Ultrahigh temperature ceramics for thermal protection systems and propulsion

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- Ing. Cantoni, CIRA.
- ISTEC staff: A. Bellosi, F. Monteverde, L. Zoli, L. Silvestroni, V. Medri
- Prof. Savino, Department of Industrial Engineering (DII) – Aerospace section, University of Naples "Federico II", for tests on TPS and rocket nozzles.

Funding

- AFOSR research grant FA8655-12-1-3004 (Contract monitor Dr. Sayir) for short fiber-UHTCs composites.
- MoD (PNRM) cofunding activity on UHTCs for propulsion applications (SMARP – Sviluppo di MAteriali ceramici ultraResistenti all'ablazione per applicazioni nella Propulsione)
- CIRA has funded ISTEC research on UHTCs through several programs and projects

Outline

- Introduction
- Short fibers - ZrB_2 composites for TPS
- SiC/C long fibers ZrB_2 composites for TPS
- Development of ultra-ablation resistant ceramics for application in the propulsion - SMARP
- Conclusions

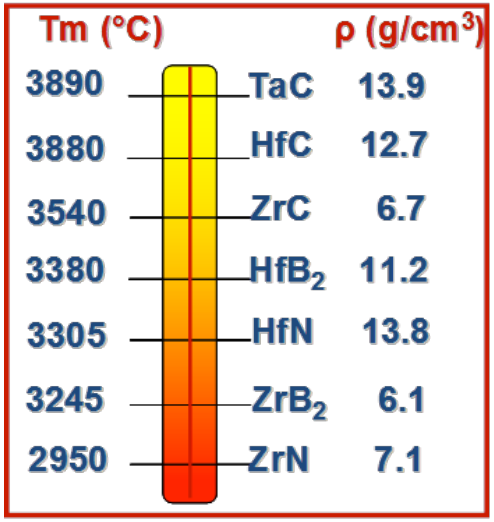
Ultra High Temperature Ceramics

Potential materials for use in extreme environments such as:

- scramjet engine components, leading edges, nosecones for hypersonic vehicles;
- Rockets nozzles
- cladding materials in generation IV nuclear reactors;

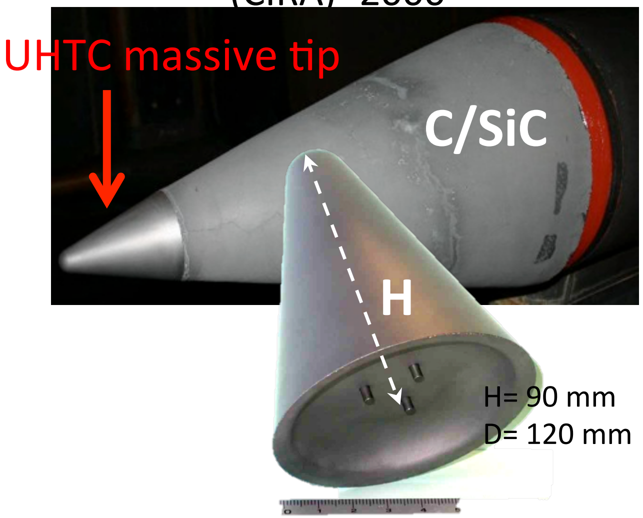
Critical challenges:

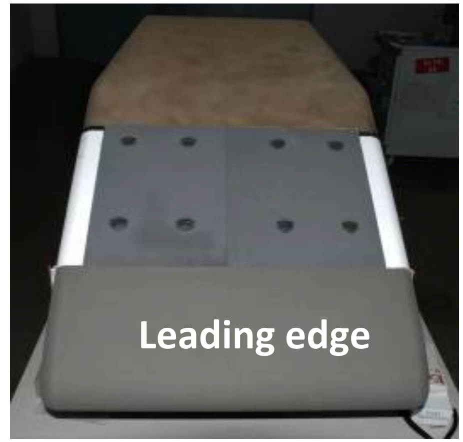
- **thermal shock resistance**
- **damage tolerance**



ISTEC – CIRA long term collaboration

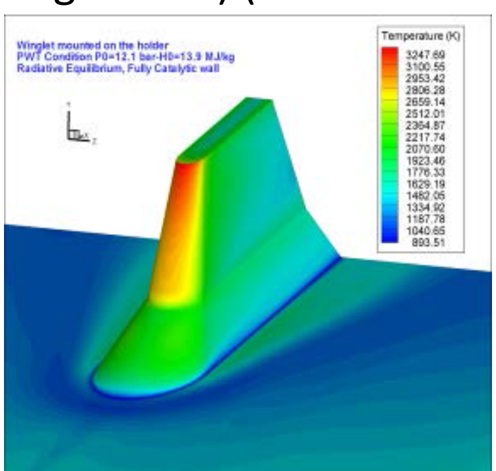
Sharp Hot Structures (CIRA)- 2000





Advanced Structural Assembly - Phase B (Thales Alenia Space) 2004-2010

UHTC Winglet in *EXPERT* (ESA Programme) (2006-on hold)



SHARK ESA project (2010)

Most of test articles in monolithic UHTCs suffered from a dramatic failure!

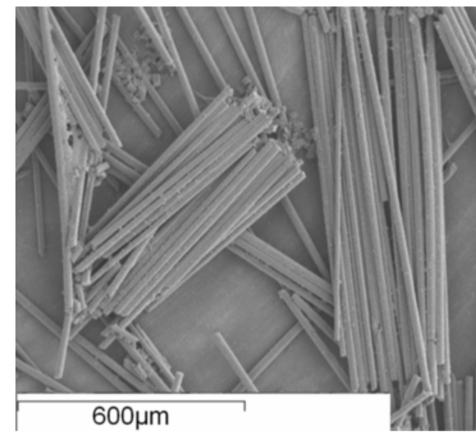
Short fibers-reinforced UHTCs

- Easy approach to increase the fracture toughness (SiC particles → SiC fibers)
- Same processing as conventional powders

SiC fibers:

✓ Hi-Nicalon, Tyranno SA3….

C fibers



UHTC powder, s.a | Short fibers

↓

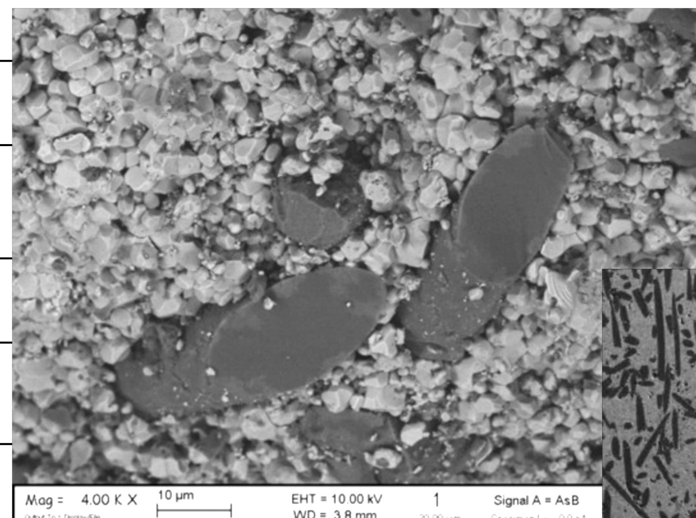
Ball milling & mixing

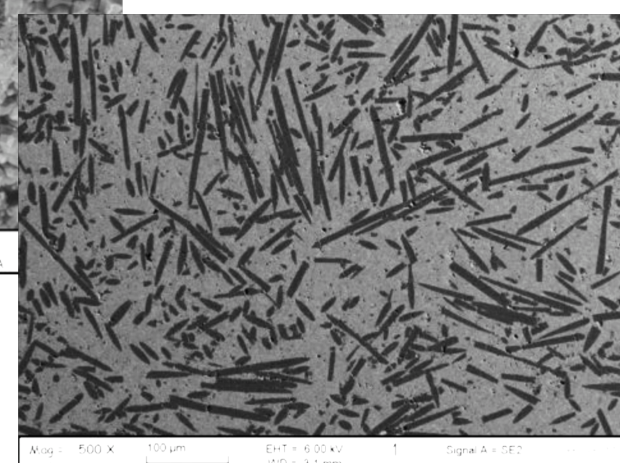
↓

Drying and crushing

↓

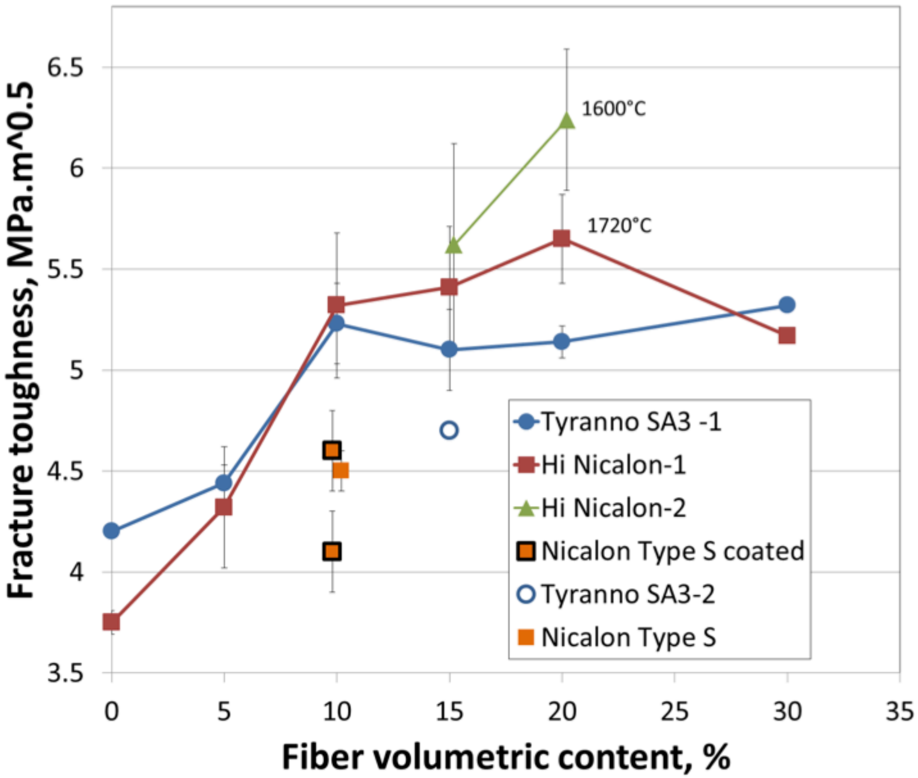
Hot Pressing

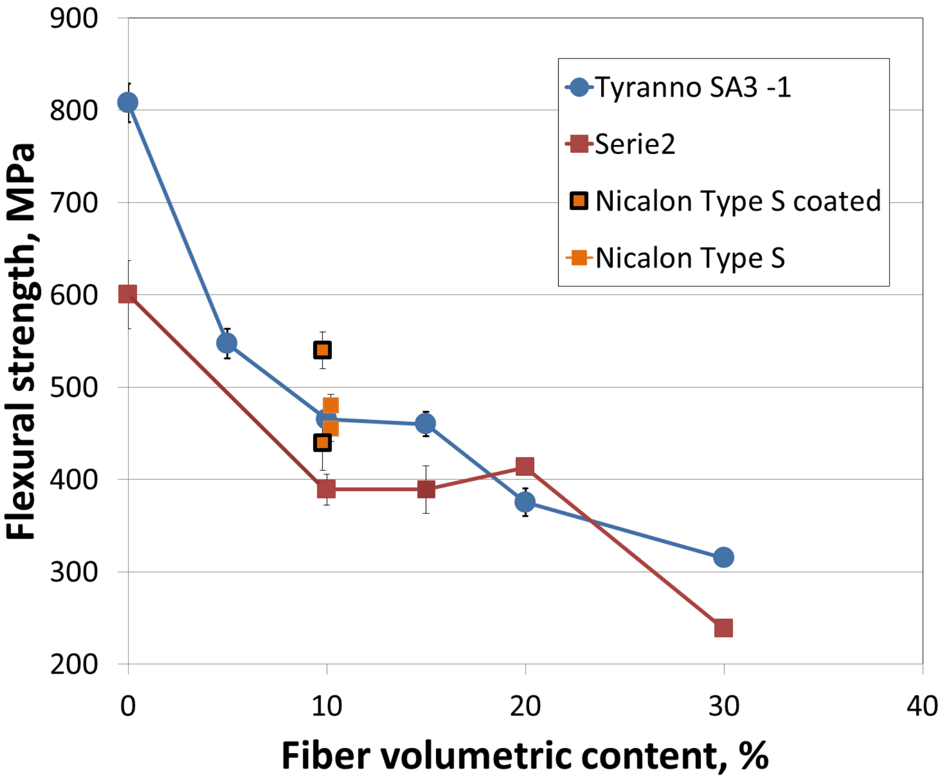




L. Silvestroni et al, JECS 30, 2155-2164 (2010)

Toughness vs Strength (SiC fibers)





-Highest values: 20% Hi Nicalon uncoated fiber, densification 1600°C
-Tyranno lower than Hi nicalon
-Type S < Hi Nicalon, Tyranno

-Strength: decreases almost linearly
-Highest values for Tyranno
-Type S (coated/uncoated) similar to Tyranno

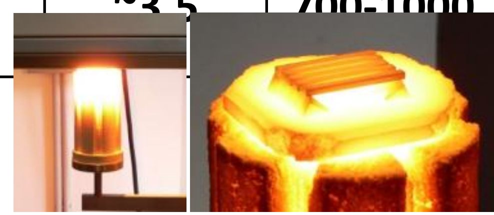
Boride –SiC_{fiber} vs Boride –$SiC_{particle}$

Sample	Sintering Temperature, °C	Density g/cm³	K_{Ic} MPam$^{1/2}$	σ_{RT} MPa	σ_{1200} MPa	σ_{1500} MPa	TSR K
ZrB_2-20SiCf	1700	5.3	5.5-6.5	400-500	300-400	200-300	450
ZrB_2-20SiCp	1900	5.3	~3.5	700-1000		200-50	385

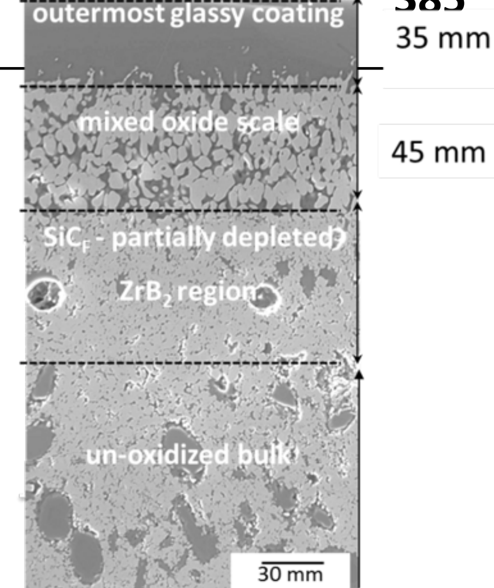
ZrB_2-SiC particles have very high strength even at 1500°C

BUT

Low damage tolerance causes failure before high temperature regimes are reached

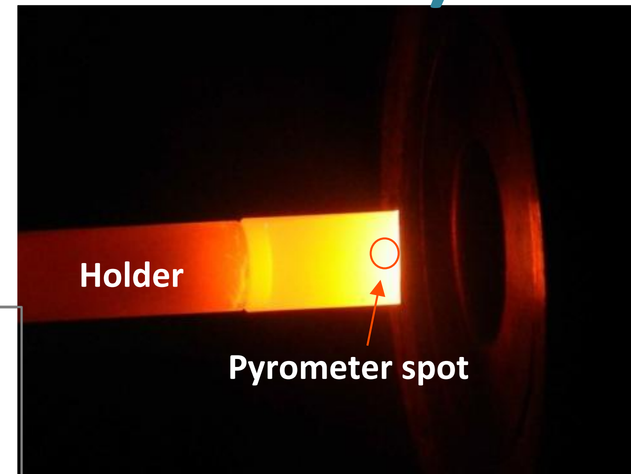


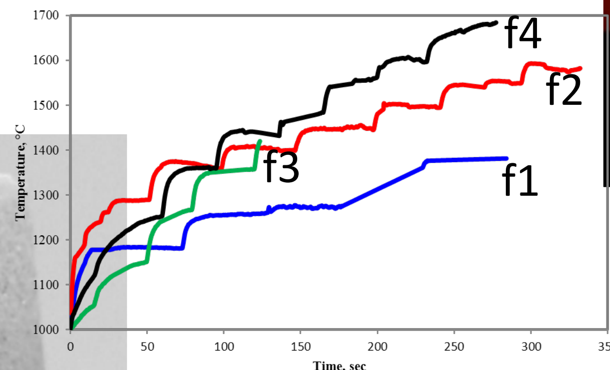
Bottom-up loading furnace



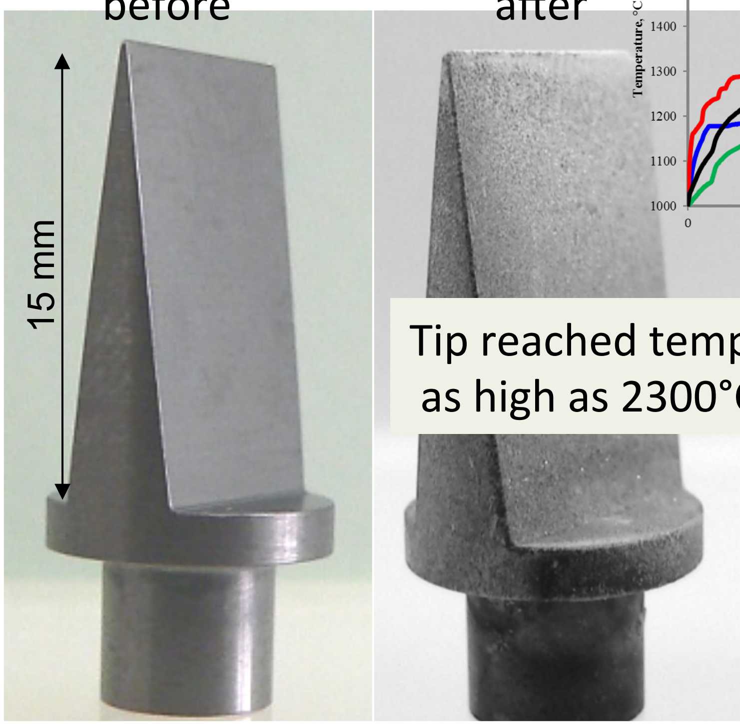
Arc Jet Tests (in collaboration with DII)

- M ≈ 3 (supersonic regime)
- 1 g/s of 80%N_2+ 20% O_2
- static pressure in the chamber ≈ 200 Pa
- specific total enthalpy 8-16.4 MJ/kg
- maximum stagnation point pressure 6-12 kPa
- 2 colour pyrometer + IR camera





before after

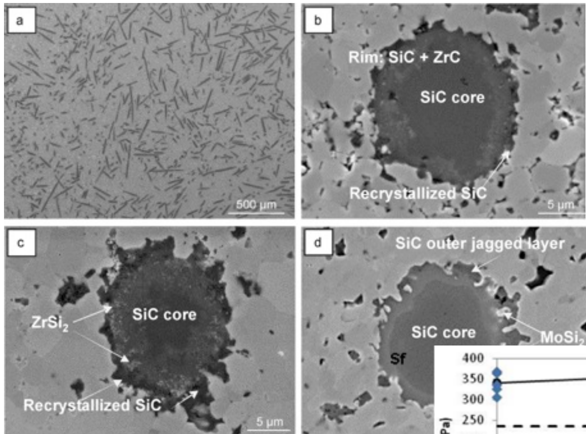


Tip reached temperatures as high as 2300°C

Test	$H0_{max}$ (MJ/kg)	time (sec)	T_{max} (°C)	$\varepsilon_{1\ \mu m}$	Tot. time
f1	13.8	285	1380	0.88	16’ 45’’
f2	17.0	330	1590	0.86	
f3	12.3	120	1395	0.65	
f4	17.0	270	1680	0.54	

The wedge survived the 4 tests!

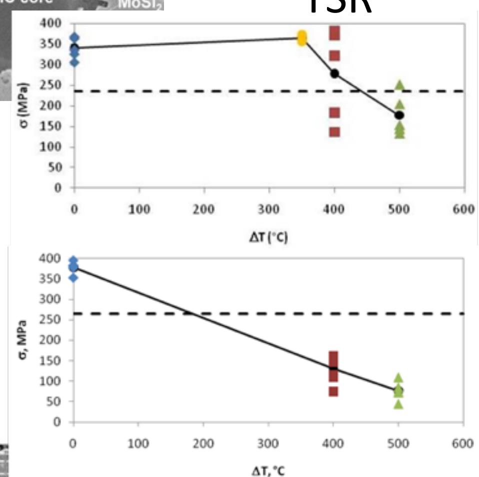
ISTEC/CIRA 2010-2013 collaboration



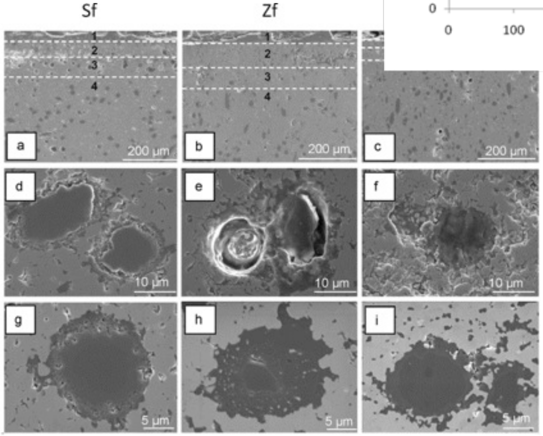
Extensive characterization campaign of short fibers-ZrB_2 composites

Temperature °C	Thermal Diffusivity mm²/s	Specific Heat J/(gK)	Thermal Conductivity W/(mK)
18	28.624	0.426	65.657
599	15.950	0.692	59.391
900	14.493	0.701	54.644
1198	13.403	0.745	53.773
1500	12.544	0.748*	50.515

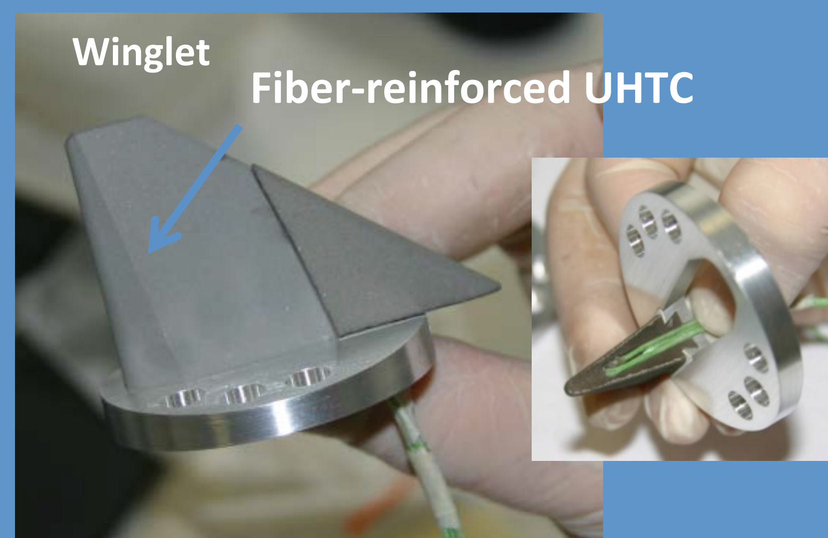
TSR



Oxidation tests



SCRAMSPACE project 2013



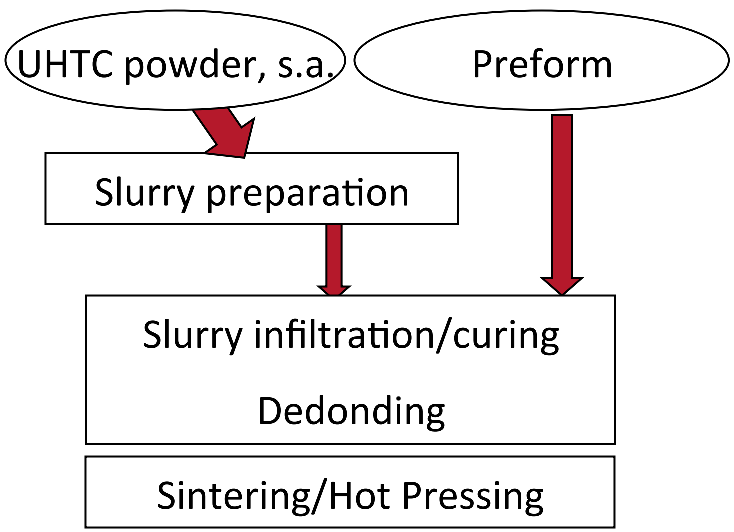
Long fiber reinforced UHTCs (ZrB_2)

-Simple preforms: tows or 1D preforms
-SiC or C fibers
-Slurry infiltration & sintering

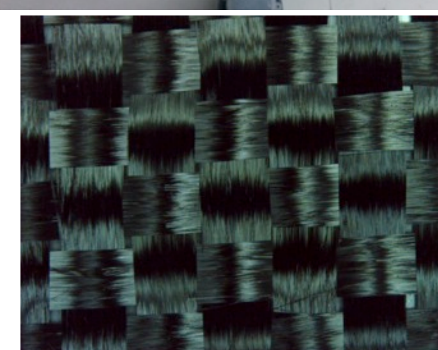
Simple arrays of fibers

GOALS

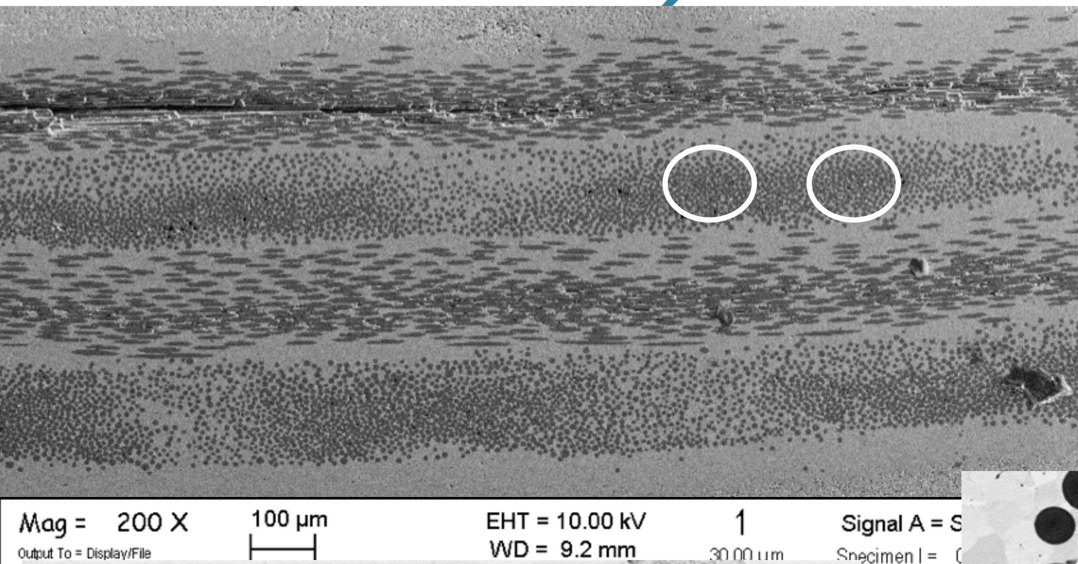
- Increase the fiber volumetric amount >40%
- Non-brittle behaviour

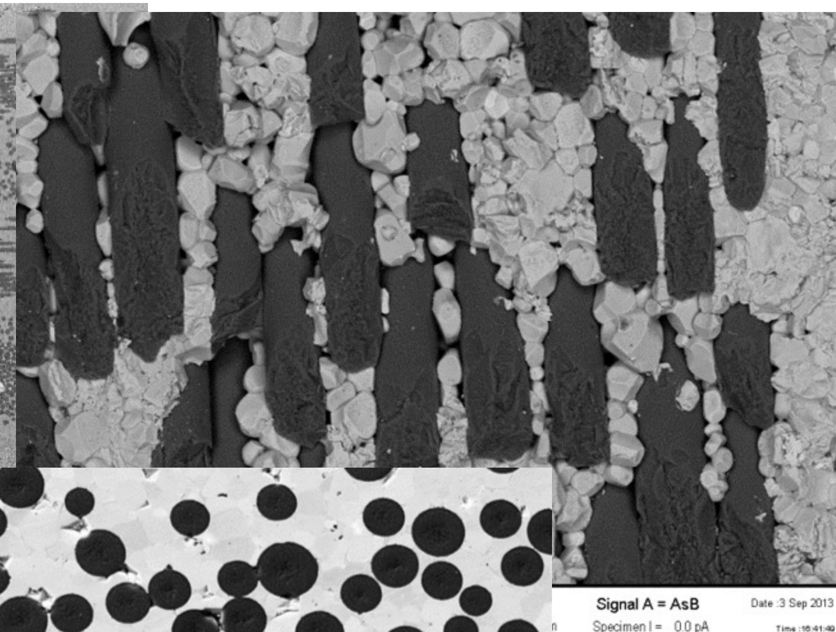


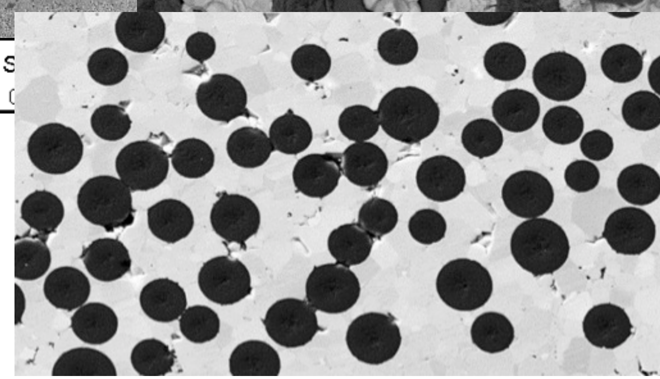
SiC plain weave

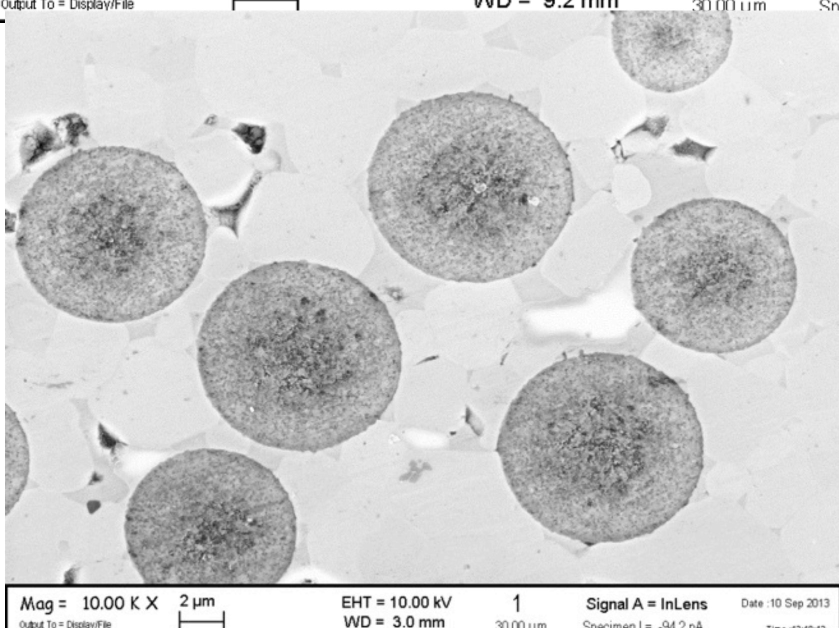


ZrB_2 – SiC 1D textiles





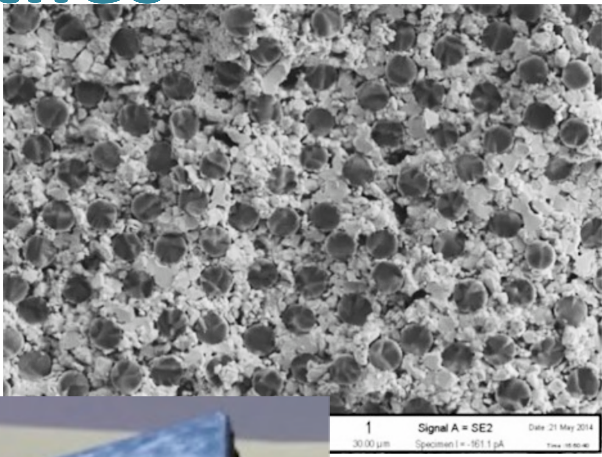


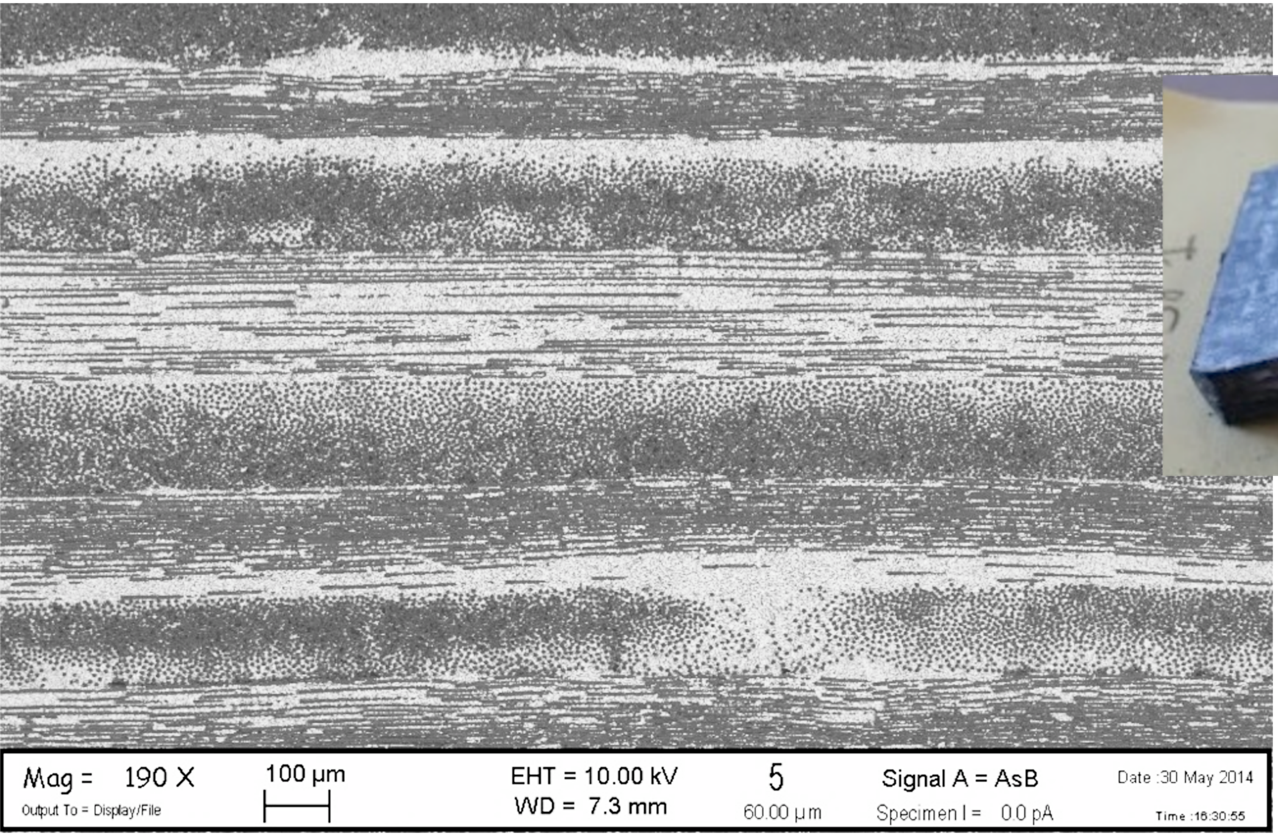


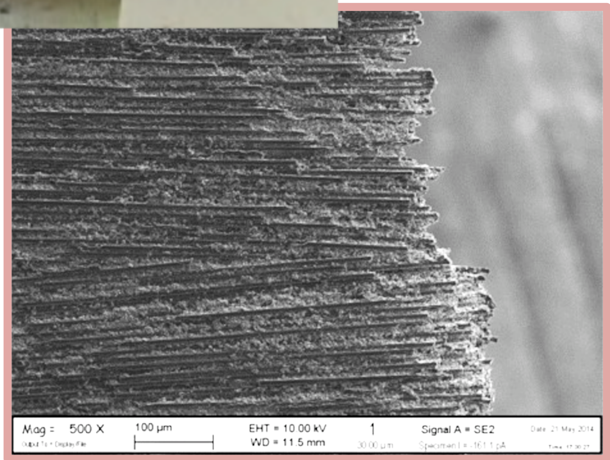
Overall: fiber vol. amount is 40%
Maximum density is 60-70%
Fracture: fiber surface is very smooth
Matrix fully dense
Problems: cracks

ZrB_2 - 1D carbon textiles

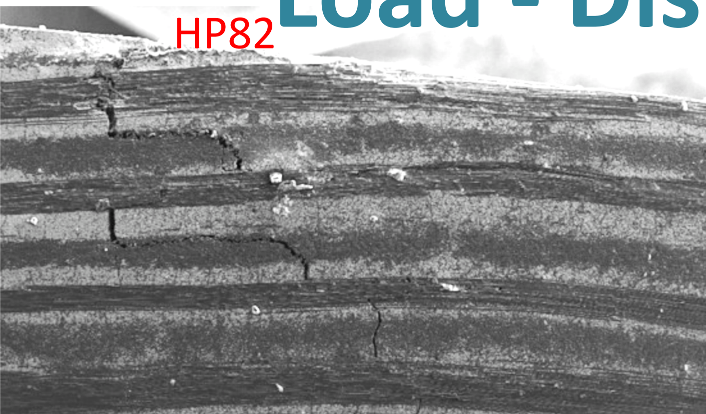
Density	~2.3 g/cm³
Fiber	~75 vol%
Relative Density	80-85%

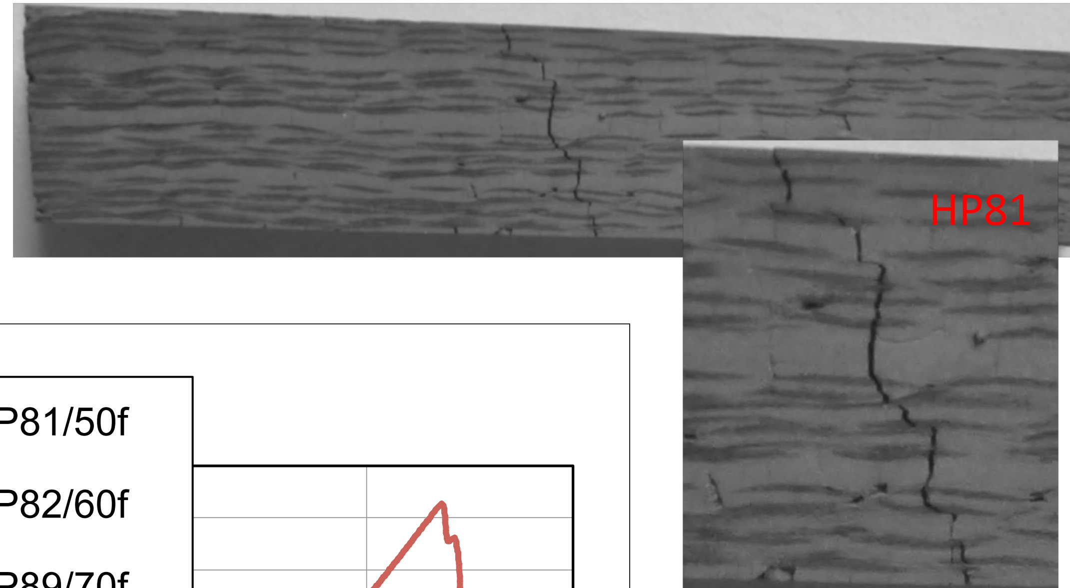


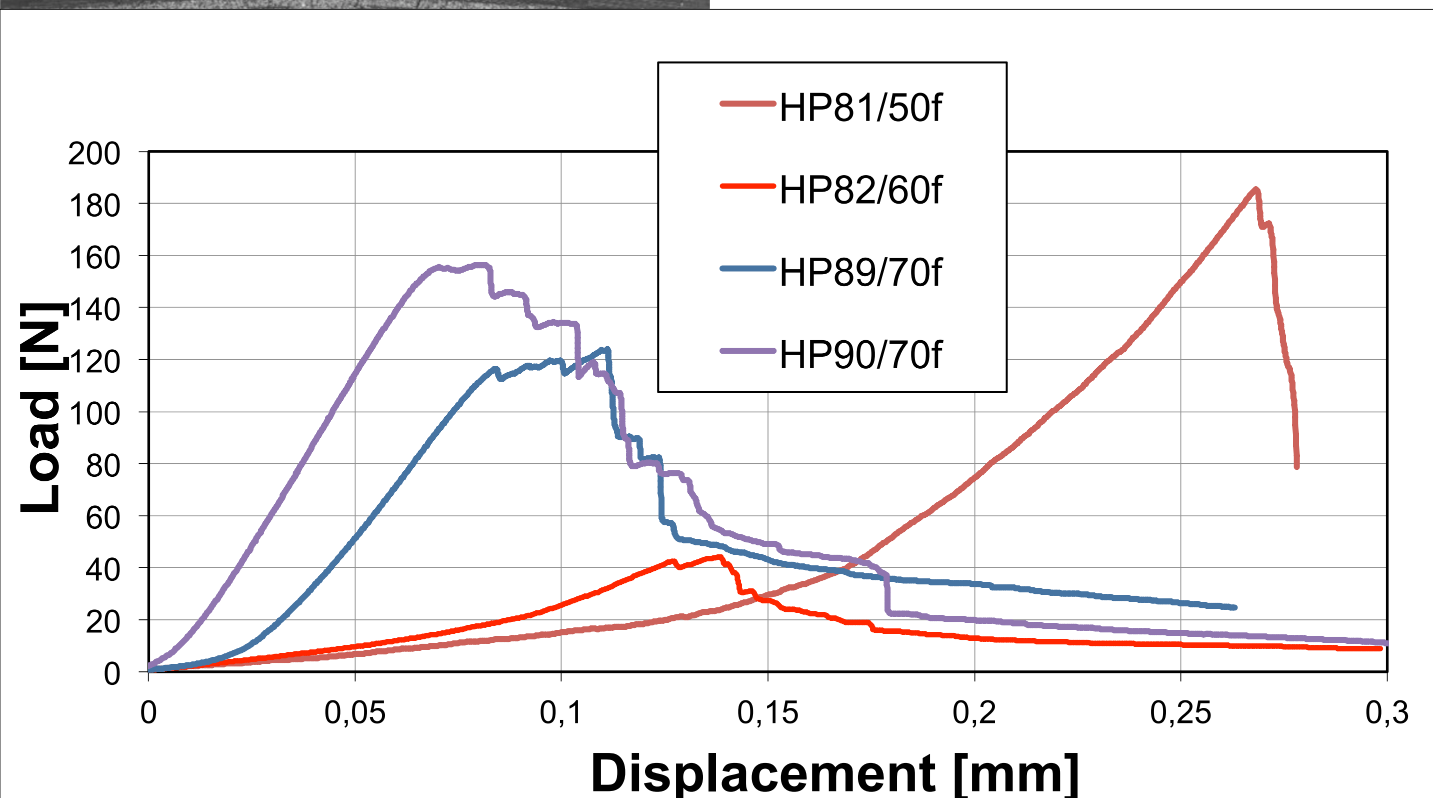


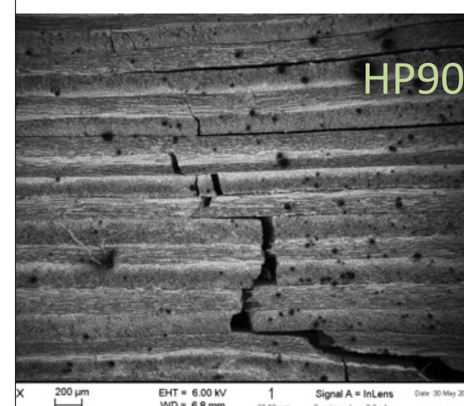


Load - Displacement behaviour



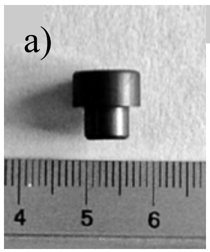


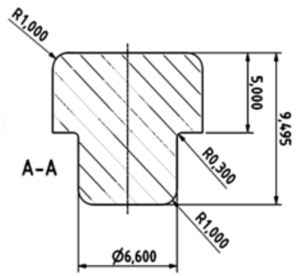




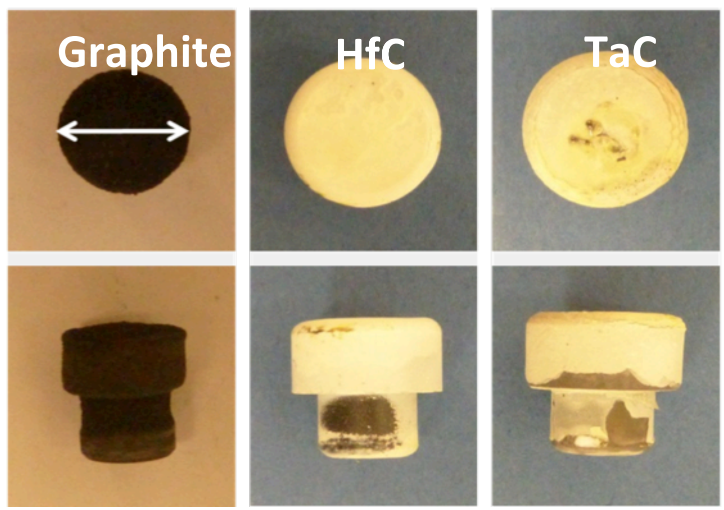
Development of ultra-ablation resistant ceramics for application in the propulsion - SMARP

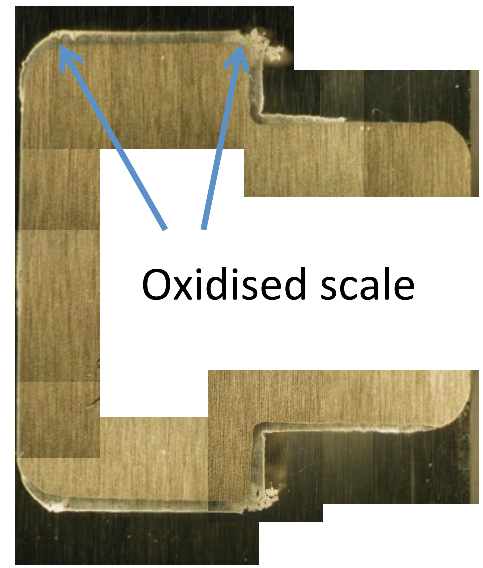
Combustion flame of oxygen and hydrocarbon gases (butane-propane), 4 min





	Max Temp. (°C)	ΔW/W0 (%)
Graphite	1600	- 26.2
HfC	1900	+ 0.2
TaC	1700	+ 0.5





Tests at DII- Univ.of Naples

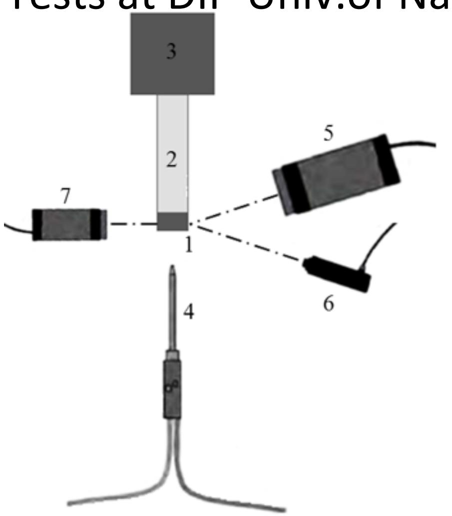
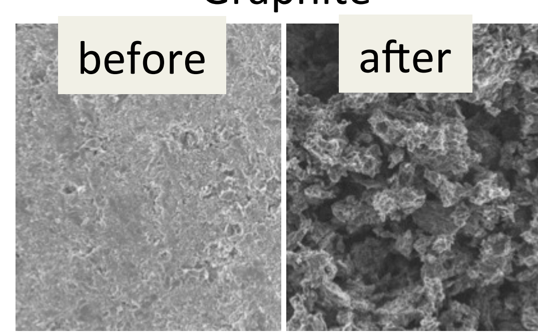
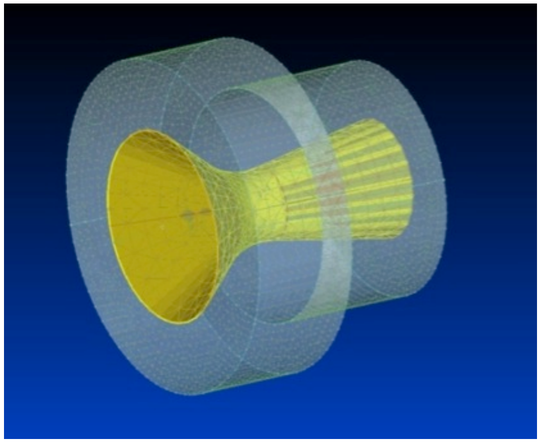


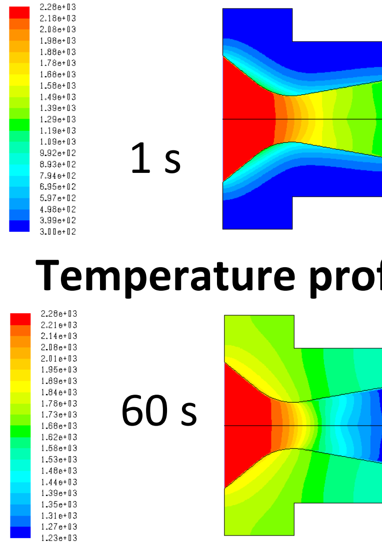
Figure 2. Experimental setup: 1) test sample, 2) alumina support, 3) ceramic support, 4) burner, 5) FLIR ThermaCAM P40 thermal camera, 6) MikronImpac ISQ5 pyrometer, 7) CCD camera.

Graphite

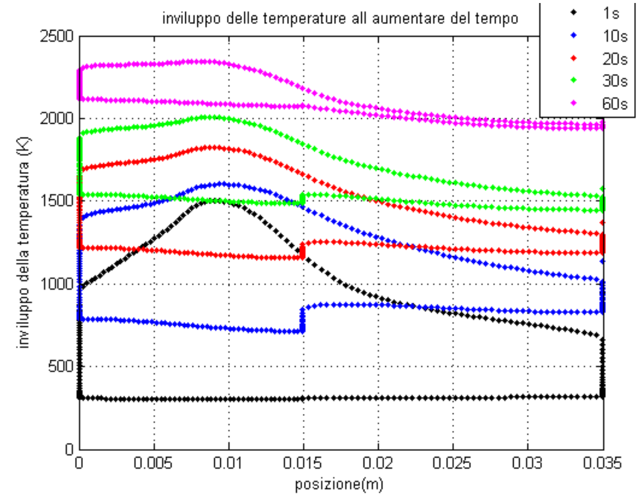


Rocket nozzle tests

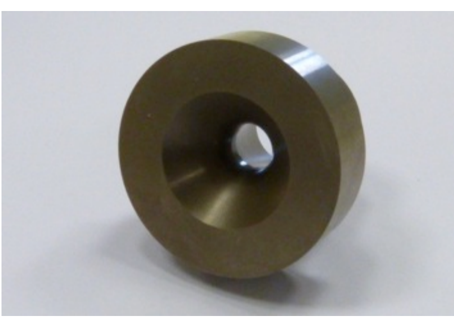


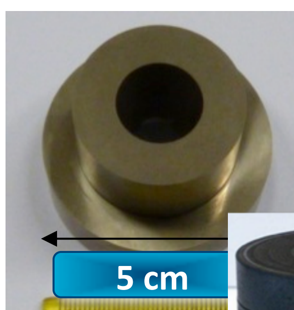


Temperature profiles

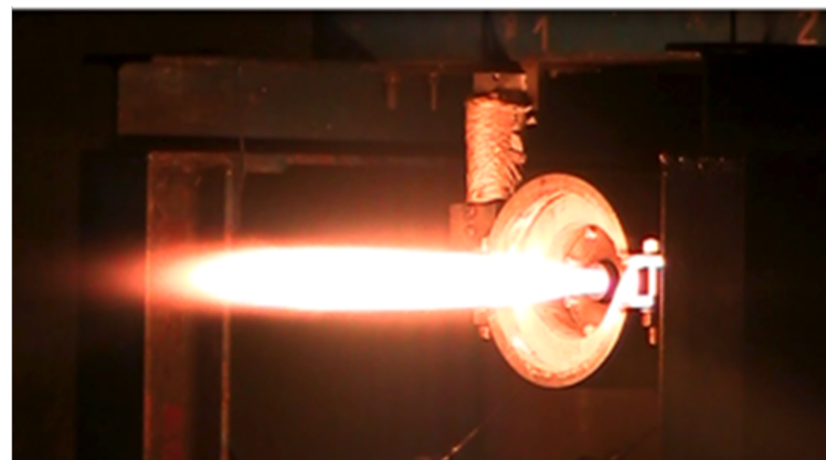


Ceramic rocket nozzle (monolithic or reinforced)





Comparison with graphite nozzles



Rocket engine test at DII - Prop lab

Conclusions

- ISTEC research is presently focused on reinforced UHTC systems (UHTC-CMCs)

- Fabrication of of short fibers-reinforced UHTCs up to 30 vol% fibers is simple, brittle behaviour, density ~ 5 g/cm^3.

- Long fibers: volumetric amount increased from 40 to 70%, non brittle behavior, density ~ 2.5 g/cm^3

Thank you for your kind attention