ESRF	**Experiment title:** **3D-MICROTOMOGRAPHIC INVESTIGATION OF POROUS BUILDING MATERIALS**	**Experiment number:** HS-70
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Names and affiliations of applicants (* indicates experimentalists):

Daniel QUENARD
Centre Scientifique et Technique du Batiment (CSTB)
Service Matériaux - 24, Rue Joseph FOURIER
38400 SAINT MARTIN D'HERES - FRANCE
Co-proposers
Dale P. BENTZ, Nicos S. MARTYS, Edward J. GARBOCZI
National Institute of Standards and Technology - (NIST)
BFRL - Building Materials Division.
Gaithersburg, Maryland 20899-0001, USA.
Hartwig KUNZEL, Fraunhofer-Institut für Bauphysik (IBP).
Postfach 11 52 - D-83601 Holzkirchen - GERMANY.

Report:

1. Introduction

To predict successfully the performance of building materials exposed to a degradative environment, transport properties must be either measured or predicted. The development of relationships between microstructure and transport properties for these materials should allow accurate prediction of the latter and an increased understanding of how microstructure influences transport.
Scanning electron microscopy is commonly used to investigated the microstructure of porous building materials. Starting with binary 2-D (two-dimensional) images, 3-D representations are estimated using computational techniques. The reconstructed 3-D microstructures are then used as input into two computational programs to compute both their relative diffusivity and their permeability.
In this poster, the results obtained with two building materials commonly exposed in building facades (a brick and a natural sandstone) are presented. The computed values compare favorably to those measured experimentally, thus demonstrating the capability of employing microstructural characterization to predict transport properties.
In order to validate this method, it is worthwhile to compare the estimated 3D images with those obtained by 3D x-ray microtomography. A first attempt was performed using beam line ID19 at ESRF. 3D images of different material samples, with an isotopic voxel size of 6.65 microns were obtained. The first results exhibited in this poster give some insight in the internal organization of the samples and how they compare to the estimations.

2. Experimental Investigation

For this investigation, two building materials were examined : a lime-silica brick formed from a mixture of lime and silica reacted in the presence of pressurized water vapor and a clinker brick which is a hard-burned clay brick. In order to determine the open porosity of these materials, the true density was measured with the aid of a helium pycnometer and compared to the bulk density. To obtain the transport properties and the necessary microstructural information, the following tests were carried out.

2.1 Microstructure investigation by SEM

The method used to investigate microstructure of porous materials using scanning electron microscope (SEM) with backscattered electron imaging. (BEI) is described in references [1,2,3]. The sample is a cylinder of dimensions : diameter 10 mm and thickness 10 mm impregnated with an epoxy resin. The size of the analyzed area depends on the phases which is analyzed. Generally, an high magnification provides a good contrast between the phases but the investigated surface is often smaller than the Elementary Representative Volume (E.V.R.) of the microstructure. The thresholding is a major point in the image analysis process. In our case, as the contrast between phases is high, a method based on the greylevel histogram is effective enough. The images obtained for the Clinker Brick (Image A) and the Lime Silica Brick (Image C) are presented in Figure 1 and the main characteristics are detailed in Table 1

Table 1 : Characteristics of the 2D SEM-Images

	Clinker Brick	Lime Silica Brick
Image Size (pixel)	512x512	512x512
SEM Magnification	200	25
Ratio micron/pixel	1 pixel = .867 μm	1 pixel = 6.94 μm
porosity (%)	19	16

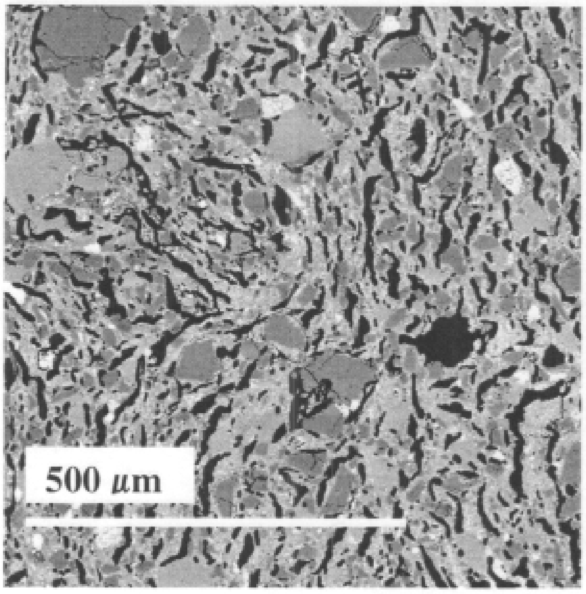

Image A : Clinker Brick (SEM)

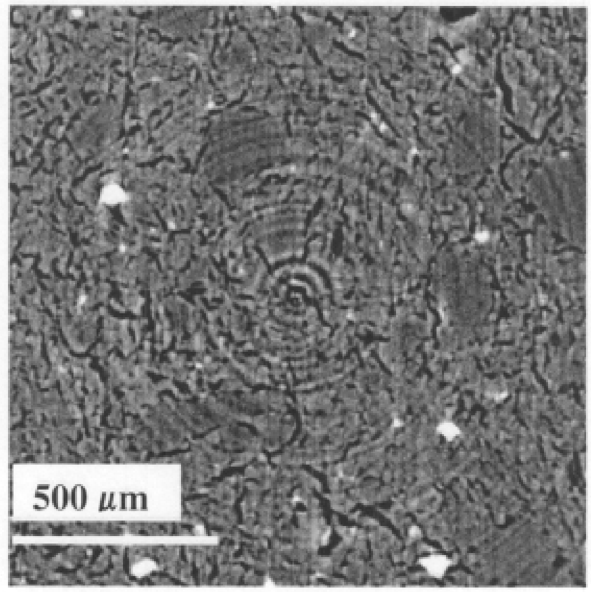

Image B : Clinker Brick (CMT)

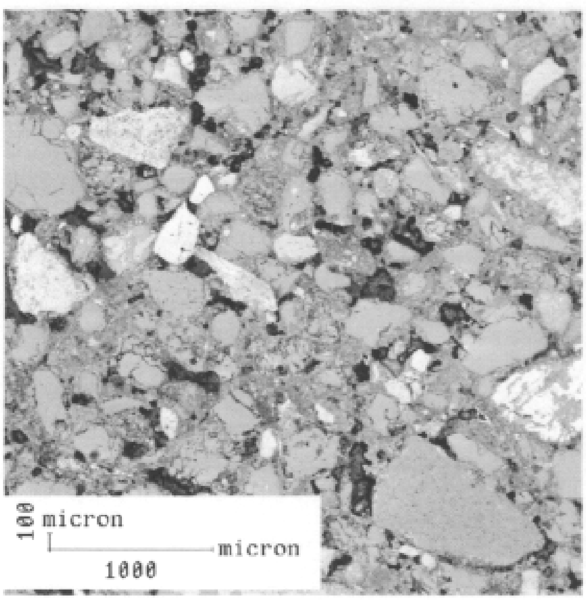

Image C : Lime Silica Brick (SEM)

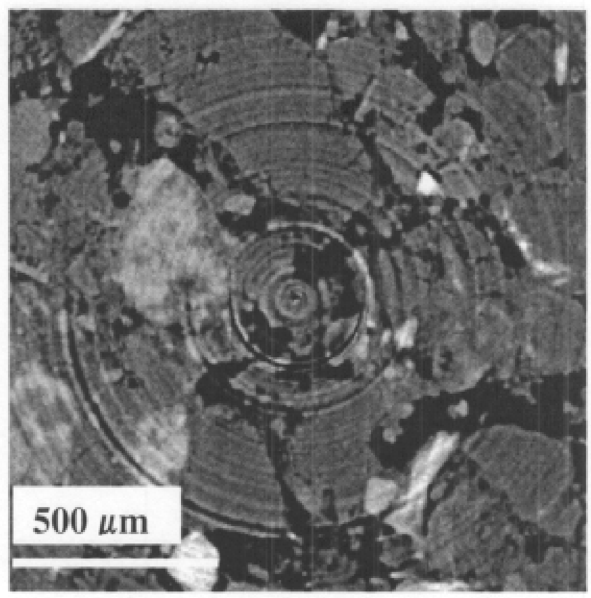

Image D : Lime Silica Brick (CMT)

Figure 1 : SEM and CMT images of the two bricks.

Porosity in black

2.2 Computed Micro-Tomography (CMT)

Within the last decade, X-ray computed microtomography (CMT) has become an important tool for investigation in materials science [4, 5, 6], biology and medecine [7].
In principle, X-ray microtomography is similar to the conventional scanners used in medical applications, with the X-ray source being a synchrotron (figure 2).
Synchrotron radiation offers the possibility to select X-rays with a small energy bandwidth from the wide and continuous energy spectrum, while at the same time keeping the photon fluence rate high enough for efficient imaging. This possibility is of great interest since it allows high spatial resolution images to be generated and avoids beam hardening artefacts, which occur with the use of polyenergetic beams for tomographic imaging.

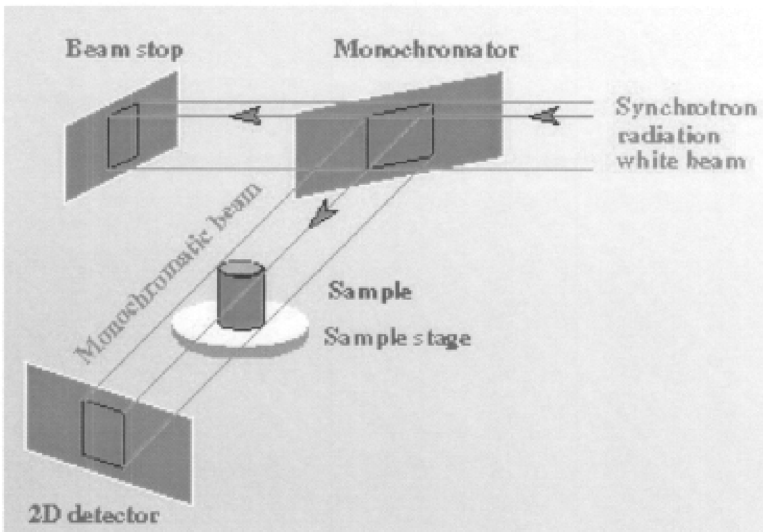

Figure 2 : Scheme of the acquisition set-up [20]

On ID 19 beam line, X-rays illuminate the sample to be imaged which is mounted on a translation/rotation stage allowing precise alignement in the beam. The specimen were scanned using 25 keV X-rays and 900 radiographic projection images were acquired over 180 degrees. After conversion to light by a fluorescent screen, these images are digitalized using the Frelon Camera [8], which consists in a 2D CCD (Charge Couple Device) array with 1024x1024 elements, each 19x19 μ, and offers a dynamic of 14 bits. The CCD camera is mounted perpendicularly to the X-ray beam in order to avoid direct interactions which cause noise in the recorded images. An optical magnification is used resulting in a pixel size of 6.65 μm on the recorded image.
A 3D filtered backprojection algorithm is used to reconstruct a 3D image of the sample from the series of 2D projections [9].

3 Results

Samples of each of the materials (4 mm x 4mm x 10 mm) were imaged using ID 19 beam line at ESRF. The voxels in the reconstructed image are cubes of side 6.65 μm.
The first step of the analysis is to compare 2D SEM images with 2D section of CMT images (figure 2). In spite of the scales which are not identical, SEM and CMT images look very similar with a contrast high enough to make possible segmentation between porosity and solid phase. The rings which appear in CMT images are artifacts resulting from instabilities and dust on the monochromator.
In figure 3, the two images seem different because the scales are different and the 3D simulated image from 2D SEM image exhibits only two phases : the porosity in black and the solid in white. Nevertheless, it should be possible to threshold the 3D-CMT image in two phases, at least within a smaller volume, to perform the computation of transport coefficients.
In figure 4, the scales are closer and considering that black and white (or yellow) correspond respectively to the macroporosity and the solid phase, both images appear very similar.

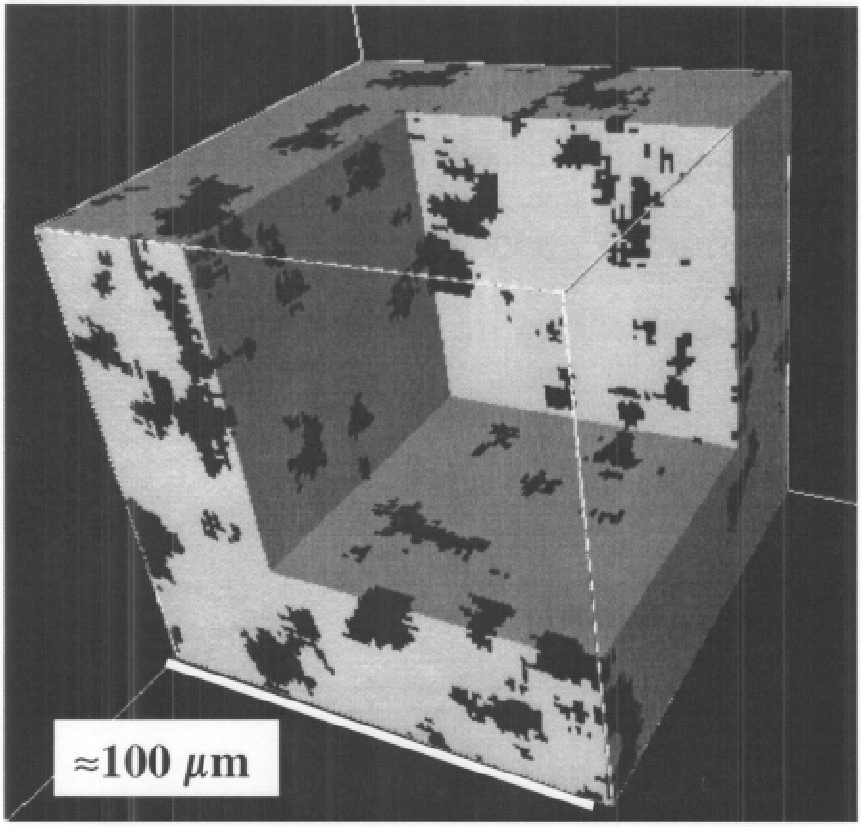

Image A : 3D Modeling from 2D SEM image (NIST)

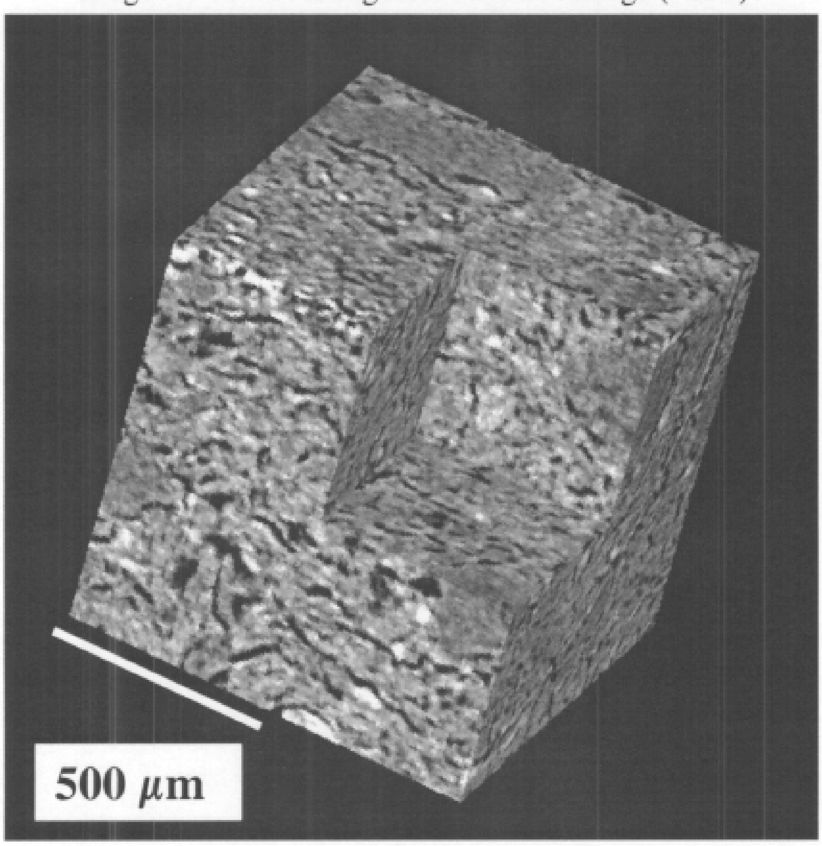

Image B : 3D CMT image (CREATIS -Lyon)

Figure 3 : Clinker Brick

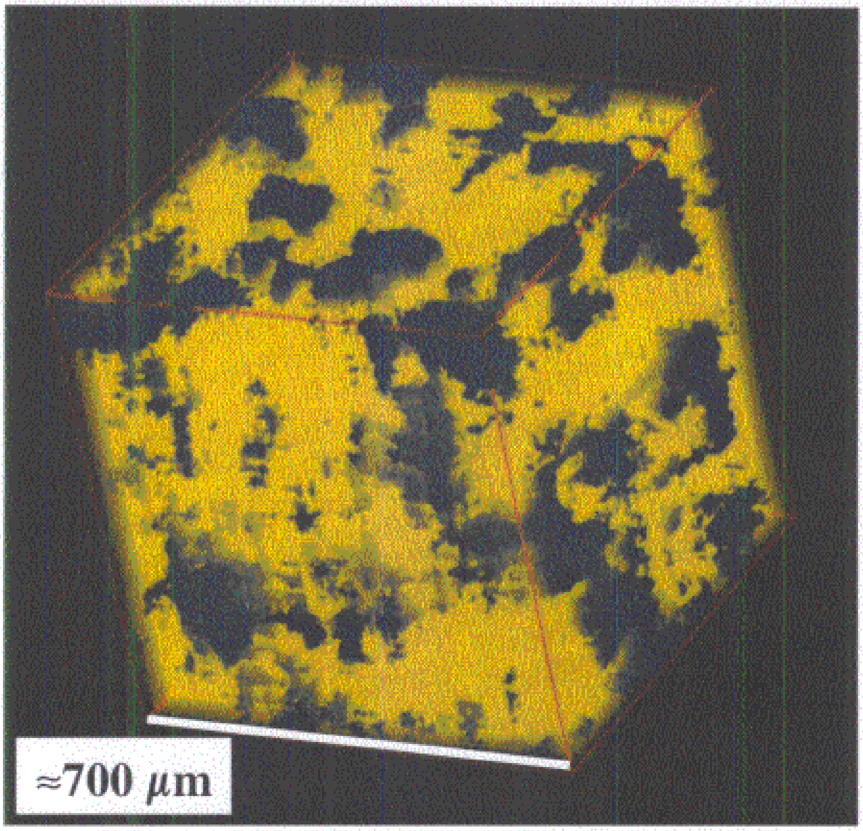

Image A : 3D Modeling from 2D SEM image (NIST)

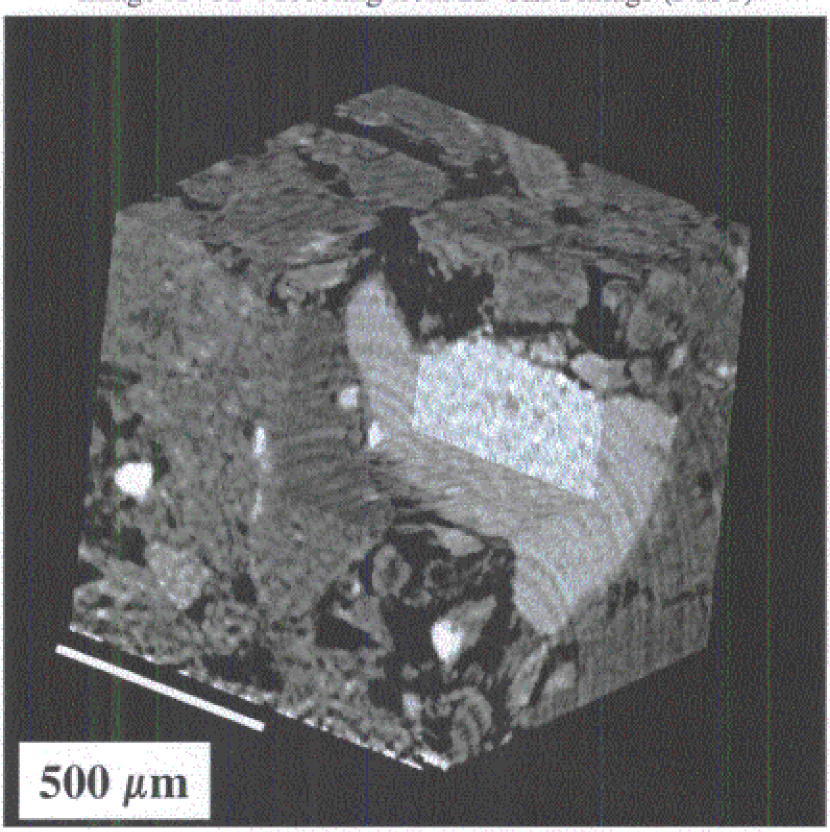

Image B : 3D CMT image (CREATIS - Lyon)

Figure 4 : Lime Silica Brick

Once again, a thresholding operation should be possible with the CMT image.
In the future, our research will be concentrated on quantitative comparison of both 3D images; several geometrical and topological parameters, such as porosity, specific area, curvature, chord size distribution, medial axis, etc ..., will be measured [10].

4. Conclusions

Two techniques (SEM and CMT) for characterizing the microstructure of porous building materials are compared. Images provided by SEM compare favorably with 2D sections of CMT images.
Moreover, the 3D microstructures obtained by a computing technique based on information extracted from 2D SEM images appear to be very similar to the ones provided by the CMT method.
Further works will focus on quantitative analysis of 3D microstructure either simulated from SEM images or provided directly by CMT.

5 : References

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