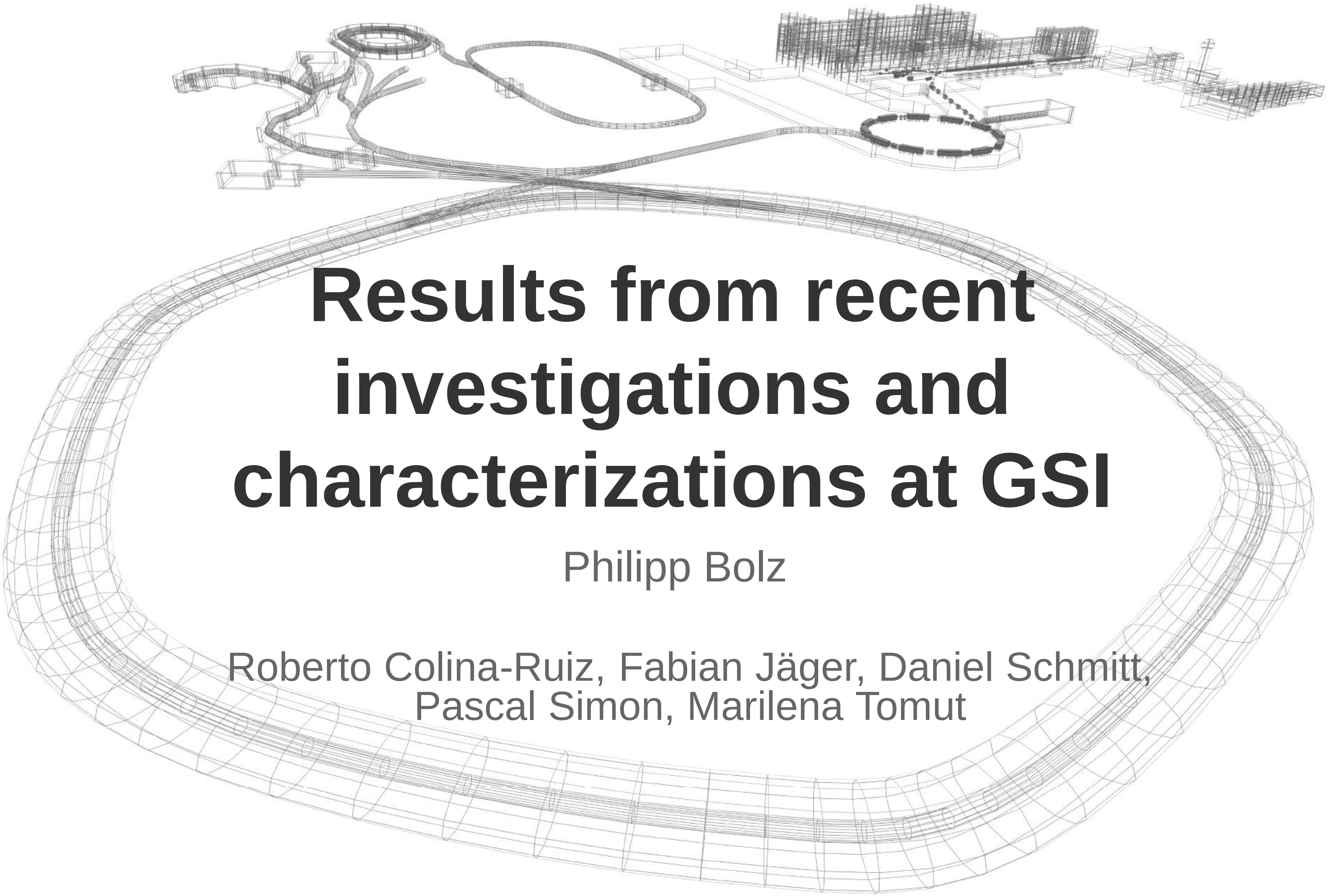


Results from recent investigations and characterizations at GSI

Philipp Bolz

Roberto Colina-Ruiz, Fabian Jäger, Daniel Schmitt, Pascal Simon, Marilena Tomut

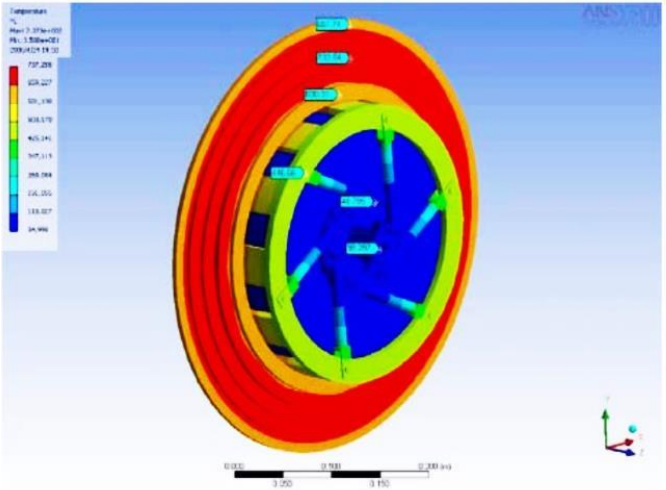
Table of content

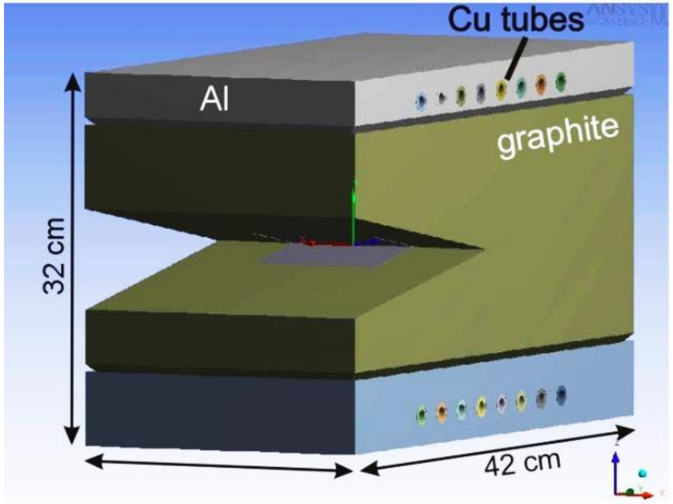
- Motivation
- Materials
- Structural characterization
  - Raman spectroscopy
  - Infrared thermography
- Functional characterization
  - Indentation
  - 3-point bending test
  - Laser flash analysis
- Conclusion and outlook

Motivation

- Properties of graphite:
  - Low density
  - High thermal conductivity
  - Low coefficient of thermal expansion
  - High service temperature

- Graphite materials are used for:
  - Target wheel and beam catchers in Super Fragment Separator
  - Beam dumps in experimental caves
  - Collimators

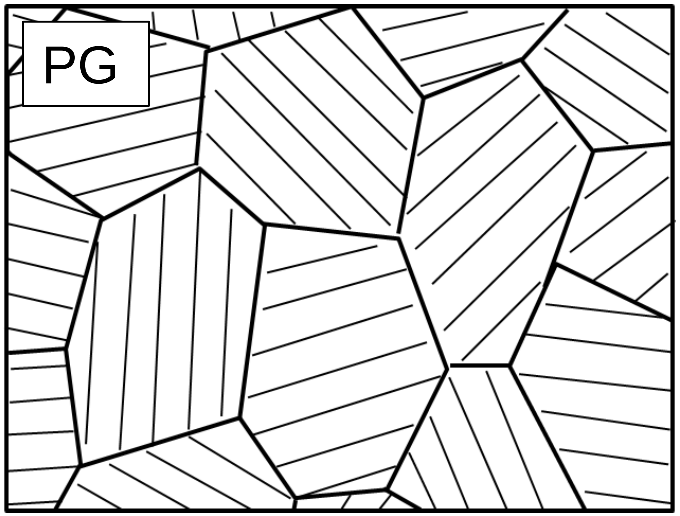




Super-FRS working group, 2008

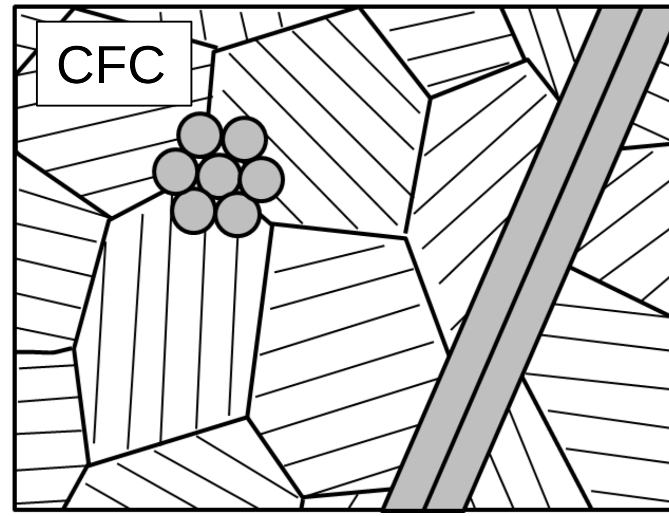
Investigated materials

- Polycrystalline graphite (PG)



Material	Particle size [µm]
R6300	20
R6500	10
R6550	10
R6650	7
POCO ZEE	1

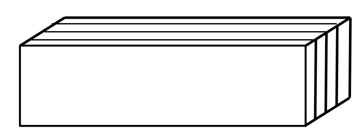
- Carbon fibre reinforced carbon (CFC)



- 2 dimensional fibre orientation
- Fibre plane parallel or perpendicular to surface

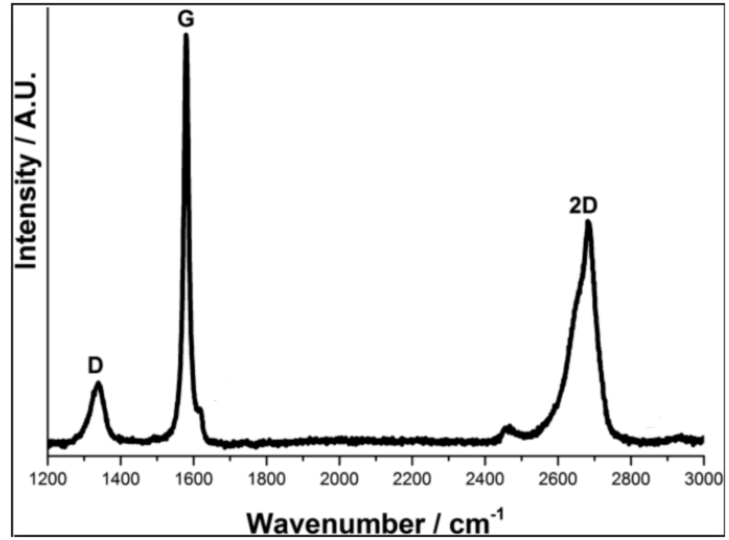
In plane ∥

Transversal ⊥



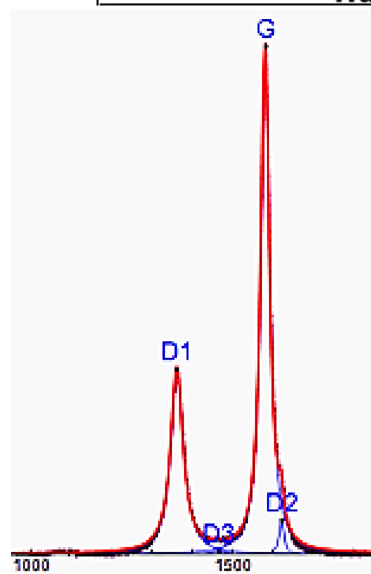
Structural characterization
Raman spectroscopy

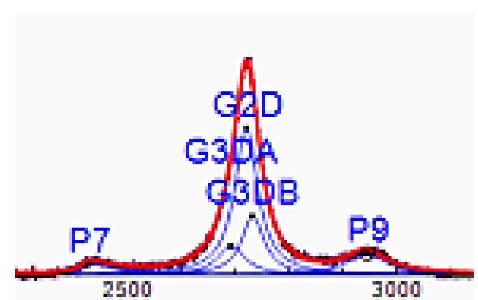
- Main peaks for graphite:
  i. D-peak ≈ 1380 cm^{-1}
  ii. G-peak ≈ 1580 cm^{-1}
  iii. 2nd D-peak ≈ 2700 cm^{-1}
- Ratio of I_D/I_G allows determination of defect concentration and lattice parameters



- Bands included for Raman spectra deconvolution:

i. D1-D3: Lorentzians
ii. D2: Gaussian
iii. G: Lorentzian
iv. G‘2D-G‘3DA-G‘3DB: Lorentzians
v. P7: Splitlorentzian
vi. P9: Lorentzian



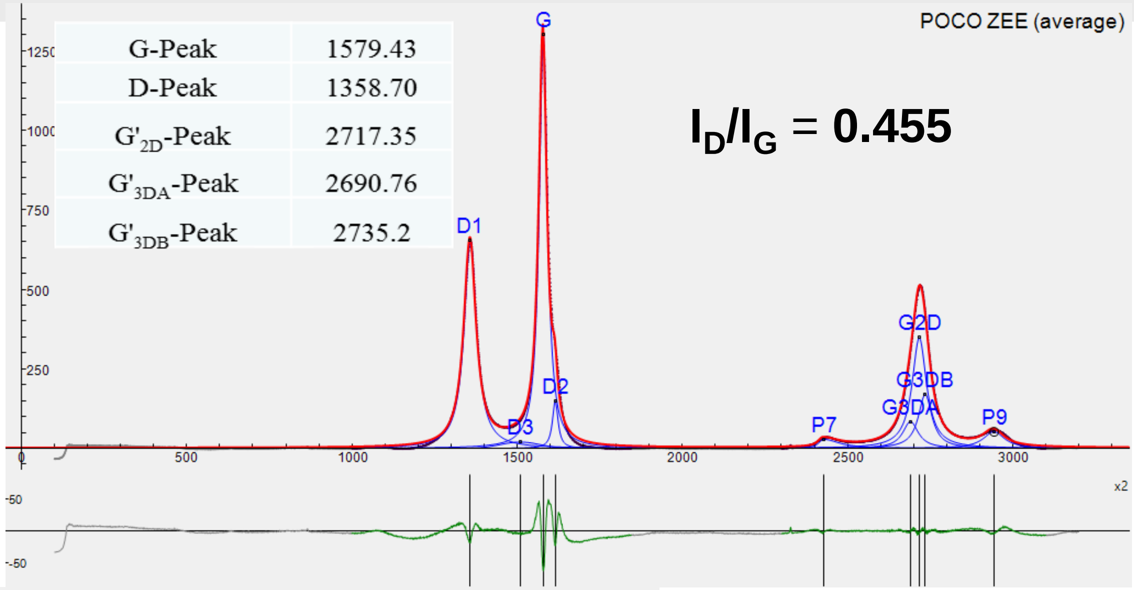


N. Larouche, B.L. Stansfield, Carbon N. Y. 48 pp. 620–629, 2009

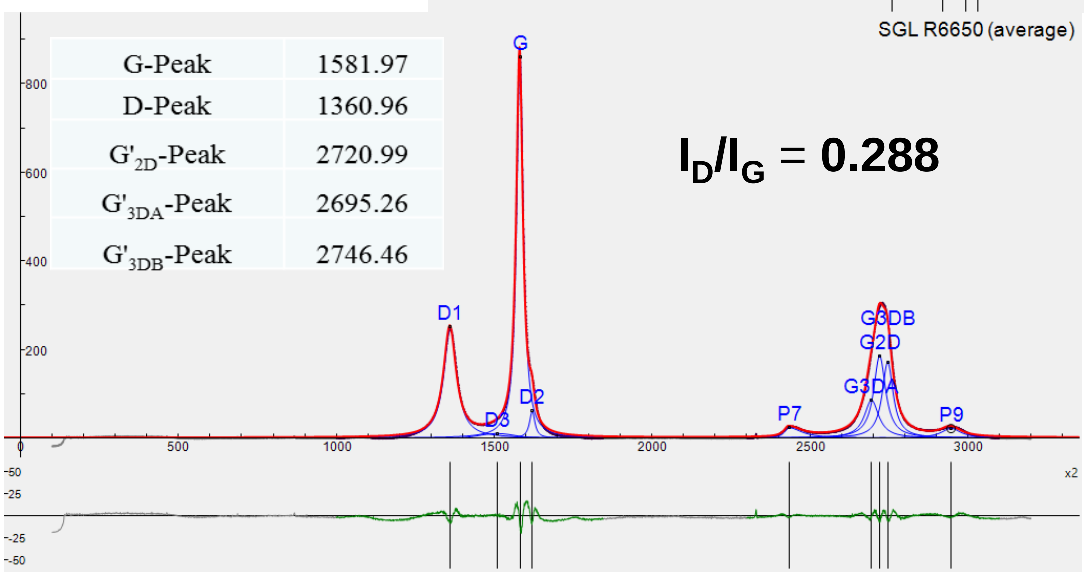
Raman results of isotropic polycrystalline graphite grades

POCO ZEE

Particle size: 1 µm
Porosity: 20%
Pore size: 0.3 µm
Density: 1.77 g/cm^3



G-Peak	1579.43
D-Peak	1358.70
G'$_{2D}$-Peak	2717.35
G'$_{3DA}$-Peak	2690.76
G'$_{3DB}$-Peak	2735.2



G-Peak	1581.97
D-Peak	1360.96
G'$_{2D}$-Peak	2720.99
G'$_{3DA}$-Peak	2695.26
G'$_{3DB}$-Peak	2746.46

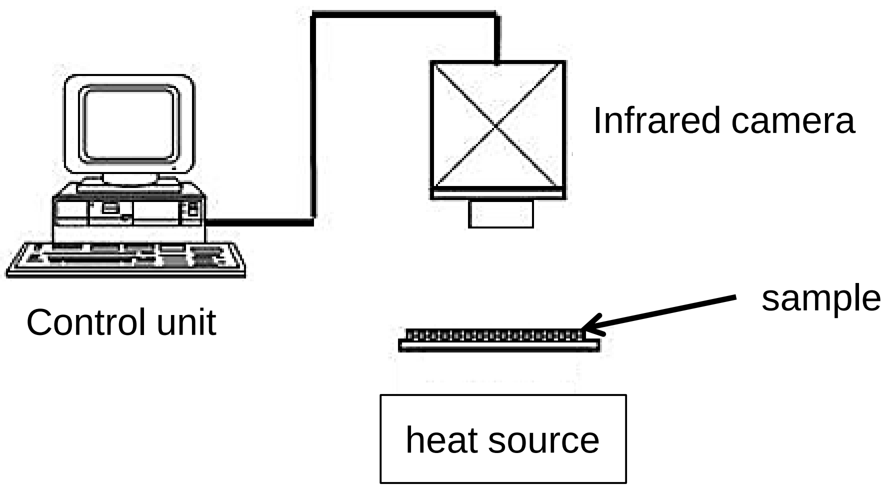
SGL R6650

Particle size: 7 µm
Porosity: 10%
Pore size: 1.1 µm
Density: 1.84 g/cm^3

Infrared thermography

- Infrared thermography acquires and processes thermal information from non-contact measurement devices

Experimental setup

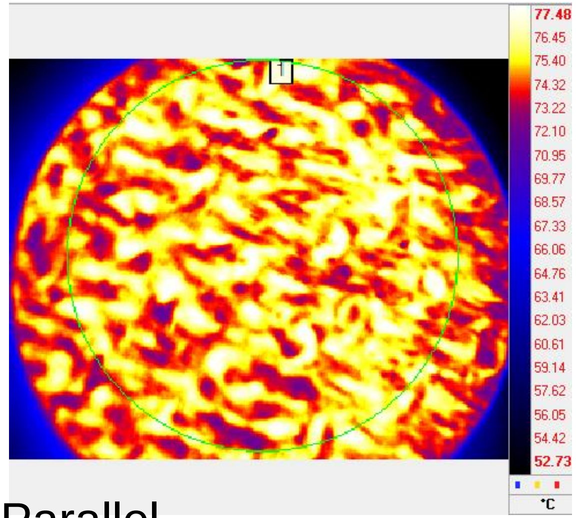


Infrared thermography

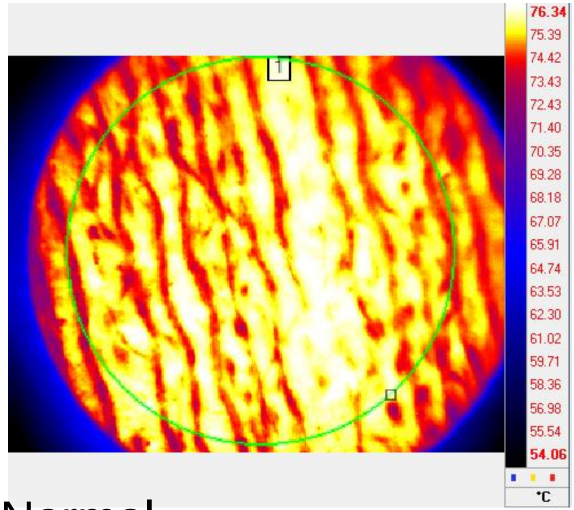
2D-CFC

- *SGL Mechanical*

Fibre plane parallel to surface



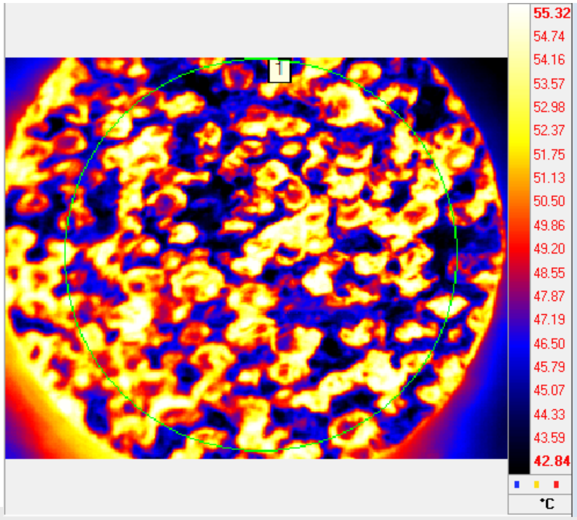
Fibre plane normal to surface



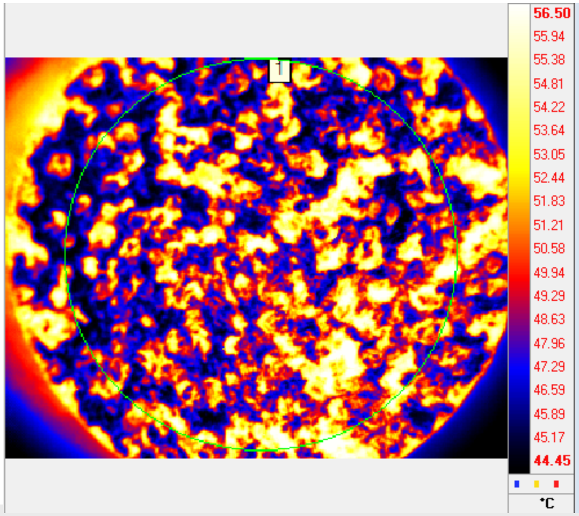
Graphitic Foam

- *POCO FOAM*

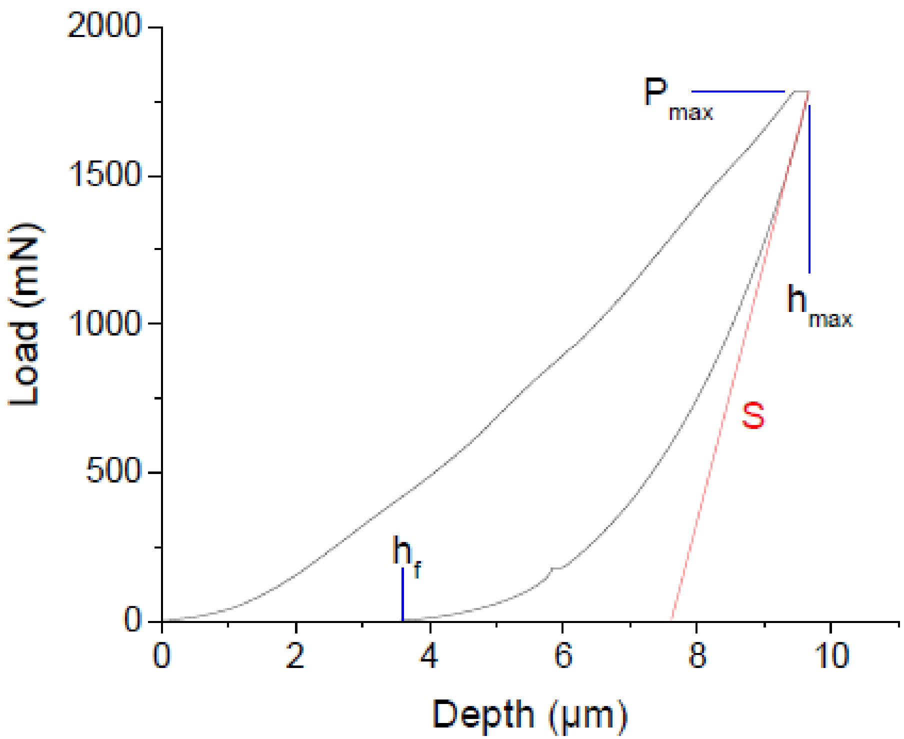
Parallel



Normal



Functional characterization
Microindentation



Area function A:

$$A = 24.5\, h_f^2$$

Hardness H:

$$H = \frac{P_{max}}{A}$$

Reduced modulus E_r:

$$E_r = \frac{\sqrt{\pi} \cdot S}{2 \cdot \beta \cdot \sqrt{A}}$$

Young's modulus E:

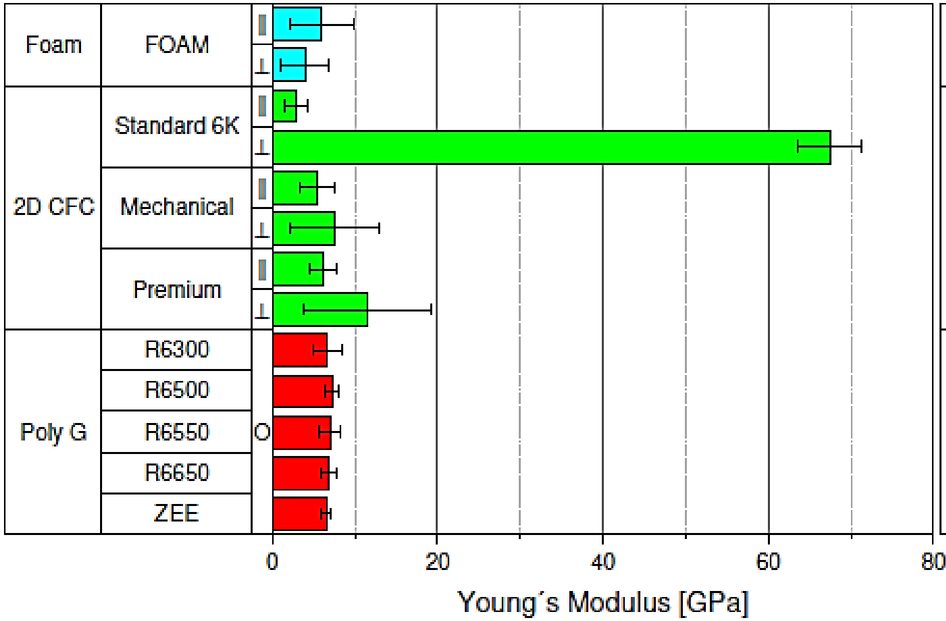
$$\frac{1}{E_r} = \frac{1-\vartheta^2}{E} + \frac{1-\vartheta_i^2}{E_i}$$

β = Geometrical correction factor
ϑ = Poisson's ratio

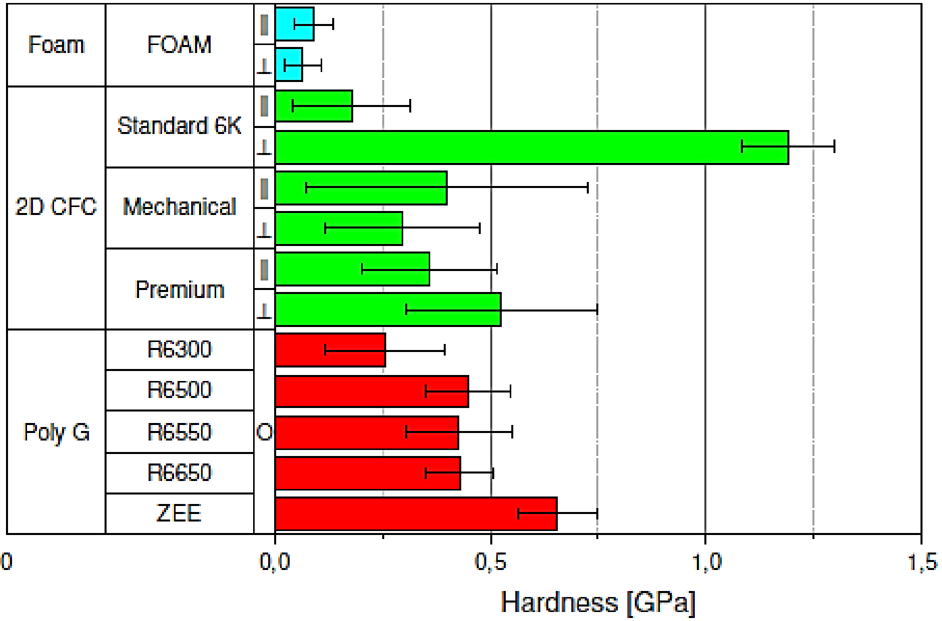
Oliver and Pharr, *J. Mater. Res.*, vol. 7, no. 6, pp. 1564-1583, 1992

Microindentation of investigated materials

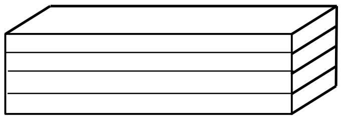
Young's modulus



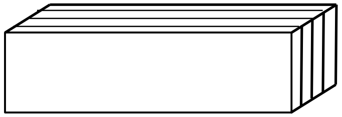
Hardness



In plane ‖



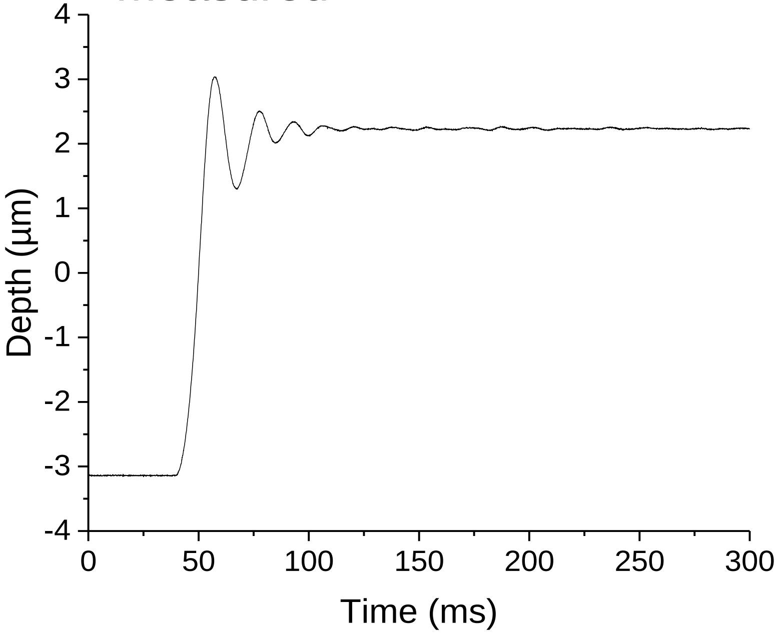
Transversal ⊥

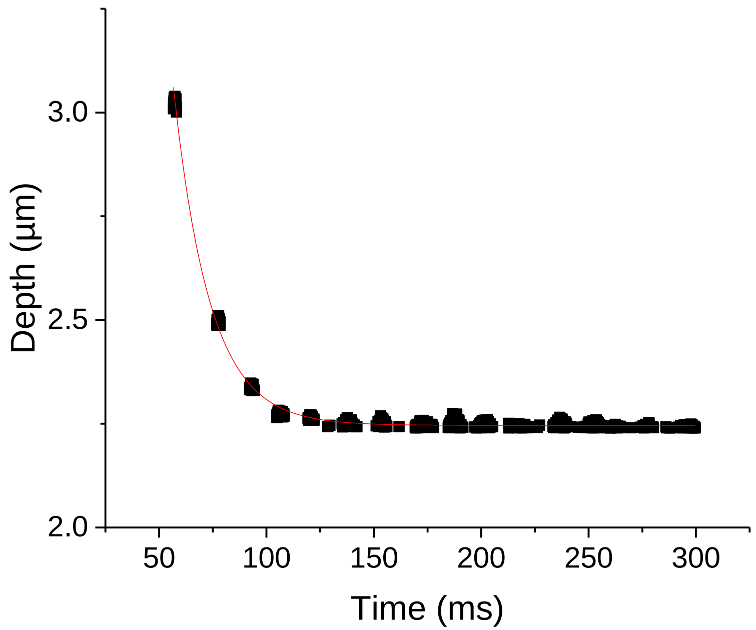


Material	Particle size [µm]
R6300	20
R6500	10
R6550	10
R6650	7
POCO ZEE	1

Dynamic indentation

- Indenter is accelerated with constant force towards the sample
- Resulting bouncing of the indenter on the surface of the sample is measured





Dynamic hardness DH:

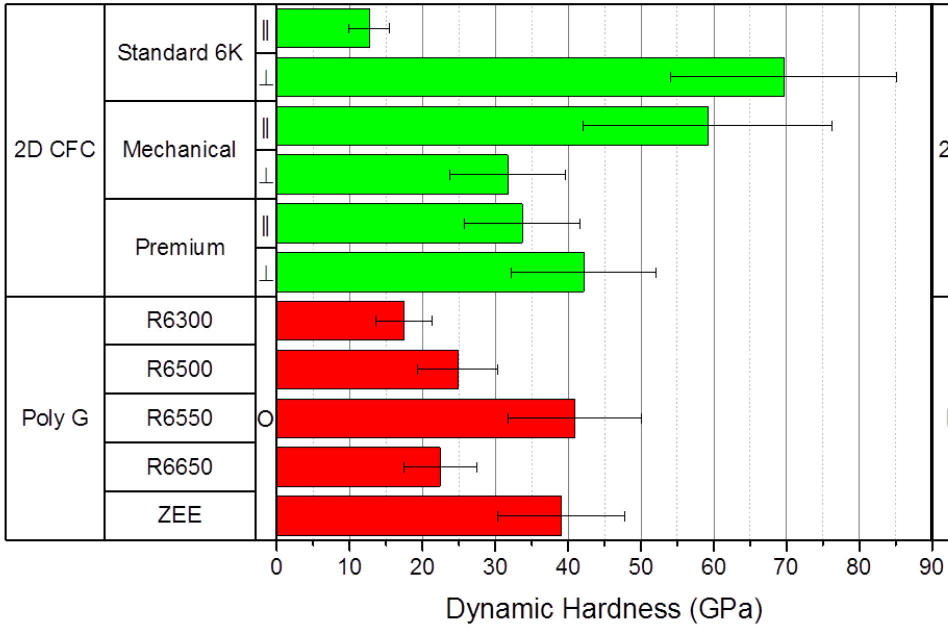
$$DH \propto \frac{v_{out}}{v_{in}}$$

Damping constant k:

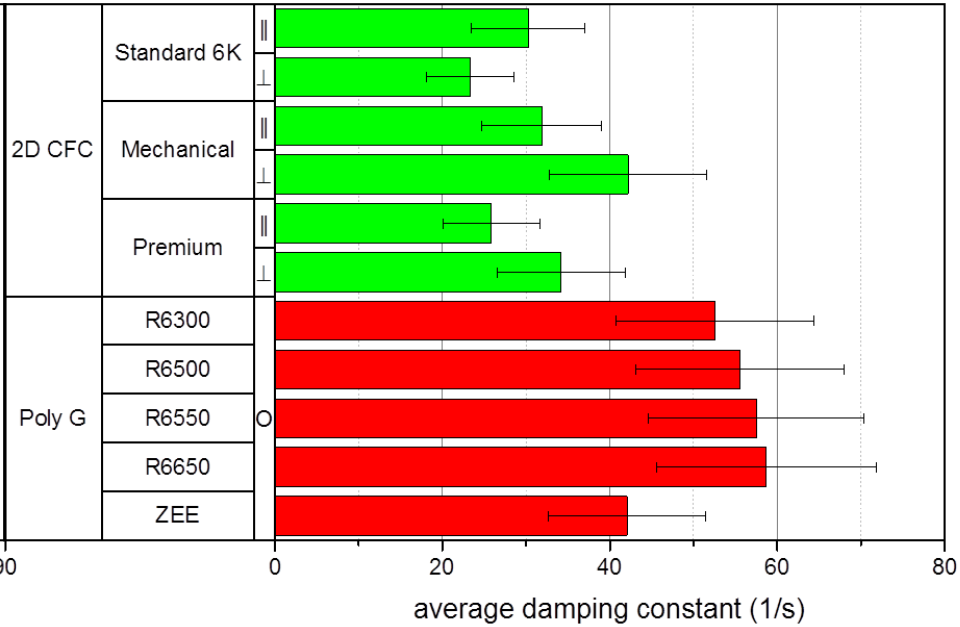
$$d = Ae^{-kt} + d_0$$

Dynamic indentation results of polycrystalline graphite and carbon fibre reinforced carbon grades

Dynamic hardness

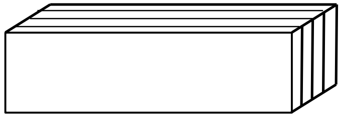


Damping constant



In plane ∥

Transversal ⊥

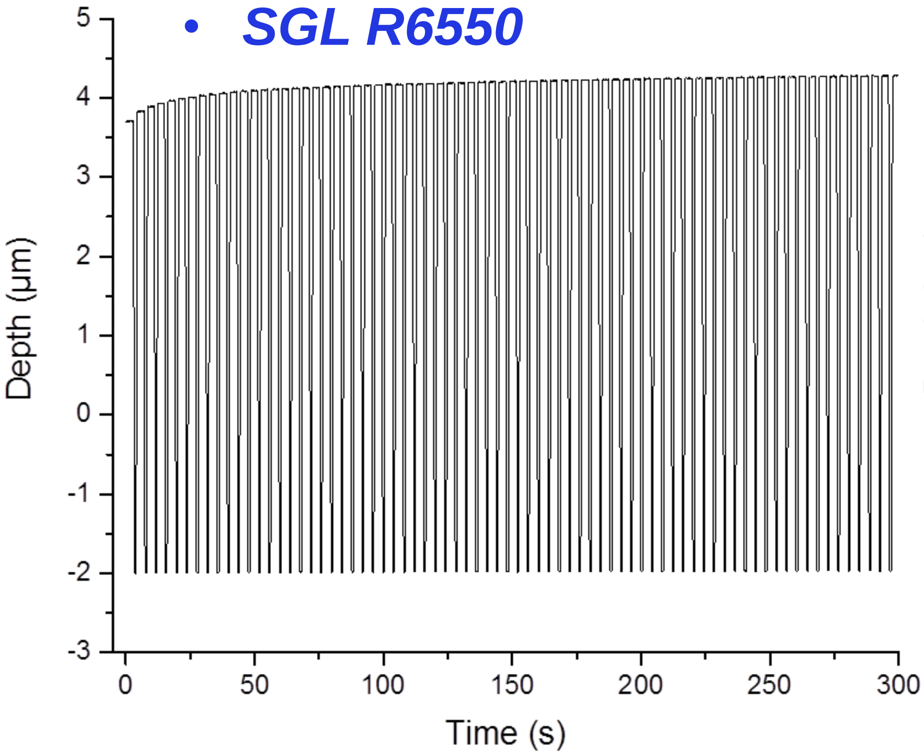


Multiple Impulse indentation

- Indenter is accelerated with constant force towards the sample
- Indenter is moved back to the initial position and accelerated again
- Allows measurement of fatigue

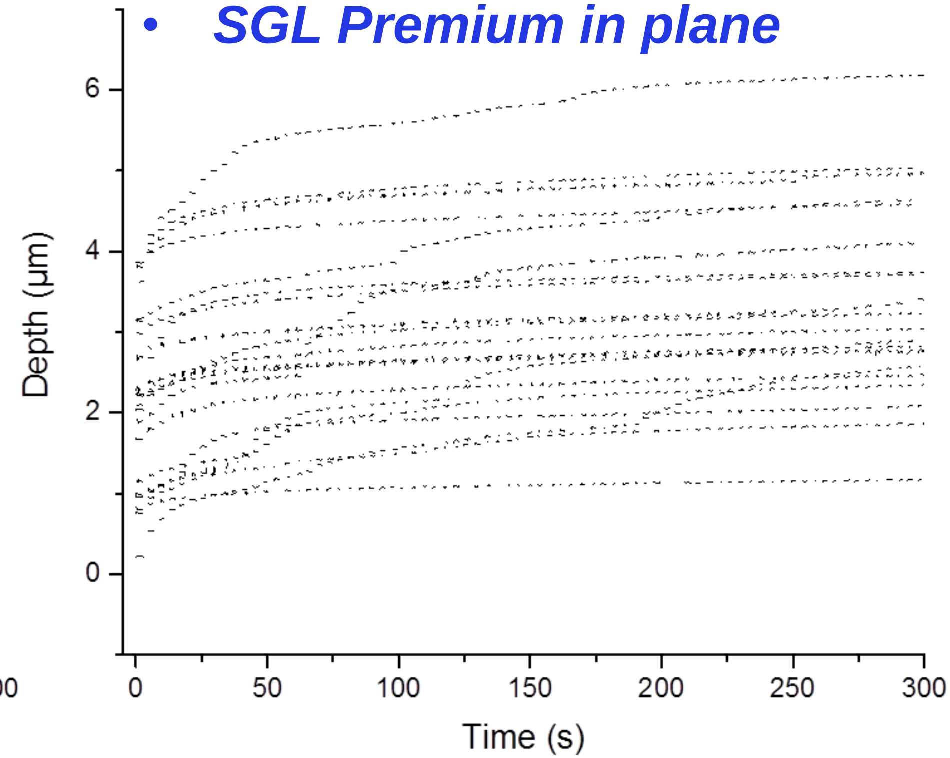
Polycrystalline graphite:

- ***SGL R6550***

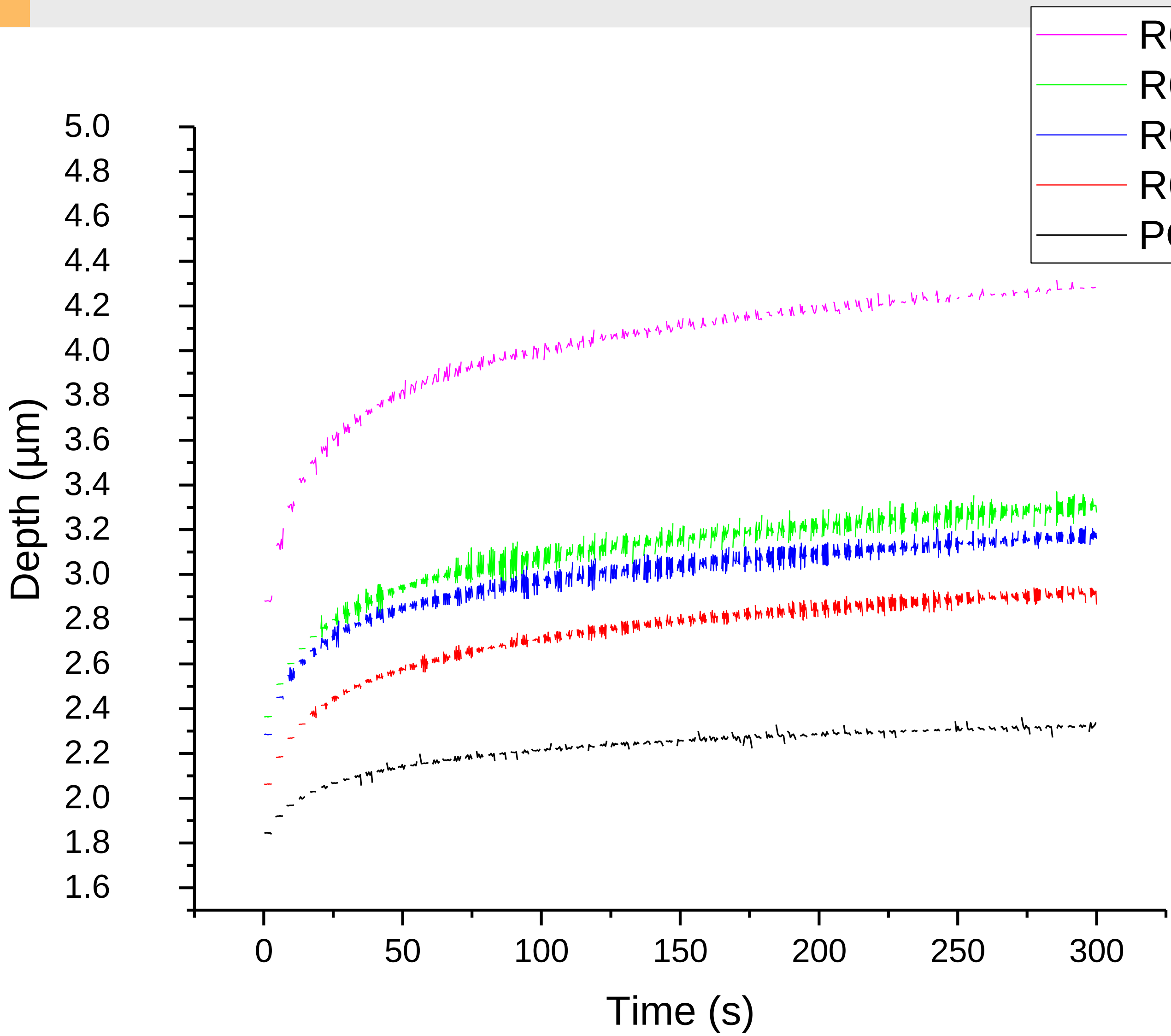


Carbon fibre reinforced carbon:

- ***SGL Premium in plane***



Multiple impulse results of polycrystalline graphite grades



Material	Particle size [µm]
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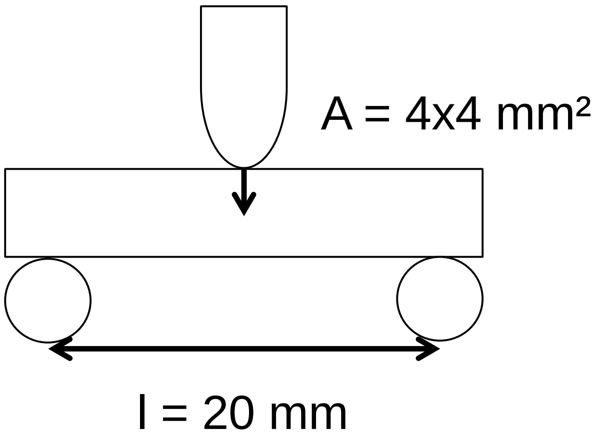
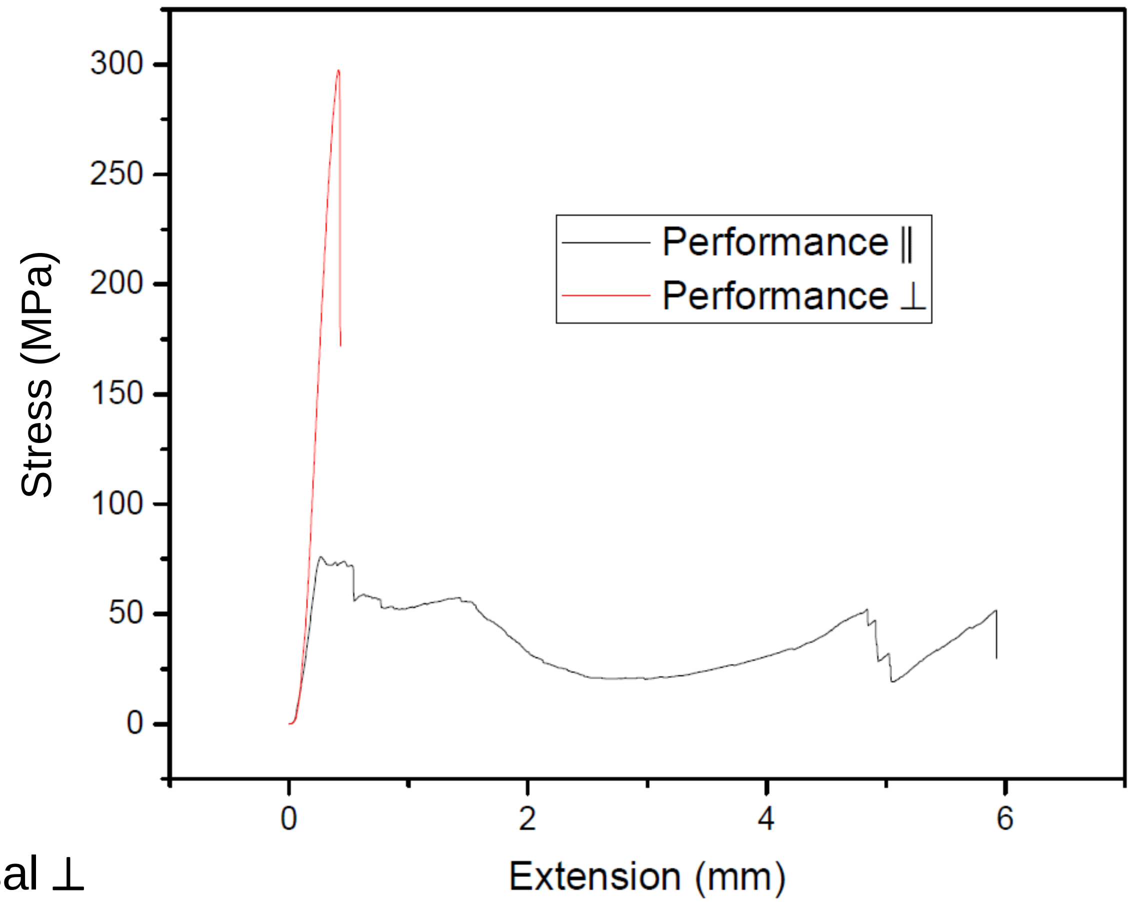
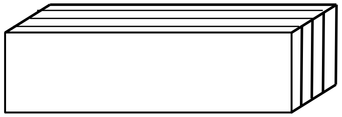
3-point bending test



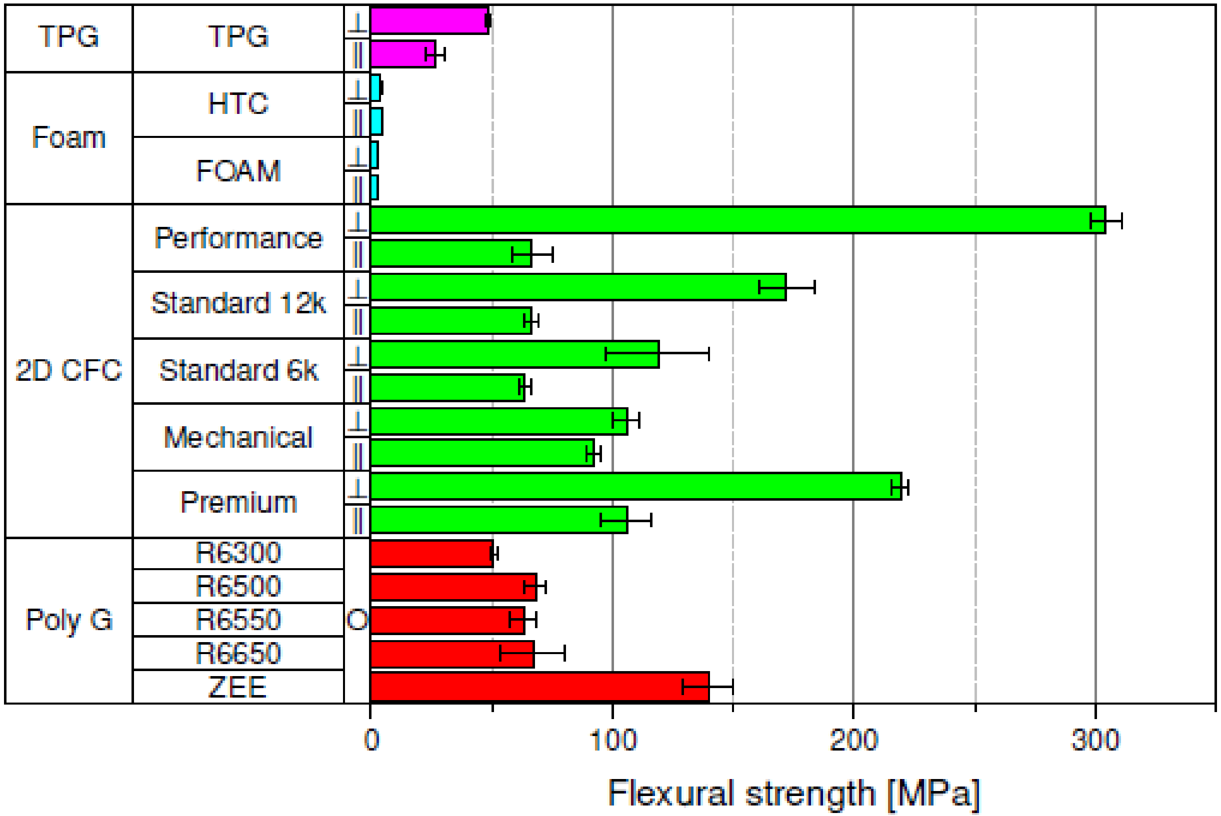


In plane ∥

Transversal ⊥

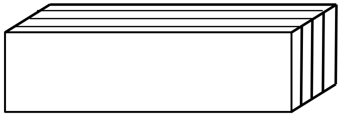


Flexural strength

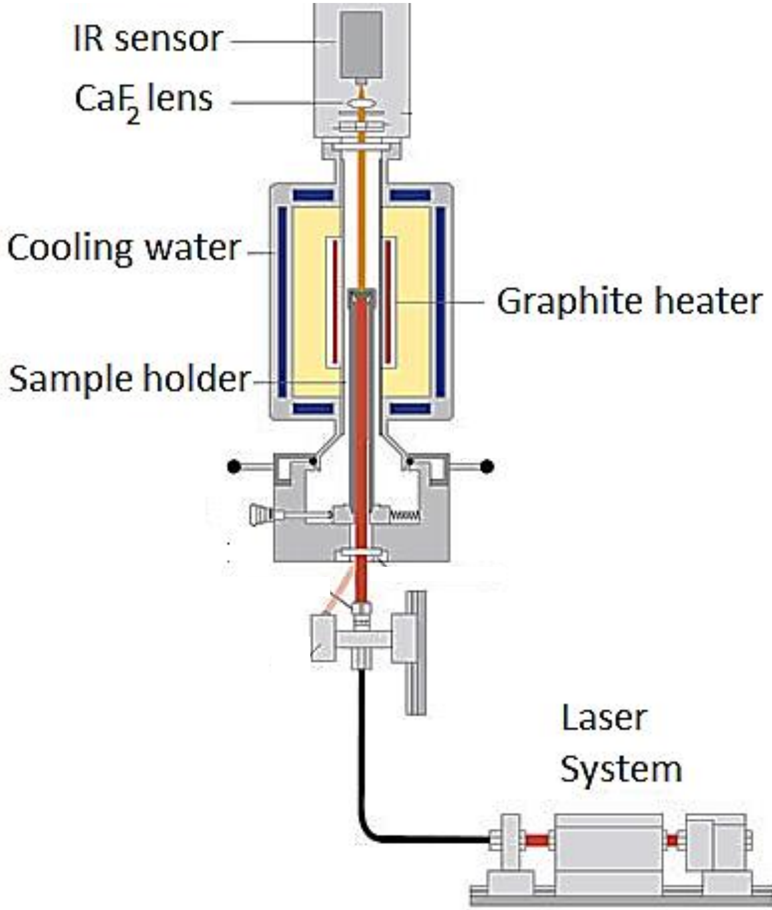


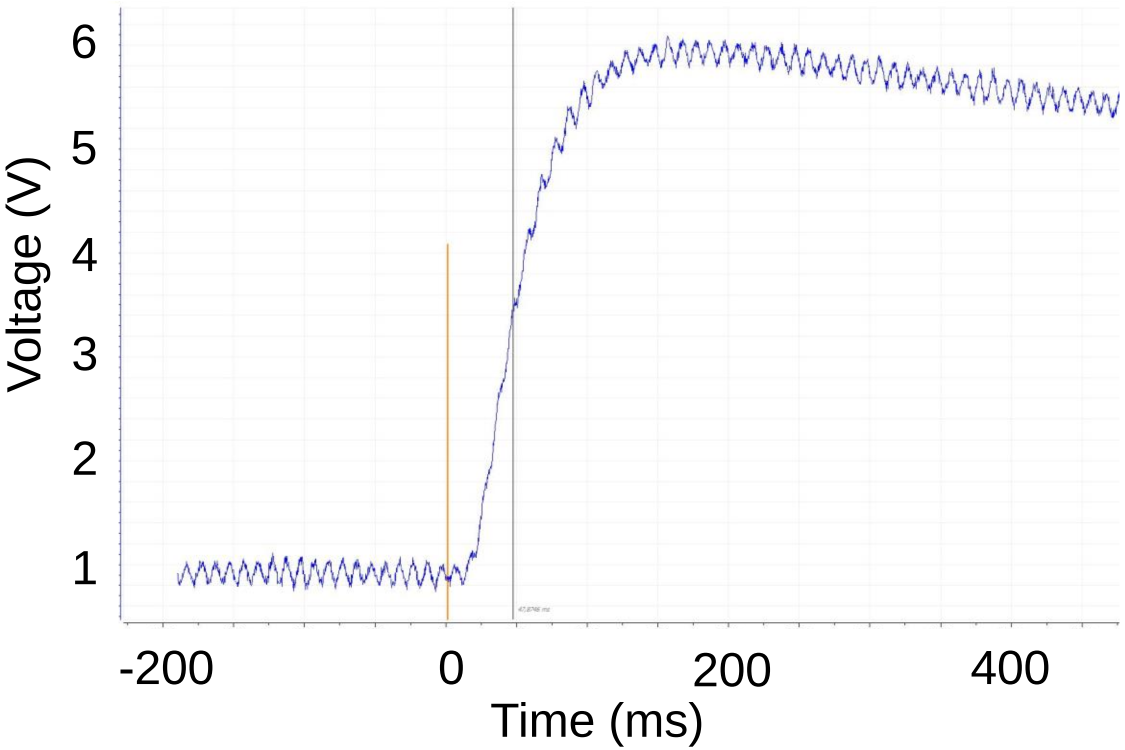
In plane ||

Transversal ⊥



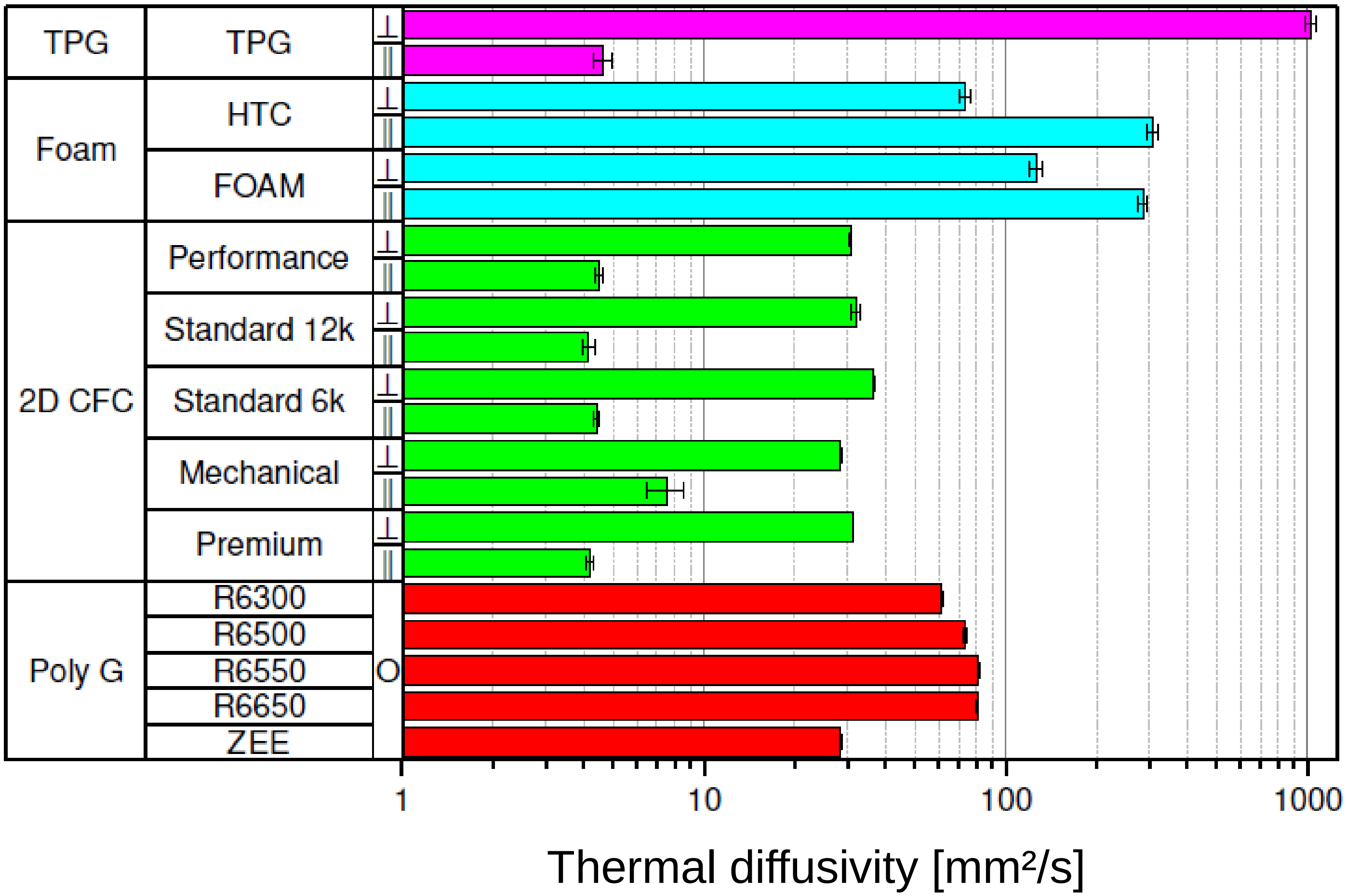
Laser flash analysis



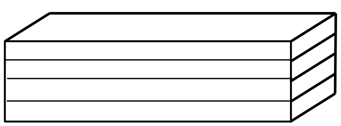


https://www.netzsch-thermal-analysis.com/de/produkte-loesungen/waerme-und-temperatur-leitfaehigkeitsbestimmung/lfa-427/ (23.11.2017)

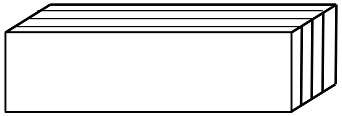
Laser flash analysis results



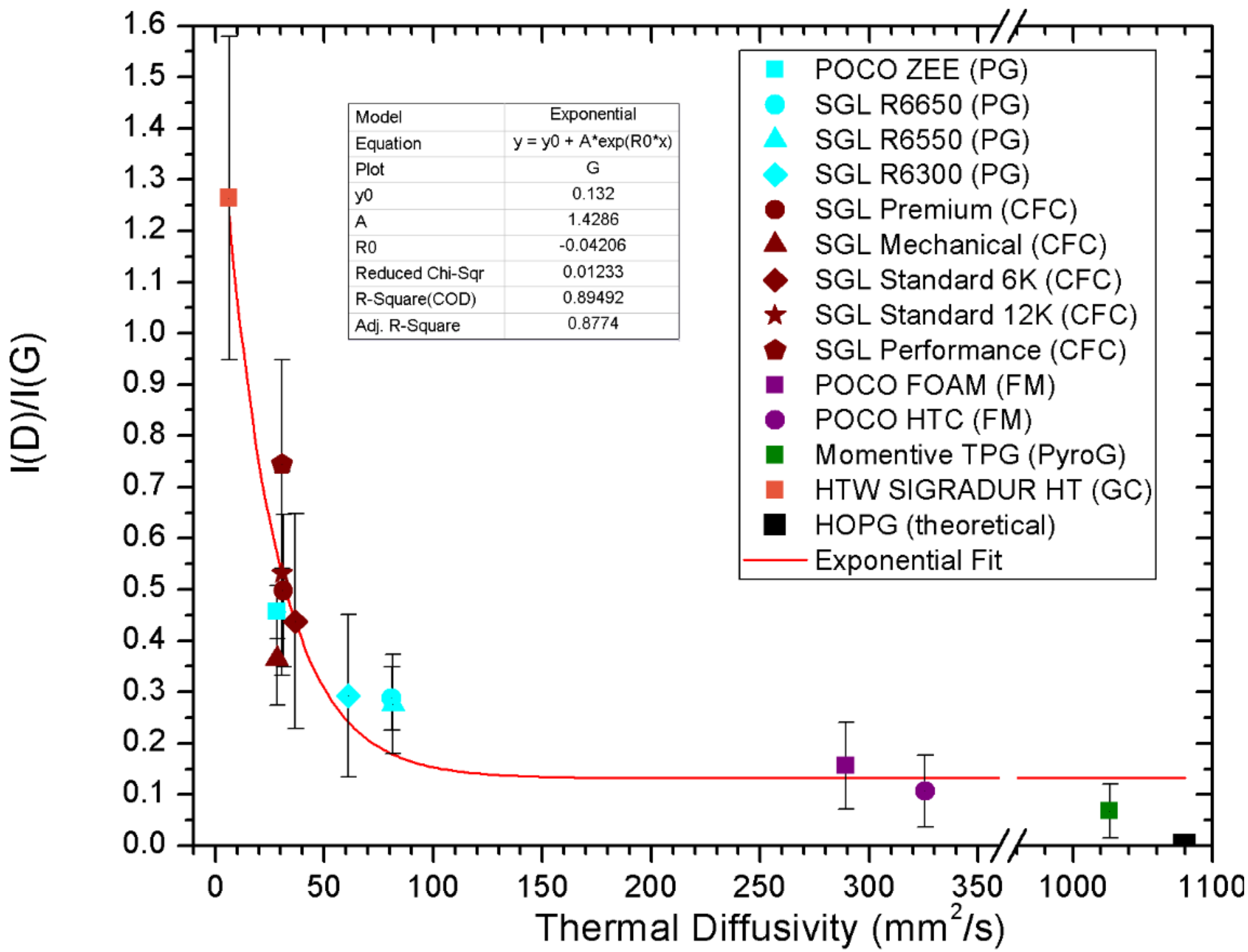
In plane ∥



Transversal ⊥



Empirical relation between I_D/I_G and thermal diffusivity



- HOPG was used as reference (Ideal case has I_D zero)
- The relation shows an exponential behavior
- Lower I_D/I_G implies larger thermal diffusivity

Conclusion and outlook

- Properties of polycrystalline graphite depend on the grain size
- Carbon fibre reinforced carbon and graphite foams properties depend on orientation with a strong and a weak direction
- The thermal diffusivity is decaying exponentially with increasing defect concentration
- Results allow calculation of figures of merit and improved simulations to predict the behaviour during irradiation
- Experiments at HiRadMat and at GSI will allow evaluation of beam induced effects and property changes of the different irradiated graphitic materials

Acknowledgment

Thank you for your attention

Thanks to Roberto Colina-Ruiz, Fabian Jäger, Daniel Schmitt, Pascal Simon and Marilena Tomut for providing data

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