Ultra-High Temperature Ceramics for hypersonic entry of slender-shaped advanced space vehicles

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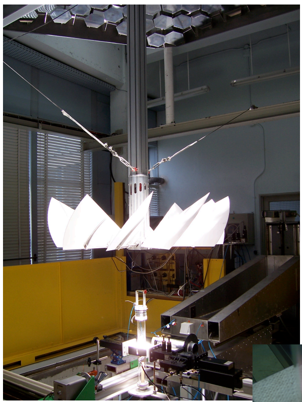
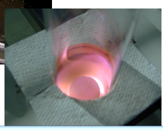
Context and goals

- Simulation of the atmospheric re-entry of reusable space vehicle at hypersonic velocity – slender-shaped hot structures – and evaluation of the catalytic recombination of oxygen atoms on thermal protection materials
- New refractory materials required: zirconium-based ceramics like ZrO_2 and Ultra-High Temperature Ceramics UHTC (ZrB_2-SiC, ZrB_2-HfB_2-SiC…) working up to 2500 K
- Evolution of the recombination coefficient γ with temperature in relation with microstructure, final shape treatment…
- No data in the literature for these materials at such high temperature levels

Experimental device and method

MESOX solar facility





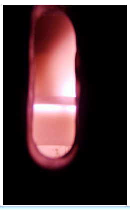
- Determination of the recombination coefficient γ by Optical Emission Spectroscopy and Actinometry using the two emission lines of O @ 844.6 nm and of Ar @ 842.4 nm; exp. cond.: P_{mw}=300 W, air flow rate=4 l/h, P=200 Pa
- Spatial resolution: 270 µm and spectral resolution: 0.2 nm

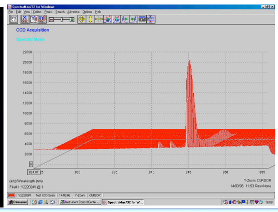
$\gamma = O_{rec}/O_{tot}$ with $0 \leq \gamma \leq 1$

$$\gamma = \left(\frac{\left.\frac{I_O}{I_{Ar}}\right|_{x=L}}{\left.\frac{I_O}{I_{Ar}}\right|_{x=0}} \cdot \frac{T_S}{T_L} - 1 \right) \frac{4.D_{O,\,air}}{V.L}$$

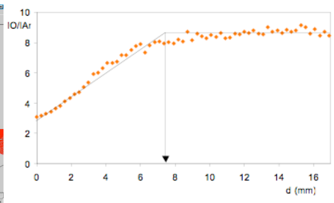
with I_O/I_{Ar} the intensity ratio, $D_{O,air}$ the binary diffusion coefficient of O in air, T_S and T_L the temperatures respectively at the surface (x=0) and far from the sample at the limit of the recombination boundary layer L (x=L) and V the mean square velocity of oxygen atoms

Images of the air plasma

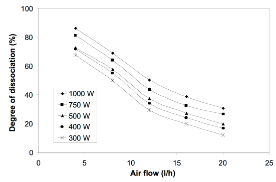




Atomic oxygen profile

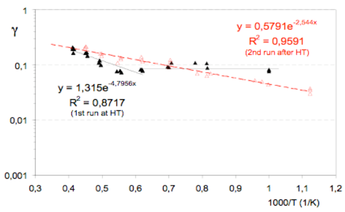


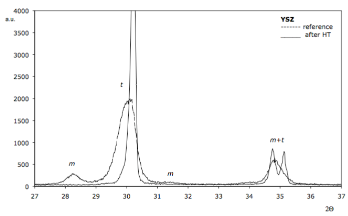
Plasma dissociation



Experimental results

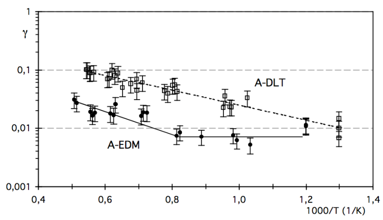
ZrO_2: influence of the microstructure

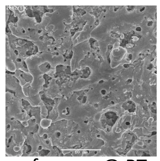




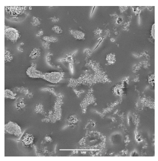
- Modification of the γ evolution with the crystalline structure of zirconia: irreversible phase transformation from monoclinic (*m*) to tetragonal (*t*) due to sintering additives (here, yttrium oxide)
- γ= 8.10^{-2} up to 1600 K before *m-t* transformation, then γ= 1.315 exp (-4796/T) with Ea=40 kJ/mol (*black triangles*)
- After stabilization of the *t* phase, γ= 0.579 exp (-2544/T) with Ea=21 kJ/mol (*red triangles*)

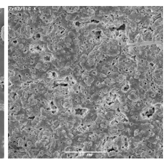
ZrB_2-SiC: influence of the surface treatment after elaboration



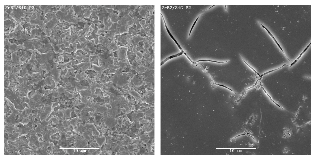


reference @ RT





1820 K mainly SiO_2 | 2000 K mainly ZrO_2

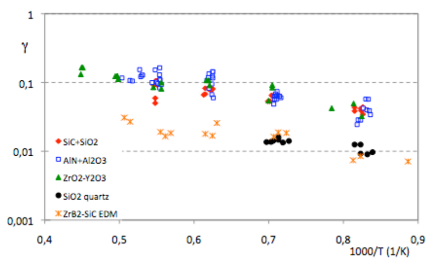


DLT: Diamond Loaded Tools | *EDM: Electric Discharge Machining*

Conclusions

- Determination of the recombination coefficient γ at high temperature on zirconium-based materials for hypersonic re-entry reusable vehicles
- Influence of the crystallographic structure and of the surface treatment during machining after the elaboration process on the recombination coefficient (EDM different from DLT)
- Zirconia-based materials present nearly same catalycity as silicon oxide (β-critobalite) and aluminum oxide: from 0.04 @ 1200 K to 0.1 @ 1800 K but can be used at higher temperatures with γ = 0.2 @ 2400 K. ZrB_2/SiC EDM presents the lower catalycity close to the one of **quartz** with possible use at higher temperatures with γ = 0.03 @ 2000 K

γ comparison for several oxides @ HT



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