

CHARACTERIZATION OF RETICULATED SI-SIC FOAMS FOR POROUS BURNERS AGED UNDER LEAN METHANE COMBUSTION

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4. Kanthal, ZN der Sandvik Materials Technology Deutschland GmbH

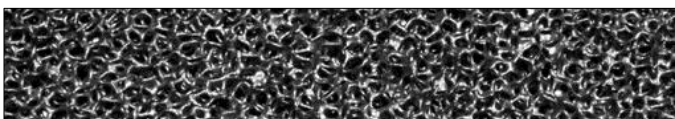
Outline



Porous Burner technology



Porous Burner application



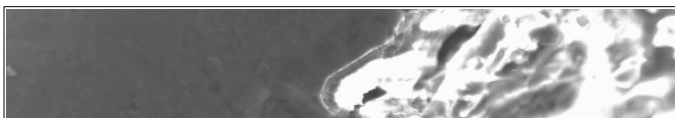
SiSiC Ceramic foam



Foam conditioning



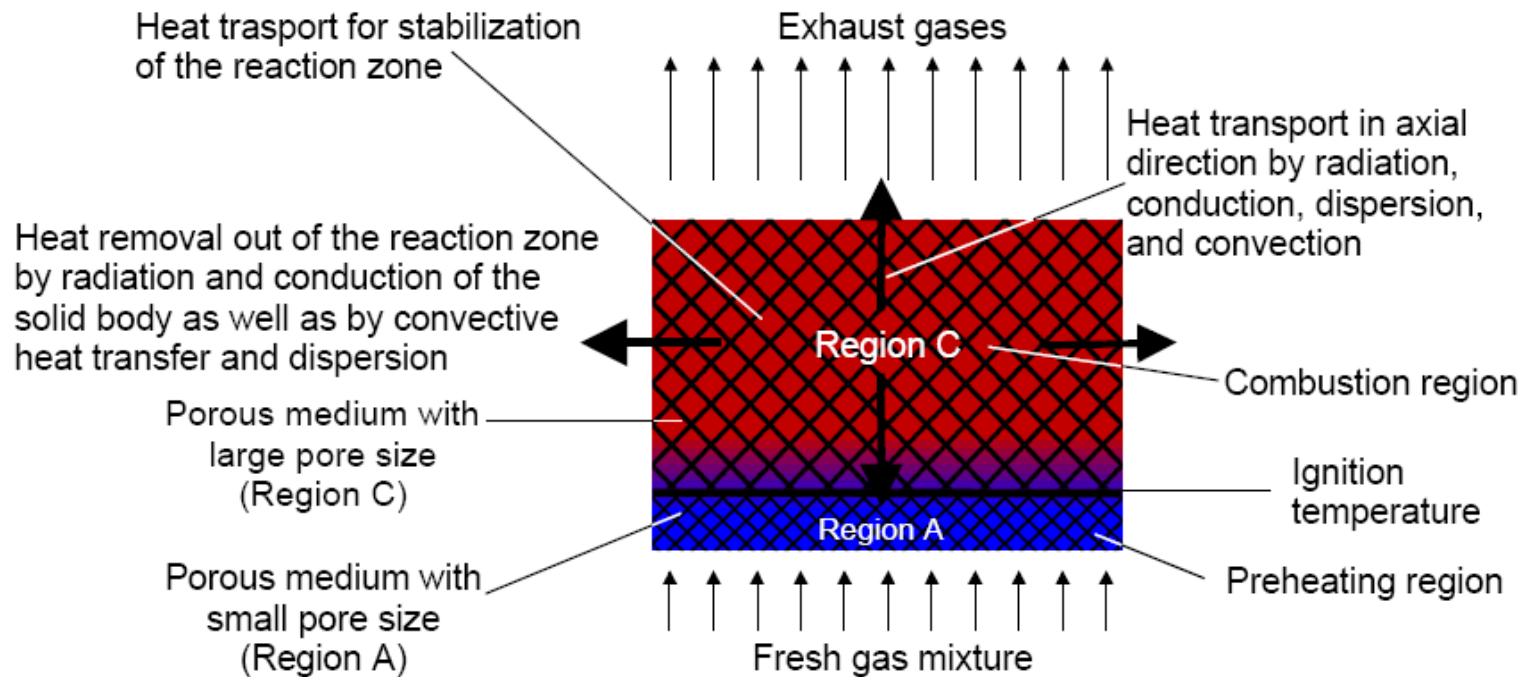
Mechanical testing and results



Further characterization

Conclusions and remarks

Porous Burner technology



POROUS BURNER RADIMAX - Use in the Paper Industry



RADIMAX test stand (Source GOGAS)

GoGaS could realize the manufacture of a burner with an extreme high thermal surface load. Compared to the conventional burners with a **thermal surface load of approx. 350 kW/m²** and a constant **temperature of 1100 °C** the porous burner **RADIMAX** reaches a temperature of 1450 °C at a thermal surface load of more than 1000 kW/m².

ErbiSiC foams

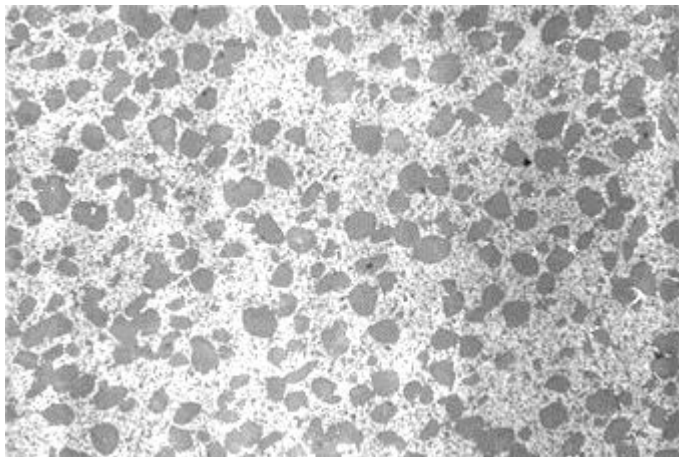


ErbiSiC-R foams from **Erbicol SA** (CH) are made via the **Schwarzwalder** production technique to make replica ceramic foams. A starting polymer, such as foamed polyurethane, is impregnated with a ceramic slurry, pyrolysed and infiltrated with molten Silicon, at high temperatures in a vacuum furnace.



K. Schwartzwalder, A. V. Somers: Method of making porous ceramic articles, US patent No. 3090094, 1963

ErbiSiC foams



10 PPI Foam properties

Foam density [g/cm ³]	0,41
Normalized density	0,14
Av. Strut Thickness [mm]	1,012
Flexural strength [MPa] (3 point bending)	3,34
Compression strength [MPa]	7,275
Strut density [g/cm ³]	2.85
Free Silicon	0,30

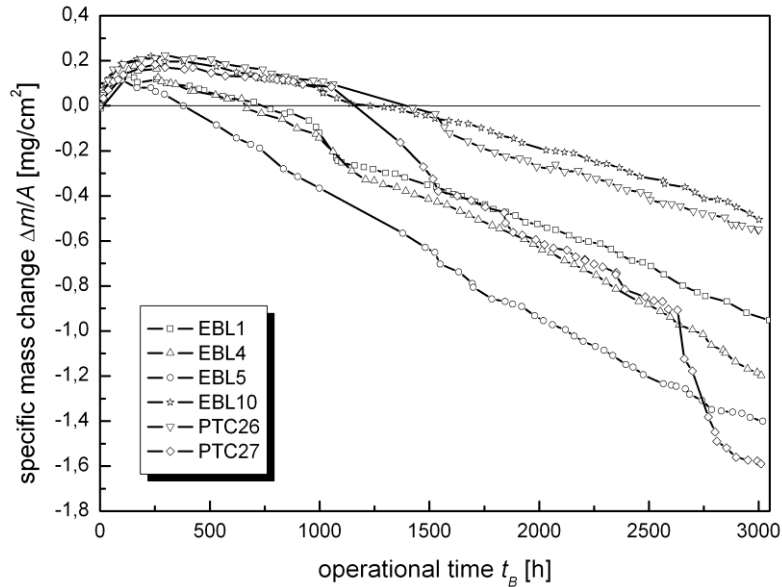
SiC foam durability studies



Testing laboratory at LSTM

For the long term test, an automated test rig consisting of twelve independent porous burners with three different reference burner geometries were constructed. There are **6 burners with a rectangular geometry** of 175 mm x 135 mm, 3 burners with ceramics of a diameter of 130 mm and 2 burners with ceramics of a diameter of 80 mm. **The burner system is assembled with well known and tested household heating components.** For each burner the power, the equivalence ratio and the running time is adjustable.

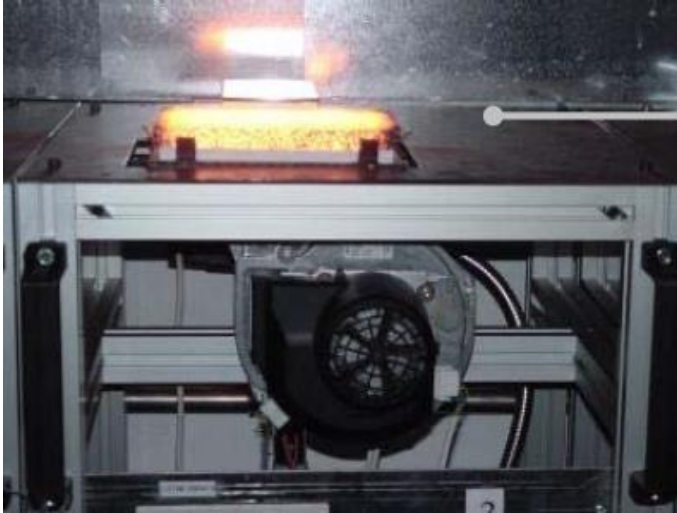
SiC foam durability studies



Mass change during operation

In a former research project (**CERPOR**), during cyclic oxidation using the porous burner test rig, the mass change of the Si SiC samples was monitored. **A common behavior of a mass gain at the beginning, followed by a mass loss** was observed for all the foams. The mass gain, as discussed in previous sections, is due to the **passive oxidation**, in which a solid phase accumulates on the foam surface, increasing its mass and protecting it from further oxidation. The mass loss can be attributed to both **active oxidation** and **spalling**.

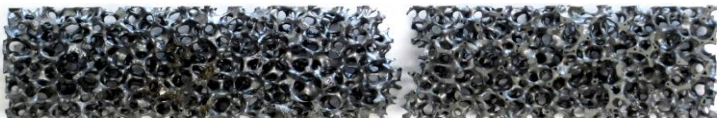
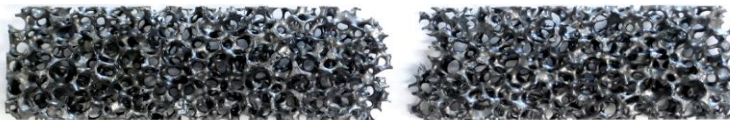
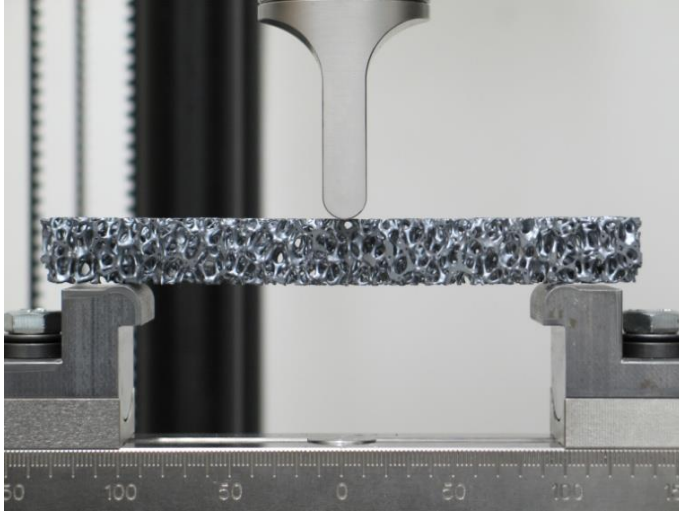
Burner test rig



Power	25 kW
excess air ratio	1.3
Temperature	1400 °C (<i>inferred</i>)
Powe ON	10 min
Power OFF	1 min
Ageing	1, 5, 10, 50, 100, 1000 h

Burner rig scheme

Bending tests



Bending was chosen because that is a **loading condition when the manufacturer fixes foams onto the burner** with a metallic net.

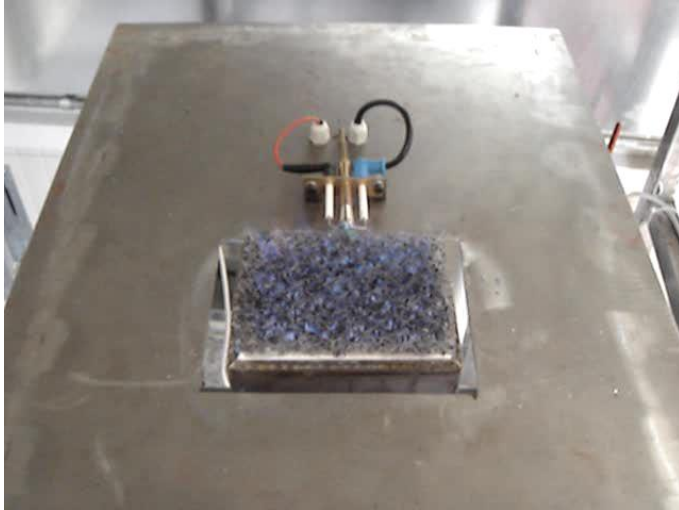
Three point bending tests were performed with a Zwick/Roell Z050 (Germany) using a span of 100 mm. Sample dimensions (139x25x15,5 mm³) were chosen trying to match the actual burner geometry with those from similar standards*. Samples have circa **three cells along the specimen thickness** when the lower limit of the standard is 5.

For this reason these values are valuable only for **relative comparison**.

Since was not possible to have a reliable strain measures, only force was acquired during tests, cross head speed was of 0.01 mm/s.

* ASTM C1674 - 08 Standard Test Method for Flexural Strength of Advanced Ceramics with Engineered Porosity (Honeycomb Cellular Channels) at Ambient Temperatures

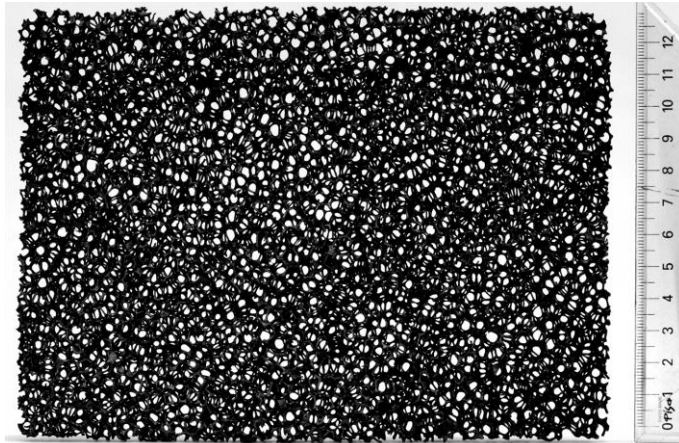
Ageing campaign



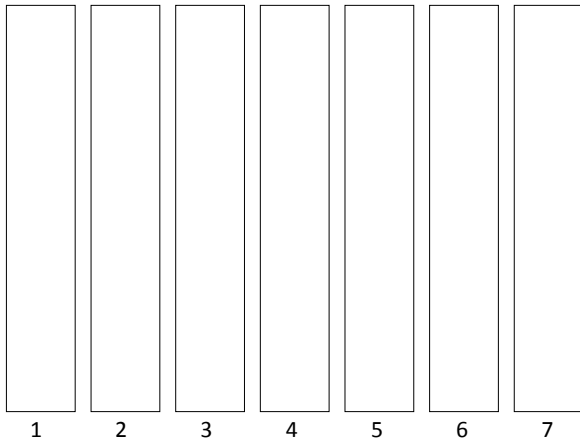
Burner start up and shut down

Foam Number	N Cycles	Aging [h]	Mass before test [g]	Mass after test [g]
32	1	0.33	154	154.13
33	2	1.00	153.7	153.72
35	3	5.00	147.42	147.63
36	15	11.50	143.89	144.11
37	35	51.00	168.74	169.11
38	153	99.50	154.88	155.04
39	3000	1000.00	n.d.	148.40

SiC foam durability, mechanical degradation studies

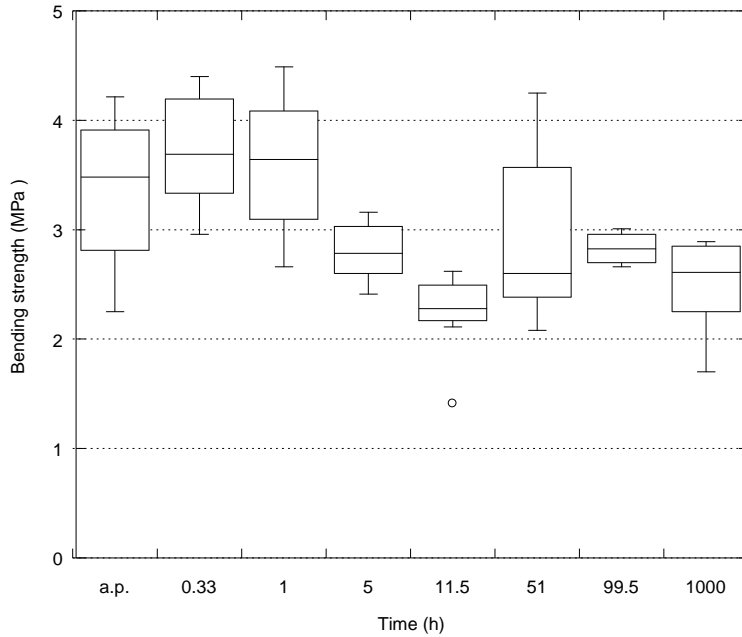


As prepared foams ($139 \times 185 \times 16 \text{ mm}^3$) were rectified on the $185 \times 139 \text{ mm}^2$ faces and cut with diamond tools in **7 pieces** ($139 \times 25 \times 15,5 \text{ mm}^3$). The first two dimensions have quite tight tolerances ($\pm 0,05 \text{ mm}$) because of machining while the third dimension was kept as produced and thus has a $\pm 1 \text{ mm}$ tolerance. Foam were thus carefully **re-assembled** and placed onto the burner rig.



Samples preparation

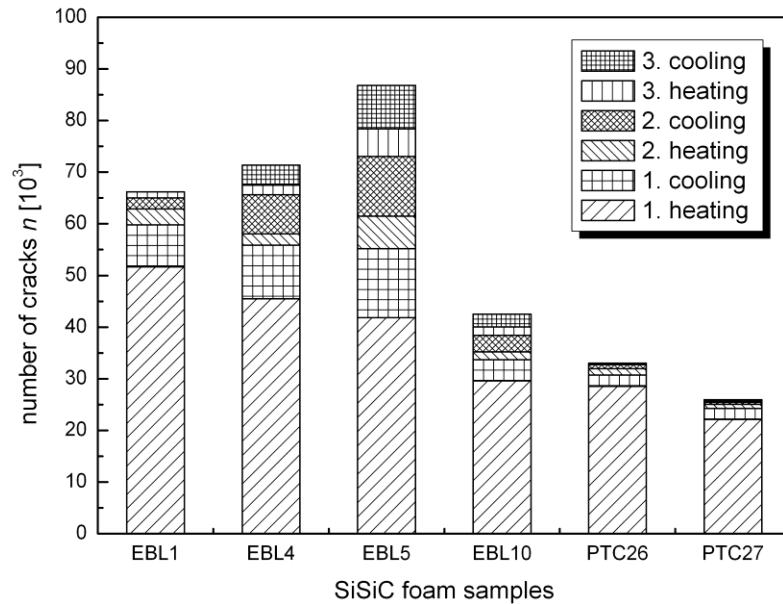
Bending tests results



Foam	Rel.Dens. ρ/ρ_s	Aging (h)	# samples	Minimum	Maximum	Mean
a.p.	0.14	0.0	7	2.25	4.21	3.34
32	0.15	0.3	7	2.96	4.40	3.73
34	0.14	1.0	7	2.66	4.49	3.59
35	0.14	5.0	6	2.41	3.16	2.80
36	0.13	11.5	7	1.41	2.62	2.23
37	0.13	51.0	7	2.08	4.25	2.98
38	0.16	99.5	4	2.66	3.01	2.83
39	0.15	1000.0	7	1.70	2.89	2.49

Bending strength vs ageing time

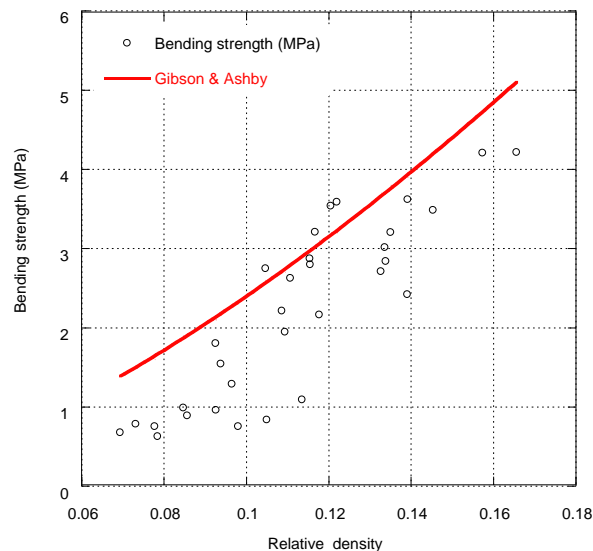
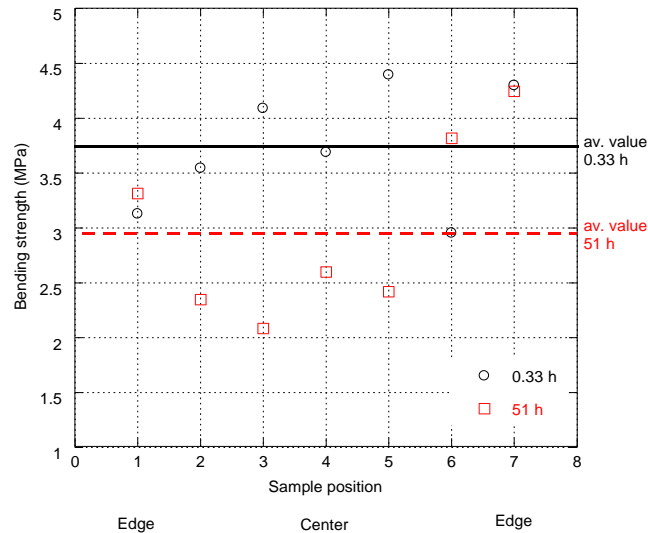
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Total number of cracks obtained during the initial three heating-cooling cycles

Bending tests results

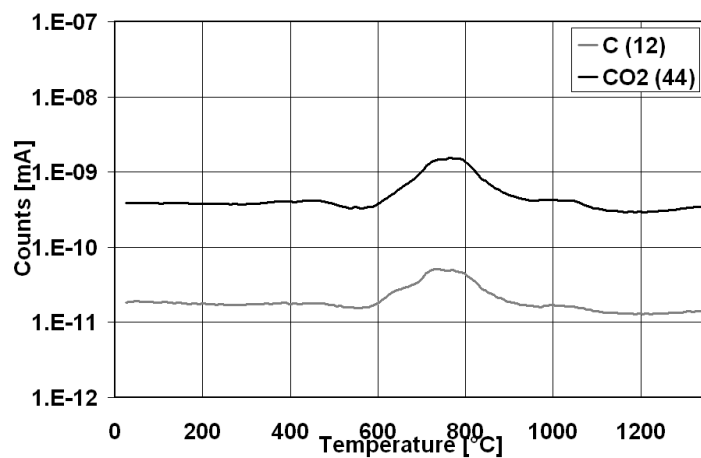
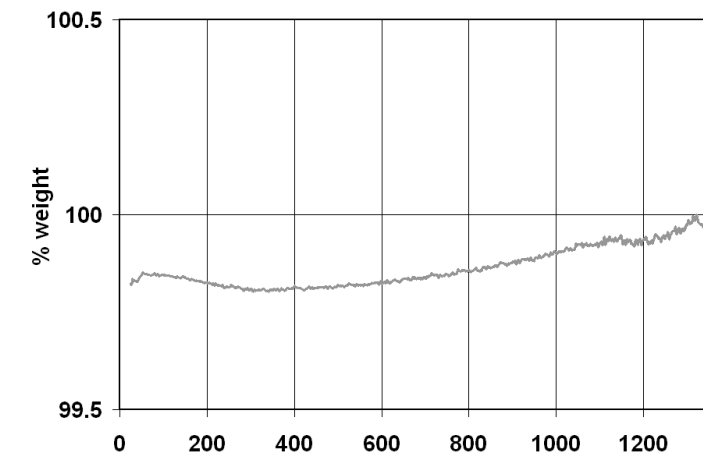


Within the burner, central samples (#4) in long-aged foams seem to have lower mechanical properties than lateral ones (#1,#7) that is probably due to local active oxidation phenomena, but in general **sample position within the burner, does not seem to be more significant than other factors affecting foams bending strengths.**

These are: cell shape and orientation, strut thickness, strut cross-section, strut cavity sharpness.

three point bending strength of un-aged Si-SiC foams are compared with those calculated from the Gibson and Ashby model which does not consider local defects.

TG/MS Analysis



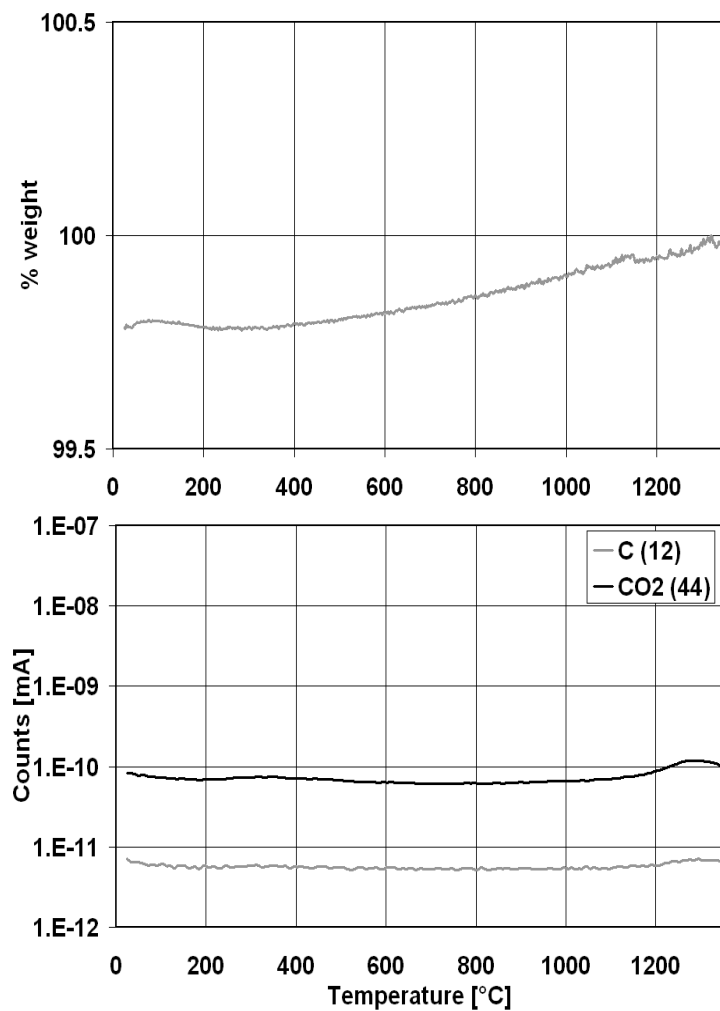
The thermal stability evolution has been tested by using TG analyses coupled with mass spectrometer.

The measurement has been performed in a first step on the as produced material.

It is possible to note that little weight variation is appreciable in 25-1350°C test range.

In the same range low CO₂ emission is detected especially close to 800°C.

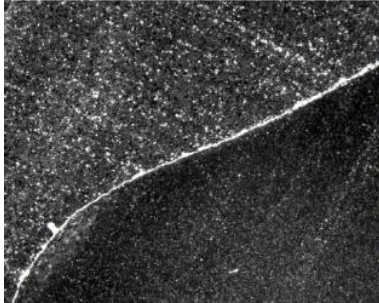
TG/MS Analysis



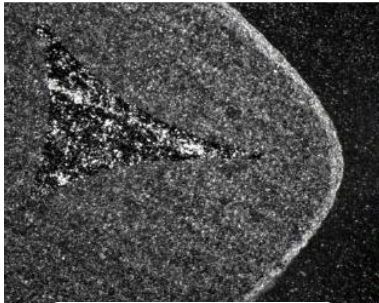
The measurement of thermal stability has been performed also on samples after ageing (1000 h at 1400°C).

There is no difference against the behavior of untreated foams.

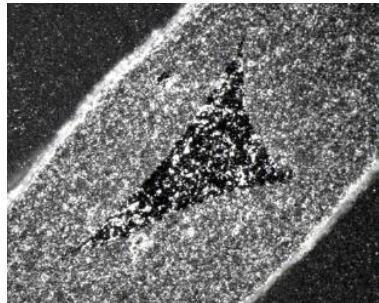
Optical microscopy with darkfield illumination



As produced – 250X



100h – 250X



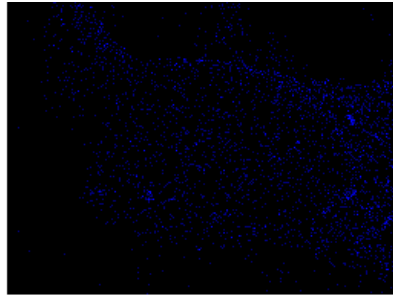
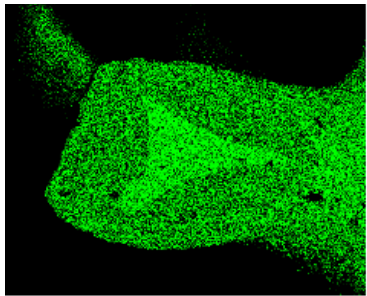
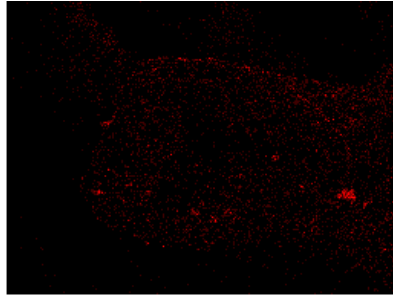
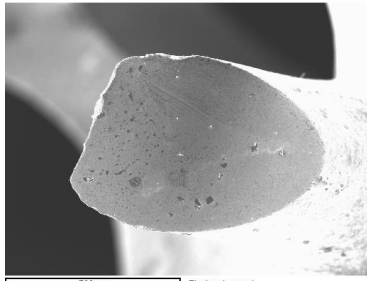
1000h – 250X

Silica formation is uneven throughout the foam. That is because of the different oxidation phenomena that occurs into the porous body.

These pictures depict an average silica layer for different ageing.

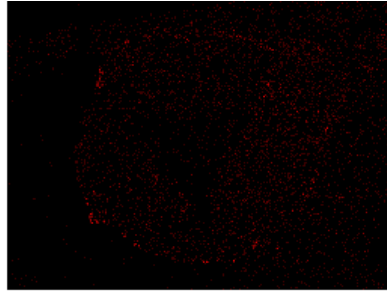
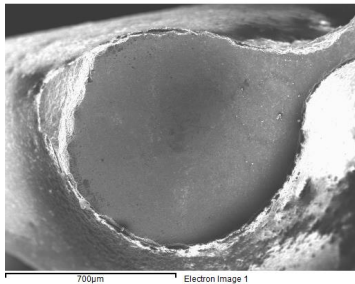
As said the **scale thickness increases during the first 1000 h.**

SEM-EDS analysis

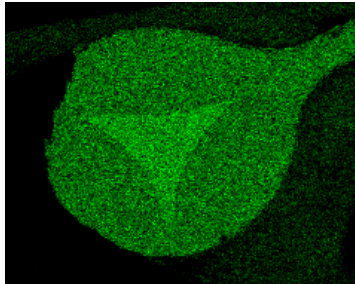


Morphology of a polished cross section of untreated sample

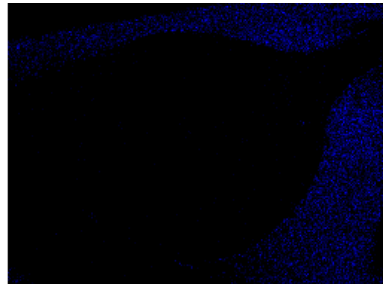
SEM-EDS analysis



C Ka1_2



Ka1

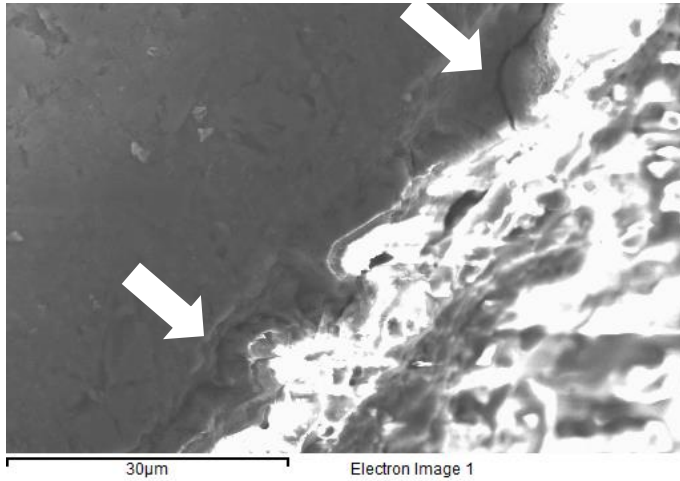


O Ka1

Morphology of a polished cross section of a foam aged at 1400°C for 1000 hrs.

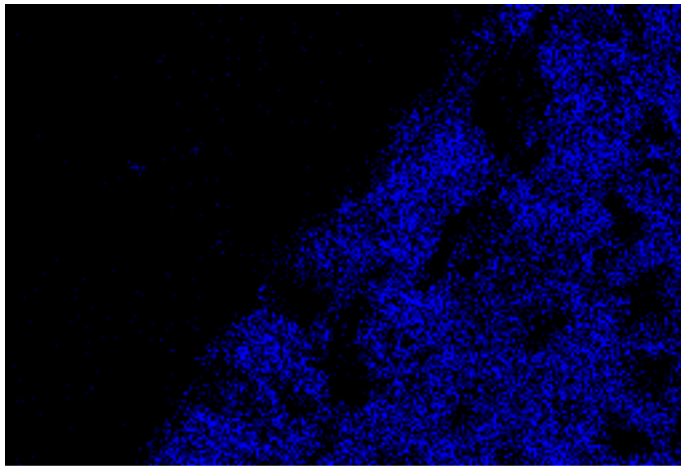
There are no differences with the untreated one

SEM-EDS analysis



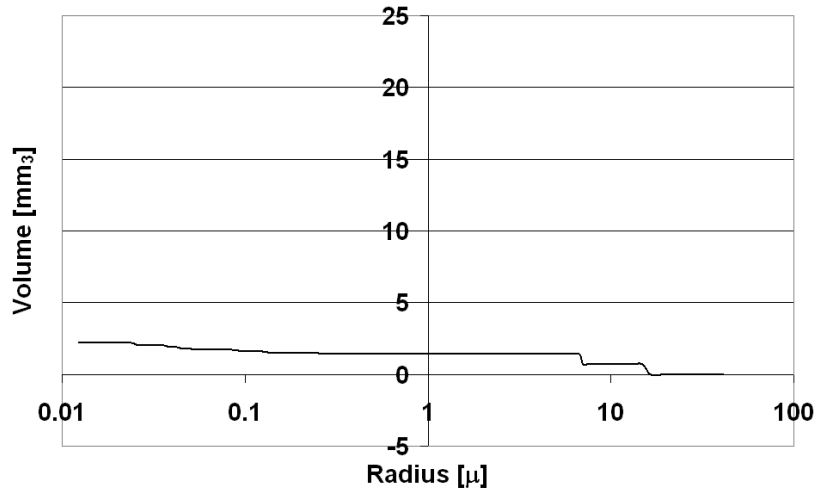
Foam aged 1000h

Beginning of silica scale disconnection.



Porosity

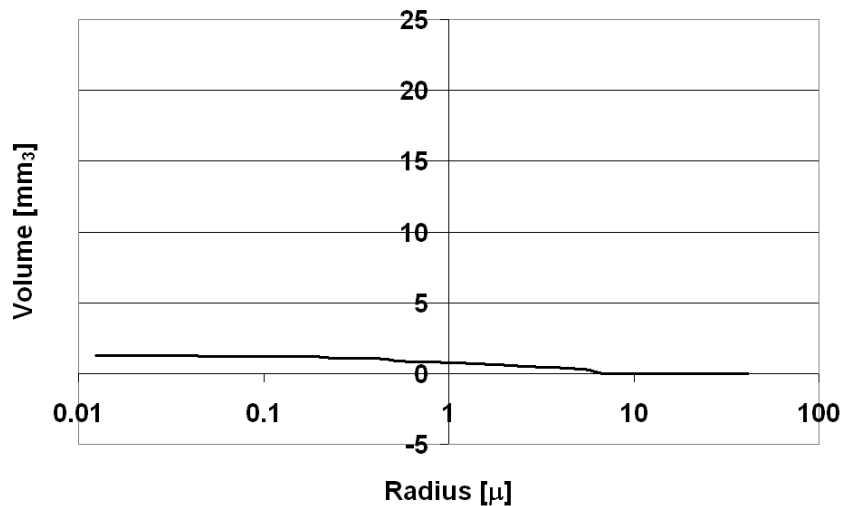
Sample untreated



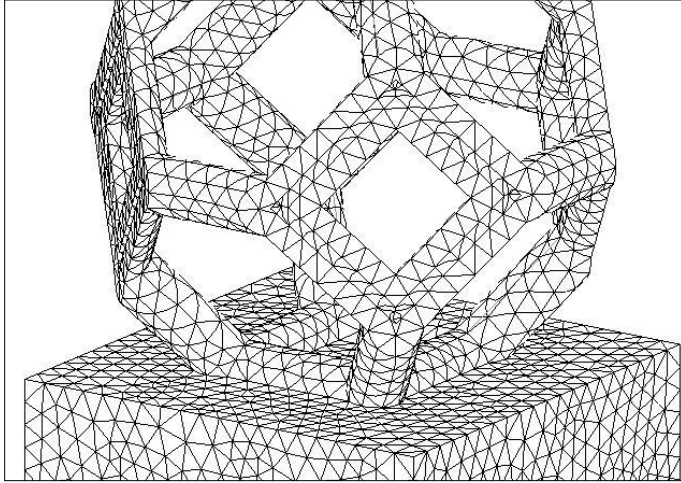
The mercury intrusion porosimetry put in evidence the very little surface area of the samples.

Also after thermal degradation of the surface due to the high temperature exposure the surface of the foams is very smooth.

Sample treated 1000 ° at 1400°C

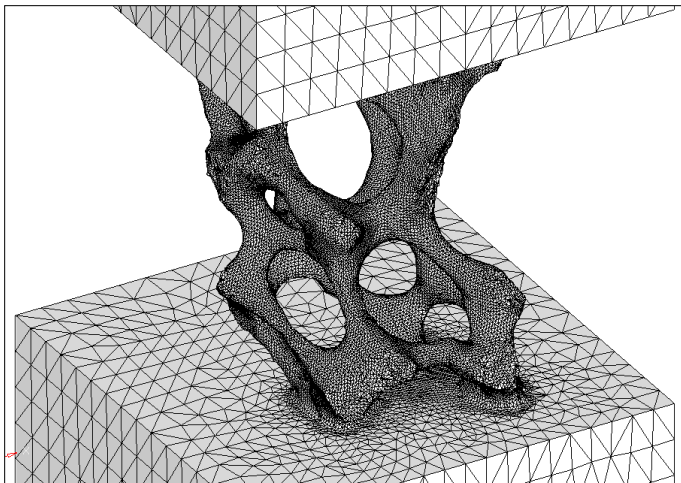


Further work



Foam behavior simulation

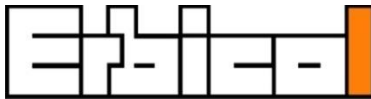
- Direct tetrakaidecahedron simulation



- Real foam meshing
 - X-ray micro CT
 - Image processing
 - Meshing
 - Mesh conditioning
 - Simulation

Acknowledgements

Authors wish to thank:



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GoGaS GmbH, Dortmund, D

which provided the porous burners

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- Politecnico di Torino, Dipartimento di Scienza dei Materiali e Ingegneria Chimica, Torino, Italy.
- Institute of Fluid Mechanics, University of Erlangen, Cauerstr. 4, D-91058 Erlangen, Germany