Powder particle density and distribution in injection moulded green metallic and ceramic micro parts investigated by synchrotron microtomography

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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. Therefore in the present work synchrotron microtomography and subsequent image analysis were used to measure local particle size distributions and local particle densities in samples produced by MicroPIM. The research is based on former experiments at ANKA which are described in literature [1]. The feedstocks used for the presented experiments consisted of a wax-polymer binder and a gas atomized 17-4PH steel powder with a median particle size of 6.5 µm and a powder loading of 63 vol.%. The specimens produced by PIM have a quadratic cross section with an edge length of about 260 µm and an overall length of more than 3 mm. First measurements at ESRF with a voxel edge length of 0.28 µm made the calculation of local density distribution and local particle size distribution possible. Using the MAVI tool [2] the particle size distribution after injection moulding was calculated and corresponds well with the results obtained by laser scattering methods of the starting powder (Figure 1).

Several further experiments accomplished at ANKA (TOPO-TOMO) allowed for the characterisation of systematic variation of the PIM process parameters on separation effects [Figure 2]. With respect to measurement time a voxel size of 0.9 µm^3 and accordingly a coarser steel powder with a median particle size of about 16 µm was used.

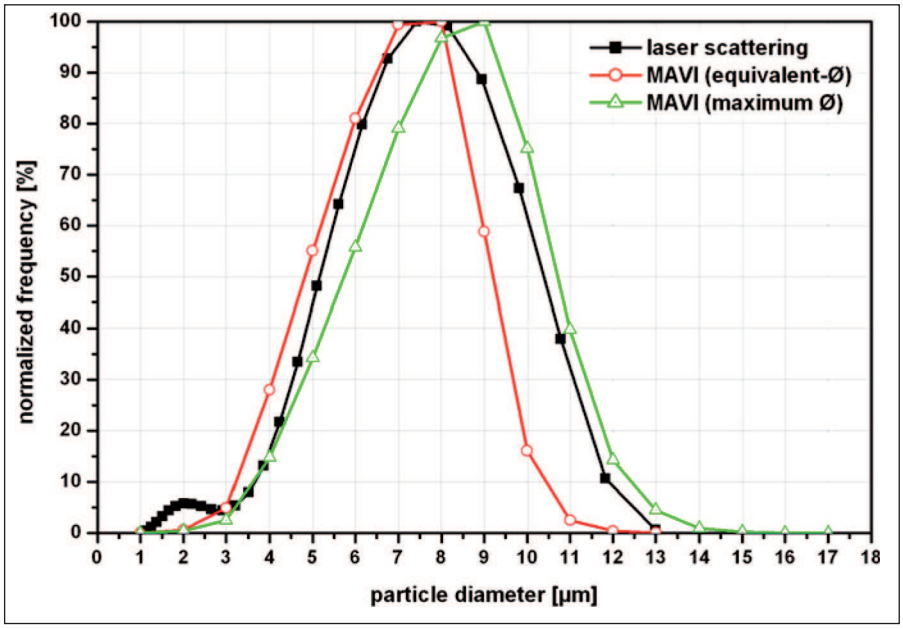

Figure 1: *Particle size distribution of the starting powder measured with laser scattering and the results from image analysis using the MAVI software.*

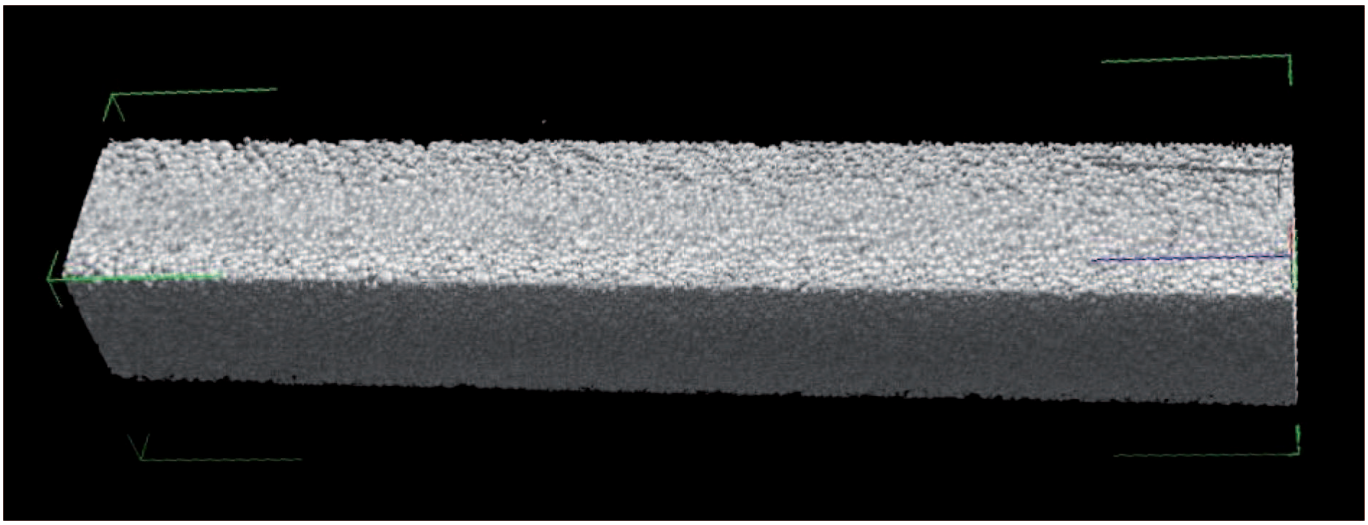

Figure 2: *3D tomograph of a micro test geometry measured at ANKA.*

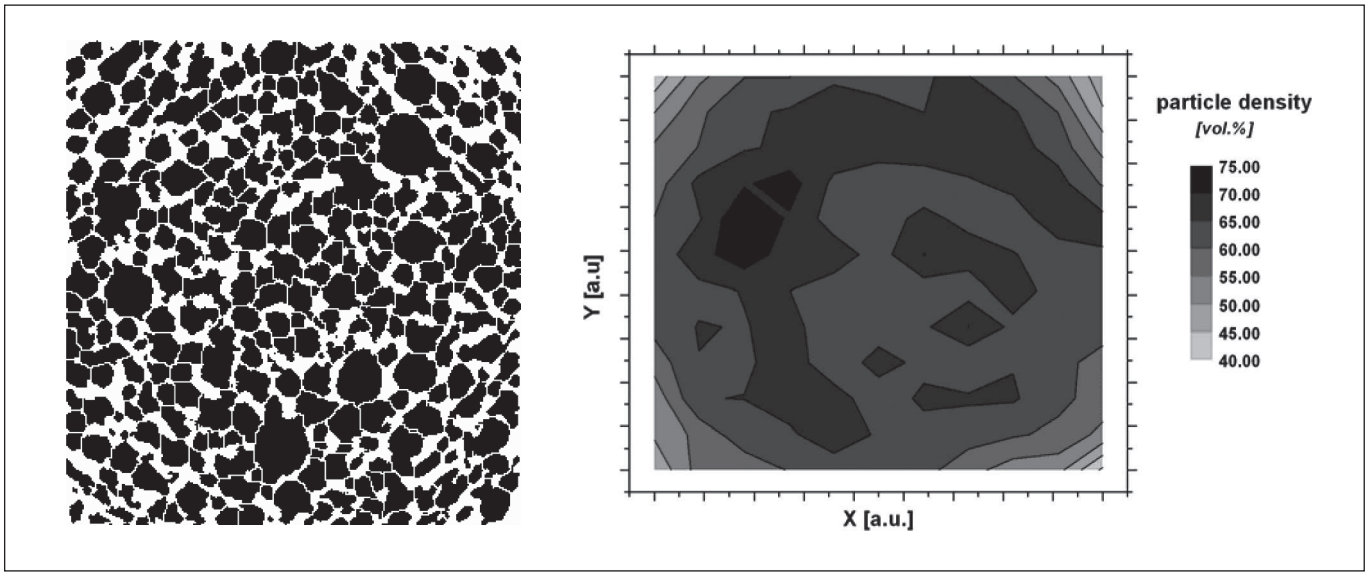

Figure 3: *(left) Filtered slice through the cross section of the micro specimen. (right) Calculated local density distribution calculated from a stack of 240 slices.*

Figure 3 shows one binarized slice through the sample after image processing (median filtering, watershed). A stack of 240 similarly processed slices were used to calculate the local particle density through the cross section of the sample. Significant density gradients and variations can be detected using this method.
The results allow for an explanation of distortions during sintering caused by an inhomogeneous density distribution as well as pores. For modelling these separation effects further tomographic experiments at ANKA are necessary to calculate particle flux parameters which can be implemented in appropriate simulation tools.
In order to obtain the high resolutions necessary for image analysis the sample handling and preparation as well as local drift of the specimen because of thermal expansion of the equipment has to be optimized.

Acknowledgements

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References

[1] Heldele, R., Rath, S., Merz, L., Butzbach, R., Hagelstein, M., Haußelt, J.: X-ray tomography of powder injection moulded micro parts using synchrotron radiation; Nuclear Instruments and Methods in Physics Research B 246 (2006), pp. 211 – 216.
[2] http://www.itwm.fraunhofer.de/bv/projects/MAVI