

applications and scientific insights [1]. It is also closely related to carbon nanotubes, fullerenes or graphite, which can be described as rolled-up or stacked graphene sheets [2]. Hence, the graphene structure and its defects are of outstanding interest for the science and applications of all these new materials. Static deformations, topological defects, various vacancy configurations or the two-dimensional equivalent of dislocations can be studied by aberration-corrected transmission electron microscopy (TEM) [2-5]. Existing defects in as-synthesized graphene, CVD synthesized graphene, and reduced graphene oxide are analyzed [6-7]. The formation and evolution of defects under electron irradiation is observed in real time with atomic resolution. High-energy electron irradiation provides a “randomization” of some atoms, which then allows new insights into the complicated bonding behaviour in carbon materials [5]. Further, we show that the charge distribution in graphene defects or other 2-D materials can be analyzed on the basis of high-resolution TEM images [8]. I will also discuss important aspects of radiation damage and instrumental performance in the context of radiation sensitive nano-carbon materials.

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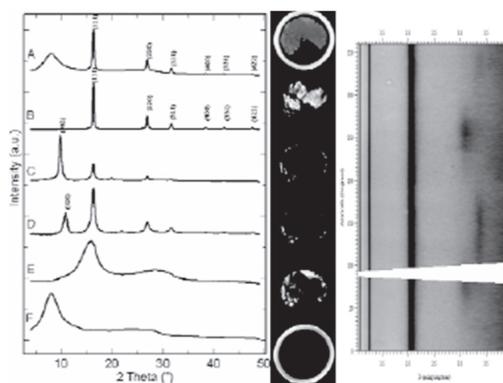
Carbon polyamorphs in pressure-crushed C60 analyzed by scattering tomography

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Laboratory carbon samples can be heterogeneous. Among them, the high-pressure polymorphs of C60 are of particular interest for the study of their complex phase diagram and structural properties. We have used scattering resolved tomography (or XRD-CT) as a non-destructive local probe, with depth resolution and structural selectivity [1], to investigate this multi-phased carbon system.

Under hydrostatic pressures C60 molecules are stable up to 20 GPa, but at higher non-hydrostatic compression at room temperature they collapse into polycrystalline cubic diamond and insulating sp³ amorphous phase. Quasi-hydrostatic loading at high temperature produce a conductive graphitic amorphous sp² structure. Furthermore, HP-HT experiments on C60 allow synthesis of several polymeric and disordered allotropes with a wide range of physical properties [2].

Scattering resolved tomography uses micro-focused synchrotron radiation, allows to extract the spatial distribution of four carbon phases in this system and to recover selectively their scattering diagram (Fig. BCDE). First tomography image corresponds to a full phase reconstruction (all Bragg peaks of the phase were taken into account for the reconstruction). Using selective scattering signal, this probe reveals concentric spatial distribution of cubic diamond, sp³-amorphous diamond, sp² graphite-like carbon phase and a new ill-ordered carbon phase having an unexpected short interlayer distance of 3.11 Å (Fig. D + unrolled pattern) [3].



Usually in “sp²-graphitic” phases, interlayer distance is found larger than 3.34Å, however it should be noted that in shocked meteorites it was reported a compressed “graphite-like phase” with an 3.19Å interlayer distance and a similar spatial concentric relationship between different carbon polymorphs [4]. Additionally, lattice d-spacing maps on the low compressible diamond phase reveal different strained diamond crystallites in the core of the sample.

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In-situ characterization of the carbon nanotube growth process by X-ray diffraction

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In the last few years carbon nanotubes (CNT) attract more and more attention due to their interesting physical properties especially in the field of micro electronics. Synthesis of CNTs with tailored properties is still a critical point, especially for their applications as interconnects. The growth of CNT by chemical vapor deposition (CVD) is an established synthesis route and it was used in this study.

In-situ X-ray diffraction experiments during growth conditions are performed at the beamline BM20 at the ESRF operated by the Helmholtz-Zentrum Dresden-Rossendorf using a high temperature annealing chamber suitable for reactive gases. Acetylene was used as carbon precursor for the CVD of CNT. Different catalyst systems were studied. For iron nano-particles acting as CNT catalyst, there is still a debate which species can be catalytically active: metallic iron and/or iron carbide. The formation of the metal nano-particles by dewetting and respective crystallisation of the initial thin film was followed by X-Ray reflectivity (XRR) and diffraction (XRD) measurements. We proved that CNT growth can occur without the presence of Fe₃C. In general two reaction pathways denoted by a high and low (or zero) iron carbide concentration were observed. Which route the growth follows depends in a statistical way on the ratio of different iron phases (α - and γ -Fe) present in the nano-particles. This can be influenced also by the reaction conditions like temperature and kind of the buffer