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Analysis Method

Investigating the influence of X-ray CT parameters on porosity measurement of laser sintered PA12 parts using a design-of-experiment approach

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A R T I C L E I N F O

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A B S T R A C T

Polymeric parts produced by Laser Sintering (LS) present a consistent amount of pores with a wide pore size distribution. The knowledge about the porosity value and its distribution within the part allows to perform a quality control of the LS process, and gives insights in how to improve it. In this work the influence of the sample size on the porosity measurement is discussed, as well as its representativeness of the LS process itself. The reliability of porosity measurement of those parts using X-ray Computed Tomography (CT) is assessed taking into account the influence of the CT scanning parameters, the reconstruction and noise reduction algorithms using a design-of-experiment approach. The quality of the CT datasets obtained is assessed by calculating Contrast-to-Noise Ratio (CNR) and Signal-to-Noise Ratio (SNR) on representative reconstructed images. Repeatability of the measurements is also assessed along the entire workflow: CT acquisition, reconstruction and porosity measurement using commercial software packages.

1. Introduction

Additive Manufacturing (AM) processes are increasingly being used to produce end-use functional parts instead of prototypes. Laser Sintering (LS) is the most promising polymer AM technique that can aim to meet the requirements needed to become a genuine manufacturing technique. However, some aspects of the LS process still need to be improved, in order to reduce the spread in part quality [1]. The porosity of laser sintered polymeric parts is a main issue for the quality, since it directly affects the mechanical properties. Beside the total porosity, also its distribution and the maximum pore size are essential information for fine tuning the LS process and to perform an efficient quality control of the process. Nowadays, there are several porosity measurement techniques available. However different techniques often deliver different results depending on measurement parameters and conditions [2] or might be inadequate for specific samples. In order to define the reliability of porosity measurement techniques for a particular application it is often necessary to compare its results with results coming from other reference techniques. Sperings et al. [2] presents a comparison between the most common techniques for porosity measurement for metallic parts produced by SLM or Electron Beam Melting (EBM), namely Archimedes' method, CT and microscopic images. They reported CT to deliver lower porosity value compared to Archimedes' method and attributed this difference to the minimum pore size measurable by CT. For high density parts (> 98%) the difference between Archimedes' method and micrographs was estimated to be around 1%. Archimedes' method was assumed to provide a more reliable result compared to micrographs due to the fact that the complete volume is taken into account. The authors highlighted the importance of the ratio between the density of the solvent and the part in Archimedes' method, suggesting it to be lower than 1/5. This is obviously not achievable for plastic parts since their density is close to the one of the solvent. However even with a precise density measurement, there is still the problem to translate it to a porosity measurement. This step implies knowing exactly the density of the polymer, which is strongly dependent on the amount of crystalline phase present in the sample. For example, for polyamide 12 (PA12), which is the most used polymer in LS, densities of 0.99 g/cm^3 and 1.07 g/cm^3 have been reported for the amorphous and crystalline phases respectively. Depending on the sample's thermal history, the share of crystalline phase differs, leading to different final samples densities [3]. The thermal history of the laser sintered objects is function of the time spent within the LS build and of the temperature profile at which they are exposed. The time is mostly influenced by the position of the part within the build and its dimensions. In fact the higher the build the longer will be the time taken to be ready, causing parts positioned at the top or at the bottom of the build

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Table 1
CT scanning parameters and noise reduction algorithm levels used to obtain the CT datasets in the first part of the study.* 1500 projections and 1415 ms exposure have been used for all the scans.

Parameters*	Levels
Target Material	Cu; Mo
Voltage (kV)	60; 70; 90; 110
Tube Power (W)	14 (low); 18 (high)
Noise Reduction Algorithm	Yes (Noise Red.); No

Table 2
CT scanning parameters and noise reduction algorithm levels used to obtain the CT datasets in the second part of the study.** 1415 ms exposure has been used for all the scans.*** The scan at 60 kV with 3000 projections, has not be carried out due to an error in the system.

Parameters**	Levels
Target Material	Mo
Voltage (kV)	60***; 70; 90; 110
Tube Power (W)	14 (low)
Noise Reduction Algorithm	No
Projections	1500; 3000

Table 3
CT scanning parameters used to check the repeatability of the porosity measurement and the influence of the voxel size on the measured porosity content.** 1415 ms exposure has been used for all the scans.

Parameters**	Value
Target Material	Mo
Voltage (kV)	110
Tube Power (W)	14 (low)
Noise Reduction Algorithm	No
Projections	3000

to spend a significantly different amount of time inside the build. Walls and bottom of the building envelope are normally maintained at a temperature around 150 °C, to limit the temperature differences in different part of the build. However, this is not sufficient to ensure a homogeneous temperature, as shown in Ref. [4]. The non-homogeneous temperature distribution causes parts in different positions to experience different thermal histories, even if they are positioned at the same height. According to the curves reported in Ref. [3], this yields a different crystallinity in the parts, which leads to a variability in the final density of the parts. Zarringhalam et al. [5] showed that a part's crystallinity is also influenced by the process parameters used during sintering with a different energy input determining a change in the magnitude of the Differential Scanning Calorimetry's (DSC) peak related to the unmolten crystals in the core of the powder's particles. This leads to a different degree of crystallinity in the final part and consequently to a different final density. The limited knowledge about the real density of the polymeric microstructure does not allow to translate density measurements obtained using Archimedes' method into porosity measurements. This implies that porosity information for polymeric parts produced by LS have to be obtained independently from techniques used to measure density. Techniques which meet this requirement are: mercury porosimetry, light microscopy and CT.

Mercury Porosimetry (MP) is an interesting technique which provides also information about the pore size distribution. However, the technique relies on the fact that the mercury should permeate the part through the open porosity, meaning that only this kind of porosity is measured. For LS polymeric parts, which are characterized by a denser skin in the parts [6,7], this technique would deliver inaccurate porosity levels. Optical Tomography (OT) offers the possibility to measure porosity by taking images of different sections of the samples. This technique is particularly reliable and widely applied for metals, while it presents some drawbacks for polymers. In fact the sample needs to be polished after being cut, which is a critical phase for polymers because of the low Young's modulus and the low melting temperature, which can lead to local deformations and consequently to a wrong estimation of the porosity. Beside this the measurement is destructive and, in order to have a reliable measurement and a correct estimation of the pore size distribution, it is necessary to take several micrographs, which might be very time consuming. Compared to OT and MP, CT has a competitive advantage since it allows to inspect the internal defects of a sample in a non-destructive way.

Literature presents several cases where micro-CT has been used to perform quantitative and qualitative porosity measurements on laser sintered polymeric parts [8,9,21]. However different authors report to use different CT parameter sets and so far it is neither clear to what extend the different CT scanning parameters influence the porosity measurements, nor has a clear parameter selection procedure been proposed that warrants obtaining the most reliable porosity values. A parameter that has certainly to be considered is the resolution of the scan, which is related to the voxel size, and affects the smallest pore size that can be detected or resolute. This resolution depends on the magnification applied in the scan, which in turn strongly depends on the size of the sample: in order to image the sample it needs to fit fully inside the X-ray cone beam. The typical minimum defect size one aims to detect within the part determine the maximum voxel size, hence the maximum sample size.

Therefore, a tradeoff must be found between the sample representativeness of the LS process and the accuracy of the measurement itself, which implies the definition of a minimal pore size which should be the focus of the measurement. An important step in the measurement process is also the selection of the actual CT scanning parameters. Following the approach suggested by Kerckhofs [10] a possible way to

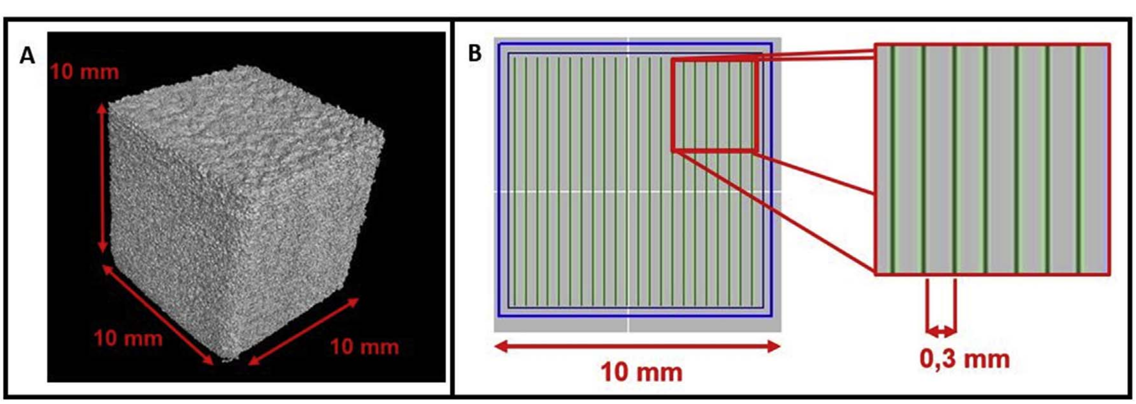

Fig. 1. (a) Voxel model of the 10 mm side cubic sample used during the investigation. (b) Scanning pattern used to manufacture the PA12 cubic sample by LS.