Investigation of the early stages of the chemical vapor deposition of pyrolytic carbon by atomic force microscopy

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Introduction

Pyrolytic carbon is used in a wide variety of applications, e.g. as coatings or in carbon/carbon composites due to its mechanical properties and its temperature stability. Therefore, the understanding of the chemical vapor deposition process of pyrolytic carbon is of great technological as well as scientific interest. The present paper is concerned with studies of the early stages of chemical vapor deposition of pyrolytic carbon.

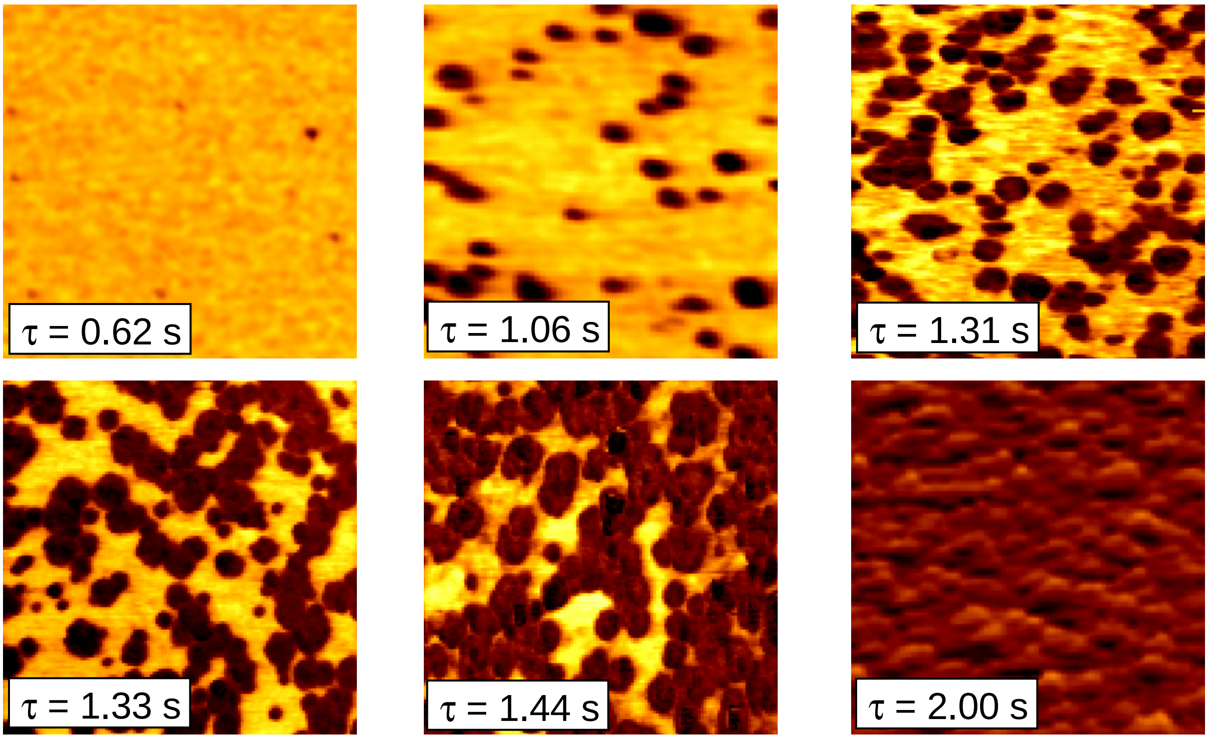

Figure 1: Pyrolytic carbon deposited on silicon, deposition time 5 min, τ is the residence time. Force modulation microscopy, modulation amplitude 6 nm, scan area 1 µm x 1 µm.

For this purpose, pyrolytic carbon was deposited on planar silicon substrates from methane in a hot-wall reactor at a temperature of 1100 °C. Methane / argon mixtures were used at a total pressure of 100 kPa (methane pressure 10 or 20 kPa). Deposition times were chosen between 3 and 90 minutes. The gas flow was adjusted to reach maximum residence times up to 3.2 s where the residence time is the time the gas needs from its entrance of the hot reaction zone to the point where the pyrocarbon deposition takes place. The early stages of pyrolytic carbon deposition were investigated by atomic force microscopy (AFM), friction force microscopy (FFM) and force modulation microscopy (FMM).

Results

At short residence times single pyrolytic carbon islands were found. Force modulation microscopy allowed to achieve a clear material contrast between the silicon substrate and carbon islands. This allows the identification of the carbon islands. Three different nucleation mechanisms were found: random nucleation of single islands, nucleation of carbon islands along lines and secondary nucleation, the nucleation of carbon islands at edges of already existing carbon islands.

The transition from individual carbon islands to a complete carbon film was observed with increasing residence time (see Fig. 1). Similar sizes are observed for individual islands close to this transition (see Fig. 1) and for the individual grains within the granular structure of the complete carbon films.

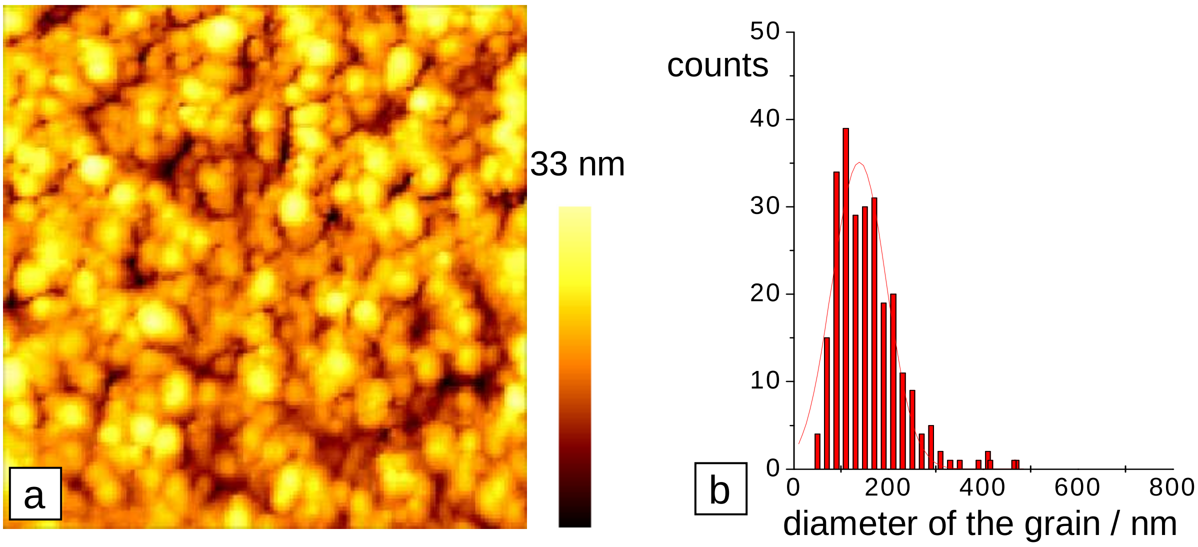

Figure 2: Carbon film on silicon, deposition time 90 min, a) AFM topography, scan area 2 µm x 2 µm, b) distribution of the grain sizes.

The investigation of the grain structure and the distribution of the grain sizes (see Fig. 2) of carbon films showed a correlation between the degree of texture of the carbon film and the average grain size: for high-textured carbon layers larger average grain sizes are found than for less textured carbon layers.