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Testing ultra-high temperature ceramics for thermal protection and rocket applications

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Testing Ultra-High-Temperature Ceramics for thermal protection and rocket applications

Ultra-High Temperature Ceramics: Materials for Extreme Environment Applications IV

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Cumberland Lodge,Windsor, UK

Outline

- Introduction to Ultra High temperature Ceramics (UHTC)
- Aerothermodynamic characterisation
  - Hypersonic arc-jet facility (SPES)
  - Experimental characterization in simulated atmospheric re-entry conditions
  - Numerical models and simulation of arc-jet tests
- Rockets Propulsion
  - Aerospace Propulsion Laboratory
  - Experimental characterization of materials in combustion environments
  - Numerical simulations
- Current research activities
- Conclusions

Introduction

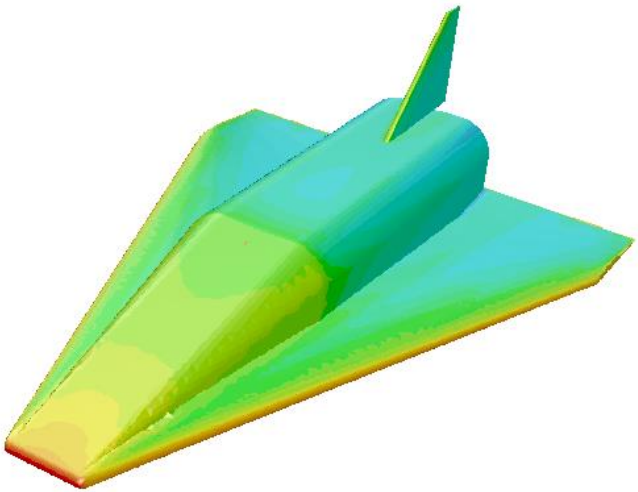
Ultra-High Temperature Ceramic (UHTC) Materials

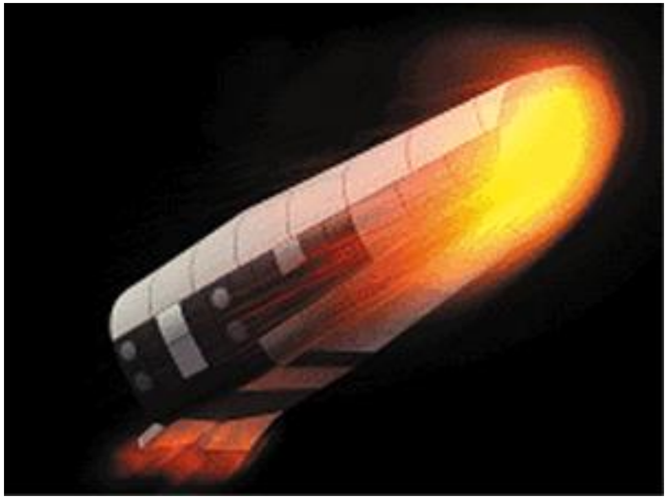
- Transition metal Diborides (i.e. ZrB_2, HfB) and transition metal Carbides (i.e. ZrC, HfC, TaC) with very high Melting Point (>3500K), high temperature strength and capability to manage and conduct heat at T>2000K
- One-phase UHTC Materials
    - Disadvantages of one-phase UHTC Materials
        - Low fracture toughness
        - Low thermal shock resistance
        - Low oxidation resistance
- UHTCs composites with particles, short fibers or whiskers
    - Very good oxidation resistance
    - Poor resistance to large thermal expansion for larger components

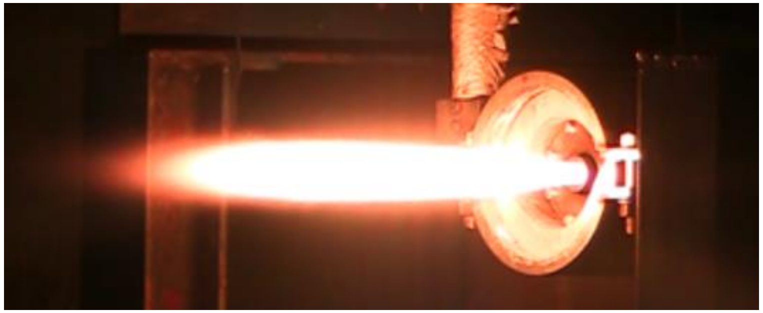
Recent interest in Ultra-High Temperature Ceramic Matrix Composites (UHTCMC) materials based on C or SiC fibers in UHTC matrix

Ultra-High Temperature Ceramic (UHTC) Materials

- Potential applications
  - Aerothermodynamics:
    - TPS for hypersonic re-entry
    - Leading edge for hypersonic vehicles
  - Propulsion:
    - Combustion chamber
    - Rocket nozzles

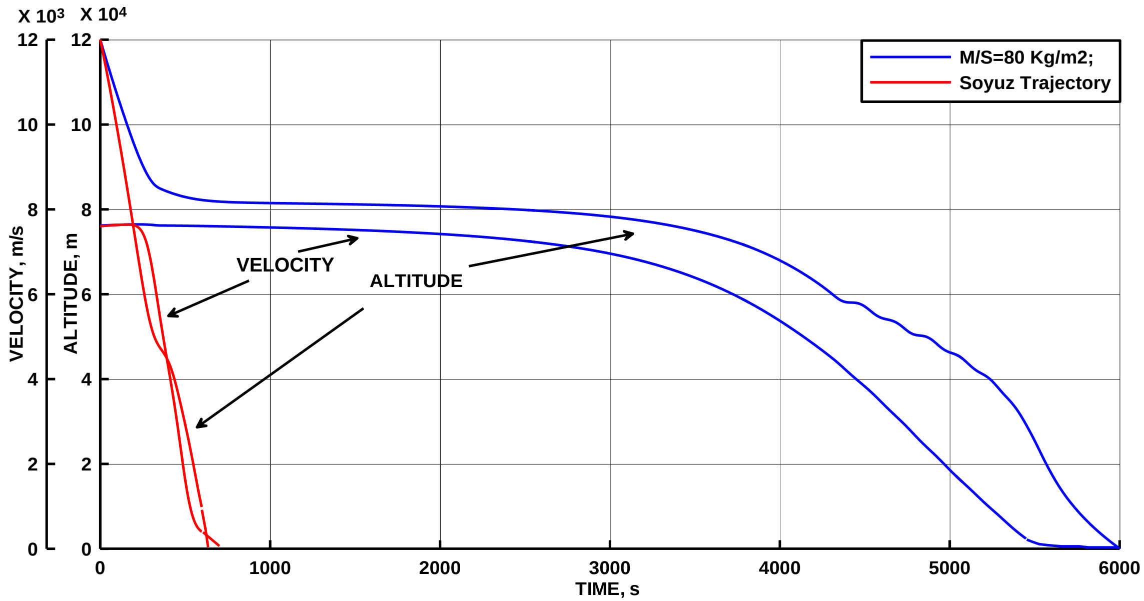




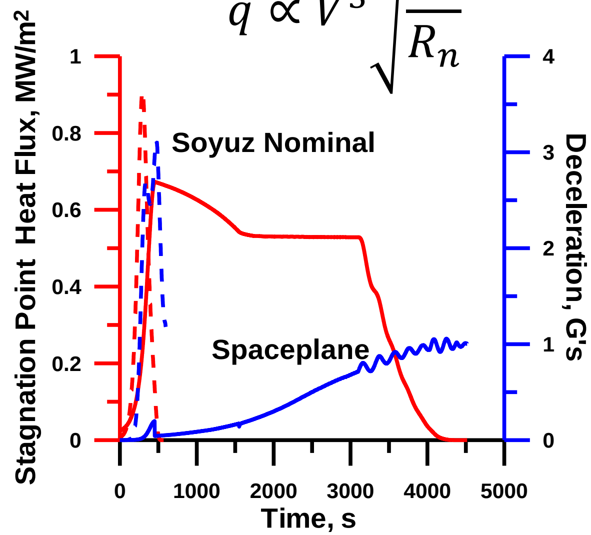


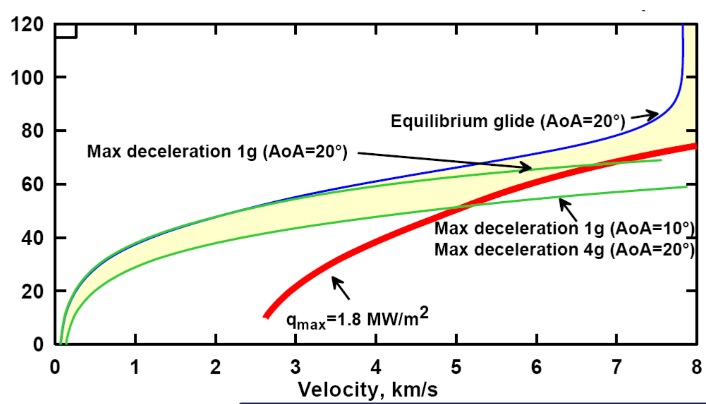
Hypersonic re-entry corridors

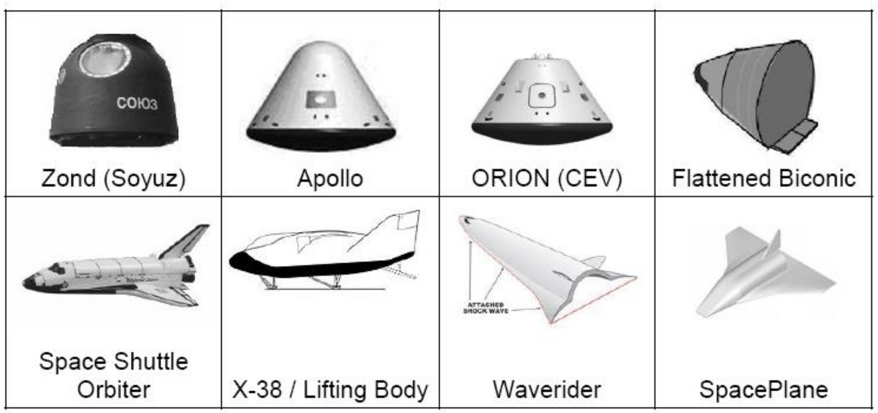
Lifting or ballistic hypersonic re-entry vehicles



$$\dot{q} \propto V^3 \sqrt{\frac{\rho}{R_n}}$$







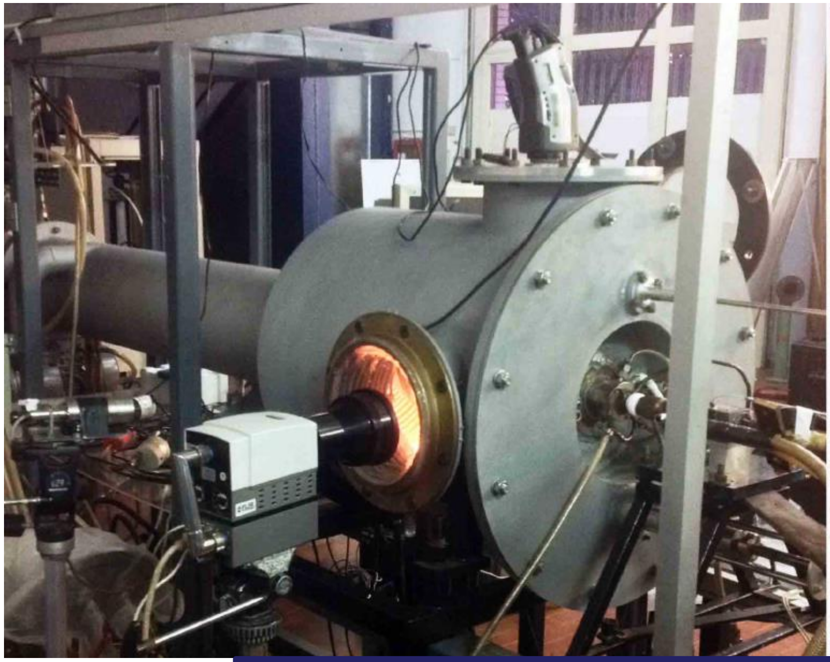
SPES (Small Planetary Entry Simulator)

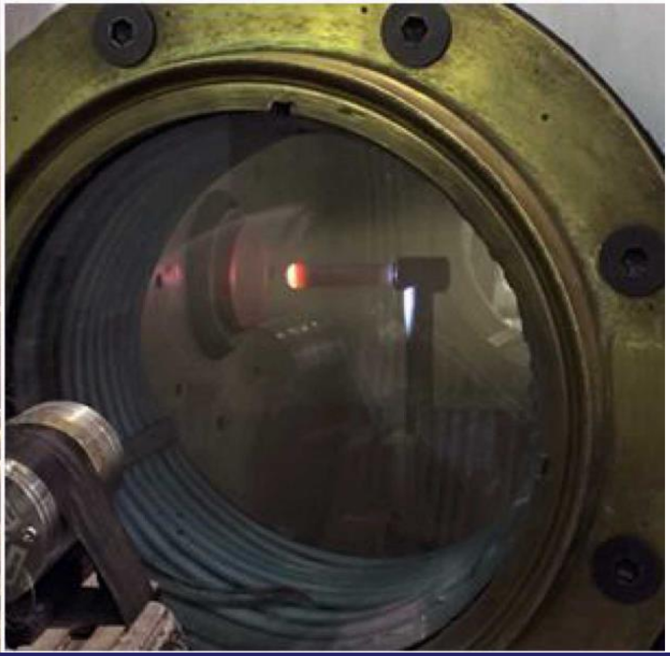
Main technical specifications:

- Max power: 80 Kw
- Running time, [s]: continuous
- Freestream Averaged Total Enthalpy: 3 - 30 MJ/Kg
- Mach number: 3 or 5 (nominal) with different nozzles
- Stagnation-point pressure: 500 – 10000 [Pa]
- Gas mass flow rate : 0.5 - 5 [g/s]

Main measurement techniques

- Total enthalpy: energy balance (global measurement)
- Stagnation-point pressure : water-cooled Pitot probe
- Pressures: precision vacuum transducers
- Mass flow : thermal mass flowmeters
- Surface temperatures : IR Thermography, pyrometers, thermocouples



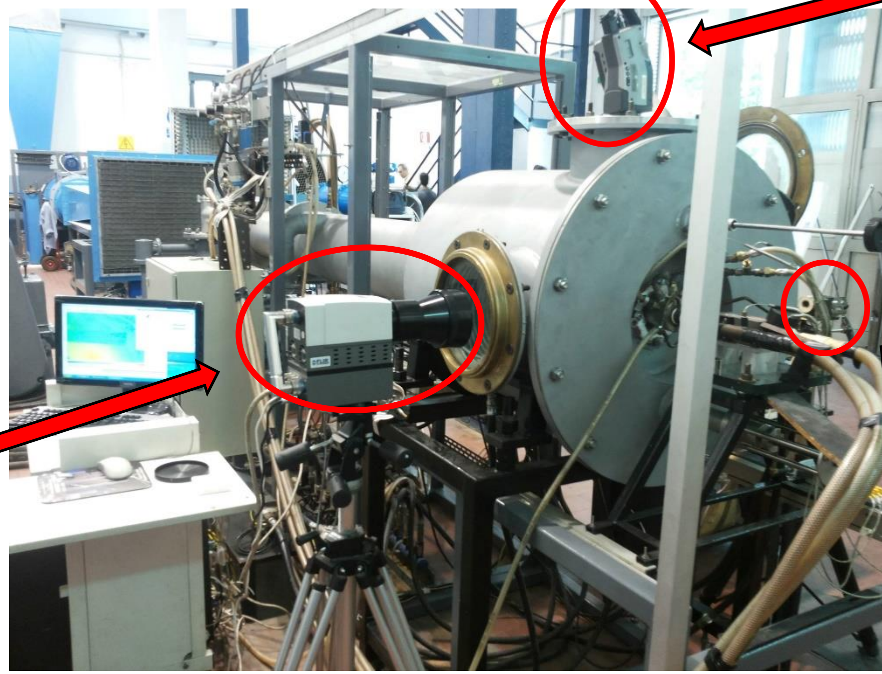


SPES (Small Planetary Entry Simulator)

T = 1200°C	Δλ (μm)	Temperature error
Emissivity error = 10%	0.8-1.1	1.3%
	8-14	> 11%

Non-intrusive diagnostic equipment:

IR Camera LW (8-13 micron)

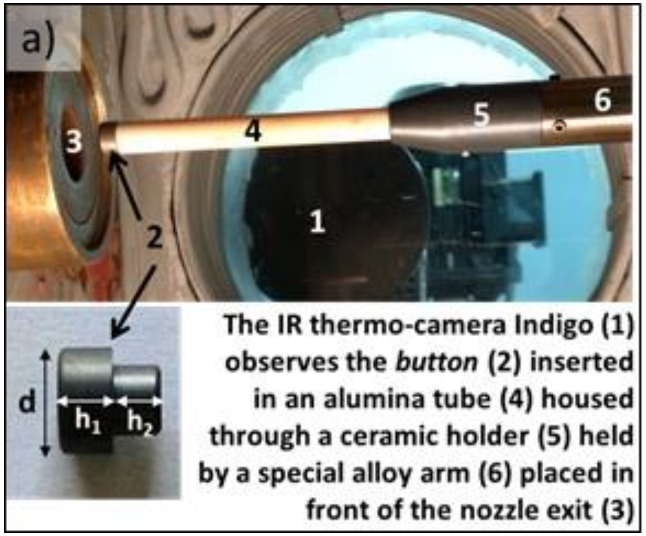


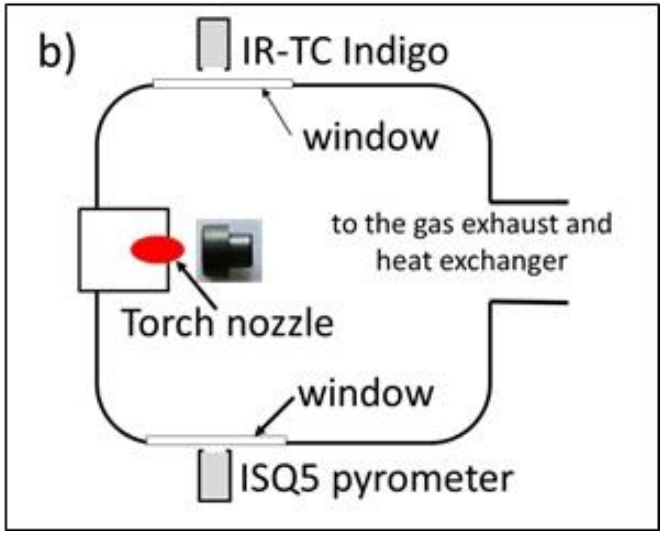
IR Camera MW (3-5 micron)

Two-color pyrometer IMPAC ISQ5

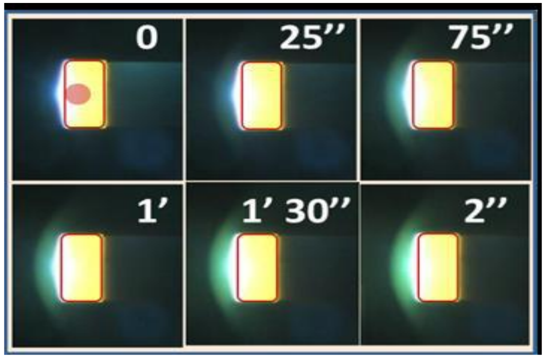
Experimental characterization in simulated atmospheric re-entry conditions

Experimental set-up inside the testing chamber during arc-jet





The inset shows a virgin cylindrical flat sample: h1= 5 mm, h2= 5 mm, and d=10 mm as diameter

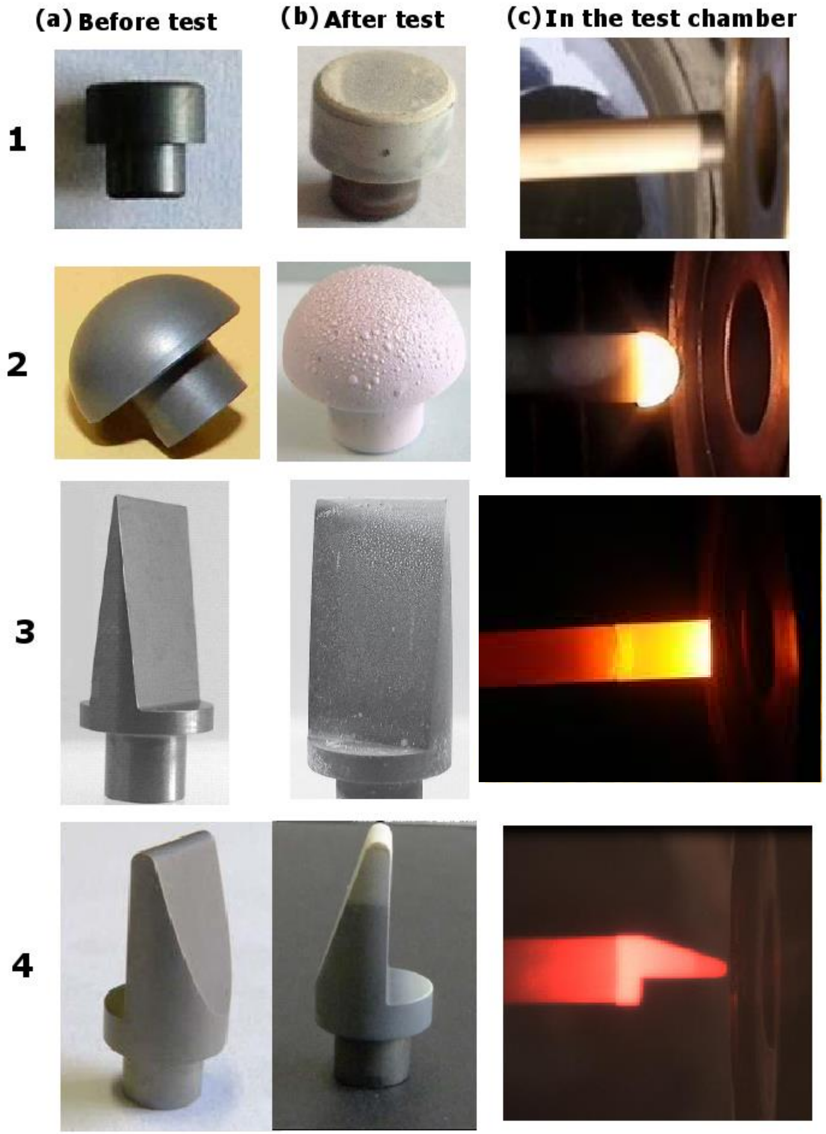


Time-resolved onset of the bow-shock during the early stages of aero-heating (2 s full duration).

The red full circle locates the measurement area of the two-color ISQ5 pyrometer.

F. Monteverde, A. Cecere and R. Savino, "Thermo-chemical surface instabilities of SiC-ZrB2 ceramics in high enthalpy dissociated supersonic air flows," Journal of the European Ceramic Society, 37 (6), 2325-2341 (2017).

Experimental characterization in simulated atmospheric re-entry conditions



Materials developed at CNR-ISTEC (Faenza, Italy)

Flat cylindrical sample (botton-like shape)

Hemispherical sample

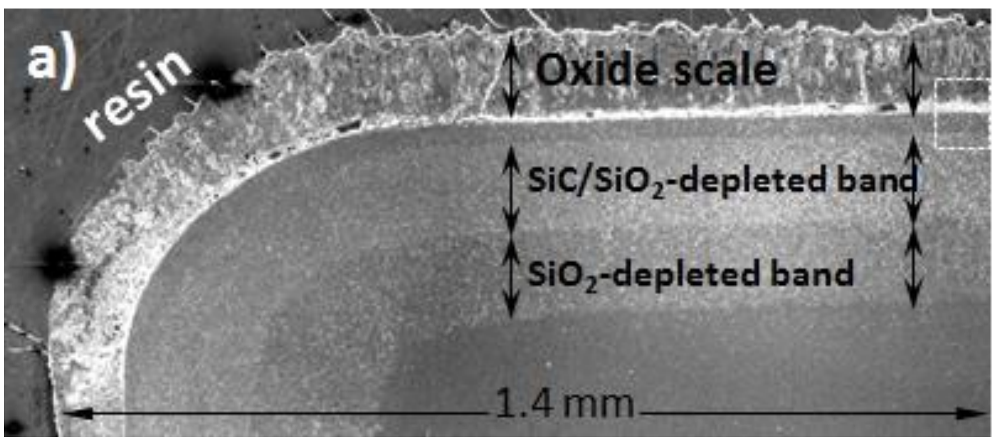
Symmetrical sharp leading edge

Asymmetrical round leading edge

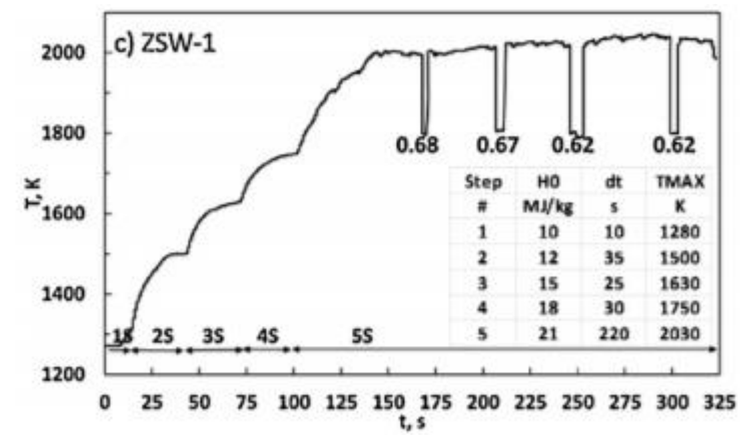
Experimental characterization of blunt UHTC specimens simulated atmospheric re-entry conditions

Post test materials analysis at CNR-ISTEC (Faenza, Italy)

Flat cylindrical sample (button-like shape) for the simulation of the conditions reached in the stagnation point of a re-entry vehicle



The layered multiphase configuration of the oxidized scale microstructures (SEM micrograph) of the ZSW button after ZSW-1 test.

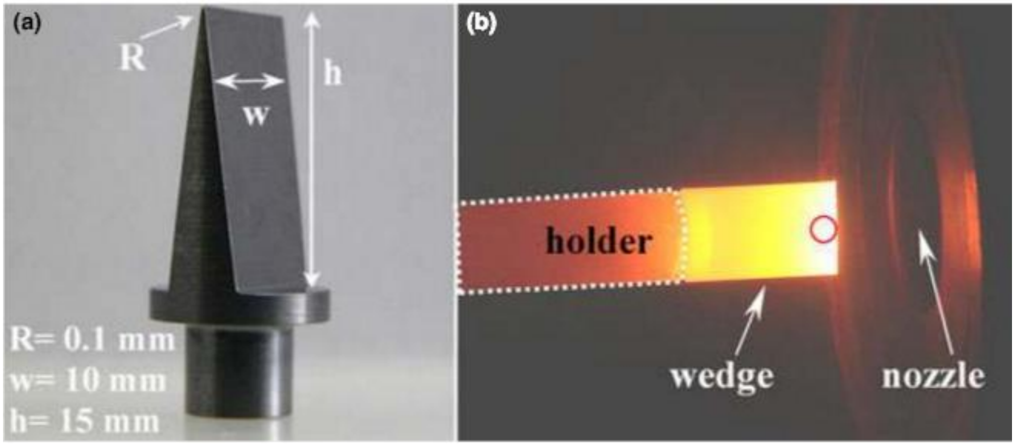


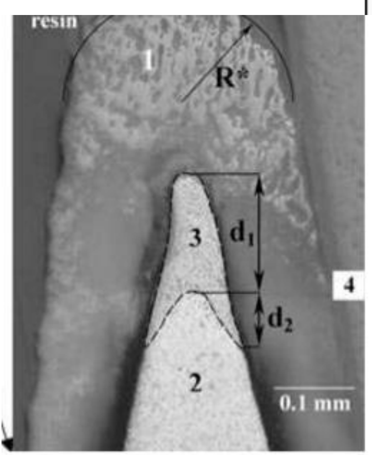
Temperature and emissivity evaluated with pyrometer

F. Monteverde, A. Cecere and R. Savino, "Thermo-chemical surface instabilities of SiC-ZrB2 ceramics in high enthalpy dissociated supersonic air flows," Journal of the European Ceramic Society, 37 (6), 2325-2341 (2017).

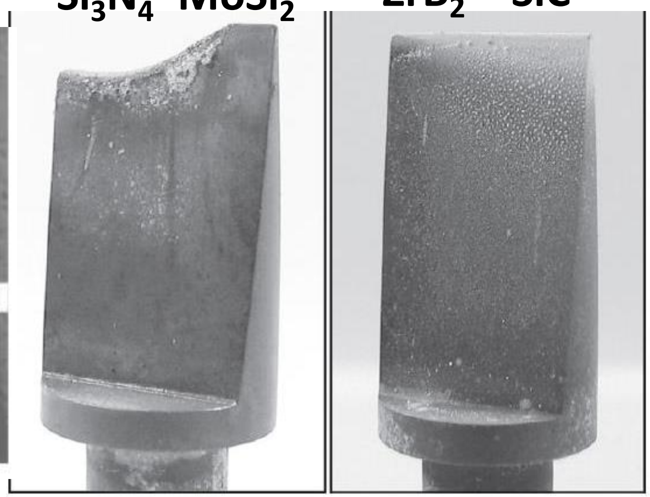
Experimental characterization of UHTC leading edges in simulated atmospheric re-entry conditions

Sharp models for the simulation of the conditions reached on leading edges of winged hypersonic vehicles





Si_3N_4–$MoSi_2$ ZrB_2 – SiC



Wedge	Test	Step	H_0 (MJ/kg)	t (s)	Max T_{pyr} (°C)	$\varepsilon_{1\ \mu m}$
ZS	T_1	T_1-1	8.2	45	1149	0.74
		T_1-2	10.2	40	1278	0.74
		T_1-3	10.8	35	1373	0.75
		T_1-4	12.2	45	1408	0.75
		T_1-5	13.8	35	1447	0.75
		T_1-6	15.1	30	1491	0.75
		T_1-7	16.4	85	1537	0.75
	T_2	T_2-1	8.1	40	1197	0.74
		T_2-2	9.8	35	1300	0.74
		T_2-3	10.5	30	1402	0.74
		T_2-4	11.9	25	1472	0.74
		T_2-5	13.5	25	1507	0.74
		T_2-6	14.8	35	1545	0.73
		T_2-7	16.0	70	1577	0.73
SM	T_3	T_3-1	10.1	50	1370	–
		T_3-2	11.6	40	1435	–
		T_3-3	13.8	50	1500	–
		T_3-4	15.6	20	1570	–
		T_3-5	16.5	45	1735	–

Sharp wedge models after the tests in SPES arc jet facility:

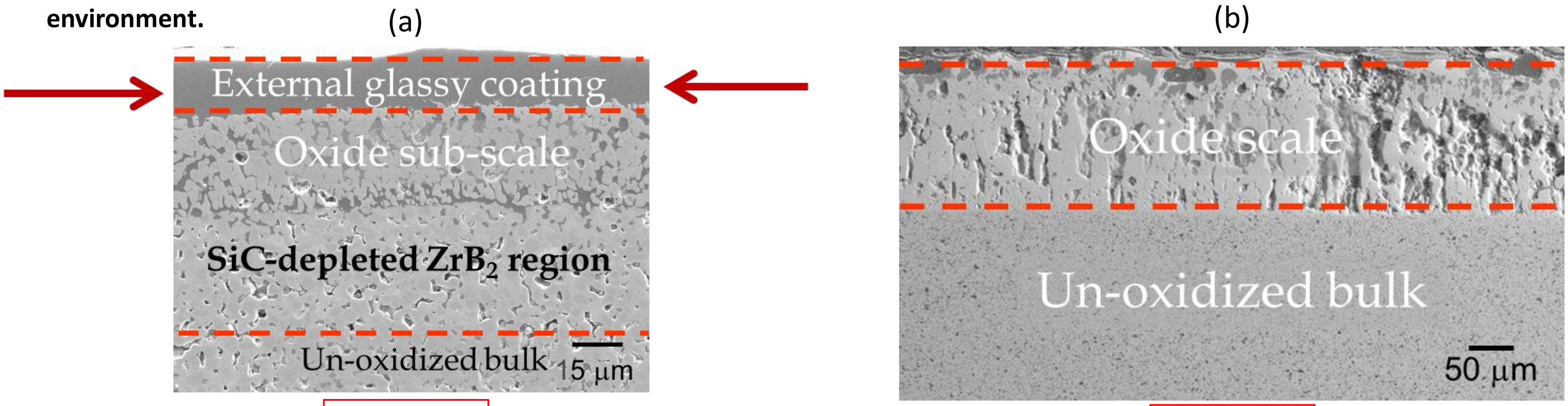
(a) lower thermal conductivity ceramic Si3N4–MoSi2 wedge after one test: the sharp tip reached high temperatures and the material underwent partial melting;
(b) UHTC (ZrB_2 – SiC) wedge after two consecutive tests: the higher thermal conductivity allowed to re-distribute the heat over cold regions and the material survived the extreme test conditions

F. Monteverde, R. Savino, ZrB2–SiC sharp leading edges in high enthalpy supersonic flows, Journal of the American Ceramic Society 95 (7), 2282-2289 (2012).

Effect of the test conditions on the materials response

When UHTC materials are characterized with plasma or oxyacetylene torches or even with other heating systems in gas environments that are not properly reproducing the gas composition, pressure and shear conditions, different surface modifications are possible and these may alter important surface properties like **emissivity** and **catalyticity**. This is true both for reentry and rocket applications, because the temperature and exhaust velocity of rocket plumes may be about 3200 °C and 1600 m/s, respectively. Hence, the recession rate of the composites in rocket plumes is expected to be higher than that in oxyacetylene torch with temperature of 3000 °C and gas flow of 10 m/s.

Even if the heat flux is the same, **the materials response may be very different, compared to what happens in real flight environment.**



ε = 0.9

ε = 0.7

Cross-section of a ZrB_2–15SiC material (a) after subsonic plasma torch testing (at atmospheric pressure)[1] and (b) after supersonic arc-jet testing (at low pressure)[2]

[1]F. Monteverde, R. Savino, «Stability of ZrB2–SiC ceramics under simulated atmospheric re-entry conditions" Journal of the European Ceramic Society, 37, 4797 (2007)

[2] F. Monteverde, A. Cecere and R. Savino, "Thermo-chemical surface instabilities of SiC-ZrB2 ceramics in high enthalpy dissociated supersonic air flows," Journal of the European Ceramic Society, 37, 2325-2341 (2017)

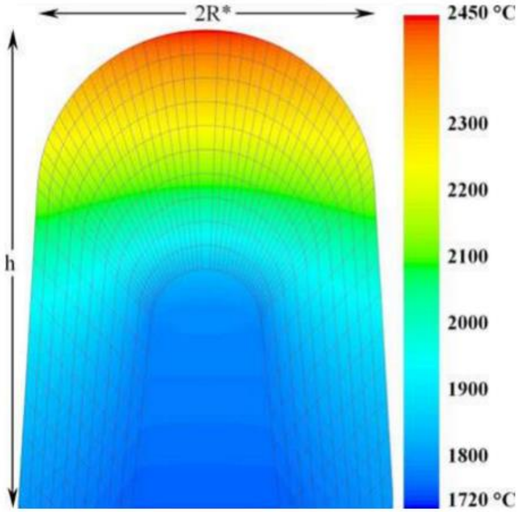
Numerical simulations to support experimental activities

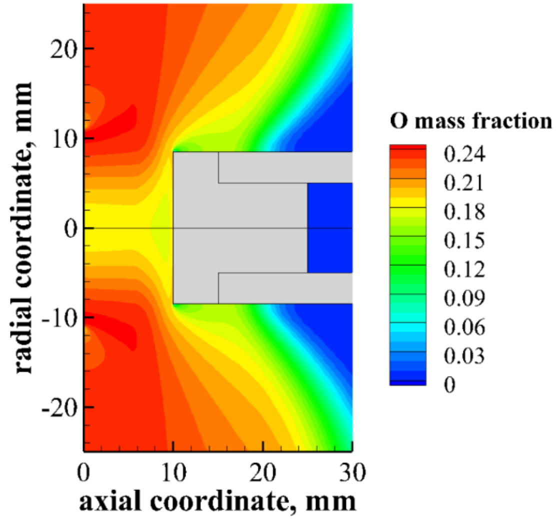
- High complexity of the flow conditions generated in the high-enthalpy plasma wind tunnel or in the rocket combustion chamber

  → The verification of the operating conditions must be a combined effort of experimental diagnostics and CFD simulations

- Difficulties to experimentally collect significant quantities

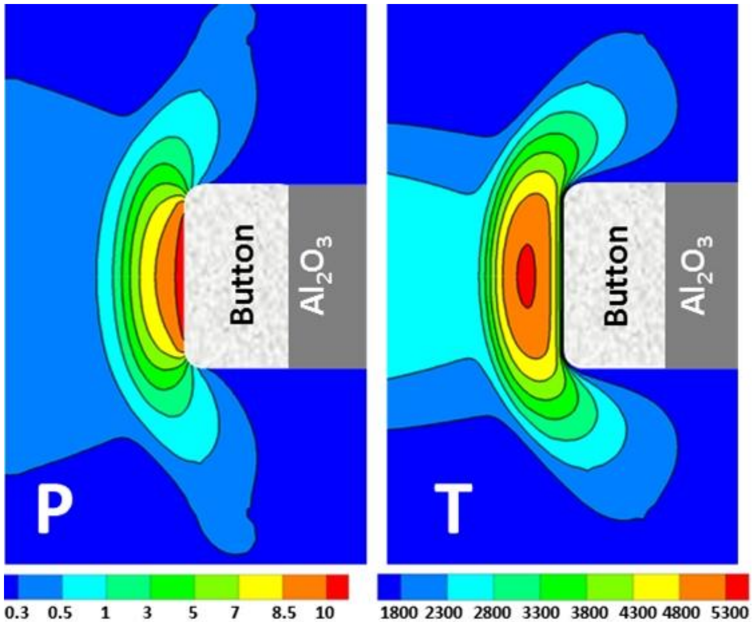
  → Numerical simulations are a viable and largely recommended tool to predict the thermo-chemical evolution of the gas and to characterize the flow-field surrounding the proof articles



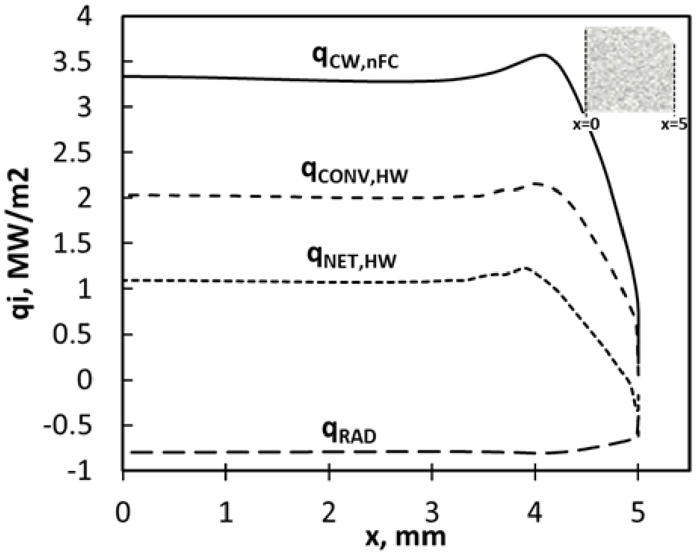


Numerical models and simulation of arc-jet tests

Model for samples thermal analysis



Static pressure P_{STAT} (Pa) and temperature T_{STAT} (K) around the flat cylinder sample computed by CFD analysis



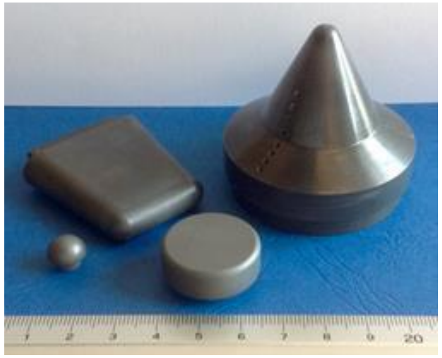
Various components of the heat flux (qi) vs x coordinate of the sample computed by CFD analysis for H0=21 MJ/kg, ε=0.7 and γ=0.1: hot-wall convective (CONV, HW), radiative (RAD) and net hot-wall (NET, HW). The initial cold-wall zero-catalytic heat flux was also calculated (i.e. γ=0, CW, nFC)

F. Monteverde, A. Cecere and R. Savino, "Thermo-chemical surface instabilities of SiC-ZrB2 ceramics in high enthalpy dissociated supersonic air flows," Journal of the European Ceramic Society, 37 (6), 2325-2341 (2017).

C^{3}HARME European Project

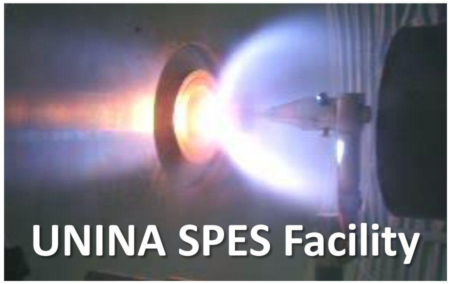
- **Design, development, manufacturing and testing of a new class o UHTCMCs for application in severe aerospace environments**:
  - **Application 1: Near ZERO-Erosion nozzle** inserts that can maintain dimensional stability during firing in combustion chambers of high performances rockets for civil aerospace propulsion
  - **Application 2: Near ZERO-Ablation thermal protection systems** for launch and re-entry into Earth's atmosphere

- **Partners of the project**:
  CNR ISTEC, IN Srl, University of Birmingham, TECNALIA, UNINA, DLR, AVIO, NANOKER, HPS, Airbus, GMBH, Trinity College.



Current Research Activities in the frame of C³HARME European Project

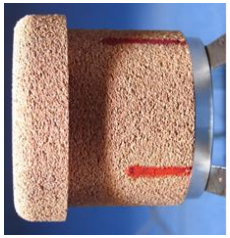
Incremental approach for materials qualification



SMALL SPECIMENS

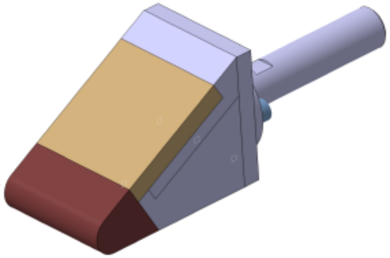
SIZE : **15mm**

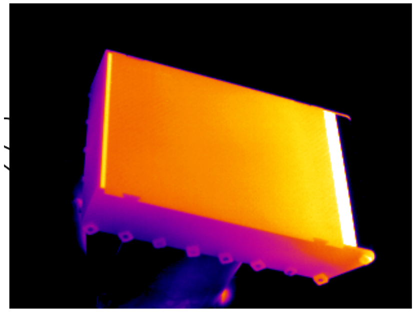
MEDIUM/LARGE SAMPLES



SIZE: **30/50mm**

FLAT PLATE/WEDGE MODEL





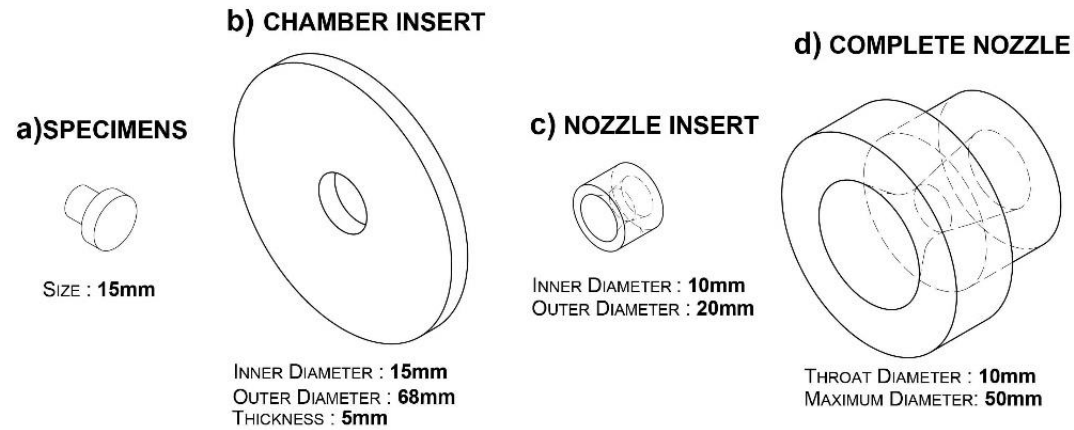
SIZE : **50x50mm/240x190mm**

TRL →

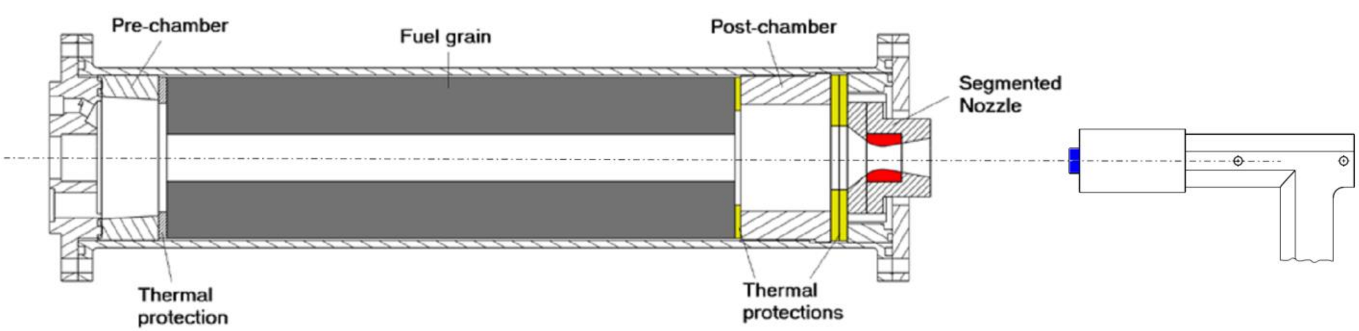
- Arc jet test at UNINA
- Plasma Wind Tunnel Test at DLR
- Plasma Wind Tunnel Test at DLR

Current Research Activities on UHTCMC nozzles in the frame of C³HARME European Project

Incremental approach for testing in hybrid rocket

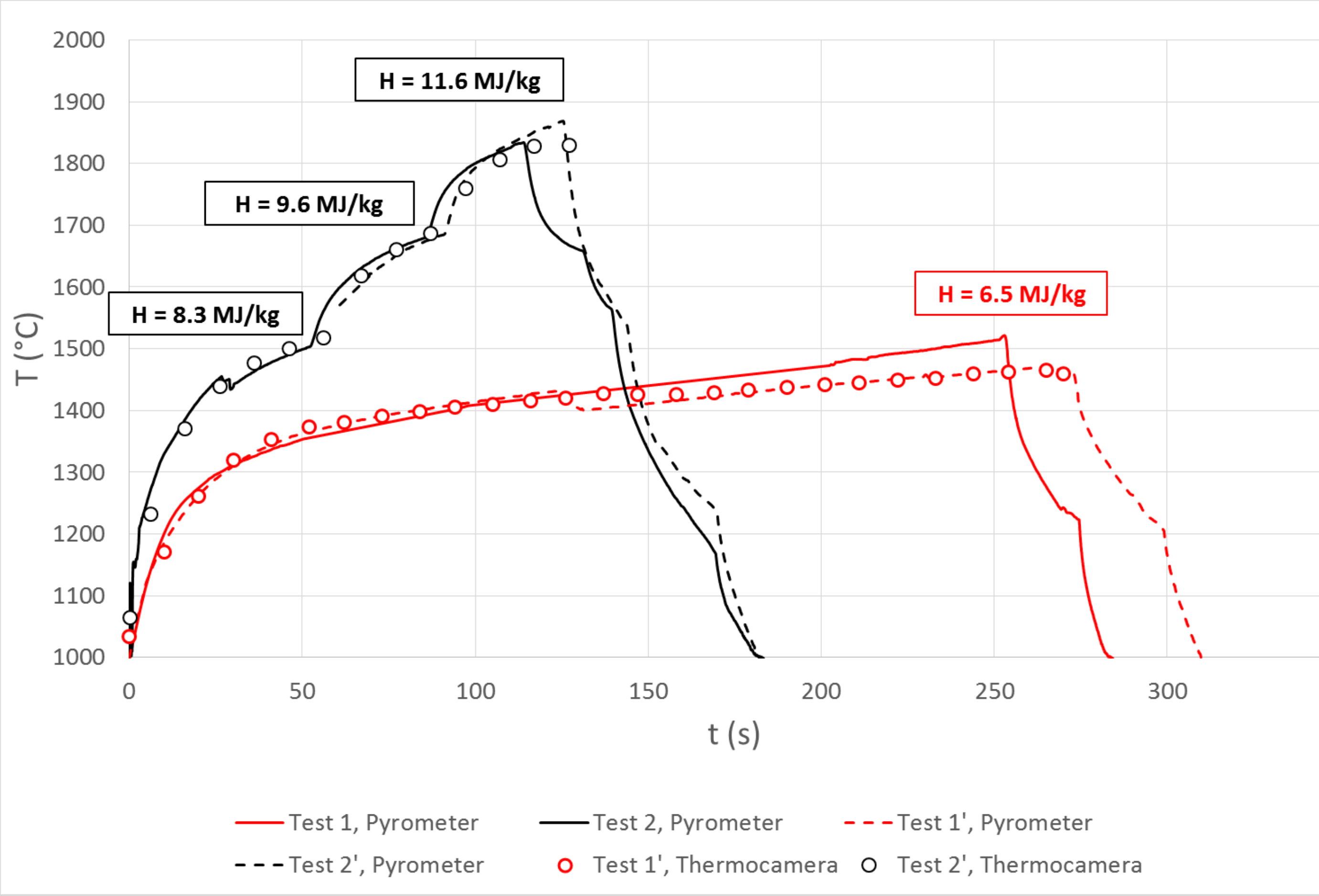


Design of the test articles for C³HARME experimental campaign at Hybrid Rocket Propulsion Laboratory



Schematic section of Hybrid Rocket Motors and Free jet test experimental setup with test articles: Flat cylindrical specimen (Blue); Chamber inserts (Yellow); Nozzle Insert (Red)

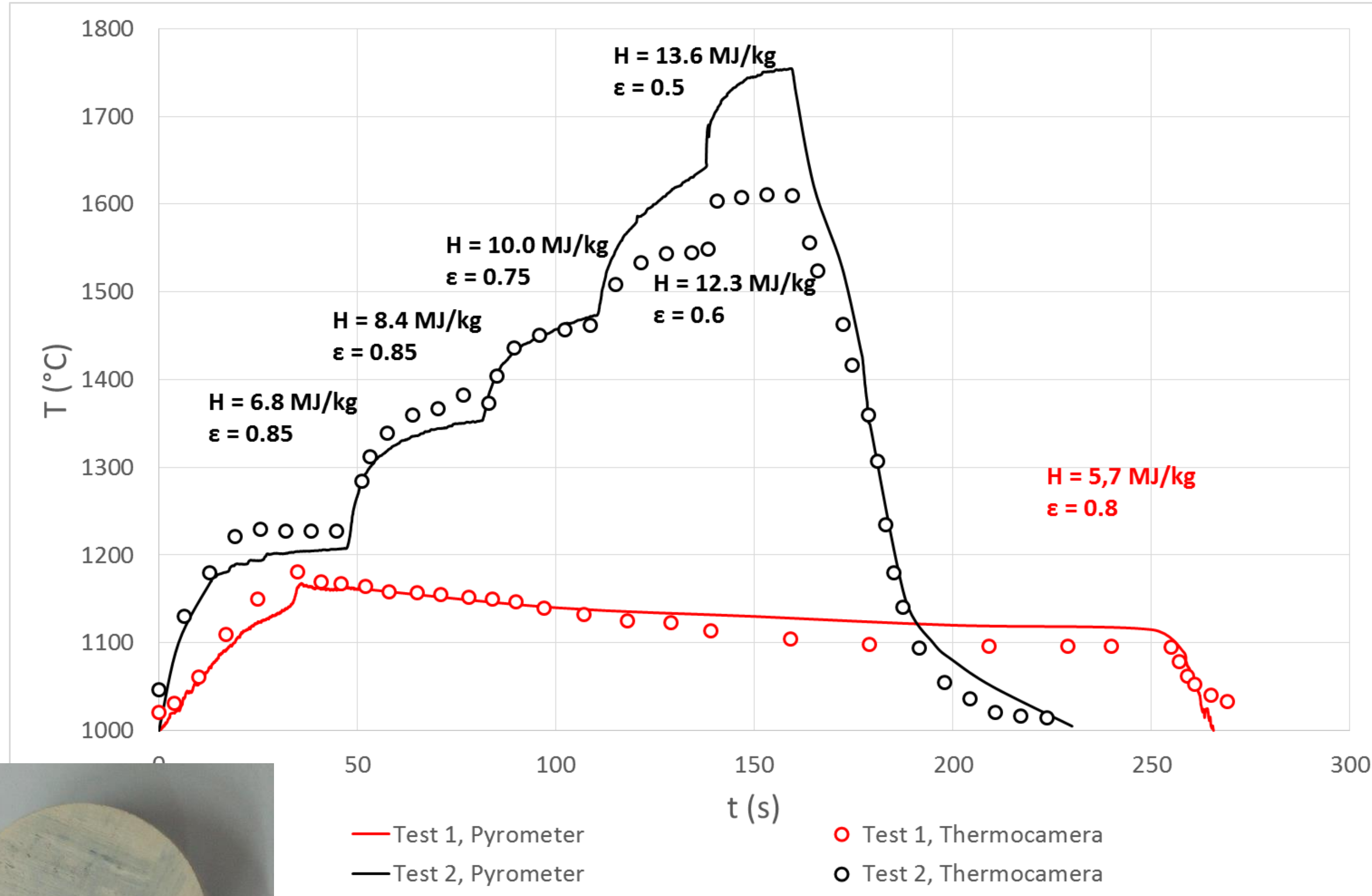
Current Research Activities in the frame of C^3HARME European Project – Graphite Test



Graphite – Sample before test 1 (Mass=4.4 g)

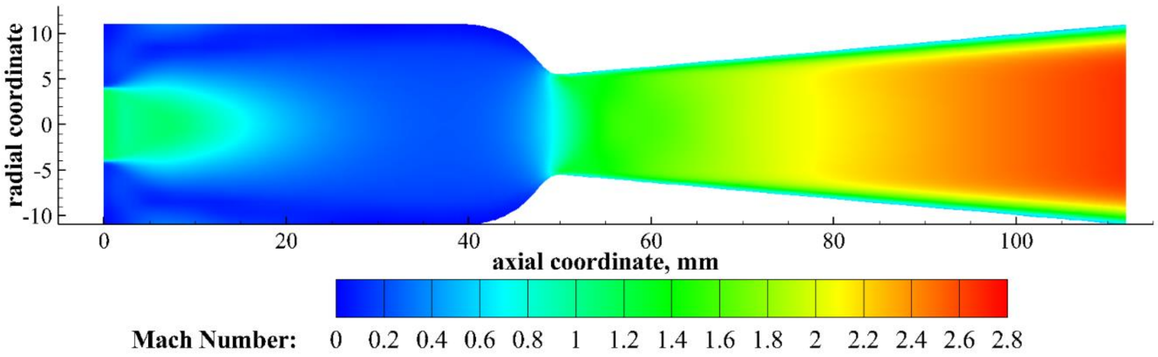
Graphite – Sample after test 1 (Mass=3.2 g)

Current Research Activities in the frame of C^3HARME European Project – UHTCMC Test



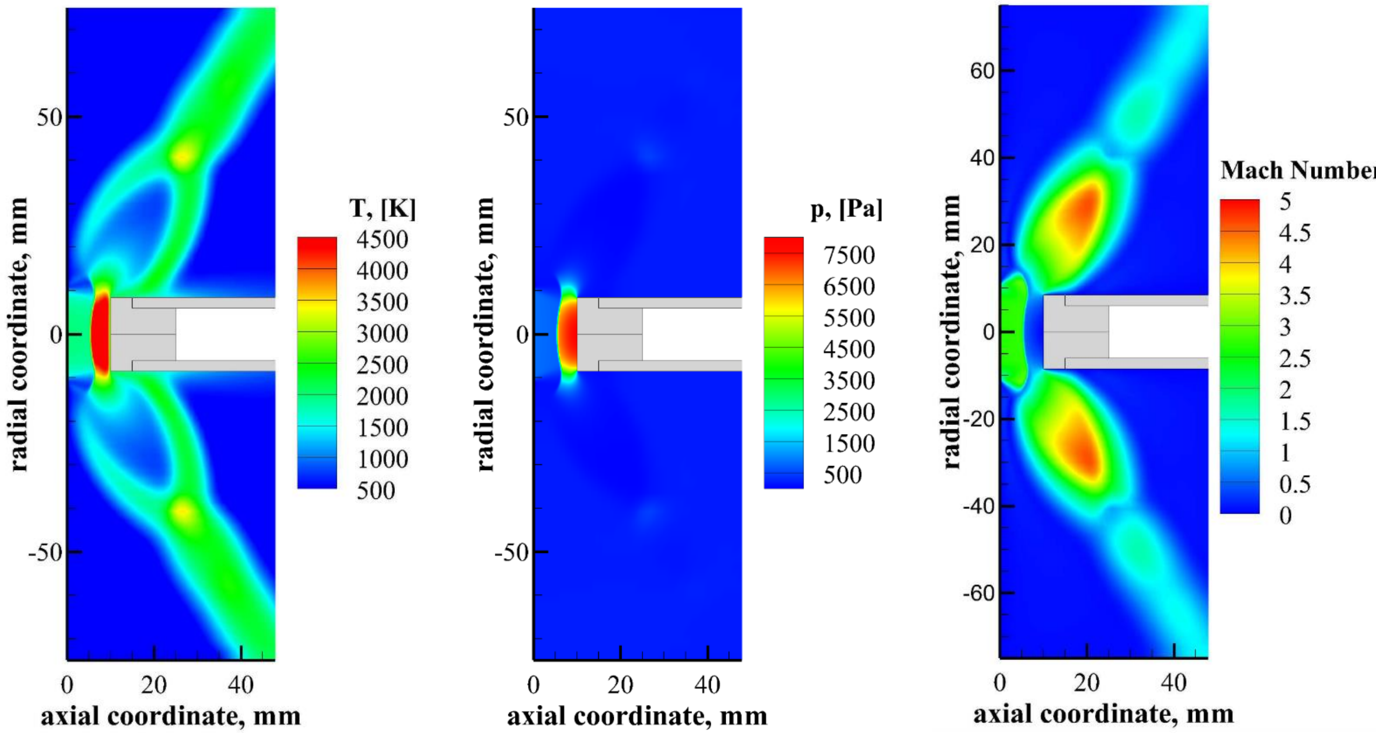
UHTCMC - Sample before test 2 (Mass=4.8 g)
UHTCMC – Sample after test 2 (T=1750°C, Mass=4.8 g)

Numerical simulations of arc-jet tests on UHTCMC samples



The flow field inside the nozzle of SPES arc-jet facility is simulated by means of CFD calculations, at different conditions, corresponding to the experimental total enthalpy levels

The values of the thermo-fluid-dynamic quantities and the species concentrations, achieved at nozzle exit, are used as inputs for the CFD simulation of the flow field inside the test chamber

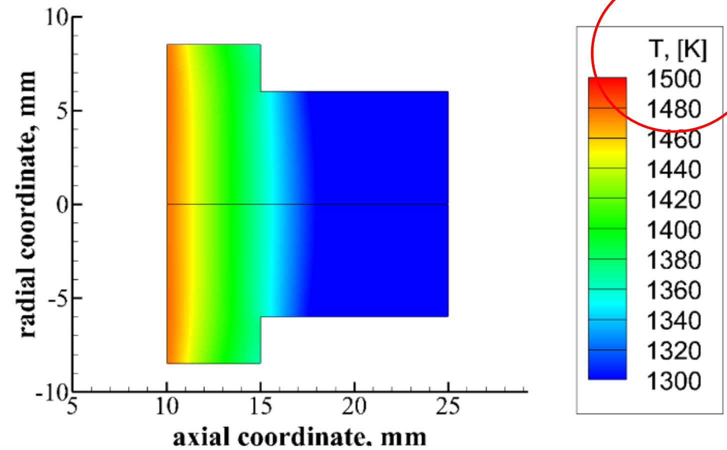


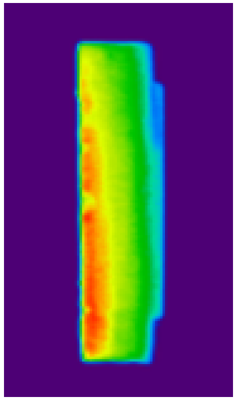
Numerical simulations of arc-jet tests on UHTCMC samples

Thermal analyses of the samples are carried out solving the energy equation inside the solid material, taking into account the coupling with the fluid field

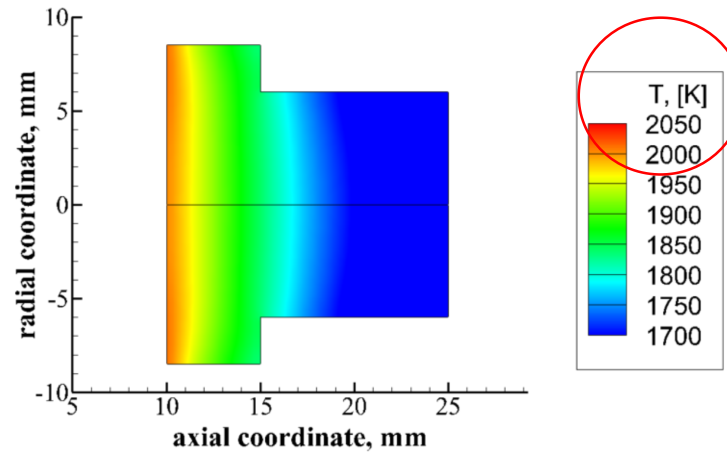
CFD | **Experimental**

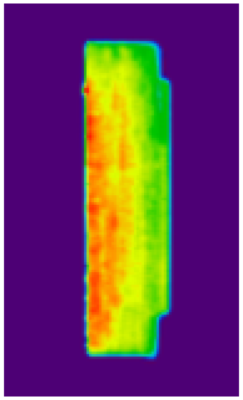
H_0 = 6.8 MJ/kg
ε = 0.85
Not catalytic wall



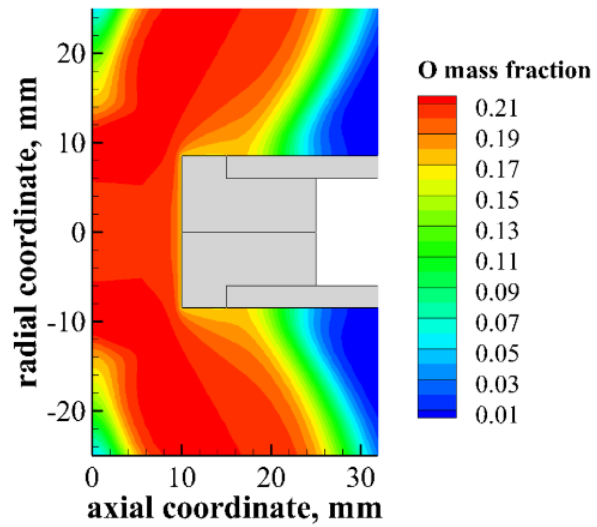


H_0 = 13.6 MJ/kg
ε = 0.5
γ = 0.0085



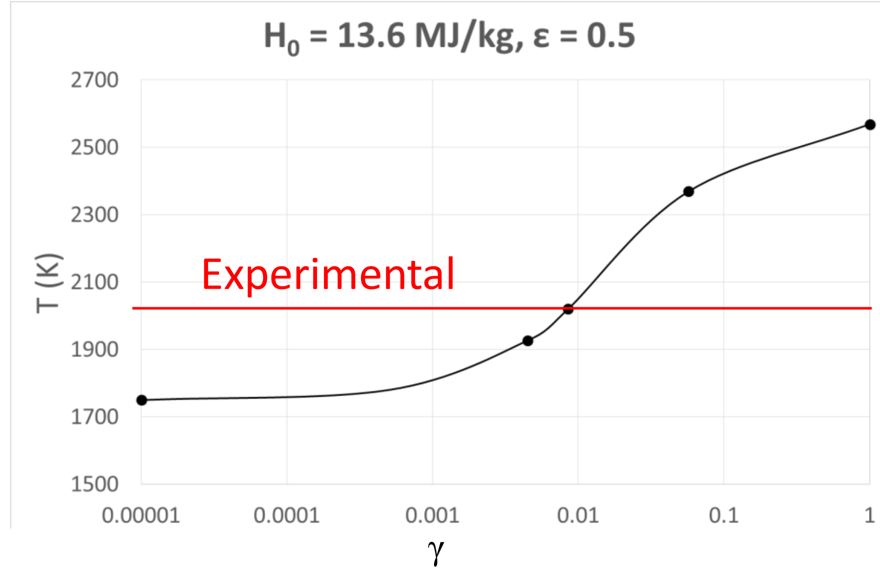


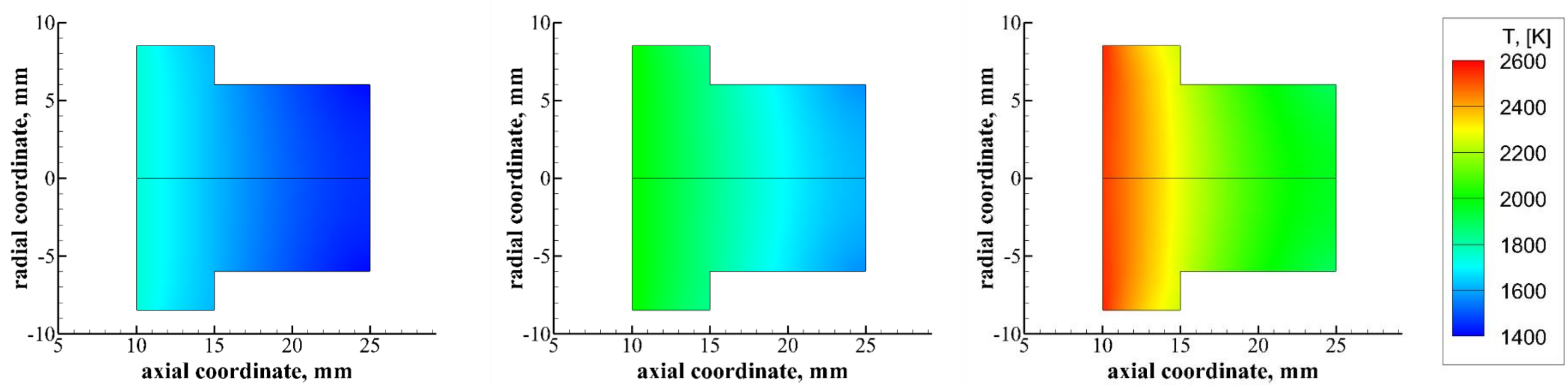
Numerical simulations – Effect of catalyticity



$$\frac{\partial C_i}{\partial n} = \frac{C_i K_w}{D_i}$$

$$K_w = \gamma \sqrt{\frac{R_0 T_w}{2\pi m_i}}$$





Not catalytic wall | **γ = 0.0085 (Experimental)** | **Fully catalytic wall**

Experimental characterization of materials in combustion environment

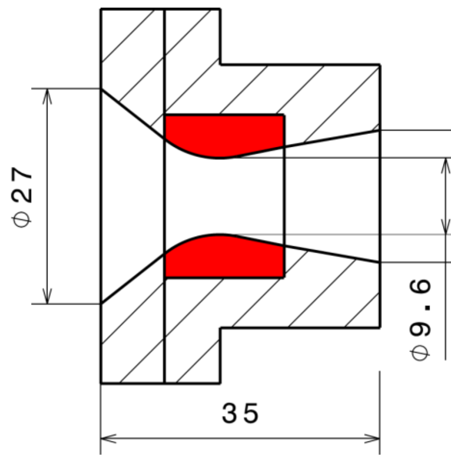
Characterization of ceramic nozzle: test setup

Test 1: Graphite nozzle

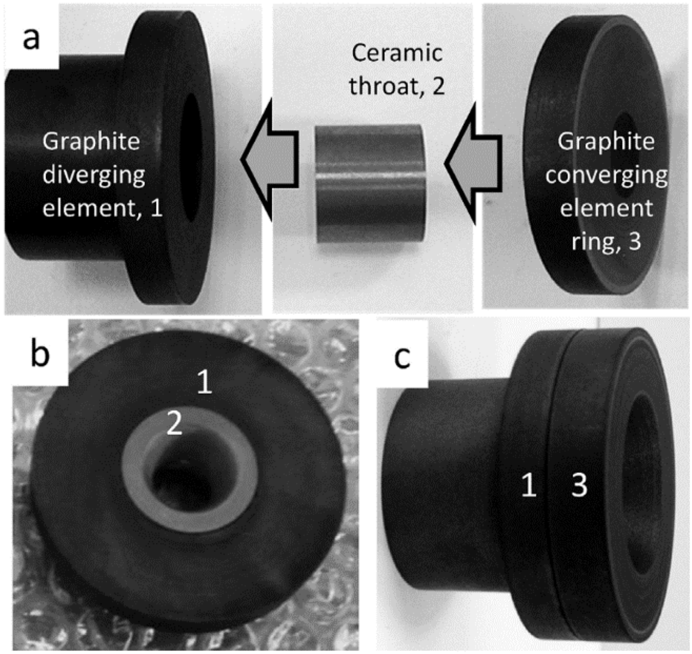
Test 2: Segmented nozzle with TaC throat insert

Test conditions

Propellants	Gaseous Oxygen + HDPE
$\dot{m}_{ox}$	27 g/s
Test duration	10 s
Grain length	240 mm
Initial port diameter	25 mm



Layout De Laval nozzle geometry (dimensions in millimiter)



Segmented nozzle, composed of a graphite diverging element (1), ceramic throat made of TaC-based composite material (2), and outer graphite converging element (3).

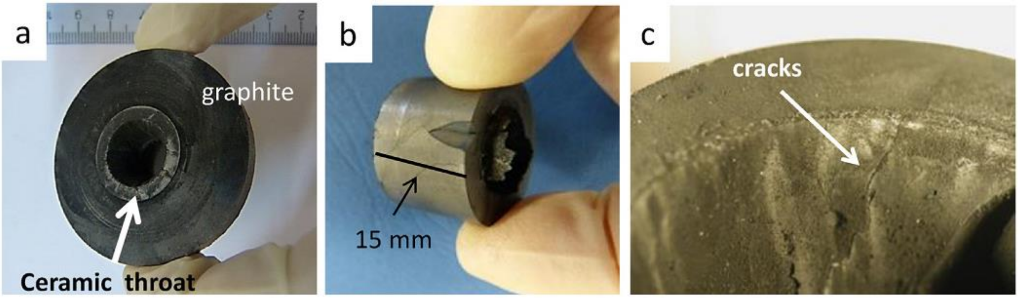
R.Savino, G.Festa, A. Cecere and L. P. a. D.Sciti., "Experimental set up for characterization of carbide-based materials in propulsion environment.," Journal of the European Ceramic Society, pp. 1715-1723, 2015.

Characterization of ceramic nozzle: results

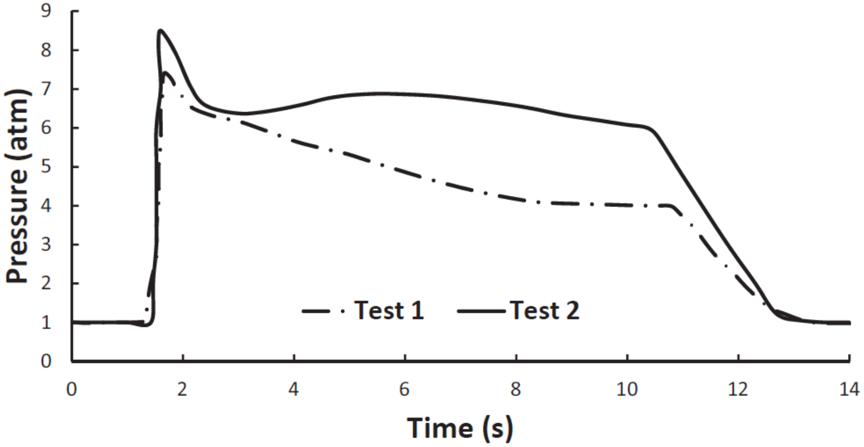
Throat erosion rate:

Test 1: $0.28\ mm/s$

Test 2: ~ 0



(a) Segmented nozzle (after removal of the converging outer element), (b) ceramic throat, (c) details of the ceramic throat showing radial cracks



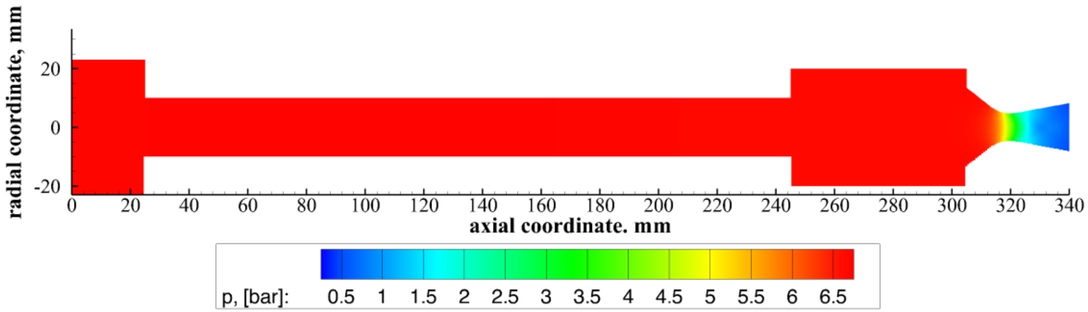
Chamber pressure time profile for the tests

- Zero-erosion behavior allows the performances to be much more stable during the whole operating time
- The presence of the cracks demonstrates the fragility of these UHTC materials, which may be overcome by the use of fiber-based Ceramic Matrix Composites

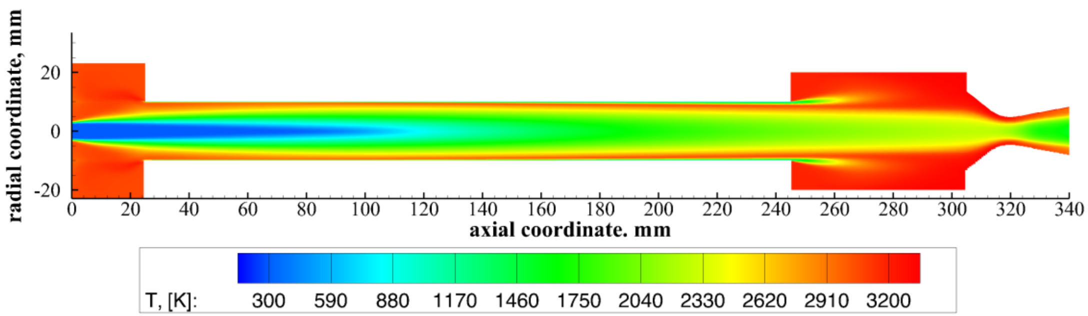
R.Savino, G.Festa, A. Cecere and L. P. a. D.Sciti., "Experimental set up for characterization of carbide-based materials in propulsion environment.," Journal of the European Ceramic Society, pp. 1715-1723, 2015.

Numerical models and simulation of hybrid rocket tests

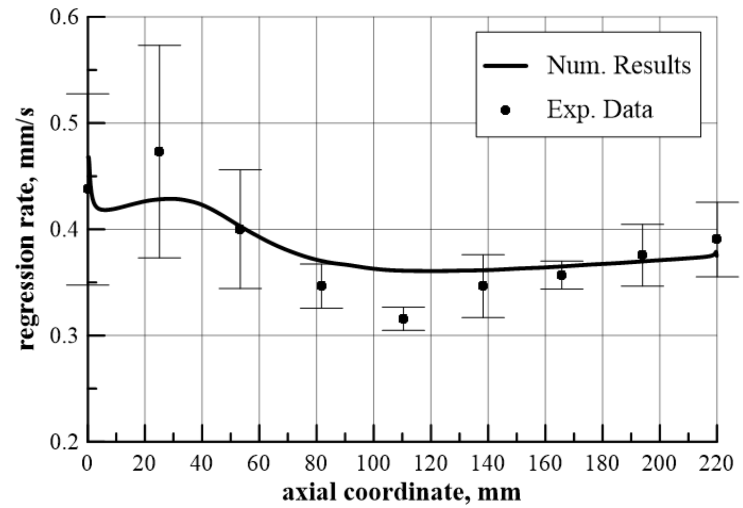
Model for the simulation of hybrid rockets internal ballistics



Pressure contour plot



Temperature contour plot

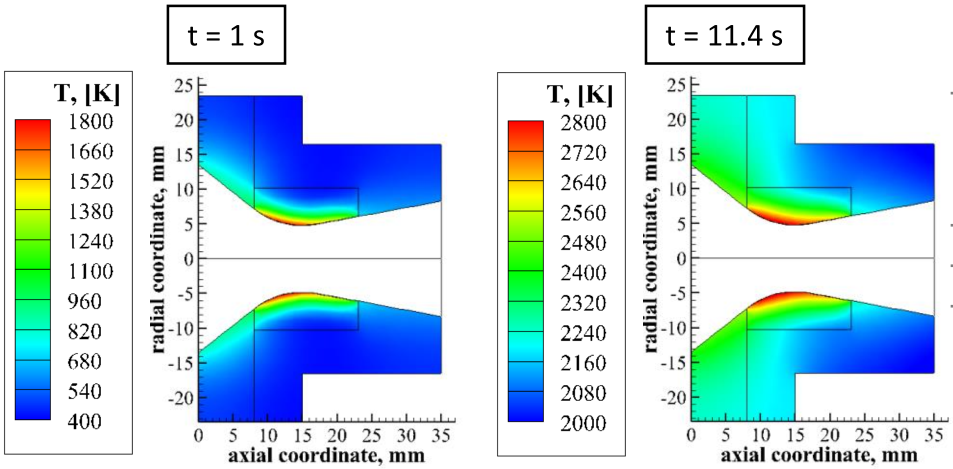


Comparison between measured and calculated after-burn port diameter

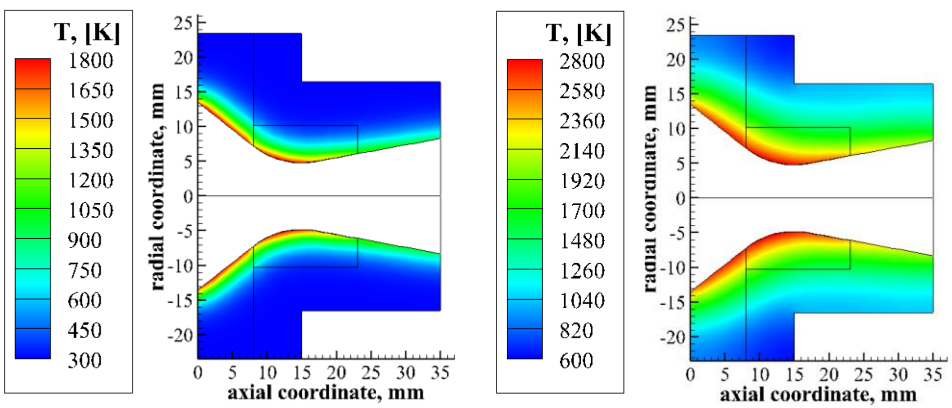
Good agreement between the numerical and experimental results, both in terms of chamber pressure (the calculated average pressure in the aft-mixing chamber is equal to 6.52 bar) and in terms of the fuel regression rate profile.

Numerical models and simulation of hybrid rocket tests

Model for nozzle thermal analysis



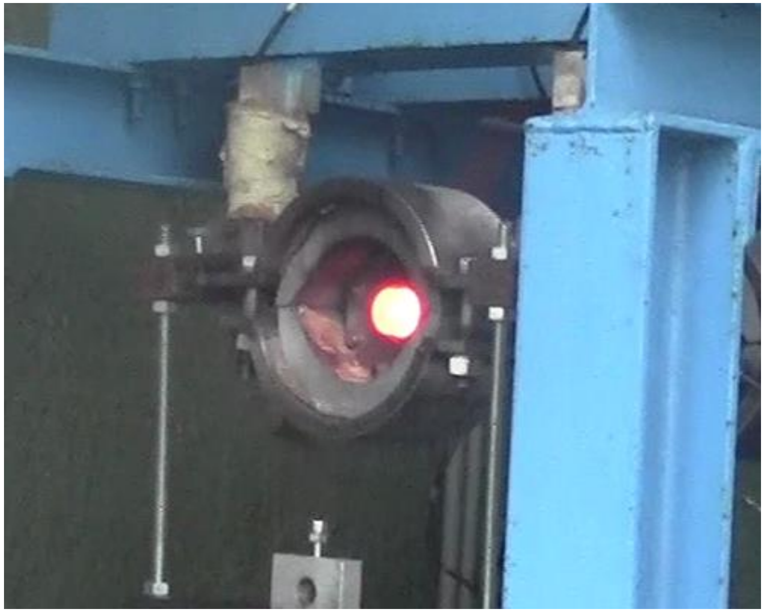
Ceramic Throat Insert



Ceramic Nozzle

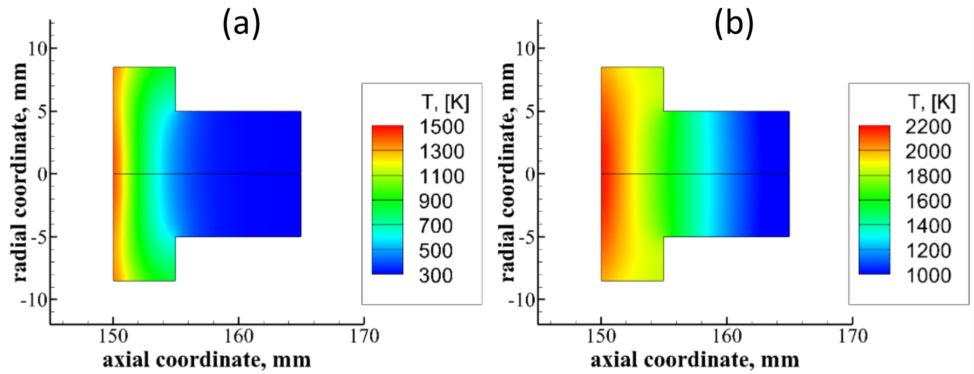
Solid materials properties

Material	Density [kg/m³]	Thermal conductivity [W/(m K)]	Specific heat [kJ/(kg K)]	Surface Emissivity
Graphite	1800	104	710	0.75
Ceramic	3210	10	750	0.8



Numerical models and simulation of hybrid rocket tests

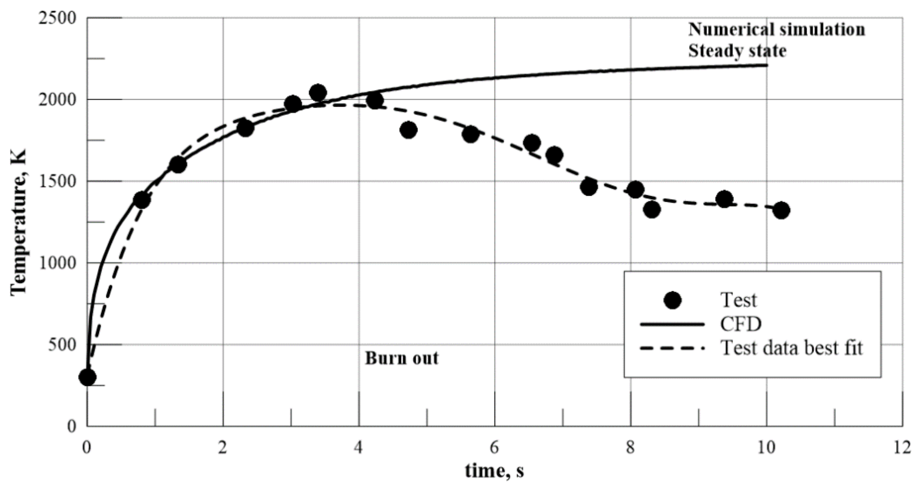
Model for free-jet exhausting rocket plume

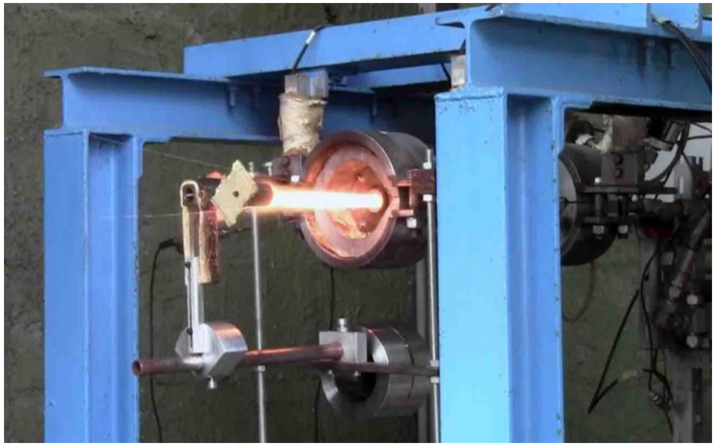


t = 1 s

t = 10 s

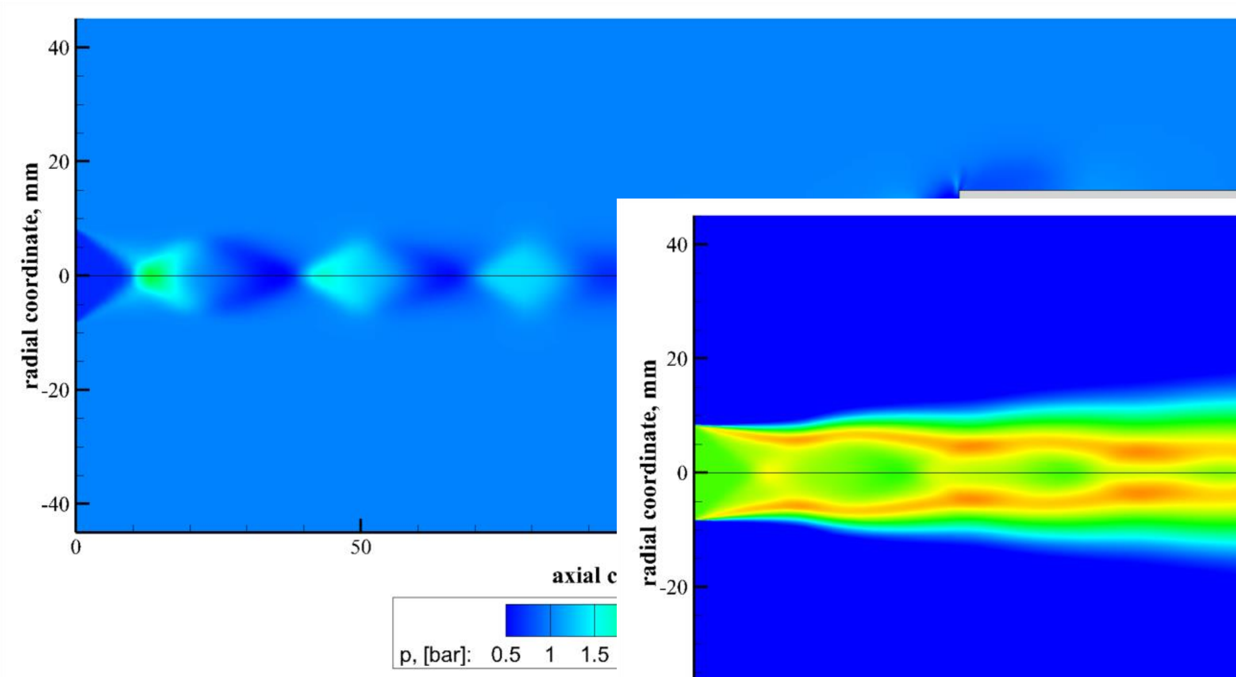
	Density [kg/m³]	Thermal conductivity [W/(m K)]	Specific heat [kJ/(kg K)]	Surface Emissivity
Ceramic tube	3000	6	900	0.8
Graphite holder and sample	1800	104	710	0.75



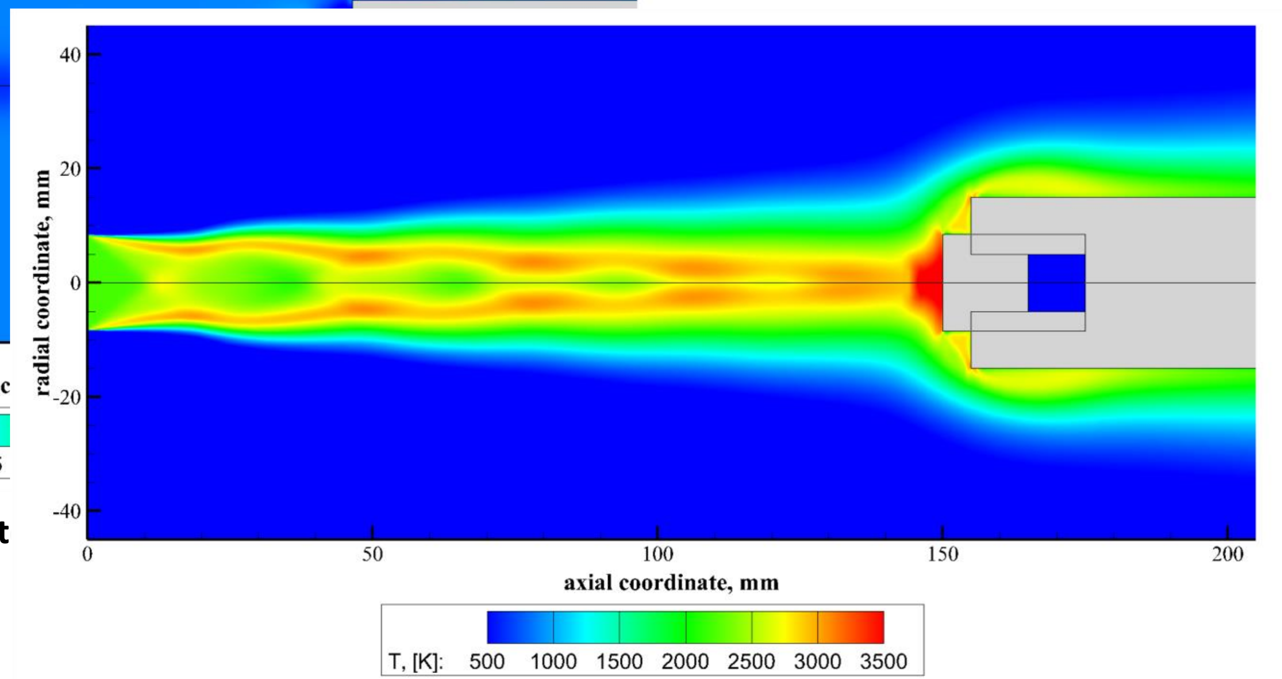


Numerical models and simulation of hybrid rocket tests

Model for free-jet (over-expanded) exhausting rocket plume



Contour of static pressure around the t



Contour of static temperature around the test article for free jet test

Conclusions

- Importance of UHTC and UHTCMC materials for application in extremely demanding environmental conditions
- Main activities at University of Naples “Federico II”:
  - Identification of test conditions and design of test articles for experimental campaigns
  - Experimental tests on material samples and components in either re-entry or combustion environments
  - Numerical modelling and simulation of reacting flows around test articles
- Experimental facilities:
  - Hypersonic arc-jet facility:
    - High total entalphy flow (>20 MJ/kg)
    - Supersonic Mach number (3-5)
    - High material temperature (>2000°C)
  - Aerospace Propulsion Laboratory:
    - Characterization of hybrid rocket performances
    - Testing of materials in combustion environment
- Current research activities in the framework of the C3HARME research project are focused on a new class of UHTCMCs

Acknowledgments

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