

DETECTION OF POWDER-BINDER SEPARATIONS DURING MICROPIM BY SYNCHROTRON COMPUTER TOMOGRAPHY

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ABSTRACT

Powder binder separations are a well known phenomenon in powder injection moulding (PIM). Due to the high surface to volume ratio in MicroPIM surface effects and also separation effects get more and more dominant. Hence, new characterization methods have to be used to detect local particle density also in micro dimensions. Therefore in the present work synchrotron computer tomography and subsequent image analysis were used to measure local particle size distributions and local particle densities in a specimen produced by MicroMIM. The feedstock consisted of a wax-polymer binder and a fine gas atomized steel powder (17-4PH) with a powder loading of 63 vol.%. A simple micro bending bar structure was manufactured and analyzed with synchrotron CT. With the high voxel resolution of 0.28 μm even single particles were visualized in the green body. Image analysis allowed for the local measurement of particle size distributions and showed that no significant size separation was introduced during injection moulding. Nevertheless, the local particle density distribution over the cross section of the bending bar structure showed massive separations with maximum density differences about 20 vol.%. The qualitative distribution corresponded very well with the theory of particle migration introduced by Phillips et al..

INTRODUCTION

Powder Injection Moulding (PIM) of metallic and ceramic compounds is an established industrial process for the production of complex shaped parts in medium to large lots. Due to the increasing demand of miniaturized devices especially in the fields of medicine, telecommunication and engineering several workgroups deal with the development of the Micro Powder Injection Moulding (MicroPIM) process [1,2,3]. The understanding of the whole process chain becomes more and more important in micro dimensions because increased requirements considering low surface roughness, reproduction accuracy or mechanical properties in the sintered state have to be met.

Compounds for PIM, so called feedstocks, consist of a polymeric binder, which acts as a flowing vehicle, and powder with loadings typically in the range of 50-70 vol.%, depending on powder size and morphology. During compounding different surface active agents (surfactants) are added to the formulation to ensure a homogeneous distribution of the powder particles in the binder matrix. During injection moulding a separation between the particles and the binder can occur and consequently lead to an inhomogeneous density distribution. During the sintering step a non isotropic power distribution

is likely to result in distortions or local porosity in the final part and therefore affect its functionality or reliability.

These separations may be introduced by a steric hindrance of the particles between each other [4]. This happens if the particle size is in the range of the structural details of the mould which must be passed by the feedstock during mould filling. Consequently structural details can be minimized if the size of the powder particles used is significantly reduced.

Beyond that, anisotropic powder particle densities can also be introduced by flow effects during mould filling. This phenomenon is known under the synonym ‘particle migration’ and is mathematically described by Phillips et al. [5]. The effect is strongly influenced by the boundary conditions during injection moulding and the properties of the feedstock material. Especially high shear rates and shear rate gradients as well as a strong dependence of feedstock viscosity on powder loading increases particle flux from regions of high shearing to regions of low shearing. Powder particle size also affects particle migration because large particles have a higher collision probability than smaller particles and therefore tend to drift away from regions of high shearing more distinctive. Therefore especially micro metal injection moulding (MicroMIM) is very susceptible to separation effects because of the extremely high shear rates during injection moulding and the comparatively coarse particles in the micrometer range. Based on this physical model new simulation tools can theoretically calculate the local powder density during mould filling [6,7]. However, the experimental detection of local particle densities is difficult but necessary to calculate the flux parameters for the different feedstock materials and to verify the simulation results.

Different experimental approaches for the detection of particle densities are discussed in literature. Scanning electronic images of breaking edges or grinded surfaces can be used to judge for the homogeneity of powder filled compounds but in most cases cannot be used to extract local quantitative results [8,9]. Many authors tried to use nuclear magnetic resonance (NMR) spectroscopy to visualize density differences in specimens [5,10,11,12,13]. Only simple geometries can be studied and the achievable resolutions are too low to adapt this method on micro parts. Zorzi et al. used x-ray absorption images for the detection of inhomogeneities in powder filled parts [14]. These studies were carried out with simple geometries and resolutions in the millimetre range. Nevertheless, standard x-ray computer tomography (CT) is one of the most promising methods for the detection of local particle densities because of the continuous enhancement of CT equipment and technology in the past years which allows for resolutions in the micrometer range [15,16].

Beside standard x-ray CT, synchrotron CT establishes itself as characterization method in the field of materials research [17,18]. The highly parallel beam characteristic and the possibility to use monochromatic radiation in synchrotron CT makes quantitative reconstructions based on the absorption coefficient possible.

Rabe et al. use synchrotron x-ray computer tomography (CT) to detect imperfections and density differences in green and sintered ceramic compacts [19].

In the present work, synchrotron CT was used to visualize fine 17-4PH particles in a simple bending bar specimen produced by micro powder injection moulding. The tomogram was used to calculate local particle densities and particle size distributions by semi automated image analysis.

MATERIALS AND METHODS

Feedstock preparation:

A typical wax-polymer binder system used in MicroPIM was selected for the studies presented here. The binder mainly consisted of a low density polyethylene, a paraffin wax and stearic acid as a dispersant. A separated 17-4PH steel powder produced by gas atomization was used. Figure 1 (left) shows the powder size distribution measured by laser scattering. With a median particle size of about 6.5 μm the powder is fine enough for micro specimens and its spherical morphology (Figure 1 (right)) allows high powder loadings during compounding. The particle shape also makes subsequent image analysis of the tomographic pictures much easier.

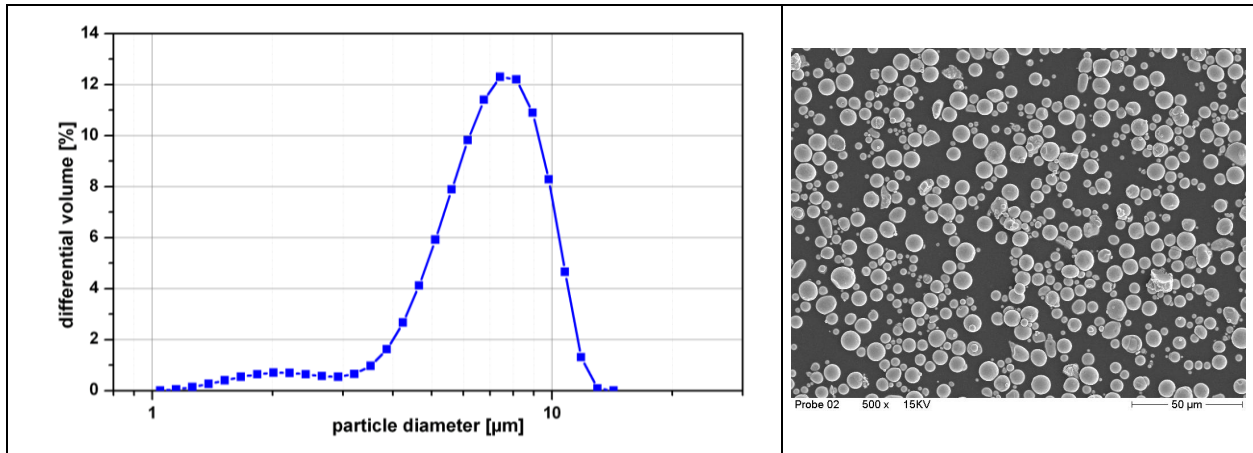


Figure 1: (left) Differential powder particle diameter distribution of the used 17-4PH powder. (right) SEM image of the powder shows its spherical powder morphology.

The feedstock was kneaded for one hour at a temperature of 125°C with a constant rotation speed of 30 rpm using a Brabender W50 EHT compounding unit. After preparation the material was granulated for further processing. The powder loading of the feedstock was 63 Vol.%.

Injection moulding:

For the tomographic experiments a simple standardized flexural test geometry developed for material testing of ceramics in micro dimensions was chosen. Figure 2 shows a 3D-model of the gate system and the micro specimen. The flexural specimen has a quadratic cross section with a side length of 260 μm and an overall length of 3250 μm. The gate of the mould is at the far end of the geometry so that the melt has to cross the complete gating and demoulding system and fills the microstructure at the end of the filling stage.

The injection moulding experiments were carried out on a MicroSystem50 plunger injection moulding machine distributed by Battenfeld GmbH, Austria.

A variothermal process with a mould temperature during injection of 75°C and a demoulding temperature of 50°C was selected. The plunger speed was set to 250 mm s⁻¹.

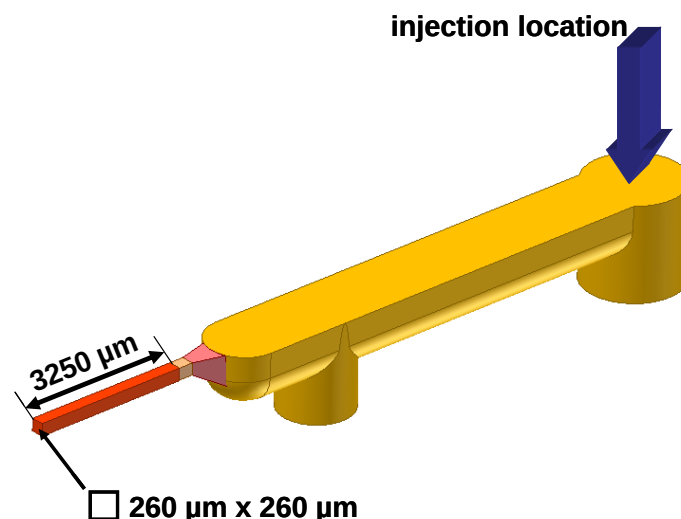


Figure 2: 3d-model of the micro bending bar specimen used for the experiments.

Synchrotron Computer Tomography:

The principle setup for synchrotron CT is shown in Figure 3. The sample is mounted on a highly precise stage which allows for the positioning of the specimen in the synchrotron beam. The parallel

and monochromatic beam enters the cross section of the sample and is locally absorbed by the material. The transmitted x-ray beam is converted to visual light by a thin mono-crystal scintillator. Thereafter the image is projected on a charge-coupled device (CCD) camera by an adequate optical system. Rotation of the sample by at least 180° around the z-axis (ω) allows for the reconstruction of a three dimensional tomogram. The experiments were carried out at the ID19 beamline at the european synchrotron radiation facility (ESRF) in grenoble [20].

The basic feedstock properties and the parameters for image recording are summarized in Table 1.

Table 1: Summary of the basic feedstock properties and the beamline parameters for CT.

binder composition	50% polyethylene, 45% paraffin, 5% stearic
particle size	$\sim 1\text{-}10\ \mu\text{m}$
powder loading	63 Vol.%
voxel size	$0.28 \times 0.28 \times 0.28\ \mu\text{m}^3$
beam parameters	monochromatic , $E=27\ \text{kV}$
angle increment	0.15° (1200 absorption images)
camera/optic	FReLoN (2048x2048 pixel chip)

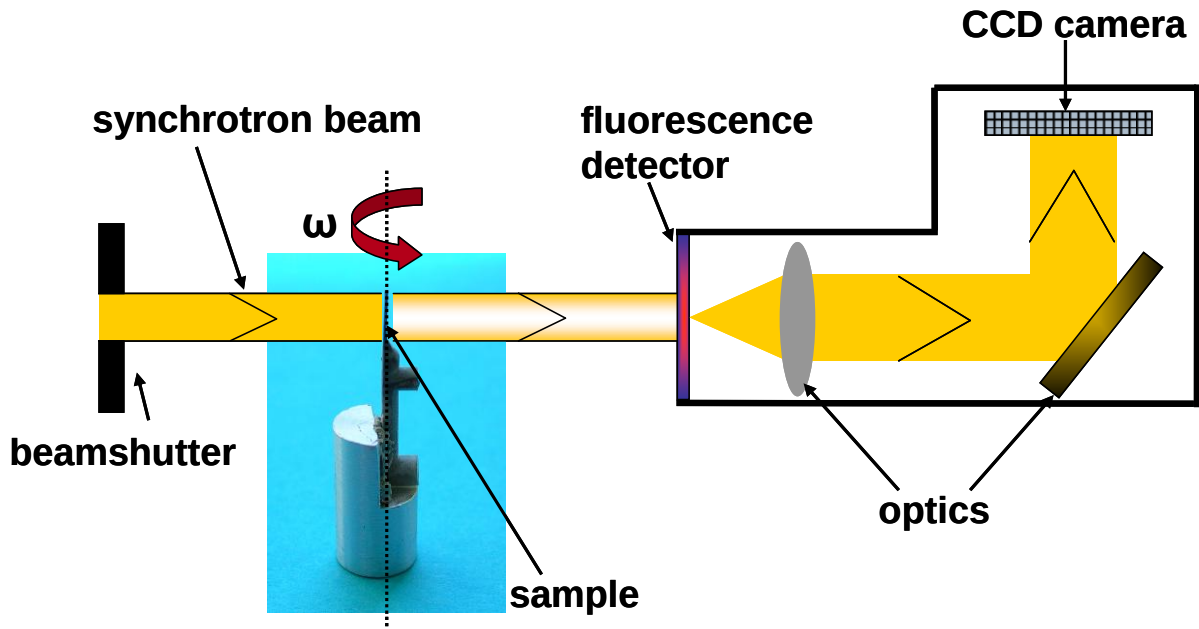


Figure 3: Principle setup for computer tomographic experiments using synchrotron radiation.

RESULTS AND DISCUSSION

Figure 4 shows the three dimensional tomogram of the micro flexural test structure. Due to the extremely high real resolution of about $0.6\ \mu\text{m}$ and the non diverting x-ray beam only a small part of the micro specimen can be visualized in one experiment. Here the region between the gating area and the beginning micro bending geometry is shown. The feedstock entered the structure from the right side and filled the channel in the x-direction.

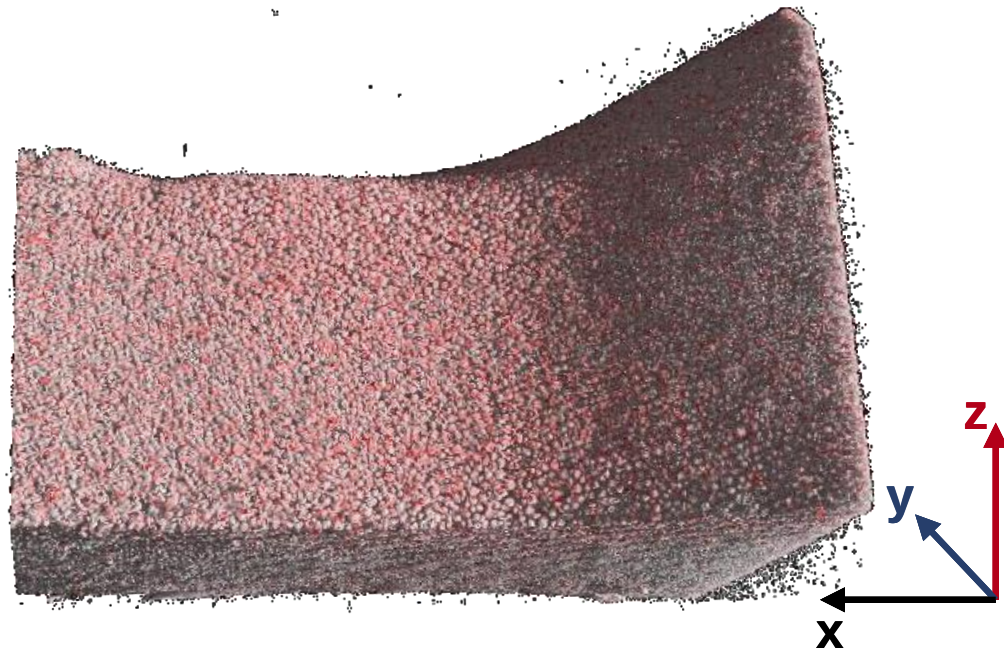


Figure 4: Three dimensional tomogram of the micro bending bar structure.

This tomogram shows the possibilities of synchrotron CT. Even the smallest particles of the powder used can be visualized with this method and therefore allows a view into the microstructure of MicroPIM parts. This image enables a qualitative inspection of the micro part and the detection of pores, inclusions and other imperfections. One way for a quantitative analysis is to cut the three dimensional tomogram into two dimensional slices perpendicular to the x-axis which then can be investigated by methods of image analysis afterwards.

The approach for image analysis is outlined in Figure 5. The Figure shows a part of a two dimensional slice perpendicular to the flow direction x (y-z plane from Figure 4). In the upper left corner the original 16 bit picture is shown (Figure 5, a)). Due to the different absorption coefficient of powder and binder the different materials are represented by different grey values. For image analysis typically binary pictures are used. Therefore the grey value distribution has to be divided in a region which corresponds to the powder material and will further on be displayed by black pixels and a second region which corresponds to the binder and the surrounding material and will be replaced by white pixels (Figure 5, b)). The optimum threshold value can be determined by a method named after Otsu [21].

The binary picture on the upper right cell of Figure 5 shows high noise i.e. diffuse particle boundaries which can be minimized by using a median filter on the picture (Figure 5, c)). The filtered image shows interconnections between the particle sections because of the limited resolution of the reconstruction and because of the median filtering. However, for particle size analysis the connected particle sections have to be completely separated from each other. This can be accomplished with the so called watershed algorithm provided by image processing and analysis software tools like 'ImageJ' [22] (Figure 5, d)).

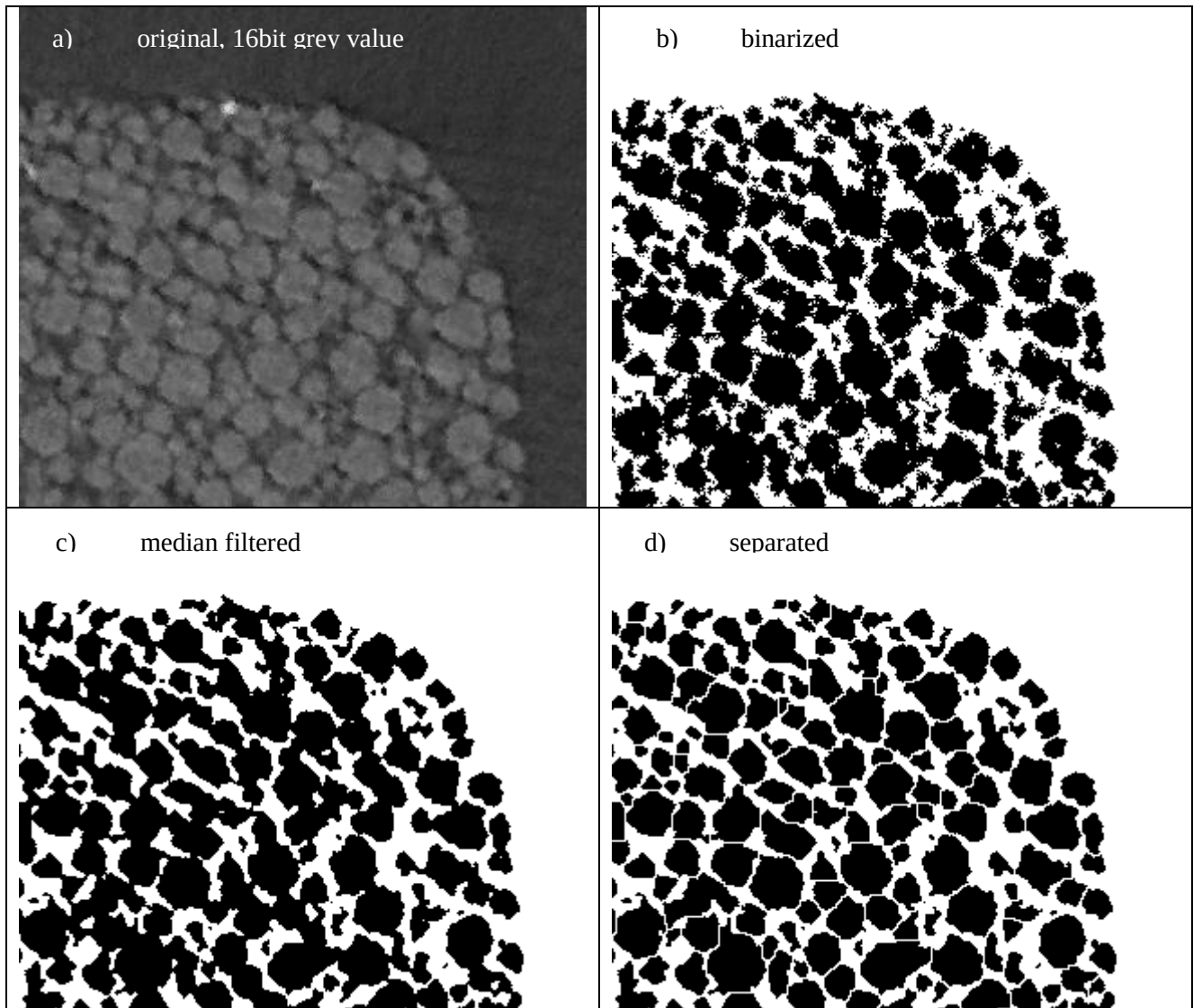


Figure 5: a) Original grey value section through the 3d tomogram. b) Binarized image. Black pixels correspond to powder material and white pixels to binder and surrounding material. c) Noise in the picture is minimized by using a median filter. d) Connected particles are separated with the watershed algorithm.

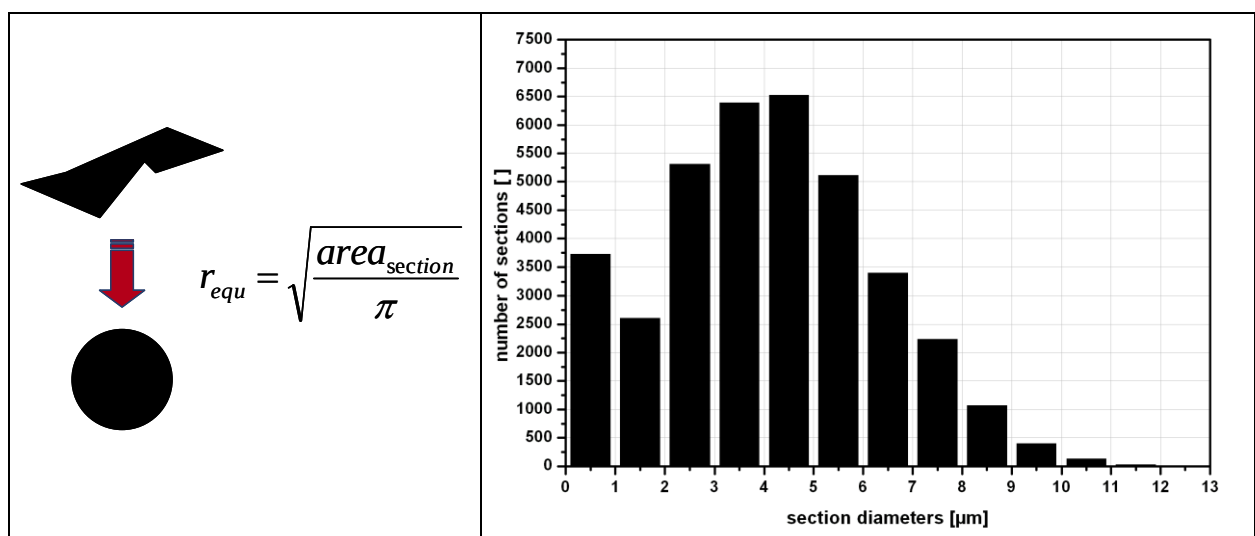


Figure 6: (left) Calculation principle of the equivalent radius of an irregular shaped particle. (right) Example of a particle section diameter distribution of a stack of 15 slices.

The area of the separated particle sections can now be calculated. Particle size distribution are typically based on the diameters of equivalent spheres. Hence the particles detected are not perfectly spherical the sectional areas have to be converted to the corresponding equivalent radius of a perfectly shaped sphere. The conversion is schematically shown in Figure 6 (left). The section diameter distribution of a stack of consecutive slices perpendicular to the flow direction is shown in Figure 6 (right). The class width of the distribution is 1 μm . Due to the fact that sections through spheres are not normally distributed it is not possible to directly convert the section diameter distribution to the particle size distribution. This problem is known as the Wicksell's problem and can be solved by a mathematical algorithm described in literature [23,24,25].

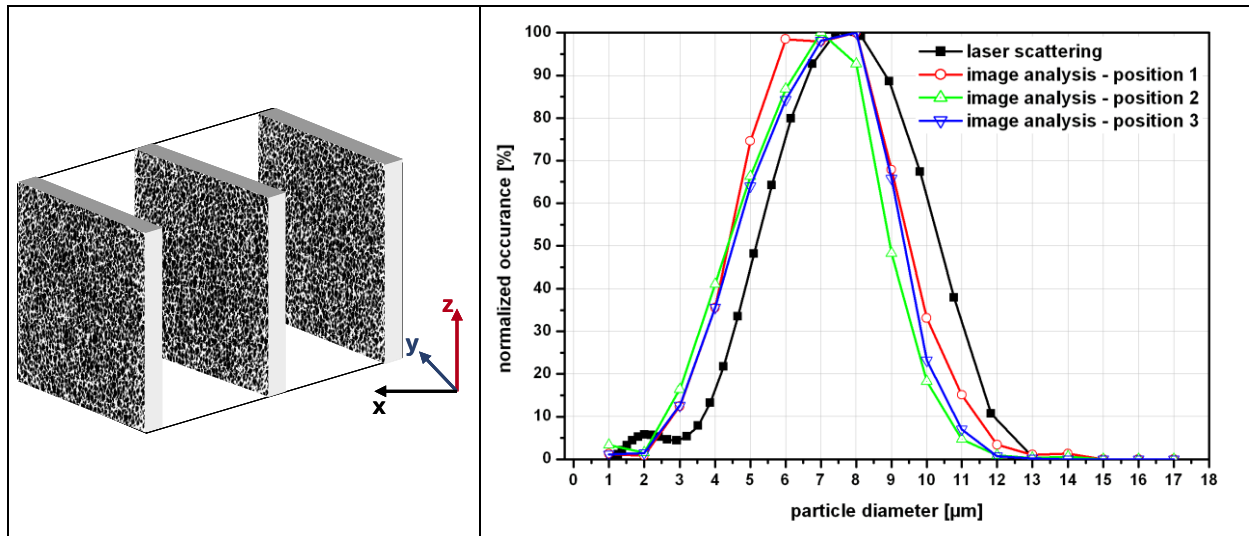


Figure 7: (left) Stack principle of the reconstruction algorithm. A stack of 15 slices in x direction are cut out of the tomogram and are used for image analysis. (right) Comparison of the reconstructed size distributions at the binning, the middle and the end of the tomogram with the results from laser scattering.

Figure 7 shows the results of three different stacks of 15 slices through the tomogram and its corresponding local particle size distribution calculated out of the sectional data. The stacks are located equidistantly along the x-axis i.e. flow direction at the beginning, the middle and the end of the reconstructed volume. A stack of 15 slices corresponds to about 40000 sections of particles. These amounts of sections are necessary to get resilient statistical data to calculate the particle size distribution. In Figure 7 (right) the normalized distributions are directly compared to the data measured by laser scattering. The differences between the calculated powder size distributions from the three considered stacks are not very significant and show that no deagglomeration and/or particle size separation along the short flow section of the sample occurred. The calculated distributions correlate very well with the data of the laser scattering experiment but show a slight shift to smaller particle sizes. One possible explanation is a real particle size separation during injection moulding. More likely the shift is caused by the binarization or over-segmentation by the watershed algorithm.

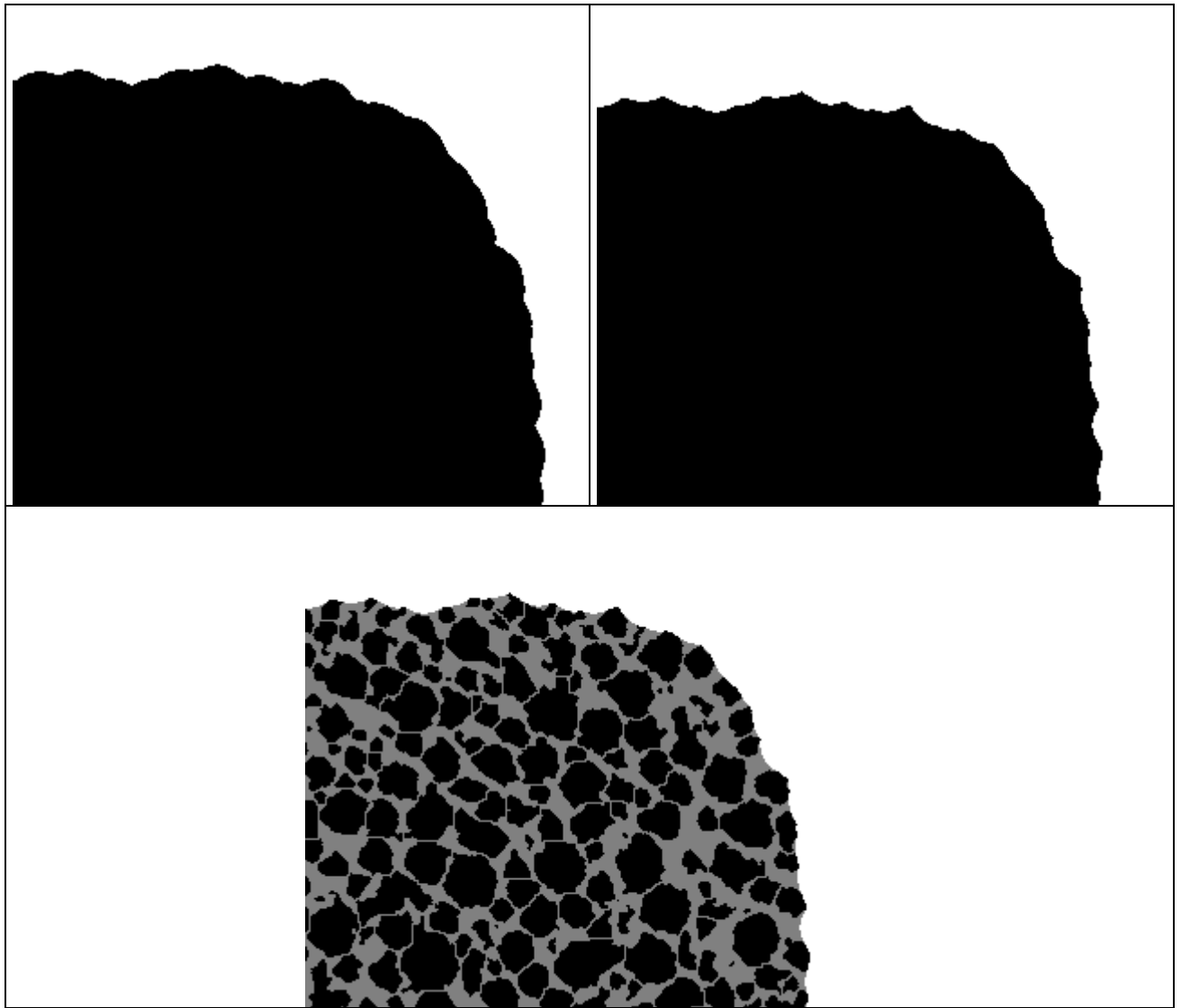


Figure 8: Approach of image analysis to calculate the cross sectional area of the sample out of the separated particle image. (upper left) The particles are dilated by a constant radius. (upper right) The resulting black area is eroded by the same radius to get the cross sectional area. (lower middle) This image shows the superimposed original image with the cross section.

Beside the local determination of the particle size distribution the calculation of particle density distribution is important because it directly affects the flow behaviour during mould filling and distortion during sintering. The local particle density in a slice is calculated out of the black to white ratio of the pixels normalized to the sample cross section. Because binder and surrounding area cannot be differentiated during binarization the cross sectional area of the sample has to be calculated separately. The approach is shown in Figure 8. For every black pixel in the image the surrounding horizontal and vertical pixels are also converted to black. The number of iteration steps necessary to fill all gaps between the particle (black) sections is counted. The resulting area relates to the cross section of the sample but is expanded by the dilation. Consequently, to get the correct cross section, the area has to be eroded using the counted number of iterations. A superposition of the original slice and the calculated cross section shows a good correspondence (lower middle).

With this image preparation it is possible to calculate the local particle density slice by slice in x-direction (Figure 9). This section of the sample shows that only a slight fluctuation in particle density can be measured along the flow direction. The calculated density corresponds very well with the reference powder loading of 63 vol.%. The slight difference can be explained either by a steric separation during injection moulding or inaccuracies during image preparation. Nevertheless a relative comparison is possible because all images were prepared by the same image algorithms.

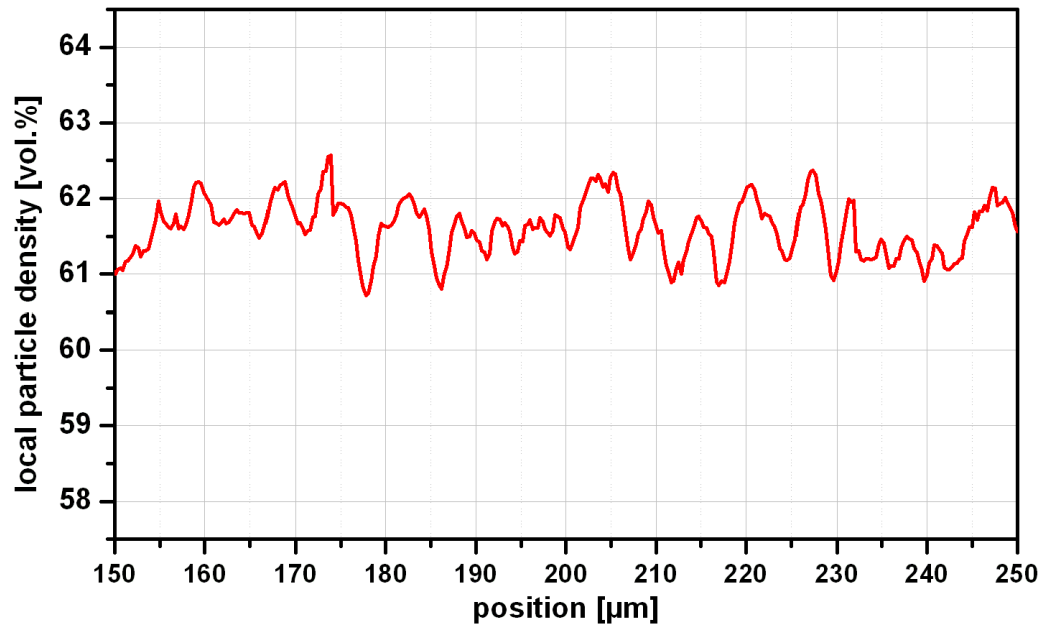


Figure 9: Local particle density along the flow direction (x-direction) calculated out of the black to white ratio of the corresponding y-z-sections.

Of even more interest is the particle density in flow direction over the cross section (y-z-layer) of the flexural specimen. Therefore a stack of one hundred slices is divided in equidistant regions of interest (ROI) of the same size. In these ROI the average density is calculated and plotted in a gradient graph (Figure 10).

The plot shows massive density differences over the cross section of the sample. At the border regions the local density drops to about 58 vol.% and at the edges the local density lies below 50 vol.%. In the middle of the cross section the local density reaches values of 70 vol.%. Hence, the resulting maximum density difference lies around 20 vol.% which can have an influence on the flow characteristic during mould filling and on the sintering behaviour.

These results correspond very well with the theoretical considerations. In the edges and at the border regions the highest shear rates and the highest shear gradients occur which leads to a particle migration in the middle of the structure where the lowest shear rates are expected.

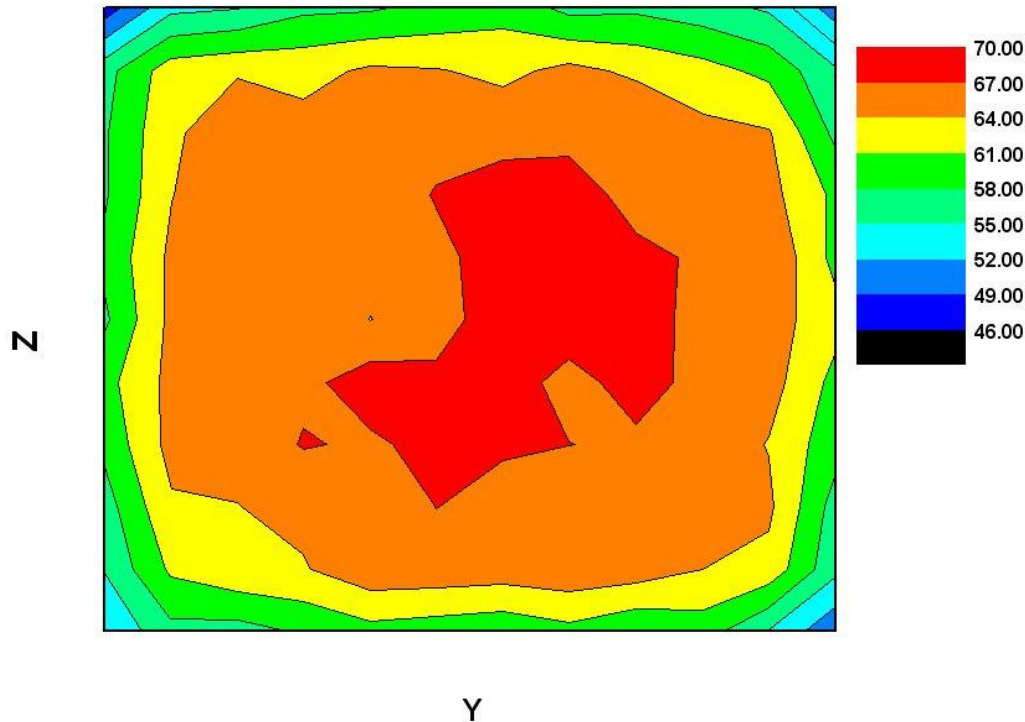


Figure 10: The gradient plot shows the local particle density across the bending bar structure. The local density was calculated from the average of one hundred slices.

CONCLUSION

In the present work synchrotron CT was successfully used to calculate local particle distributions and local particle densities in a micro structured sample prepared by MicroMIM. The determined particle size distributions over the whole cross section correspond very well with the results from laser scattering. The particle density in flow direction shows no significant fluctuations and compare very well with the reference powder loading of 63 vol.%. In contrast to this, the density distribution in a cross section of the sample shows massive powder-binder-separations with maximum differences of about 20 vol.%. The qualitative distribution corresponds with the theoretical considerations and shows that the lowest densities in the parts result in regions of highest shearing during mould filling. Due to the fact that the surface to volume ratio in micro parts is much larger than in standard MIM parts these separations can get even higher if smaller structures are considered. To really understand this phenomenon further experiments are necessary to calculate the flux parameters and implement these models in appropriate simulation tools. This approach is even more necessary because image analysis gets more difficult if complex geometries have to be measured. Hence, the understanding of particle migration and the successful implementation in simulation tools allows for an optimization of the MicroPIM process and maximized the accuracy.

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