

Synthesis of Refractory Metals Fluorides for Chemical Vapor Deposition: Applications to Rotating Anodes For Medicals X-Ray Tubes

X-ray Medical Imaging

X-ray tube focal spot

Meteor entry
Rocket nozzle interior
Fission reactor core
Brain surgery

100-500
3300-4200
10
1-2

W/mm²

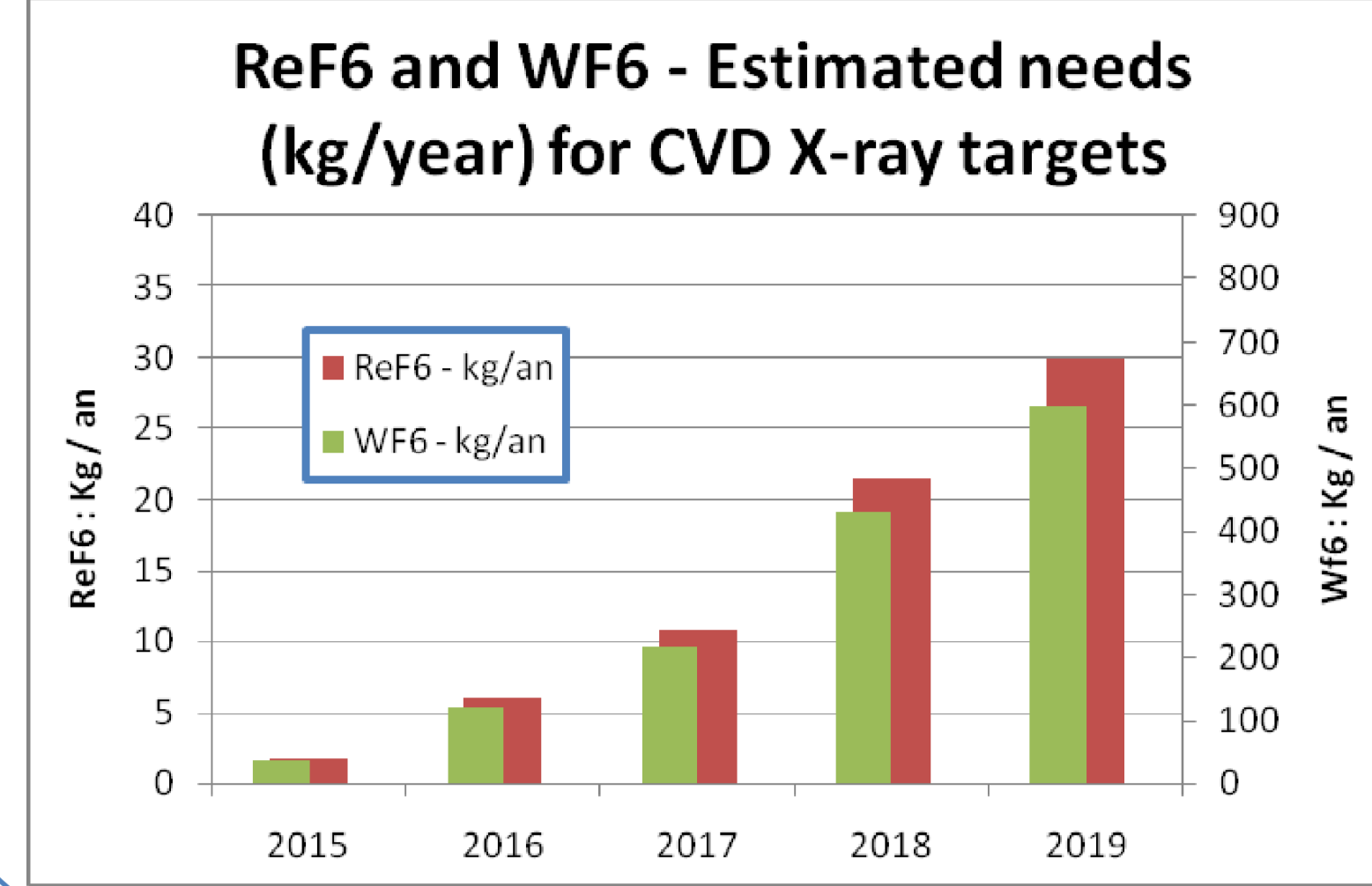
Philips iMRC CT

Spot temperature ~3000°C
Trace temp. ~2000°C
Bulk Temp. ~1000°C
High Rotary Speed : 10 000rpm

Refractory and High Strength Materials :

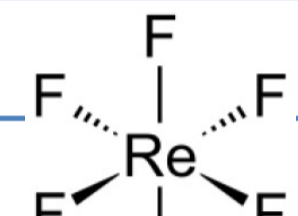
- W, Re, Mo,
- Graphite and Carbon Fibers Composite

- ACERDE: founded in 2006 and based in Savoie, France, near Chambéry, 12 high skilled employees
- Main activities are develop light and chip X-ray targets and rework costly « on the market » graphite brazed targets
- Annual consumption of Fluorides gases :
- Objective: to be self-dependant on ReF₆ supply according our specifications



- Bare Substrate
 - Substrate with CVD coating
 - Coating after grinding
-

ReF₆ Synthesis



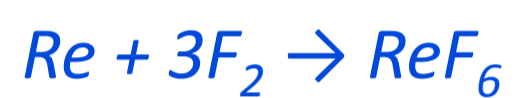
ReF₆ Purity

Element	Unit	Lower Limit	Upper Limit	Element	Unit	Lower Limit	Upper Limit
Re	%	99.90	none	Mn	ppm	none	10
O	ppm	none	3500	Mo	ppm	none	10
N	ppm	none	200	Na	ppm	none	100
H	ppm	none	200	Ni	ppm	none	10
Al	ppm	none	10	P	ppm	none	10
B	ppm	none	10	Pb	ppm	none	10
Cd	ppm	none	10	S	ppm	none	10
Ca	ppm	none	100	Si	ppm	none	10
Cr	ppm	none	10	Ti	ppm	none	20
Cu	ppm	none	10	V	ppm	none	10
Fe	ppm	none	20	W	ppm	none	20
K	ppm	none	100	Zn	ppm	none	10
Mg	ppm	none	10	Zr	ppm	none	10

ReF₆ Specifications

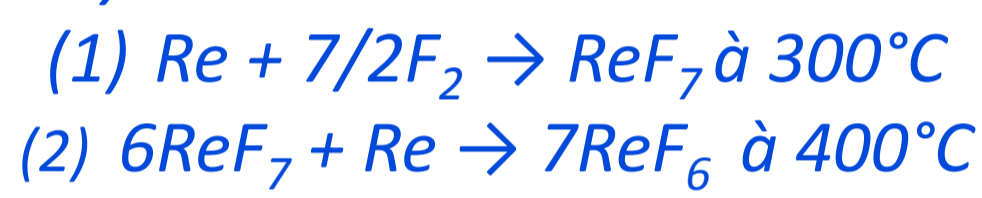
Molar Mass	300,20 g/mol [1]
Appearance	Yellow Crystals [1]
Fusion Point	18,5°C [1]
Boiling Point	33,7°C [1]
Density	4,94 g/mL [2]

•Direct synthesis :



Yield <80% [3]

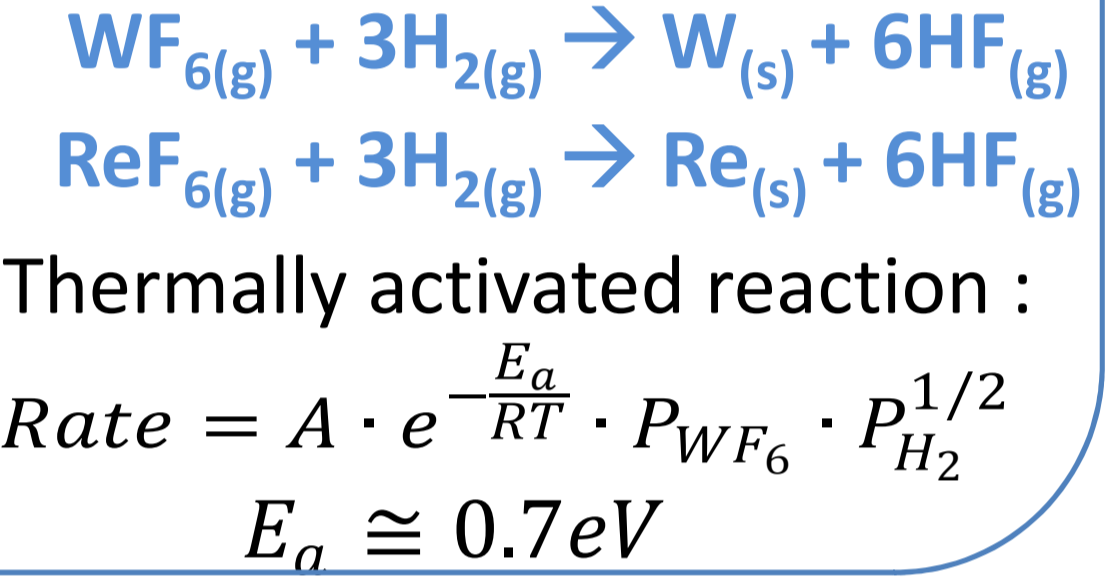
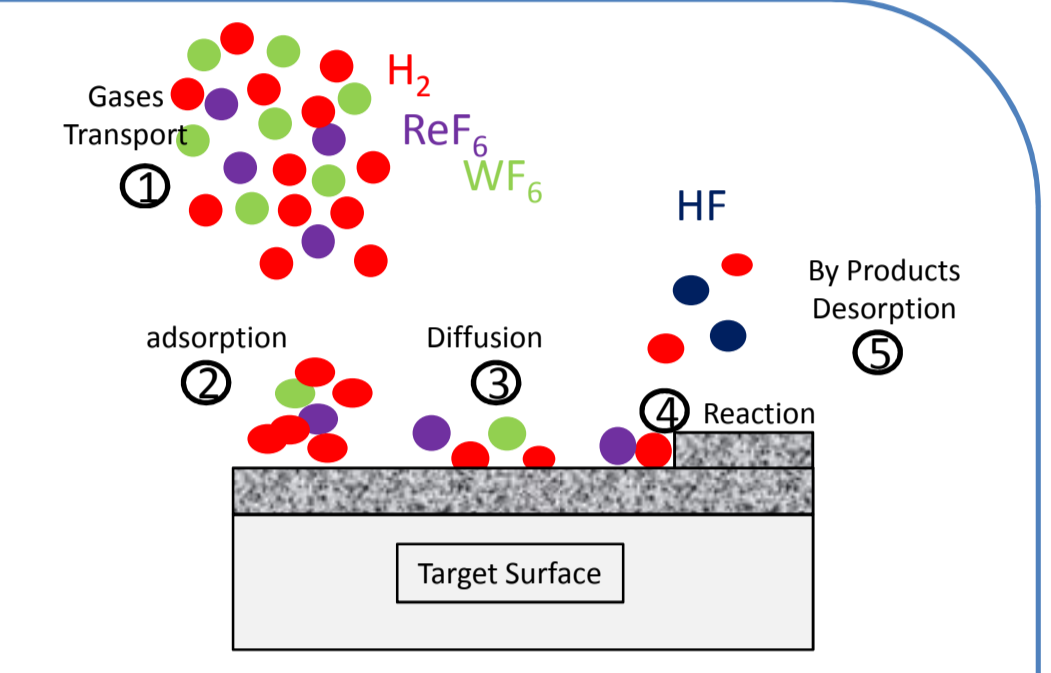
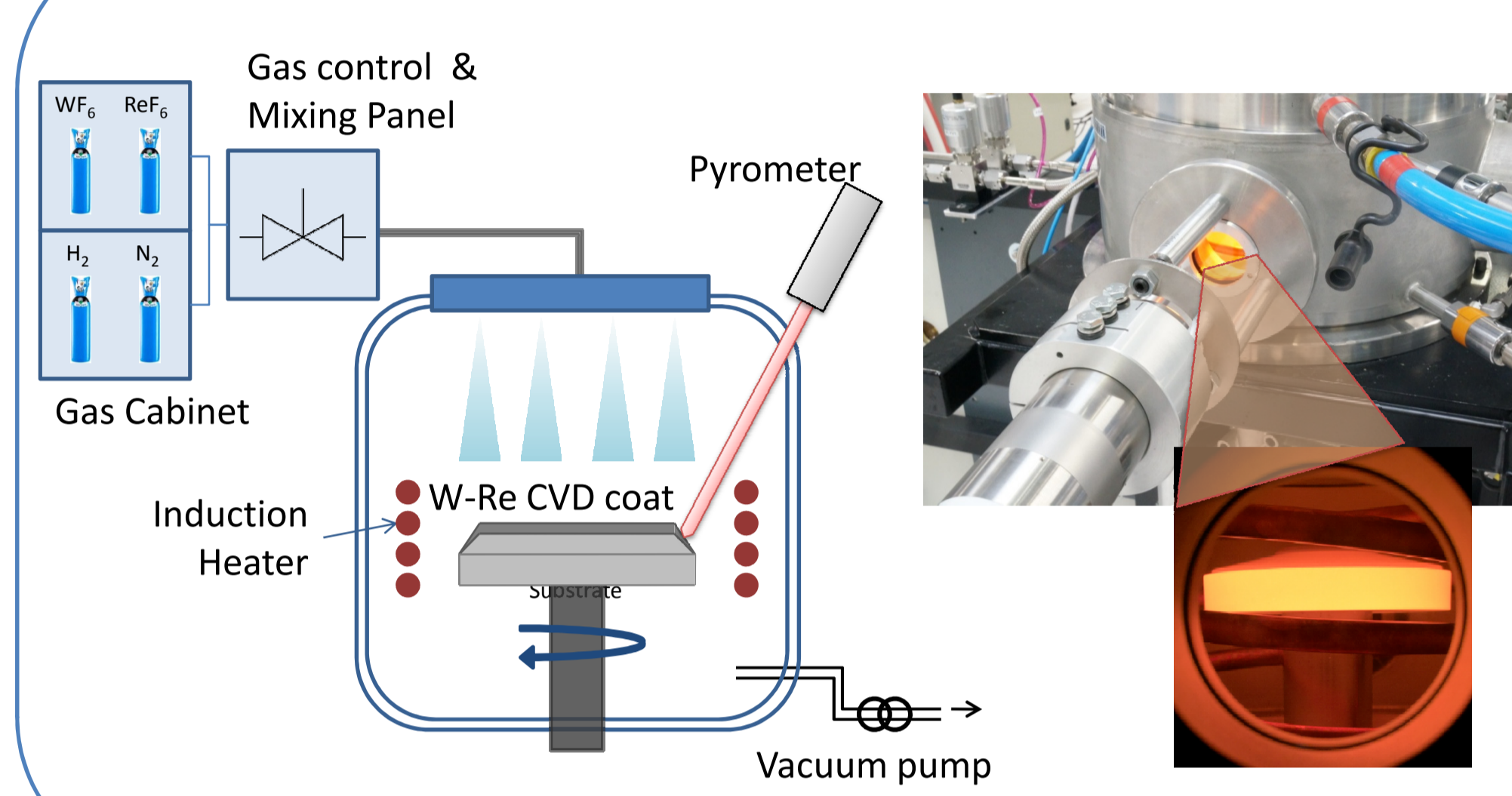
•2 steps synthesis :



Yield ~100% [3]

[1] CRC Handbook of Chemistry and Physics, 90th Edition, CRC Press, Boca Raton, Florida, 2009, ISBN 978-1-4200-9084-0, Section 4, Physical Constants of Inorganic Compounds, p. 4-85.
[2] T. Dreves, J. Supel, A. Hagenbach, K. Seppelt: "Solid State Molecular Structures of Transition Metal Hexafluorides", in: *Inorganic Chemistry*, 2006, 45 (9), S. 3782-3788, doi:10.1021/ci052029f, PMID: 16834814.
[3] V. Vybyvanets, AV Kouzulin, AV Cherenkov and GS Shilkin, REES2015, IOP Conf Series Materials Science and Engineering 112, (2016) 012041.

W-Re CVD Coating



ReF₆ Reactor

- Reactor designed and assembly by Acerde i.e. equipment usage without IP restrictions
- All developments (machine, software, recipe,...) are under Acerde's IP
- F₂ and F₂/N₂ delivery system (Solvay)
- Monel made cylindrical Furnace with temperature regulation up to 600°C and pressure up to 2.4bars
- Products trap with temperature regulation from -180°C to +120°C for both trapping Fluorides and ReF₆ purification
- Solid trap and LN2 trap to eliminate exhausted fluoride compounds
- 1kg of Re powder consumption by run
- In situ IR spectrometer for gas analysis
- All parameters are monitored trough a OPTO22 controller with a PC display
- Customable automatic recipe

PC display

Schematic of the reactor

IR Spectrometer

W-Re Layer

- The tungsten coating is deposited in several successive layers with rhenium interlayers.
- Between each W-layer, the system is cooled to release thermal stresses. (Patents FR2962591 and FR1451695)
- The interface between each layer will stop to the cracks propagation
- Rhenium-multilayer system is a barrier against carbon thermal diffusion for high temperature applications
- Re interlayer provides diffusion bonding of W-Re CVD layer
- Accomplished during existing outgassing process
- Patent US-2015-0248988-A1

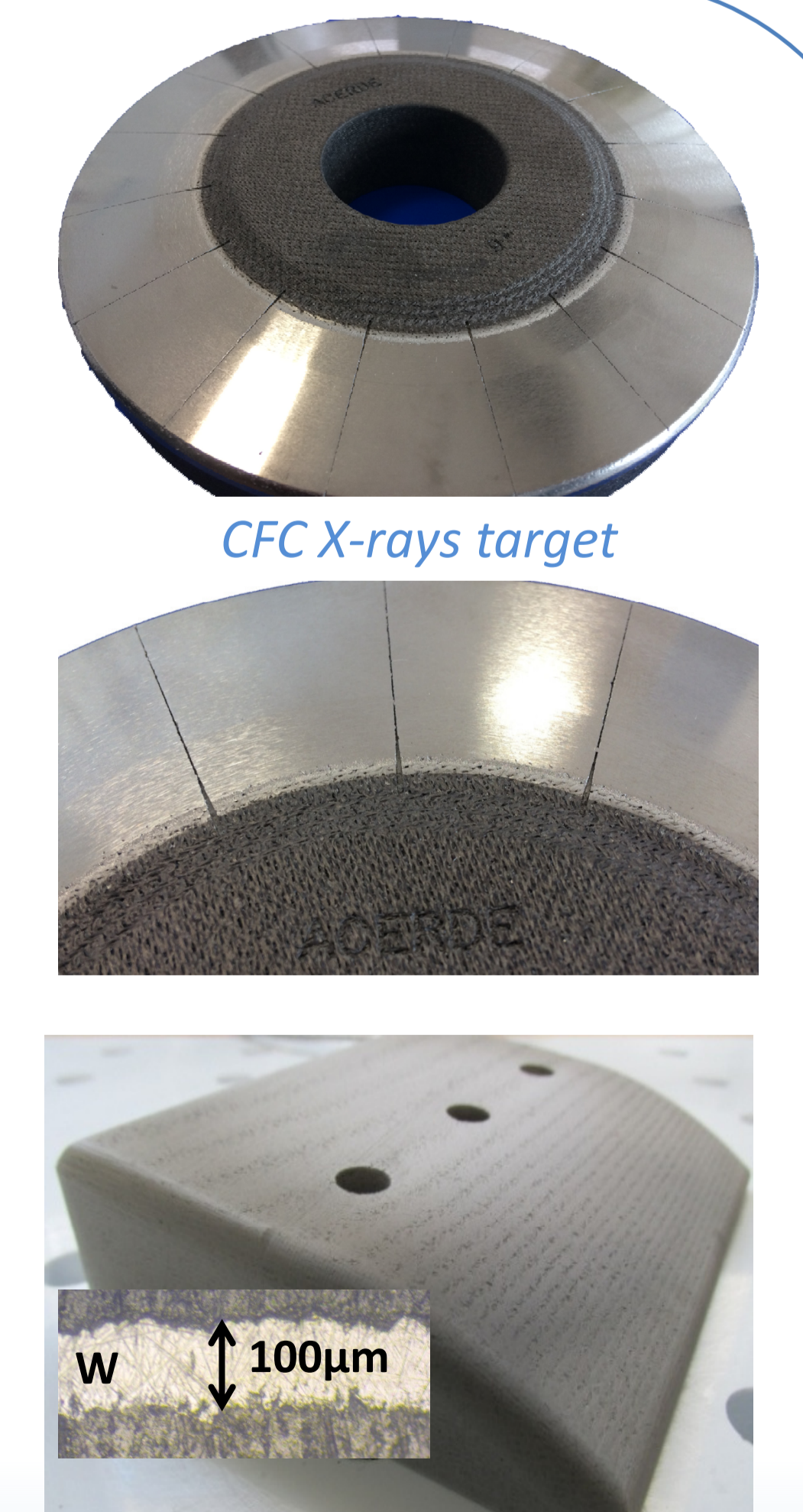
Acerde Products Portfolio

- Reclaimed TZM and Graphite brazed target
- CVD graphite Target :
- CVD Carbon Fiber Composite Target
- Special Parts



Reclaim and graphite X-rays targets

ACERDE also realized W-deposition experiments on CFC bricks for WEST Tokamak first wall



CFC brick with W-coating for first wall

Conclusion

- Acerde has designed and built his own Fluorination equipment to supply his own demand of ReF₆
- ReF₆ production capability has to be 1kg/month
- ReF₆ is used for CVD
- Acerde's CVD reactor has been develop for special Applications like X-ray targets
- Actual anode capacity is 1000pcs/year and We plan to multiply by 2 the capacity during the next Year
- Possibility to use the reactor for other metals fluorination like Mo or Ir, that can have industrial applications with CVD

ACERDE SAS

Bâtiment CLEANSPACE
354 Voie Magellan
Parc d'Activités ALPESPACE
FR-73800 STE HELENE DU LAC



Contact : P.O. ROBERT
Standard : +33 (0)4 79 26 48 27
Fax : +33 (0)4 26 23 67 38
E-Mail : porobert@acerde.com
Web : <http://www.acerde.com>

