

Direct observation of intermediate phases of pyrolytic carbon by atomic force microscopy

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Pyrolytic carbon was deposited on planar substrates in a hot-wall reactor at 1100 °C. For the investigation of the early stages of deposition, residence times up to 3.2 s and deposition times between 5 and 90 minutes were chosen. Methane / argon mixtures at a total pressure of 100 kPa were used. Scanning force microscopy-based methods were applied to achieve a material contrast between silicon substrate and individual carbon islands revealing two types of islands by different chemical contrast. This observation can be interpreted in terms of an intermediate phase of pyrolytic carbon according to a model by Hüttinger et al. [1]: for deposition in a regime where the nucleation mechanism dominates, an intermediate phase of pyrolytic carbon is predicted which is expected to have deviating mechanical properties. This agrees with our results of island removal experiments carried out using atomic force microscopy where defined lateral forces are applied to the islands: two types of carbon islands are found that differ significantly in the shear stress necessary for island removal.

Additionally, complete pyrolytic carbon layers were deposited on cordierite channel structures at methane pressures between 4 kPa and 50 kPa and residence times up to 1 s. Atomic force microscopy of these layers shows hillocks with typical diameters between 5 µm and 50 µm. At sufficiently high methane pressures where adsorption saturation is reached [2], additional carbon structures exhibiting different chemical contrast at typical length scales between 100 nm and 5 µm were found additionally on top of the hillocks. This observation is a direct experimental evidence for the existence of an intermediate phase of carbon as postulated for deposition in the nucleation mechanism.

[1] Z.J. Hu, K.J. Hüttinger. Carbon **40** (2002), 617-636.

- [2] Z.J. Hu, W.G. Zhang, K.J. Hüttinger, B. Reznik, D. Gerthsen. Carbon **41** (2003), 749-758.