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# Polymer to Carbon Transition: In Situ Pyrolysis of 3D Printed Microstructures

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Carbon has been well known as a compelling material to compete with silicon in the field of microchips. One promising method to fabricate micro- or nanoscale carbon architectures is to pyrolyze a pre-patterned polymer precursor [1]. Pyrolysis is, therefore, a crucial processing parameter. However, a mechanistic understanding of the pyrolysis process for sub-micrometer polymer geometries is still missing. In particular, for the temperature regime, where the highest mass loss and, in turn, the highest shrinkage is observed, a detailed analysis of the pyrolysis-induced shrinkage kinetics, geometry dependence shape deformation, and the roles of atmosphere and surface area is still lacking [2].
In this work, we aim to fill this knowledge gap and provide a comprehensive understanding of morphological deformation upon pyrolysis. Here, we report a systematic study of in situ pyrolysis process by varying environment pressure, heating temperature, and sample geometry (surface-to-volume ratio) in an environmental SEM (ESEM). We use direct laser writing of IP-Dip photoresist to print microstructures directly on MEMS heating chips [3]. We focus on the early stage of carbonization from 450 to 550 ℃, where polymer precursor experiences the greatest mass loss and structural shrinkage. The structural changes are directly tracked by secondary electron imaging.
We reveal the pyrolysis kinetics illustrating the temporal and temperature dependency of the deformation. When evaluating the data pool by generating model-free master curves, we fully describe the dynamic process and extract the effective activation energy (Ea). After changing the environmental conditions, the shrinkage behavior turns out to be fundamentally different, largely kinetically hindered. A prevalence of the aspect ratio on kinetics and the final size becomes apparent, causing a dramatically lower Ea.
To complete the picture of the structural changes during pyrolysis-induced transformation, our ongoing experiments tackle focused ion beam (FIB) micromachined cross sections of the microstruts after isothermal exposure using scanning transmission electron microscopy (STEM). Not only morphological changes can be characterized but also local structural and chemical information by electron energy loss spectroscopy in 4D-spectrum images (EELS-SI).
In conclusion, our in situ and scale-bridging study paves the way to a thorough understanding of morphological mechanisms upon pyrolysis and correlated property strengthening. The precise tuning of functional metamaterials by pyrolysis will facilitate the development of industrially relevant carbon device fabrication.

## Category

Solid State (Experiment)

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