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Design and Additive Manufacturing of Periodic Ceramic Architectures

G. Bianchi[2], S. Gianella[1], A. Ortona[*2]

[1]EngiCer Sa, Balerna, Switzerland
[2]MEMTI - SUPSI, Manno, Switzerland

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Abstract

Cellular ceramics are attractive material solutions for high-temperature applications thanks to their outstanding properties. Although ceramic foams are already widely employed in industry, they exhibit scattered properties because of their randomness and fragility. Moreover, there are only few parameters that can be varied in order to engineer their properties.

We begin with finite element simulations to show how periodic cellular ceramics can be designed to meet user requirements.

Then, thanks to additive manufacturing (AM), the numerical domains can be readily transformed into physical objects. Complementing the many AM techniques available today, we developed an original method in which polymeric lattices templates are produced by means of 3D printing, further coated by replica with ceramic slurries and finally heat treated. The advantage of this technique is its flexibility. Practically any ceramic material already produced in bulk form can be realized.

We then present some case studies in which we were able to design and produce components for concentrated volumetric solar receivers, thermal protection for aerospace entry vehicles, and heat exchangers.

Keywords: Additive manufacturing, cellular ceramics, high-temperature applications, periodic architectures

I. Introduction

Porous ceramics are a category of hybrid materials composed of a ceramic constituent and empty spaces usually filled with a gas [1]. This material architecture endows the resulting structure with a unique property-to-density ratio [2]. Such material configurations are indeed quite common in nature, often related to organic components. Examples of natural porous solids [3] are bones, wood, sponges and pumice stones formed during volcanic eruptions. Engineers have adopted these structures, made of different materials, for many applications such as hot and cold filtration [4, 5], noise reduction [6], shock absorption [7], mechanical [2], thermal insulation [5] and wide-ranging high-temperature applications [8], some of which are detailed in the following paragraphs.

In this paper, we shall focus on hybrid materials characterized by a continuous ceramic solid phase and macrovoids [9], and particularly on macroporous SiC [10] on account of its outstanding high-temperature properties. Ceramic foams can be produced in several ways [11, 12], the principal methods being direct foaming, gelation-freezing, burnout of fugitive pore formers and, last but not least, replication of a sacrificial plastic foam [13]. A plastic foam (usually polyurethane) is soaked in a slurry composed of ceramic powders, plastic binders and solvents. Then the excess slurry is removed by squeezing. The result is a green foam with a layer of slurry around its struts. This green body is then thermally treated (sintering for oxides) to obtain the final component. In the case of SiSiC, the body is pyrolysed at 1000 °C in an inert atmosphere and then infiltrated with molten silicon in vacuum at 1500 °C [14].

Slurry replication [13] of oxide ceramics is by far the most industrialized method, its huge development is due to the filtration of molten metals for the metal casting industry [15, 16]. Millions of pieces are used every year to filter different metal alloys before these are cast.

In applications such as the one described above, the randomness of a ceramic foam architecture is a huge advantage [17–19].

On the other hand, precisely this randomness can be a limiting factor. The polyurethane foams used as templates are derived from a liquid phase in which, before its polymerization, gas bubbles of different size grow inside. Because of that, the struts, i.e. the skeleton of these foams, are random in shape, length, distribution and orientation in the space (Fig. 1). As a consequence, the local properties of the porous body change dramatically [20] over its volume. The foam properties are thus calculated using homogenization techniques in which the selected volume has to be rather large in terms of number of pores in order to be treated as representative [21–23]. Moreover, using homogenization techniques do not take account of the boundaries at which cells and struts are interrupted by machining: this operation has a huge influence on the overall properties of the macroporous ceramic component with a discrete volume [24, 25] (Fig. 1).

* Corresponding author: alberto.ortona@supsi.ch

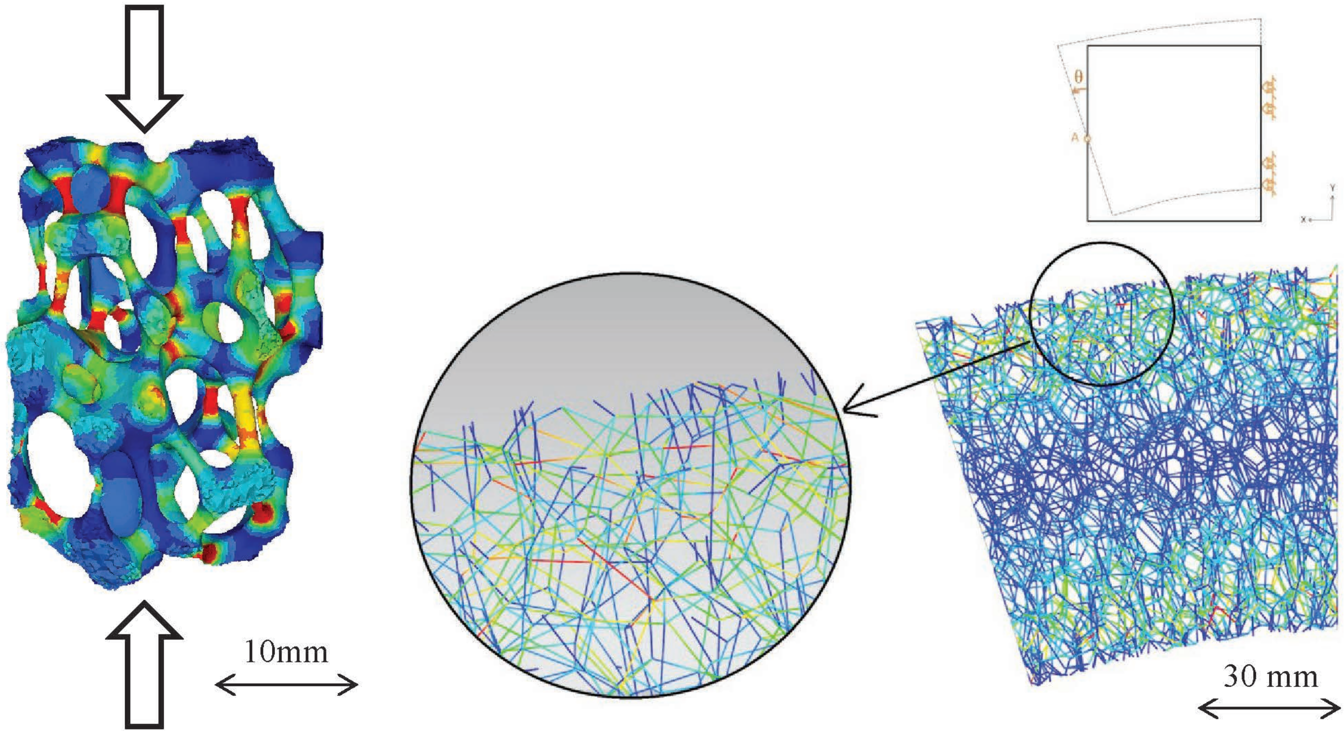

Fig. 1: Stress distribution (blue low, red high) from the FEM analysis of a random foam under compression (left) [23] and bending (right) [26].

Depending on user requirements, to be fully reliable, macroporous ceramics components should be realized as structures with a repeatable architecture. With random foams, this is impossible.

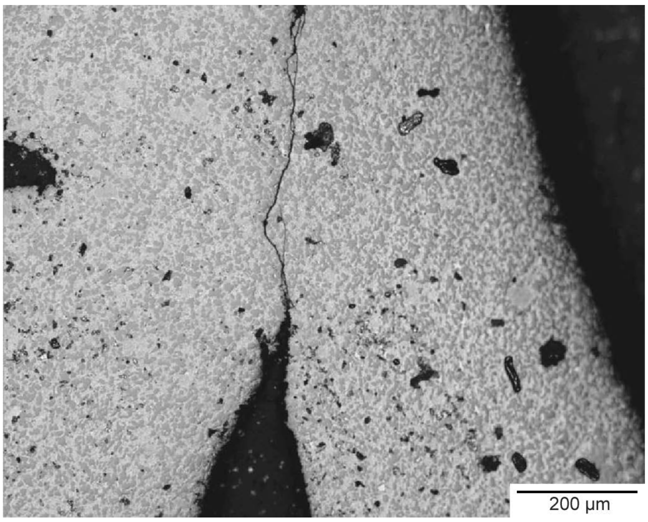

Fig. 2: Crack propagating from the sharp corner of a hollow strut produced by replication of a polyurethane foam.

Another issue with random ceramic foams is the shape of their struts; this has huge consequences for the foams' mechanical properties [23]. Replication is accomplished employing polyurethane foams which, because of their manufacturing process [5], have struts with piercing corners. At the end of the process each ceramic strut presents a hollow shape with sharp corners imposed by the former polyurethane foam. Sharp corners act as notches from where, under mechanical or thermal loading, a crack can easily propagate (Fig. 2) [27].

All these issues could be resolved with a periodic cellular architecture with smooth, regular and interconnected struts. This need was met with the advent of additive manufacturing.

Additive manufacturing or 3D printing is a near-net shape production technique developed several years ago that allows the assembly of a material starting from a CAD model. The main advantage of this technique is that it allows the direct production of components with a shape that is impossible to realize with conventional manufacturing methods. This is especially true for porous materials. At present, this technique has reached industrial maturity for metallic and plastic materials while it is still at the development phase for ceramics. Our idea was thus to combine plastic AM, the most mature method, with replication [28].

The following paragraphs in this paper will describe how CAD files of cellular architectures are generated and how they can be converted into ceramic objects. Finally, some applications in different industrial fields will be reviewed.

II. CAD Lattice Generation

Lattice structures can increase the properties of a porous material by orders of magnitudes [1, 29]. The repetitive unit cell of these structures (Fig. 3) can be designed from topological studies (tetrakaidecahedron), inspired from crystallography (octet), or developed specifically (Gibson and Ashby cell) [2].

In Fig. 3, it can be seen immediately that the first two structures already solve the problem of having unconnected struts (Fig. 1), this feature improves the mechanical properties of the resulting structures [20]. The next advantage is that the struts can be designed to overcome the problems highlighted in the introduction [22, 27, 30]. We test-

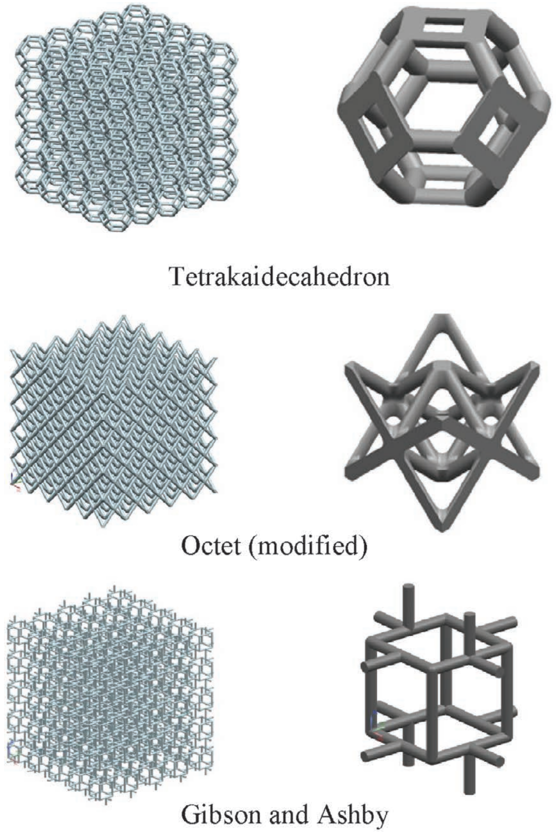

Fig. 3: Different unit cells and the relevant lattices obtained by means of CAD modelling.

ed several strut shapes, but the easiest solution is to assign to the strut a cylindrical shape which will be, depending on the following transformation technique, a tube or a solid bar. If for any reason the cell chosen does not allow the formation of unconnected external struts, we have developed a software tool which cuts the numerical domain while keeping the struts connected (Fig. 4). To do this, solid lattices were made and cut into the desired form (Fig. 4, left). An NX (Siemens, Munich, D) application-programming interface (API) was used together with a MATLAB script (The Mathworks, Inc., Natick, MA) to convert all the nodes and edges into spheres and cylinders (Fig. 4, right).

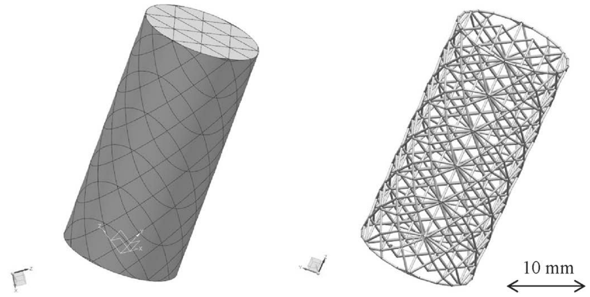

Fig. 4: Numerically solving the problem of unconnected struts in a rotated cube configuration adapted to fit a cylindrical volume (Ehsan Rezaei's PhD thesis – ongoing).

If the problem is to fit a given volume with cells allowing distortion of the cells, another MATLAB script can solve it (Fig. 5). This code receives a meshed structure as the input and converts the structure into any of tetrakaidecahedron, cubic or octet lattices. Basically, this code can be further developed for any type of cell that can fit into a hexahedron.

Once the numerical model has been designed, it can be utilized to simulate its behaviour in different applications. Mechanical, thermal, fluid dynamic, electromagnetic simulations have been used to optimize the performance of a porous component [17, 20, 22, 23, 25, 30–32].

III. Additive Manufacturing of Periodic Cellular Architectures

There are several methods to build a porous ceramic architecture with AM [33, 34]. Two different techniques were utilized in this work to transform a CAD file into a ceramic periodic cellular architecture. They can both be considered as two-step manufacturing methods, in the sense that 3D printing was utilized to produce green bodies that were subsequently thermally treated to transform them into ceramics. We are studying these two techniques because they are complementary in terms of printing volume capability, resolution and ceramic powder choice.

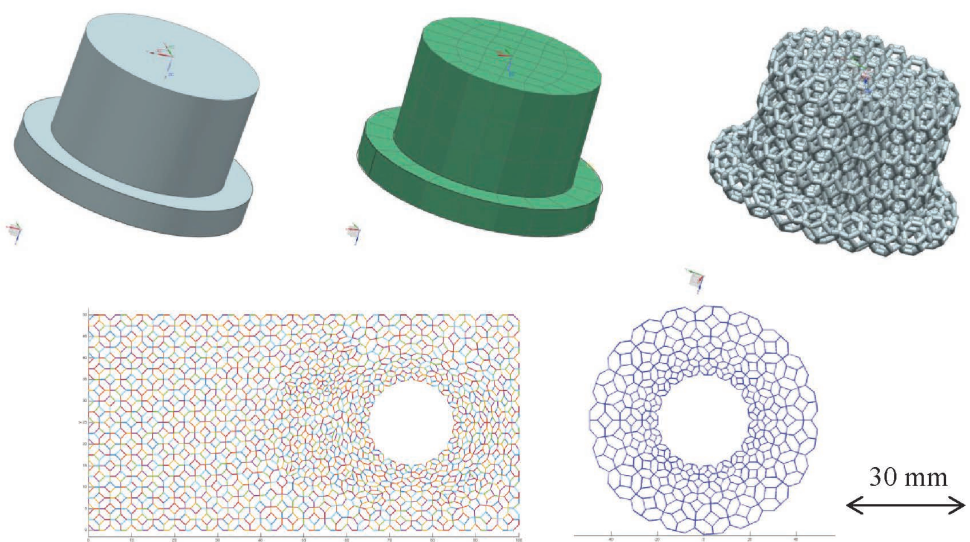

Fig. 5: A numerical way to mesh a specific volume with a given unit cell (a tetrakaidecahedron in this case) (Ehsan Rezaei's PhD thesis – ongoing).

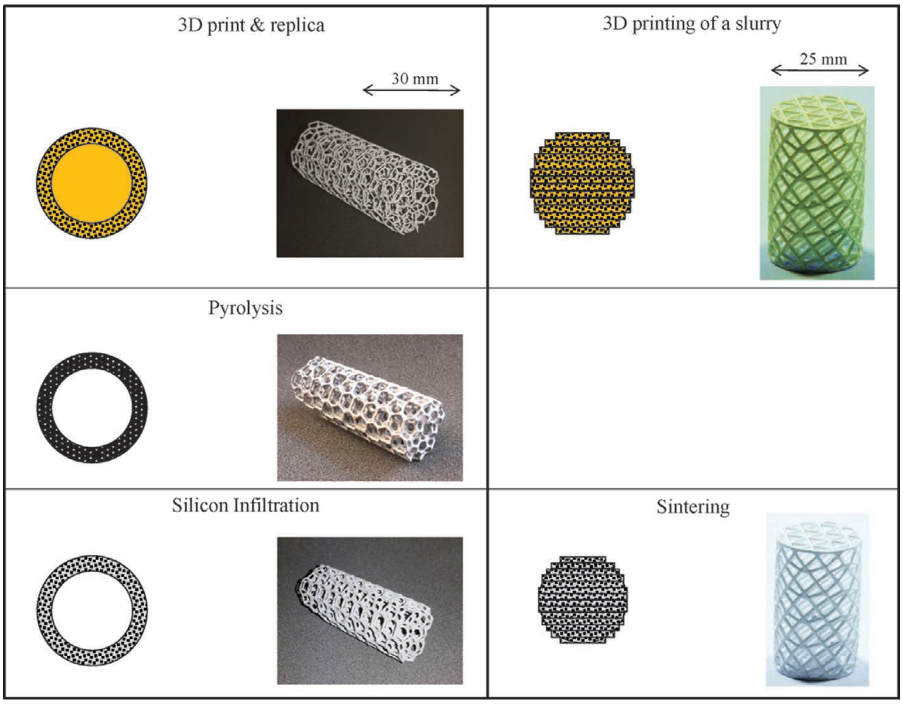

Fig. 6: A representation of the two AM processes described in this paper: a sketch of a generic strut cross-section along with pictures of two samples. On the left, the replication and thermal treatments of a 3D-printed plastic template realized from a CAD file. On the right, the production steps of an Al_2O_3 periodic architecture obtained by printing a slurry.

(1) 3D printing and replication

A plastic periodic architecture is 3D-printed by means of stereolithography and then used as template for a subsequent replication process (Fig. 6 left) [13].

This simple method allows the production of virtually any pore architecture with different slurries and thus different final ceramic materials [28]. SiSiC ceramics have been produced by means of molten silicon infiltration of carbon-bonded SiC particles but, as an example, graphite-based architectures can also be produced [35]. For details regarding materials and methods relevant to this technique, we encourage interested readers to refer to our previous work [28].

The advantages of this method are that large bodies can be realized, the material added by means of replication wraps the sacrificial template, endowing the final porous architecture with improved mechanical properties [11, 28] and finally the surface of the ceramic end-product is very smooth.

Drawbacks are also present and are described in the following.

There are dimensional deviations of the strut shape from the nominal CAD file owing to the replica deposition phase, which is gravity dependent because of the viscous movement of the slurry before its consolidation. Slurry viscosity is the cause of probably the biggest disadvantage of this technique: owing to its high values (because of the high solid loading) small windows, and thus small cells, will be occluded. Since one feature of a porous body in several applications is its large surface area (which exponentially increases with the decrease in the cell size), this technique cannot be applied to produce porous bodies with a surface area larger than 2000 $m^2 \cdot m^{-3}$.

One peculiarity of the replication process, which can be considered as an advantage or a disadvantage depending on the application, is that the ceramic struts are hollow owing to the disappearance of the polymeric template on pyrolysis.

(2) Stereolithography

The slurry recipe comprises photopolymeric resin TPGDA (Allnex, Luxemburg, Luxemburg) mixed with ceramic powders (average diameter 2 μm) 42 vol% (Nabaltec, Schwandorf, Germany) and UV photoinitiator Irgacure 819 1.2 wt% (BASF, Ludwigshafen, Germany). The slurry was thoroughly mixed by means of ball mixing for 24 h with zirconia balls D = 8.5 mm, 30 vol% [36]. The green cellular architectures were then built layer by layer (Fig. 6 right), projecting with UV light a sequence of images with a dark background obtained by slicing the CAD file of the object. Printing was performed with a 3DL Printer – HD 2.0 (Robot Factory, Mirano, Italy). The parameters were set as following: slice thickness: 0.05 mm, slice exposure time: 1.1 s. This technique is well suited for the production of porous bodies because, when these are sliced, the projected area to be cured at each shot is confined to small regions. A well-cured, monomer-free slurry with highly packed and well-dispersed powders will result in a denser layer-less bulk ceramic after sintering [37–39]. 3D printing of large bulk ceramics with low surface-area-to-volume ratio (e.g. a sphere) is indeed the most challenging goal at present. The advantages of this technique are: final geometries (provided that the distortions upon sintering are limited) do adhere to their nominal CAD geometries, small features can be printed (the present resolution is in the range of tens of microns) and thus small struts and consequently cells can be realized, the process is completed in two steps.

The disadvantages are as follows: After the CAD file has been sliced, this technique selectively cures regions of the slurry by projecting UV light onto it. Once one slice has been cured, the system moves on and a new layer is printed on top of the previous one. This step-wise production method limits the accuracy in reproducing tilted objects because the thickness of the slices cannot be infinitesimal. The result is the so-called stair-casing effect (Fig. 6 right) where printed objects present a zig-zag surface. In ceramic materials, this phenomenon can be highly detrimental under load because it acts as a notch and thus as a stress concentration point. This issue has been recently solved for plastic AM [40]. Printing area and printing time are two other technological issues that will be resolved as knowledge of this technique increases. The biggest limitation is that not all the powders can be employed to make a UV-curable slurry. Slurries with several ceramic powders are not UV curable simply because they reflect or absorb the UV radiation. In our work, we used Al_2O_3, which is easy to print.

IV. Applications

The following paragraphs briefly present some applications of periodic cellular architectures for high-temperature, harsh working conditions. They are the outcome of research projects whose results can be disclosed. Other applications developed under non-disclosure agreements will be omitted.

(1) Porous burner

The porous burner used in this work is based on the stabilization of the combustion process within a porous medium where flameless combustion takes places in its empty spaces [19]. Si-SiC porous architectures are fundamental components for flame stabilization technology because they can guarantee the system functionality in oxidative conditions at high temperatures (~ 1400 °C) for several thousand hours [41]. With the use of a Si-SiC lattice, it is possible to further stabilize the flame, ensuring better temperature uniformity (Fig. 7).

In industrial applications with SiSiC ceramic foams, the power is mostly limited to 1 MW/m^2, with SiSiC cellular ceramics made with periodic architectures, output power can be increased. Furthermore, the major improvement of this solution over the random foams is during the transient regime of switching the burner ON and OFF. Rapid heating and cooling will lead to lower local failures because of thermal shock. As a consequence, the problem of local stress concentration because of the weaker struts in a foam (Fig. 1) is clearly reduced.

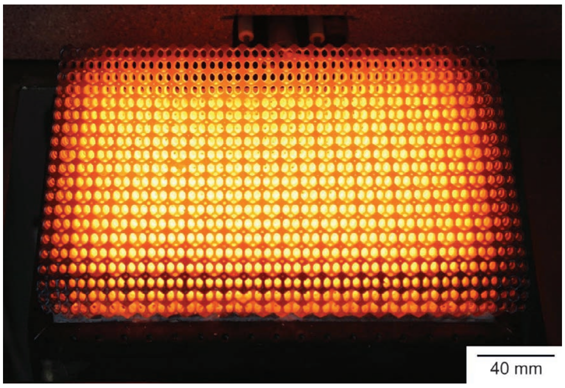

Fig. 7: A lattice glowing at ~ 1400 °C on a CH_4/air porous burner [42].

(2) TPS

Innovative thermal protection solutions (TPS) for space vehicles entering planets' atmospheres were developed within the FP7 European research project THOR (www. http://thor-space.eu/) [43]. One of them exploited a sandwich-structured ceramic matrix composite [44] with its core working as an active cooling system (Fig. 8). A prototypic leading edge for plasma wind tunnel tests was designed, its core, made of ad hoc cellular ceramics, was realized by means of AM and joined to CMC skins [45].

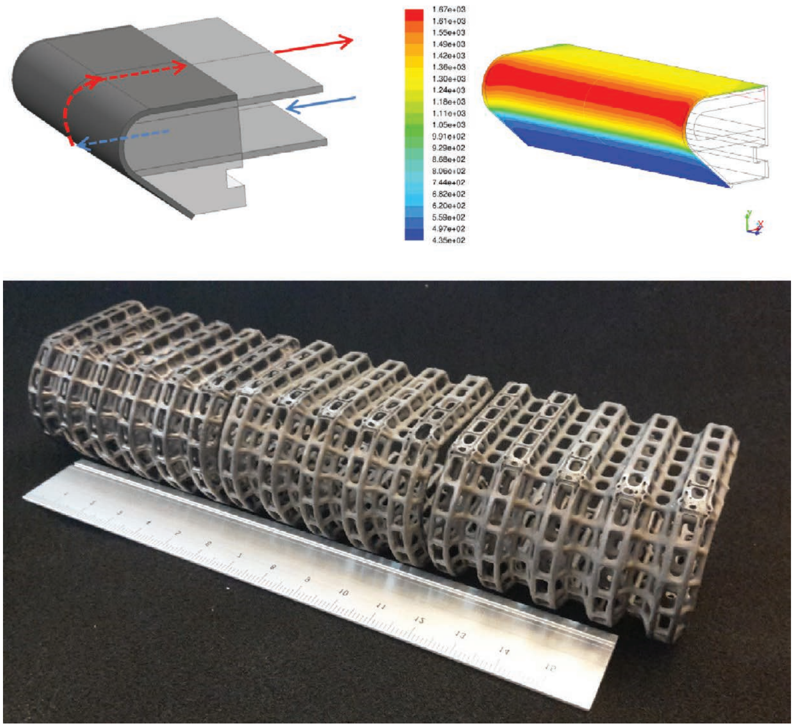

Fig. 8: Top: Cooling gases flow simulation inside the leading edge. The colour bar shows the static temperature [K] on the leading edge surface. Bottom: The cellular architecture developed for the active TPS solution above.

Following an extensive test campaign, active-cooling TPS proved to be an effective way to lower the outside temperatures of the hottest parts of re-entry vehicles [31].

(3) Catalytic supports

A lattice with rotated cubes as the unit cell (Fig. 9) was developed within the FP7 BioRobur European research project (http://www.biorobur.org/).

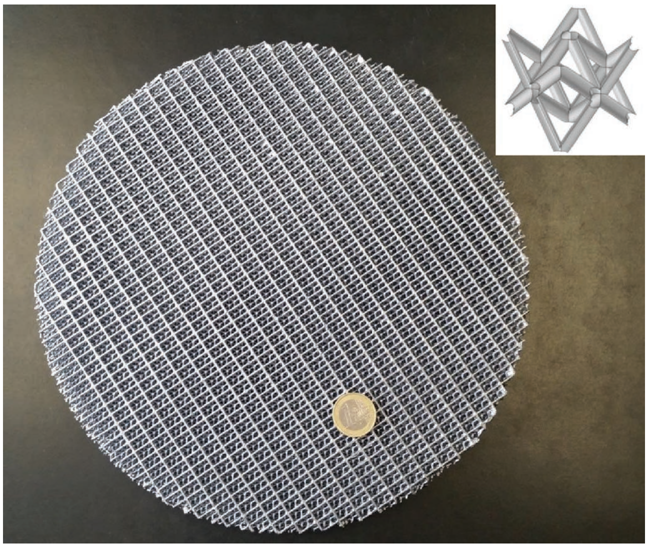

Fig. 9: One slice (20 mm thick, 250 mm diameter) of a SiSiC periodic architecture based on a rotated cube cell top right.

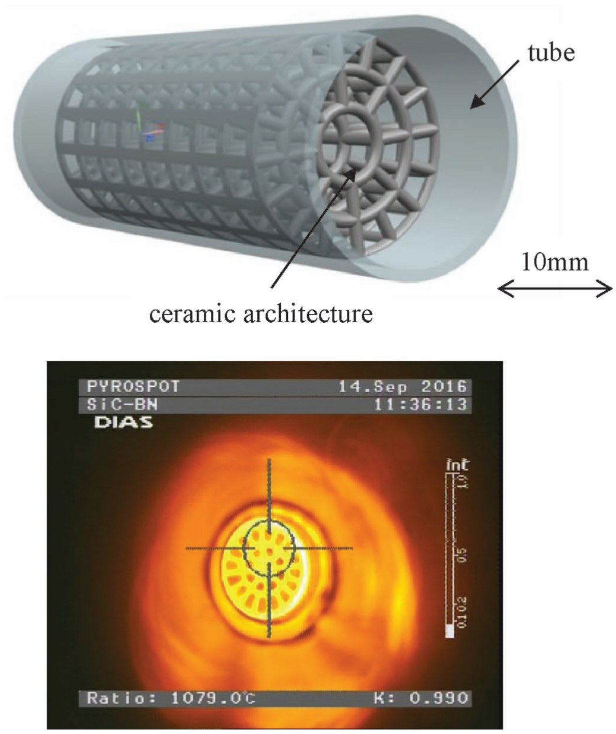

Fig. 10: Top: The solution adopted enhances heat exchange with engineered porous architectures. Bottom: Camera view of the inside of the tube during preliminary tests in a high-temperature experimental apparatus. The image shows the inlet of the tube with an air mass flow of 5 g·sec^{-1} while the outer tube walls are kept at 1280 °C.

The BioRobur project focussed on green hydrogen production by means of direct biogas auto thermal reforming (ATR). The novelty is based on the use of period architectures as catalyst support for the ATR reaction. ATR was favoured owing to the ability of SiSiC periodic cellular materials to disperse the heat axially in the reactor [46, 47].

(4) Heat exchange

Tubular Si-infiltrated SiC_f/SiC_m composites with a periodic architecture inside were fabricated by means of the electrophoretic deposition of matrix phases followed by Si-infiltration for concentrated solar receiver applications [48, 49]. Cellular ceramics were produced with the above-mentioned method.

Results show that by using engineered porous architectures such as cubic cells inside a tube (Fig. 10), heat transfer efficiency in operative conditions increased ~ 4 times compared with a hollow tube [50].

V. Conclusions

Ceramic foams are employed in several industrial fields today. These porous architectures show several limitations in high-tech applications where materials have to be engineered. These limitations consist in the lack of design ability and variations in local properties because of the foam's randomness. These problems were overcome with the advent of ceramic AM. Thanks to this near-net shaping technique, periodic cellular ceramics can be designed, their performance simulated and finally produced close to their nominal dimensions. This paper shows the outcome of long-term research work in this field, dedicated to the complete production cycle of these architectures.

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