3D Reconstruction of Porous Polymers using FIB-SEM

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3D reconstruction of microstructures of materials by serial sectioning using a combined focused ion beam with scanning electron microscopy (FIB-SEM) is a well-established procedure. However, beam sensitive and poor conduction materials are challenging. The electron beam may give rise to accumulation of charges at the surface due to poor electrical conduction and cause image distortion. Another challenge is that the ion beam can destroy the sample by surface melting or induce curtaining effects [1]. The presence of pores introduces a third challenge. The pores cause difficulties during the subsequent 3D reconstruction due to their intensity distribution in contrast to the constant level to the surrounding planar material and where the intensity level from the planar material may overlap with the intensity distribution of the pores.

We have addressed the three challenges above in coatings for controlled drug release. A drug core is coated with a phase-separated polymer film [2]. The films consist of ethyl cellulose (EC), water insoluble, and hydroxypropyl cellulose (HPC), water soluble, mixed with ethanol as solvent. A phase-separation occurs when the ethanol evaporates. The two polymers form a bi-continuous network where the HPC can be removed by leaching the films in body fluids. Provided that the HPC phase forms a continuous network the resulting porous film provides transport paths for the drug. Therefore, the drug release partly depends on the HPC volume fraction [3]. Other factors that affect the drug release characteristics are the connectivity and the tortuosity. Previous studies have shown an onset of drug release at a volume fraction of 22% of HPC. Lower volume fractions give lower film permeability while the permeability increases for higher volume fractions. Therefore, we chose to study the porous network in leached EC/HPC films with the volume fractions 22 % (HPC22), 30 % (HPC30) and 45 % (HPC45).

We chose to work with the electron beam acceleration voltage 700 V and current below measurable value of 1 pA. The lower voltage of 700 V was selected to enhance the surface sensitivity. The lower electron beam current reduced the charging effect. In addition, a carbon gas was injected prior to neutralise the charge accumulation at the surface and thus avoid image distortions [4]. The surface melting and curtaining effects, caused by the ion beam, were removed by selecting the current 100 pA and the voltage 30 kV. The challenge concerning the binarization was solved by developing a self-learning algorithm that can separate the pores from the planar surface despite the overlap between the corresponding intensity distributions. The binary data are used for the 3D reconstruction and the determination of pore size distribution, connectivity, tortuosity, morphology and to perform diffusion simulations. Finally, this information is used to correlate the microstructure with mass transport through the films.

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References

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