requirements have led to the development of a new gauge, called NanoXSpot (NxS) gauge, with better contrast properties than the existing gauges. The new design also allows the characterisation of the patterns which is a condition for traceability of the measurements. The uncertainty budgets of both selected methods have been analysed in detail and calculated. For both methods the uncertainty budget is below 10%.

4.2 To develop traceable methods to characterise nanometre gauges

Objective 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.

The aim of the objective was to provide traceable methods to characterise nanometre gauges used for the measurement of the spot size, shape, and position of target designs. Line group structures and holes in NxS sample were selected for characterisation. Some characterisations were made also for JIMA target.

The measurements were started with preliminary studies on the HiCo sample (High Contrast gauge designed at Comet Yxlon). Two NxS gauges were characterized, SN 0001 and SN 0007. The structures to be characterized were selected to allow traceable determination of the spot size. Simulations in were utilized to select the measured parameters. The NxS SN 0001 gauge was characterised at PTB. As agreed with the partners of the project in the beginning of 2021, the 3 μ m, 4 μ m and 8 μ m line groups were measured in 0° (horizontal) and 90° (vertical) orientations.

AFM, SEM, and different optical microscopy methods were used in the characterisation.

Target uncertainty 1% - 5% depending on the feature size and measurement method were reached. For the calibration of the parameters of the line groups AFM measurements were carried out at PTB. For the determination of the hole diameters both AFM and optical microscopy with different instruments complementing each other were applied. In total three instruments were involved into the calibration campaign:

- 1. The Metrological Large Range Scanning Probe Microscope (Met. LR-SPM)
- 2. The 3D- or Critical Dimension (CD) AFM
- 3. The Confocal 3D-Laser Scanning Optical Microscope LEXT OLS5100

PSIA XE-100 was used in AFM measurements at VTT. Team Nanotec ISC high aspect ratio tips were used in the measurement. Tip size was estimated using blind reconstruction algorithm. The lateral scales of the AFM were calibrated with gratings calibrated by laser diffraction at VTT.

Ring gauges were characterised on the METAS μ CMM [METAS-1]. Nine circular profiles were recorded in different positions parallel to the cylindrical axis of the gauge. Roundness analyses were performed using a Gauss filter wavelength of 50 UPR.

Since the NxS gauge cannot be measured on the µCMM due to limitations of the probing sphere diameter, a radiographic calibration procedure for 2D microstructures was established at METAS. It relies on a well-aligned gauge parallel to the flat panel detector. Whereas radiographic calibration does not reach the low measurement

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uncertainties of AFM, it is comparably faster, not affected by resist residuals and provides the actual "radiographic" diameter that is required for scale calibration of the focal spot measurements.

There are several different parameters that might affect the focal spot analysis.

We have identified several parameters that can affect the focal spot determination. The parameters are schematically shown in Figure 6. The effects of the parameters were estimated by simulations. Some parameters were estimated insignificant by user experience. The feature size in NxS sample, especially the height of the structures made the AFM challenging. The characterized parameters are bolded in the list:

- Line width and gap width and variation in LW and GW
- Bending/waviness of the lines
- Variation in step height
- Side wall angle
- Line edge roughness
- Surface roughness on top surface
- Surface roughness on bottom surface
- Line width roughness
- Corner rounding

	Top view
Side view	
Line width LW Pitch or period	Pitch
	Line Groove width width
Side wall angle	Corner rounding
Jong Man	
Surface roughness	Line edge

Line edge roughness LER



Figure 6: Schematical illustration of different parameters of line structures.

The NxS gauges were inspected using optical microscopy methods. Some residues from the production process can be seen in the 3 μ m and 4 μ m line groups (see Figure 7). The residue is assumed not to affect the XCT measurements, but it can affect the AFM characterisation. Inspection by white light interferometer microscope (WLI) shows that the residue is only on the bottom surface and not filling the gaps between the lines (see Figure 8).



Figure 7: Optical image of the 3 µm line groups. Some residues can be seen between the lines.



Figure 8: WLI image of the 3 µm line group and line profiles along a gap and across the lines showing that the residue is present only at the bottom of the gaps.

The step height, evaluated according to ISO 5436-1 at PTB (Profile in Figure 9a), has shown inhomogeneities not only for different measurement fields over a single line (Figure 9b) but also for the five lines within a line group (Figure 9c). The straightness deviation was measured for 3 μ m and 8 μ m vertical and horizontal line groups and the results are shown in Figure 10.



Figure 9: Parameter selection for step height calculation in analogy to ISO 5436-1 (a), inhomogeneity of step height for nine measurement positions distributed over the line length (b) and inhomogeneity of step height for every of the five lines within a group (c) exemplary for the 8 µm vertical line group



Figure 10: The straightness deviations (line bending) for the 3 µm and 8 µm line groups

Figure 11 shows a comparison between the radiographic structure calibration from METAS and the AFM results from VTT. Deviations were mostly within the uncertainty and mainly attributed to the different measurement modalities: AFM determines a tactile diameter on the upper edge of the hole, whereas radiographic calibration determines an average diameter of the materials with high radiographic contrast.







Figure 11: Comparison of radiographic structure calibration (METAS) to AFM (VTT).

In summary, objective 2 was achieved by developing traceable methods to characterise nanometre gauges used for the measurement of the spot size, shape, and position of target designs. Line group structures and holes in NxS sample were characterized. The target uncertainty was reached in all characterizations.

4.3 To develop numerical algorithms for the calculation of spot parameter

Objective 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.

CEA designed and implemented a new software tool based which include several algorithms for the selected patterns. The work took into account the inputs from Objective 1 and Objective 2, in particular the design of the NxS gauge. All the algorithms were implemented entirely in Python language, and the approaches and methods were evolved as a joint effort of the partners of the consortium, principally BAM, METAS, Comet Yxlon, Excillum, and KOWOTEST.

Figure 12 presents the synthesis of the numerical algorithms implemented in NxS Tool, the associated standard drafts and the link to the patterns of NxS Gauge.





The software *NxS Tool* is a stand-alone Windows executable with a graphical user interface (GUI). For the ease of use, it is provided through an installation kit, which copies all the necessary files to an installation folder together with few sample images. The main distribution channel is a dedicated website: <u>https://nanoxspotproject.cea.fr/ and can also be found here: DOI: 10.5281/zenodo.7806038</u>. A <u>user manual</u> is provided along with the software, which describes the installation procedure and the usage for the implemented methods which can be found here: DOI: <u>10.5281/zenodo.7806096</u>. A set of reference test images is provided through a page of the <u>website</u> and also through a Zenodo repository: DOI: <u>10.5281/zenodo.7625671</u>.

The graphical user interface is designed to be simple and intuitive. It is composed of a main window containing the choice of the evaluation method, the visualisation of the images and the selection of regions of interest which are used by the algorithms. Since the algorithms are different with distinct parameters, for each case a second window is used to provide the necessary information. The results are displayed on the second window and follow the recommendations of the drafts of standards prepared by NanoXSpot consortium (EN 12543-x).





An illustration of the main window including two sample images is displayed in Figure 13.



Figure 13: The main window of NxS Tool v2022.06

Once the regions of interest are well set, the button "Run evaluation algorithm" will open the second window. Figure 14 displays an example of the secondary window, for the *EN 12543-6 Manufacturer Method* case, before and after the computation.



Figure 14: Secondary window for EN 12543-6 Manufacturer Method, before and after computation

The results are presented on the second window, graphically and with estimated values. The table at the bottom of the window follows the recommendation of the proposed draft of the standard, with the measured dimensions (X/Y), the reported values, spot size values, the nominal spot size and the class, as defined by the standard.

The validation process of the implemented algorithms and implicitly of NxS Tool was divided into two main parts. In a first phase, the robustness of the algorithms was assessed with respect to variations of physical and geometrical parameters, such as noise and magnification. The objective was to ensure a consistent measurement value within reasonable limits of variations that could be expected for the main user chosen parameters. In a second phase, the validation assessment consisted in verifying that potential user errors and inaccuracies in the experimental setups still allow to provide expected results with acceptable errors. In this case, misalignments were tested through horizontal and vertical offsets, as well as rotations of the gauge with respect to its ideal or expected position.

Several sets of test images were defined and used for the robustness evaluation process. Based on the recommendations from Objective 1, the general approach was to use one or few typical cases, for which the errors should be minimal and at least two extreme cases, at or beyond the limits of recommendations. For the line group pattern, several combinations parameters were generated, for the LP10, LP3 and LP1 patterns, with

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source diameters ranging from 0.1 μ m to 30 μ m, magnification factor in the range 200 to 5600 and contrast to noise ratio between 10 and 60. For the hole type (aperture) pattern, several combinations of parameters were generated, for the diameters 100 μ m, 20 μ m and 10 μ m, with source diameters ranging from 0.3 μ m to 30 μ m, magnification factor in the range 200 to 2000 and contrast to noise ratio between 10 and 60.

For a better interpretation, the results were split into two ranges of the tested focal spot sizes. Figure 15 presents the results in the range 0.1 μ m to 1 μ m and Figure 16 presents the range 5 μ m to 30 μ m. The green bars represent the references, which include error bars of 5%. Two main algorithms were tested, the line group fit method for the line group pattern and the focal spot reconstruction for the hole type pattern. The values are represented by blue and respectively yellow bars in the figures. As described in the overview, the data sets contain images generated using the recommended acquisition parameters, but also images using parameters outside the recommended ranges. These cases for which the output values are expected to have a higher error, are indicated on the bars with red border (for both line group and hole type cases).



Figure 15: Synthesis of results of the robustness evaluation for the range 0.1 µm - 1.0 µm



Figure 16: Synthesis of the robustness evaluation for the range 5 μ m – 30 μ m





The results showed a homogeneous behaviour of the evaluation algorithms. All the cases with parameters within the recommended range have values with relative errors within the limit of 5%. The cases with parameters set outside the recommended range have higher errors, up to more than 10% as relative difference.

The validation study as the second phase of the validation process, aimed to be an exhaustive set of tests of the main algorithms, in order to cover different potential scenarios for measurements. The main objectives here were to ensure that the results obtained with NxS Tool were similar to the ones obtained with other implementations of the algorithms, and also very important was to ensure a correct output for cases with inaccuracies in the experimental setup. As a general rule, the variations in the final estimations were expected to be within 5% as relative deviations from the ground truth.

In addition to the images generated in the first phase, new datasets were generated for the studied test cases. For the line group pattern, combinations of parameters were generated for the LP10, LP3 and LP1 patterns, with source diameters ranging from 0.1 μ m to 10 μ m, magnification factor in the range 280 to 5600, transversal misalignment 1x and 2x of the estimated source diameter, rotation and vertical tilt of 1° and 2°. For the hole type (aperture) pattern, combinations of parameters were generated for the diameters 100 μ m, 20 μ m and 10 μ m, with source diameters ranging from 0.3 μ m to 30 μ m, magnification factor in the range 280 to 5600, transversal misalignment 1x and 2x of the estimated source diameter, rotation, and vertical tilt of 1° and 2°.

The results were split into two ranges of the tested focal spot sizes. Figure 17 presents the results in the range 0.3 μ m to 1 μ m and Figure 18 presents the range 5 μ m to 30 μ m. The same representation is used, namely the green bars represent the references (which include error bars of 5%) and the values of the two main algorithms are represented by blue and respectively yellow bars. The results for images using parameters outside the recommended ranges are highlighted by the bars with red border (for both line group and hole type cases).



Figure 17: Synthesis of results for the validation evaluation, for the range 0.3 µm - 1.0 µm



Figure 18: Synthesis of results for the validation evaluation, for the range 5 μ m – 30 μ m

The results confirm a homogeneous behaviour of the algorithms over the tested focal spot ranges and for a variety of test cases. The majority of images generated with the recommended parameters have a relative deviation inferior to 5%. For the spot sizes 0.3 μ m and 0.5 μ m, some of the deviations are slightly higher than 5%. The chosen combination of gauge pattern and sampling parameters are close to the limits of the algorithms therefore higher deviations were to be expected. For the range 5 μ m to 30 μ m, the majority of values show less than 5% of relative error, including most of the cases with parameters outside the recommendations.

In summary, objective 3 was achieved by the development and implementation of numerical algorithms for the evaluation of spot parameters. The proposed methods are well adapted to the purpose and to the analysis range. The methods and the software implementation have been validated in terms of simulated and experimental reference images which can be used by the public together with the reference implementation and the user manual. It can be concluded that the achievements exceeded the initial objective, both in terms of number of algorithms and in terms of the range of use.

4.4 To perform inter- and intra-laboratory comparisons

Objective 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.

The primary goals set by objective 4 were to incorporate the developed methods into a draft standard and to validate their ability to meet the uncertainty requirements set in objective 1, as well as to validate the reference software developed to meet objective 3 in the process.

To evaluate applicability of the methods and determine corresponding uncertainties, it was necessary to cover a broad selection of focal spot sizes and shapes, as well as to involve a variety of personnel in the acquisition and evaluation phases. Ultimately 12 participants contributed to the inter- and intra-laboratory comparisons:

- 6 project partners: BAM, Excillum, METAS, VTT, X-RAY WorX, Zeiss, and Comet Yxlon
- 2 collaborators: FAU and, Waygate
- 4 stakeholders: Comet, LANL, NIST, and VJ Technologies

The first step needed was the collection of information about the various systems available to the consortium. Based on this information a measurement plan was developed, taking into consideration learnings from the earlier, smaller measurement campaign in Objective 1 and gauge characterization in Objective 2.





Microfocus spots are in general not stable over longer periods of time, so that it was not possible to have multiple participants measure the size of identical focal spots. Consequently, every participant was asked to provide several radiographic images for each available focal spot, acquired with specified parameter variations to test the limits of the methods.

Due to time constraints and the needs of other project activities, only one NanoXSpot gauge metrologically characterized within Objective 2 could be used. An additional uncharacterized NanoXSpot gauge was used to facilitate progress by carrying out measurements in parallel. In addition, one gauge of an alternative design, HiCo by Comet Yxlon, was used in some measurements to ensure usability of the methods and software with various gauges meeting the methods' requirements. No issues pertaining to gauge type were identified.

Newly written standard drafts, prEN 12543-6 for line group evaluation and prEN 12543-7 for focal spot reconstruction from hole type structures, were used as primary instructions for carrying out the measurements, to maximize probability of identifying any issues with the drafts. Additional instructions were only provided for specifics of the validation tests, i.e. to ensure suitability of the data for the intended analysis. All evaluations were carried out using the current version of the reference software (NxS Tool).

In total 849 evaluation results were generated from 540 radiographic images, with 406 evaluation results meeting all requirements and being considered for further analysis. A subset of 46 evaluations, ca. 12.5% of the considered evaluation results, was evaluated by more than one partner. The main analysis was carried out by Comet Yxlon, with support on methodology provided by BAM, METAS, and KOWOTEST.

For the majority of focal spot evaluations that met the minimum requirements set by the draft standards, a standard uncertainty of 10% or below was found, in line with objective 1. Specifically, 95% of the considered line group evaluations and 100% of the considered hole evaluations exhibited standard uncertainties of 10% or below (see Figure 19). For those cases exhibiting larger standard uncertainties, the causes could be identified, and recommendations be made for extension of the reference implementation, with no changes required to the standard drafts.



Figure 19: Histogram of deviations for both evaluated methods

In summary, objective 4 was achieved by validating the developed methods and incorporating them into two new standard drafts, one for each method, for measurement of focal spot sizes down to well below 5 µm.





Suitability of the accompanying reference software implementation of the evaluation methods was demonstrated as well.

4.5 To contribute to the standards development work

Objective 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.

The goal of the project was the critical review of the standard series EN 12543 and the development of one new standard, extending the series of EN 12543 with an additional part to cover the measurement range for focal spots of $0,2-5 \mu m$.

This was successfully finalised. Additionally, to the development of one new standard draft, the outcome of the project was extended to four revisions and two new standard drafts to guarantee a holistic system of supporting standards.

During the project period KOWOTEST and BAM contributed to the revision of

- **ASTM E 1165**, which was revised and published in 2020 as "Standard Test Method for Measurement of Focal Spots of Industrial X-Ray Tubes by Pinhole Imaging".
- EN 12543-2, which was revised and published in 2021. This standard was submitted to ISO/TC 135/SC 5 and will be published as EN ISO 32543-1.

As outcome of the project NanoXSpot, after critical review of the standard series EN 12543, the NanoXSpot consortium decided to develop revisions of **EN 12543 part 4 and part 5**. The following revisions were submitted from KOWOTEST and BAM with support of the NanoXSpot consortium to CEN/TC 138 WG 1 via the secretariat of DIN:

- EN 12543: "Non-destructive testing Characteristics of focal spots in industrial X-ray systems for use in non-destructive testing Part 4: Edge method with hole type gauges".
- EN 12543: "Non-destructive testing Characteristics of focal spots in industrial X-ray systems for use in non-destructive testing — Part 5: Measurement of the effective focal spot size of mini and micro focus X-ray tubes".

Additionally, two new standard drafts, **prEN 12543-6 and prEN 12453-7**, were submitted from the NanoXSpot consortium to CEN/TC 138 WG 1 via the secretariat of DIN:

 prEN 12543: "Non-destructive testing — Characteristics of focal spots in industrial X-ray systems for use in non-destructive testing — Part 6: Measurement of the effective focal spot size of micro- and nanofocus X-ray tubes with spot sizes < 100 µm".

This European standard specifies a method for the measurement of effective focal spot dimensions between 0,2 μ m and 100 μ m of X-ray tubes up to and including 225 kV tube voltage by means of the line group fit method and evaluation of digital images. ... This test method provides instructions for determining the effective size (dimensions) of nano and micro focal spots of industrial X-ray tubes. This determination is based on the evaluation of an X-ray image taken with line group gauges. The standard defines the design of the line group test gauges with structures in the nm and μ m range and it provides the procedure, how to determine the focal spot size from the line group radiographs.

• **prEN 12543:** "Non-destructive testing — Characteristics of focal spots in industrial X-ray systems for use in non-destructive testing — Part 7: Focal spot reconstruction technique from hole images".

This European standard specifies a method for the measurement of effective focal spot dimensions below 0,1 mm of X-ray systems by means of the reconstruction technique, applied to digital images taken from hole type or disk type gauges. This method can also be applied for the measurement of effective focal spot dimensions above or equal to 0,1 mm, but the application of the pin hole method of EN 12543-2 is preferable. This document provides instructions for determining the effective size (dimensions) of mini and micro focal spots of industrial X-ray tubes for users in applications where the



pin hole method of EN12543-2 is not practicable. This determination is based on the measurement of profiles of an image of a hole or disk type gauge and the reconstruction of the 2D distribution. The use of the pin hole method (ASTM E 1165, EN 12543-2) requires the application of special pin hole cameras. This procedure describes, how the 2D spot shape and size of focal spots can be determined from hole or disk exposures, which are easier to produce in comparison to pin holes and the related cameras. Microfocus (1 μ m \leq spot sizes < 0,1mm) and nanofocus tubes (spot sizes < 1 μ m) have a significantly lower photon flux than mini and macro focus tubes (spot sizes \geq 0.1 mm). Furthermore, pin holes (see EN 12543-2 or ASTM E 1165) with diameters in the nm and μ m region are difficult to manufacture and will not permit a sufficient photon statistic for digital imaging with the required magnifications.

The following official recommendations were published in the minutes of the CEN/TC 138/WG 1 minutes of Oct. 26th, 2022:

- **Recommendation 02/2022**: CEN/TC 138/WG 1 proposes to prepare the revision of EN 12543-5 based on the discussed draft (N 684). WG 1 recommends CEN/TC 138 to establish EN 12543-5 rev with Uwe Ewert as project leader until December of 2022.
- Recommendation 03/2022: CEN/TC 138/WG 1 proposes to prepare the development of a new "part 6" of series EN 12543 based on the discussed draft (N 682) to cover the procedures for focal spot measurements < 5 μm. WG 1 recommends CEN/TC 138 to establish EN 12543-6 with Uwe Ewert as Project Leader until March of 2023.
- Recommendation 04/2022: CEN/TC 138/WG 1 proposes to prepare the revision of EN 12543-4 rev (N 685) and the new "part 7" (N 686) of series EN 12543 in a parallel process. WG 1 recommends CEN/TC 138 to establish EN 12543-4 rev with Uwe Ewert as Project Leader until December of 2023. EN 12543-7 will extend the range of the hole camera measurement < 100 μm. WG 1 recommends CEN/TC 138 to establish EN 12543-7 with Uwe Ewert as Project Leader until December of 2023.

5 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 μ m 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 μ m", 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 "Nondestructive 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-optoelectro-mechanical systems (MOEMS), medical devices like hollow needles and surgery equipment, microbioengineering, 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.

6 List of publications

- 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: <u>https://doi.org/10.1088/1361-6501/ac6225</u>
- 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 Mikround Nanometerbereich. Proceedings of the DGZfP Jahrestagung 2022, Kassel, Germany, 23.-25.05.2022, <u>https://jt2022.dgzfp.de/Portals/jt2022/bb/P10.pdf</u>
- 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: <u>https://www.euramet.org/repository/research-publications-repository-link/</u>