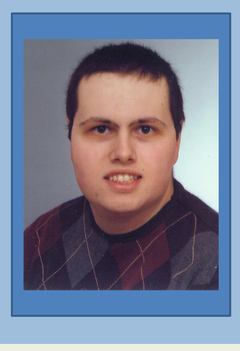

CarbX – An evaluation programme for wide-angle x-ray (WAXS) and neutron (WANS) scattering data of non-graphitic carbons (NGCs)

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Introduction

- Structure of NGCs, a promising material class for several applications:
  - Small stacks of graphene layers, distorted and displaced to each other.
  - Termed turbostratic arrangement of graphene layers in stacks (Fig. 1).
    - → No long range crystallographic order leading to diffuse scattering patterns with broad, asymmetric and overlapping maxima (Fig. 2).
- Structure determination not possible with standard approaches like Scherrer equation or profile analysis (separation of size and disorder).
- Microstructure of NGCs has to be known to link macroscopic (electrical, mechanical) properties to it and to tune production processes properly.

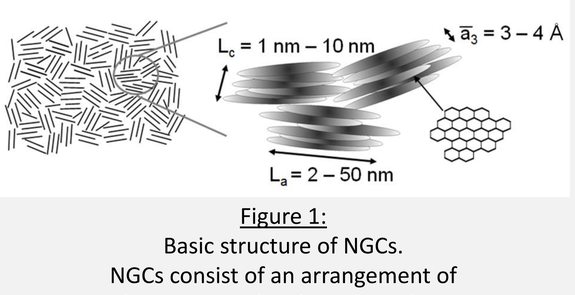


Figure 1:
Basic structure of NGCs.
NGCs consist of an arrangement of turbostratic stacks of graphene layers.
Adapted from [2].

- Microstructure determination possible by acquiring wide-angle x-ray scattering or neutron scattering data and subsequently fitting the whole scattering curve using an algorithm published in 2002.[1]
- Basic idea: theoretically observable intensity $I_{theoretical}$ described to be proportional to the sum of coherent scattering I_{coh} and incoherent scattering I_{incoh}: $I_{theoretical} \propto (I_{coh} + I_{incoh})$
- Coherent scattering: scattering on the hexagonal structure of each graphene layer (intralayer scattering) and on each stack of graphene layers (interlayer scattering), see Fig. 3.

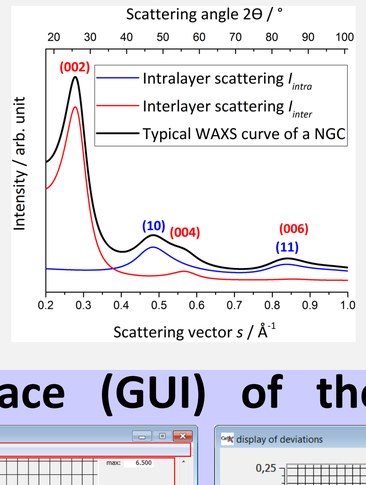


Figure 2:
Typical scattering curve of NGCs resulting fr. overlapping asymmetric maxima of interlayer and intralayer scattering.

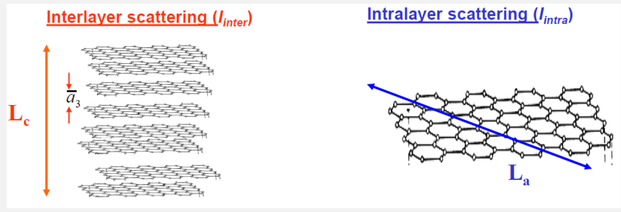


Figure 3:
Origin of the interlayer and intralayer scattering intensities of NGCs. Both intensities are influenced by several microstructural parameters of which three are shown here.

- Incoherent scattering: Inelastic Compton scattering by carbon atoms.
- Scattering by impurities (sp^3 carbon, oxygen) has to be considered, too.
- Further mathematical details can be found in reference [1] , [3] and [4].
- CarbX has been developed to improve the usability of this algorithm.
- CarbX is an unique solution for the microstructural analysis of NGCs.
- CarbX hides the complex mathematics required behind an "easy to use" GUI (graphical user interface), which is depicted in Fig. 4.
- The steps required for fitting scattering data and CarbX' unique features are listed below, Figs. 5 and 6 show reproductions of some fits.

Graphical User Interface (GUI) of the computer programme CarbX

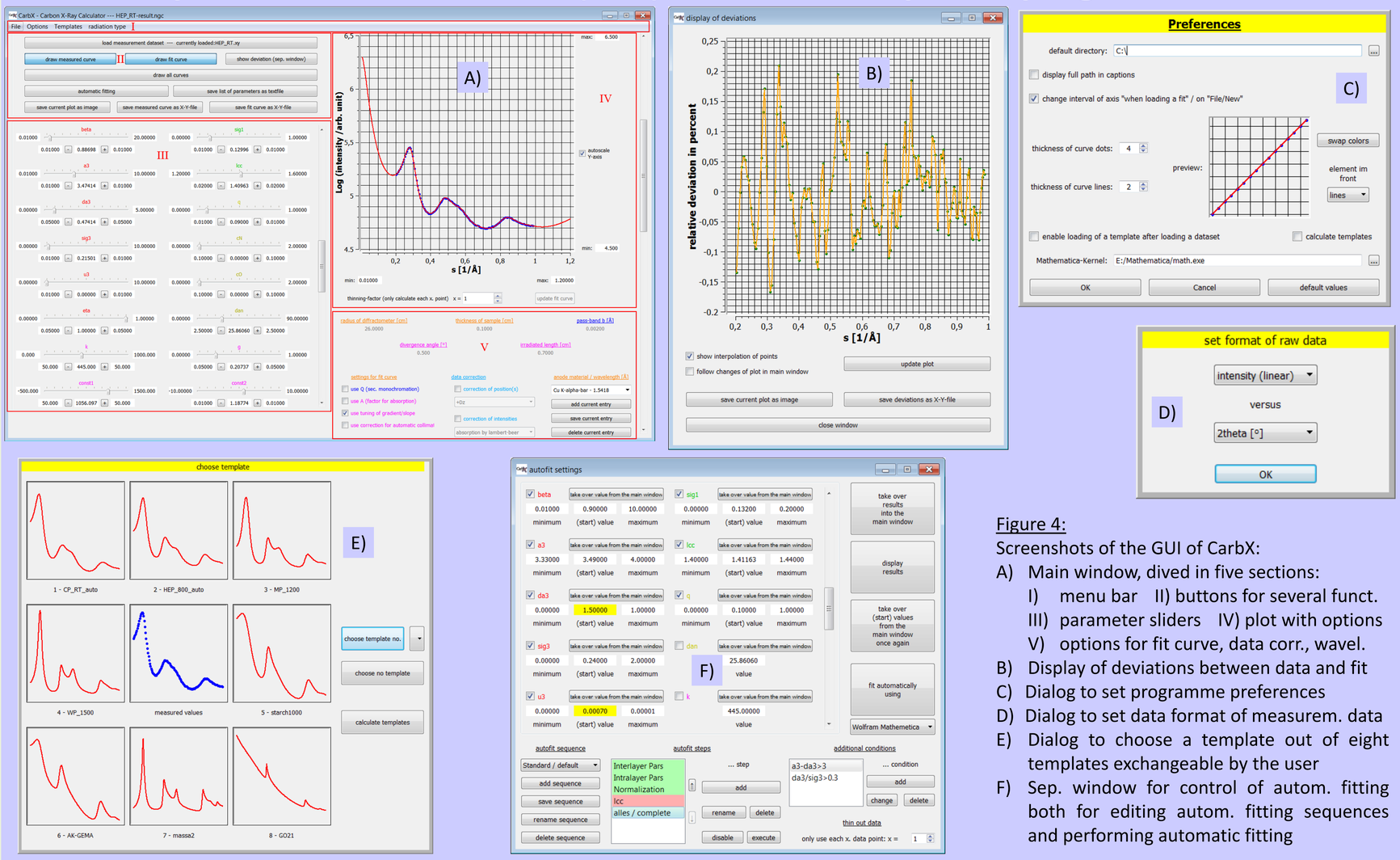


Figure 4:
Screenshots of the GUI of CarbX:
A) Main window, dived in five sections:
I) menu bar II) buttons for several funct.
III) parameter sliders IV) plot with options
V) options for fit curve, data corr., wavel.
B) Display of deviations between data and fit
C) Dialog to set programme preferences
D) Dialog to set data format of measurem. data
E) Dialog to choose a template out of eight templates exchangeable by the user
F) Sep. window for control of autom. fitting both for editing autom. fitting sequences and performing automatic fitting

Fits of WAXS data of four NGCs

$\lambda = 1.5418$ Å $\qquad s = \frac{1}{d} = \frac{2 \cdot \sin\theta}{\lambda}$

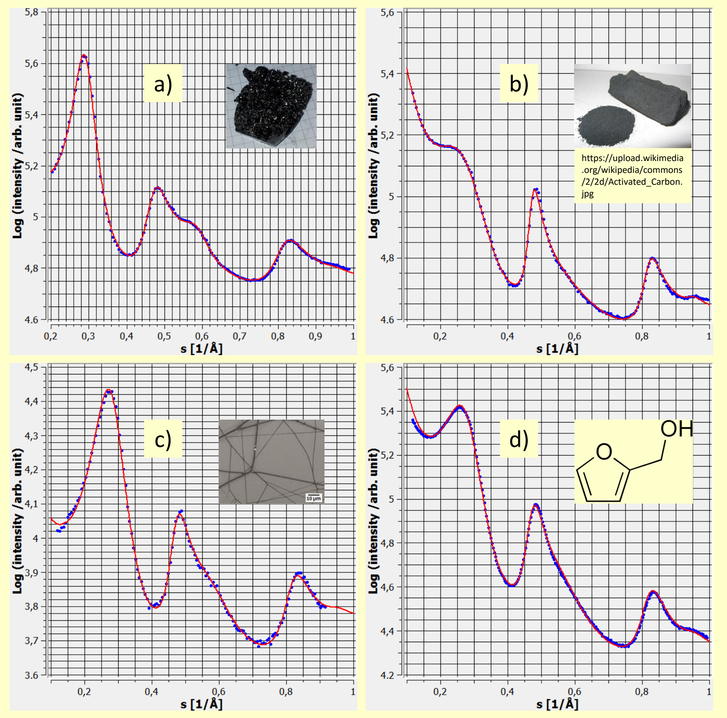
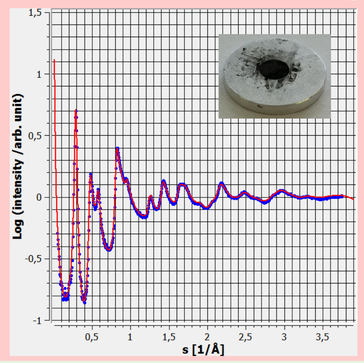


Figure 5:
WAXS data and fits of four different NGCs reproduced with CarbX along with the structural parameters obtained from the result files:

		l_{cc}/Å	L_a/Å	L_c/Å	a_3/Å
a)	Coal tar pitch (800 °C) (with photograph)	1.415	19	13	3.44
b)	Activated carbon (with photograph)	1.409	32	7.4	3.60
c)	Polymerized ionic liquid carbon fibre (with SEM image)	1.411	21	11	3.63
d)	Carbonized furfuryl alcohol (850 °C)	1.409	24	8.0	3.59

Fit of neutron scattering data of a pyrocarbon



$\lambda = 0.4972$ Å $\qquad s = \frac{1}{d} = \frac{2 \cdot \sin\theta}{\lambda}$

Figure 6:
Neutron scattering data and fit of a pyrocarbon reproduced with CarbX. Reflexes occur here over an larger s-range. The structural parameters obtained are:
$l_{cc} = 1.415$ Å, $L_a = 29$ Å
$L_c = 38$ Å, $a_3 = 3.47$ Å

Steps required to fit scattering data using the programme CarbX

1. Extract ZIP-archive of the programme "CarbX.zip" into an empty folder.
2. Set general programme options using the dialog depicted in Fig. 4C.
3. Set radiation type using the menu in section I of Fig. 4A and measurement parameters using control elements in section V of Fig. 4A.
4. Load measurement data (stored in X-Y-file) using the correspond. button in section II of Fig. 4A, set data format (via dialog, see Fig. 4D). If enabled, the dialog to choose a template (Fig. 4E) is shown afterwards. Data will then be displayed automatically in section IV of Fig. 4A.
5. Set data correction (regarding position and/or intensity of the data points), if necessary, using control elements in section V of Fig. 4A
6. Set start param. for fit in sect. III of Fig. 4A using the sliders or choose a templ. after calling the correspond. dialog via a menu in sect. I of Fig. 4A and set options for the fit curve using contr. elemen. in sect. V of Fig. 4A.
7. Display the fit using the corresponding button in section II of Fig. 4A, adapt parameters in section III until achieving a good manual fit.
8. If available, use Wolfram Mathematica for automatic fitting. It is performed and customized using the window depicted in Fig. 4F.
9. Transfer automatic fitting results in the main window after previewing.
10. Display deviation between fit and data, export data, fit and deviation as X-Y-files, export plots as images and export fit results in a text file.

Features of CarbX

- **Complex mathematical calculations hidden behind an "easy to use" GUI.**
- Determination of up to 14 microstructural parameters, e.g.
  - L_a (average graphene layer extent) and L_c (average stack height).
  - a_3 (average interlayer spacing) and several disorder parameters.
- Consideration of several correction terms and support for different X-ray wavelengths.
- Support for both wide angle X-ray and neutron scattering data if supplied as X-Y-file.
- Thinning factor for both faster calculation of fit curve and faster automatic fitting.
- Separate window to display the deviation between data and fit (customizable).
- Several export possibilities (XY-data, graphs, result list) for data, fit and deviation.
- Support for templates and automatic fitting via Mathematica (customizable).
- Data display completely customizable and programme available in German and English.

Conclusion

- CarbX - an unique software solution for easy analysis of the microstructure of NGCS - has been developed.
- CarbX will be available as open-source software after publication - to be submitted to the *J. Appl. Cryst.*
- CarbX will be the first programme available free of charge in this field offering a convenient GUI.
- CarbX has been used successfully for reproduction of fits of both WAXS and neutron scattering data.
- Further development steps planned:
  - Support for two NGC phases in one material.
  - Access of remote Wolfram Mathematica kernels.
  - Range selection for automatic fitting.
  - Cutting out of artefacts for automatic fitting.
  - Self-written routines for automatic fitting.
    - → eliminate the need of third party software.

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