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Application of X-ray CT method for discontinuity and porosity detection in 316L stainless steel parts produced with SLM technology



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ABSTRACT

Industrial tomography (XCT) is a nondestructive test method that provides information about spatial distribution of X-ray absorption in the analyzed structures. The aim of this paper was to examine the possibility and accuracy of application of XCT method for discontinuity and porosity detection in parts made of 316L stainless steel powder produced by Selective Laser Melting technology. Analysis conducted on three produced test samples showed that the application of XCT as a method of quality control of specimens produced with an additive manufacturing technology offers a wide range of possibilities to detect porosity within materials. Parameters such as the amount of porosity, pore size and pore shape are presented. Accuracy of XCT method strongly depends on the size of the samples analyzed, but the possibility of obtaining information in 3D nondestructively shows considerable advantages of XCT method over traditional metallographic cross-sectional analysis.

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1. Introduction

Selective Laser Melting (SLM), which is one of the additive manufacturing technologies, enables layer manufacturing of different types of metallic and non-metallic materials using a 3D CAD volume model. The process of melting and solidifying a series of successive layers of metallic or composite powder materials on top of each other generates complex threedimensional parts [1]. Such elements can be produced without fixing and assembling, which enables production of complex structures such as conformal channels, customized patient implants or mechanisms with recess, ribs, cavities and internal features. Metallic powders such as steel, titanium, cobalt-based alloys, nickel-based alloys or aluminum alloys can be used in this technology. The SLM process is affected by many factors associated with the material used in the process and with the process itself [2]. These factors make it difficult to achieve 100% density in the manufactured elements because SLM process is affected by temperature, gravity and capillary

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effects, and there is no mechanical pressure present during the forming processes [3]. The number of layers that are affected by the process parameters and position of elements on the platform increase the risk of discontinuity in the test pieces [4]. Determining the position, direction and shape of the pores inside the components manufactured with SLM technology provides information on the impact of these factors on the final density of the material.

Currently the methods most widely used for the analysis of porosity such as metallographic cross-section of parts are expensive, time-consuming and destructive. The accuracy of this method is affected by the magnification of metallographic cross-section and cutting plane. Archimedes method allows to determine the density of the sample, but fails to provide information about the shape and position of the pores within the material. Analysis of internal structure of the elements with computed tomography is an alternative.

X-ray computed tomography (XCT) with cone-beam geometry has been used for non-destructive characterization and evaluation of materials since the 1980s [5]. XCT is a nondestructive test method that provides information about the spatial distribution of X-ray absorption in the analyzed structures. Computed tomography has become an important and powerful tool in the analysis of internal structure of materials, allowing to visualize and evaluate pores, cracks, inclusions or defects in the materials [6–9]. For example, in the case of additive manufacturing, XCT is applied to study the effect of geometry and build direction on defects in titanium components [10] or density measurements of additive manufactured metallic parts [11]. In this article, information about spatial structure provided by XCT of steel elements manufactured with SLM technology will be used to determine the impact of building strategy on porosity. For this purpose, reconstructed volumes are oriented in space in relation to the build platform. Space registration corresponding to the actual positioning of specimens in the SLM chamber will allow visualization and evaluation of the spatial distribution of porosity in the test samples.

The influence of measurement parameters, especially the impact of voxel size, on the ability to register the test specimens is an important factor which determines the applicability of the XCT method. Pores smaller than the voxel size cannot be distinguished from the reconstructed CT data set; however, they affect the recorded area by a Partial Volume Effect (PVE). This means that the reconstructed grayscale value of each voxel is the average value of the linear absorption coefficient of the radiation in the entire volume of the voxel. In the case of registering a small pore with a high surface area to volume ratio, this effect may introduce a large error determining porosity [12]. Resolution in industrial computed tomography is limited not only by the measurement system, but also by the dimension of the sample. Because of the high radiation absorption coefficients for metals, the reconstruction of these elements is associated with measurement noise and artifacts, such as beam hardening or the already mentioned PVE. For this reason, the information about the ability to register internal structure depending on the size of tested sample allows the estimation of accuracy of the XCT method for registering porosity.

2. Materials and methods

2.1. Fabrication of specimens

Specimens were fabricated from 1.4404 (AISI 316L) stainless steel powder using SLM Realiser II (MCP HEK GmbH). The SLM machine is equipped with continuous wavelength Ytterbium fiber laser with a maximum power of 100 W and beam spot size of 200 $\mu m.$ The machine is also equipped with a reduced chamber, which ensures efficient use of the powder and shortens manufacturing time. Selective Laser Melting is a technology based on the processing of metal powder. In the process metal powder is locally melted, layer by layer, using a high-power laser. Subsequent layers are built with a newly applied powder, directly on previous layers, which provides a permanent connection in the whole build. The metal powder is administered by a movable tray, which also aligns the applied layer. Particle size of AISI 316L powder used in the process is less than 63 μ m. A schematic diagram of a SLM device is shown in Fig. 1.

All samples had the same dimensions illustrated in Fig. 2. The thinnest part of the samples had a diameter of 4 mm, which provides the same resolution measurement as XCT.

Technological parameters of the SLM process, which were used to manufacture the specimens (A, B and C), are presented in Table 1.

All of the process parameters were the same for the three presented samples. The only difference was the distribution (building orientation) of the specimens within the chamber of the SLM machine (see Fig. 3).

Sample A was built up along the Z-axis, perpendicular to the platform, and required the highest number of melted layers. In this case, the surface of the melted layer was the smallest. Sample B was built parallel to the platform with the lowest number of layers and the largest melting area. Sample C was built up at a 45° angle to the XY plane.



Fig. 1 – Schematic of SLM device [12].



Fig. 2 – Geometry of specimens under investigation. Dashed line marks the area reconstructed by XCT.

2.2. X-ray tomography

The 316L specimens were scanned on a Carl Zeiss CT machine. The system was equipped with a 225 kV X-ray source with a minimum focal spot size of 7 μm and a Perkin Elmer XRD 1640 of 1024 \times 1024 pixels 16-bit amorphous silicon sensor flat panel detector. The source-to-detector distance was 1530 mm. During the measurement the sample was illuminated by cone beam X-rays (Fig. 4). X-rays were transmitted through the specimen and detected using a flat panel detector. The sample was rotated over 360° and a series of two-dimensional images were taken, to perform the 3D reconstruction.

All samples were scanned under the same measurement conditions. In order to obtain the maximum resolution of the measurement, only the center of each specimen was reconstructed (dashed line in Fig. 2). During scanning, the X-ray



Fig. 3 - Building orientation of A, B and C specimens. XCT.

beam was filtered using a 0.25 mm Cu filter in order to reduce beam-hardening effects. Selection of parameters was determined by density and size of the tested samples. The scanning parameters are shown in Table 2.

The FDK with Sheep-Logan filtering reconstruction was performed and the obtained data were processed and visualized with advanced 3D voxel analysis and visualization software package. Segmentation was performed using global and local gray value thresholds. An "Enhanced" algorithm was used for segmentation of the pores in the specimens.

Volumetric models obtained from CT reconstruction were registered (taking into account the rotation along the long axis of the specimens) in the XYZ coordinate system corresponding to their position in the chamber of the SLM machine (Fig. 3).

The XY plane highlighted in red in Fig. 3 corresponds to the plane of the build platform. This arrangement of volumetric models in the coordinate system allowed to determine the distribution and shape of pores in relation to the position of the build platform.

| Table 1 – SLM p | process parameters. | | | |
|--------------------|----------------------------|---|--|-----------------------|
| Power laser [W] | Layer thickness [µm] | Distance between scanning lines [µm] | Distance between scanning points [µm] | Exposure time [µs] |
| 97 | 50 | 125 | 80 | 400 |
| | | | | |

| Table 2 – Parameters of CT measurements. | | | | | | |
|--|--------------------|-----------------|-----------------|--------------------------|-----------------------|----------------------|
| Specimen | Voxel size [µm] | Voltage [kV] | Current [µA] | Prefiltration Cu [mm] | Exposure time [ms] | Number of projection |
| A–C | 37 | 210 | 160 | 0.25 | 2000 | 500 |



Fig. 4 - Schematic of X-ray industrial tomography.



Fig. 5 – 3D visualization of porosity of manufactured specimens in the XYZ coordinate system corresponding to their position in the chamber of the SLM machine. XCT.

3. Results of porosity examination

The summary of porosity analysis for samples A, B and C is shown in Table 3. Pore sizes were calculated as the equivalent diameter for a spherical representation of pores.

For all specimens the minimum equivalent diameter was determined as 70μ m. This means that the resolution for the presented measurement conditions is around 0.07 mm. The limited resolution does not allow to detect smaller pores. The maximum equivalent diameter varied between samples from



Fig. 6 – Pore size distribution histogram. Pore sizes were calculated as the equivalent diameter of the circumscribed sphere of pores.

2.85 mm for sample A to 1.24 mm for sample C. The highest volumetric porosity, amounting to almost 3%, was observed in sample B, contrary to sample C, which is characterized by the lowest porosity in the range of 0.15% (see Fig. 5).

The average value of porosity of the measured samples was in the range of 330 and 540 $\mu m.$ Most of the pores had a diameter of less than 1 mm (Fig. 6). Larger pores with a diameter greater than 1 mm were mainly found in samples A and B.

The sphericity (S) factor [13] has been introduced to describe the three-dimensional shape of registered pores according to the following relation:

$$S = \frac{\pi^{1/3} (6V)^{2/3}}{A} \tag{1}$$

where $S \in [0, 1]$ and for the ideal sphere S = 1.

| Table 3 – XCT porosity measurements. | | | | | | |
|--------------------------------------|---------------------|----------|------------------------------------|----------------------------------|--------------|--|
| Specimens | Equivalent diameter | | Material volume [mm ³] | Defect volume [mm ³] | Porosity [%] | |
| | Min [mm] | Max [mm] | | | | |
| А | 0.07 | 2.85 | 683.14 | 11.21 | 1.61 | |
| В | 0.07 | 2.27 | 708.56 | 21.71 | 2.97 | |
| С | 0.07 | 1.24 | 688.04 | 1.06 | 0.15 | |



Fig. 7 - Distribution of shape factor (sphericity) for specimens A, B and C.



Fig. 8 – Average pore size in the X, Y and Z directions. XY plane is the platform of SLM machine chamber.

Sphericity allows to specify the nature of the porosity that occurred within the material. Smaller pore sphericity may indicate higher directionality of the pores or lower consistency. Most of the pores detected in samples A and B have a sphericity factor at the level of 0.6. Pores found in sample C were characterized by sphericity at the level of 0.5 (see Fig. 7).

With the arrangement of volumetric models of specimens in the SLM coordinate system, it was possible to measure the elongation of the pores. Directionality of the pores, i.e. the degree of elongation, was measured by comparing their dimensions in the X, Y and Z axes. The average pore dimensions measured in the three directions are shown in Fig. 8.



Fig. 9 – Picture showing the longitudinal cross-section of specimen A, CM.

For all specimens the X and Y dimensions were larger than the dimension in the Z axis, which means that the sample was elongated along the plane of the build platform.

To estimate the porosity of specimens made by SLM technology and verify the presented XCT method in this article, the 316L samples were observed using a Confocal Microscope (CM). The study was conducted on a polished cross-section, prepared on the longitudinal section extending through the central part of each sample (along Z axis). Due to the large size of the 316L specimens, the analysis consisted of combining a series of images using stitching mode, taken at a magnification ratio of $54 \times$, which allowed to analyze the entire area of the tested sample (see Fig. 9).

Porosity analysis carried out for samples A, B and C was performed using a 2D image analysis software "ImageJ". Recorded images were subjected to binarization process, which allowed to determine the relative contribution of pores on the surfaces of the studied samples. Then, using the

| Table 4 – Microscopic porosity measurements. | | | | | | |
|--|--|--|--|--|--|--|
| ea [mm²] Porosity [%] | | | | | | |
| | | | | | | |
| 92 4.74 | | | | | | |
| 00 2.58 | | | | | | |
| 71 0.14 | | | | | | |
| 1 3 1 | | | | | | |



Fig. 10 – Metallographic cross-sectional examination of specimen A; (a) plane obtained from a CT analysis, (b) image taken with a CM – after binarization process.

formulas for the designation of Feret's diameter, the total area of examined objects (pores) was determined in two-dimensional space (2D), based on the analysis of the objects identified in the longitudinal plane. The summary of analysis of porosity for specimens A–C using microscopic methods is shown in Table 4.

Analysis of samples B and C by microscopic methods showed porosity values similar to those obtained with XCT analysis, despite the considerable difference in the resulting resolution of both methods (Table 4).These values are, respectively, 2.58% and 0.14%, and were slightly lower than those obtained through the 3D reconstruction (Table 3).

In the case of sample A the porosity obtained is 4.74%. The difference in porosity values obtained from the cross-section of sample A with respect to XCT method is almost two times higher. The difference is due to the limitation of the CT system, to not be able to distinguish defects smaller than 70 μ m, imposed by the analyzed samples. Example picture of the pore distribution in the cross-section of the sample A after binarization process with a cross-sectional plane which were obtained from the CT analysis are presented in Fig. 10.

Summarized comparison shows that the low porosity for sample A is due to the fact that the vast majority of the small defects (pores) are uniformly distributed and therefore impossible to detect by the XCT method. Analysis of specimens with small dimensions allows the reconstruction of defects with a higher resolution.

4. Conclusions

In the presented work, X-ray tomography was presented as a method for discontinuity and porosity detection, including the size, shape and orientation of pores in 316L stainless steel specimens built by SLM technology. In order to validate the nondestructive XCT method, microscopic analysis of porosity using Confocal Microscope, was conducted. The XCT reconstruction was performed with the voxel size of 37 μ m, which allowed for registration of defects greater than 70 μ m. The arrangement of volumetric models in the coordinate system allowed to determine the distribution and shape of porosity in relation to the position of the build platform. In sample A, built perpendicular to the platform, the number of pores decreased with the number of applied layers. In sample B, the highest number of pores was recorded in the middle layers as opposed

to sample C, where pores were found at the beginning and at the end of the specimen. Most pores were not spherical and their average shape factor was 0.6. The pores were arranged in a direction parallel to the applied layers, and their sizes were larger in the X and Y direction than in the Z direction.

The lowest porosity (0.15%) was found in sample C, which was confirmed by metallographic analysis. For the analysis of sample B, the difference in porosity determined in relation to the microscopic analysis was smaller and amounted to only 0.39%. Porosity analysis of sample A revealed the occurrence of defects at a level of 1.61%, but this value was found to be too low, due to the presence of small-size pores in the internal structure of the analyzed specimen.

The nondestructive XCT porosity analysis method, presented in this study, provides information on the internal defects that occur within material, in comparison to traditional methods.

Orientation of the reconstructed data in the coordinate system corresponding to their position in the chamber of the SLM machine allows to obtain information describing the shape and position of the defect in the direction of the internal structure of the test specimens. Based on the data obtained by the XCT measurement analysis, it is possible to determine the course of the cracks and diagnose places that may be the point of crack initiation, especially fatigue.

The presented porosity analysis method of specimens produced by generative technologies allows the use of tomographic analysis to assess the impact of sample orientation, which is one of the SLM process parameters.

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