MICROSCOPIC INVESTIGATIONS OF PYROLYTIC CARBON ISLANDS DEPOSITED ON SILICON SUBSTRATES

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Introduction

The physical properties of pyrolytic carbon in carbon fibre / carbon matrix composites are strongly determined by the texture, *i.e.* the preferential alignment of the graphene planes with respect to the substrate. To gain insight into the mechanisms of texture formation during chemical vapour deposition, the early stages of carbon deposition were studied. For this purpose, pyrolytic carbon was deposited on planar substrates (silicon wafers) in a hot-wall reactor. Conventional and high-resolution transmission electron microscopy (TEM, HRTEM) were applied for the investigation of the structure and the texture of the deposited material. The surface topography of the layers was studied by atomic force microscopy (AFM).

Experimental

The pyrolytic carbon was deposited in a hot-wall reactor with a graphite reactor chamber from methane / argon mixtures at a total pressure of 100 kPa (10 or 20 kPa methane partial pressure) and a temperature of 1100 °C. The silicon substrate was oriented parallel to the gas flow. The residence time τ of the gas, which increases along the substrate, was adjusted to 2 or 3.2 s at the end of the substrate. The substrate surface area / reactor volume ratio was 0.26 mm^{-1}. Short deposition times between 5 and 90 min were chosen.

TEM was carried out using a Philips CM 200 FEG/ST electron microscope. The texture of pyrolytic carbon was quantitatively analysed applying selected area electron diffraction. The value of the full width at half maximum intensity of the azimuthal opening of the arc of 00.2 reflections is used for this texture quantification [1]. Applying the recently suggested terminology and definitions for different texture degrees of pyrolytic carbon [2], high-textured carbon exhibits a strong preferential orientation of the (00.1) basal planes parallel to the substrate surface. From medium-textured to low-textured material, the degree of preferential orientation of the basal planes decreases.

AFM measurements were performed with a home-built microscope equipped with a commercial control electronics (Park Scientific Instruments). The experiments were performed at room temperature and in air. Lateral force microscopy was applied to achieve a material contrast between silicon substrate and carbon islands. This allows the identification of the isolated islands on the silicon substrate.

Results and Discussion

The carbon deposition starts with the formation of isolated carbon islands with sizes in the order of 60 nm at very short residence times (Fig. 1, τ = 0.04 s, deposition time of 90 min). These islands are nucleated directly on the silicon wafer without a continuous carbon layer underneath. The size and number density of the islands increase along the substrate, *i.e.* with increasing residence times, until a closed carbon layer is obtained for τ > 1 s.

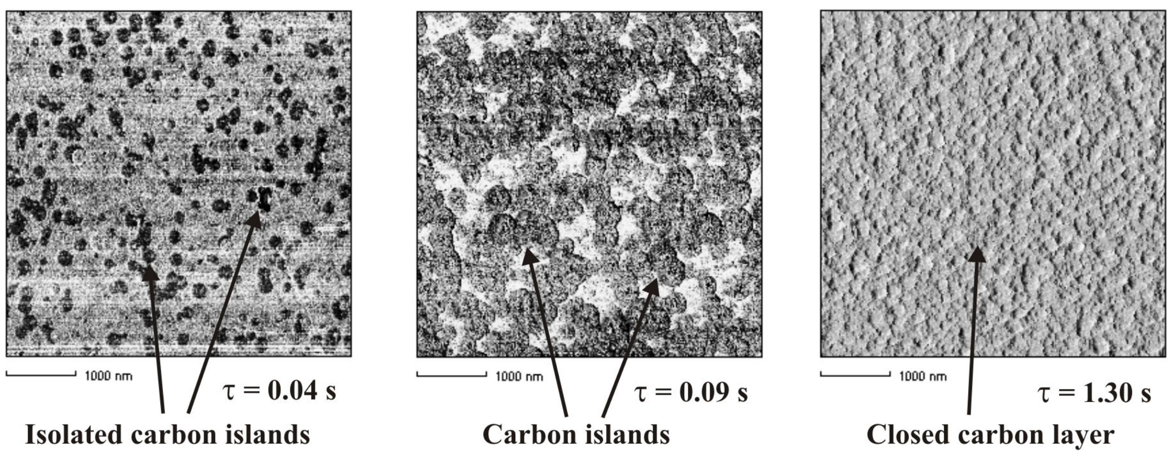

Figure 1. Lateral force micrographs taken by AFM showing pyrolytic carbon islands on the substrate for very short residence times and a closed carbon layer for a residence time of 1.3 s (deposition time of 90 min)

TEM cross-section images at τ = 0.3 s show a thin (~60 nm) carbon layer on the silicon surface and two types of isolated islands: “flat” and “round” islands can be distinguished (Fig. 2). The island sizes range from 90 nm to 1200 nm.

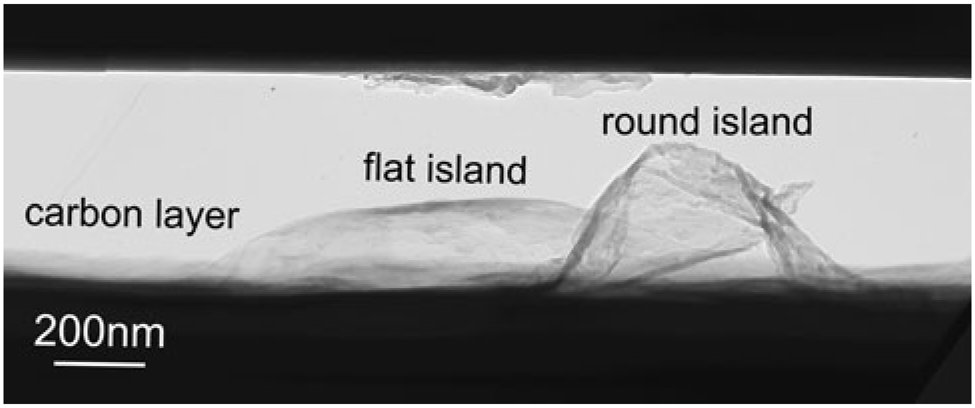

Figure 2. TEM micrograph showing a pyrolytic carbon layer on the silicon surface and the two types of carbon islands (τ = 0.3 s)

HRTEM reveals that single islands deposited at τ = 0.3 s are wrapped by a thin - approximately 10 nm - high-textured shell and contain low-textured material and some long high-textured branches between 7 and 16 nm in thickness (Fig. 3).

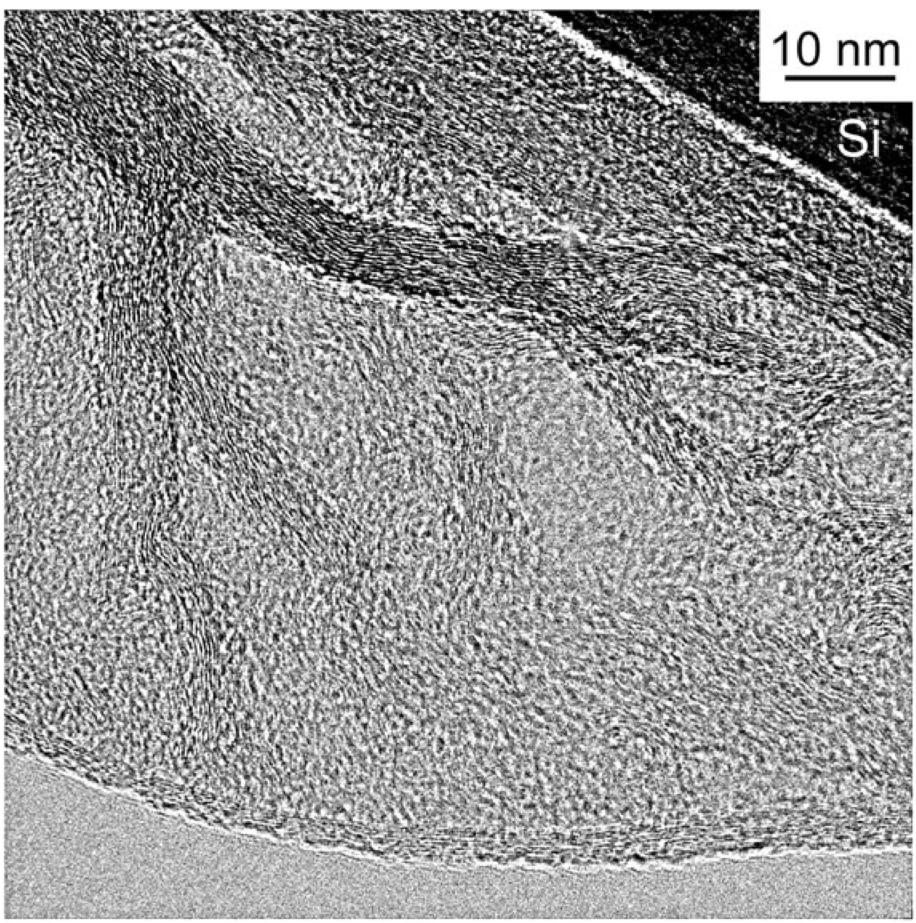

Figure 3. HRTEM micrograph of an isolated carbon island deposited on a silicon substrate (τ = 0.3 s)

The increase of the deposition time up to 240 min leads to the formation of a closed medium-textured carbon layer for $\tau < 1.2$ s.

Conclusions

HRTEM and AFM analyses allow the characterization of the earliest stages of the carbon deposition. The results show that isolated islands are first deposited. The increase of the residence time leads to higher island densities and finally to the formation of a continuous carbon layer.

Acknowledgements

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