This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under GA No 101004730.

WP4 Managing Innovation: Beam Windows and Composite Materials

I.FAST 3rd Annual Meeting

18th April 2024

Paris, France

F. Carra (CERN)

With contributions from WP4.3 & WP4.4 members, CERN IRRAD and FLUKA teams

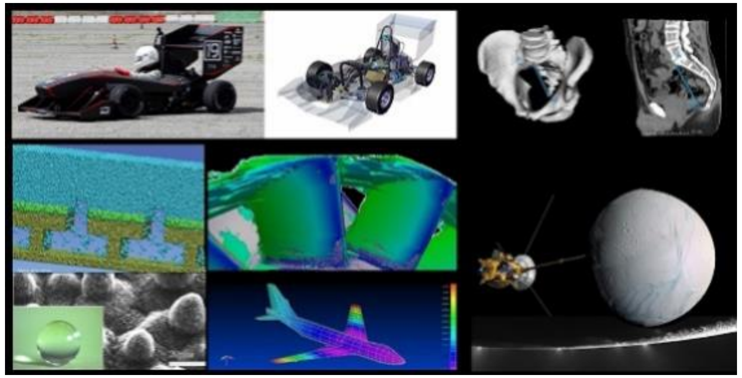
IFAST

Outline

- WP4.3 – Innovative beam windows for high-power accelerator applications
- WP4.4 – Carbide-Carbon Materials for Multipurpose Applications
- SAC recommendations and Technology Readiness Levels (TRL)
- Conclusions

WP4.3 – Innovative beam windows for high-power accelerator applications

- **Beam window**: separating environment at different pressures (vacuum/atmosphere or differential vacuum levels)
- Two technical solutions investigated within WP4.3
  - **Metallic windows** (tantalum, T91 steel, aluminium alloys)
  - **Graphenic windows** (thin graphene membranes)
- Task participants: **GSI (DE), RHP (AT)** and **CERN** + collaboration with **La Sapienza University - DIMA (IT)**



WP4.3 – Innovative beam windows for high-power accelerator applications

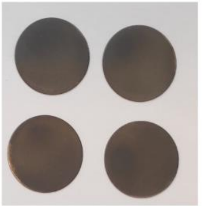
Milestone/Deliverable Number	Title	Lead beneficiary	Type	Dissemination level	Due Date (in months)
MS13	First characterisation of beam windows materials under thermomechanical load and extended radiation damage	GSI	Report	Public	18
D4.3	Manufacturing and testing of two beam-windows prototypes	CERN	Demonstrator	Public	32

Milestone MS13 *achieved in 2022* (*link*)

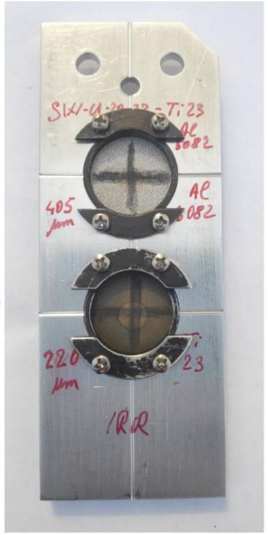
- **Ion irradiation of window foils** of different materials at GSI M-Branch UNILAC → T91 steel, Titanium grade 23, Inconel 718, Aluminium 6082 T6, graphene
- **Online and post-mortem characterization** of mechanical and thermal properties
- Well documented in **Marilena's *talk*** at 2nd IFAST Annual Meeting
- **Scientific production** on top of milestone report (**L. Notari** master thesis, international papers → *see "Scientific Contributions" section at the end*)

Courtesy of M. Tomut (GSI), L. Notari (La Sapienza)

Titanium Grade 23 (0.22 μm thickness)



Aluminium 6082 T6 (0.405 μm thickness)



WP4.3 – Innovative beam windows for high-power accelerator applications

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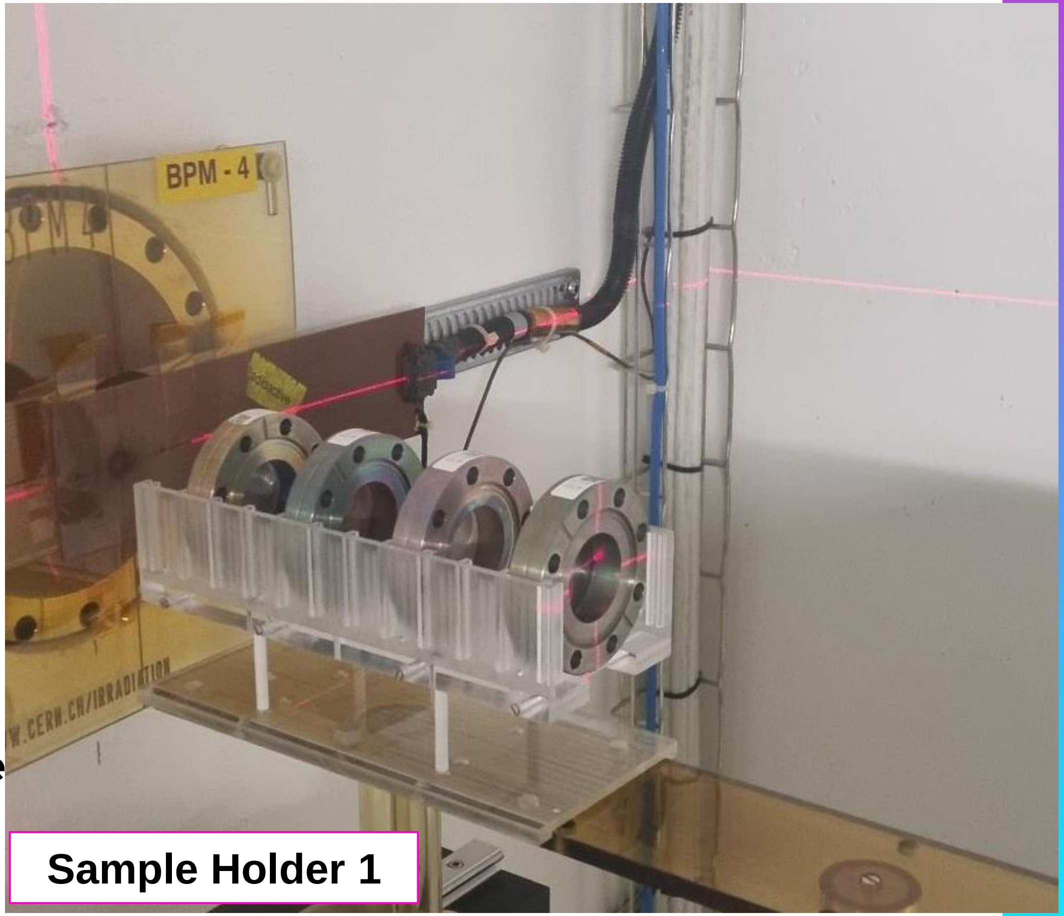
Deliverable D4.3

- Postponed by 6 months (June 2024) due to beam unavailability at GSI in ‘23
- Alternative solution found → **proton irradiation at CERN IRRAD in Sep-Oct ’23**
- Samples cooldown until March ’24 → **now thermomechanical characterization starting** (non-irradiated vs irradiated samples evaluation)
- Aim of completing a deliverable **draft in early May to the project office**

WP4.3 – Innovative beam windows for high-power accelerator applications

- **Facility**: IRRAD (PS proton beam, T8 beam-line at the CERN PS East Hall building 157) [***www.cern.ch/ps-irrad***](http://www.cern.ch/ps-irrad)
- **Beam**: protons @24 GeV/c
- **Samples**: thin foils brazed to metallic flange produced by **RHP**
  - **Sample Holder 1** (Irradiation period **6 September – 11 October 2023**, fluence up to 2e16 p/cm², dose up to 4.5 MGy)
    - Sample #7 → **Tantalum**, diameter 50mm, thickness 0.3mm
    - Sample #15 → **Tantalum**, diameter 50mm, thickness 0.4mm
    - Sample #13 → **T91**, diameter 50mm, thickness 0.4mm
    - Sample #10 → **T91**, diameter 50mm, thickness 0.6mm
  - **Sample Holder 2** (Irradiation period **April – June 2024, target fluence 1e17 p/cm²**)
    - Sample #8 → **Tantalum**, diameter 50mm, thickness 0.6mm
    - Sample #9 → **T91**, diameter 50mm, thickness 0.4mm
    - Sample #11 → **T91**, diameter 50mm, thickness 0.6mm
    - Sample #10 → **Graphene**, 10x10 mm, thickness 1 um
- D4.3 due by June 2024→ only **Sample Holder 1 will be in its scope**
- **Sample Holder 2** can then be part of the final report

Courtesy of G. Pezzullo, F. Ravotti (CERN)

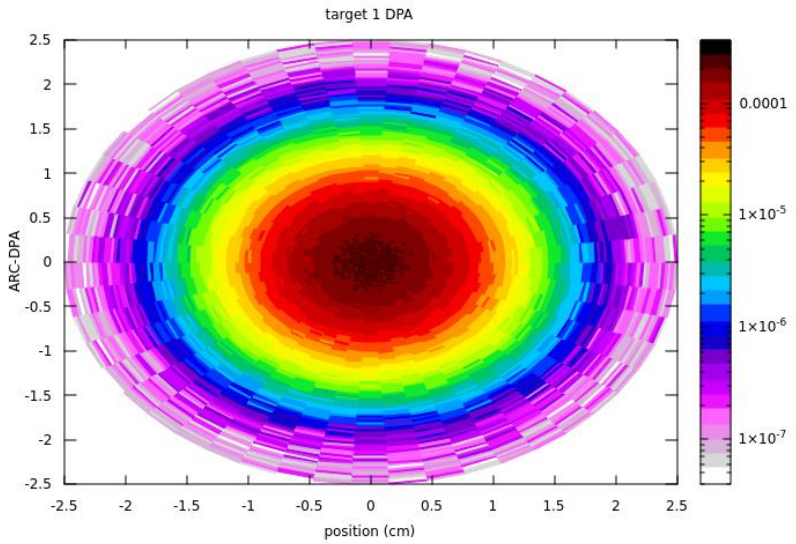


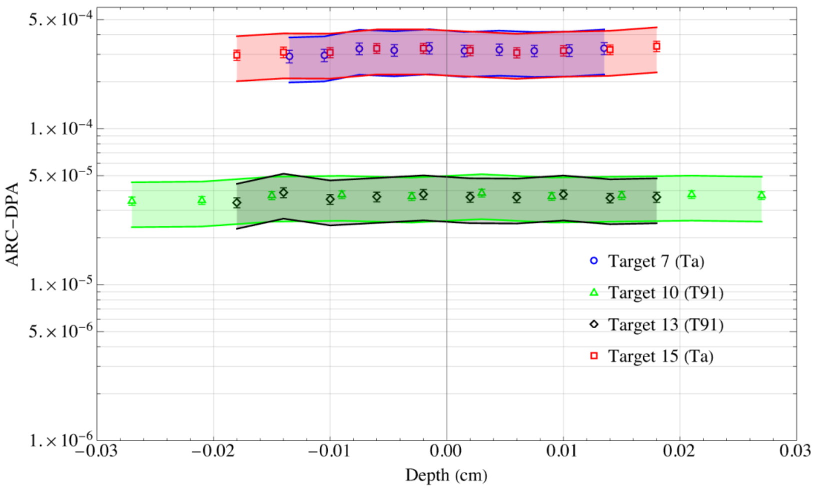
Sample Holder 1

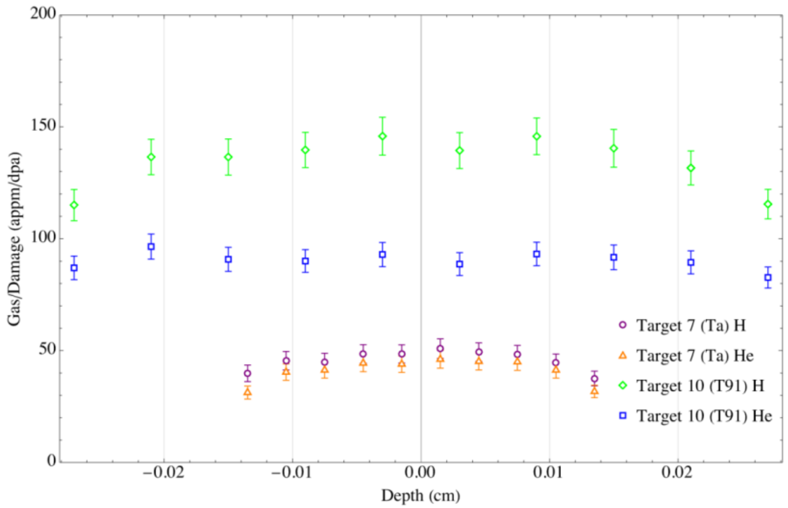
WP4.3 – Innovative beam windows for high-power accelerator applications

- Change in the thermomechanical properties will be correlated to the **estimated displacement-per-atom (DPA) and gas production** (big thanks to the FLUKA team at CERN SY-STI!)
- FLUKA simulations also allow to estimate the **temperature during irradiation** (preliminary: looks to be close to T_{room})
- Highest level of DPA in Sample Holder 1 **~5e-4 DPA** (ONGOING / PRELIMINARY ESTIMATION)

Courtesy of S. Marin, F. Salvat Pujol (CERN)
PRELIMINARY RESULTS!







WP4.4 – Carbide-Carbon Materials for Multipurpose Applications

- Large scale Carbide-Carbon Materials for multipurpose applications (M1 – M48)
  - **Promote the use of carbide-carbon materials (CCM)** in future particle physics facilities and open up the market to commercial applications (thermal transfer, aerospace light components, fusion, etc.)
  - Thermal conductivity 2-3 times higher than Cu! Stronger, low density
- How?

3x larger volume per sintering cycle

\+

1.5x lower sintering temperature material

→

2x Cost Reduction

Higher efficiency

- Who?

TAKING PROFIT FROM THE SMALL

WP4.4 – Carbide-Carbon Materials for Multipurpose Applications

Milestone/Deliverable Number	Title	Lead beneficiary	Type	Dissemination level	Due Date (in months)
MS14	Evaluation of a CCM alternative to Molybdenum-Graphite	CERN	Report	Public	16
D4.4	Production of large-size CCM plates	CERN	Demonstrator	Public	24

Milestone MS14 ***achieved in 2022*** *(**link**)*

- **Chromium-Graphite** (CrGr) proposed (and produced) as a valid alternative to MoGr
- Presented at the 1st IFAST Annual Meeting (see ***here***)

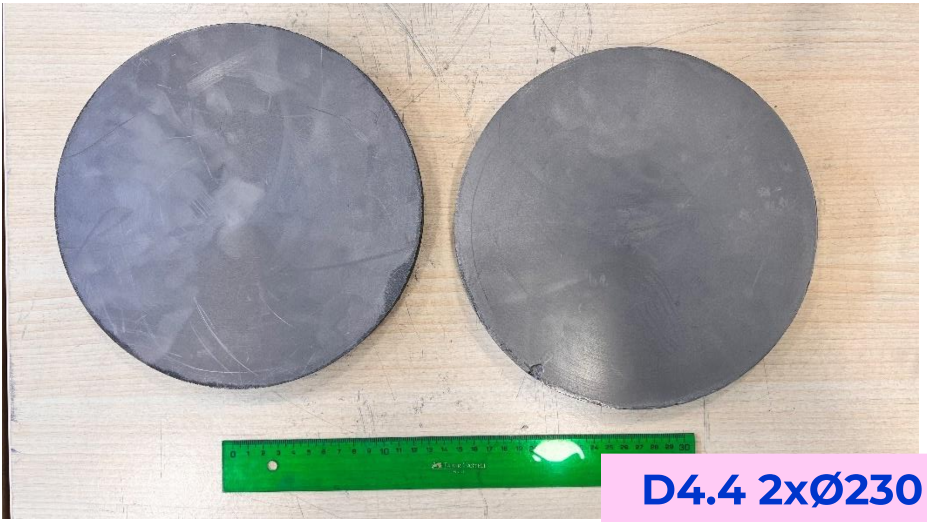
D4.4 ***achieved in 2023*** *(**link**)*

- **Two big (Ø230 mm) Chromium-Graphite disks produced in a single machine cycle** (doubling of disk cross-section, decreasing of the sintering temperature wrt MoGr)
- Presented at the 2nd IFAST Annual Meeting (see ***here***)

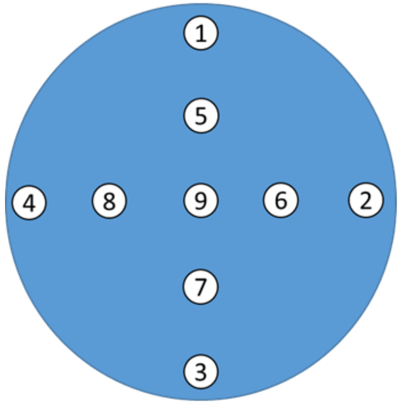
WP4.4 – Carbide-Carbon Materials for Multipurpose Applications

Possible additional activities until the end of the project

- **Already ongoing:** in-lab characterization at CERN of the full thermomechanical properties in temperature of the CrGr big disks (thanks to Nanoker for cutting > 100 samples!)
- One more sintering test with **increased disk thickness** (> 30 mm) to achieve record volume sintered per cycle?
  - Potentially followed by fine machining + metrology + UHV test?
- Scientific paper on CrGr → never disclosed in an international journal so far!



D4.4 2xØ230 mm CrGr plates



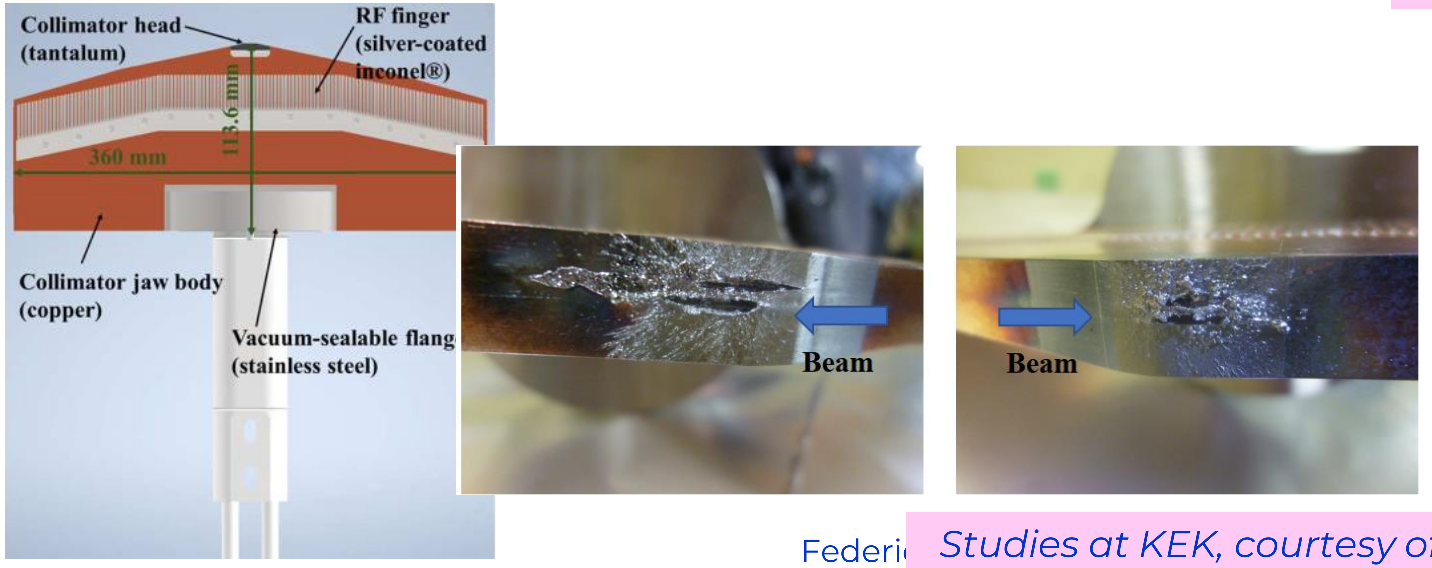
Conductivity (MS/m)		
Position	Side 1	Side 2
1	1,04	0,74
2	1,02	0,75
3	1,03	0,79
4	1,04	0,78
5	1,08	0,89
6	1,06	0,94
7	1,01	0,88
8	1,09	0,96
9	1,05	1,00
Average	**1,05**	**0,86**

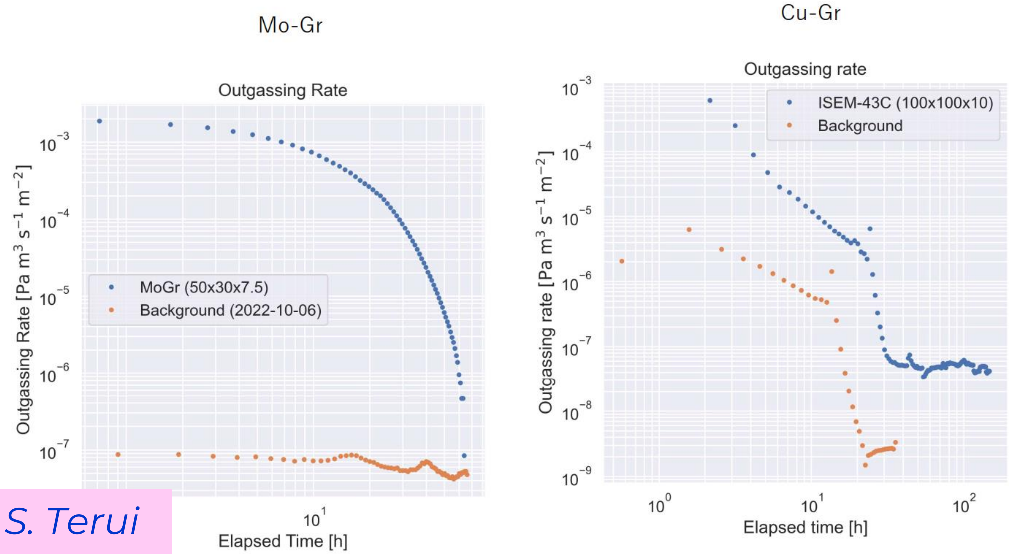
Conductivity (MS/m)		
Position	Side 1	Side 2
1	0,95	0,92
2	0,94	0,93
3	0,94	0,90
4	0,95	0,88
5	0,93	0,91
6	0,93	0,93
7	0,93	0,92
8	0,94	0,91
9	0,92	0,93
Average	**0,94**	**0,91**

SAC recommendations and TRL

- **SAC Feedback from 2nd annual meeting:**
  - Carbide materials: it is a very successful development work good for HL-LHC at CERN and also for KEK-SuperKEKB.
- **15 HL-LHC collimators with MoGr already operating** in the LHC, CrGr development can provide cheaper solution
- **KEK collaboration via IFAST**: several material samples sent to KEK for study on SuperKEKB collimators

Big MoGr block for KEK studies, courtesy of S. Rivera





Studies at KEK, courtesy of S. Terui

SAC recommendations and TRL

- **SAC further (general) suggestions**
  - I.FAST technologies should be critically assessed with regards to a realistic pathway to identified markets and the time scales required.
  - For each technology, it would be good to get idea of technology readiness level (TRL) as not all developments seem to have the same level of market-readiness.

Technology	TRL	TRL definition	Time until market readiness (TRL=9)*	Comments
Ta and T91 beam windows	**6**	Technology demonstrated in relevant environment (industrially relevant environment in the case of key enabling technologies)	**2 years**	Assembly (window + flange bonding) requires optimization and testing at higher does / flux
Molybenum-Graphite	**9**	Actual system proven in operational environment (competitive manufacturing in the case of key enabling technologies; or in space)	**–**	Installed in 12 HL-LHC collimators, currently under operation!
Chromium-Graphite	**4**	Technology validated in lab	**2 years**	Based on the experience with MoGr

**in presence of budget and a driving application scenario concretizes*

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Conclusions

- **WP4.3 Beam Windows:**
  - Beam windows assemblies prepared by RHP and irradiated at CERN at IRRAD in 2023
  - Cooldown completed in April 2024 and samples now under post-irradiation characterization + DPA / gas production calculations
  - Results will be included in deliverable D4.3 (aiming at a draft in May)
- **WP4.4 Carbide Materials:**
  - MS and D completed, but additional activities already agreed / ongoing or under discussion
  - Main one is the thermomechanical characterization of the two big disks of Chromium-Graphite
- **Extensive scientific contribution** through master thesis and papers already produced and some more is coming!

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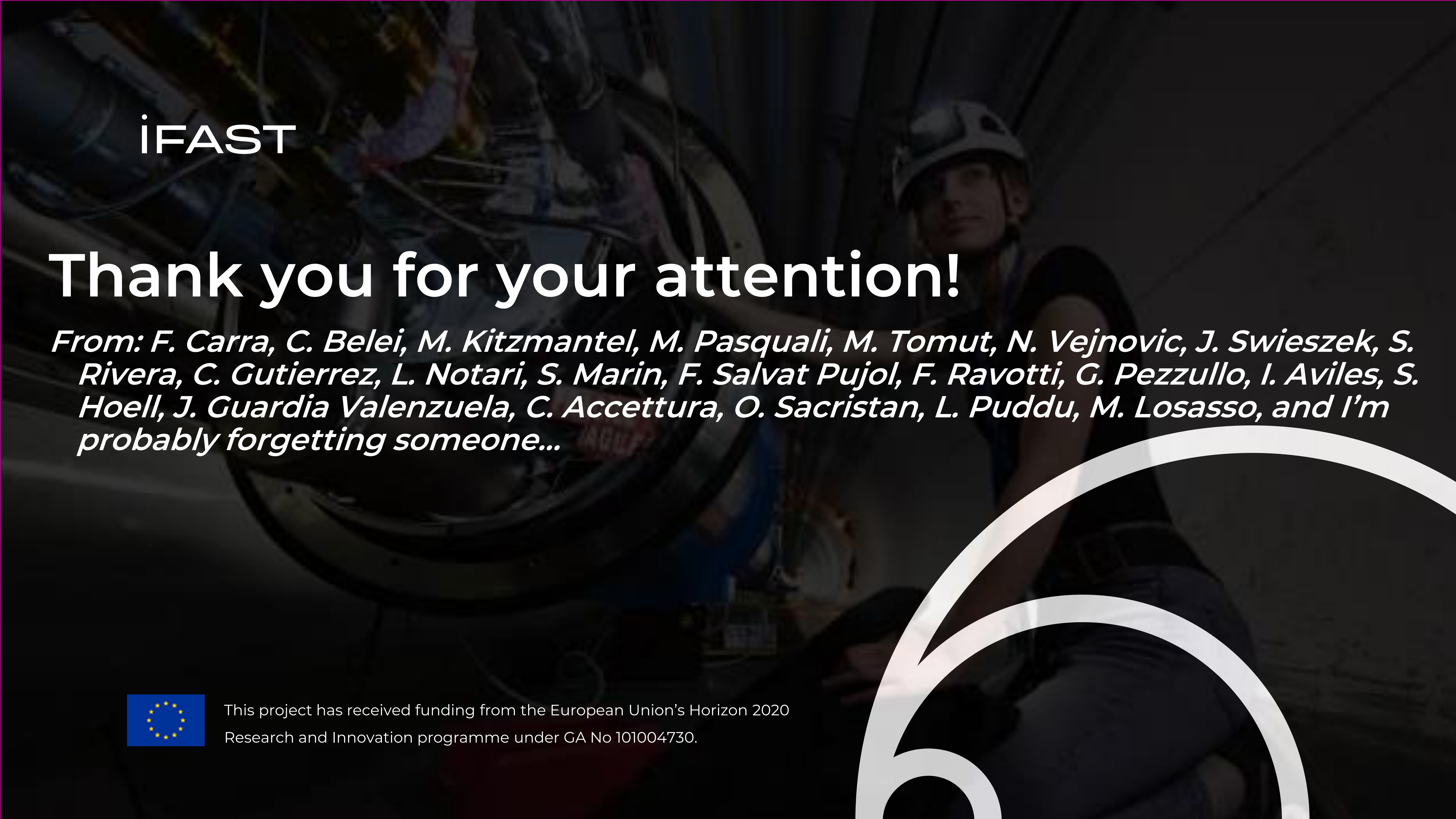
Scientific Contributions

- L. Notari, "*Dynamic radiation effects induced by short-pulsed U-ion beams in metallic targets*", Master Thesis, 10.5281/zenodo.7484054
- L. Notari, M. Pasquali, F. Carra, M. Losasso, M. Tomut, "*Materials adopted for particle beam windows in relevant experimental facilities*", https://zenodo.org/uploads/10964349
- L. Notari, M. Pasquali, F. Carra, M. Losasso, J. Guardia-Valenzuela, M. Tomut, "*Dynamic response to short-pulsed U-ion beams of material candidates for vacuum beam windows manufacturing*", submitted to Heliyon and under review.
- Additional paper "wishlist":
  - L. Notari on simulations of 2022 windows irradiation – ongoing
  - F. Carra on 2023/24 windows irradiation
  - M. Tomut on graphenic windows
  - J. Guardia on Chromium-Graphite



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Thank you for your attention!

From: F. Carra, C. Belei, M. Kitzmantel, M. Pasquali, M. Tomut, N. Vejnovic, J. Swieszek, S. Rivera, C. Gutierrez, L. Notari, S. Marin, F. Salvat Pujol, F. Ravotti, G. Pezzullo, I. Aviles, S. Hoell, J. Guardia Valenzuela, C. Accettura, O. Sacristan, L. Puddu, M. Losasso, and I'm probably forgetting someone…

This project has received funding from the European Union's Horizon 2020 Research and Innovation programme under GA No 101004730.

CCM Production Cycle

Selection and sieving of the powders

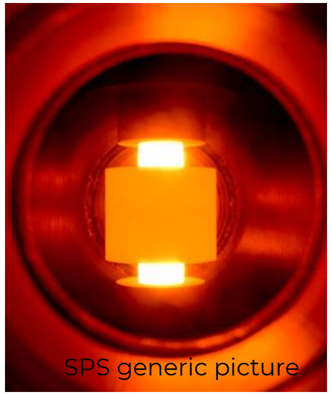
Preparation of the desired composition

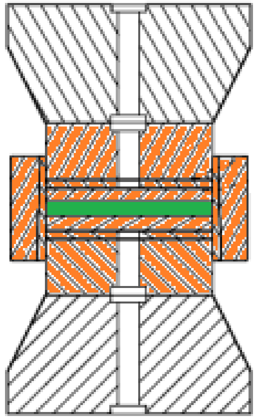
Powder	Vol. %
Graphite	93.9
Mo	5.5
Ti	0.6

Mixing

Green body

Spark Plasma Sintering (SPS) >25 MPa, >2600 °C

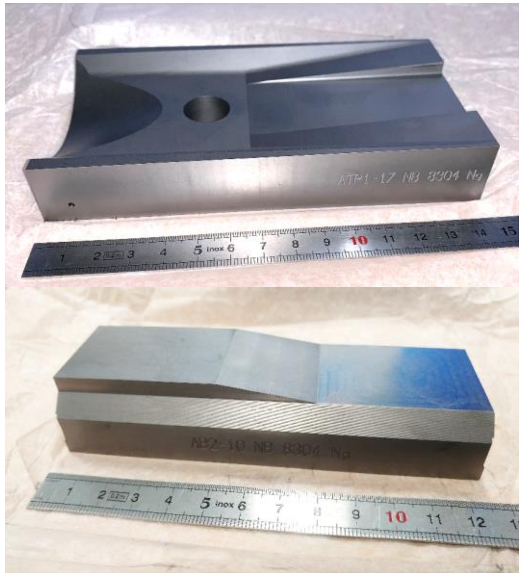




Sintered CCM
Single-use mould elements (graphite)

Stress relieving thermal treatment 0 MPa, >2400 °C

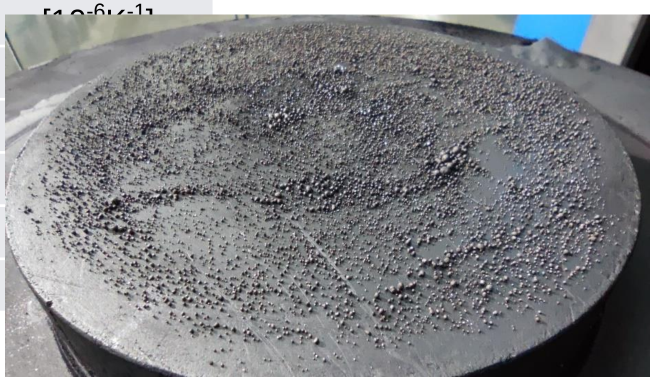
Machining of the pieces



Milestone MS14 – CrGr Characterization

- **Full thermomechanical characterization** done at CERN Mechanical Lab in April 2022
- **Very promising results**, some parameters to be improved during the next years of the project. Mechanical strength increased by a factor of 5 wrt ARIES!

	Specification		**CrGr Plate #1**		
Property	**∥***	**⊥**	**∥***	**⊥**	**Unit**
Density at 20ºC	2.40 – 2.60		2.32		[g/cm³]
Specific heat at 20ºC	> 0.6		0.687		[J/(g·K)]
Electrical conductivity at 20ºC	> 0.75		1.02		[MS/m]
Thermal Diffusivity 20ºC /at 300ºC	> 350/100	> 20/6	470/120	33/9	[mm^2/s]
Thermal conductivity at 20ºC /at 300ºC	> 500/280	> 35/20	750/350	52/27	[W/(m·K)]
Volumetric CTE 20-1000ºC	< 7		6.7		$[10^{-6}K^{-1}]$
Coefficient of thermal expansion 20-1000ºC	< 2.9	< 15	4.0	12.0	
Young's Modulus at 20ºC	35 < E < 75	5 < E < 8	46	3	
Flexural strength at 20ºC	> 60	> 10	58	8	
Flexural strain to rupture at 20ºC	> 2500	> 4000	3280	4200	
Dimensional stability*	< 0.05	< 0.25	-0.05	0.45	



Reusable Mold and Parts → Important Cost Reduction

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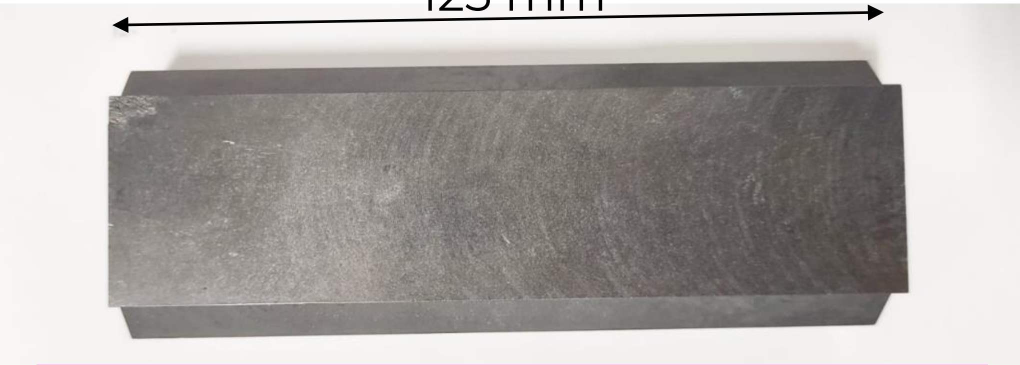
Additional CrGr Characterization

- **Component of complex shape and tolerances** produced, to prove machinability and test UHV behaviour

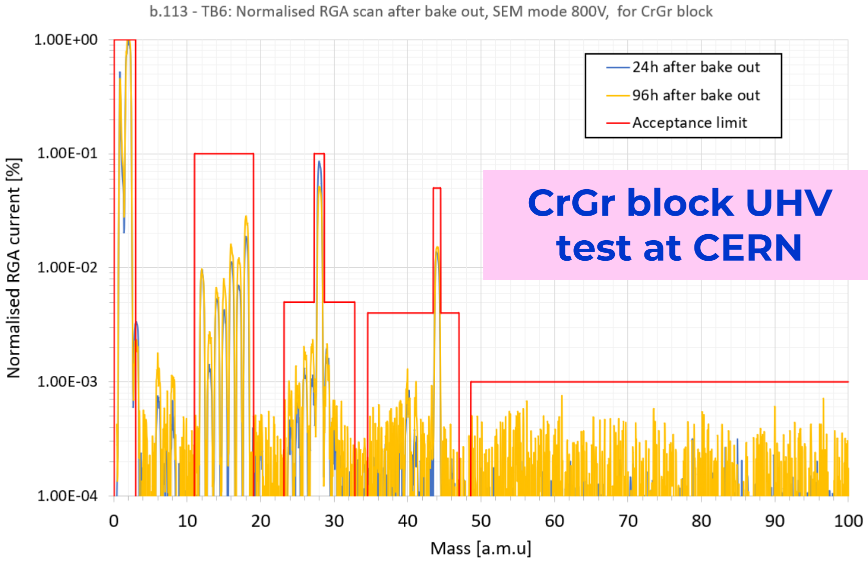
125 mm



CrGr block, machined in the shape of HL-LHC collimator absorber

CrGr block metrology

Tolerance*	Time interval after machining			
	7 days	1 month	2 months	3 months
Flatness of back face [mm]	0.010	0.011	0.011	0.012
Flatness of top face [mm]	0.005	0.011	0.011	0.010
Parallelism of top face wrt back face [mm]	0.022	0.019	0.021	0.038
Position of top face wrt back face [mm]	0.025	0.022	0.021	0.042



CrGr block UHV test at CERN

Material compliant for installation in the LHC!

Courtesy of G. Cattenoz (CERN)

Deliverables and Budget

Milestone/Deliverable Number	Title	Lead beneficiary	Type	Dissemination level	Due Date (in months)
MS14	Evaluation of a CCM alternative to Molybdenum-Graphite	CERN	Report	Public	16
D4.4	Production of large-size CCM plates	CERN	Demonstrator	Public	24

Deliverable D4.4 *due date April 2023, submitted to I.FAST project office 3 weeks ago*

- Produce two large CCM plates (cross section >400 cm^2) in a single sintering cycle
- This means: moving from the sintering of Ø170 mm to Ø230 mm plates → **doubling the cross-section** (and: 2 plates per cycle!)
- Given the good results achieved on CrGr, and the advantages provided over MoGr → **decision to select CrGr as D4.4 baseline material**
- Sintering run: **9th March 2023**

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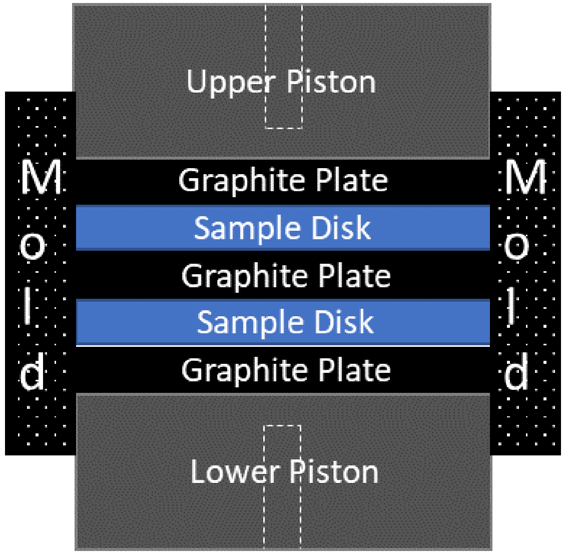
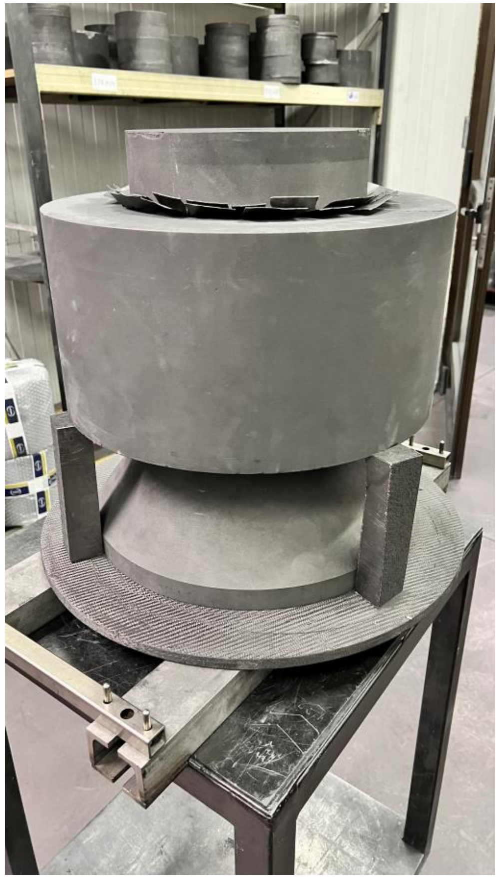
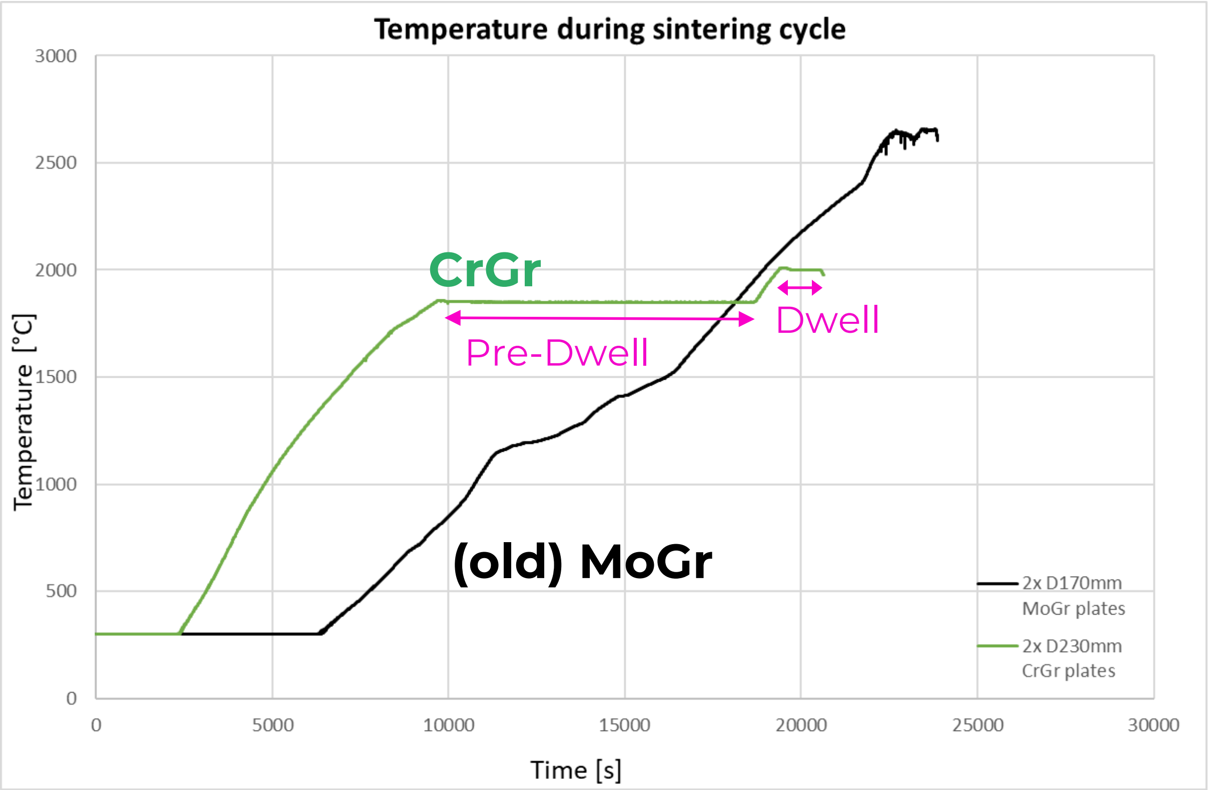
Deliverable D4.4 – CrGr 2xØ230 mm production

- **One single mould** for two Ø230 mm CrGr disks (+ graphite spacers)
- Shorter cycle, lower sintering temperature wrt MoGr





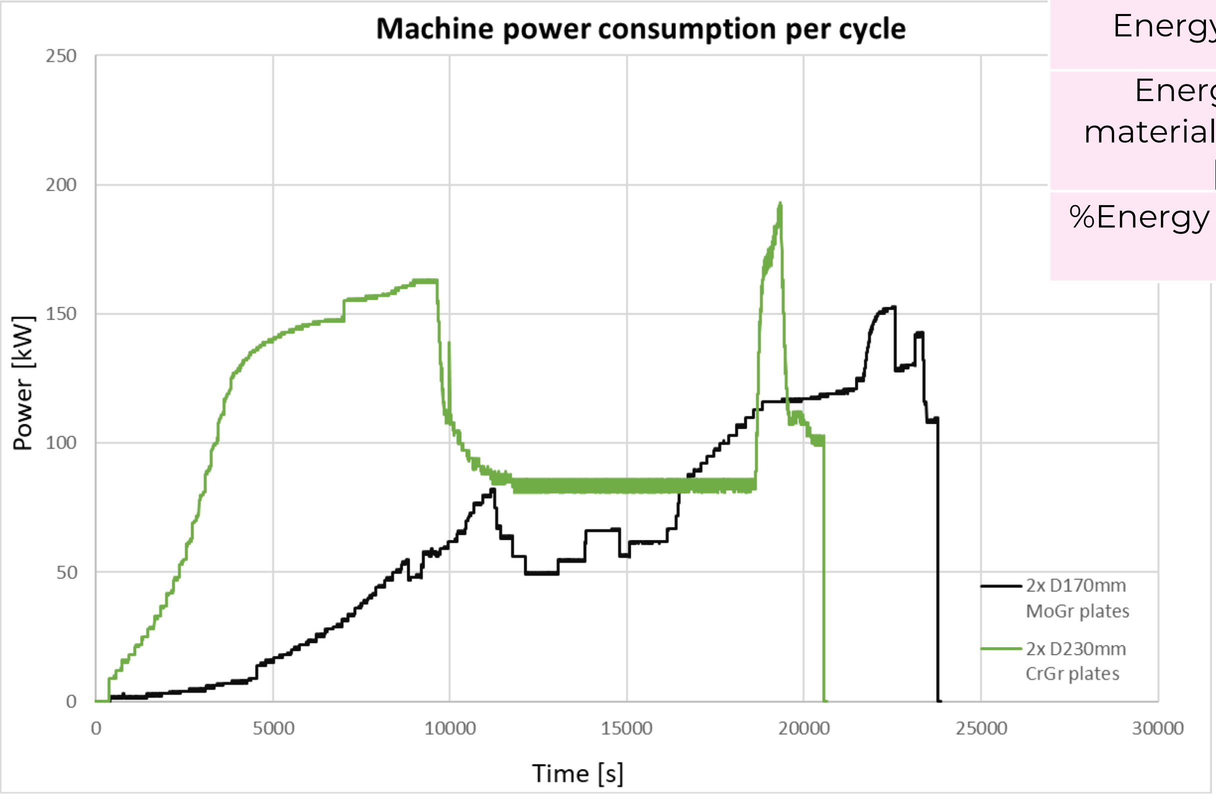


Deliverable D4.4 – CrGr 2xØ230 mm production

- **Energy saving** (similar peak power, shorter time, bigger volume produced)

*estimation

	2x Ø170 MoGr plates	2x Ø230 CrGr plates	2x Ø230 CrGr plates without pre-dwell*
Energy per cycle [kWh]	408	577	214
Energy per cycle per material volume produced [kWh/cm³]	0.36	0.28	0.10
%Energy saving per material volume	***Pre-I.FAST baseline***	**-23%**	**-72%**

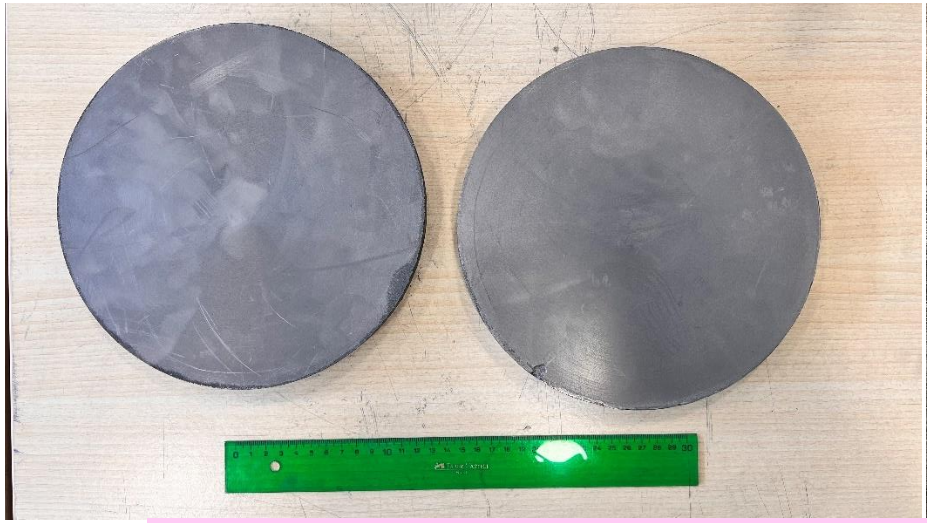


Deliverable D4.4 – CrGr 2xØ230 mm production

- **Mould saving**

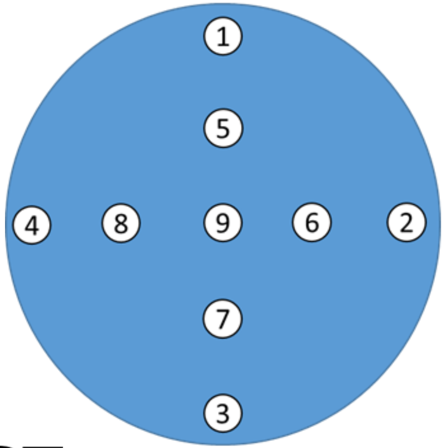
Deliverable D4.4 – CrGr 2xØ230 mm production

- Preliminary characterization at **Nanoker**
- **Very promising** electrical conductivity and compaction (Density)
- **CTE now to be measured at CERN**, to evaluate best annealing cycle (and: can we remove annealing cycle?)



D4.4 2xØ230 mm CrGr plates

Density 2.44 g/cm^3



Conductivity (MS/m)		
Position	Side 1	Side 2
1	1,04	0,74
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4	1,04	0,78
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Average	**1,05**	**0,86**

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Position	Side 1	Side 2
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3	0,94	0,90
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5	0,93	0,91
6	0,93	0,93
7	0,93	0,92
8	0,94	0,91
9	0,92	0,93
Average	**0,94**	**0,91**

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Conclusions and Next Steps

- All milestones and deliverables of I.FAST task 4.4 **have been reached in the first two years of the project**
- However, in the scope of the task objective (reduction of cost of carbon-carbide materials), **several actions are still foreseen in the last two years of I.FAST**:
  - **Complete the in-lab characterization of the CrGr** produced in the scope of deliverable D4.4, and publish the results on an international journal (record achieved in CrGr thermophysical properties and record in a CCM size sintering)
  - Optimize and, if possible, remove production steps related to material **pre-dwell and annealing**
  - Study the machine insulation system to understand if further improvements are needed to **reduce power losses**
  - **Further increase the material volume produced per cycle**: increase the plate thickness (up to 5 cm?) and/or increase the number of plates (up to 4?)
  - Optimize the material composition to **reduce spilling of molten metal**

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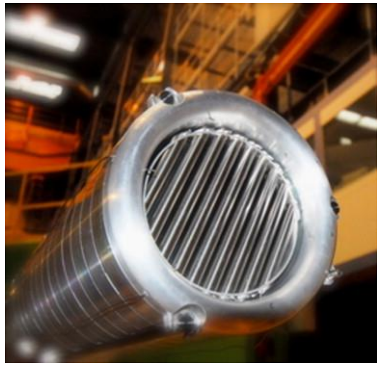
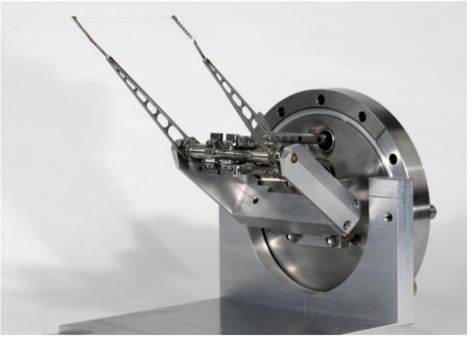
Motivation

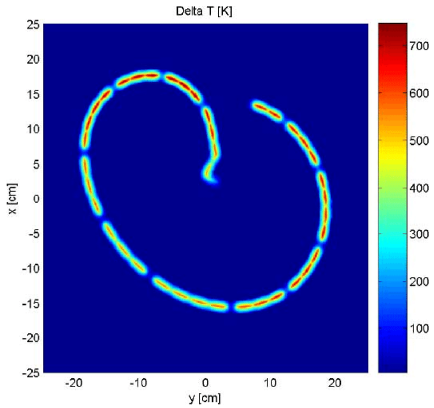
- **Increasing worldwide request for thermal management materials** (high thermal diffusivity and specific heat, low density)
- **Cost still high**: CCM are limited to high-end applications (nuclear energy, particle physics, aerospace, …)
- Decrease of energy consumption, improvement of production cycle **efficiency and sustainability** are also a must
- In particle physics: very interesting for **beam-intercepting devices and beam instrumentation, beam windows, etc.**



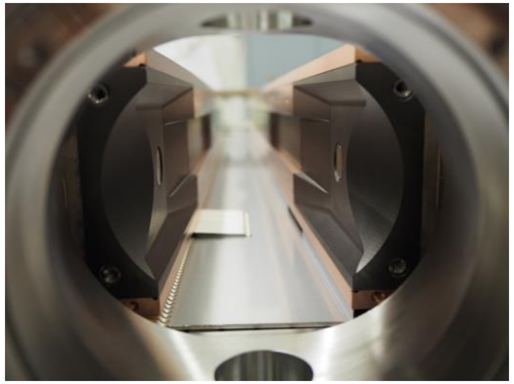
Targets



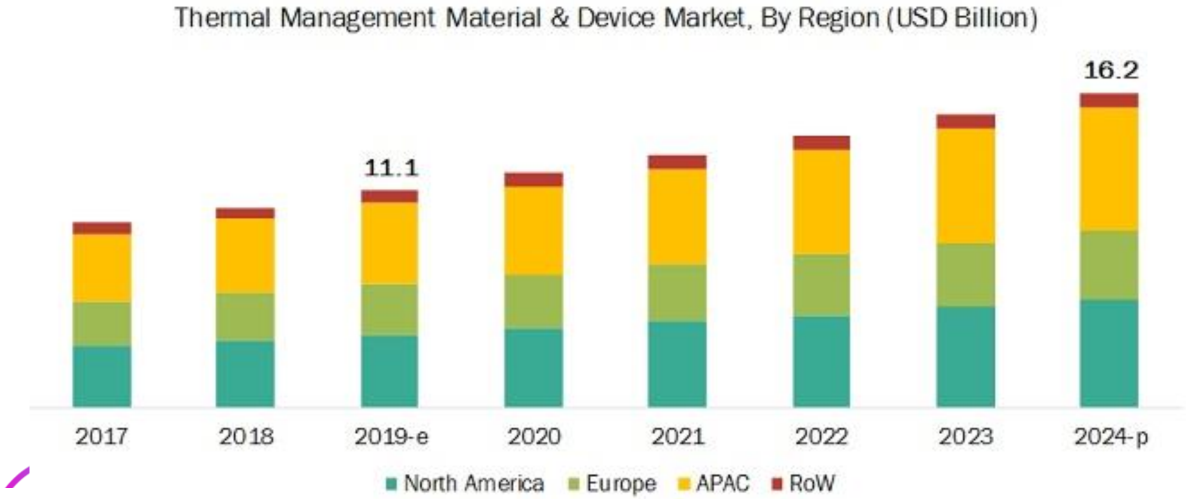
Beam wire scanners



Dumps



Collimators



Expected figures for the development of the thermal management market in the next years, source: https://www.marketsandmarkets.com/Market-Reports/thermal-management-market-155049228.html

Year 1 activities – Technical Specification

- Definition of the **minimum thermophysical properties** for a use in HEP beam-intercepting devices and in thermal-management applications

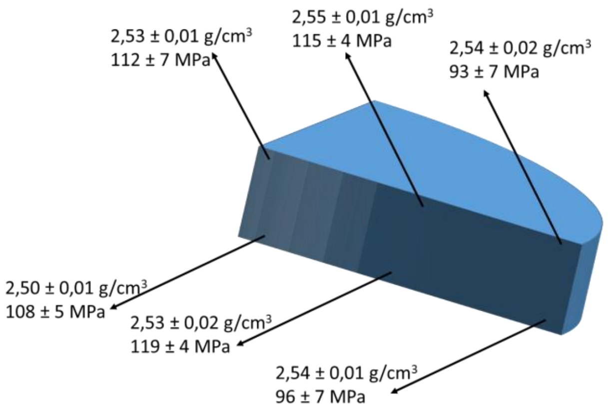
	Specification		
Property	**II***	**Ⱶ**	**Unit**
Density at 20ºC	2.40 – 2.60		[g/cm³]
Specific heat at 20ºC	> 0.6		[J/(g·K)]
Electrical conductivity at 20ºC	> 0.75		[MS/m]
Thermal Diffusivity 20ºC /at 300ºC	> 350/100	> 20/6	[mm^2/s]
Thermal conductivity at 20ºC /at 300ºC	> 500/280	> 35/20	[W/(m·K)]
Volumetric CTE 20-1000ºC	< 7		$[10^{-6}K^{-1}]$
Coefficient of thermal expansion 20-1000ºC	< 2.9	< 15	$[10^{-6}K^{-1}]$
Young's Modulus at 20ºC	35 < E < 75	5 < E < 8	[GPa]
Flexural strength at 20ºC	> 60	> 10	[MPa]
Flexural strain to rupture at 20ºC	> 2500	> 4000	[µm/m]
Dimensional stability*	< 0.05	< 0.25	%

**The dimensional stability shall be ensured after the following thermal cycle: heating of the specimen up to 1950ºC with a ramp of 5ºC/min. Cooling of the specimen down to room temperature with the same ramp.*

Year 1 – Increase of volume per cycle

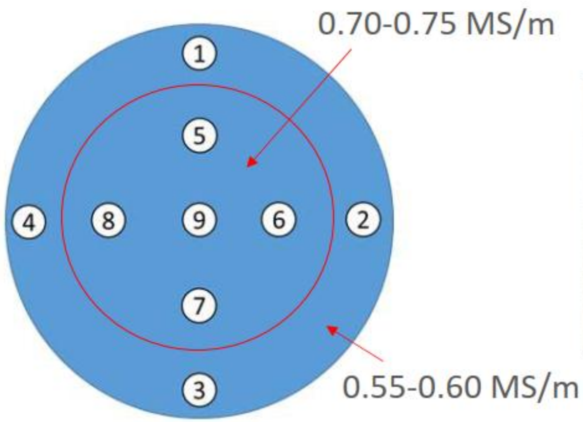
- **Molybdenum-Graphite** (sintered at 2640°C)

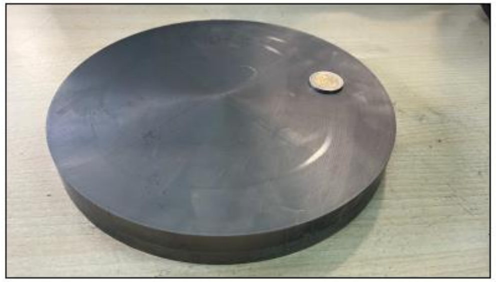
2 plates produced with 230 mm Diameter (2x bigger section than before IFAST)



Disk (230 mm diameter)	Density (g/cm³)	Electrical Conductivity (Mean values on each side) (MS/m)
Plate #1 (p=26 MPa)	2,53	0,6 – 0,63
Plate #2 (p=40 MPa)	2,60	0,65 – 0,68
Specification	2,3 ÷2,6	>0,8

Lower electrical conductivity values than in the 170 mm diameter disks





Pre-compaction of the green powder:

Maximum Applied Force Uniaxial Hydraulic Press ~ 900 kN

- 170 mm Ø → 40 MPa → 2,00 g/cm³
- 230 mm Ø → 21 MPa → 1,65 g/cm³

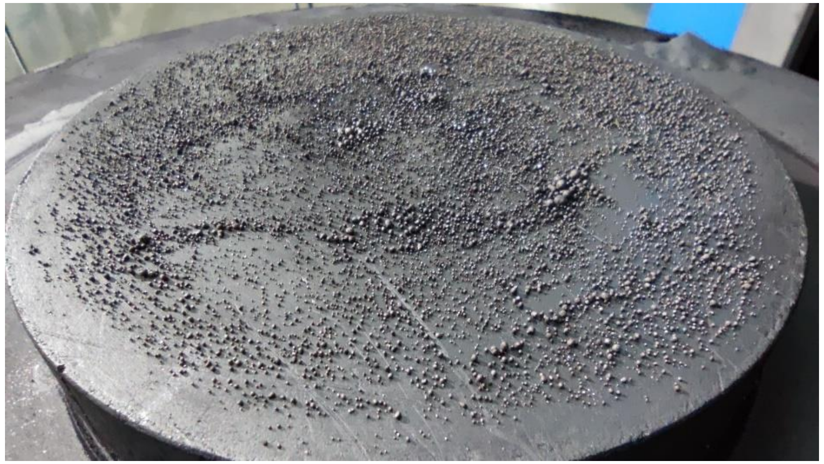
Next: increase the metal content, together with the higher sintering pressure

Year 1 – Decrease of sintering Temperature

- **Chromium-Graphite** (sintered at 2000°C – 1.3x lower T)
- Concept proposed by **Jorge Guardia** within ARIES WP14 & WP17, technically was not demonstrated yet (very poor mechanical properties)

3 plates produced with 170 mm Diameter

Disk (170 mm diameter)	Density (g/cm^3)	Electrical Conductivity (MS/m)
Plate #1	2,30	1,00 – 1,07
Plates #2 & #3	2,30	0.75/0.81
Specification	2,3 ÷2,6	>0,8



Reusable Mold and Parts → Important Cost Reduction

- **Plate #1 produced in a single plate per cycle**, very promising properties, decision for full characterization at CERN
- **Plates #2 and #3 double-plate per cycle**, losing a bit in conductivity → composition and cycle to be optimized

iFAST

Year 1 – CrGr Characterization