ESRF	**Experiment title:** Multiscale determination of structural and transport properties of porous C/C composite preforms during Chemical Vapor Infiltration	**Experiment number**: ME-289
Beamline: ID19	**Date of experiment**: from: to:	**Date of report**: Feb. 27, 2012
Shifts: 12	**Local contact(s)**: P. Cloetens/E. Boller (today: P. Tafforeau)	*Received at ESRF:*

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Report:

A long-term aim of LCTS is the modeling of C/C composites. C/C composites are constituted of a fibrous architecture that is progressively filled by a carbon matrix by Chemical Vapor Infiltration (CVI). These fibrous architectures, or preforms, are commonly made of complex arrangements of layered patterns such as woven fabrics, felts, mats, etc. . . held together by needlings or interlocking patterns in the perpendicular direction. A rational study of the influence of the precise arrangement of these patterns on the infiltrability (that is, the ability to reach a high final matrix density by CVI) urges to combine structural and transport information at at least two scales: (i) the fibre (and micropore) scale (one fibre diameter is roughly 7 µm), and (ii) the weave (and macropore) scale (3 to 4 mm). Infiltrability may be characterized through the evaluation of transport properties such as permeability , diffusivity, and Knudsen (rarefied gas) diffusivity, as well as the pore radius distribution and the specific surface. We proposed to perform a two-scale assessment of several structural and transport properties for different kinds of preforms, taking advantage of the excellent spatial resolution available at ESRF and the unique holotomography capabilities.
The aims of this study were:
- A complete 3D visualisation and characterisation of the pore space at two resolutions (including the fibre diameter resolution) in different carbon fibre preform architectures.

- Comparison with other analyses carried out on the same samples (BET, Hg porosimetry, optical micrographs, permeation and diffusion measures).
- Comparison between different CVI conditions is expected to help understand the relevant issues for optimisation.
- A 3D computer model of pore structure as a starting point for numerical computation of transport properties and a validation tool for densification simulations.

All of these objectives were fulfilled, by combining results of the ME-289 experiment and of the subsequent IN-593 experiments, as briefly listed below. The further details are available in the quoted references.

- High-resolution, high-quality images of C/C composites were obtained at 0.7 µm resolution [2,4,5]. Phase-contrast images were successfully processed, with a sufficiently good fluid/matrix labeling for posterior computations.
- Holotomographic images were obtained and successfully processed [1], however it was not possible to use systematically this method for fibre bundles perpendicular to the tomographic rotation axis [11] .
- Comparison of the geometrical properties with other methods (BET, Hg porosimetry, etc.) has proved successful [2,4,5]
- The high-resolution images were taken as a base for several effective transfer properties computations:
    - Heat and mass transfer [7]
    - Nonisothermal rarefied gas transfer (i.e. “thermal transpiration”) [3,8]
- Modelling of CVI, featuring gas transfer, chemical reaction, and pore morphology evolution [6], has been implemented and applied on the X-ray CT images, at fibre scale [9,11,12] and tow scale [10]. The results compare favorably with experiments and allow a rational approach of infiltrability.

More work is ongoing, principally on the basis of IN-593 experiments.

Publications, in published time order :

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2. “ Computing Structural and Transport Properties of C/C Composites from 3D Tomographic Images” , O. COINDREAU and G. L .VIGNOLES, “ *Advanced Materials Forum II ”* , R. Martins, E. Fortunato, I. Ferreira and C. Dias eds., *Mater. Sci. Forum* vol. **455-456,** Trans. Tech. Publications, Zurich (2004), pp . 751-754.

3. “ Influence of porous media geometry on rarefied non-isothermal gas transport ”, C. PREUX, G. L. VIGNOLES, P. CHARRIER and B. DUBROCA, “ *Progress in Computational Heat and Mass Transfer ”*, R. Bennacer, A. A. Mohamad, M. El Ganaoui and J. Sicard eds., Lavoisier, Paris, France (2005), vol. **1**, pp. 447-452.

4. “ Assessment of structural and transport properties in fibrous C/C composite preforms as digitized by X-ray CMT. Part I : Image acquisition and geometrical properties”, O. COINDREAU and G. L. VIGNOLES, *J. Mater. Res.* vol. **20** (2005), pp. 2328-2339.

5. “ Multiscale X-ray CMT of C/C composites : a tool for properties assessment”, O. COINDREAU, G. L. VIGNOLES, and J.-M. GOYHENECHE, in *“Advances in Ceramic-Matrix Composites XI”*, N. P. Bansal, J. P. Singh and W. M. Kriven eds., *Ceram. Trans.* **175**, Wiley, New York, (2005), pp. 77-84.

6. “ Modelling of CVI Processes”, G. L. VIGNOLES, in “ *Advanced Fibrous Inorganic Composites V ”*, P. Vicenzini ed., *Adv. Sci. Technol.* vol. **50**, Trans Tech Publications, Zürich, (2006), pp. 97-106.

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8. “ Rarefied pure gas transport in non-isothermal porous media: Validation and tests of the model”, G. L. VIGNOLES, C. PREUX, P. CHARRIER and B. DUBROCA, *Transp. In Porous Media* vol. **75** (2008), pp. 295-317.

9. “ Fibre-scale modelling of C/C processing by Chemical Vapour Infiltration using X-ray CMT images and random walkers ”, G. L. VIGNOLES, C. GERMAIN, O. COINDREAU, C. MULAT, W. ROS, *ICVD XVII & EuroCVD 17*, Vienna, 4-9 Octobre 2009, M. T. Swihart, D. Barreca, R. A. Adomaitis and K. Wörkhoff, eds., *ECS Transactions* vol. **25** no. 8, The Electrochemical Society, Pennington, NJ, (2009), pp. 1275-1284.

10. “ Modelling Chemical Vapour Infiltration in C/C composites : numerical tools based on µ-CT images”, G. L. VIGNOLES, O. COINDREAU, W. ROS, I. SZELENGOWICZ, C. MULAT, C. GERMAIN, M. DONIAS, “*HT-CMC 7 – 7th Intl. Conf. On High-Temperature Ceramic-Matrix Composites* », W. Krenkel & J. Lamon eds. (2010), pp. 598-606

11. “ Benefits of X-ray CMT for the modelling of C/C composites”, G. L. VIGNOLES, C. MULAT, C. GERMAIN, O. COINDREAU, and J. LACHAUD, *Advanced Engineering Materials* (2011), vol. **13**, pp. 178–185. Also in Virtual Journal “*Materials Views*”.

12. “ Modeling of infiltration of fiber preforms based on X-ray tomographic imaging”, G. L. VIGNOLES, C. GERMAIN, C. MULAT, O. COINDREAU, W. ROS, “*12th International Ceramics Congress Part J.*”, P. Vicenzini, M. Ferrar and M. Singh eds., *Adv. Sci. Technol* (2011) vol. **71**, pp. 108-117.