In-Situ X-Ray Tomography on Permeability Evolution of C/SiC Porous Ceramic for Hypersonic Vehicles

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Abstract

Transpiration cooling system in hypersonic vehicles still remains a challenge due to the limitations of observing permeability and microstructure evolution of porous medium filled with coolant. To tackle this problem, a novel compression-permeation device is designed with high-resolution X-ray tomography system, and then an investigation on permeability evolution mechanism of a C/SiC porous ceramic under pressure is performed using in-situ X-ray imaging and the compression-permeation device. The experimental results indicate that the pore-space fluid flow is displayed in terms of three-dimensional streamlines, making the permeability mechanism clear. Meanwhile the porosity along the thickness of ceramic under pressure has been obtained by synchrotron tomography testing, and it is also verified that the porosity of C/SiC ceramic fabricated in our research group is basically uniform (>95.4%) along the thickness. Furthermore, we have found the evolution rule for permeability of porous ceramic with water, which depends on the variation of its microstructure under different loads.

1. Introduction

Transpiration cooling is regarded as the most potential thermal protection system in future hypersonic cruise vehicles (see Fig. 1) for its superior properties such as reusability and maintaining the aerodynamic shape of the advanced vehicles [1, 2]. Porous medium is usually used as a counter flow heat exchanger and channels for coolant, which shows an essential effect on the cooling performance of vehicle surfaces [3–5]. Whereas this cooling system is hardly utilized due to the low reliability of porous medium. For examples, the porosity of cross sections along the thickness of porous ceramic cannot be measured accurately, and this internal characteristics due to complex microstructures shows a crucial influence on the transpiration cooling performance [6, 7]. Furthermore, during the flight process at super high speed, the porous medium is necessarily subjected to complex loads including the external aerodynamic load, seepage pressure of coolant, heat stress caused by aerodynamic heating, and structural load of the vehicles [8–12]. Thus, it is a significant issue whether the flow of coolant can be precisely calibrated and the uniformity of coolant in the porous medium can still remain unchanged or not while the porous medium undergoes heavy loads [13–15]. It is urgent to elucidate the permeability evolution subjected to external loads. Unfortunately, none of works have been conducted due to technological cause.

It is worth that the permeability evolution rules during the deformation and failure of rock specimens have been investigated in the geotechnical engineering including drilling for oil and gas [16, 17], stability analysis and micro-crack propagation of materials [18, 19], and seepage flows in porous media [19–21]. These researches indicate that the rock has a distinct permeability difference during deformation stages [22–24]. The microstructural evolution of the materials with high porosity has been preliminarily discussed [25–27], but the effect of permeability behaviors has not been elucidated. Nevertheless, the methodology used can be referenced to investigate the permeability and microstructural features for porous medium

(C/SiC porous ceramic in this work can be thought as one type of porous medium) subjected to dynamic loads during transpiration cooling.

In this work, a novel compression-permeation device is firstly designed in order to reveal the mechanism of permeability evolution of porous ceramic. Then, a detailed investigation on the permeability evolution mechanism of C/SiC porous ceramic potentially applied into hypersonic vehicles exposed to compression was tentatively carried out by using the third generation high-resolution synchrotron radiation X-ray micro computed tomography (SR-μCT) with in-situ compression-permeation device.

2. Experimental Procedure

2.1 C/SiC prous ceramic

The C/SiC porous ceramic sample studied here with the diameter of 4 mm and the height of 4 mm was prepared from as-received ceramic raw material using grinding-mould pressing-sintering process [28, 29]. In detail, the chopped carbon fibers with the diameter of 7 μm are firstly added into the prepared SiC ceramic slurry. The dried mixture including carbon fibers and SiC particles (usually with the average diameter of 10 μm) is then grinded into hybrid powders of micron, which are pressed at 100 MPa in the cylindrical mould to form green bodies. The green bodies are further sintered at 1300°C in a pure nitrogen flow atmosphere. This final material physically presents low density, high compressive strength, uniform pore distribution, and excellent permeability. Particularly, it has a superior transpiration cooling performance during the oxyacetylene flame test, which usually shows huge potential for the application into advanced hypersonic vehicles. Figure 2 presents fractured features before and after compression tests of C/SiC ceramic.

2.2 In-situ X-ray imaging

High-resolution BL13W1 is an X-ray imaging beamline at Shanghai Synchrotron Radiation Facility (SSRF, China) with an optimized 3.5 GeV electron storage ring [30−32]. It can devote to various scientific fields [33−37], including advanced material and structural characterization due to its unique advantages of high brightness, high purity and high collimation [38−40] over traditional laboratory X-ray machine. Figure 3 shows the schematics of in-situ testing system compatible with the BL13W1 of the SSRF. It can be illustrated that high energy X-rays emitted from the emitter can penetrate the C/SiC porous ceramic sample (totally 6 samples were here used with 4.0 mm height and 4.0 mm diameter) installed at an in situ compression-permeation device, and the penetration signal could be accurately captured using the CCD detector (16 bit dynamic, 2048×2048 pixel array) with a pixel size of 3.25 μm, which was capable of identifying the mixture.

The compress-permeation machine consists of a miniature loading device, a water pump, an electronic balance, and some auxiliary equipment [41−43]. The device is capable of carrying out the permeability test while the sample is subjected to different interrupted loading. Figure 4 gives the compression-permeation

testing diagram. Stainless steel sinks are often utilized to pass the stress to the porous ceramic sample and are also used as a water inlet and a water outlet, respectively. Filter plates with uniform holes can evenly disperse water on the surface of the porous sample. Silicone tube and sealing tape can avoid the water flow permeate along the outer wall surface of the porous sample. During this compression process, the miniature loading device was set on the sample stage, which would be rotated 180 degrees for getting photos with different angles of the porous sample.

In this compression experiment, the optimized photon energy of X-ray beam was about 25 keV, the distance from the ceramic sample to the CCD camera was 18 cm as shown in Fig. 4b, and an exposure time was selected to be 1s or so. This in-situ compression process can be monitored by a user controlling software and then can be initially analyzed as illustrated in Fig. 4b. A total of 720 radiographs were acquired over an angular range of 0-180° for each SR-μCT. For each scanning, we additionally record 5 dark fields and 22 flat field images to correct the raw projections. During the permeability testing, the water pressure difference was 0.08 MPa, the gravity of water was neglected.

3. Results And Discussions

3.1 Permeability mechanism

Totally 747 original images including projections, dark field and flat field images were captured in one cycle. However, they only show side elevation instead of directly reflecting the inner structure of this porous ceramic, as shown in Fig. 5(a). So it needs to transfer the original images to slice images through ReNamer, P3 and P3B software.

These slice images can visually show cross-section views. The cross section (Z = 3.068 mm) of ceramic sample after failure is selected to display the cracks in Fig. 5(b), while the rendered models are chosen from the ceramic sample before loading in Fig. 5(d), (e) and (f). Gray values of pixels in binary images are strictly divided into two gray values for presenting information more distinct. White region represents the matrix of porous ceramic, and black gray value exhibits the pore structures information (Fig. 5(c)). The image processing from slice images to binary images is achieved by Image J software. The image analysis for the greyscale pixel intensity value of ceramic matrix and pore structure is carried out using this software. There is a marked difference between the greyscale value of the ceramic matrix pixels and the pore structure pixels. Then the threshold has been selected according to the image segmentation [44–46] in order to transform to black and white ones. Three-dimensional reconstruction of this porous ceramic is achieved through these slice images with Avizo software in this paper. Each voxel represents a physical volume of ~ $3.25 \times 3.25 \times 3.25$ μm^3. Random part of this porous ceramic (diameter of 337.9 μm, height of 737 μm) has been selected to show the pore structures and permeability process, as shown in Fig. 5(d), (e) and (f). It has a uniform distribution for pores inside the porous ceramic (see Fig. 5(d)). White region represents the matrix and blue region is regarded as pores inside the porous ceramic in 5(d).

Pore characteristics are extracted from three-dimensional model of porous ceramic (see Fig. 5(e)). Figure 5(f) shows the state of permeability test of porous ceramic, and three-dimensional streamlines obtained by using Avizo software are used to simulate the pore fluid flow based on the Darcy's law and the Navier-stokes equation. Streamlines with different colors represent flow rates in this permeability simulation process, it also indicates uniformity of permeability during the permeability test. The red streamlines denote faster flow rate, while blue streamlines show slower flow rate. According to mass conservation of incompressible fluid, the smaller area of flow passing can contribute to a faster flow rate, so the streamlines can visually display the porosity and size of pores. Note that where streamlines are concentrated, pores are also more concentrated, which indicates that these streamlines can also be used to qualitatively and visually identify the pore zones information inside the porous ceramic. As we all know, fluid usually escapes the region with bigger flow resistance, and prefers to flow through the pores structures with smaller flow resistance, this phenomenon may cause heat transfer deterioration in the transpiration cooling. On the basis of streamlines, porosity uniformity of porous ceramic can still be further improved by optimizing the fabrication technology even though the porosity is basically uniform. The streamlines video associated with this permeability process can be found in the supplementary material.

3.2 Influence of Loads on permeability

Figure 6 shows the stress-strain curve and permeability-strain curve under the loads of F = 0 MPa, F = 5.28 MPa, F = 10 MPa and failure (F = 12.4 MPa), which respectively corresponds to A, B, C, D in stress-strain curve and a, b, c, d in permeability-strain curve.

Due to mechanical transmission of the miniature loading device, there are two stress relaxation stages of BB' and CC' where the scan imaging and permeability testing are carried out, respectively. The strains of ceramic sample in these two stages are virtually remained unchanged, so they can't affect the experimental results and the height of the sample has little variation. During the experiment, A point is the original state that has no loading and no permeability test, in this state, the x-ray imaging, permeability test and x-ray imaging are successively carried out in turn. AB is first loading, B'C is second loading, C'D is last loading until the sample has been broken. The x-ray imaging, permeability test and x-ray imaging are successively carried out after each loading. For permeability-strain curve, as the strain increases, the permeability rate has a tendency to decrease for ab, and a slight rise for bc, the last a sharp rise for cd.

3.3 Permeability variation mechanism

Permeability rate is mainly determined by the inner pore structures of the sample. 3D rendering of porous ceramic with pores and matrix (blue represents matrix, yellow is pore) has been built in Fig. 7, it has obvious changes for pores under different loading stages, while the pores of sample before and after permeability test have little noticeable change under the same load.

The visual cross-section views of a sample under different loads are exhibited in Fig. 8. For clarity, cross-section views are chosen from six different positions (Z_1 = 0.64 mm, Z_2 = 1.31 mm, Z_3 = 1.97 mm, Z_4 =

2.64 mm, Z_5 = 2.97 mm and Z_6 = 3.30 mm) of sample, as clearly shown in Fig. 8(a). The porosities of these six positions under different loads were also illustrated Fig. 8(b), it is obvious that the porosities at different positions firstly decrease and then increase with the increasing of loads. Similarly, the slice images of these six positions can also reflect this tendency (see Fig. 8(c)). The black areas of these six cross-section positions after compression of 5 MPa are smaller than those before loading, which indicates that pores inside the sample become smaller under this load. This trend corresponds to the permeability rate of sample after compression of 5 MPa. As the stress increases to 10 MPa, the porosity and the permeability rate have some increase. The stress continues to be increased, it is clear that the large cracks begin to appear for some cross-section views like Z_2 = 1.311 mm, Z_3 = 1.971 mm, Z_4 = 2.636 mm, Z_5 = 2.969 mm and Z_6 = 3.301 mm. These cracks provide the bigger channels for deionized water, which causes a sharp rise in the permeability rate of the sample. The cross-section views of each position before and after permeability have been displayed, respectively. Compared with cross-section views before permeability test, the pore structures of these cross-section views after permeability test have little noticeable change. It indicates that the water pressure (0.08 MPa) inside sample almost doesn't impact on the pore structures at this stage.

Figure 9 displays the porosity distribution along the thickness (from Z_7 = 0.256 mm to Z_8 = 3.424 mm) of porous ceramic with different loads. Note that it is verified that porosity of C/SiC ceramic without any loads ranges from 10.6−15.1% along the thickness, namely uniformity > 95.4%. In these curves, the porosity includes volume fraction of pores and cracks caused by loading. The changing trends of porosity of porous ceramic with different loads are consistent with those of permeability, which indicates cracks can also be the channels for deionized water in permeability tests. To investigate the variation of porosity of different cross-sections in porous ceramics under different loads, respectively, the dispersion degree of porosity of different cross-sections has been estimated according to the discrete variance formula accepted widely by researchers:

$$D(X) = \sum_{i=1}^{n} p_i \cdot (x_i - \mu)^2 \qquad (1)$$

$$\mu = E(X) \qquad (2)$$

where D represents the dispersion of porosity of different cross-sections in the porous ceramic, E is mathematical expectation of the porosity. The dispersion degree under loads of 0 MPa, 5.28 MPa, and 10 MPa are 0.94, 1.1, 0.61, separately. Note that dispersion degree after failure is 8.1. It distinctly indicates pores along the thickness of porous ceramic without large cracks are uniform, which shows that the uniform distribution of pores can provide reasonable channels for deionized water in the transpiration cooling.

The three-dimensional structure model of porous ceramic (the diameter of 4 mm, and the height of 737 μm) has been restructured with Avizo software in order to see the pore structures clearly, pores have been uniformly distributed in the porous ceramic (see Fig. 10(a)). After maximum loading, the pore structures including pores and cracks are displayed in Fig. 10(b), blue region represents pores and red part shows cracks. Compared to porous ceramic before loading in Fig. 10(a), it is clearly seen that large cracks have randomly appeared in the porous ceramic, and these cracks can also be the channels for deionized water in the experiment, so the permeability has a sharp increase in cd in Fig. 6. The porosity formed from cracks is very non uniform along the thickness direction of porous ceramic (see Fig. 9). Therefore, these pore channels of porous ceramic after achieving maximum loading cannot control accurately and uniformly water flow.

4. Conclusions

In order to quantitatively elucidate the permeability evolution behaviors of a C/SiC porous ceramic, an in situ compression-permeation testing rig was designed with high-resolution synchrotron radiation X-ray imaging beam line BL13W1 at the SSRF. Through interrupted pressure process, the following conclusions can be drawn:

1. The visualization for permeability mechanism of porous ceramic can be displayed by three-dimensional streamlines, which can indirectly reflect the porosity and the size distribution of open pores of the porous ceramic.
2. The porosity along the thickness of ceramic under different loads can be obtained based on in-situ synchrotron X-ray tomography investigation. It's worth noting that the uniform distribution of porosity along the thickness of C/SiC porous ceramic sample without loads has been verified.
3. The permeability evolution rule of porous ceramic with water is obtained under different loads. As the load increases, the permeability rate firstly decreases and then conversely becomes larger. This phenomenon is determined by the evolution of pore channels inside the porous ceramic.

Declarations

Acknowledgement

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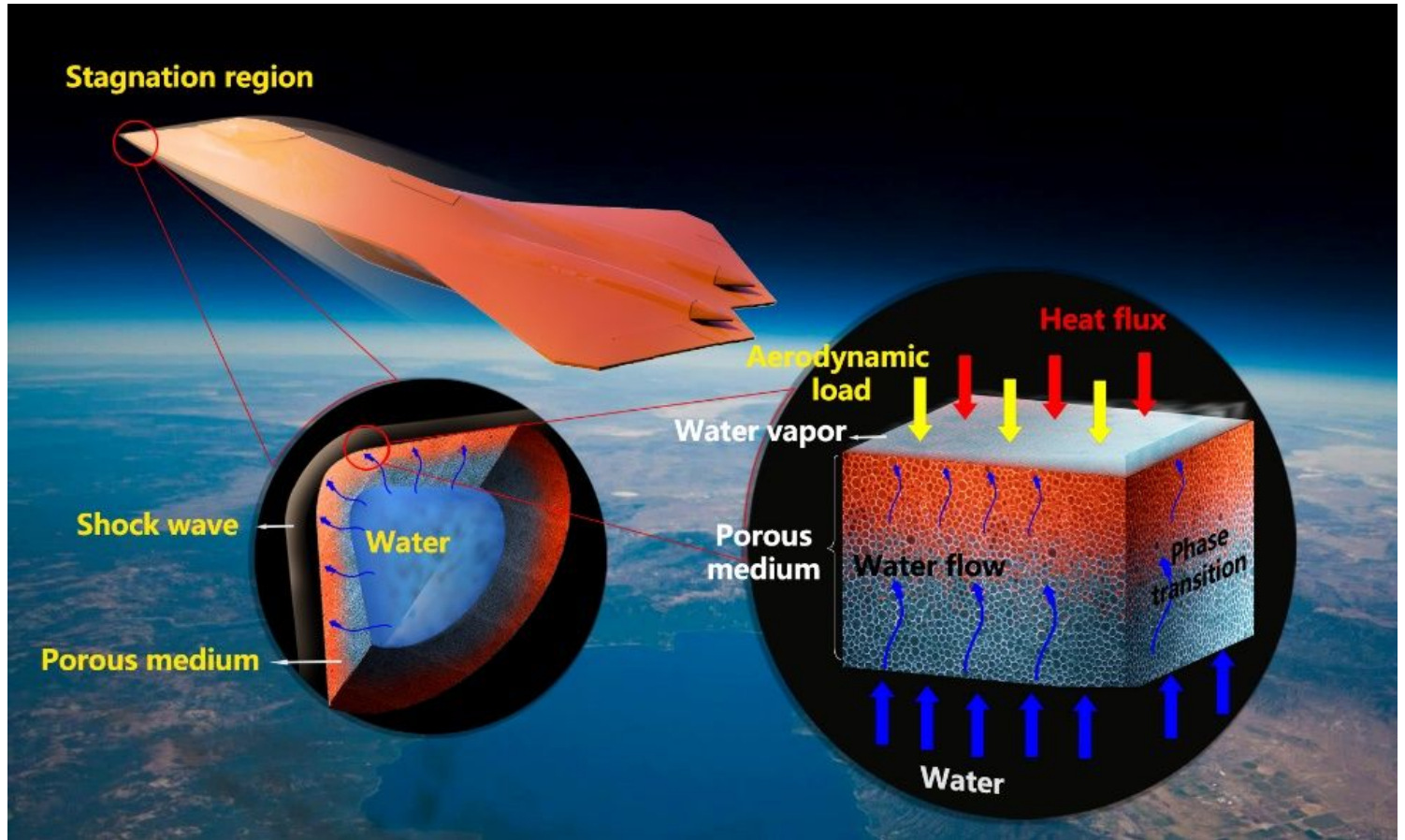
Figures

Figure 1

The schematics of future hypersonic vehicles where external surfaces of the stagnation region show excellent heat exchange.
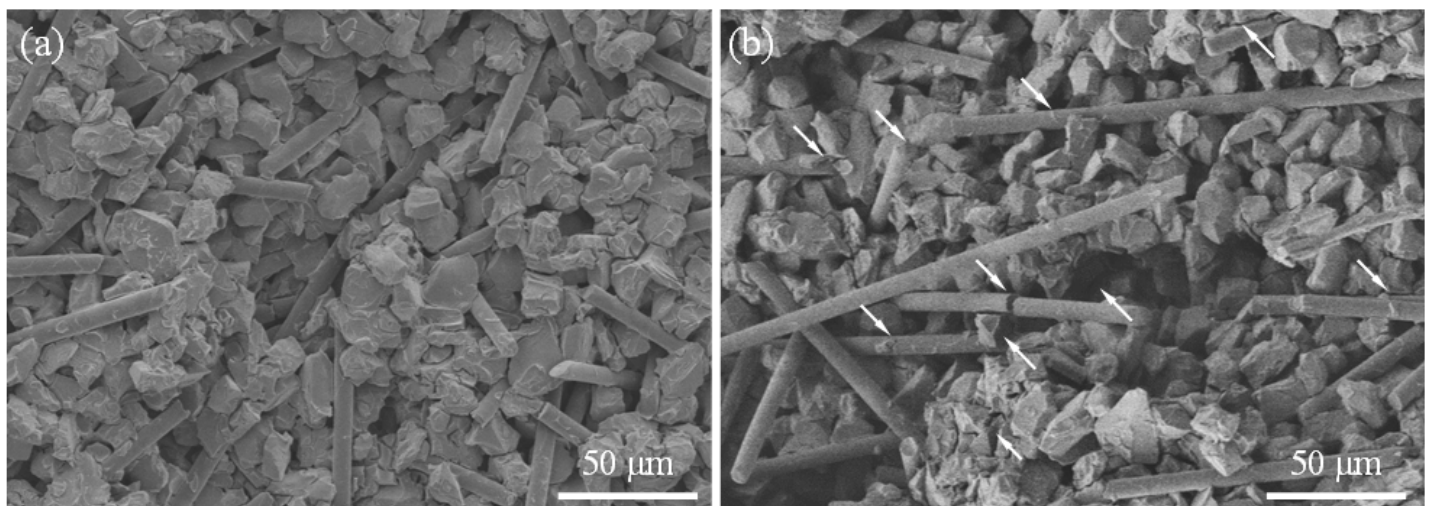

Figure 2

The fractured C/SiC porous ceramic showing distinct failure features of fibers and particles in (a) static tensile sample; (b) compressed sample.
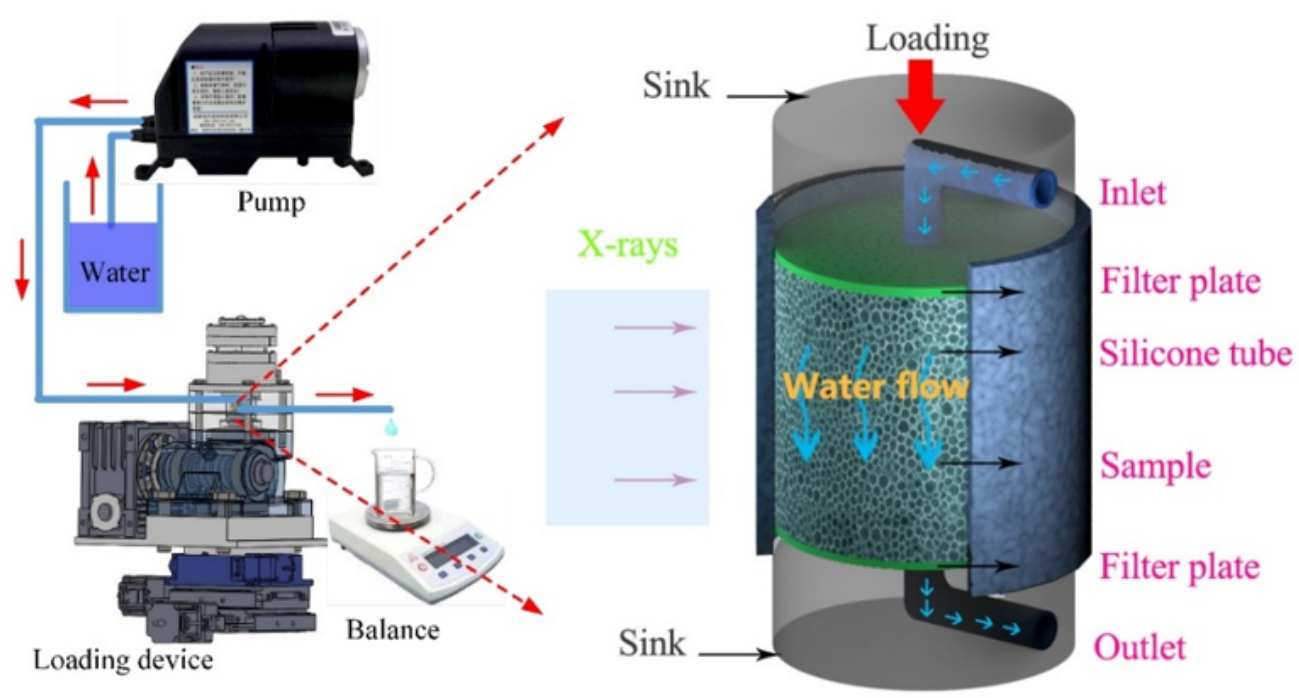

Figure 3

The schematics of in-situ miniature permeability compression testing rig and thorough experimental process at the BL13W1 of SSRF.
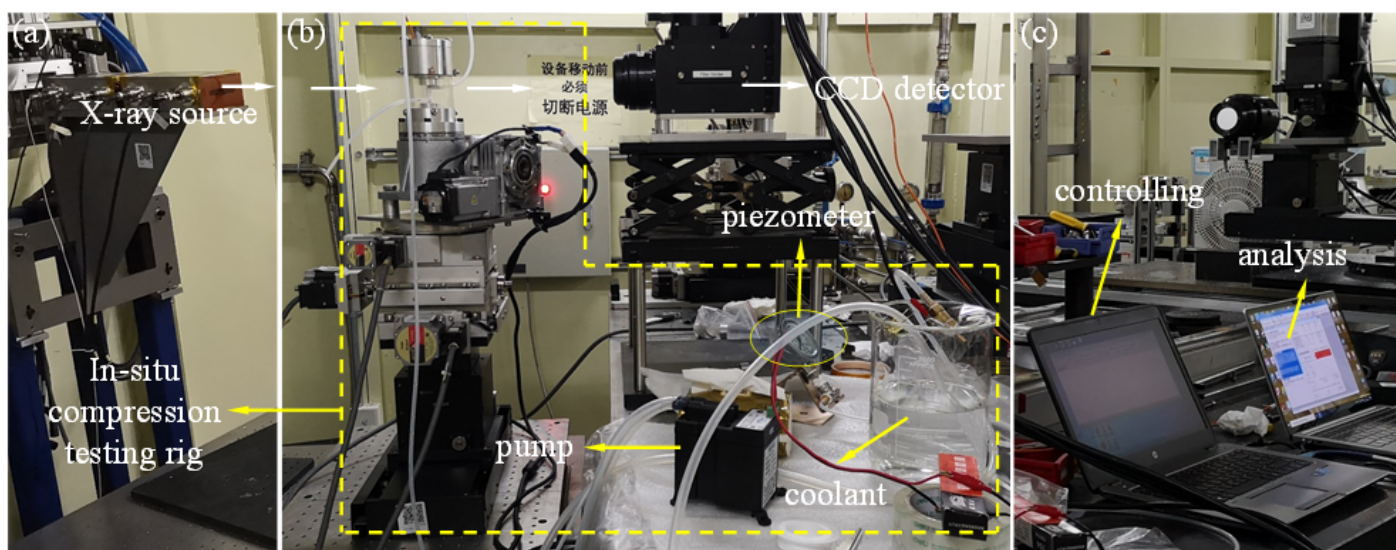

Figure 4

Field experimental pictures with X-ray source, in-situ compression device, CCD detector, inlet / outlet water devices and user controling.

Figure 5

Schematic permeability mechanism of C/SiC porous ceramic through synchrotron X-ray tomography and data construction.

Figure 6

Permeability behavior of C/SiC porous ceramics subjected to interrupted negative loading with synchrotron X-ray tomography.
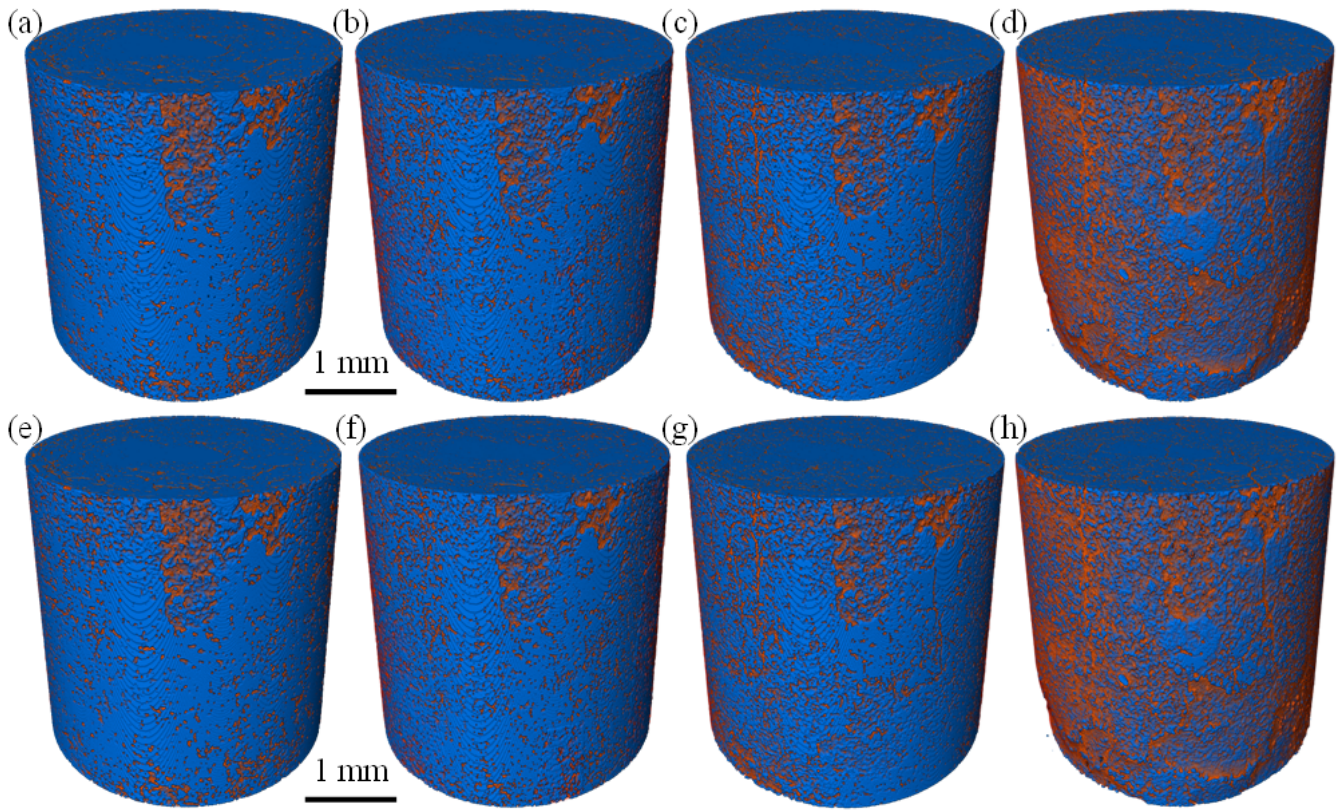
Figure 7

3D rendering of permeability under different loading stages (a)-(d) before permeability and (e)-(h) after permeability, and (a) and (e) under F=0 MPa, (b) and (f) under F=5.28 MPa, (c) and (g) under F=10 MPa, (d) and (h) under F=12.4 MPa.

Figure 8
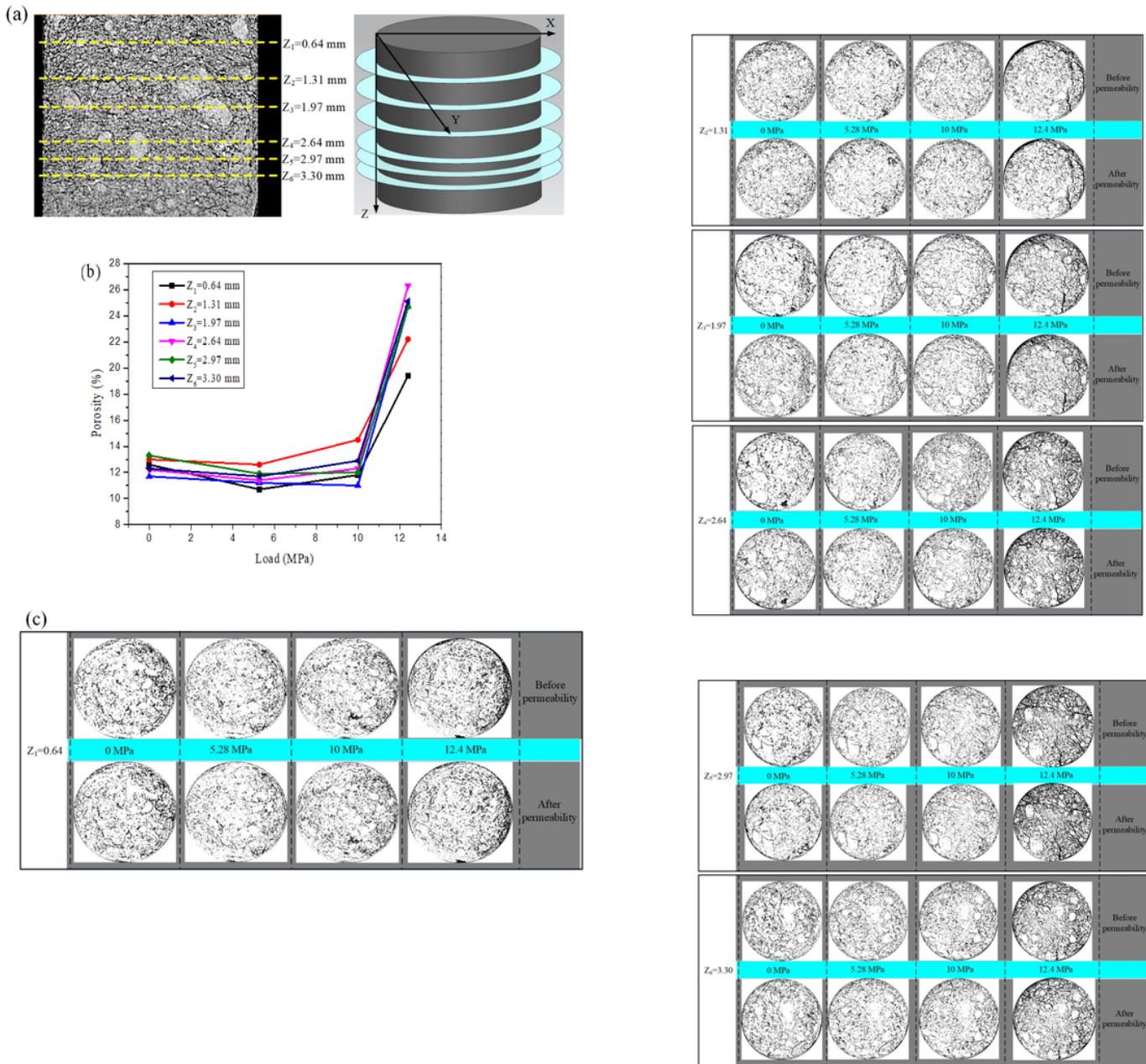
Effect of different loading stages on the porosities across different cross-sections: (a) cross-sections extraction; (b) curves; (c) slice images
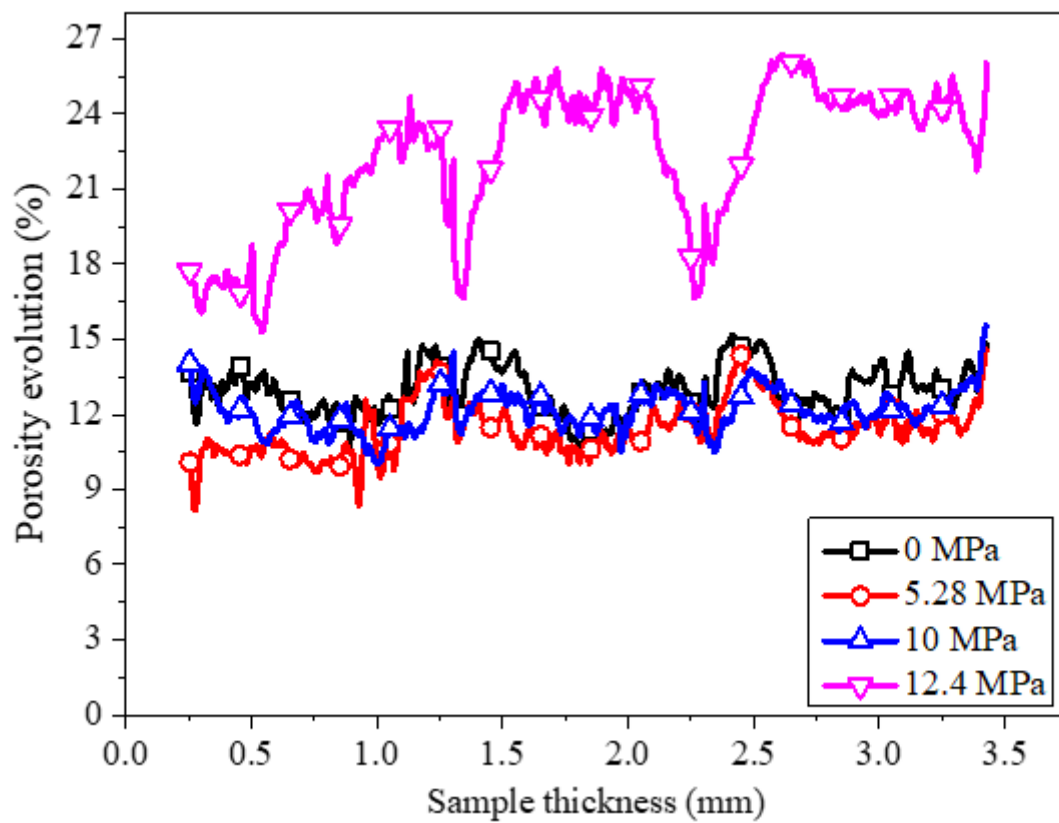
Figure 9

Measured porosity distribution curves along the thickness direction of C/SiC porous ceramic under four loading levels.
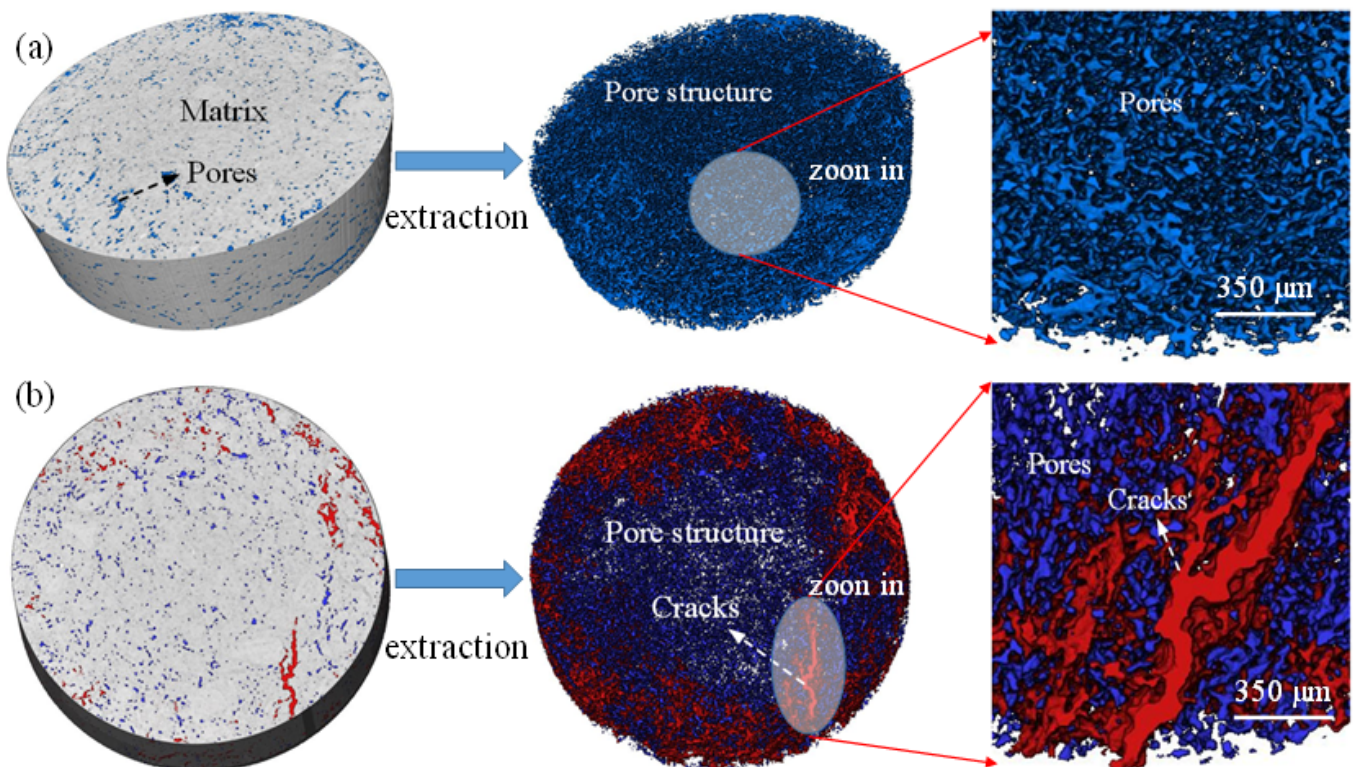
Figure 10

Microstructural evolution features of C/SiC porous ceramic using high-resolution X-ray tomography: (a) without load; (b) failure after loading.

Supplementary Files

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- Streamlines000000000030.gif