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Using 4D-Imaging to describe the impact of the microstructure on sublimation front patterns

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Freeze-drying is a gentle drying technique for high value products such as pharmaceuticals. The process can mainly be separated in three steps. (1) The freezing step, here the actual microstructure of the final product can be formed. (2) The primary drying removes the ice out of the product by sublimation. (3) The secondary drying is used to remove the bound water inside the matrix by desorption to get a stable product. While freeze-drying is still a very time and energy consuming process, it is still the aim to accelerate the process [1]. Here one important factor is the microstructure of the product [2]. In literature often the influence of the pore size is described and stated that bigger pores lead to a faster drying because of the lower mass transport resistance. However, other structural parameters such as pore shape and orientation are neglected. Recently we could demonstrate the impact of pore shape and orientation on the movement of the sublimation front in 2D [3]. In this work, we will present a more detailed study on the impact of the microstructure on drying kinetics by using in-situ freeze-drying experiments. For that freeze-drying experiments were conducted with a custom made freeze-drying stage in the 4D tomography system DynaTom. Here, continuous tomography scans were conducted during the freeze-drying experiments to observe the movement of the sublimation front in 3D. At the end of each experiment a high resolution scan of the final microstructure was made. To generate different microstructures, different freezing protocols were used (different solid concentrations and annealing treatment). The experiments are conducted at -15℃ shelf temperature and the chamber pressure is 10 Pa. By the use of an inhouse MATLAB and python script the microstructure in terms of pore size, shape and orientation as well as the movement of the sublimation front are analyzed. It can be demonstrated that depending on the pore shape, either pore size or orientation can be the more dominating factor.

Participation

In-Person

References

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