Dark Field Photoelectron Emission Microscopy of Micron Scale Few Layer Graphene

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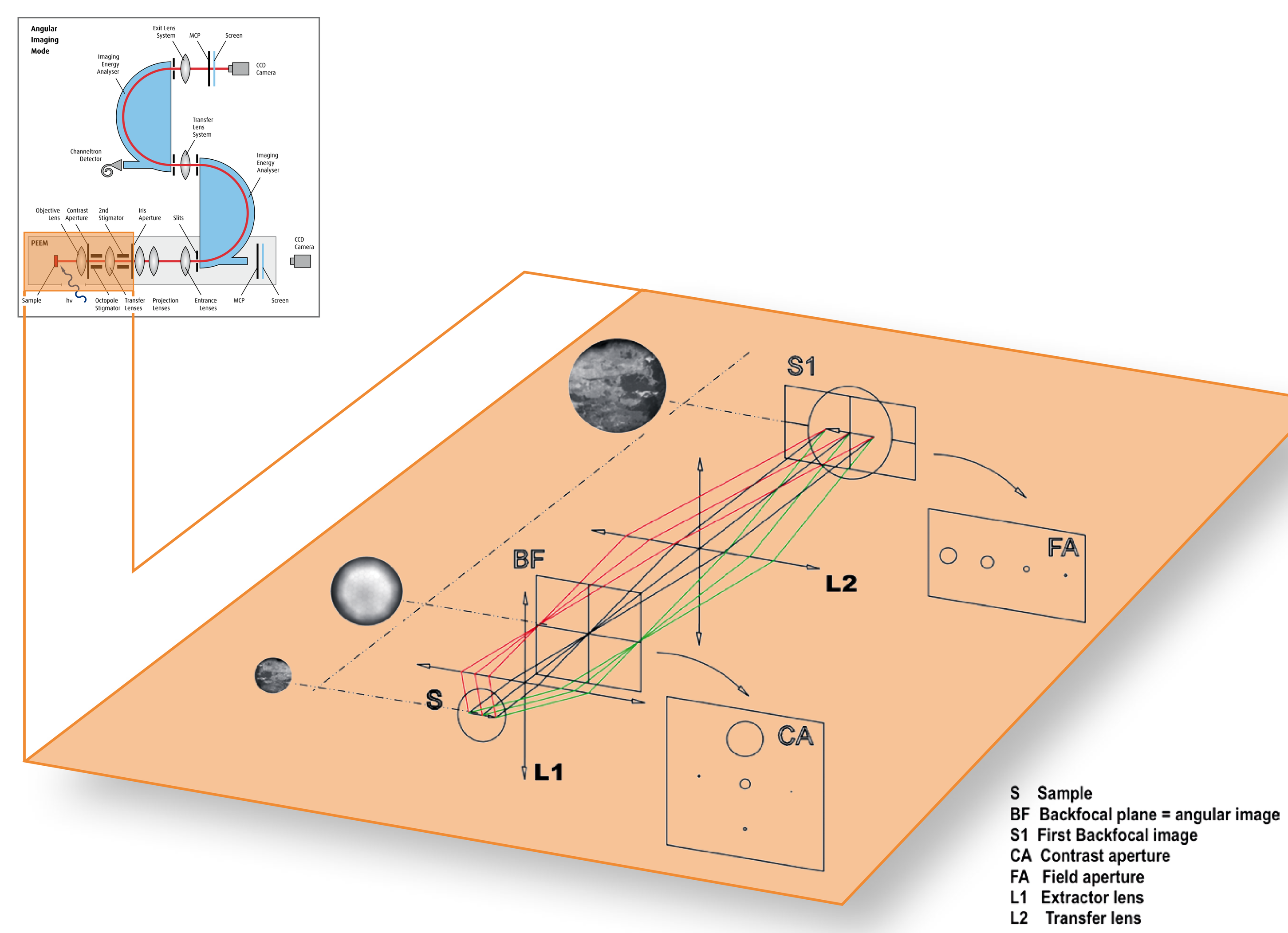
Introduction

We present dark field Photoelectron Emission Microscopy and energy filtered electron emission spectromicroscopy with high spatial and wave-vector resolution on few-layer epitaxial graphene on SiC(000-1) grown by furnace annealing.

Conventional electron spectroscopy methods are limited in providing simultaneous real and reciprocal or k-space information from small areas under laboratory conditions. Therefore, the characterization of materials with only micron scale sample homogeneity such as epitaxially grown graphene requires new instrumentation. Recent improvements in aberration compensated energy-filtered photoelectron emission microscopy (PEEM) can overcome the known limitations in both synchrotron and laboratory environments.

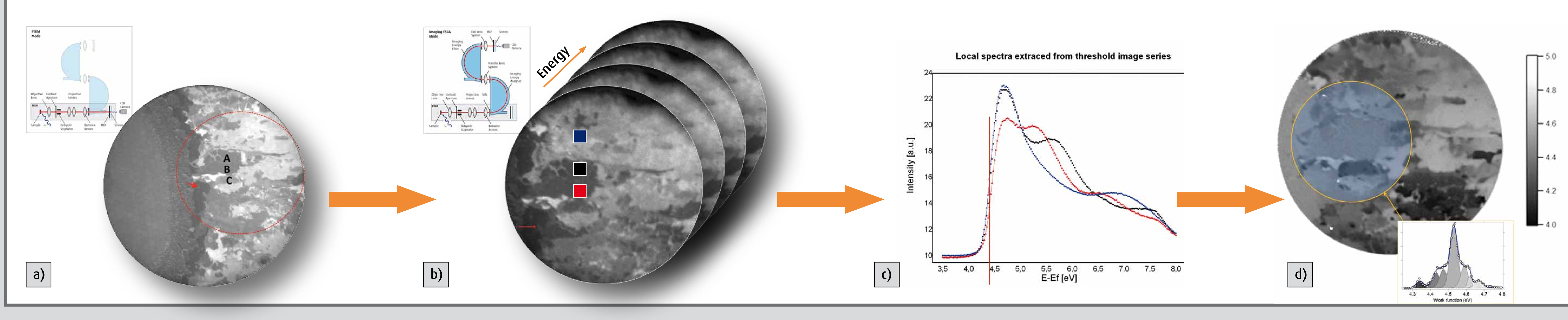
Energy-filtered, threshold PEEM is used to locate distinct zones of FLG graphene. In each region, selected by a field aperture, the k-space information is imaged using appropriate transfer optics. By selecting the photoelectron intensity at a given wave vector and using the inverse transfer optics, dark field PEEM gives a spatial distribution of the angular photoelectron emission. Only the combination of high lateral, high energy, high k-resolution and controlled switching between real space and k-space allows detailed understanding of micron size sample sites with 1–3 layers graphene.

Instrumental Details



Advantage of the NanoESCA lens design is that the analysis area for k-space analysis can precisely defined when the instrument is operated in real space and vice versa. Both apertures CA and FA are operated independently from each other.
In the back focal plane of the first lens L1 the angular image is formed. In this plane a contrast aperture CA is situated. This CA allows to define the angular acceptance of the instrument. A large CA is choosen for a maximum k acceptance a small CA for dark field imaging. After the transfer lens L2 the first intermediate image is formed. In the plane of the first intermediate image a field aperture (iris- or a novel micro-aperture) can be activated to precisely define the local analysis area on the sample for space k-space imaging.
Since the k-space and real space acceptance are defined in the front part of the instrument, the actual switching process has no influence on the area and angular definition. Switching is done by using the projective lenses to shift the focal plane or the image plane into the entrance of the energy analyser. As a result the NanoESCA lens design allows controlled switching between real space and k-space.

Secondary Electron Spectra and Work-Function Map



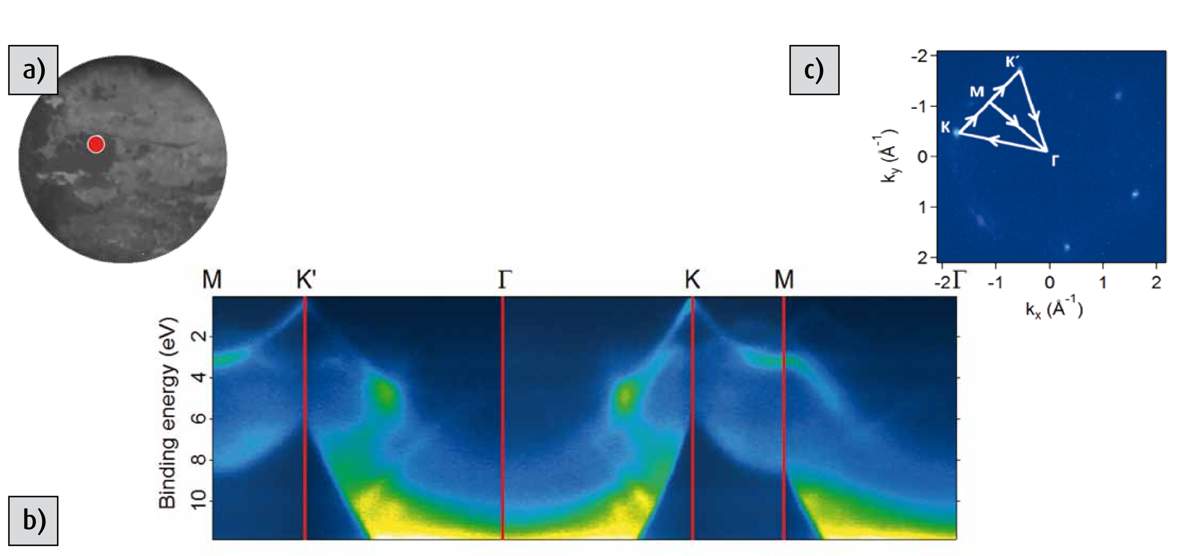
a) PEEM Mode, 120 µm FOV

b) Secondary electron scan:
Dwell time 20 sec,
total time 1h E-Ef 3.5 – 8.0 eV
Step 0.025eV

c) Local spectra extracted from threshold image series b). The Position of emission onset can be analysed to generate local work function map

d) Corresponding high resolution work function map

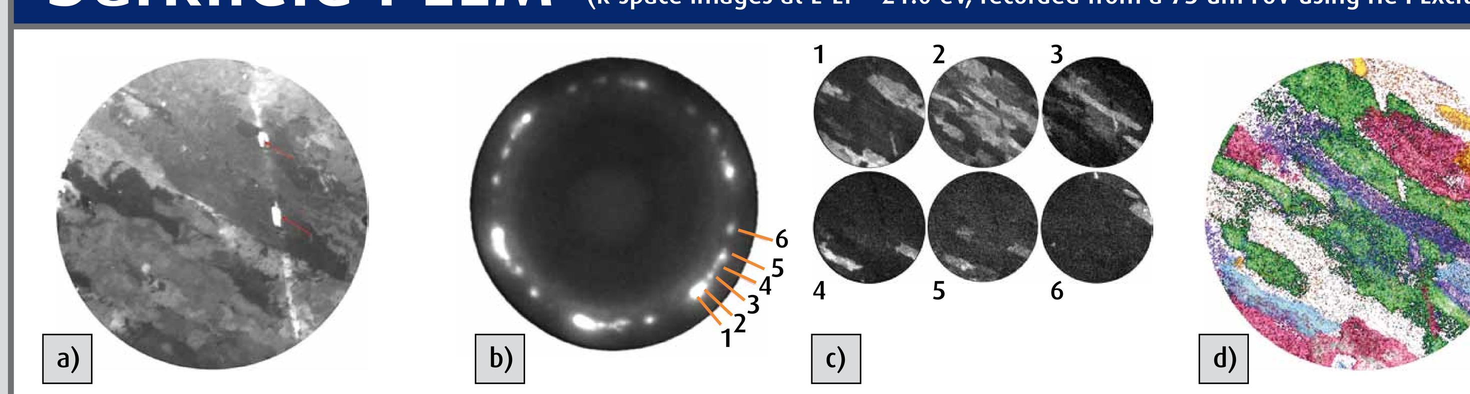
High resolution µ-ARUPS (from 5.7 µm area on single graphene site)



Small area ARUPS using a dedicated micro aperture, He I excitation:

a) Analysis position with a diameter of 5.7 µm diameter has been selected for further analysis
b) K-space landscape generated from the area position A:
Dwell time 100 sec,scanned energy from -0.1eV... 12.0 eV BE, 50 meV step
k-resolution < 0.05 Å^{-1}
c) Constant energy cut at the Dirac point giving direction for the landscape

Darkfield PEEM (k-space images at E-Ef = 21.0 eV, recorded from a 73 um FoV using He I Excitation)



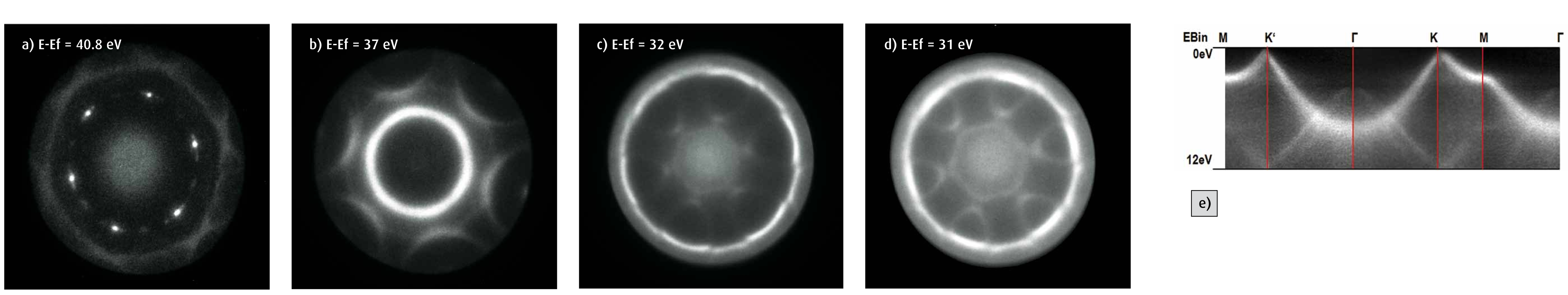
d) Darkfield map composed by colour overlay of the individual darkfield maps from
1) red 0°,
2) green 3°,
3) purple 9°,
4) light blue 20°,
6) yellow 35°

a) Energy filtered secondary electron image, E-Ef = 4.7 eV

b) Area integral k-space map shows multiple dirac cones originating from differently oriented graphene layers.
c) Darkfield images (E-Ef = 21.0 eV) from distinct k-points 1-6 all recorded with an acceptance area of 0.11 Å^{-1} in diameter selected by contrast aperture.

The composed image allows to identify areas where more than one graphene layer contribute to the photoemission at E-Ef = 21.0 eV

He II: Early results using He II excitation



An analyses area of about 35 um was selected for an energy scan from Scan E-Ef 29-41 eV, Step 0.1 eV

a-d: individual constant energy cuts at diferent energies.

e) K-space landscape generated from the energy scan. The heterogene sample caused a slight broadening of the band structure due to fact that photoemission originates from several graphene sheets with slight orientation missmatch.