

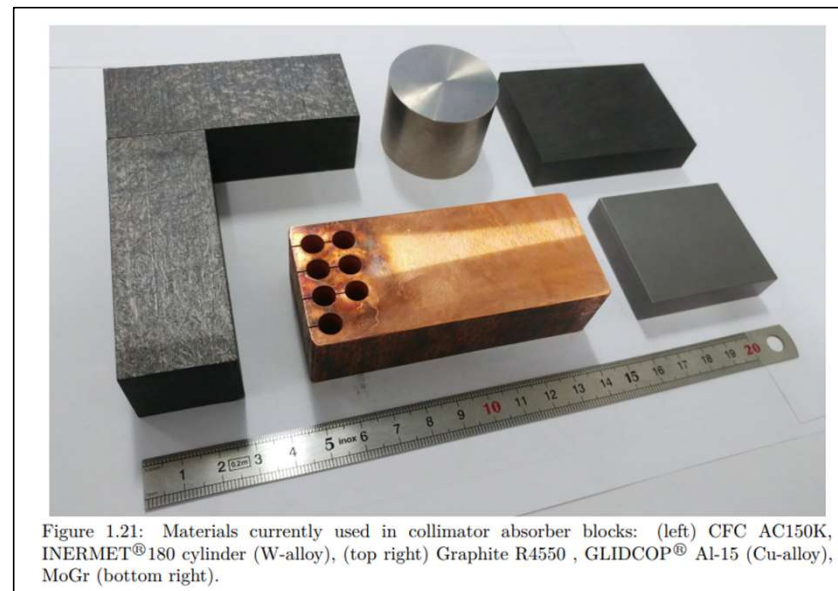


“MAKING THE SMALL PROFITABLE”

I-FAST: Graphite-Metal carbide materials

BACKGROUND INFORMATION

	MoGr		CFC AC150K		R4550	GLIDCOP	INERMET	SS-316L
		⊥		⊥	Isotr.	Isotr.	Isotr.	Isotr.
ρ [$g\ cm^{-3}$]	2.5-2.6		1.89		1.86	8.75	17.94	7.95
T_m [$^{\circ}C$]	2589		3650		3650	1083	1350	1400
c_p [$J\ g^{-1}K^{-1}$]	0.6-0.65		0.71		0.71	0.39	0.15	0.45
Z	6.6-6.8		6		6	28.96	67.66	25.5
A	13.5-13.8		12.01		12.01	63.62	166.67	54.7
γ_e [$MS\ m^{-1}$]	0.9-1.2	0.05-0.07	0.24-0.18	0.03	0.08	53.8	8.7	1.35
a [$mm^2\ s^{-1}$]	430-530	28-37	174-227	40	73	106	34	4
k [$W\ m^{-1}K^{-1}$]	650-900	45-65	233-304	54	100	365	91	14
α [$10^{-6}K^{-1}$]	1.7-2.7	8-12	-0.8	11	4.2	18.5	5.3	15
R_M [MPa]	60-80	10-12	105-140	10	60	375 (T)	683 (T)	515 (T)
E [GPa]	60-85	4-5	\approx 50	5	11.5	128	360	195
ε_{adm} [%]	0.18-0.26	0.45-0.72	0.14-0.2	0.43	0.7	10	3	40
X_0 [$g\ cm^{-2}$]	39-42		43.0		43.0	13.2	7.2	14.4
X_g [m]	15-17		22.8		23.1	1.5	0.4	1.8
ΔT_q [K]	2.4-2.9		1.5		1.5	57.1	646	40.6
TRI	141-367		1362		3445	6.3	0.6	10.9
TSI	43-73		47		23	0.9	0.1	0.1
RFI	0.9-1.1		0.4		0.3	7.3	2.9	1.2



Information (pictures) from PhD Jorge Guardia Valenzuela:

“OPTIMISATION OF GRAPHITE-MATRIX COMPOSITES FOR COLLIMATORS IN THE LHC UPGRADE”

Tender IT 4201

MoGr series production for LS2

- Material to equip **10 TCSPMs and 5 TCPPMs** (+20% spare material)
- Total of 360 pieces (blocks + tapered extremities)
- Contract for uncoated blocks and taperings awarded to Nanoker (ES)**
- Production rate: 0.5 and later 1 batch*/month
- Conditioning and Acceptance tests at CERN:** thermal treatments, US, metrology, thermophysical and UHV tests
- Technical officer: F. Carra (EN/MME)




- Contract for **coating TCSPM blocks awarded to DTI (DK)**, after successful qualification of samples and, later, of two preseries!
- Conditioning and Acceptance tests at CERN:** thermal treatments, UHV, adherence, coating thickness and electrical conductivity
- Technical officer: W. Vollenberg (TE/VSC)

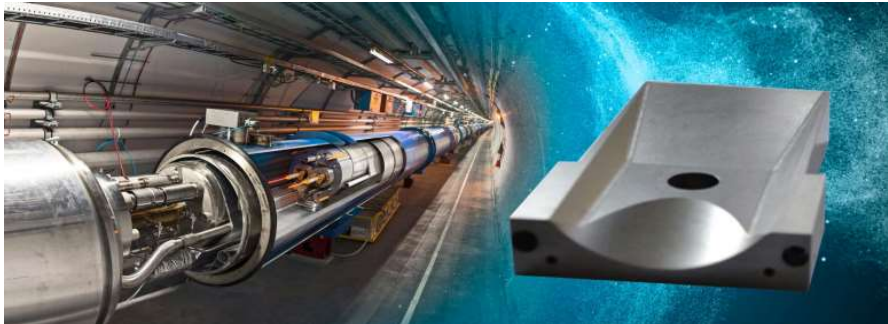





1 batch* = material to equip 1 collimator

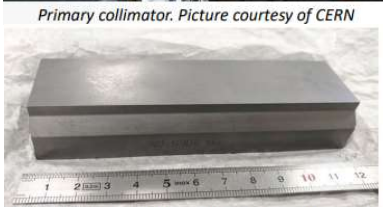
F. Carra (CERN), 2 September 2020

Picture taken from presentation F.Carra (CERN) Sep 2020

GRAPHITE-METAL CARBIDE COMPOSITES

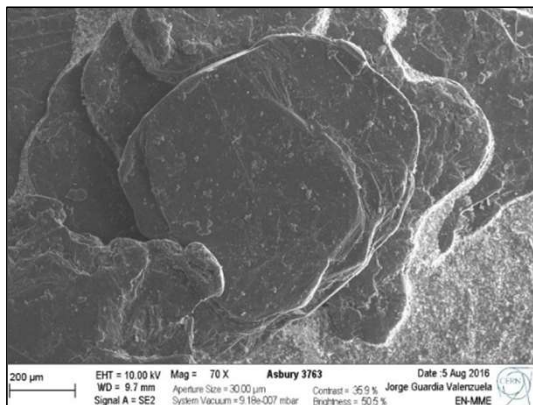
Primary collimator. Picture courtesy of CERN



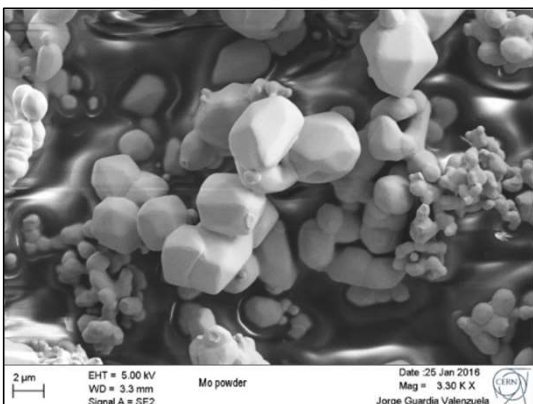
Block for collimator (position tolerance of 5 micron)

Physical properties		
Parameters	Units	X,Y Z ¹
Density	g/cm ³	2.57
Flexural Strength	MPa	102.1 16.9
Flexural Strain to rupture	µm/m	2580 5900
Young Modulus	GPa	69.7 5.5
Thermal conductivity (@20°C/300°C)	W/m-k	650/310 45/23
Thermal Diffusivity (@20°C/300°C)	mm ² /s	390/110 27/8
CTE average (20-1000°C)	10 ⁻⁶ K ⁻¹	6.5
CTE ² (20-1000°C)	10 ⁻⁶ K ⁻¹	2.4 14.7
Specific heat	J/g-K	0.65
Electrical conductivity	MS/m	0.8
Dimensional stability	%	0 0.1

All properties measured at 20°C unless otherwise stated
¹ XY - Parallel to the grain direction; Z - Perpendicular to the grain direction
² CTE adjustability according to chemical composition



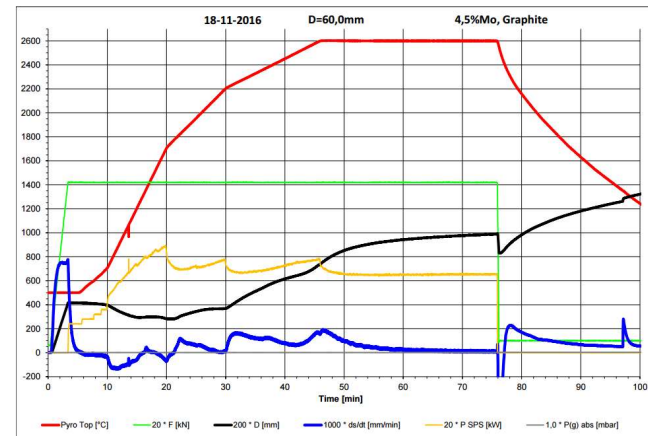
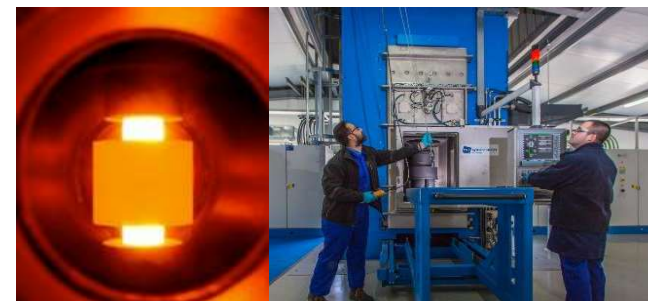
Graphite flakes



Mo powder

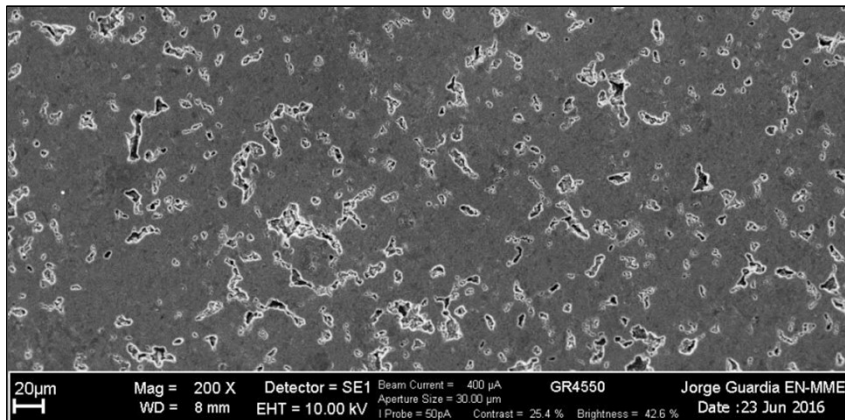


Attrition milling

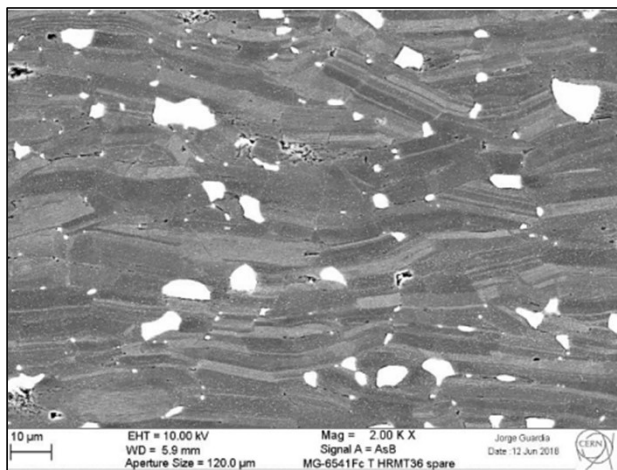


Phase I: SPS, 2650°C 25 MPa

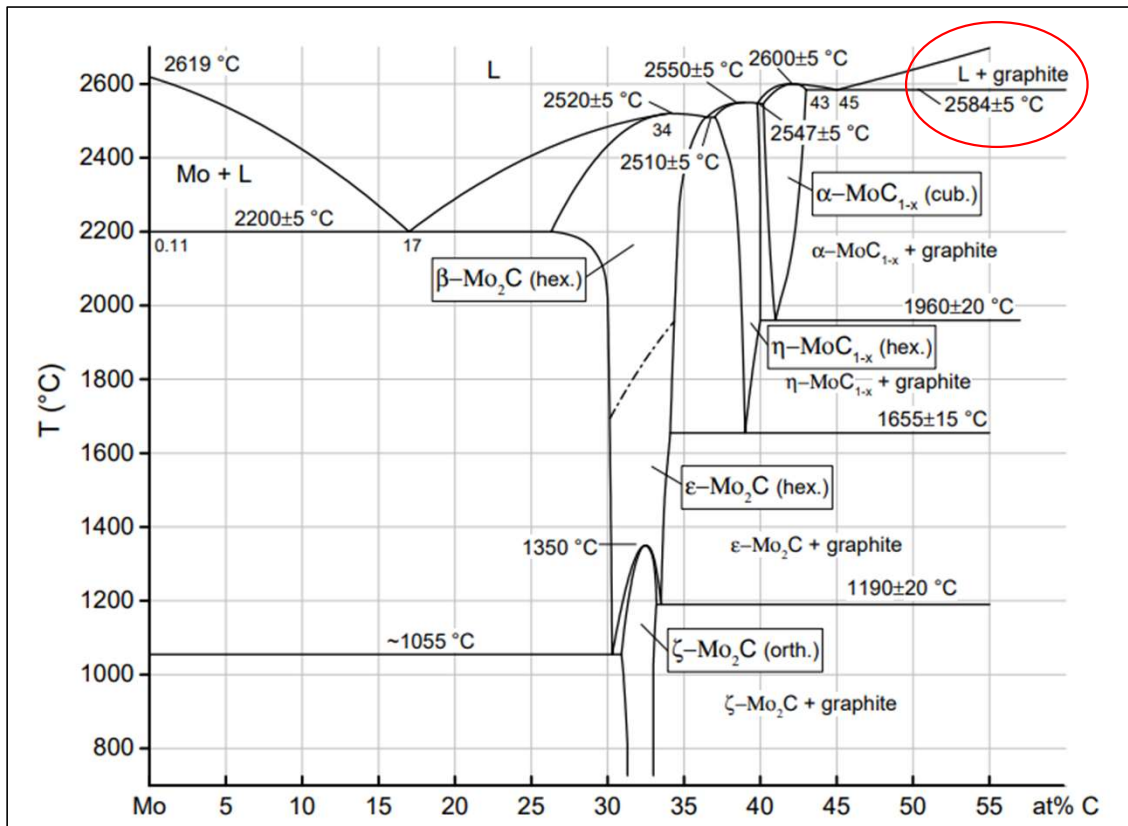
Phase II: Annealing 2350°C



Microstructure of Isostatic graphite



Microstructure of LP Gr-Mo



Phase diagram Mo-C (Equilibrium point)



Molten metal-carbide



Generation of defects

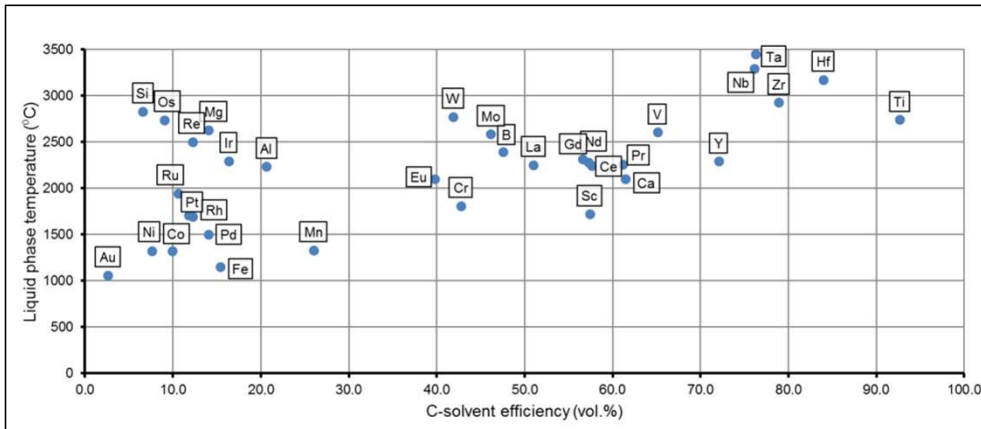
(In-plane) CTE Gr-Mo: $2.4 \cdot K^{-1}$
CTE Graphite: $4.2 \cdot K^{-1}$

Graphite-Molybdenum carbide:

- Expensive material (16-20 €/cc)
 - High consumable cost (mould sacrifice)
 - High energy cost (2650°C + annealing at 2350°C)
 - Effect of scale (2 x D175 mm blanks)

I-FAST activities:

- Alternative material to Graphite-Molybdenum
- Reduce the cost of material
 - Lower consumable cost (no graphite sacrifice)
 - Lower energy (reducing sintering T^a)
 - Upscaling (2x D230 mm blanks)



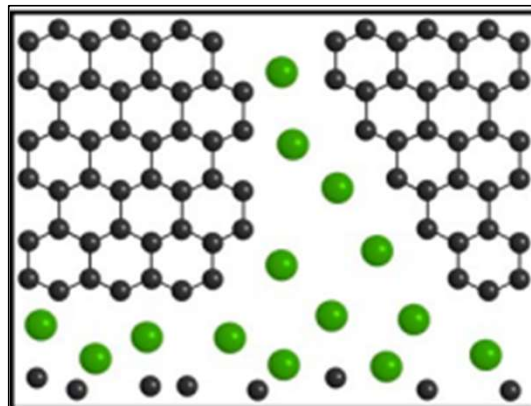
Liquid phase temperatures

Selection of Chromium driven by:

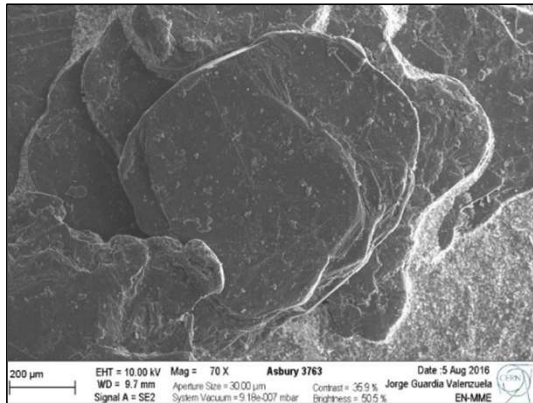
- Liquid phase formation of carbide < 2000°C
- Low cost of metal powder
- High C-solvent efficiency
- Promote solution-precipitation mechanism

	Min.	Max.	Market [€/g]
Sc	652	804	5-13*
Cr	0.4	2.2	
Mn	1.0	4.3	
Fe	0.04	0.06	0.08 (1 kg)
Co	1.1	7	29.71 (1 kg)
Ni	1.0	6.6	11.67 (1 kg)
Ru	65.4	65.4	7.50
Rh	528	528	86.38
Pd	89	264	38.8
Pt	181	609	25.86
Au	394	529	37.15
Mo	0.5	15	

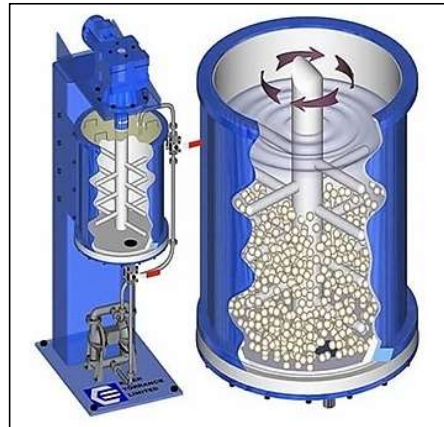
Prices



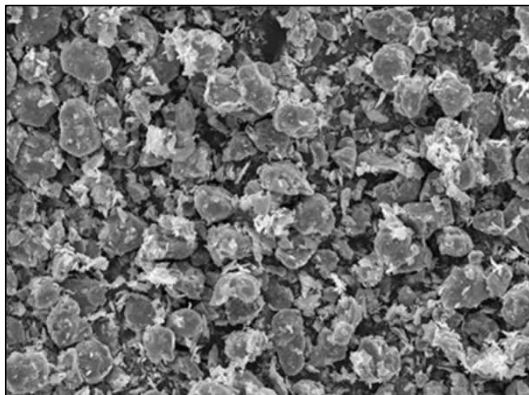
Solution - precipitation



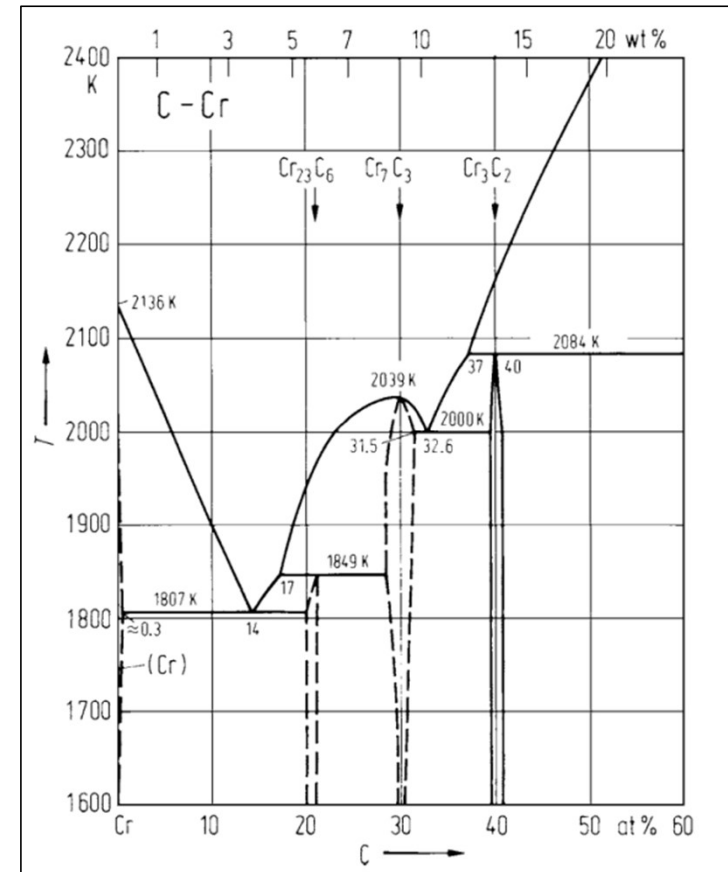
Graphite flakes



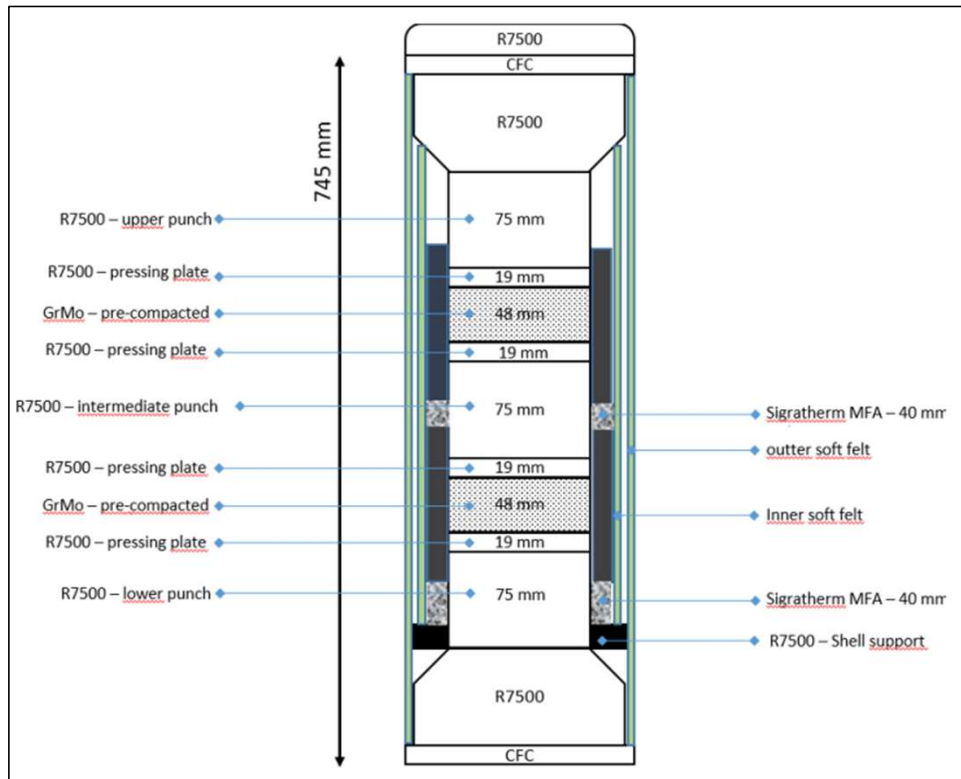
Attrition milling



Cr powder



Phase Diagram (liquid phase at 1850 °C)



Mould configuration



2x Mould configuration



1^o Insulation layer



2^o Insulation layer



Mould filling

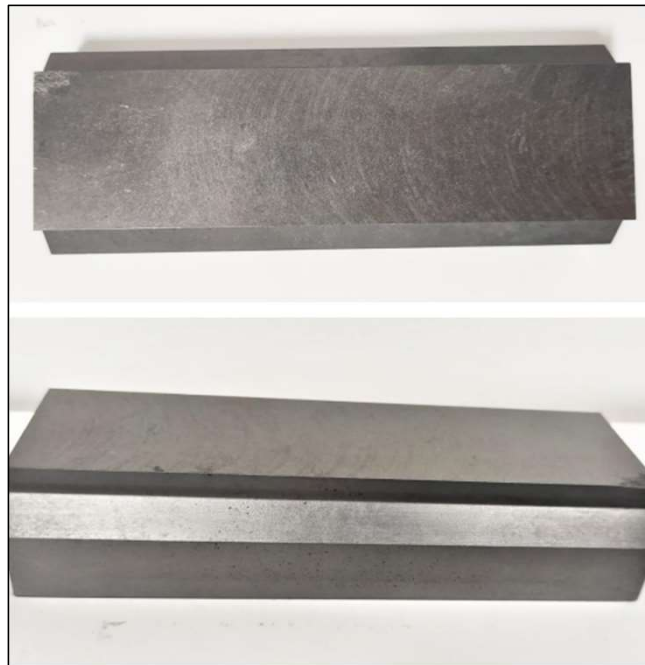
Sintering Run	Production period	Plate number	Diameter (cm)	Density (g/cm ³)	Electrical Conductivity (MS/m)
#1*	November 2021	Plate #1	170	2.30	1,00 – 1,07
#2*	March 2022	Plate #2	170	2.23	0.75 – 0.76
		Plate #3	170	2.29	0.80 – 0.81
#3 (D4.4)	March 2023	Plate #4	230	2.44	0.86 – 1.05
		Plate #5	230	2.44	0.91 – 0.94

**values after stress-relaxation annealing cycle at 1700°C*

Test performed to Cr-Gr in the framework of I-FAST

Property	Specification		CrGr Plate #1		
	I*	I*	I*	I*	Unit
Density at 20°C	2.40 – 2.60		2.32		[g/cm ³]
Specific heat at 20°C	> 0.6		0.687		[J/(g·K)]
Electrical conductivity at 20°C	> 0.75		1.02		[MS/m]
Thermal Diffusivity 20°C / 300°C	> 350/100	> 20/6	470/120	33/9	[mm ² /s]
Thermal conductivity at 20°C / 300°C	> 500/280	> 35/20	750/350	52/27	[W/(m·K)]
Volumetric CTE 20-1000°C	< 7		6.7		[10 ⁻⁶ K ⁻¹]
Coefficient of thermal expansion 20-1000°C	< 2.9	< 15	4.0	12.0	[10 ⁻⁶ K ⁻¹]
Young's Modulus at 20°C	35 < E < 75	5 < E < 8	46	3	[GPa]
Flexural strength at 20°C	> 60	> 10	58	8	[MPa]
Flexural strain to rupture at 20°C	> 2500	> 4000	3280	4200	[µm/m]
Dimensional stability	< 0.05	< 0.25	-0.05	0.45	%

Characterization to Plate #1



Block Type I machined on Plate #1

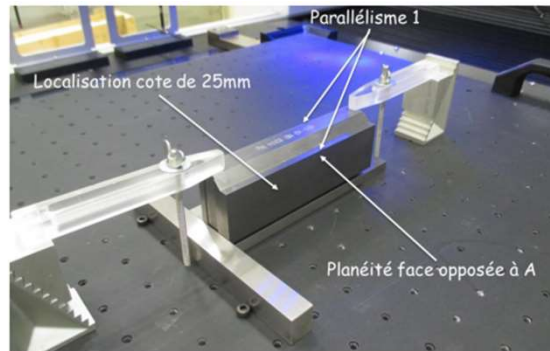


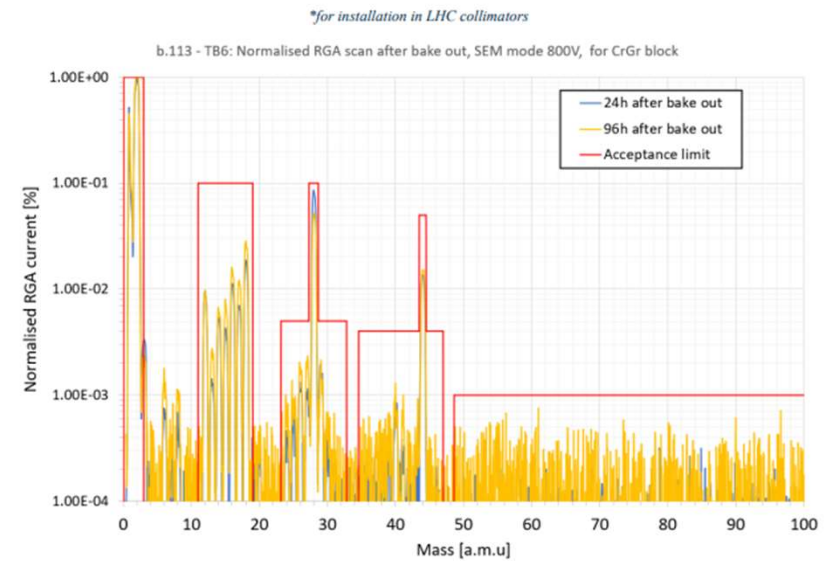
Figure 9 – CrGr absorber block, positioning for metrology measurement at CERN.

Table 5. Variation of tolerances of the CrGr absorber block after different post-machining time intervals.

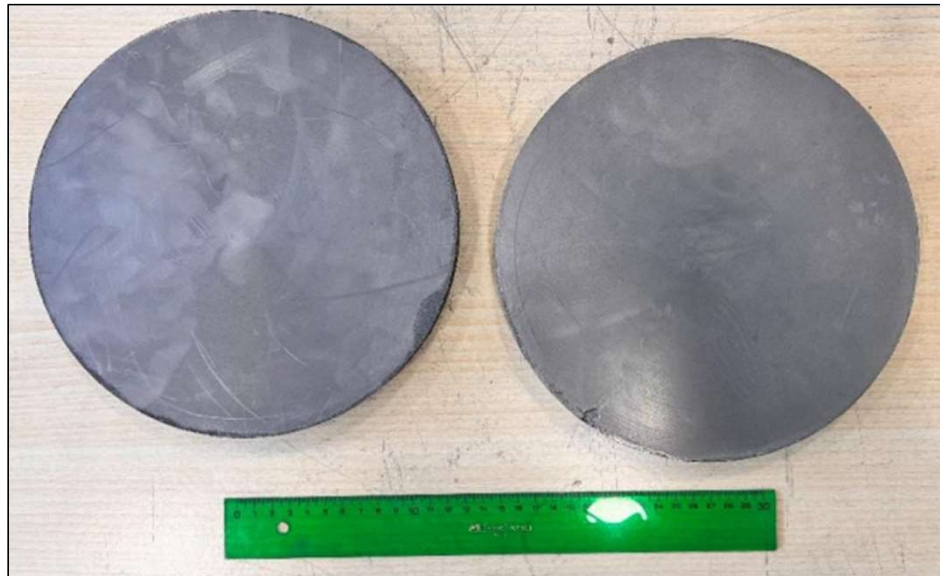
Tolerance*	Time interval after machining			
	7 days	1 month	2 months	3 months
Flatness of "A" face [mm]	0.010	0.011	0.011	0.012
Flatness of face opposite to "A" (i.e. "Beam face") [mm]	0.005	0.011	0.011	0.010
Parallelism of "Beam face" with respect to "A" [mm]	0.022	0.019	0.021	0.038
Position of "Beam face" with respect to "A" [mm]	0.025	0.022	0.021	0.042

*with reference to Annex B.

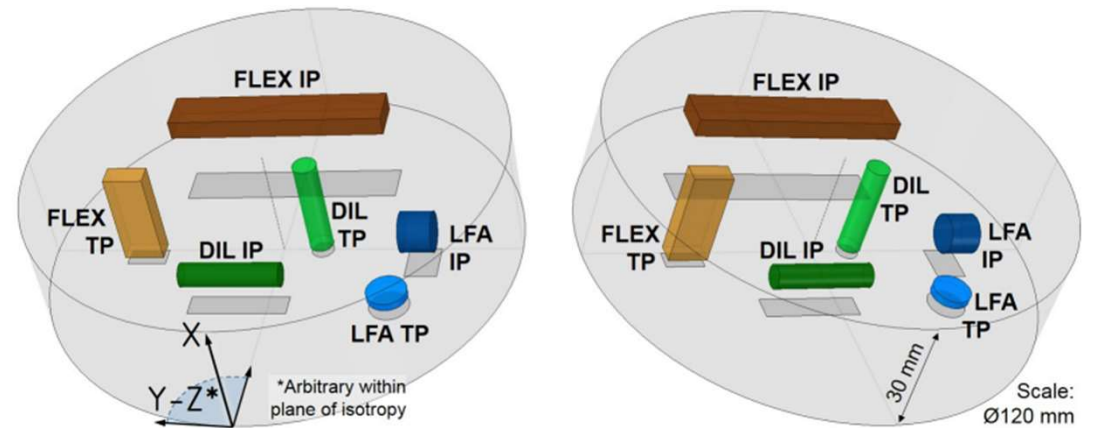
Dimensional stability



Outgassing test



2 blocks of D230 mm Cr-Gr



Characterization in D230 mm (ongoing)

Graphite-Chromium carbide:

- Reduction of cost of material (4-6 €/cc)
 - Lower consumable cost (reusable mould)
 - CTE compatibility between Cr-Gr and Graphite mould and lower T^a
 - Lower energy cost (72% reduction compared to Mo-Gr)
 - Upscaling (2 x D230 mm blanks)

- Material properties (measured in D175 mm)
 - Fulfillment of initial specs (Thermo-physical-mechanical)
 - Dimensional stability.
 - Compatible to be installed in the collimator (Outgassing)



“MAKING THE SMALL PROFITABLE”

I-FAST: Graphite-Metal carbide materials