PEERING INTO THE EVOLUTION OF THE CARBON MICROSTRUCTURE OF GLASSY CARBONS DERIVED FROM PHENOL FORMALDEHYDE RESINS

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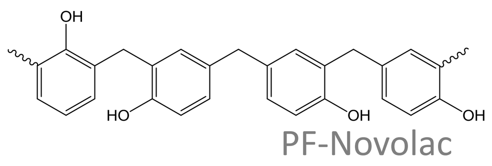
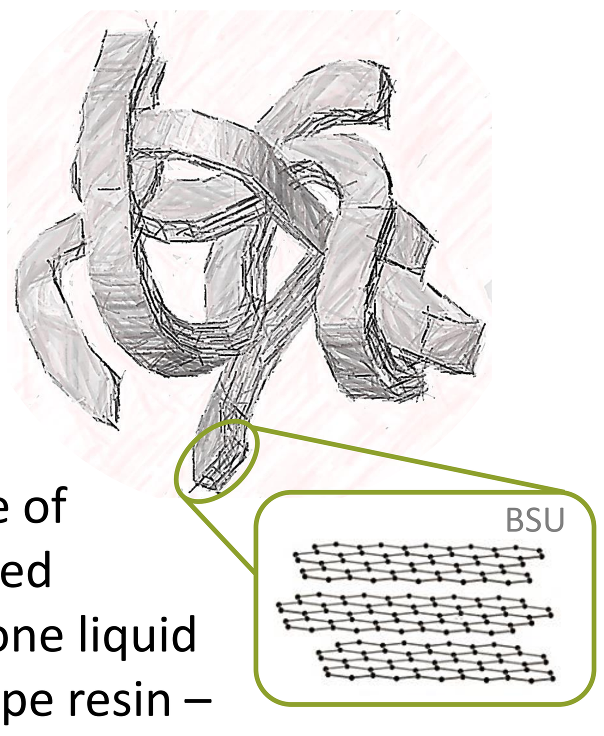
Introduction

- Main physical and chemical properties of carbon materials are closely related to the **carbon microstructure**, e.g. the finite size and disorder of the graphene layer stacks, which can be considered as the basic structural units (BSU) of most sp[2]-based carbons



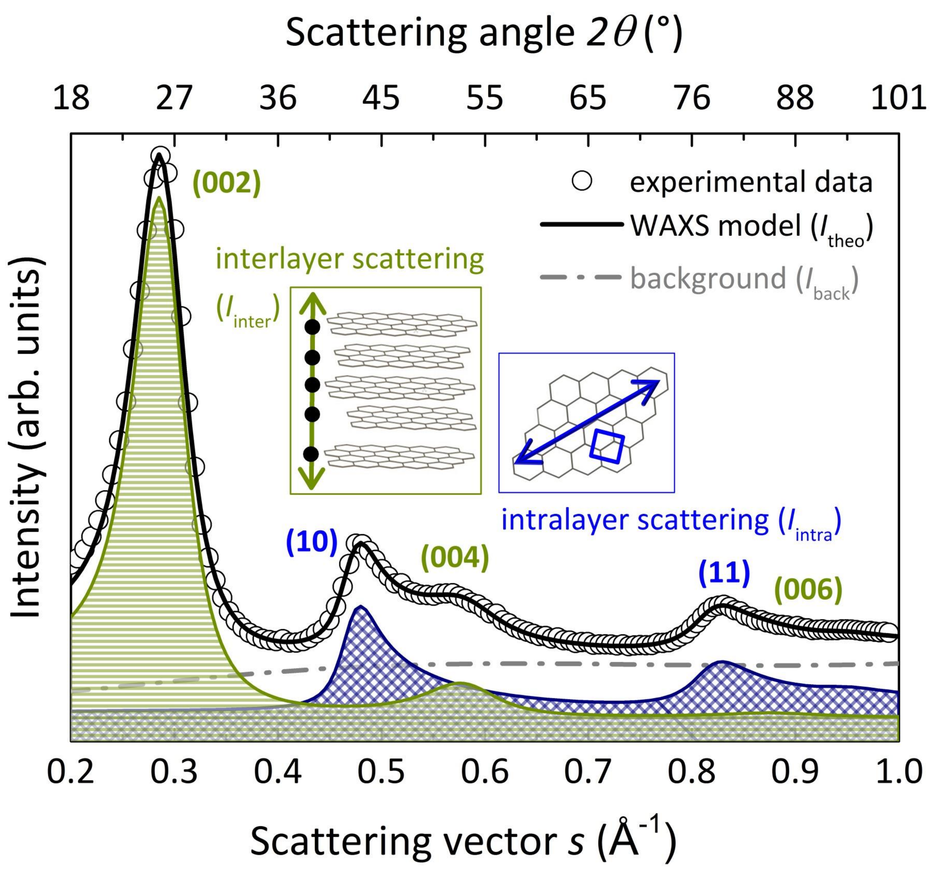
  - Chemical & thermal stability
  - Density, hardness
  - Young's modulus
  - Coefficient of thermal expansion
  - Thermal & electrical conductivity



- Investigation of the carbon microstructure of two non-graphitizing **glassy carbons** derived from **phenol formaldehyde resins (PF)** – one liquid resole-type resin and one solid novolac-type resin – as well as an easily graphitizing mesophase pitch by an advanced evaluation method of Wide Angle X-ray Scattering data (WAXS)

WAXS Evaluation Approach

- WAXS pattern of a typical non-graphitic carbon:
  - Turbostratic disorder
  - Only (*hk*)- & (00*l*)-reflections
  - Asymmetric (*hk*)-reflections
  - Pronounced overlapping
- Evaluation is based on a model of Ruland and Smarsly[1]
- Entire WAXS pattern of a non-graphitic carbon is fitted by a theoretical intensity distribution

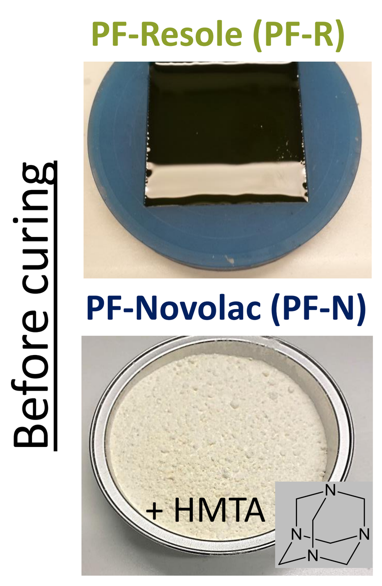


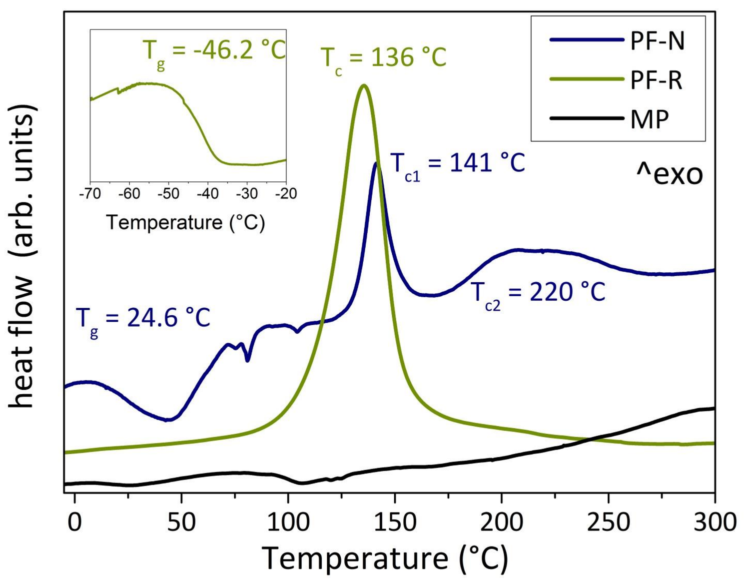
$$I_{obs} = k \cdot A \cdot P \cdot I_{theo}$$
$$I_{theo} = I_{coherent} + I_{incoherent}$$
$$I_{coherent} = f_c^2 (I_{inter} + I_{intra})$$

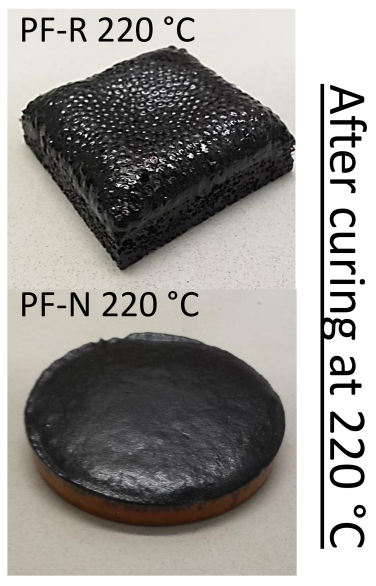
f_c: atomic form factor of carbon, P: polarization factor
$I_{incoherent}$: Compton scattering, A: absorption factor

- Output: 14 parameters quantitatively describing the carbon microstructure[2]

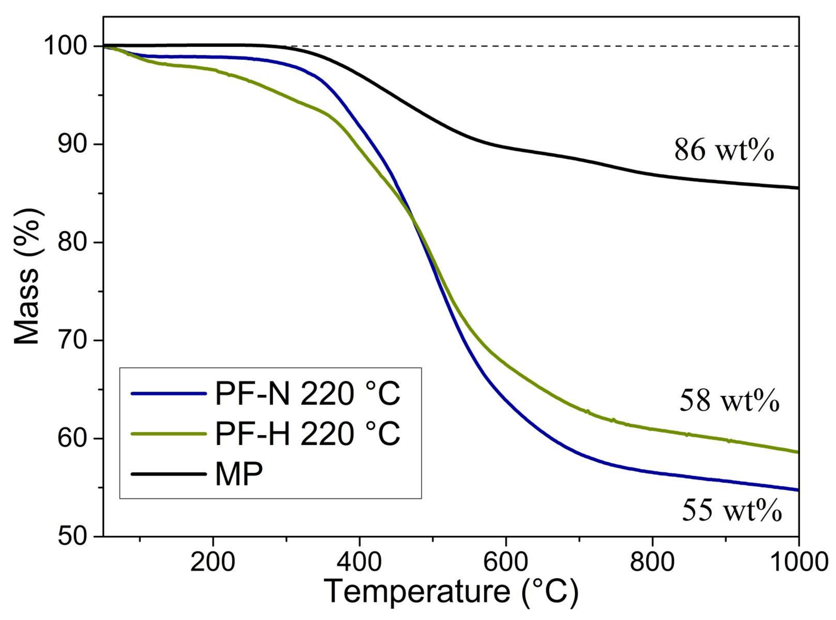
Materials

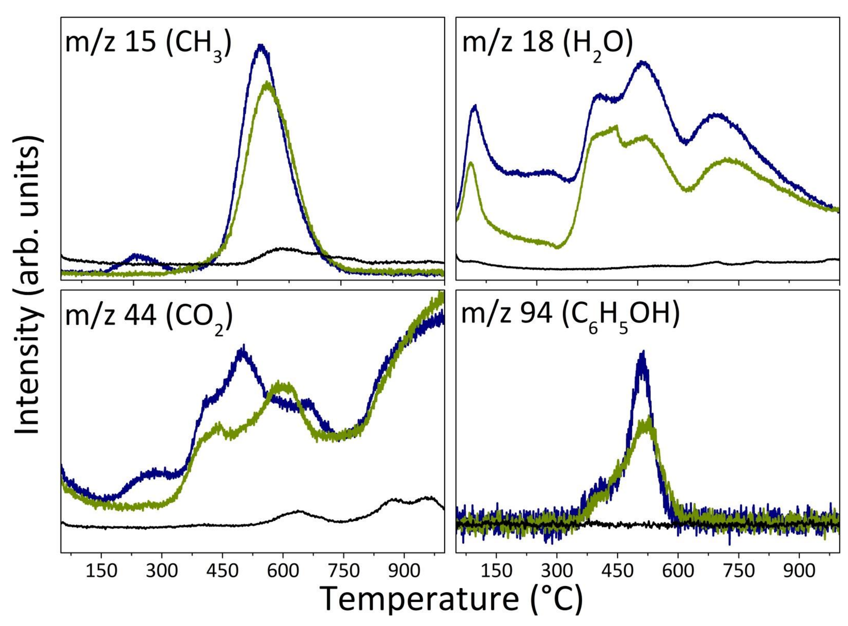






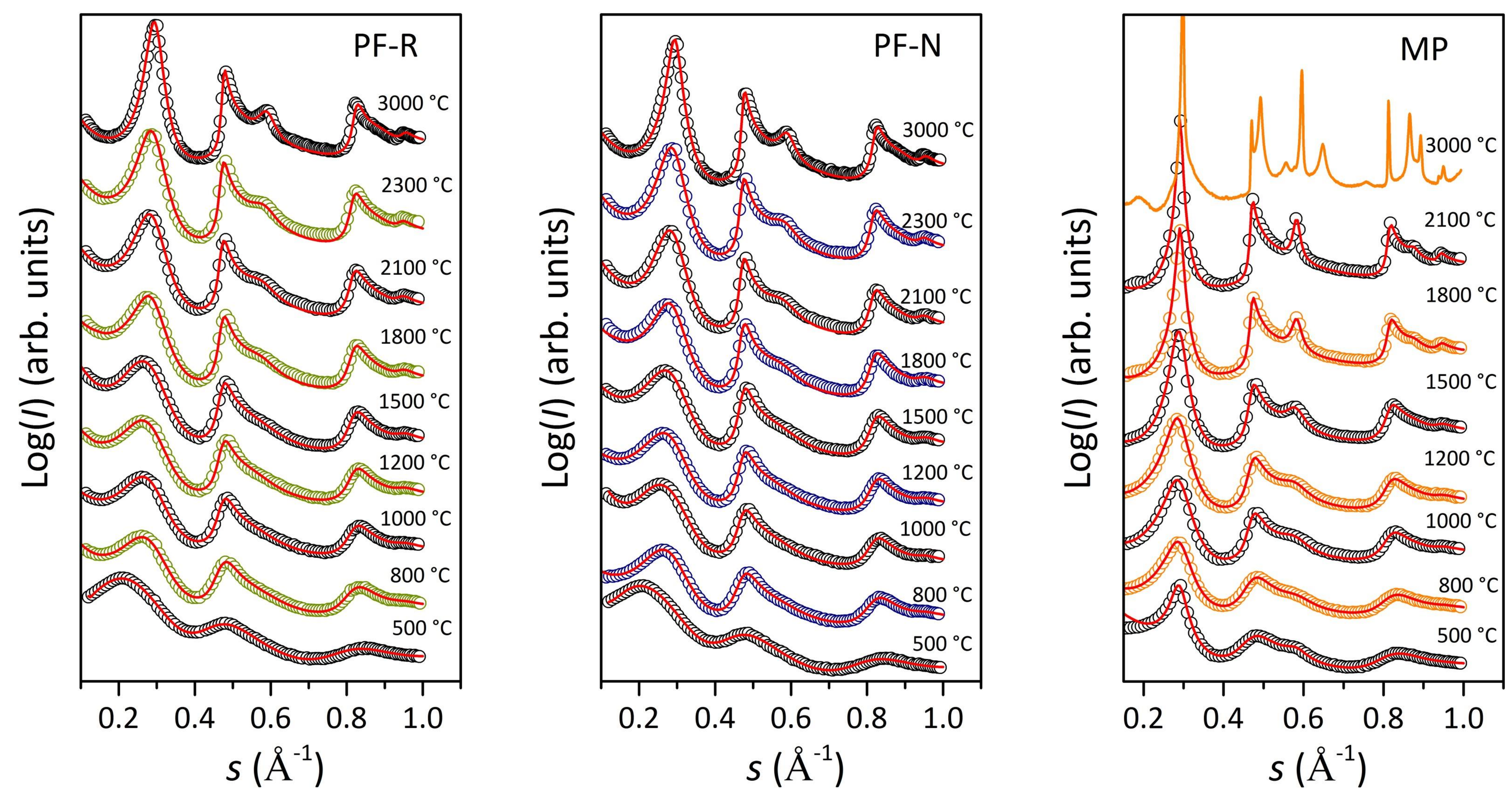
Differential scanning calorimetry (DSC); T_g glass transition temperature; T_c reaction temperature; HMTA: hexamethylenetetramine = curing agent





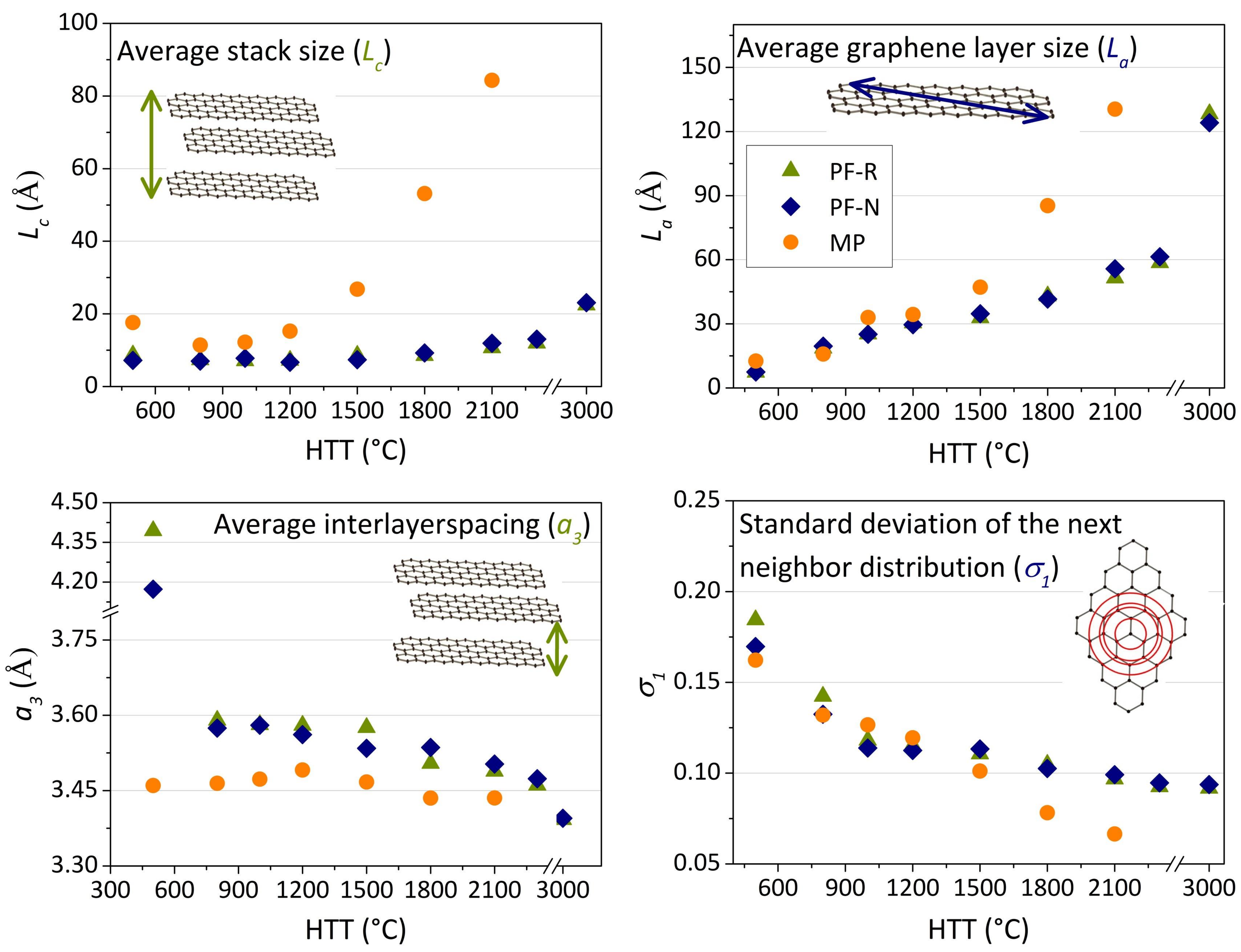
Thermogravimetric Analysis coupled with Mass Spectroscopy (TG-MS)

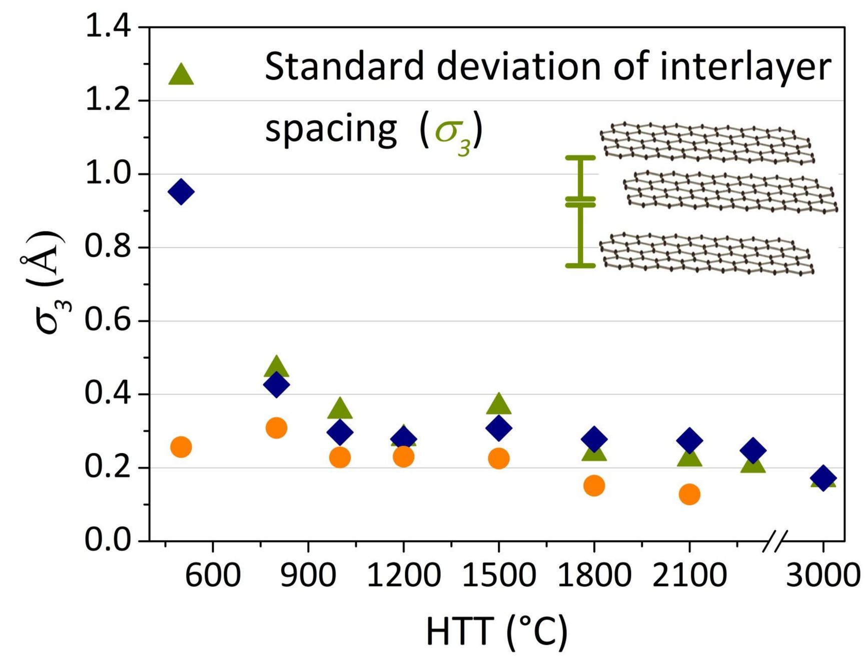
Results – WAXS Data Modeling

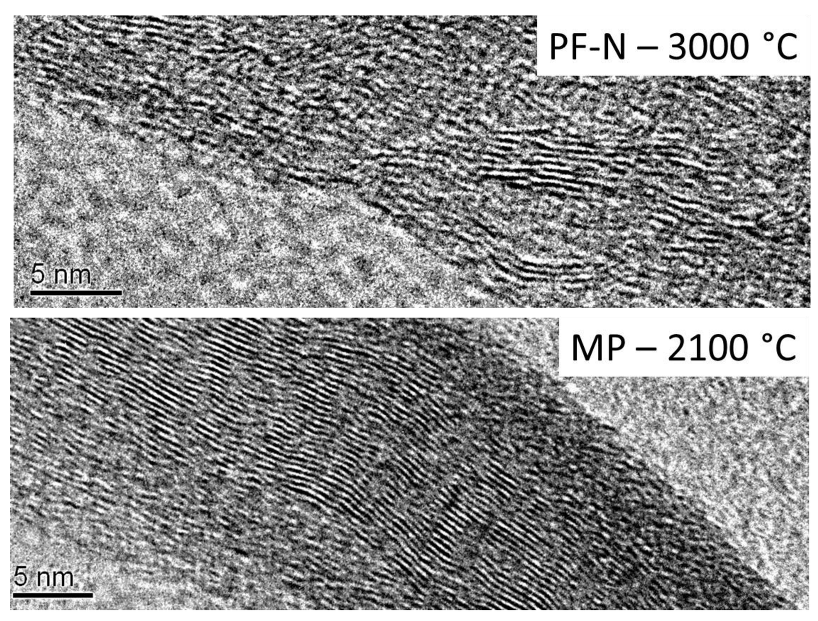


WAXS diffraction patterns: experimental data (circles), WAXS model curves (lines) as a function of the scattering vector $s = 2 \sin(\theta) \cdot \lambda_{(Cu)}^{-1}$

Evolution of the Carbon Microstructure







Conclusion

- The glassy carbons display almost no growth of the graphene layer stacks in the temperature rage up to 3000 °C, BSU preferentially grow in the lateral direction
- Both glassy carbons displayed a virtually identical development of the carbon microstructure which seems to be independent of the initial type of phenolic formaldehyde resin (Novolac/resole)
- WAXS data modeling enables quantitative insights into the carbonization and graphitization behavior of glassy carbons

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Literature
[1] W. Ruland, B. M. Smarsly, *J. Appl. Crystallogr.* **2002**, *35*, 624–633.
[2] Faber K, Smarsly B. M. ZAAC **2014**, *640*, 3107-3117.

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