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Formation of graphene and hexagonal boron nitride on Rh(111) studied by in-situ scanning tunneling microscopy

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Chapter 7 Introduction to Part II

7.1 General background

Graphene, single-layer graphite, has drawn great attention recently, both because of its special properties, and because of its potential applications, for example, in future-generation electronics [35, 36]. However, reproducibly manufacturing high-quality graphene has remained a challenge. To date, most high-quality graphene, for example for use in experiments requiring high carrier mobilities, is still obtained from graphite, for example, by the ‘scotch-tape’ method [43]. Most methods to assemble graphene directly result in material with a high defect density, caused by multilayer growth [56, 66], chemical contamination [85]. These imperfections seriously influence the properties and the stability of graphene.

Chemical vapor deposition (CVD) of hydrocarbon molecules on transition metals (TMs) provides a practical method for graphene production [25, 51-58]. Since newly arriving hydrocarbon molecules do not stick to or decompose on graphene, CVD may lead to the formation of precisely a single graphene layer, when dissolution and segregation of carbon can be ignored. However, one can also exploit the temperature dependent solubility of carbon in TMs to produce graphene from segregated carbon [51, 53, 57, 59, 60]. The graphene formation process typically takes place at high temperatures, at which the interaction between carbon and TMs is very complex. An essential part of the process, such as the carbon segregation, may be taking place while the metal substrate is cooled down. Often the properties of graphene are measured only at room temperature or below. From such “after-growth” observations it is very difficult to distinguish which source of carbon, segregated or directly deposited, has been responsible for the formation of the graphene layer, and what the precise nucleation and growth mechanisms have been. This is why only a limited number of experiments have been devoted to the kinetic processes of this system [51, 53]. In addition, most of the

experimental methods that can be used *in-situ*, i.e. under growth conditions at high temperature, do not have atomic resolution. Obviously, atomic-scale measurements during graphene growth are really necessary, as they are essential for understanding the nucleation and growth mechanisms, which in turn form the basis for strategies for improving the quality of grown graphene.

7.2 Experimental method

In this part, the formation of graphene on Rh (111), deposited by CVD, was investigated. The main goal was to understand the mechanisms behind graphene formation on transition metals (TMs). This allows us to construct the optimal recipes for producing high-quality graphene, or graphene that satisfies other, special requirements (e.g. multilayer, islanded, etcetera). The main experimental method is a special Scanning Tunneling Microscope (STM), which is applied under actual process conditions, i.e. at elevated temperatures and during substantial variations in temperature. This STM, which has been optimized for (fast) scanning and variable-temperature operation [33, 34], has enabled us to follow the reaction and growth of graphene *in-situ*. Due to its lattice mismatch with Rh(111), graphene forms moiré patterns on this substrate, which work as a 'magnifying glass' for atomic defects in the graphene. In this way, atomic-scale information about the graphene overlayer has been acquired, even from STM images without direct atomic resolution. The system chosen as an example of CVD on TMs is ethylene deposition on Rh (111). The interaction between carbon and Rh can lead to carbide formation [72], carbon dissolution [73] and graphene formation [61], as is also typical for the behavior of carbon on other TMs. Actually, carbon is the main contaminant in purified Rh crystals. Possibly, because this system is so complex, there are relatively few reports about graphite formation on Rh [58, 61]. This complexity makes Rh a good example for demonstrating, in general, the interaction between carbon and TMs. The 3-fold symmetry of the (111) surface was chosen to make it work as a template for graphene crystallization.

7.3 This part of the thesis

This part is organized as follows. The parameters that can influence graphene and carbide formation will be discussed in Chapter 8. We address not only the conventional growth parameters, in this case the temperature and the ethylene pressure, but also the influence of the starting situation. Armed with this information, we propose a first recipe for growing high-quality graphene. In Chapter 9, we further optimize this recipe by controlling the dissolved carbon in the bulk. We conclude that the final quality of the graphene overlayer is limited by the formation of special double layer Rh island structures. Based on the appearance of these structures, we conclude in Chapter 10 that Rh atoms saturate the edges of graphene during growth. In Chapter 11, we provide a detailed, quantitative analysis of various aspects of the growth kinetics. Chapter 12 presents experimental evidence to settle the controversy on the structure of graphene on Rh. Chapter 13 combines all the knowledge acquired in this investigation into one overall picture. This picture can be adapted easily to graphene formation on other TMs and is therefore useful in interpreting this class of growth systems in general and in optimizing growth recipes for each of these cases.

