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Thermophysical and mechanical characterization of advanced graphitic materials

1st Workshop of ARIES WP17 PowerMat, Politecnico di Torino
27/11/2017

Laura Bianchi (CERN)

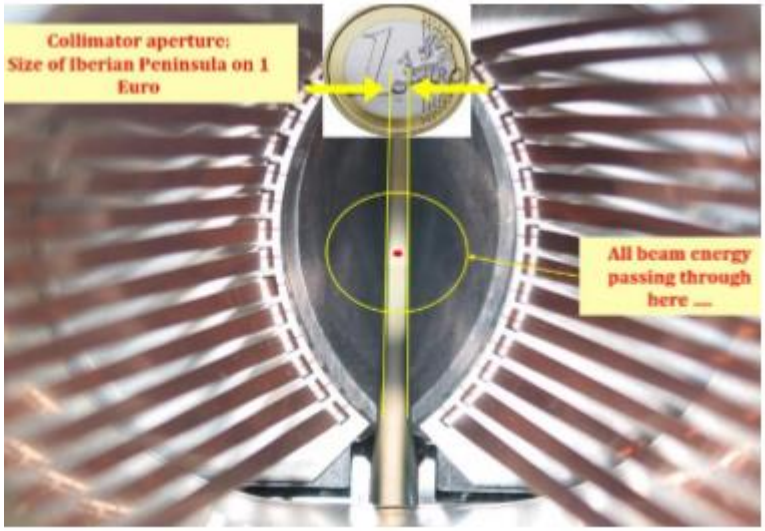
Outline

- Introduction
- Characterization techniques
- Latest characterization campaigns:
  - Highly Oriented Pyrolytic Graphite
  - Metal Carbide Reinforced Ceramics
  - Carbon Fiber – Carbon
- Summary
- Future steps

Introduction

- The collimators' jaws are made of absorber materials which interacts with high energetic particles
- They must withstand a harsh environment…
  - Thermal shocks
  - Ultra High Vacuum
  - Radiation

  while performing the required tasks
  - Cleaning
  - Low RF impedance



↑ **Thermal conductivity λ** ↑ **Electrical conductivity γ** ↓ **Density ρ**

↑ **Specific heat c_p** ↑ **Mechanical strength R_M** ↓ **CTE α** ↓ **Stiffness E**

Thermophysical and mechanical characterization

Thermo-physical characterization

Thermal Expansion & CTE | **Specific Heat cp(T)** | **Thermal Diffusivity a(T)**

PUSHROD DILATOMETER
• ASTM E228

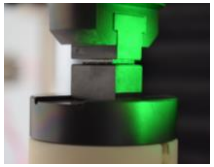
DIFFERENTIAL SCANNING CALORIMETER
• DIN 51007

LASER FLASH APPARATUS
• ASTM E2585

Thermal Conductivity
$\lambda(T) = a(T) \cdot cp(T) \cdot \rho(T)$

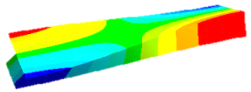
•From RT up to 2000 °C
•Solids, powders and liquids

Mechanical characterization



UNIVERSAL TESTING MACHINE
- Furnace up to **1200 °C**
- Cryostat down to **-200 °C**
- Laser-Extensometer

First Flexural Mode → E



First Torsional Mode → G

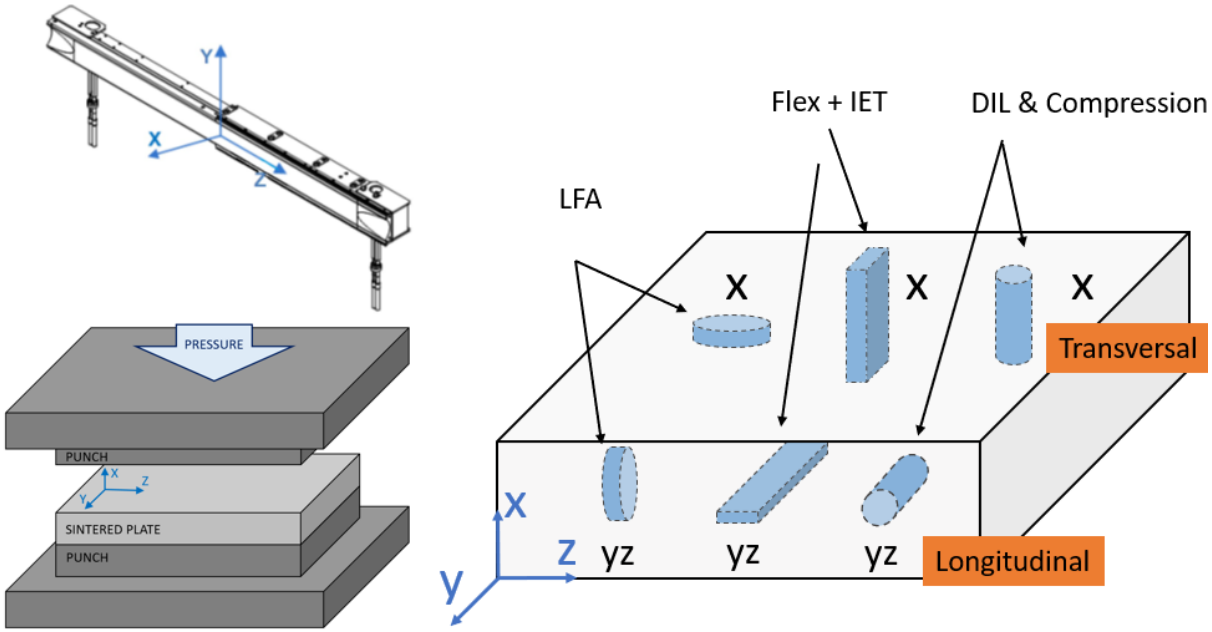
IMPACT EXCITATION TECHNIQUE
• Non-destructive test
• ASTM C1259

•From RT up to 1200 °C

Tensile, compression, bending testing | **Impact testing** | **Mechanical properties** E, G, σ_y

Preamble

- Conventional Reference System



- Thermal diffusivity results marked with * corresponds to a non-negligible underestimate of the measured true value

Highly Oriented Pyrolytic Graphite

- Manufactured by Momentive
- It features:
  - Highly oriented crystals in a layered structure
  - In-plane conductivity 4 times copper

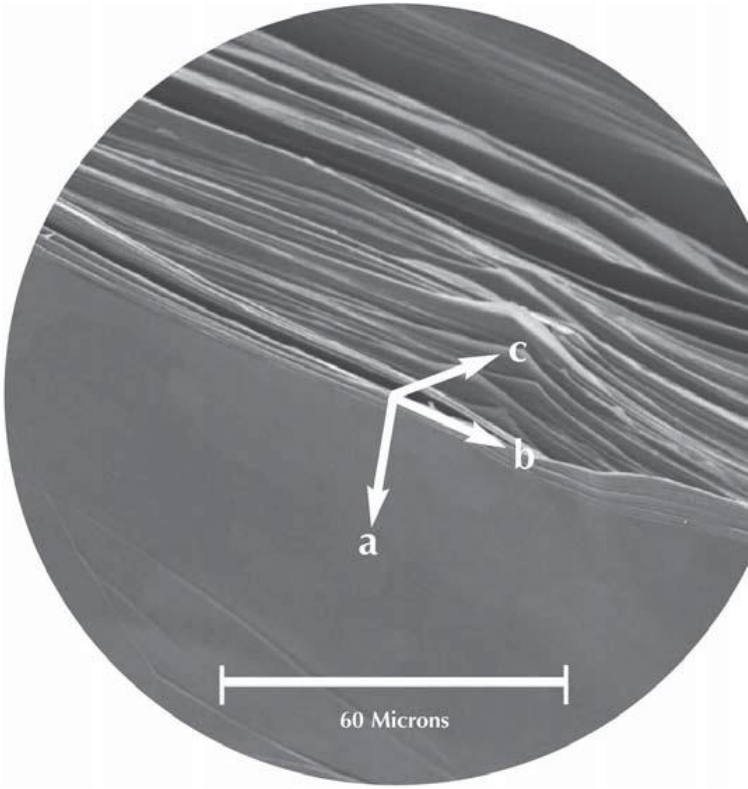
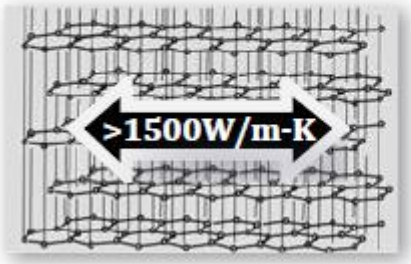
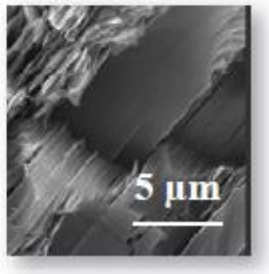
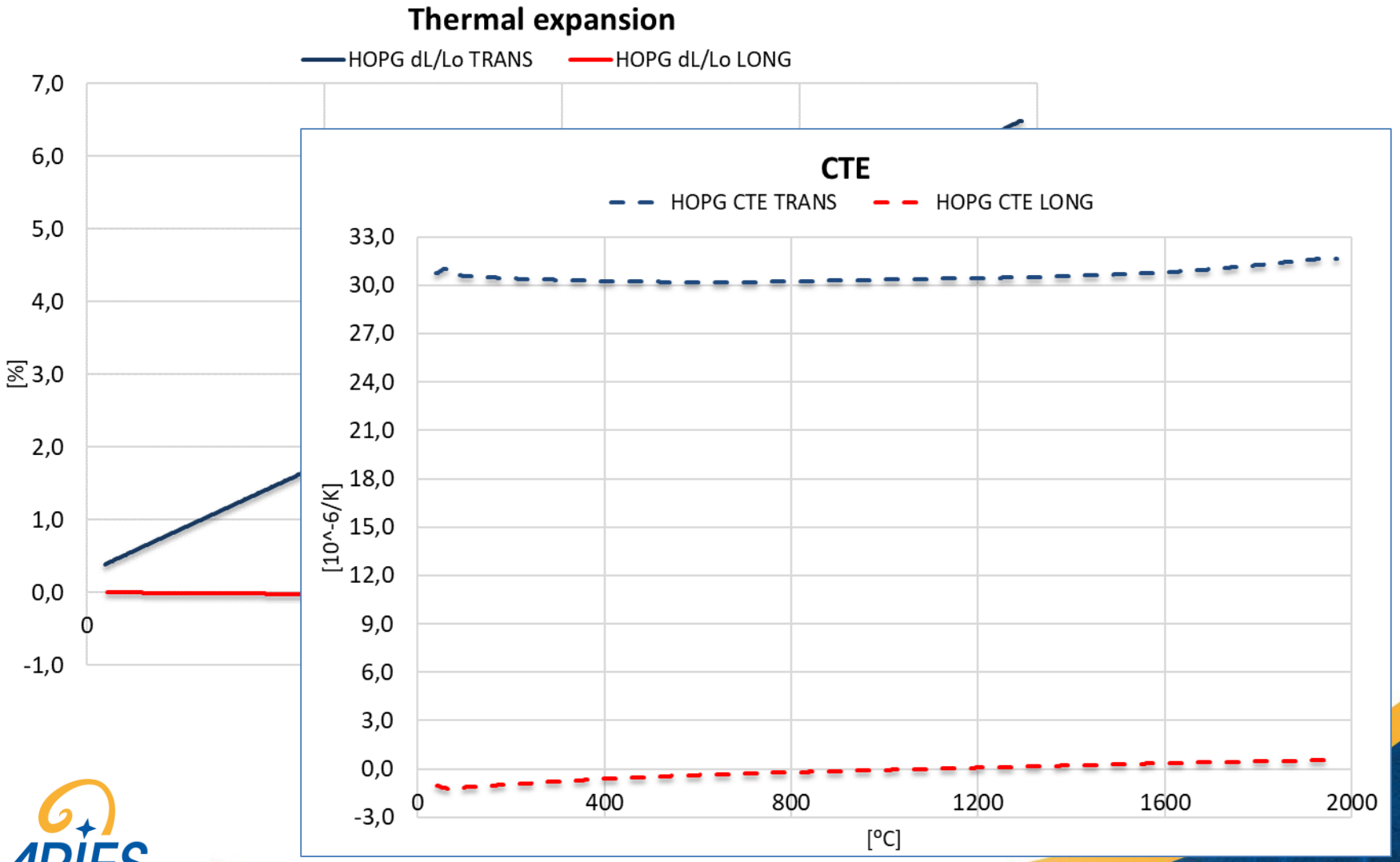


Illustration and Microscopic Image of Highly-Oriented Graphene Layers

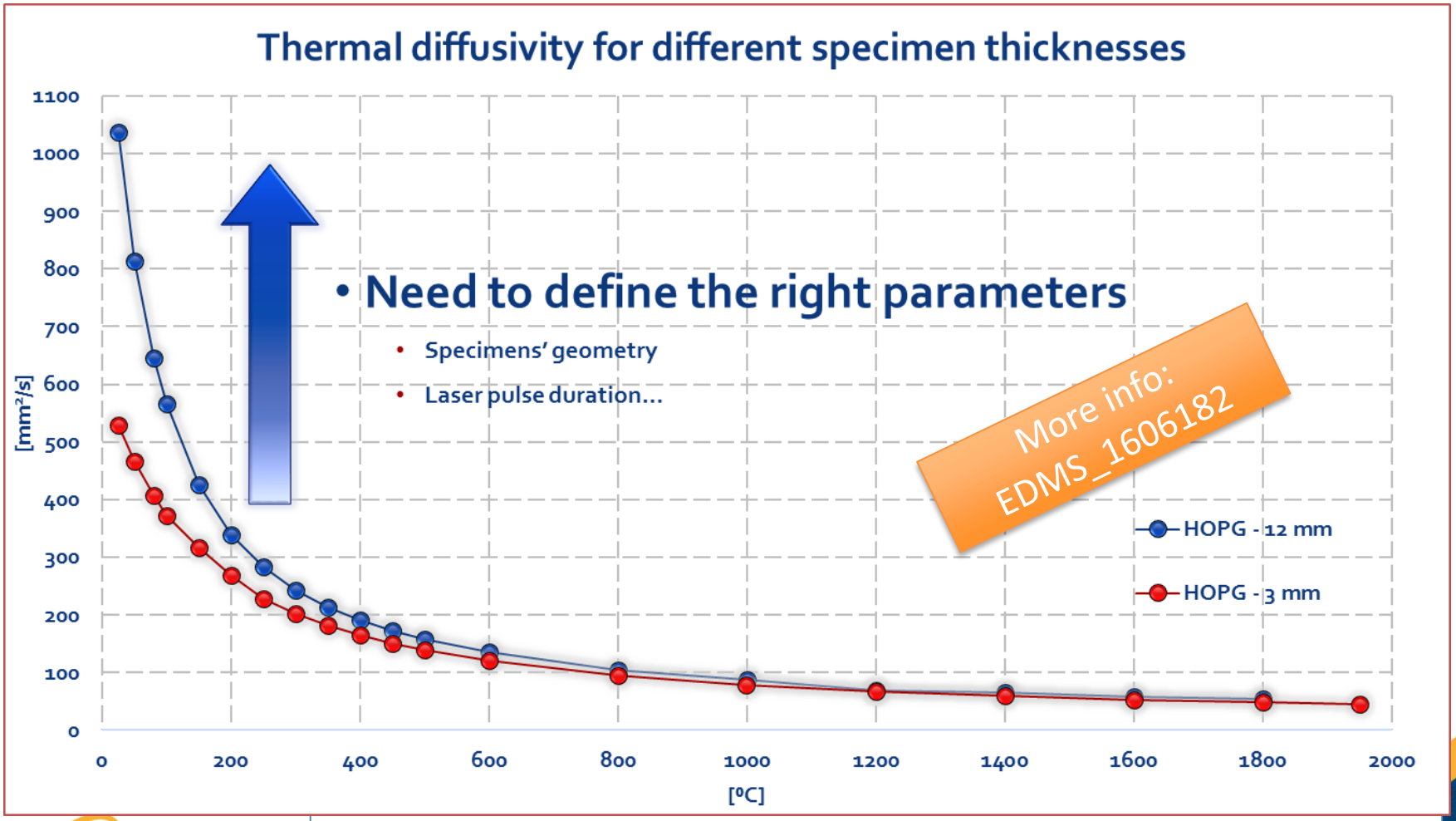




HOPG: Thermal properties 1/2



HOPG: Thermal properties 2/2

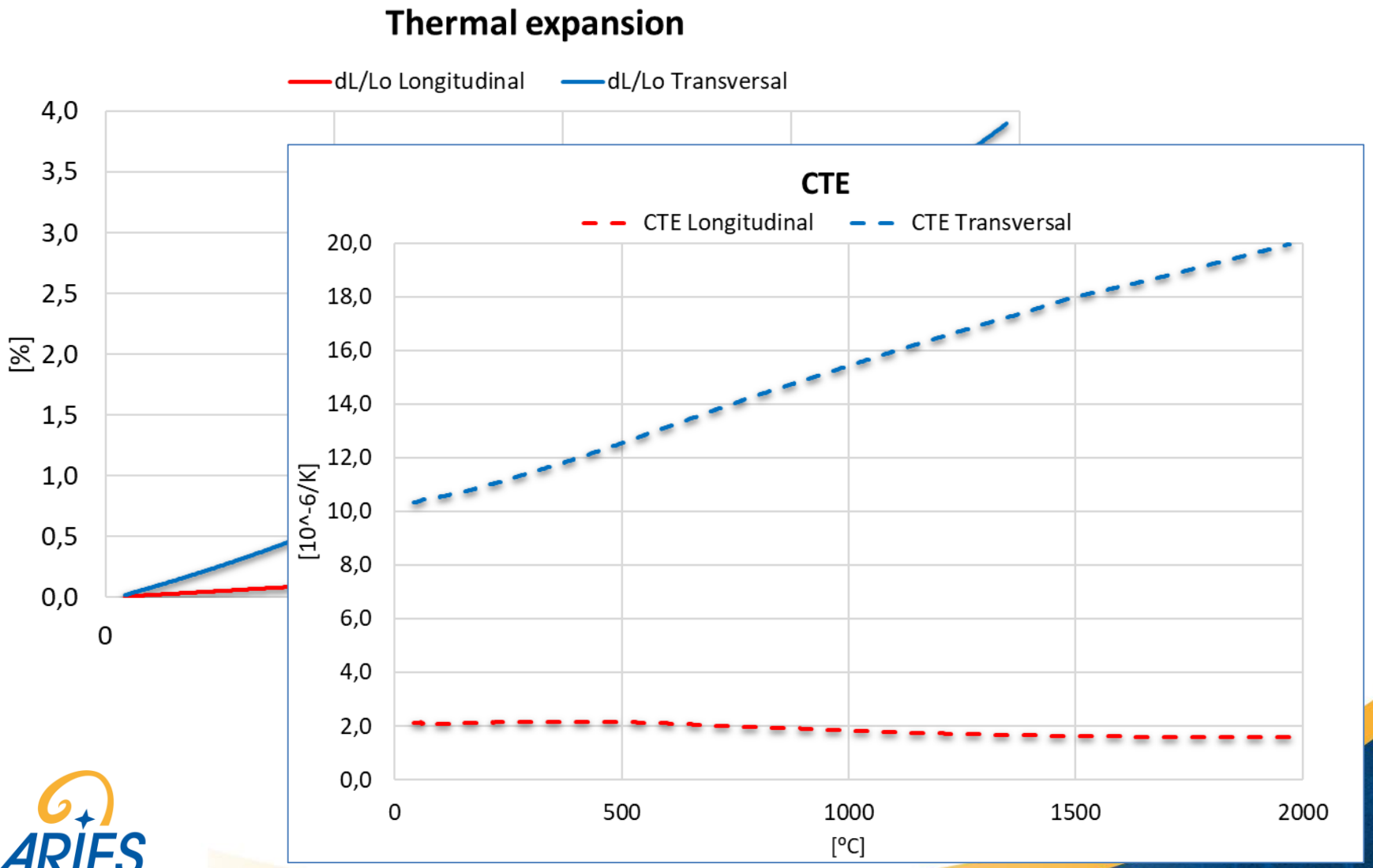


0 500 1000 1500 2000
[°C]

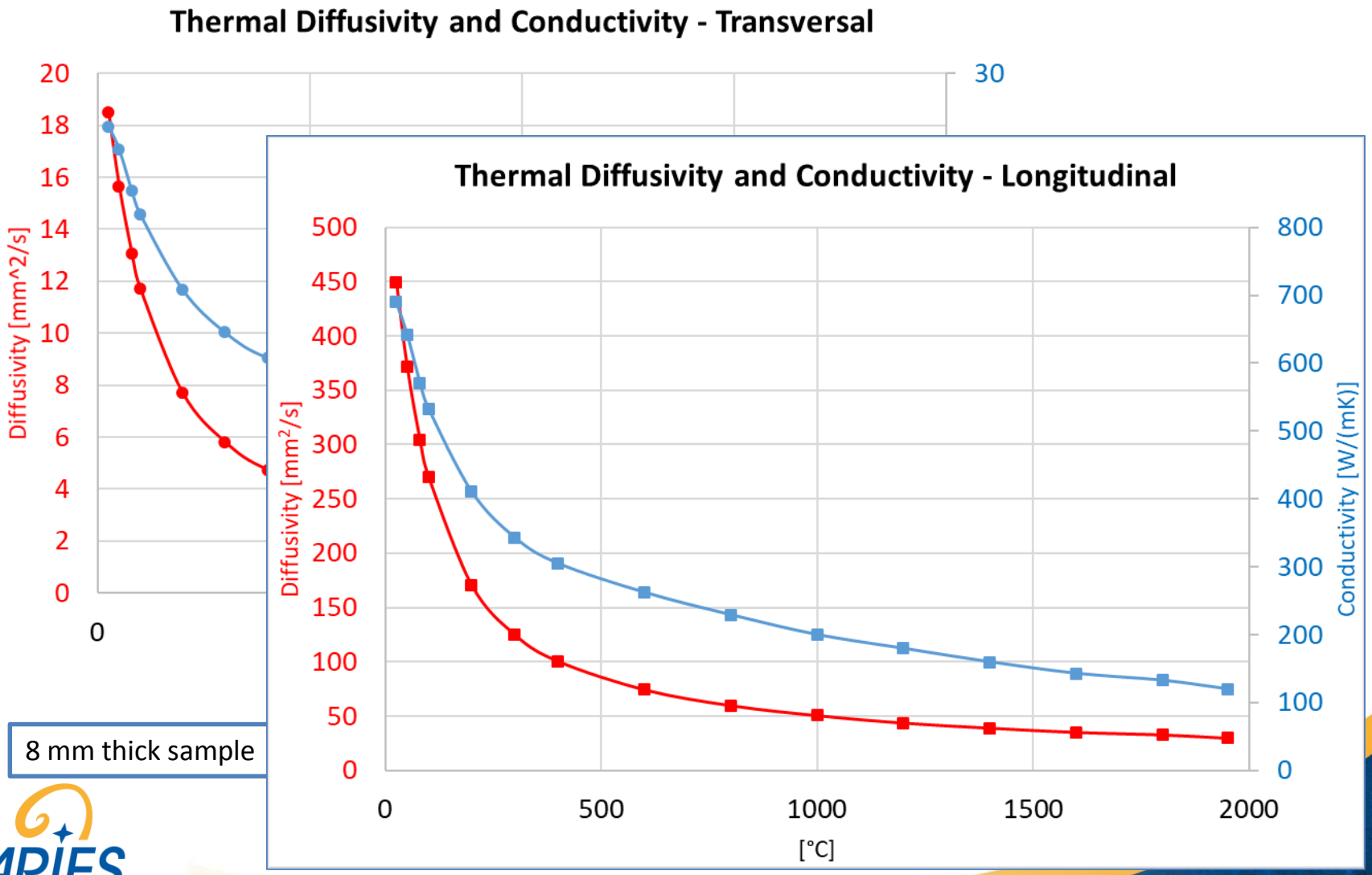
Metal Carbide Reinforced Ceramics

- Nb-8304Je by Nanoker
  - Molybdenum carbide as reinforcement
  - Ti 0.6 %vol
  - $T_{Sintering}$=2660 ºC, $T_{Post\text{-}sintering}$=2450 ºC
- TG-1100 by Brevetti BIZZ
  - Titanium carbide as reinforcement
  - Ti 5.7 %w

Nb-8304Je: Thermal properties 1/2

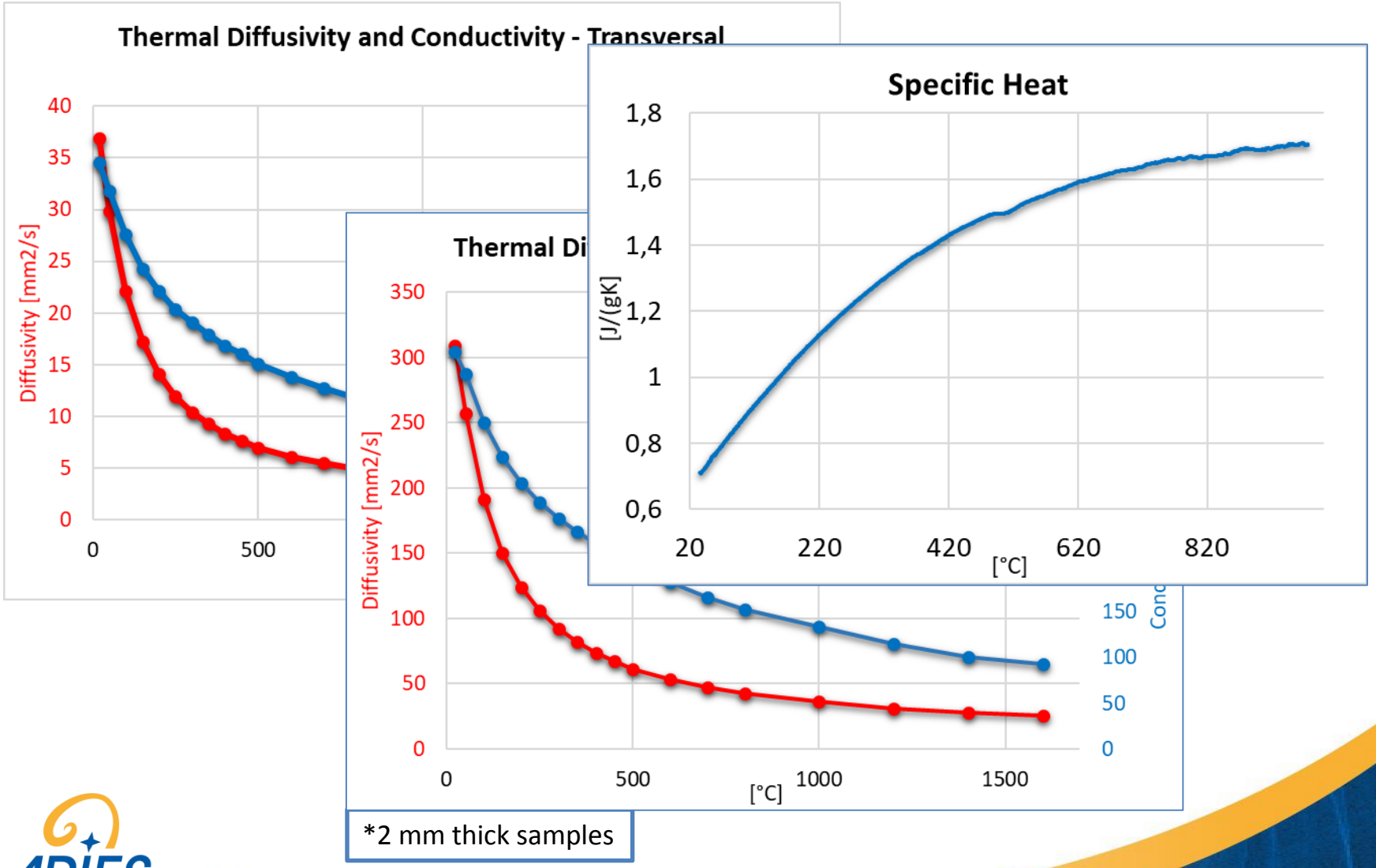


Nb-8304Je: Thermal properties 2/2



8 mm thick sample

TG-1100: Thermal properties

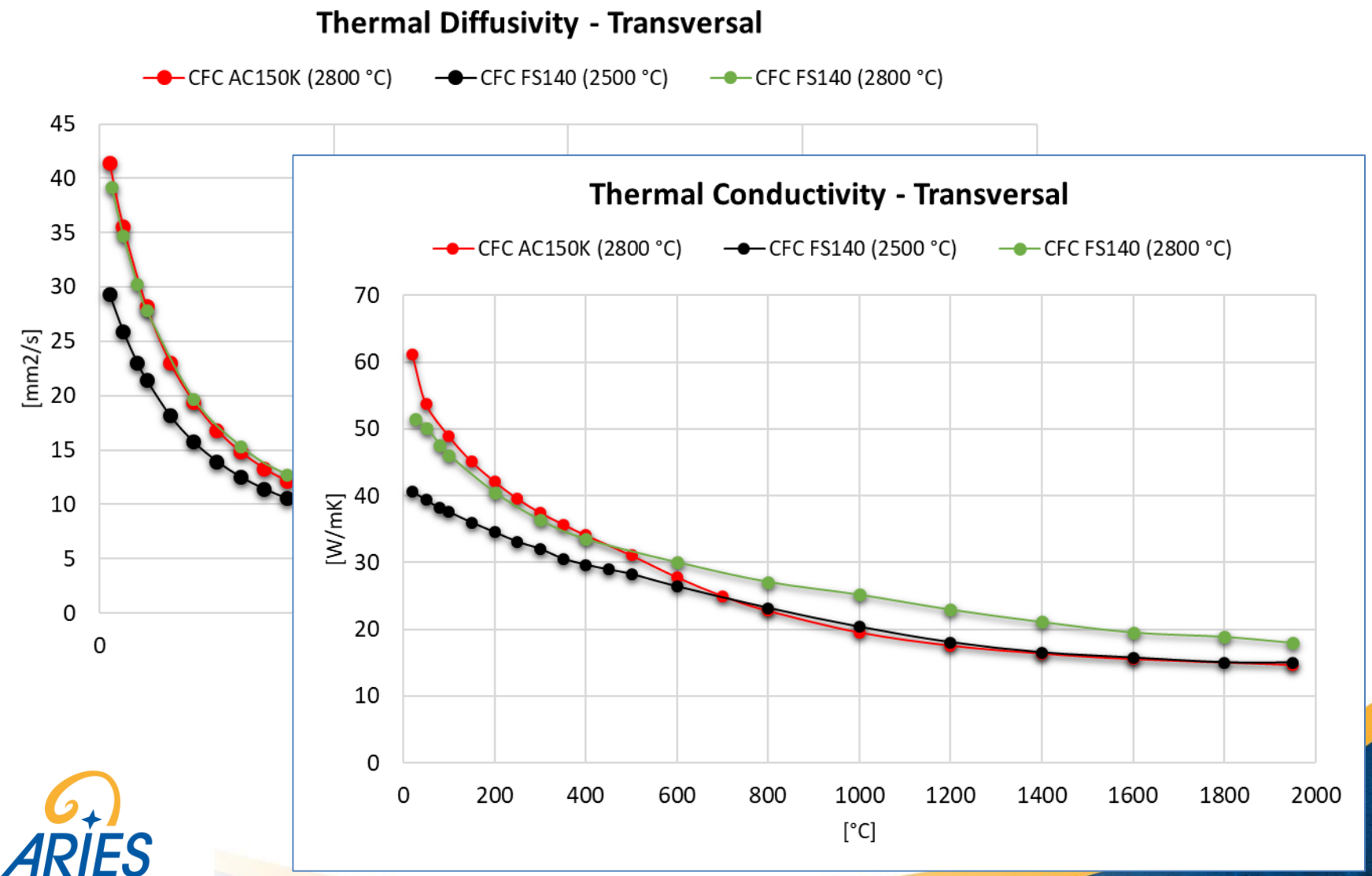


*2 mm thick samples

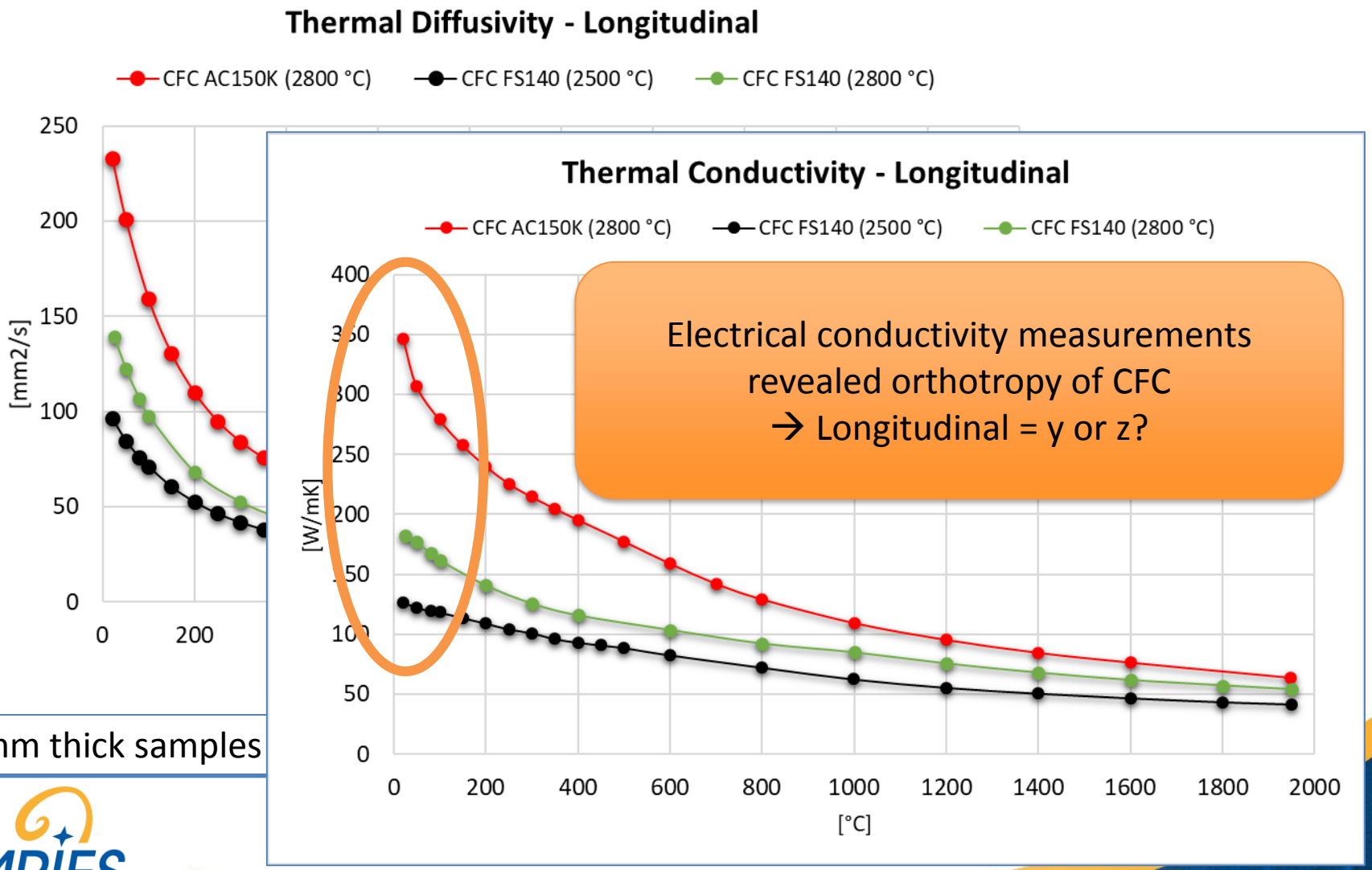
Carbon Fiber-Carbon

- Comparison of three grades whose production process differ in the final baking temperature (which plays an important role in the diffusivity properties).
  - CFC AC150K, currently embarked in the collimators, Tf= 2800 ºC
  - CFC FS140, characterised in 2016, Tf= 2500 ºC
  - CFC FS140, characterisation ongoing, Tf= 2800 ºC
- In between the first and the second two grades, Tatsuno changed the raw material (AC150 Across Co → FS140 CFC Design Co)

Carbon Fiber-Carbon: Thermal properties 1/4

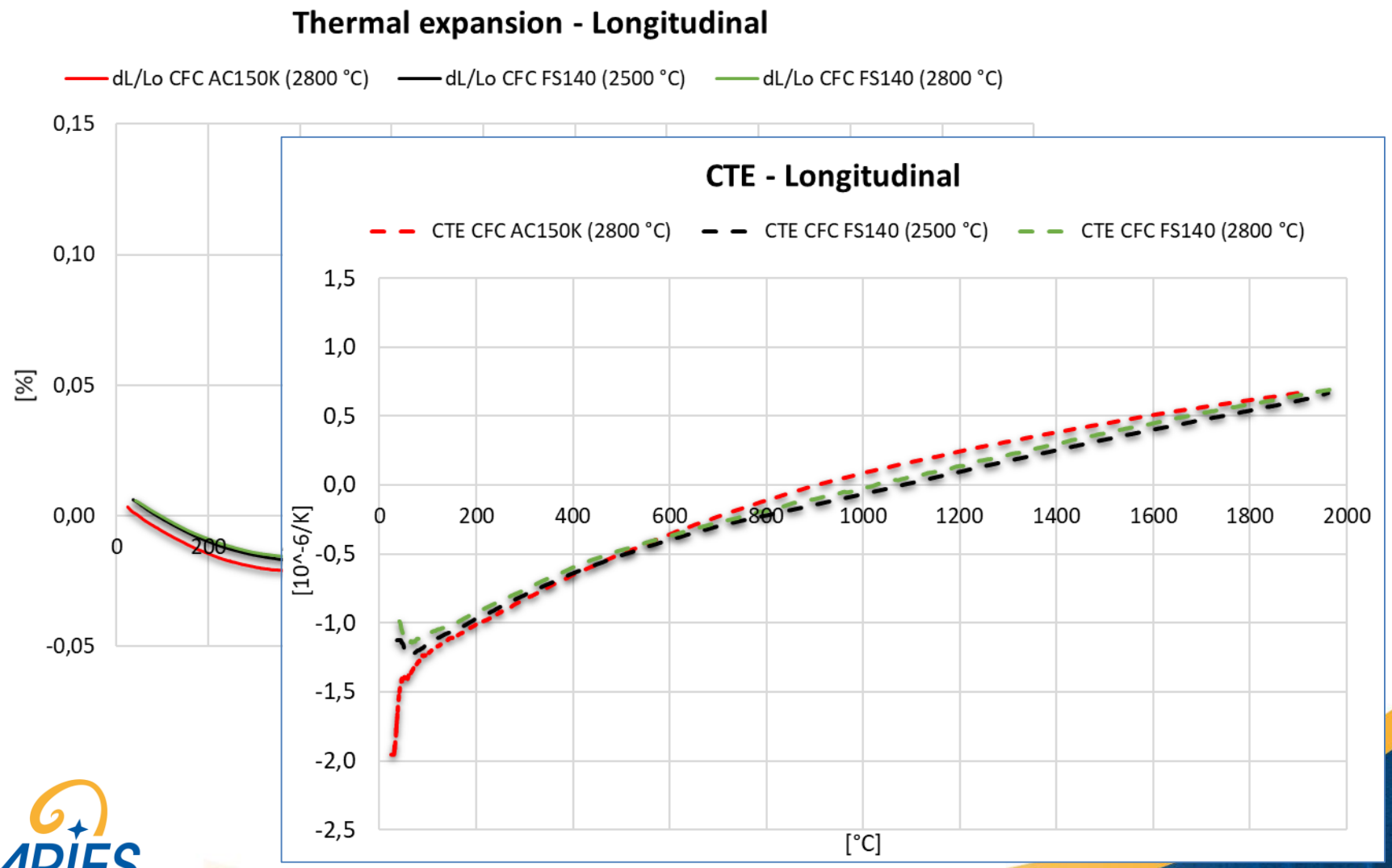


Carbon Fiber-Carbon: Thermal properties 2/4

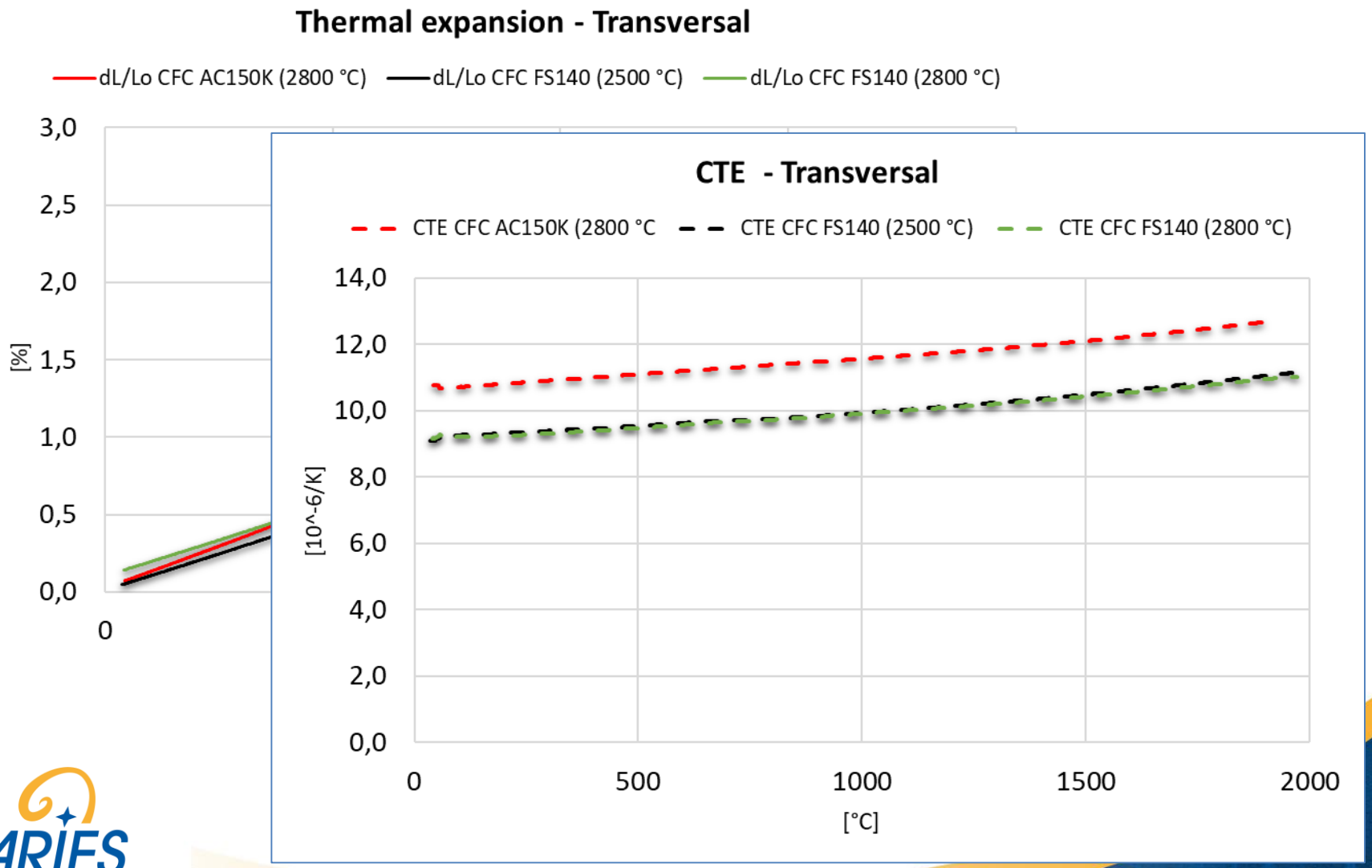


*3 mm thick samples

Carbon Fiber-Carbon: Thermal properties 3/4



Carbon Fiber-Carbon: Thermal properties 4/4



Summary

Property		CFC AC150K	CFC FS140 (2500 °C)	CFC FS140 (2800 °C)	Nb-8304Je	TG-1100	HOPG	MG-6403Fc
Density [g/cm^3]		1.89	1.87	1.92	2.57	2.19	2.25	2.54
cp @ 20°C [J/gK]		0.76	0.72	0.74	0.63	0.64	0.69	0.62
CTE RT-1000°C [10^{-6}/K]	L	0.03	-0.07	-0.04	1.77	*Tests planned*	0.04	2.82
	T	11.6	9.93	9.93	15.4	*Tests planned*	33.6	10.9
λ @ RT [W/mK]	L	233	124	171	720	435	1685	738
	T	54	42	50	29	52	7.8	50
Flexural strength [MPa]	L	139	165	156	94	*Tests planned*	25.5	58
	T	10	9	10	13	*Tests planned*	*Tests planned*	10
Reference E [GPa]		62	73	*Tests planned*	83	*Tests planned*	25	60
Electrical conductivity [MS/m]#	L	Y=0.24 Z=0.18	Y=0.12 Z=0.09	*Tests planned*	0.96*	*Tests planned*	2.5*	1.01*
	T	0.03	0.02	*Tests planned*	0.05	0.08	0.0009*	0.07

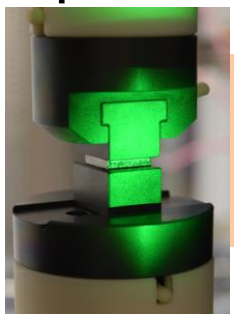
Uncertainties of measurements: EDMS_1371429, EDMS_1371432 *sigmatest **#Jorge G. Valenzuela**

Future steps

- *Ongoing*
  - CuCD (market survey)
  - CFC FS140 (2800 °C) dilatometry, IET, compression test
- Characterization of MultiMat spare specimens:
  - MG-6541-FC (New!)
  - TG-1100
  - CFOAM
  - HOPG Mechanical tests
  - …
- Investigate LFA capabilities with NETZSCH (→ meeting in Jan 2018)
- Benchmark with Material Research Facility UKAEA

Future steps

- Mechanical characterization up to 1200 °C



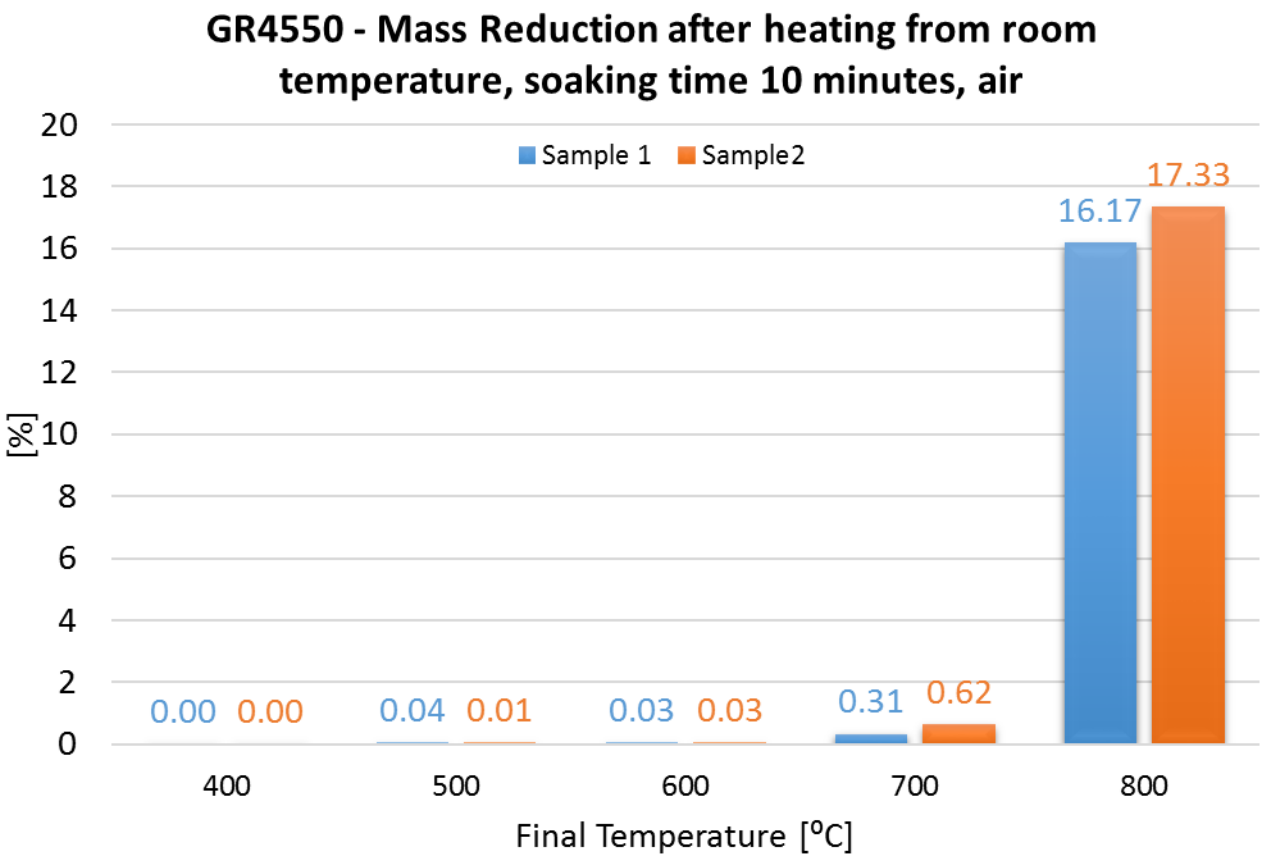
Compression test
•Cylindrical specimen
•Ø4-14.5 x h10

3- and 4-point bending test
DIN EN 843-1
Square specimen
•60x4x3mm

Tensile test
ISO 6892-2
•Ø5mm specimen
•M8 threaded rods

Future steps

- Mechanical characterization up to 1200 °C
- For graphitic material below 700 °C



Possibility to flush inert gas

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ARIES

Thank you for your attention!

Questions?

Back-up slides

Figures of Merit

Material	TRI	TSI	RFI
CFC AC150K	1372	47	0.24
CFC FS140 (2500 °C)	1943	28	0.12
CFC FS140 (2800 °C)	*ongoing*	*ongoing*	*ongoing*
Nb-8304Je	253.9	49.2	0.92
TG-1100	*not enough data*	*not enough data*	*not measured*
HOPG	*ongoing*	*ongoing*	*ongoing*

$$\mathbf{TSI} \propto \frac{\bar{\lambda}}{\bar{\alpha}\,\rho}$$

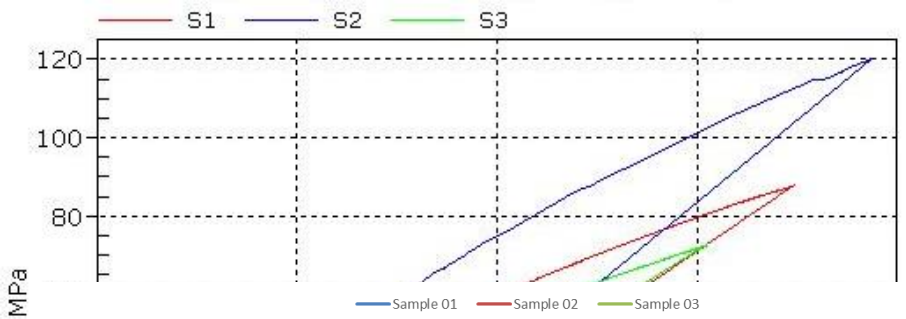
$$\mathbf{TRI} \propto \frac{R_M c_p}{\bar{E}\bar{\alpha}\rho}$$

$$RFI = \gamma$$

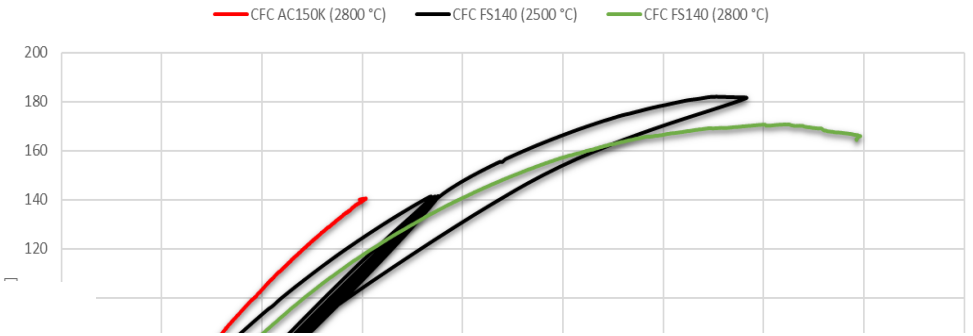
Material	TRI	TSI	RFI
MG-6530Aa	200	35	0.83
MG-6541Aa	226	37	0.98
MG-6403Fc	246	58	0.91

Flexural tests (4-point bending)

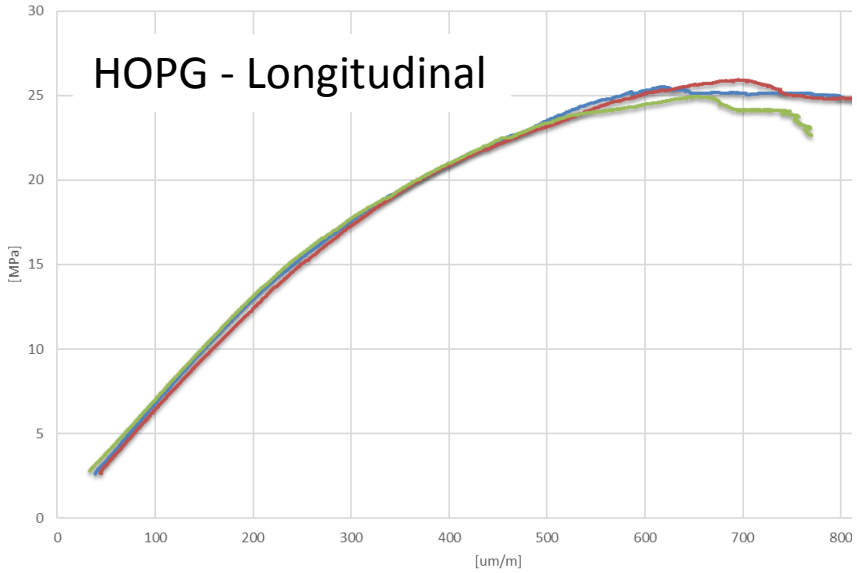
Nb-8304Je - Longitudinal

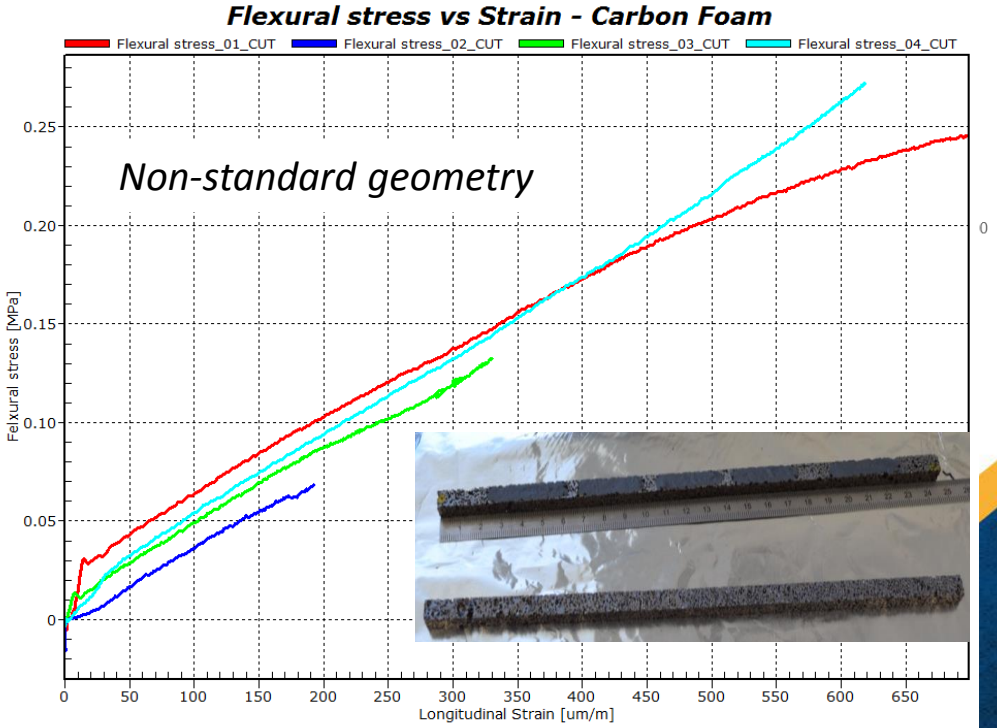


CFC - Longitudinal

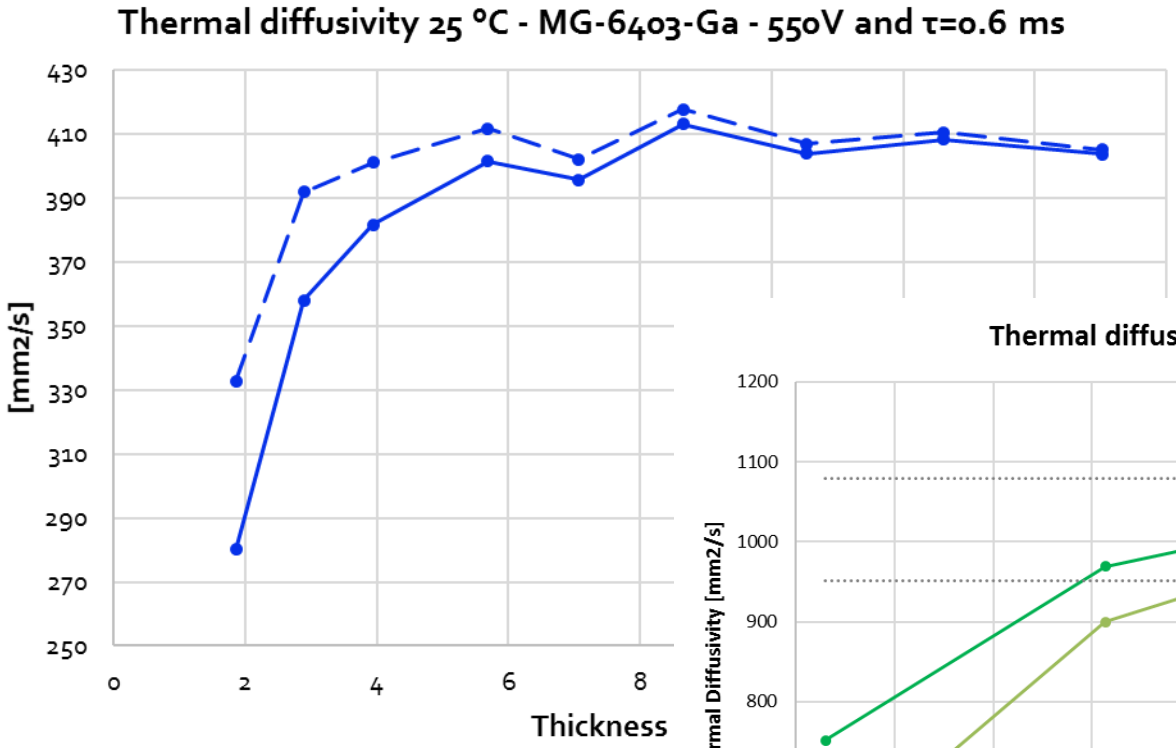


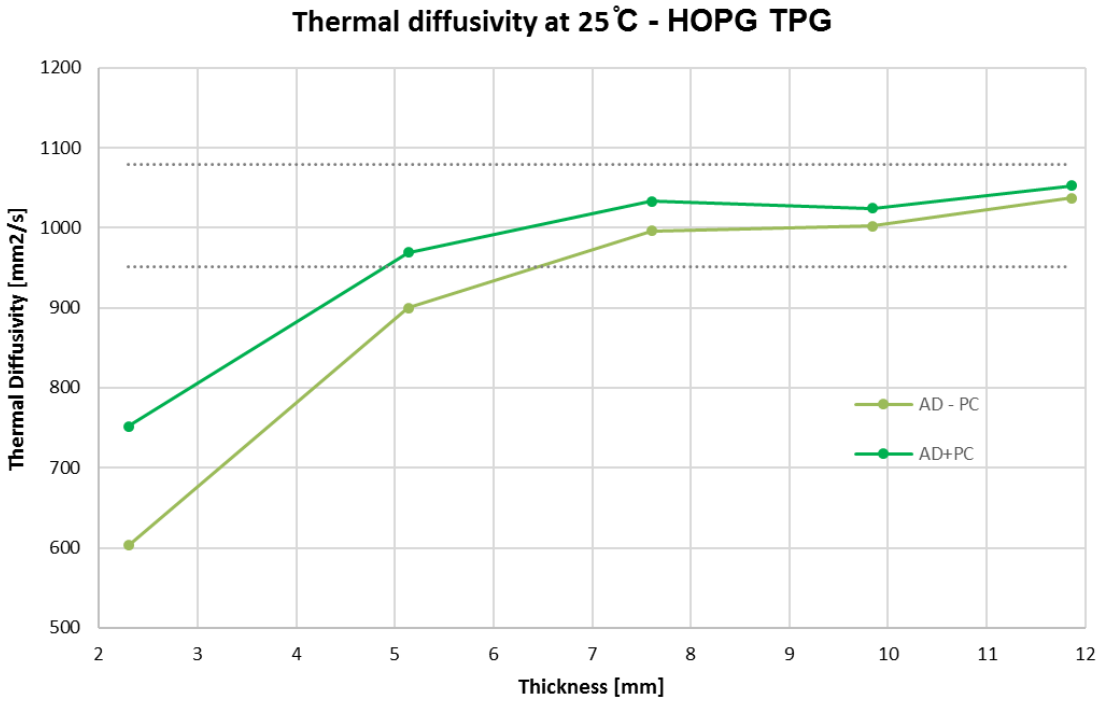
HOPG - Longitudinal





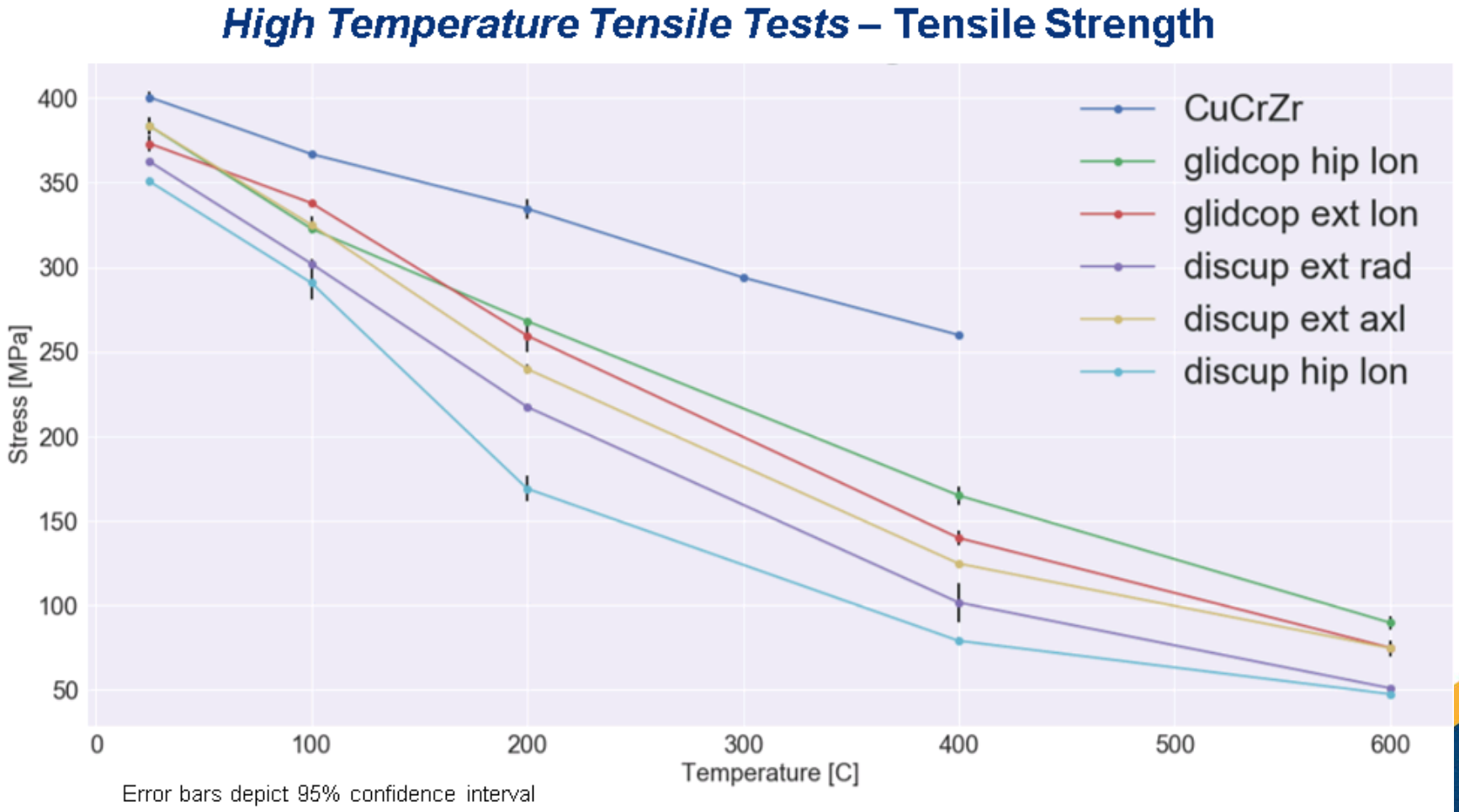
LFA limits: Thickness sensitivity





Mechanical testing at HT – Cu alloys

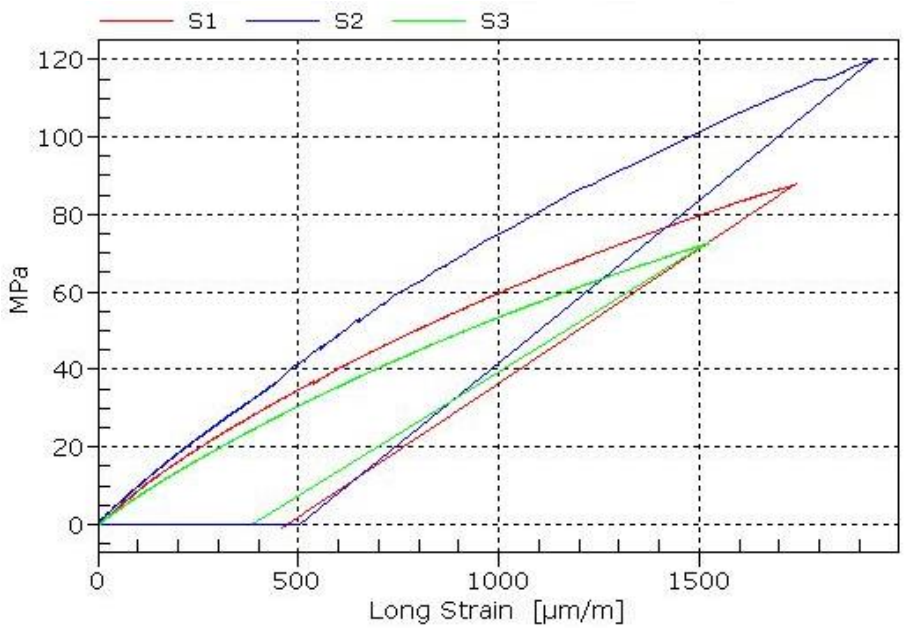
- Mechanical characterization up to 1200 °C



Nb-8304Je: Flexural test

4-point bending tests

Longitudinal



Transversal