Microstructure of graphite

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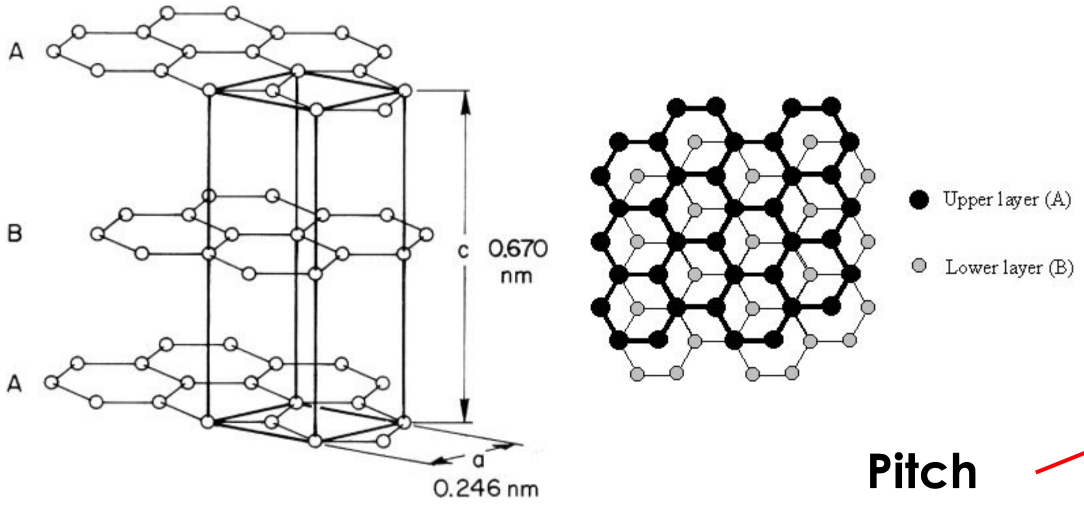
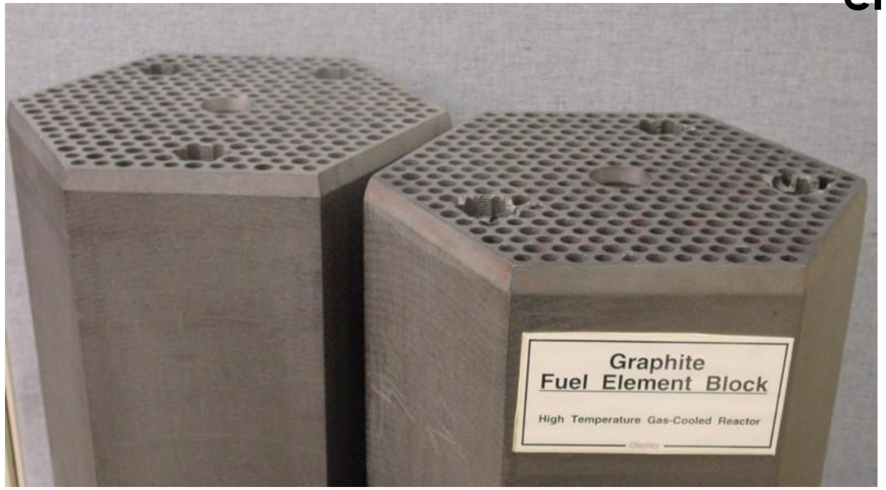
ART Gas-Cooled Reactor Program Review

July 14, 2022

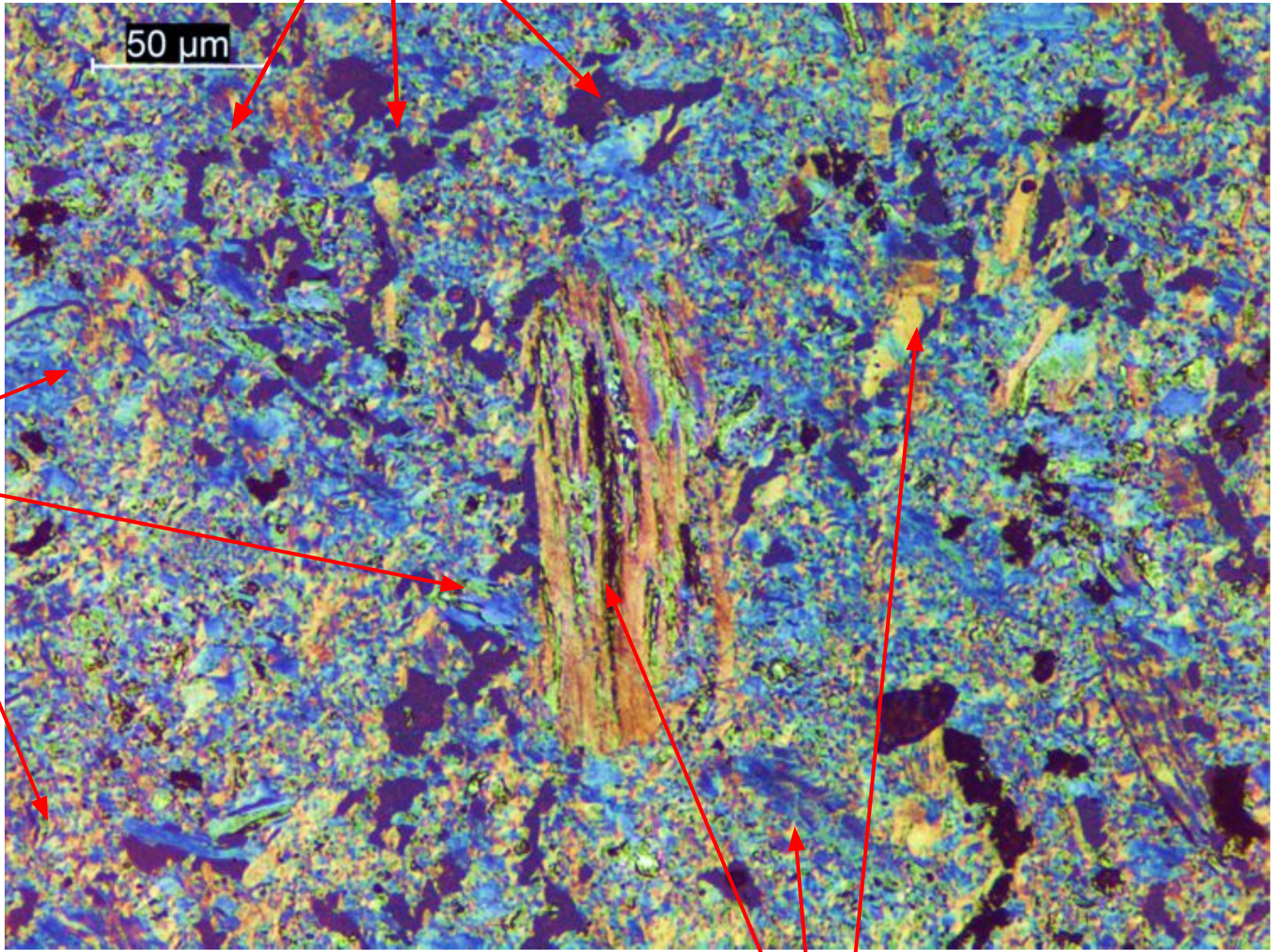
Graphite





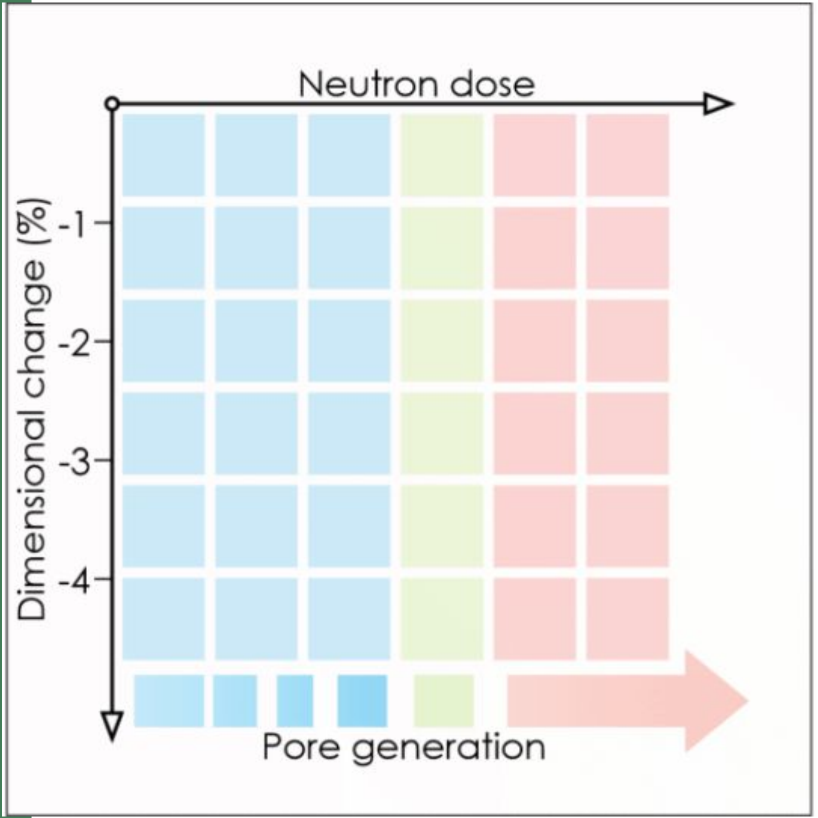
Pores

Pitch bind er

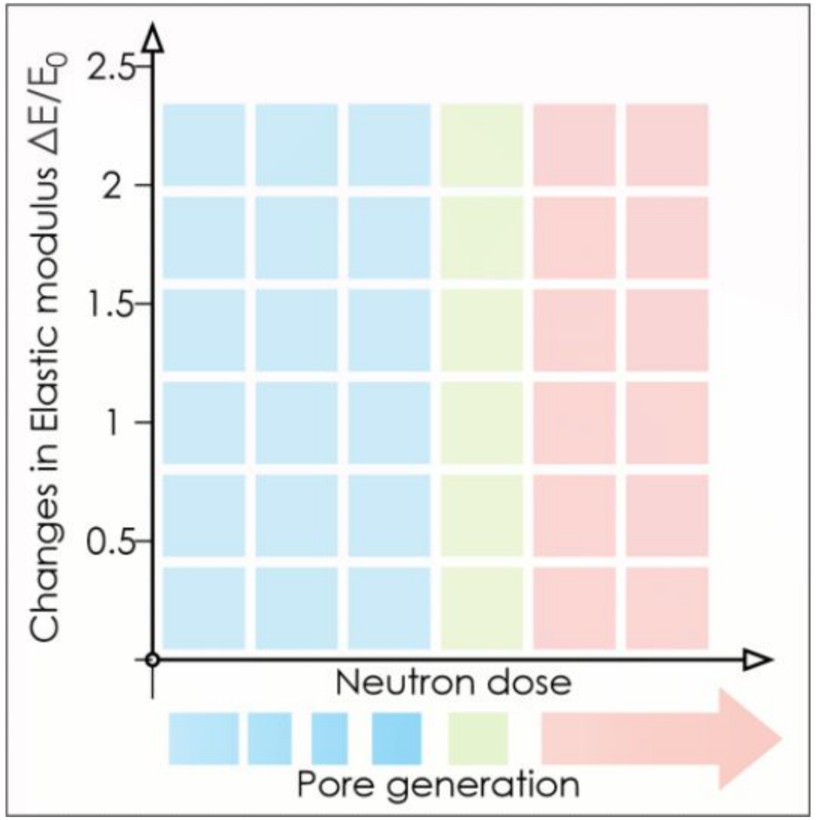


Filler particles

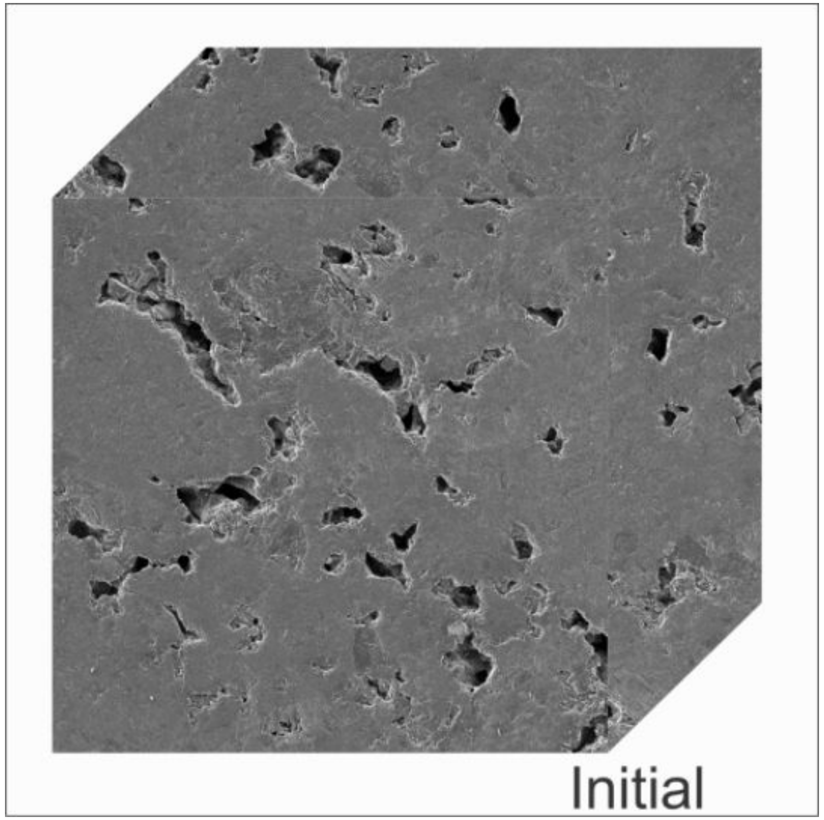
Graphite neutron irradiation effects in a nutshell



Dimensional change



Material properties



Microstructure

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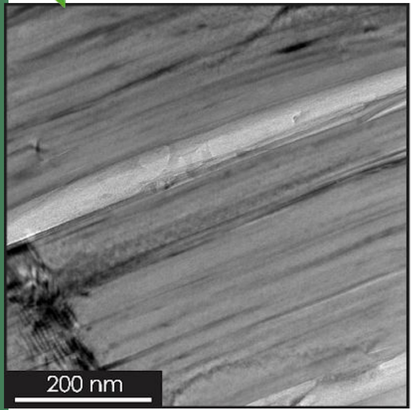
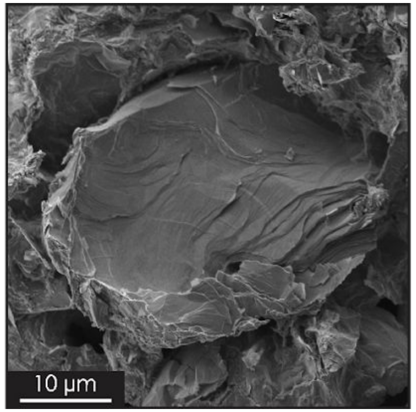
Graphite as a multiscale material

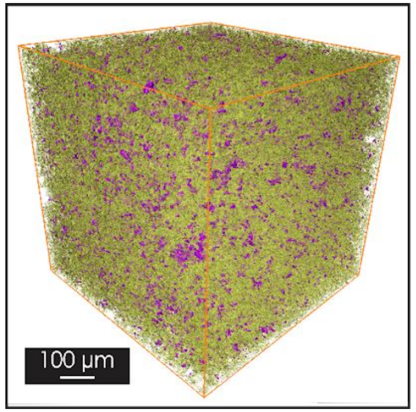
- Bridge the gaps between different length scales and response of graphite

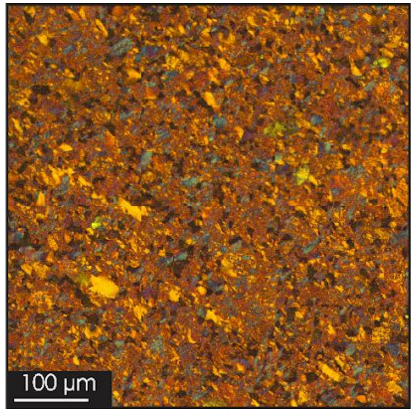
Nano to micro scale	Material scale	Engineering scale	Structural scale

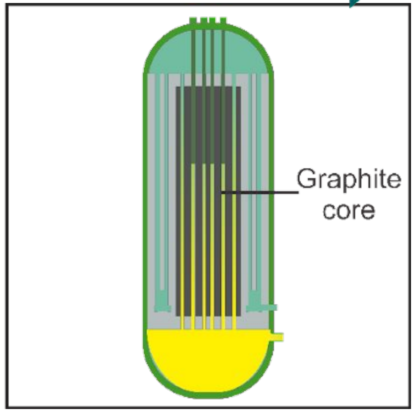
Basic science ⟷ **Applied science**









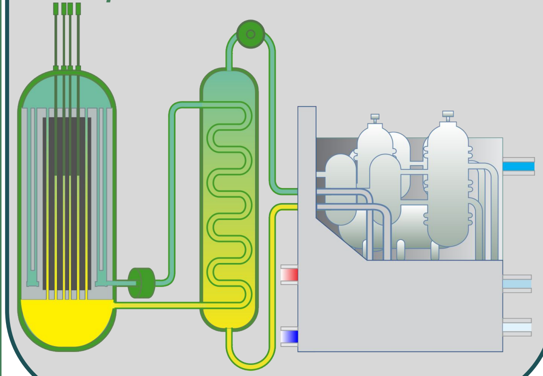
nm | nm-µm | µm | cm | m | m

Length scale

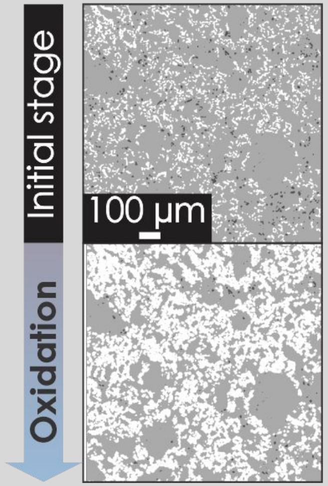
General Vision and Strategy

INPUT

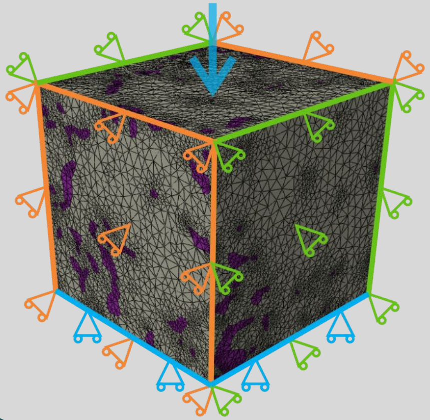
Plant design and operation conditions



Studies on environmental effects



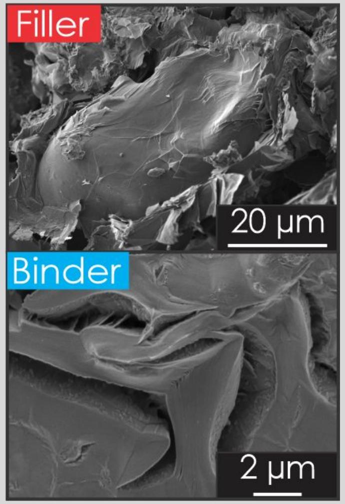
Microstructural based modeling of irradiation and environmental effects



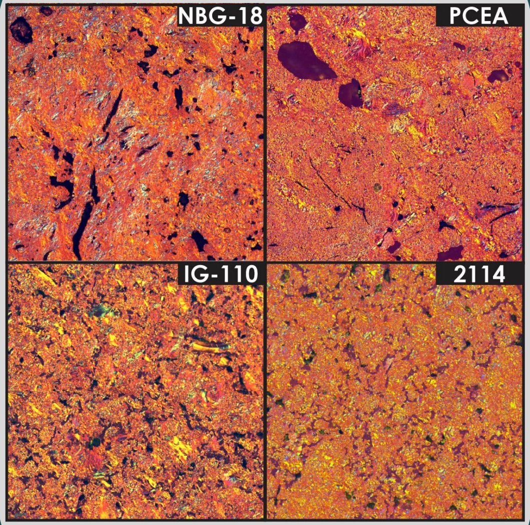
OUTPUT

Assessments of long-term graphite core performance

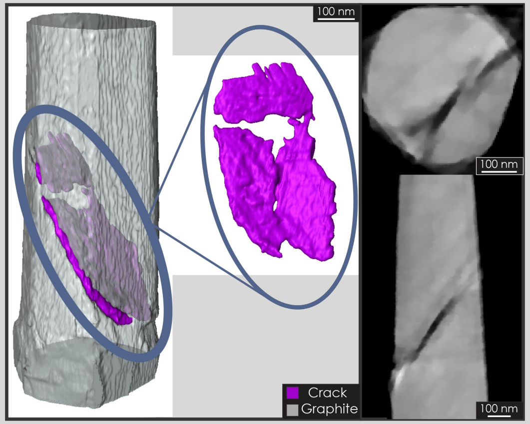
Characterization of nuclear graphite materials microstructure



Library of microstructures



Development of new characterization techniques



Electron tomography

Surveillance of graphite core

General strategy to assess the irradiation-tolerance of nuclear graphite – Microstructural analysis

- **ART/ORNL objective:** Develop a rational classification of irradiation induced defects that help determine the damage on graphite components

Approach

1. Characterization and phase identification of unirradiated graphite using various imaging techniques

2. Phase identification of unirradiated phases to create a robust baseline of grades characteristics

3. Forensic fingerprints identify which phases and techniques can be used assess and predict the lifetime of graphite components

4. Model the influence of microstructure on the irradiation performance of different graphite grades

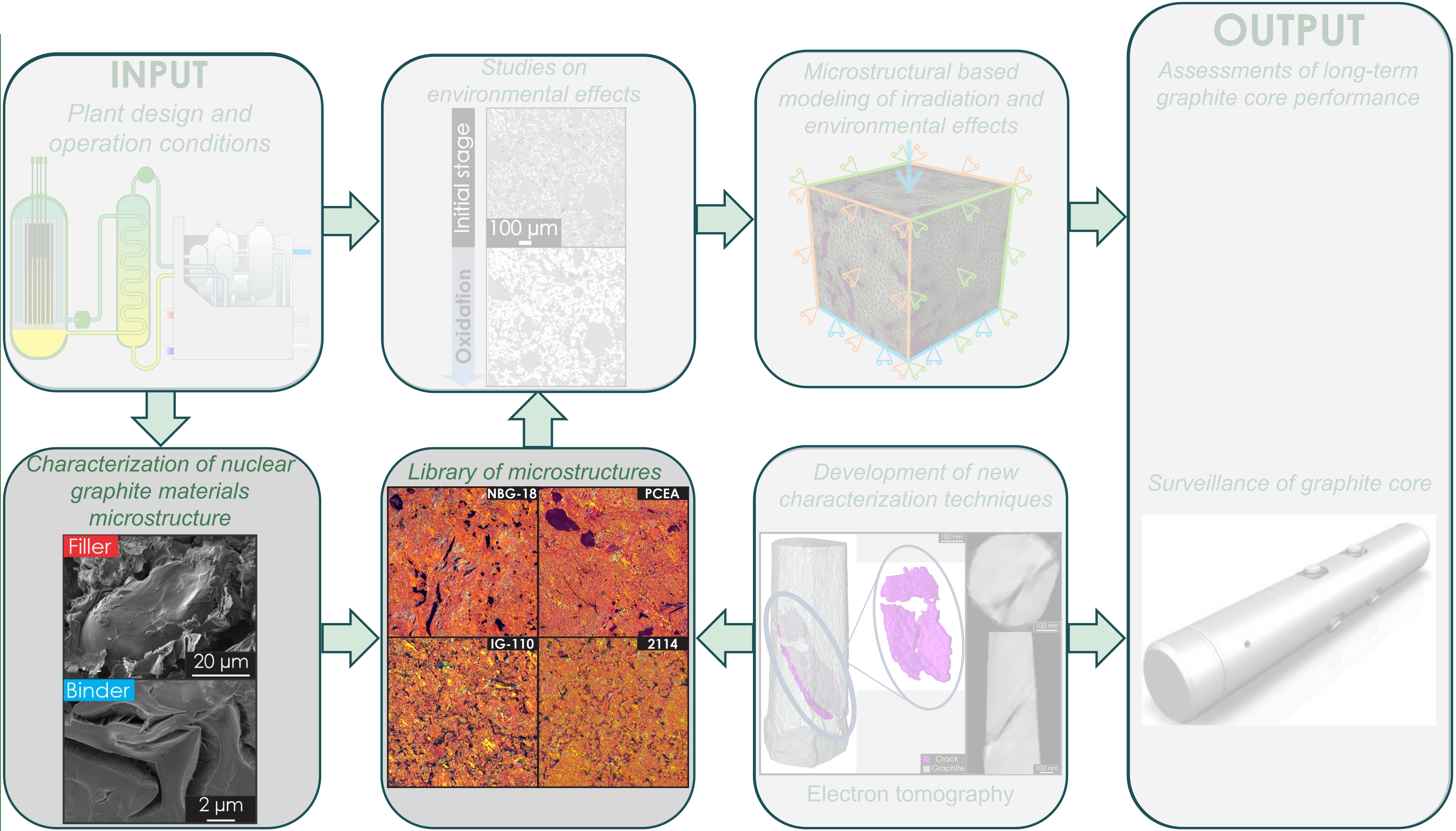
Context and research needs

Limited knowledge/lack of data – on the potential microstructural effects on the microstructure of graphite

Research problem: Can microstructural characterization can be used to infer the damage of a trepanned sample or used to predict the lifetime of a graphite component?

Approach

Baseline characterization	Environmental and irradiated characterization	Microstructural Modeling	Full size modeling
Historical grades Candidates' grades for US reactors	Oxidized samples Irradiated samples (ATR and HFIR)	Image-based models Random field models	Random field models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Plant design and operation conditions
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Initial stage
100 µm
Oxidation
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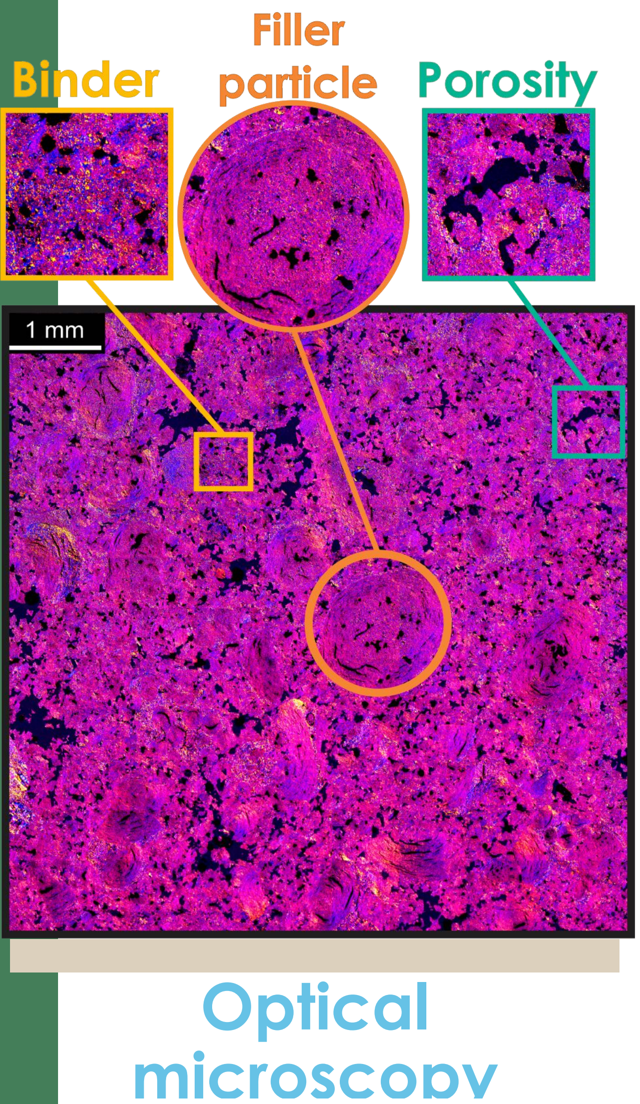
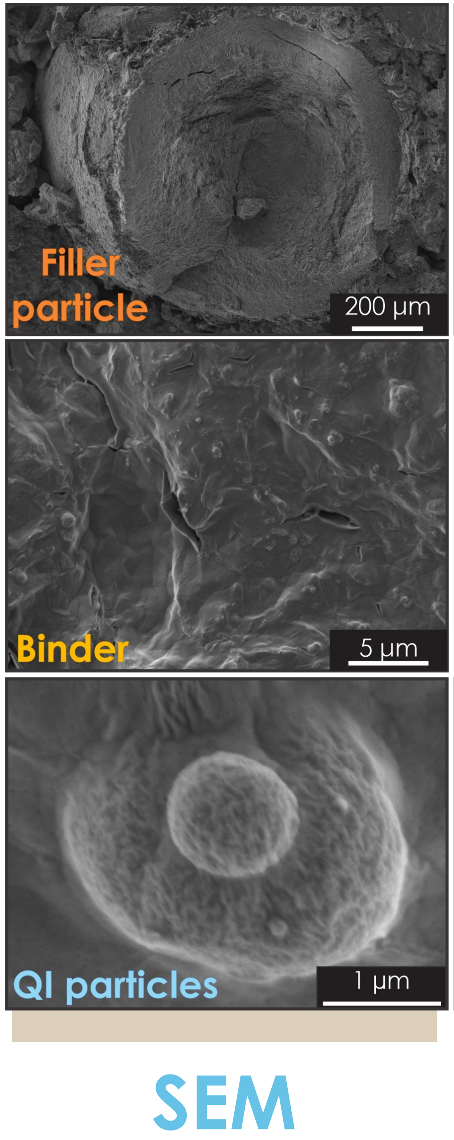
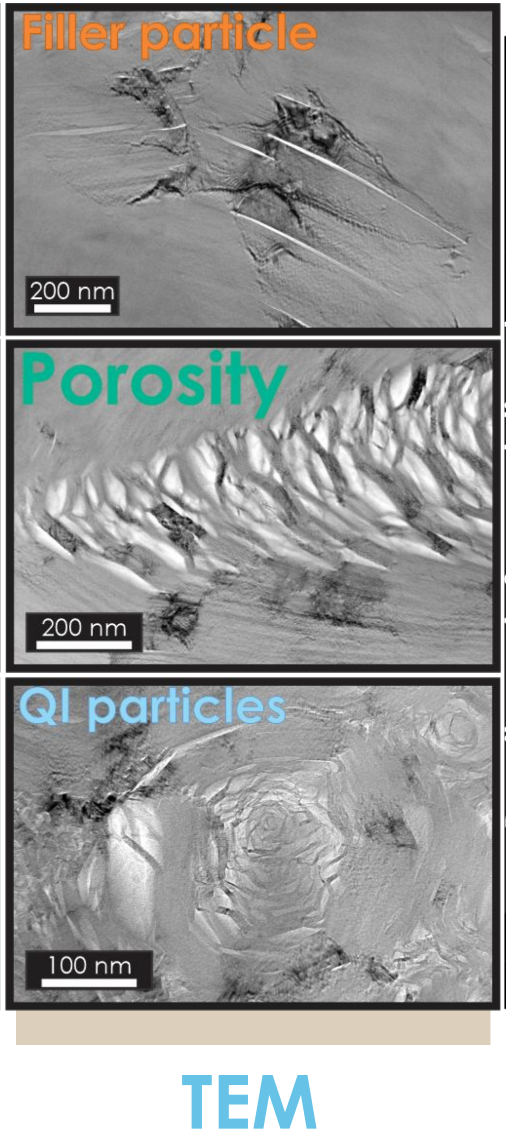
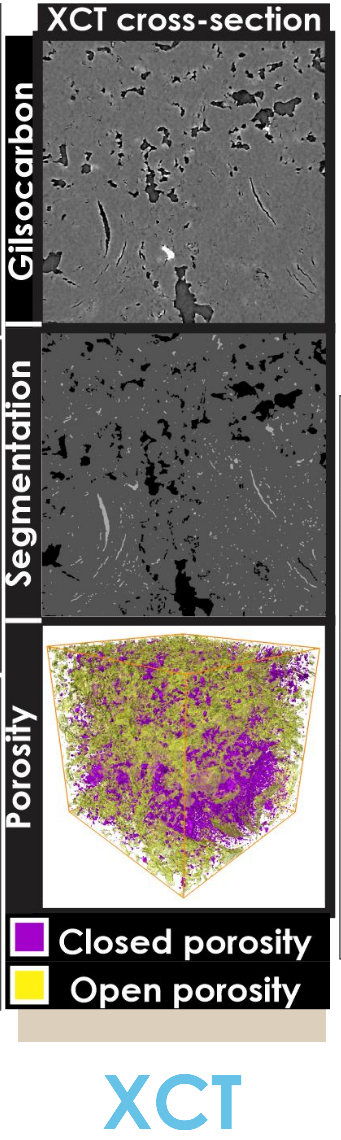
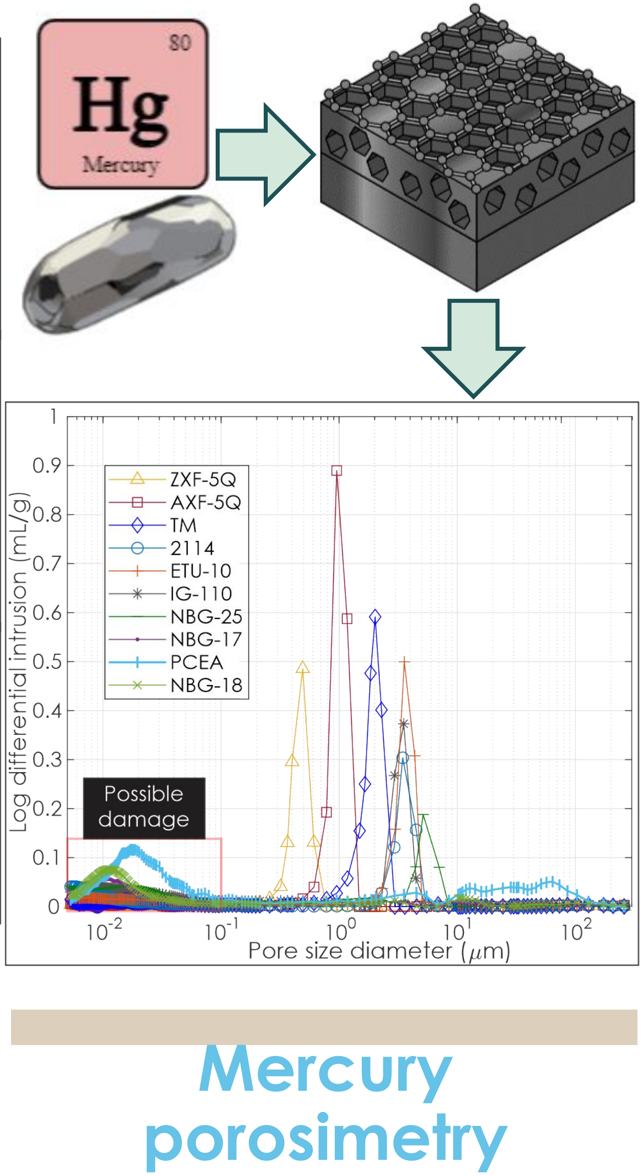
Library of microstructure



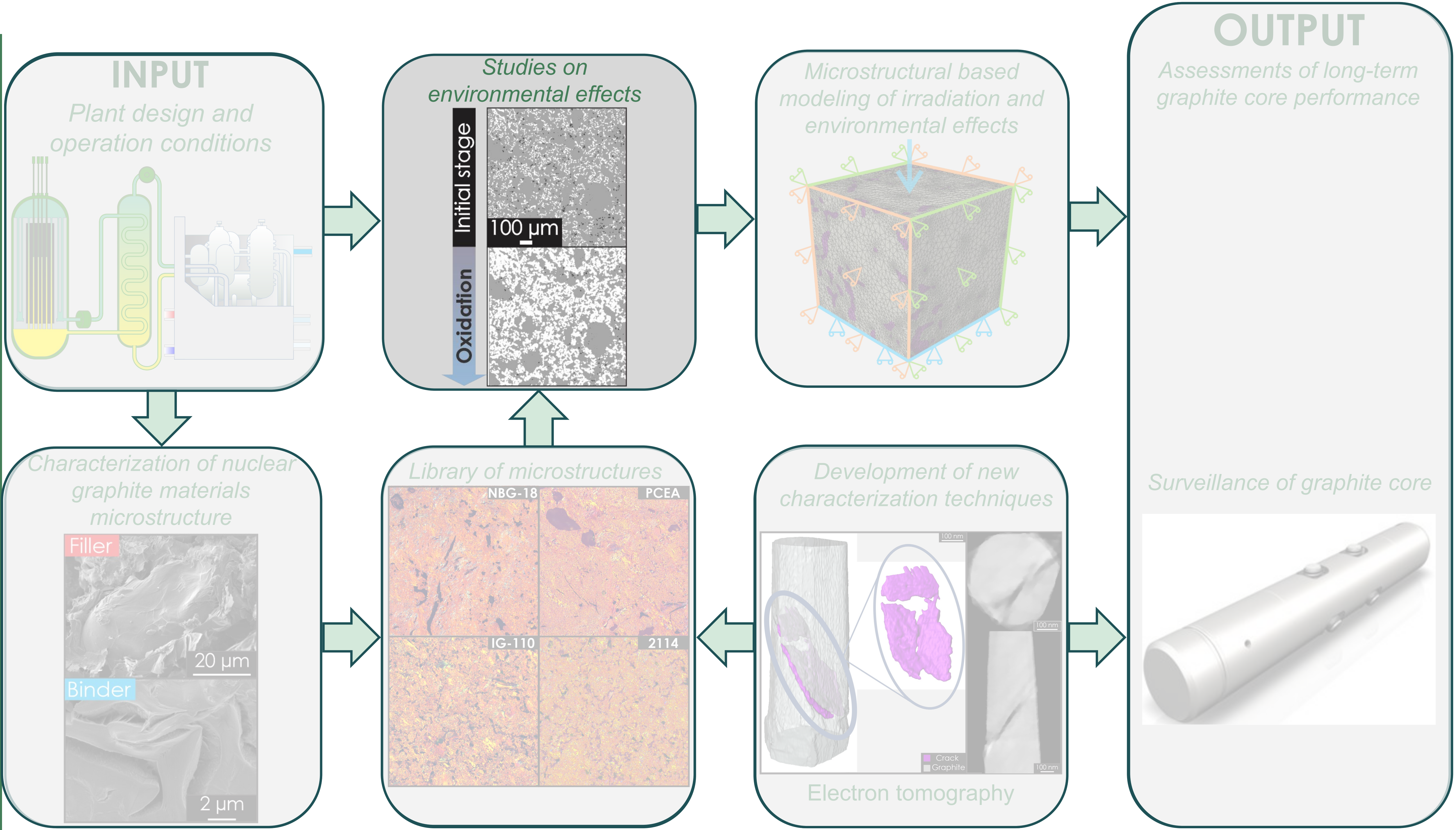








J.D. Arregui-Mena, R.N. Worth, W. Bodel, B. März, W. Li, A.A. Campbell, E. Cakmak, N. Gallego, C. Contescu, P.D. Edmondson, Multiscale characterization and comparison of historical and modern nuclear graphite grades, Materials Characterization 190 (2022) 112047. https://doi.org/https://doi.org/10.1016/j.matchar.2022.112047.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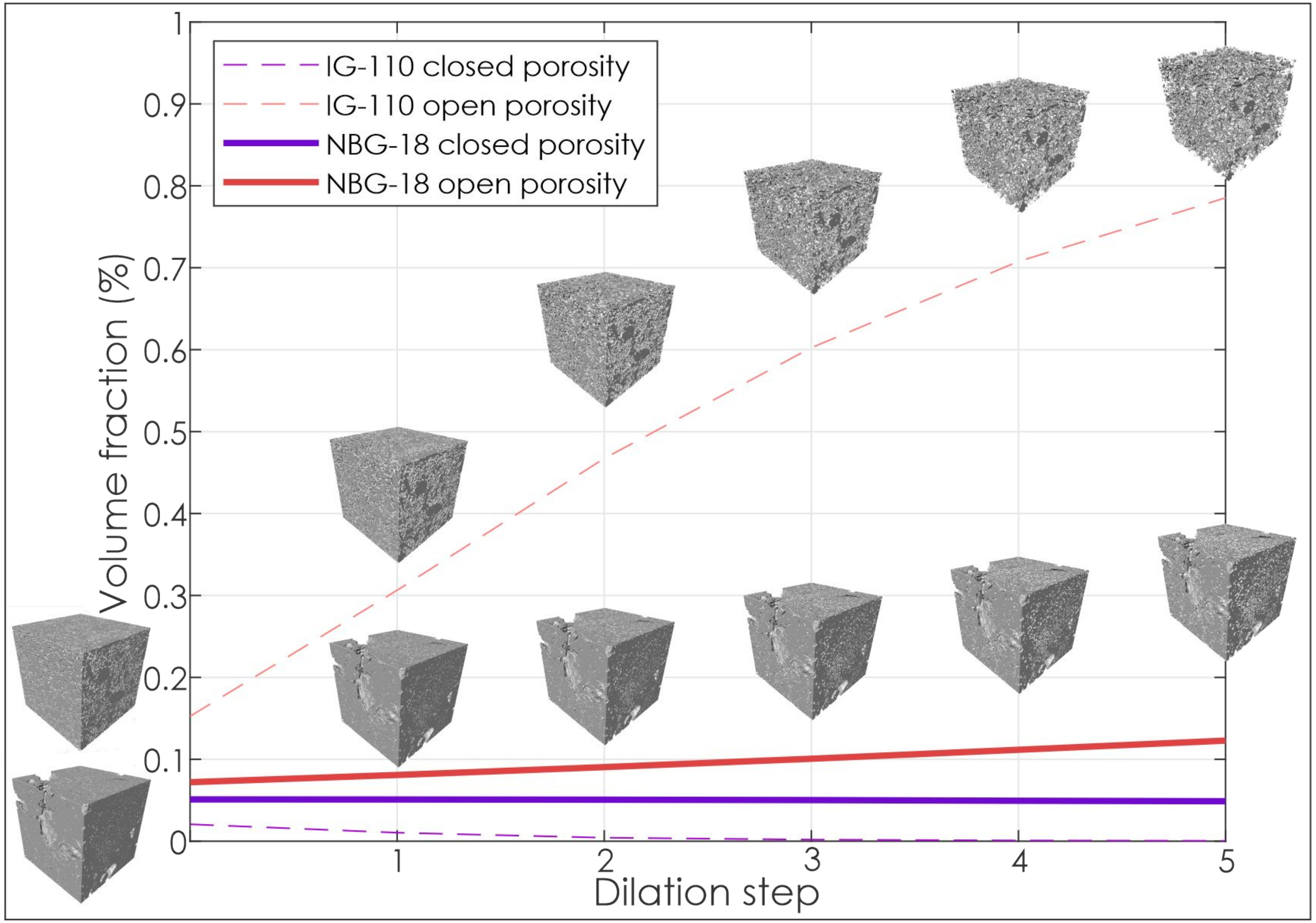
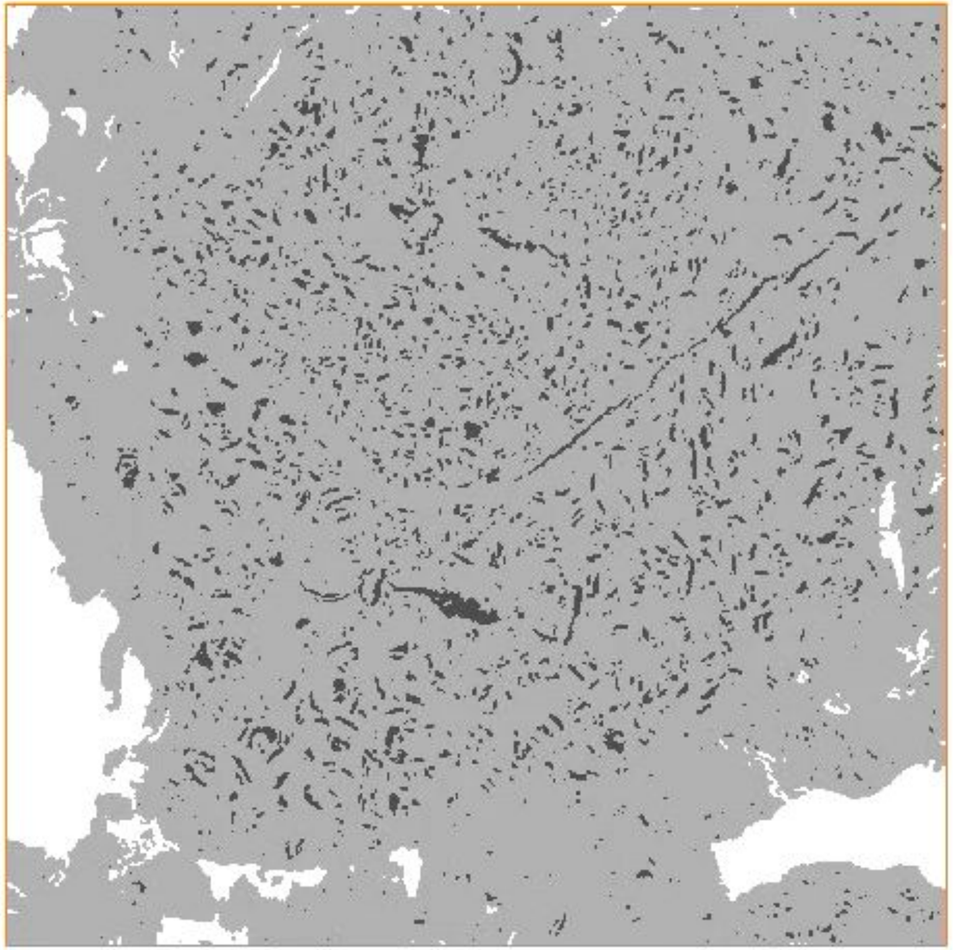
Environmental effects



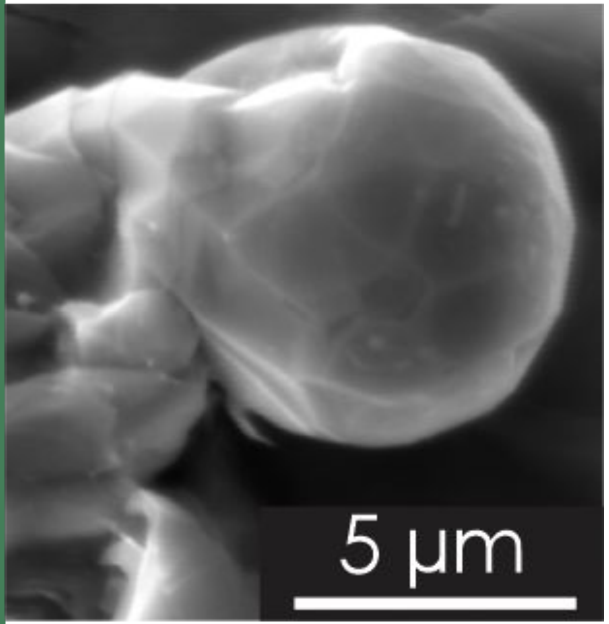
Uniform oxidation of NBG-18

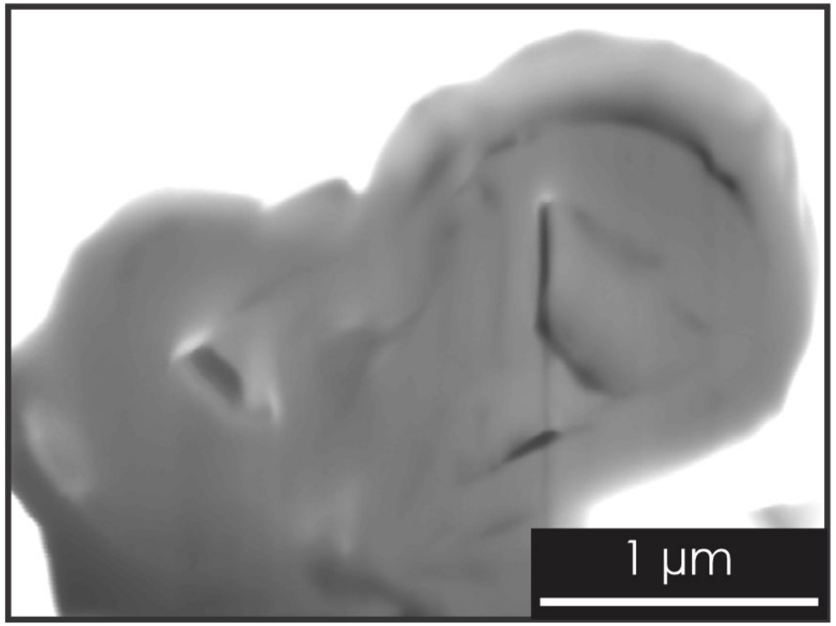


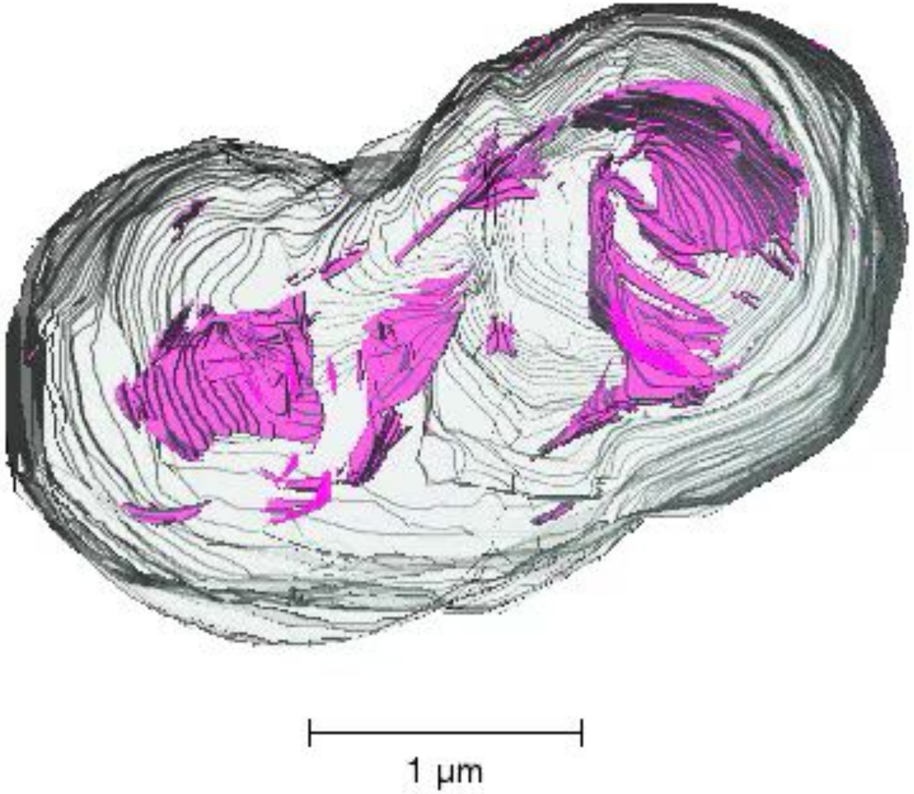
Oxidation generate new porosity and degrades the mechanical properties

QI particles – A forensic fingerprint of irradiation effects

- QI particles are a phase of graphite commonly found in the binder material







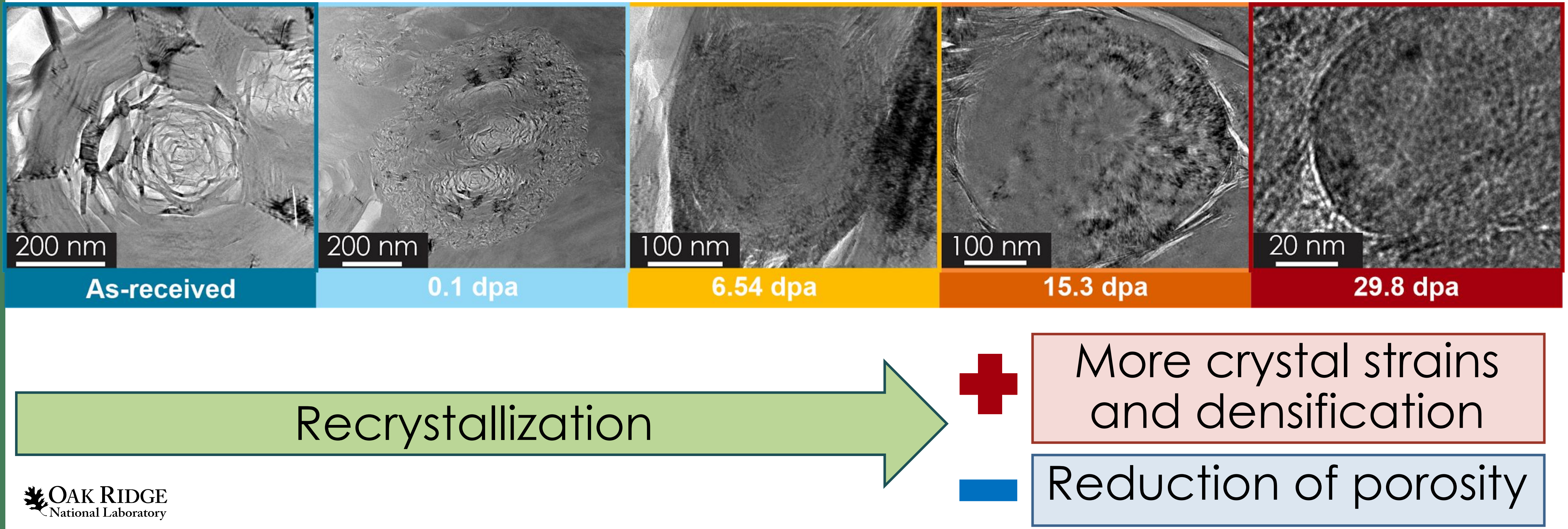
Surface

Cross-section

3D model

QI particles a marker to estimate neutron irradiation effects

- QI particles undergo morphological changes induced by neutron irradiation
- The changes in these particles may be used as a marker to determine the degree of neutron induce damage in graphite components or trepanned samples



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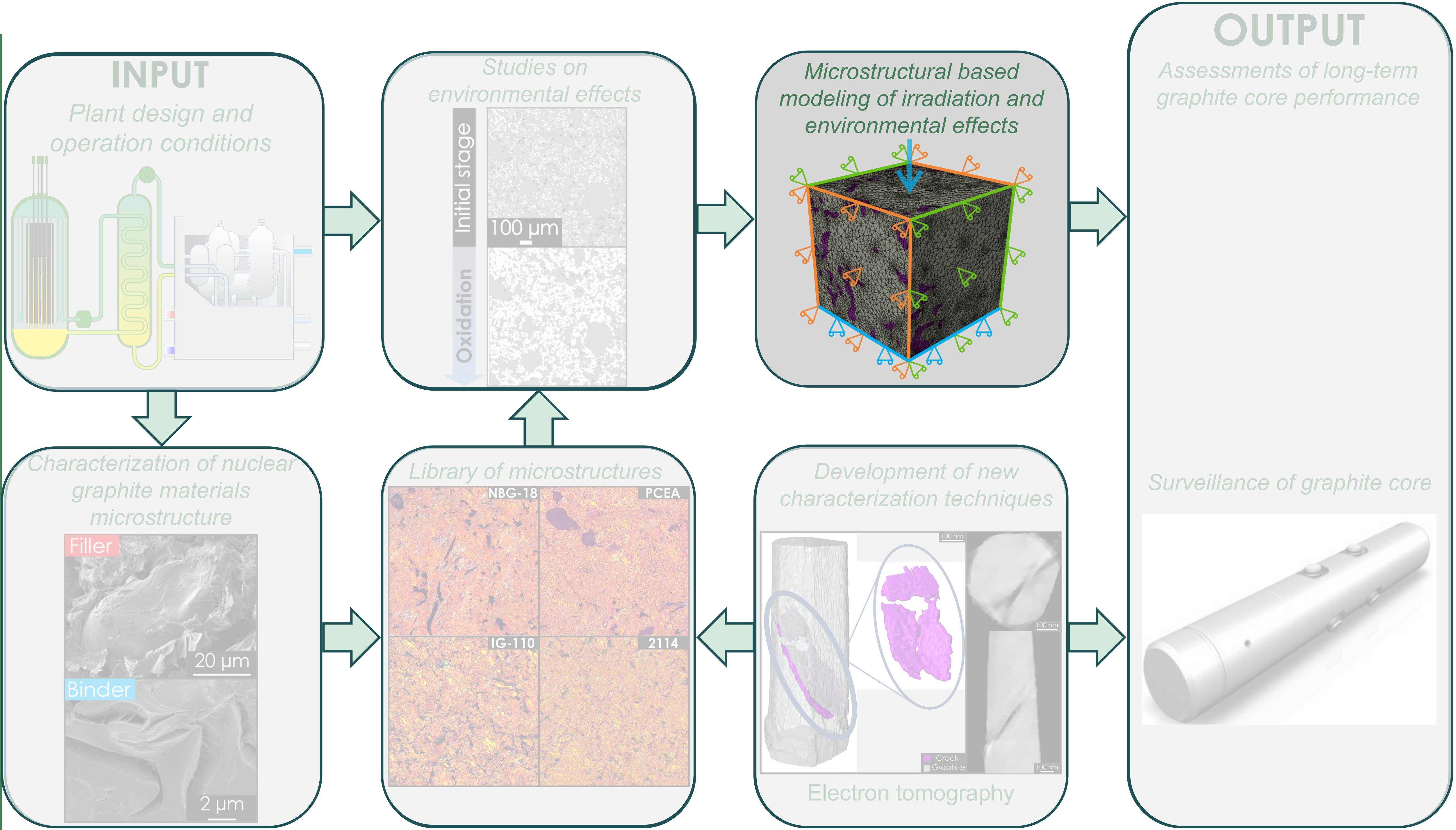
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Modeling of graphite: linking microstructure and full-length components

Objective

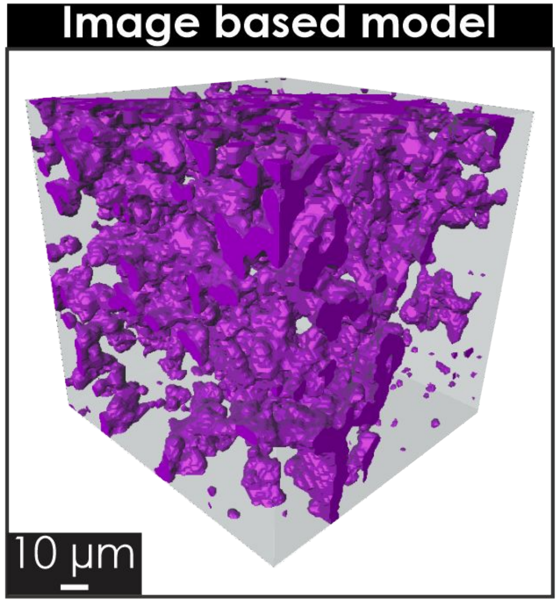
- Modeling the irradiation effects of graphite can benefit from accounting for defects created from oxidation or irradiation effects

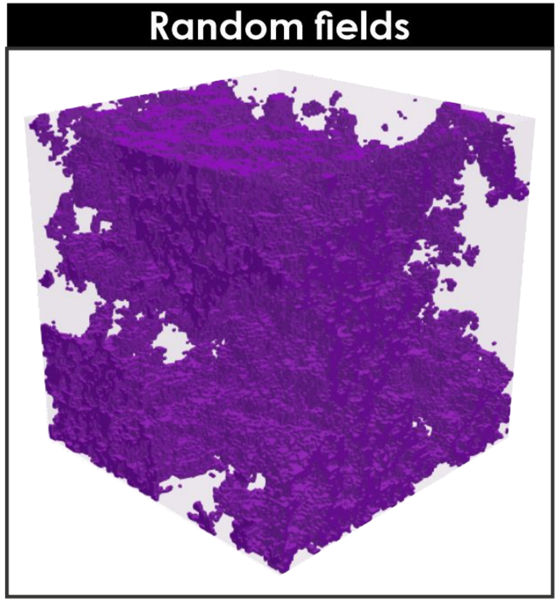
Strategies

- We have adopted two modeling strategies: Random field theory and Image-based modeling

Benefits

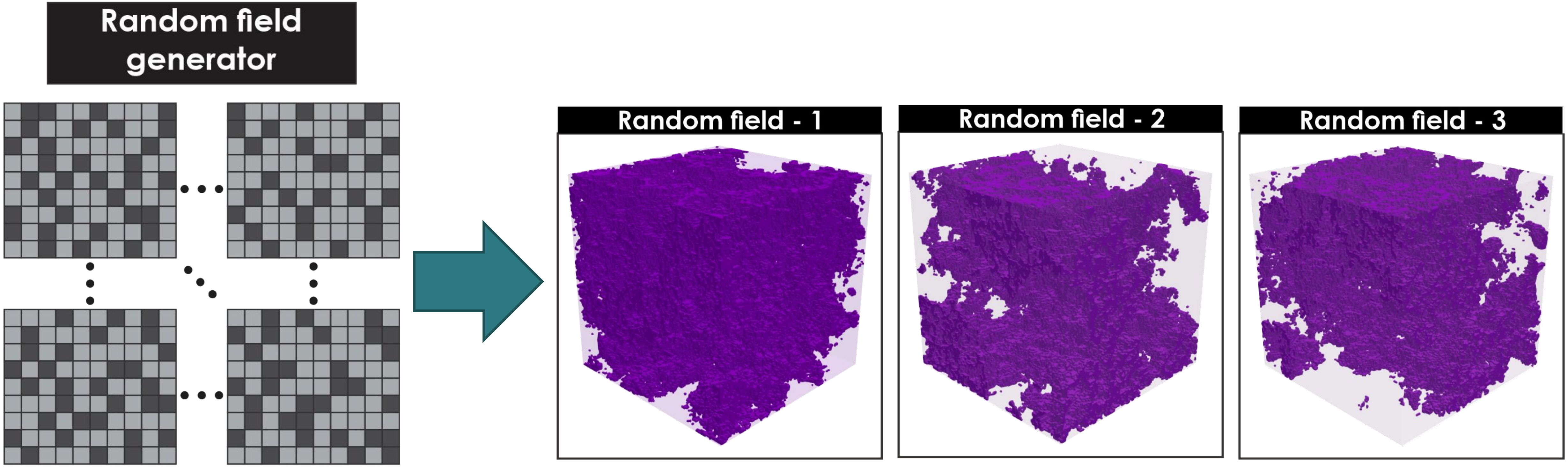
- Account for microstructural variations in the same type and different grades of graphite





Microstructural based models – Random field theory

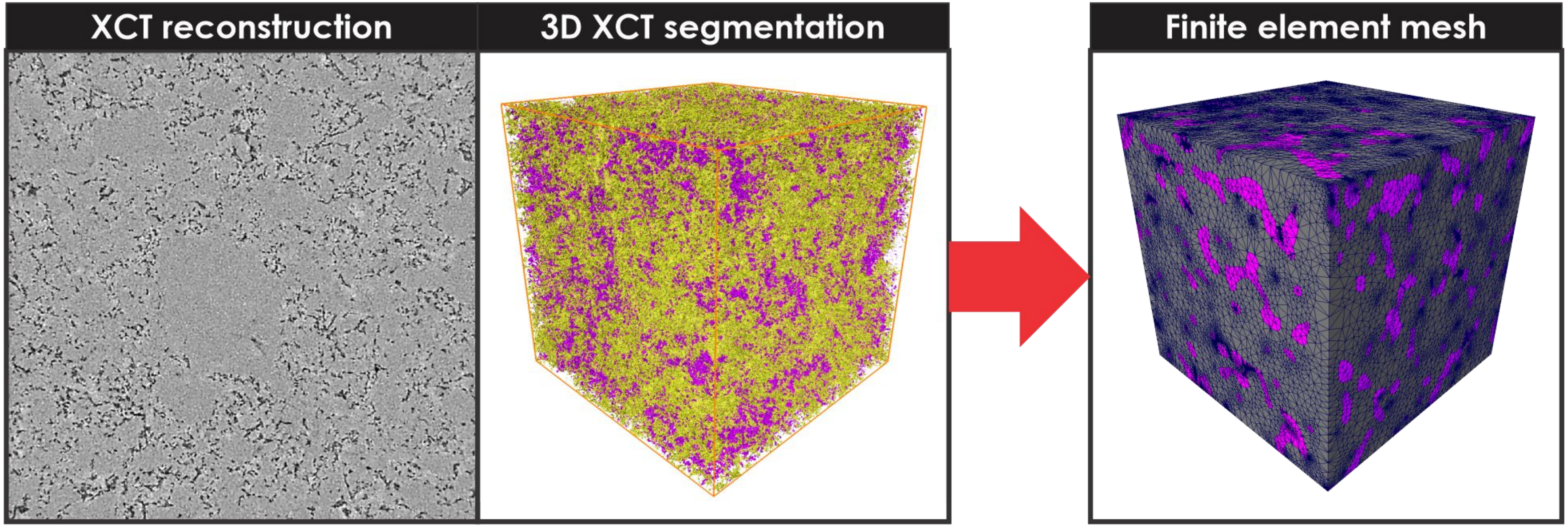
- *In-silico* or artificial microstructures can be generated by using random field theory



- Random field generators only require a few input parameters to produce the virtual microstructures of graphite

Microstructural based models – Image-based models

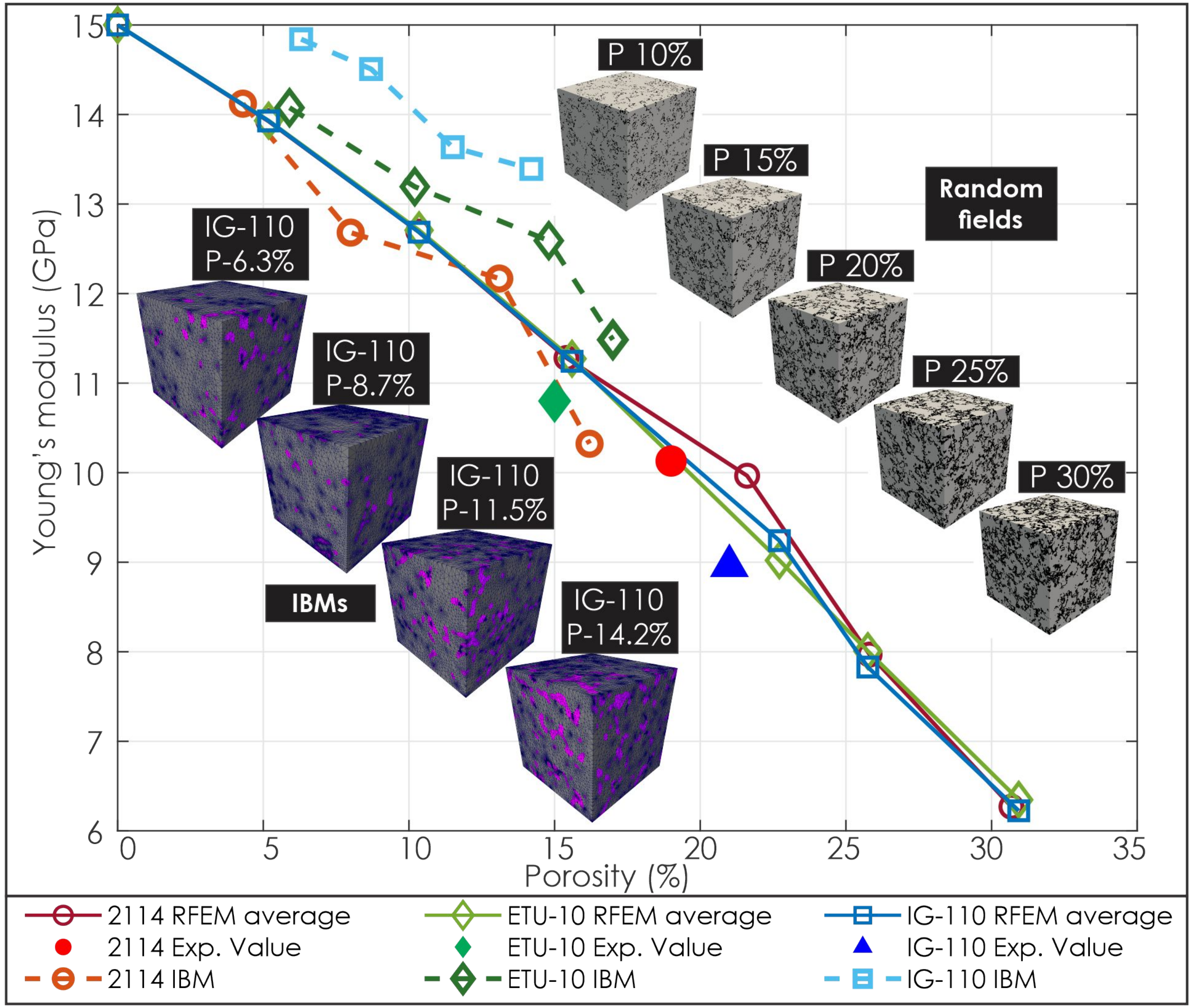
- The image-based model approach is to create digital twins of the microstructure of graphite



- For this approach is necessary to characterize the microstructure of graphite in a 2D or 3D space

Oxidation effects and elastic response

- The oxidation effects on the mechanical properties of nuclear graphite can be estimated using these two modeling techniques
- The result of this models can be used as part of a multiscale approach and help calibrate full scale components models



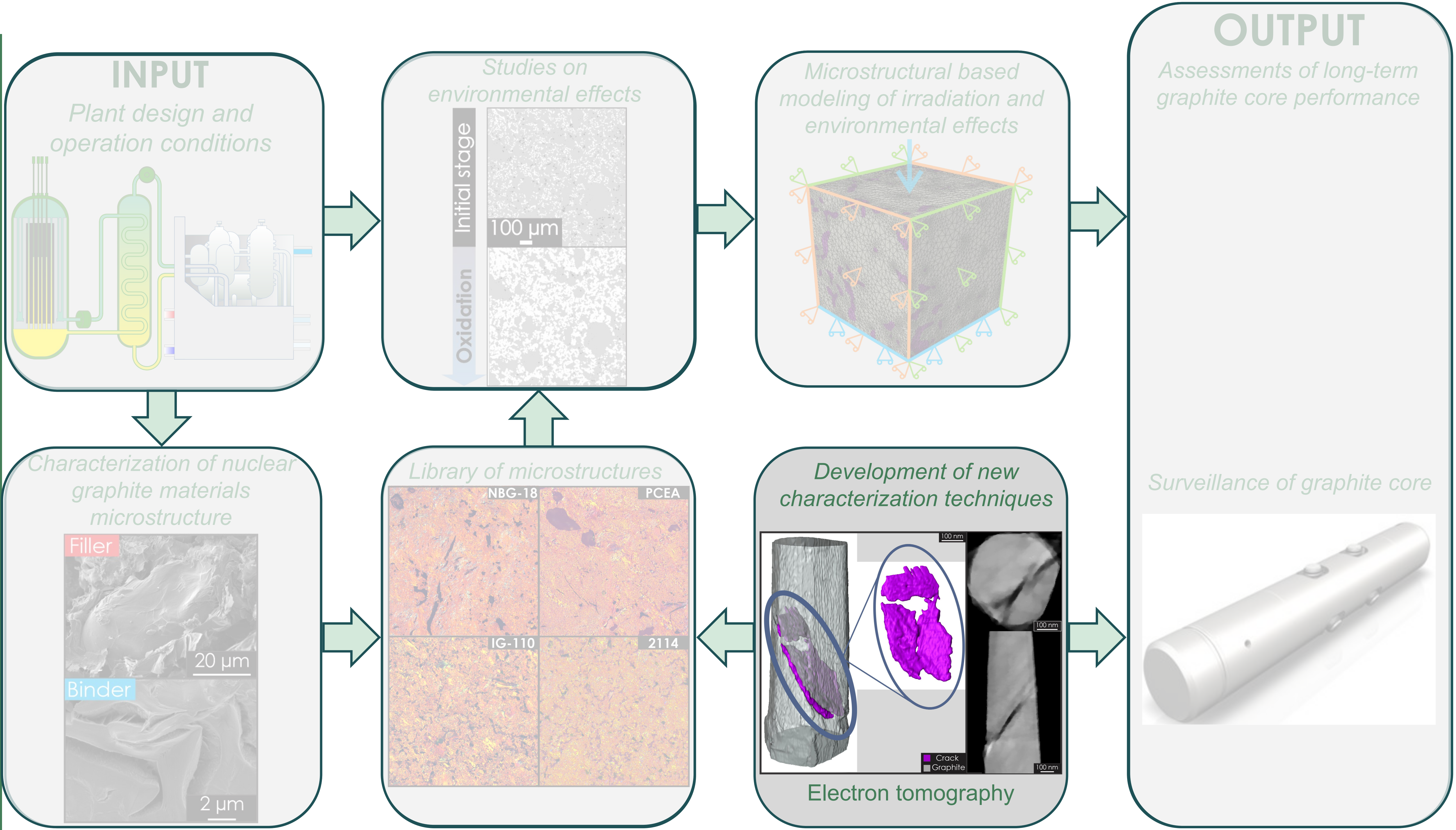
J.D. Arregui-Mena, D.V. Griffiths, R.N. Worth, C.E. Torrence, A. Selby, C. Contescu, N. Gallego, P.D. Edmondson, P.M. Mummery, L. Margetts, Using porous random fields to predict the elastic modulus of unoxidized and oxidized superfine graphite, Materials & Design 220 (2022) 110840. https://doi.org/https://doi.org/10.1016/j.matdes.2022.110840.

Image-based modeling based on XCT data

- Image-based models can also help to understand the oxidation effects on other properties such as thermal conductivity and absolute permeability

Thermal conductivity

Absolute permeability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Conclusions

- Characterization of porosity a various length scales plays a critical role to understand the behavior of graphite under various operating conditions
- Detail characterization of graphite contributes to understanding the fundamental mechanisms that lead to the degradation of graphite
- A comprehensive library of microstructures will help to inform models of graphite for multiple modeling aspects at different length scales
- Microstructural based models and other modeling techniques will help to improve the modeling of irradiated graphite components and introduce the inherit heterogeneity of graphite
- More detailed microstructural information will help to determine the crack propagation and failure mechanisms of graphite components

Publications

- **UPDATE LIST**
- CI Contescu, JD Arregui-Mena, AA Campbell, PD Edmondson, NC Gallego, K Takizawa, Y Katoh, “Development of mesopores in superfine grain graphite neutron-irradiated at high fluence” *Carbon* **141** (2019) Pages 663-675
- LR Olasov, FW Zeng, JB Spicer, NC Gallego, CI Contescu, “Modelling the effects of oxidation-induced porosity on the elastic moduli of nuclear graphites” *Carbon* **141** (2019) Pages 304-315
- CI Contescu, JB Spicer, NC Gallego, AA Campbell, JD Arregui-Mena and Tim Burchell “Assessment of Neutron Damage in Irradiated Graphite using Gas Adsorption Methods” *INGSM-2020*, September 2019
- AA Campbell, CI Contescu, E Carkmak, NC Gallego, TD Burchell, “Effects of High-Temperature Irradiation on the Microstructure of Nuclear Graphite”, ORNL Technical Report, **ORNL/TM-2019/1309**, September 2019.
- NC Gallego, CI Contescu, TD Burchell, “XRD and SANS Evaluation of HOPG and Polycrystalline Graphite”, ORNL Technical Report, **ORNL/TM-2018/871**, June 2018.
- JD Arregui-Mena, Contescu CI, Campbell AA, Edmondson PD, Gallego NC, Smith QB, Takizawa K, Katoh Y, “Nitrogen adsorption data, FIB-SEM tomography and TEM micrographs of neutron-irradiated superfine grain graphite” *Data in Brief* **2018**/11/28
- Contescu CI, RW Mee, JD Arregui-Mena, NC Gallego, TD Burchell, JJ Kane, WE Windes “Beyond the classical kinetic model for chronic graphite oxidation by moisture in high temperature gas-cooled reactors” *Carbon* **127** (2018) Pages 158-169

Thank you!

Questions???