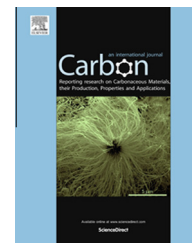


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Formation and characterization of carbon–metal nano-contacts



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ABSTRACT

Electrical contacts between iron nanoparticles and different sp^2 - and sp^1 -hybridized carbon nanomaterials are established in an *in situ* experiment in the transmission electron microscope. The starting material, consisting of FeO nanoparticles, is reduced *in situ* when contacted to graphitic carbon and by passing an electrical current. The remaining Fe particles form covalent bonds with the graphitic electrodes, thus allowing the extraction of different types of nanostructures by retracting one electrode. Graphene ribbons, nanotube-like objects, and monoatomic carbon chains are formed in such a technique that also allows the measurement of the electrical properties of the respective carbon nanostructures.

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1. Introduction

Low-dimensional carbon-based nano-systems are currently receiving much interest due to their high potential in nanoelectronics [1–4], spintronics [5,6], and nano-electromechanical systems [7–13]. Of particular interest are carbon nanotubes [14] and graphene nanoribbons [15]. Many approaches by different techniques have been developed towards their formation and implementation in devices [16,17]. An important step in the production of such devices is the creation of nano-junctions between these low-dimensional carbon materials and their periphery, which is often a metal contact [18,19]. Creating such contacts is already important in experimental setups where the electrical properties of carbon nano-systems are studied [20]. Different types of contacts have been realized, *e.g.*, end contacts where the cross-sectional area of a nanotube or the edge of a graphene ribbon is attached to a metal by covalent bonds [20,21]. However, in test

devices, such a well-defined and mechanically robust contact is rather the exception than the rule. More often shown and easier to realize is the side contact, where a nanotube or graphene sheet is just attached weakly with its side face to a metal surface [18]. Nevertheless, since the π -electron system of the graphitic particle overlaps with the conduction electron system of the metal, a good electrical contact can be achieved. Different metals have already been tested as contacts to carbon nanomaterials. Interesting options are transition metals that are used for the growth of carbon nanotubes or graphene. Growth and contacting can be done in one step by using the same metal. However, a general difficulty is the formation of insulating oxide layers on the metal contacts as soon as they are exposed to air. This is a particular problem since metal nanoparticles easily transform to oxides due to their high reactivity with oxygen and the high surface/volume ratio at the nanoscale. Hence, a procedure has to be applied that either avoids surface oxidation of the metal

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contacts or allows the *in situ* reduction of oxides so that reliable metal–carbon contacts can be made.

In a previous study [20], contacts between metal tips and graphitic aggregates have already been used to unravel atomic carbon chains from graphene layers. The stability of the contacts was problematic in this study and limited the lifetimes of carbon chains as well as the measurement of their electrical properties. The conditions and procedures for establishing reliable covalent contacts remained unknown. The formation of undesired insulating or semi-conducting metal oxides or carbides at the metal–carbon interface was a general concern. It became clear that a detailed study of metal–carbon nanocontacts with a wider applicability (graphene ribbons, carbon nanotubes) would be highly desirable. In the present work we study in detail the formation mechanism of contacts between iron nanoparticles and sp^2 - or sp^1 -bonded carbon materials. Since extensive work on nanotubes has already been done in the last few years [21], we concentrate on graphene nanoribbons and atomic carbon chains, the latter being an unusual carbon allotrope that has recently been synthesized [22]. Contacts between these carbon species and Fe nanoparticles are formed in an *in situ* transmission electron microscopy (TEM) study. The implementation of a scanning tunneling microscope (STM) tip in the TEM allows us to measure the electrical properties of the contacts. The chemical transformations at the contacts are studied by imaging and electron energy-loss spectroscopy (EELS).

2. Experimental and results

The formation of graphene ribbons and carbon chains as well as their contact to a metal was achieved *in situ* in the TEM. The starting material, consisting of larger few-layer graphene flakes, was synthesized by the mechanical ablation of pencil lead in an ultrasonic bath. The following acid/base purification produced flakes with a small number of layers and an average lateral size of $2\ \mu\text{m}$ [23]. At first, the few-layer graphene flakes were decorated by Fe_3O_4 nanoparticles in a solvothermal synthesis [24]. This technique ensured the formation of well-defined nanoparticles of about 10 nm in diameter with very narrow size distribution (shown in Fig. S1 in the Supplementary information). The particles are trapped on defects of the graphene surface that act as pinning sites. Then, the nanoparticles were reduced *ex-situ* under H_2 at 400°C in order to obtain metallic Fe particles. Leaving the Fe particles in air caused their transformation to iron monoxide (FeO) nanoparticles. Details of the synthesis and preparation procedure are described in the Supplementary information.

The experiments were carried out in an electron microscope (Jeol 2100F) with aberration-corrected condenser and an imaging energy filter for EELS analysis. An STM tip was integrated in a Nanofactory specimen holder with piezo manipulation in x -, y -, and z -direction [25,26]. The few-layer graphene flakes were deposited on molybdenum half-grids that allowed the contacting by a gold tip which was prepared

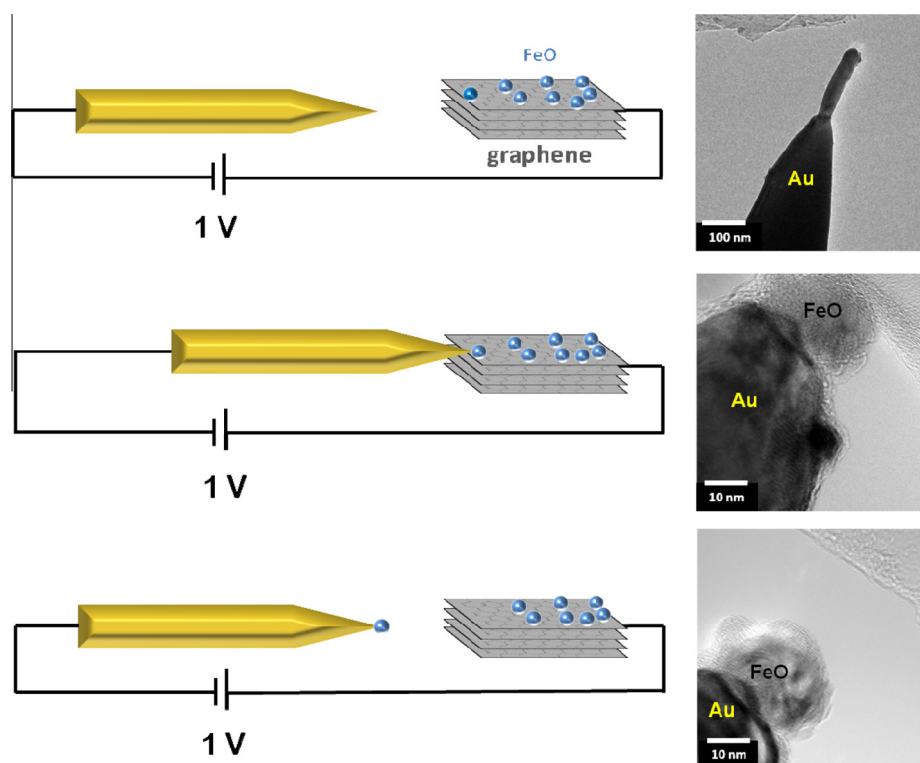


Fig. 1 – Schematic procedure showing the picking-up of an iron oxide particle from the graphene support. The respective TEM images during the procedure are shown on the right hand side (note the different magnifications). (A colour version of this figure can be viewed online.)

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