Reference object for evaluating the accuracy of porosity measurements by X-ray computed tomography

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Abstract
Internal defects such as voids and porosity directly influence mechanical properties, durability, service life and other characteristics of industrial parts. There are several non-destructive and destructive methods for defects detection and evaluation. Recently, X-ray Computed Tomography (CT) has emerged as an effective tool for geometrical characterization of internal defects. 3D information about internal voids/porosity extracted from CT datasets can be utilized in many applications, such as production processes optimization and quality control. However, there are still challenges in using CT as a traceable method for internal voids dimensional measurements. In order to enhance the accuracy and reliability of CT porosity measurements, a metrological validation method is required. This study presents the application of a new reference object for accuracy evaluation of CT porosity measurements and discusses results obtained by using it. The reference object is made of aluminium and is composed of a cylindrical body and four cylindrical inserts with micro-milled hemispherical features of calibrated sizes resembling artificial flaws. The accuracy of porosity measurements is evaluated according to various characteristics (diameters and depths measurements errors) and repeatability of measurements. Design of experiments technique is used to investigate the influence of CT parameters settings on porosity measurement accuracy.

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1. Introduction
Detection and characterization of internal defects such as voids and porosity are very important in a wide range of industrial applications as there is a direct correlation between the level of porosity and mechanical properties of industrial parts and materials [1,2]. Many publications dealing with internal porosity inspection were focused on assessment of mechanical properties related to the total void volume percentage [3,4]. However, recent studies proved significance of voids geometry and distribution on various characteristics, for instance tensile strength, fatigue life, etc. [5].

There are many techniques for internal defects evaluation applied in literature, among the most established belong: (i) ultrasonic testing, (ii) Archimedes theoretical versus actual density, (iii) materialography.

Ultrasonic testing is a non-destructive technique widely used e.g. in aeronautics industry for quality control as this method is sufficiently reliable. The attenuation coefficient of ultrasonic waves is essentially linear with the amount of porosity in the material, if the defects are of spherical shape and homogeneous distribution. However, if the condition of defects shape and distribution is not satisfied and if the voids size varies excessively, the accuracy is significantly reduced [6,7].

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http://dx.doi.org/10.1016/j.csnst.2016.05.003
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Archimedes theoretical versus actual density is another non-destructive method that uses in a first step the Archimedes principle to calculate the actual density of the sample. In a second step then, based on the theoretical density of the voids-free sample, the void volume percentage is calculated. The major drawback of this method is the fact that the exact value of the density of the sample must be known. Another limitation is that this technique provides only general information about the total void content, whereas the information about defects shape and distribution is not available [8,9].

Materialography is based on image analysis of micrographs acquired from sample cross-sections, which makes this method destructive. The testing preparation procedure is usually time-consuming as the sample must be set in resin first and then a flat cross-section is prepared. The surface is sequentially ground by abrasive paper with decreasing size of grinding particles so that a high quality surface finish is achieved. This technique is rather reliable, however during the processing of the sample some scratches can be embedded and then considered as porosity or, on the contrary, small pores can be smeared and neglected [4]. Furthermore, in case of non-homogeneous pores shape and distribution, the overall analysis result can be distorted [8].

Recent advances proved X-ray Computed Tomography (CT) to be a promising 3D technique for non-destructive characterization of materials and dimensional metrology [10]. The information that can be extracted from CT datasets provides complete volumetric information about the sample structure and composition, internal and external shape, etc. [10,11]. Full information about internal porosity/voids shape, size and distribution can be extracted without a sample destruction, which makes CT a unique tool for internal defects evaluation [12]. However, in this field of application, the accuracy of CT internal defects measurements is still under investigation and a metrological validation method is needed. In this paper, an approach to investigate the accuracy of porosity measurements through a newly developed reference object is applied. In the literature, there are several studies on evaluating the performances of CT porosity measurements using reference objects with artificial defects [13–16]. However, none of them can provide accuracy evaluation based on completely internal calibrated geometries. For example, Nikishkov et al. [13] used a reference object with micro holes that were accessible from the surface and, therefore, did not simulate real internal defects. Jansson et al. [14] designed a reference object with external and internal features, but the internal features were not measured by other measurement system than CT as they were enclosed in the material; therefore, the authors could rely only on the stability of the manufacturing process.

The reference object developed specifically for this study has embedded internal features resembling real internal defects. The object is an assembly with components that can be disassembled so that the artificial defects can be inspected and calibrated as external features by more accurate measuring systems. Calibrated values can then be compared to values extracted from CT data sets, so that the accuracy of CT measurements can be evaluated.

2. Reference object

In order to properly design a reference object for thorough porosity measurements accuracy evaluation, three main requirements were defined as follows: (i) artificial defects must be completely internal, (ii) the features must allow calibration by conventional coordinate measuring systems (i.e. the object must be disassemblable) and (iii) the material should be widely applied in industry and at the same time should be suitable for CT scanning and provide sufficient metrological stability. The geometry of the reference object developed for this purpose is shown in Fig. 1. All components of the object are made of aluminium, which satisfies all material requirements as it is frequently applied in industrial parts, the X-ray attenuation coefficient is rather low compared to other metals and the dimensional stability is sufficiently good. The object is composed of a cylindrical body (15 mm in diameter and 15 mm high) and four removable cylindrical pins (5 mm in diameter) of different heights. The faces at the interface between the hole bottom and corresponding pin are machined with high precision in order to facilitate the fitting and minimize possible gap. The hemispherical features, resembling internal
artificial defects, are milled onto the pin faces using a high-precision micro milling machining centre. Eighteen defects are manufactured per pin resulting in 72 defects in total. The size range of hemispherical calottes is 100 µm–500 µm.

Calibration of hemispherical features was performed by means of two optical measuring systems: (i) 3D optical profiler (Sensofar Plu Neox) and (ii) high-accuracy multisensor coordinate measuring machine (Werth Video Check IP 400). The former instrument was used in confocal scanning mode with a 20 x objective for complete calottes surfaces acquisition. The latter was applied for measurements of defects diameters in the top plains of pins using an image processing sensor.

The expanded calibration uncertainty of diameter and height measurements was ranging from 1.8–2.5 µm depending on the size of the defect. Reduction of the calibration uncertainty is a subject of further improvements and will be considered in future research.

3. Definition of measurands and measurement procedures

The nominal shape of the analysed features is hemispherical; therefore, the diameter $D$ is the most important measurand in this case. However, due to errors caused by the manufacturing and assembly processes, the centre of the sphere does not lie perfectly on the ideal top plane of the referred pin face. Therefore, an additional important measurand is defined – the depth of the defect, indicated as $Z$ in Fig. 2-a. The hemisphere diameter $D$ is evaluated by a least squares fitted sphere (Gaussian fitting). In order to avoid errors by edge points close to the top surface, points for spheres construction close to this interface are avoided; the area of fitting points taken into account can be observed in Fig. 2-b.

The hemisphere depth $Z$ is measured as the distance between the plane fitted to the top flat face of the hemisphere and the furthest point of the fitted sphere. Both the plane and the sphere are fitted by the least squares method.

Volumetric datasets were acquired by a metrological CT system, Nikon Metrology X-Tek MCT 225, with maximum permissible error (MPE) equal to $(9 + L/50)$ µm (where $L$ is length in mm), using $18.4 \times$ magnification. The CT data were processed and evaluated using VGStudio MAX 2.2.6. In order to achieve reliable results, the measurement procedures were kept consistent during the calibration and experimental phase.

In this publication, the newly developed reference object is used for porosity measurements accuracy evaluation of a specific CT system (see above) and material (aluminium). However, the design allows manufacturing of similar objects in wide range of materials, and even their combinations. Furthermore, the object can be used also for testing other industrial CT systems. This shows a great potential for future works with focus on accurate internal defects assessment.

4. Results and discussion

Two separate investigations are performed in this paper: (i) influence of CT parameters settings on accuracy of CT porosity measurements and (ii) repeatability of measurements. In both cases, diameter $D$ and depth $Z$ are separately evaluated and results are discussed.

4.1. Influence of CT parameters settings on CT porosity measurements accuracy

Results of a previous study on influence of CT parameters settings on Probability of Detection (PoD) [17] showed that magnification has a clear trend that enhances PoD with increasing value. Furthermore, outcomes from the PoD study demonstrated the same stable effect on measurement accuracy, hence magnification is not considered in this publication. On the contrary, the effect of current and voltage was not so clear in the previous work, so an experimental investigation on their influence on porosity measurements accuracy is described further in this paper.
In order to evaluate the effect of investigated parameters, a Design of Experiments technique (DoE) with two factors and four levels for each factor (16 combinations in total) is applied in this chapter. Parameters values are shown in Table 1. The choice of parameters settings was defined in a range where for the lowest settings the image is not too dark and on the contrary not saturated for the highest settings. Furthermore, the scanning parameters were chosen with respect to maintaining the tube power sufficiently low to achieve the focal spot size not exceeding 3 µm.

**Table 1**

CT scanning parameters for DoE.

<table>
<thead>
<tr>
<th>Nr.</th>
<th>Voltage (kV)</th>
<th>Current (µA)</th>
</tr>
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<tbody>
<tr>
<td>1</td>
<td>130</td>
<td>30</td>
</tr>
<tr>
<td>2</td>
<td>145</td>
<td>35</td>
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<td>3</td>
<td>160</td>
<td>40</td>
</tr>
<tr>
<td>4</td>
<td>175</td>
<td>45</td>
</tr>
</tbody>
</table>

**Common settings**

<table>
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<tr>
<th>Voxel size</th>
<th>Filter</th>
<th>Exposure time</th>
</tr>
</thead>
<tbody>
<tr>
<td>11 µm</td>
<td>0.25 mm Cu</td>
<td>2829 ms</td>
</tr>
</tbody>
</table>

**Fig. 3** shows absolute values of deviation of defects diameter $D$ and depth $Z$ from reference values related to the CT parameters settings. The analysis was performed on all the 72 defects; however, plotting all of them would make the charts illegible; for this reason, simplified charts are given in Fig. 3. The defects are sorted into two groups, from 100 µm to 200 µm and from 205 µm to 500 µm, as the trends for the smaller and the larger defects are slightly different. The trends are constant within the groups though, so the number of plotted planes can be reduced to three representative defects for each group.

**Fig. 3.** Influence of CT parameters settings on absolute value of deviations of diameter $D$ and depth $Z$: (a) absolute value of $D$ deviation for “smaller” defects, (b) absolute value of $D$ deviation for “larger” defects, (c) absolute value of $Z$ deviation for “smaller” defects, (d) absolute value of $Z$ deviation for “larger” defects.
Two main observations can be extracted from the charts. The first one is that measurement of defects in the range from 100 µm to 200 µm is more problematic for both $D$ and $Z$, as the absolute value of deviation is higher than for larger defects in both cases. Furthermore, it can be seen that the deviation of $D$ for “smaller” defects is about one order of magnitude higher than that of $Z$ for the same defects. However, this might be caused also by difficulties in the calibration of the smaller defects by optical instruments where the number of collected points was rather low. However, except for small defects, the deviations are within 5 µm which demonstrate good performance in porosity measurements.

The second dependency that can be observed is slightly different for the two groups of defects. For “larger” defects the effect of increasing voltage is positive, i.e. the deviation is lower, whereas the effect of current is negative, i.e. the deviation is higher. On the other hand, for “smaller” defects, the effect of current can be considered neutral, while the effect of voltage remains slightly positive. These effects were not expected, and were seen only for the conditions described in this paper; therefore, their validity should not be understood as of general value. Further investigations are needed to extend the experiments in other conditions. Nonetheless, for the conditions applied in this work, the order of magnitude of the CT parameters influence is of 4 µm or lower, which shows good performance of CT porosity measurements.

4.2. Repeatability of measurements

The repeatability of CT measurements depends on several factors: focal spot drift, thermal instability, scattering, etc. [18]. In order to evaluate the repeatability of porosity measurements by CT, 12 scans with the same system set-up were performed. Repeated CT scans were acquired under the same conditions distributed throughout the whole batch scan run comprising scans from DoE discussed in the previous section. The voltage and current values were selected based on the best performance criterion extracted from the PoD study [17]. The investigation was focused on the same characteristics, defects diameter $D$ and depth $Z$.

Fig. 4 depicts the deviations of $D$ (Fig. 4-a) and $Z$ (Fig. 4-b) measurements, showing also error bars representing ±1 standard deviations. Results confirm the fact that the smaller is the defect, the more problematic is the measurement and the higher is the deviation. The same applies for the standard deviation, i.e. the smaller is the defect, the higher is the standard deviation. Overall, apart from small defects, the deviations of $D$ measurements are within ±5 µm with standard deviation within 2.5 µm. As far as the deviations of $Z$ measurements are concerned, the error is within ±3 µm with standard deviation less than 2 µm for 90% of defects.

5. Conclusion

A new reference object for accuracy evaluation of porosity measurements performed by X-ray computed tomography has been developed in this work. Unlike other proposed objects, the one proposed in this paper offers full dismountable configuration, resulting in the main advantage that it allows dimensional calibration of artificial internal defects.
The accuracy of CT porosity measurements was evaluated using the reference object, showing the influence of defects size and CT parameters settings. Furthermore, an investigation on the repeatability of measurements was performed. The results show that for optimal voltage and current settings, the errors obtained can be below 5 µm in case of diameter measurements and below 3 µm in case of depth measurements. Furthermore, the repeatability study showed that the standard deviation for optimal CT parameters settings is below 2.5 µm for both measurands. However, the investigation on diameter measurements showed difficulties in evaluation of small defects where the error was rapidly increasing. This can be caused by insufficient resolution of scans or non-negligible uncertainty of calibrations; further investigations must be performed on this.

Further research should be focused on evaluation of porosity volume measurements (instead of diameter and depth measurements), as diameter and depth characteristics apply only to artificial defects with regular shapes, such as hemispheres.

Acknowledgements

This work has received funding from the European Union’s Seventh Framework Programme under grant agreement No. 607817.

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