



# Report from recent material characterization campaigns and new developments

ARIES WP17 2<sup>nd</sup> Annual Meeting

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<https://indico.cern.ch/event/938208/>

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With contributions from Mechanical Measurements Laboratory, Mechanical & Materials Engineering group at CERN

# Outline

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- CuCD tests
  - Thermal conductivity isotropy
  - High-temperature bending tests
- Updates on graphite-matrix composites
  - Description and results
  - Comparison with MoGr and pure carbon materials
- Conclusions
- Next steps

# CuCD composites (Copper-diamond)

# CuCD HRMT36 grade

## Composition:

- **50vol%** bi-modal **diamond** mixture with mesh size 70/80 and 120/140.
- Matrix: **copper alloy**.
- Theoretical density= $6.21 \text{ g/cm}^3$  (CD= $3.514$ , Cu-alloy= $8.9 \text{ g/cm}^3$ )

Several batches produced for different purposes.

## Measured density:

- 3025 (weight+dimensions):  **$\sim 5.7 \text{ g/cm}^3$**
- 3434 (weight+dimensions):  **$5.7 \text{ g/cm}^3$**
- 3434 (Archimedes'):  $6.06 \text{ g/cm}^3$  \*
- HT-bending (Archimedes):  $6.10 \text{ g/cm}^3$  \*
- HT-bending (weight+dimensions):  $5.1 \text{ g/cm}^3$  \*\*

**$5.7 \text{ g/cm}^3$  is used for the material model**

→ Estimated porosity content is  $\sim 8 \text{ vol}\%$



Plate for thermo-mechanical characterization (Mid-2018)



ID: RHP3025 (July 2017), tested in HRMT36 (Oct-2017)

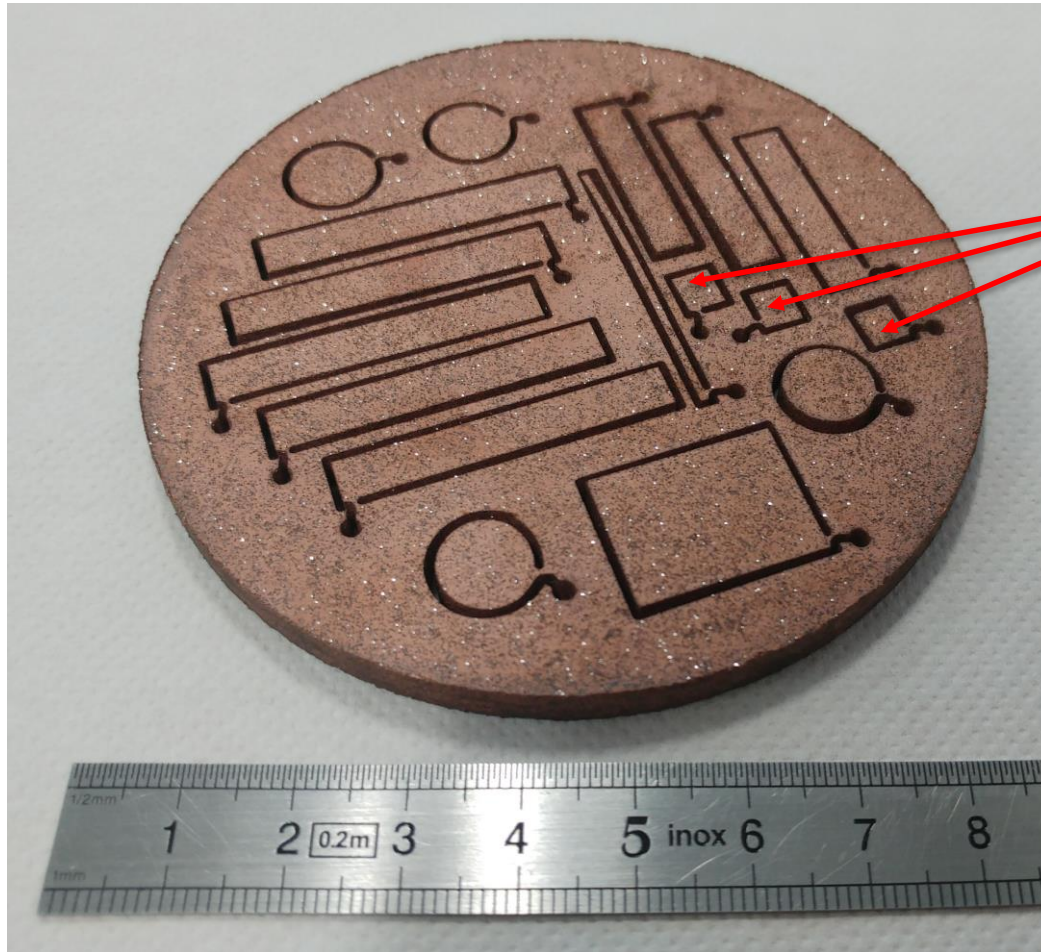
\* Large error due to liquid infiltration in open porosity

\*\* Large error due to small sample dimensions



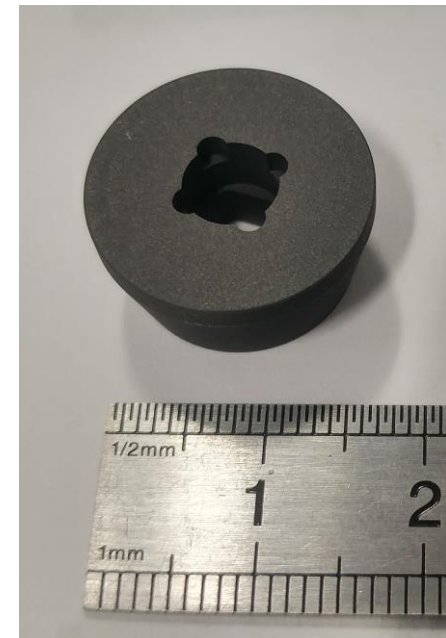
Specimens for high-temperature bending tests (February-2020)

# CuCD HRMT36 grade: isotropy



3 samples (4.5x5x5 mm) for  
in-plane Laser Flash Analysis tests

Special graphite sample holder  
manufactured for these samples

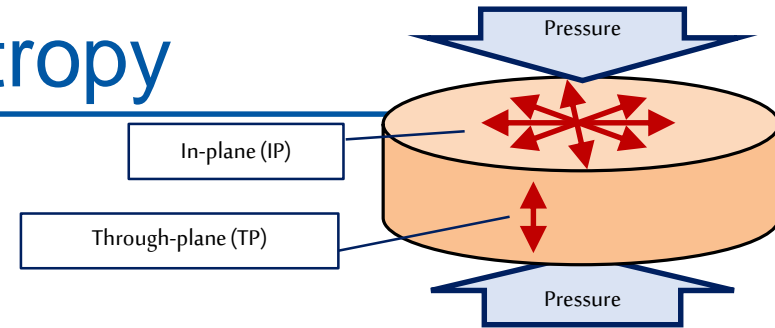


# CuCD HRMT36 grade: isotropy

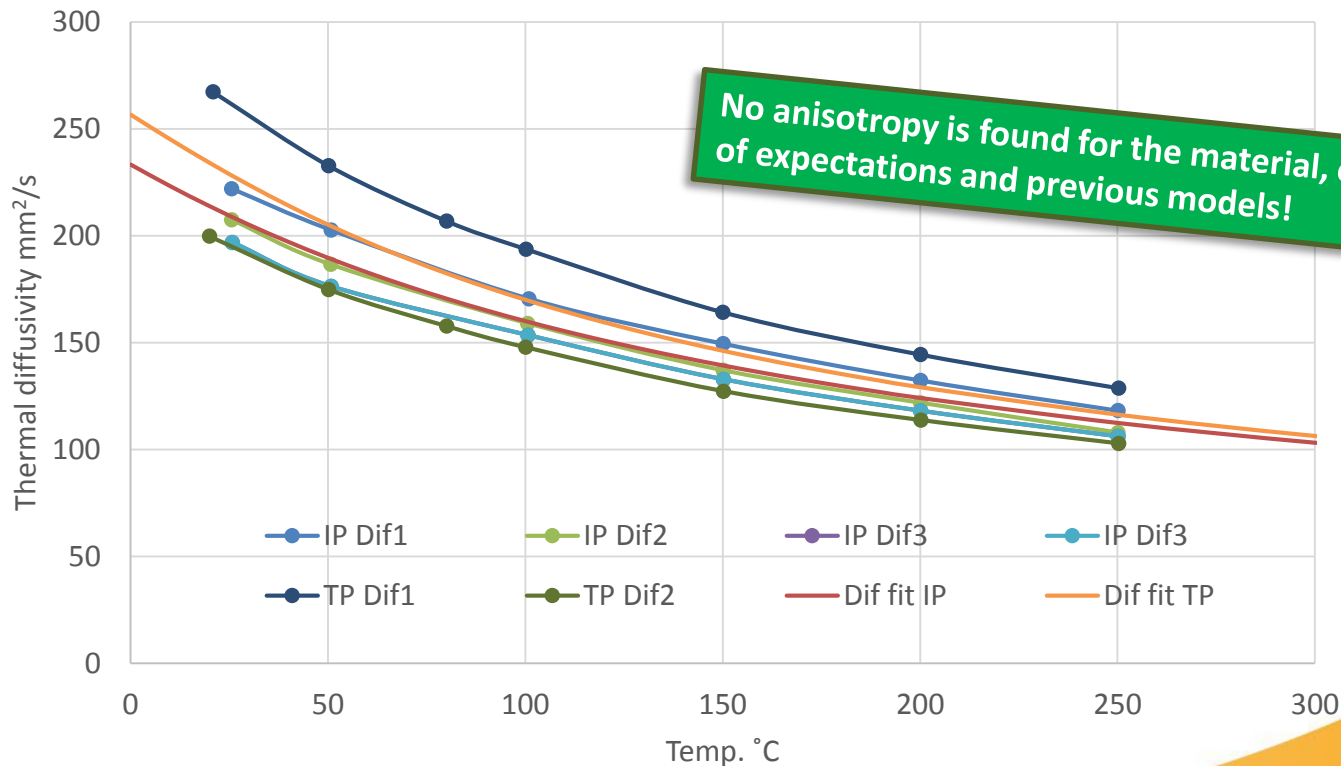
Average (fit) thermal diffusivity results at 20 °C:

- In-plane: **214** mm<sup>2</sup>/s
- Through-plane: **234** mm<sup>2</sup>/s

These values are comparable within the test method uncertainty (precise thickness measurement is challenging for this material).

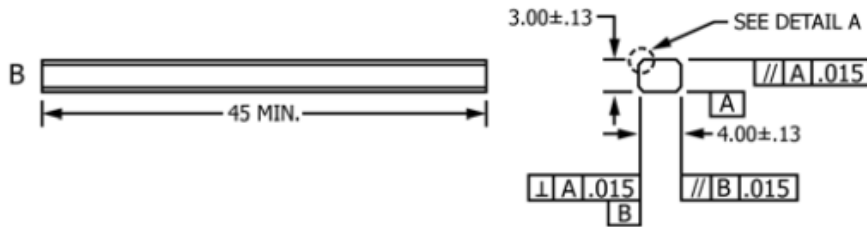


Comparison IP – TP CuCD HRMT36

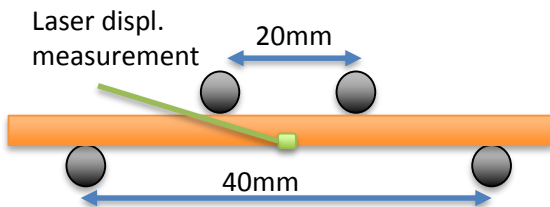
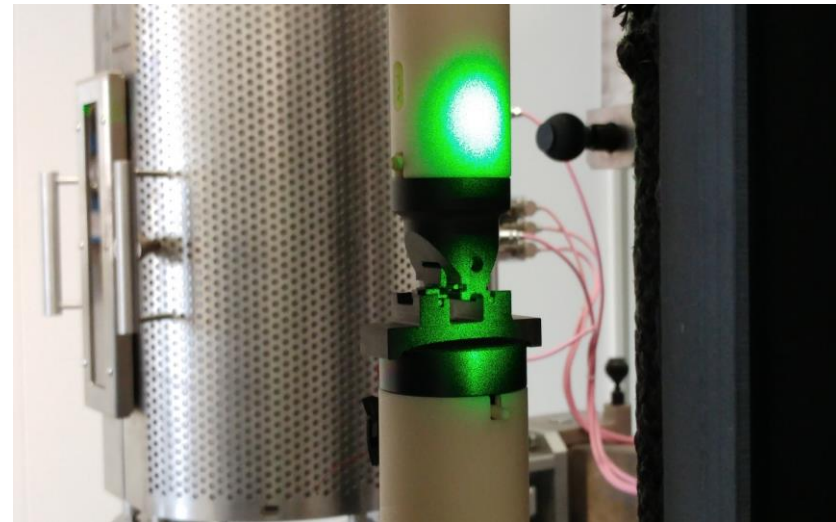


# CuCD HRMT36 grade: HT-bending tests

- **Bending tests at high temperature on the CuCD Multimat grade: requiring 20 samples with indicative dimensions:**



- **25 rods** prepared by RHP in February 2020 and shipped to CERN
- Most of the samples already tested at the CERN MME laboratory!
- Tests performed with silicon-carbide 4-pointing fixture up to 300 °C
- Laser extensometer measuring displacement at the specimen centre (side under traction) → strain
- Challenging strain measurement due to small optical aperture in the furnace

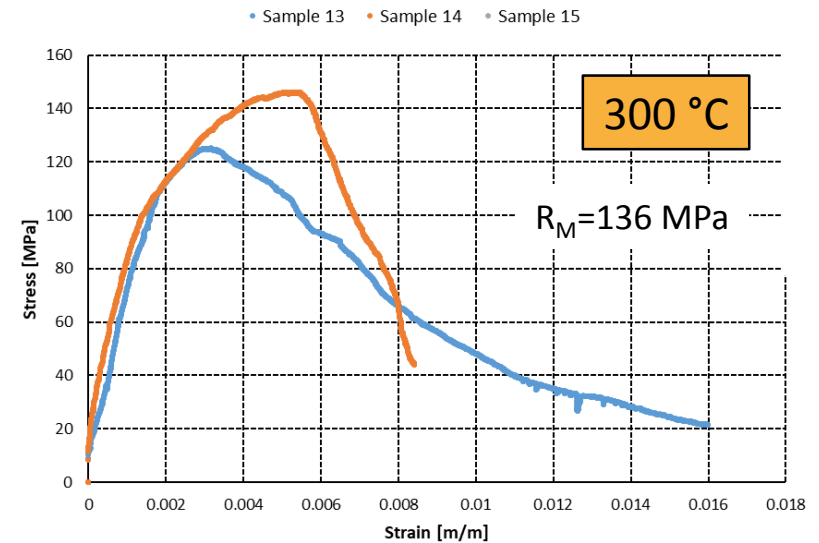
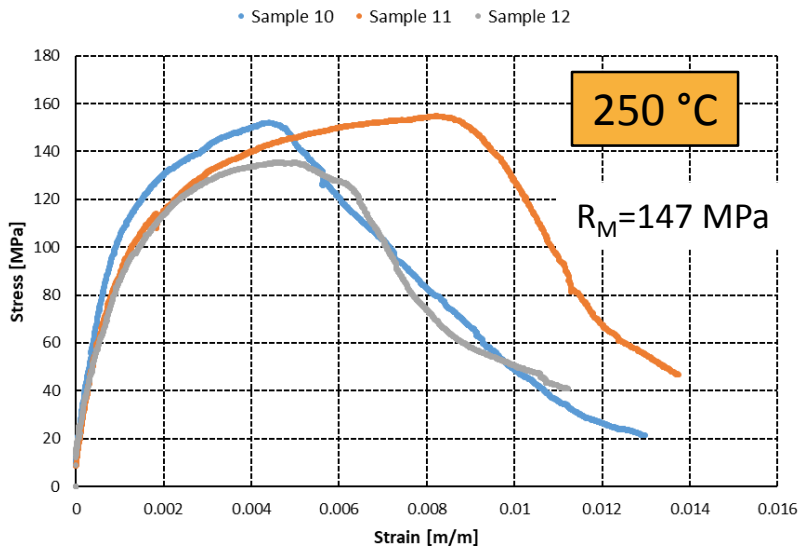
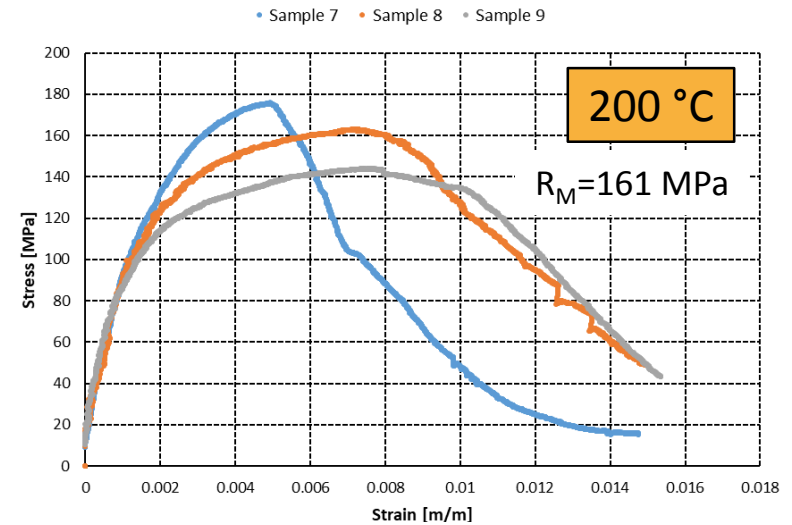
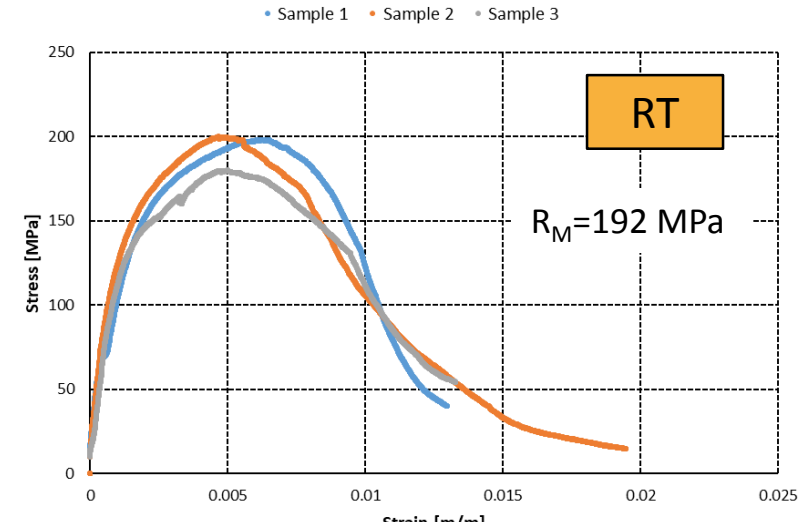


$$\varepsilon = \frac{12h}{3L^2 - 4A^2} x$$

$\varepsilon$  = strain  
 $x$  = displacement  
 $h$  = thickness = 3 mm  
 $L$  = outer span = 40 mm  
 $A$  = (outer-inner span)/2 = 10 mm

# CuCD HRMT36 grade: HT-bending tests

Preliminary results. Analysis & implementation in the material model ongoing.

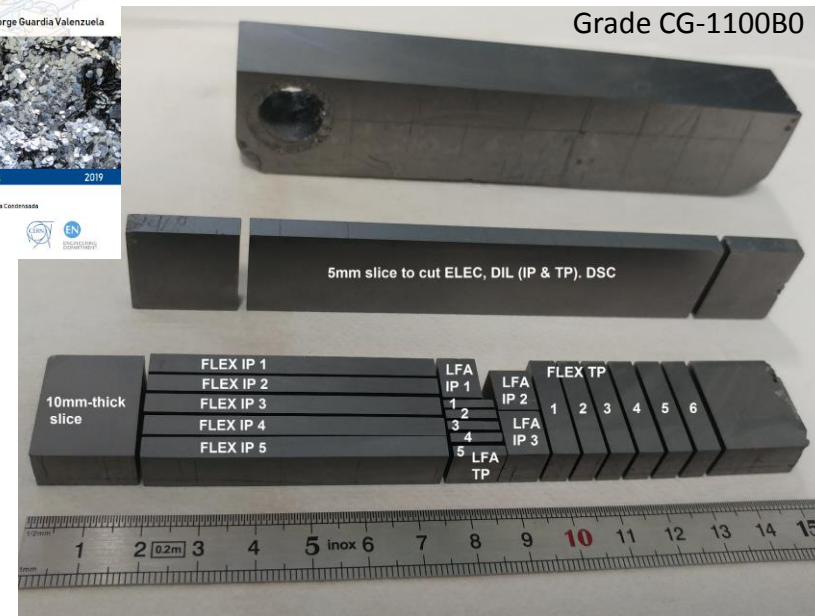
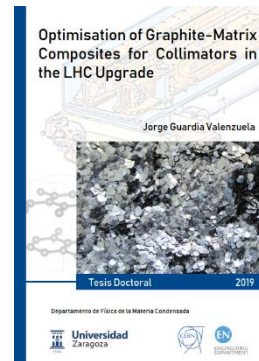
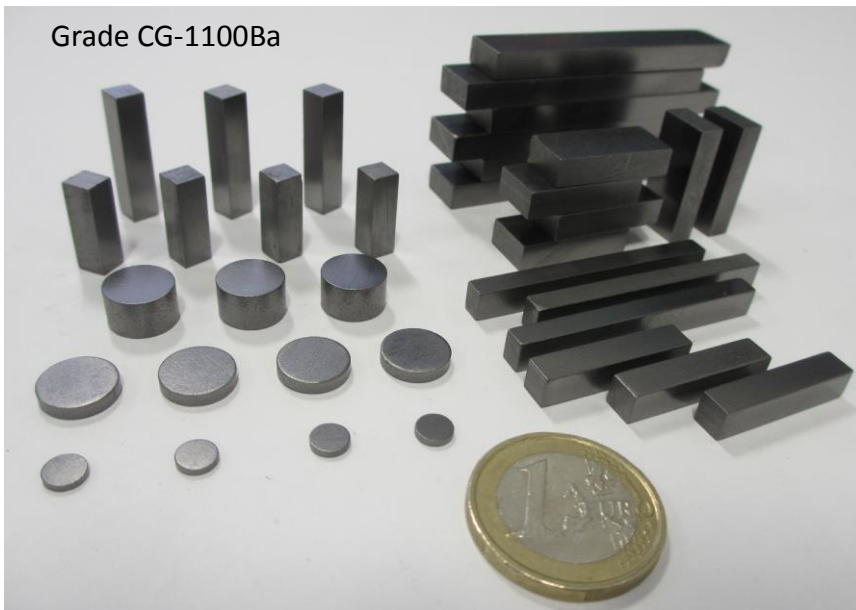




# CrGr composites (graphite-matrix)

# CrGr composite materials

- Based on the knowledge learnt from MoGr [1], new composite materials have been developed at CERN.
- The main goal is to reduce production complexity with respect to MoGr.
- Several grades of chromium-graphite have been produced
- PhD “Optimisation of graphite-matrix composites for collimators in the LHC Upgrade” (J.Guardia, 2019). Thesis not yet public for IP protection reasons.



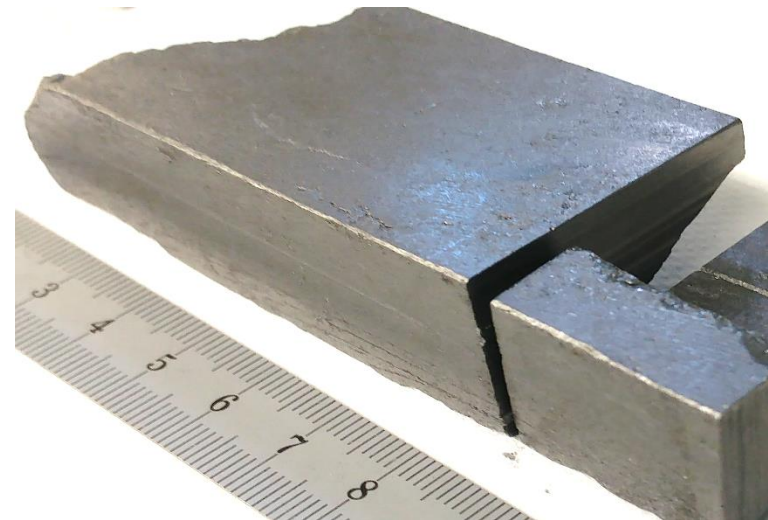
Produced by: **BREVETTI BIZZ**

[1] Development and properties of high thermal conductivity molybdenum carbide - graphite composites. J.Guardia, A.Bertarelli, F.Carra et al. Carbon 2018, Vol 135. <https://doi.org/10.1016/j.carbon.2018.04.010>

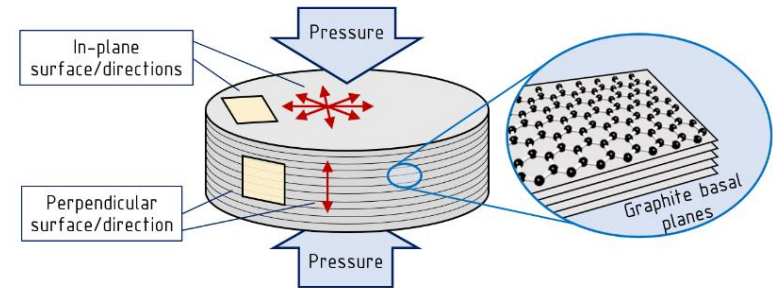


# Grade CG-1240X

- **Best in-plane electrical and thermal conductivities** amongst all CrGr grades.
- **Similar through-plane conductivity** to the other grades.
- Production failure due to the mould breakage → another trial is foreseen.



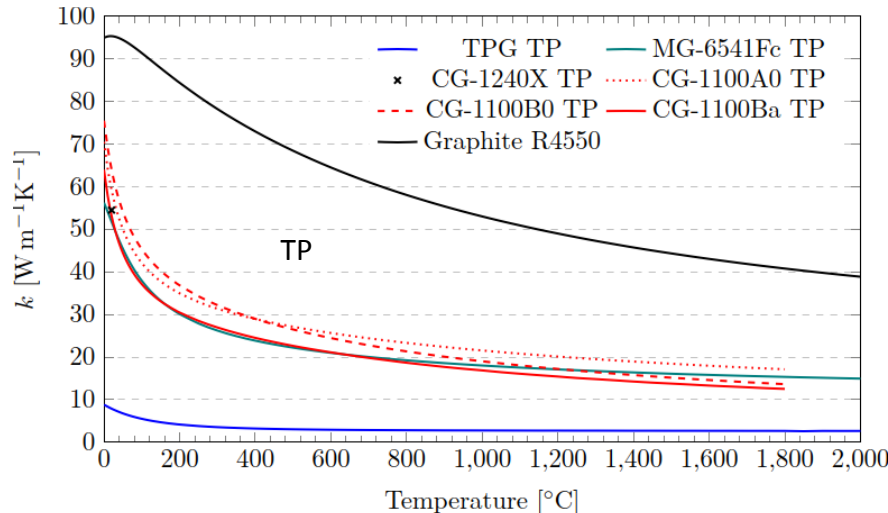
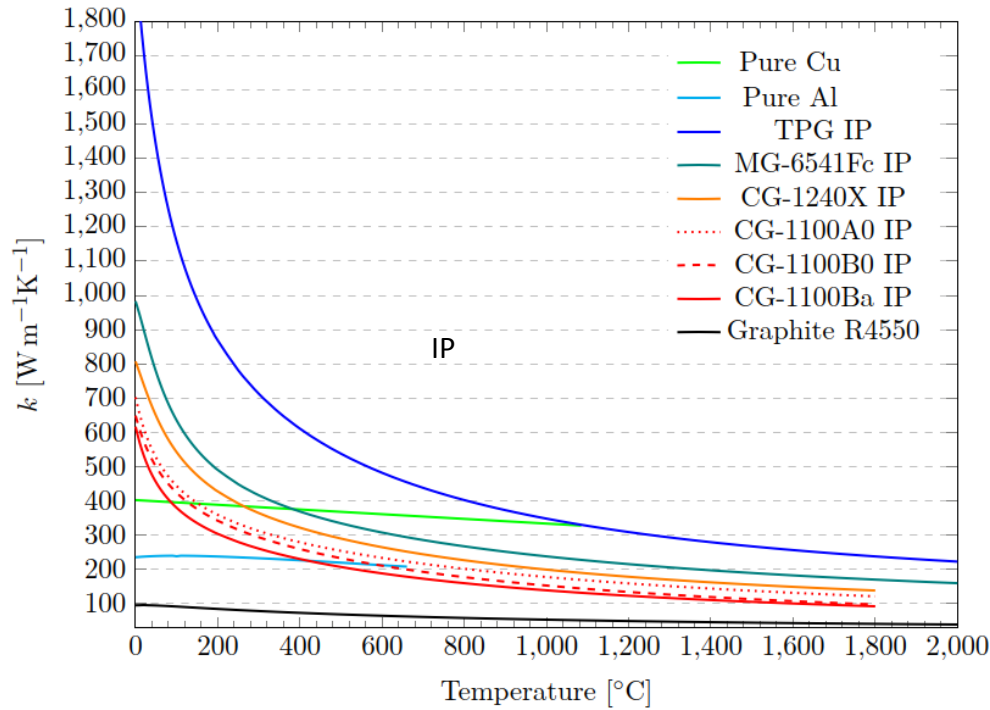
# Comparison table



Direction	MoGr		CG-1100A0		CG-1100B0		CG-1100Ba		CG-1240X	
	IP	TP	IP	TP	IP	TP	IP	TP	IP	TP
Density ( $\text{g}\times\text{cm}^{-3}$ )	2.5-2.6		2.31		2.21		2.15		2.26	
Electrical conductivity ( $\text{MS}\times\text{m}^{-1}$ )	0.9-1.1	0.05-0.07	0.84	-	1.00	0.08	1.01	0.08	1.06	-
Specific heat at 20°C ( $\text{J}\times\text{g}^{-1}\times\text{K}^{-1}$ )	0.6-0.65		0.677		0.687		0.670		0.714	
Thermal diff. at 20°C ( $\text{mm}^2\times\text{s}^{-1}$ )	430-530	28-37	391	38	378	42	370	37	458	34
Thermal cond. at 20°C ( $\text{Wm}^{-1}\text{K}^{-1}$ )	650-900	45-65	612	59	574	64	532	53	739	55
CTE 20-200°C ( $\times 10^{-6} \text{K}^{-1}$ )	1.7-2.7	8-12	4.2	8.6	2.3	7.9	2.6	8.3	1.8	11.5
Flexural strength (MPa)	60-80	10-12	-	-	31.2	6.6	26.2	5.5	15.0	-
Elastic modulus (GPa)	60-85	4-5	-	-	42.8	3.8	38.7	2.4	~20	-
Flexural strain to rupture (%)	0.18-0.26	0.45-0.72	-	-	0.16	0.42	0.12	0.42	0.24	-

Thanks to the colleagues in the Mechanical Measurements Laboratory at CERN for the comprehensive characterization campaign!

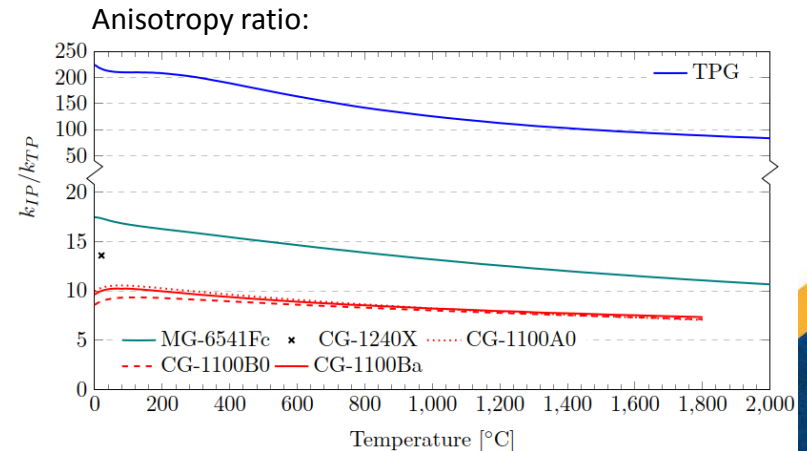
# Thermal conductivity comparison



CrGr materials are less anisotropic than MoGr or PG

k of CG-1240X is just slightly lower than best MoGr produced so far

Note that the composites have higher k than copper in its usable range → thermal management



# Reference graphitic materials

Purely oriented graphite:  
very weak TP properties

Direction <sup>1</sup>	Gr. R4550	PG (TPG)		CFC (AC150K), orthotropic		
	Isotropic		⊥	Z	Y	X
$\rho$ ( $g\ cm^{-3}$ )	1.83	2.26		1.89		
$c_p$ ( $J\ g^{-1}\ K^{-1}$ )	0.715	0.69		0.712		
$\gamma_e$ ( $MS\ m^{-1}$ )	0.08	2.5*	0.0009	0.24	0.18	0.03
$a$ ( $mm^2\ s^{-1}$ )	73	1095	5	227	174	40
$k$ ( $W\ m^{-1}\ K^{-1}$ )	95	1710	8	304	233	54
$\alpha$ RT-200°C <sup>2</sup>	4.2	-0.9	29.3 <sup>3</sup>	-0.4	-0.2	10.7
$\alpha$ RT-1000°C <sup>2</sup>	5	~0.4	~29.5 <sup>3</sup>	0.5	0.5	11.4
RD (%)	-0.01	-0.02	0.01 <sup>3</sup>	0.001	0.002	0.07
$R_M$ (MPa)	61.2±3	25.5±0.4	-	139.6±.1	104.2±3	10.3±.3
$\epsilon_{adm}$	0.72±0.04	0.08±0	-	0.14±0.01	0.20±0.04	0.43±0.03
$E$ (GPa)	11.5	~65	-	110.2	75.4	3.1
$G$ (GPa)	4.6	-	-	21.5	4.3	3.3

<sup>1</sup> In-plane (||)/through-plane (⊥) or Cartesian directions.

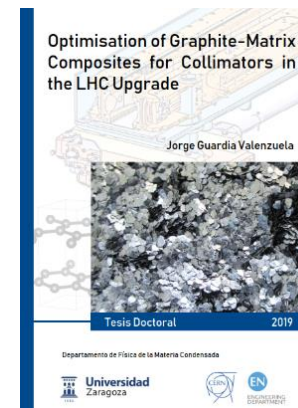
<sup>2</sup> ( $10^{-6}\ K^{-1}$ ).

<sup>3</sup> From specimen TP4, measured only up to 600 °C.  $\alpha$  RT-1000°C is extrapolated.

\* By eddy-current method on the surface, otherwise by four-wire method (average).

# Summary

- CuCD thermal conductivity isotropy tests completed: no relevant anisotropy found.
- CuCD high-temperature bending tests completed. Analysis ongoing.
- New graphite-matrix composite grade produced. Excellent thermal and electrical conductivities found. Mechanical properties to be improved.
- PhD thesis “Optimisation of graphite-matrix composites for collimators in the LHC Upgrade” by J.Guardia was completed in 2019. MoGr and CrGr material families were investigated.



# Next steps

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- Complete CuCD high-temperature bending test campaign to implement the test results in the material model.
- Repeat production of CG-1240X promising grade, which experienced mould breakage during sintering.
- Investigate other additions to reinforce the graphite matrix of CrGr materials.

**On behalf of the materials R&D team, thanks to our industrial partners and to the colleagues in the Mechanical Measurements Laboratory!!**

**BREVETTI BIZZ**

**RHP**  
TECHNOLOGY







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**Thank you for your attention!**

# CuCD HRMT36 grade (density of 3434 batch)



	Measurements						average	
	1	2	3	4	5	6		
Thickness (mm)	4.97	4.96	4.91	4.96	4.93	4.9	4.938	mm
a (mm)	20.1	20.13	20.21				20.147	mm
b (mm)	15.28	15.24	15.21				15.243	mm
Mass (g)	8.5887	8.589	8.5888				8.589	g
volume							1.517	cm <sup>3</sup>
density							5.663	
							<b>5.7</b>	<b>g/cm<sup>3</sup></b>

Comment on the accuracy of this method:

- With an estimated error of 0.1 mm in the geometry, the range of density goes from 5.49 to 5.85 g/cm<sup>3</sup>.
- Because of the microstructure (protruding diamonds) it is expected that the measured volume is overestimated, therefore the real density is likely to be on the high side (higher than the result 5.663 g/cm<sup>3</sup>). The value is therefore rounded to 5.7 g/cm<sup>3</sup>.

Estimated porosity content is around 8 vol%

Marcus' proposal: use a thin Vaseline/paraffin coating and measure the volume with the Archimedes' method, as in <http://dx.doi.org/10.1016/j.jallcom.2009.10.040>

