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Biomorphic silicon/silicon carbide ceramics from birch powder

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Abstract

A novel process has been developed for the fabrication of biomorphic silicon/silicon carbide (Si/SiC) ceramics from birch powder. Fine birch powder was hot-pressed to obtain pre-templates, which were subsequently carbonized to acquire carbon templates, and these were then converted into biomorphic Si/SiC ceramics by liquid silicon infiltration at 1550 °C. The prepared ceramics are characterized by homogeneous microstructure, high density, and superior mechanical properties compared to biomorphic Si/SiC ceramics from birch blocks. Their maximum density has been measured as 3.01 g/cm^3. The microstructure is similar to that of conventional reaction-bonded silicon carbide. The Vicker's hardness, flexural strength, elastic modulus, and fracture toughness of the biomorphic Si/SiC were 19.6 ± 2.2 GPa, 388 ± 36 MPa, 364 ± 22 GPa, and 3.5 ± 0.3 MPa m$^{1/2}$, respectively. The outstanding mechanical properties of the biomorphic Si/SiC ceramics are assessed to derive from the improved uniform microstructure of the pre-templates made from birch powder.

Keywords: B. Microstructure; C. Mechanical properties; D. SiC; D. Birch powder

1. Introduction

Liquid silicon infiltration process is a typical process for fabrication of reaction formed silicon carbide (RF-SiC) and reaction bonded silicon carbide (RB-SiC) with porous pure carbon template and template containing silicon carbide and carbon as precursors, respectively [1,2]. RB-SiC has been extensively studied and commercially produced [3–7]. RF-SiC, invented by Hucke [8,9], have not yet been manufactured in industrial scale due to the complex fabrication process and the high cost of carbon template, although the flexural strength of RF-SiC is superior to that of RB-SiC [1]. Therefore, developing simple and low-cost processes to fabricate RF-SiC remains still a great challenge to materials scientists.

In the past decade, there has been a surge of interest in fabrication of RF-SiC with renewable natural resources as starting materials. The most common practice is to transfer wood blocks (including pine, oak, maple, birch, mahogany, etc.) into porous pure carbon templates by carbonization and then to prepare silicon/silicon carbide ceramics, namely biomorphic Si/SiC ceramics, by liquid silicon infiltration [10–27]. Instead of wood blocks, medium density fiberboards (MDF) [19,28–31] and wood-based composites [31,32] were also used as starting materials with relatively homogeneous structure compared to natural wood. In addition, similar studies were conducted on a number of non-wood materials, such as bamboo [19,33,34], cotton fabric [35], and lotus root [36]. Relevant study indicated that some biomorphic Si/SiC ceramics from wood blocks and wood based composites obtained flexural strengths and elastic moduli comparable to those of conventional RB-SiC ceramics [11–17,31,32].

Compared to Hucke's method, the biomorphic Si/SiC fabrication process is simple and cost effective because it gets rid of preparation of porous polymer pre-template, in which expensive chemicals are necessary. However, the starting materials for biomorphic ceramic fabrication, such as wood block, bamboo and MDF, normally exhibit the following disadvantages [37–41]: relatively low and uncontrollable density, non-uniform density distribution, evident variation of composition and structure, inevitable existence of structural

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flaws, fluctuant shrinkages and weight loss during carbonization, and so on. These defects will lead in unpredictable and uncontrollable microstructure and mechanical properties for the ceramics.

The aim of the present work is to fabricate biomorphic Si/SiC ceramics with improved uniform structure and outstanding mechanical properties from birch powder. Fine birch powder was pressed into blocks to acquire pre-templates, which were subsequently carbonized to form porous carbon templates and then infiltrated with liquid silicon to obtain dense biomorphic Si/SiC ceramics. The hot-pressing process, the carbonization behavior of the pre-templates made from birch powder, and the microstructures of both the carbon templates and the sintered bodies have been investigated in detail. Furthermore, evaluations of the mechanical properties of the ceramics have also been carried out. For a comparison, studies have also been conducted on birch blocks for Si/SiC ceramic fabrication.

2. Experimental procedure

Birch (*Betula platyphylla suk*), a kind of dicotyledonous plant of Northern China, was milled to powder with an average diameter of 35 μm. A temperature and humidity regulator (SETH-Z-022R, Shanghai ESPEC Environment Equipment Co., Ltd., China) was used to adjust the moisture content of the powder to (10.0 ± 2.0) wt%. The damp powder was added into a stainless steel mold without any additive or adhesive and pressed into blocks of dimensions 100 mm × 80 mm × 10 mm at 180 °C and 10 MPa for 45 min by means of a flat vulcanizer (YXD-50, Shanghai Zimmerli Weili Rubber & Plastic Machinery Co., Ltd., China) to obtain the pre-templates.

The pre-templates were carbonized at up to 1200 °C and kept for 2 h in a furnace (ZT-100-20, Shanghai Chenrong Electric Furnace Co., Ltd., China) under a low positive pressure of flowing nitrogen as a protective medium. To avoid cracking of the pre-templates during carbonization, the temperature was increased at a rate of 0.5 °C/min in the range 200–500 °C. The carbonized plates were cut into blocks of dimensions 50 mm × 40 mm × 6 mm as carbon templates and then infiltrated with liquid silicon (purity 99.0 wt%, Beijing Yuanchuang Magnesium Co., Ltd., China) at 1550 °C for 30 min under a vacuum (10–30 Pa) in a graphite element furnace (EJ-13, General Research Institute for Nonferrous Metals) to yield the Si/SiC ceramics.

For a comparison, birch blocks were also carbonized and then infiltrated with liquid silicon together with the pre-templates made from birch powder to fabricate biomorphic Si/SiC ceramics.

The densities of pre-templates made from birch powder, birch blocks, and carbon templates were determined by the weight and dimensions of the samples. The vertical density profiles (VDP, which refers to the density variation in the height direction of man-made board from wood [42]) of the pre-templates made from birch powder were measured with an X-ray scanning densitometer (DA-X/DA-1, GreCon Greten GmbH & Co. KG, Germany). The densities of the Si/SiC ceramics were characterized by the Archimedes' method. The microstructures of the carbon templates and ceramics were observed by scanning electron microscopy (SSX-550, Shimadzu Corp., Japan) and light microscopy (KH–1 000, Shanghai Hirox Instrument Technology Co., Ltd., China), respectively. The porosity distributions of the carbon templates were determined with a mercury porosimeter (Autopore-9220, Micromeritics Instrument Corp., USA).

The Vickers hardnesses of the biomorphic Si/SiC ceramics were measured using a Vickers micro indentation apparatus (HVS-5, Laizhou Huayin Testing Instrument Co., Ltd., China) with a 1000 g load and a residence period of 10 s. At least eight indentations were made for each sample to get the average value and the standard deviation. Flexural strength, elastic modulus and fracture toughness were tested at room temperature with a universal testing machine (AG-2000A, Shimadzu Corp., Japan). The specimens for the above three tests were ground and polished to an 1 μm finish. The corresponding specimen sizes are 45 mm × 4 mm × 3 mm, 45 mm × 4 mm × 2.4 mm, 36 mm × 6 mm × 3 mm, respectively. For flexural strength tests three-point bending method was performed with a span of 30 mm and a crosshead speed of 0.5 mm/min. Fracture toughness tests on the ceramics were performed by the single-edge-precracked-beam (SEPB) method. For those measurements, eight to twelve specimens were tested for each measurement to get the average values and standard deviations.

3. Results and discussion

By hot-pressing, we hoped to obtain pre-templates made from birch powder with defined density and uniform VDP. However, the shape of VDP is influenced by the combined effects of many factors, including wood species, closure rate of the press machine, moisture content, press temperature, and so on, which makes it difficult to achieve a uniform VDP [43]. Through preliminary experiments, it was established that a four-step hot-pressing process (Fig. 1) was advantageous for obtaining pre-templates made from birch powder with uniform VDP and densities in the range of 0.60–1.10 g/cm^3 (Fig. 2).

Birch blocks and pre-templates made from birch powder were carbonized under the same conditions and their weight

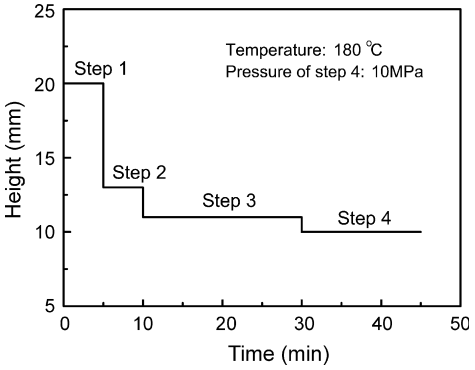

Fig. 1. Mold-pressing schedule for pre-templates made from birch powder with a moisture content of 15 wt%. Height refers to the height of pre-templates in the mold-pressing process.

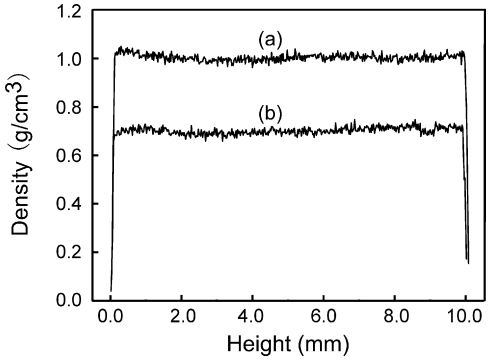

Fig. 2. Vertical density profile (VDP) measurement results of pre-templates with average densities of 1.00 g/cm^3 (curve (a)) and 0.70 g/cm^3 (curve (b)) made from birch powder.

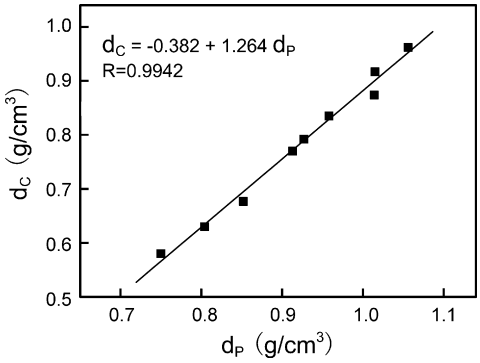

Fig. 4. The linear relationship obtained between the densities of pre-templates made from birch powder (d_P) and those of the resulting carbon templates (d_C).

losses and shrinkages during carbonization were calculated. The results are shown in Fig. 3. They exhibited similar weight losses but apparently different shrinkages. The birch blocks displayed different shrinkages in three different directions, whereas the pre-templates made from birch powder displayed equal shrinkages in their lengths and widths but a different degree of shrinkage in the height direction.

Such behavior of carbonization can be explained in terms of the similarities and differences in the components and structures of these samples. Wood is a naturally grown composite material with a highly anisotropic cellular structure at both the macroscopic and microscopic levels. Consequently, it exhibits different shrinkages in three directions. Furthermore, there is also variation in the structure and chemical components among trees of a given species and even within a single tree. Therefore, both the weight losses and shrinkages of birch blocks during carbonization are subjected to large standard deviation, which implies that the density of carbon template from birch varies within a wide range. The milling treatment of birch and the hot-pressing of birch powder do not change the chemical composition, and so the pre-templates made from birch powder display a similar weight loss to birch blocks. The pre-templates can be seen as a reorganized wooden material, in which the structural characteristics in the width and length directions are the same, but differ in the height direction. Consequently, the pre-templates shrink to the same extent in the width and length directions, but to a different extent in the height direction. Compared to birch blocks, the structure of the pre-templates made from birch powder is more homogeneous and less variable, and the standard deviations in the shrinkages are correspondingly small.

Fig. 4 shows the correlation between the densities of the pre-templates made from birch powder and the corresponding carbon templates. By linear fitting, it can be seen that the two densities are highly linearly related, with a correlation coefficient of 0.9942. On this basis, it should be possible to

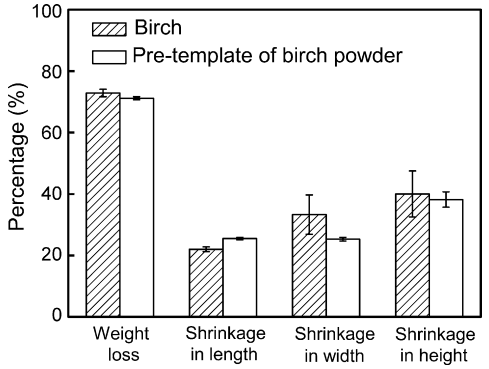

Fig. 3. Weight loss and shrinkage of birch blocks and pre-templates made from birch powder during carbonization. For the birch blocks, the length, width, and height directions correspond to the axial, radial, and tangential directions of the birch trunk.

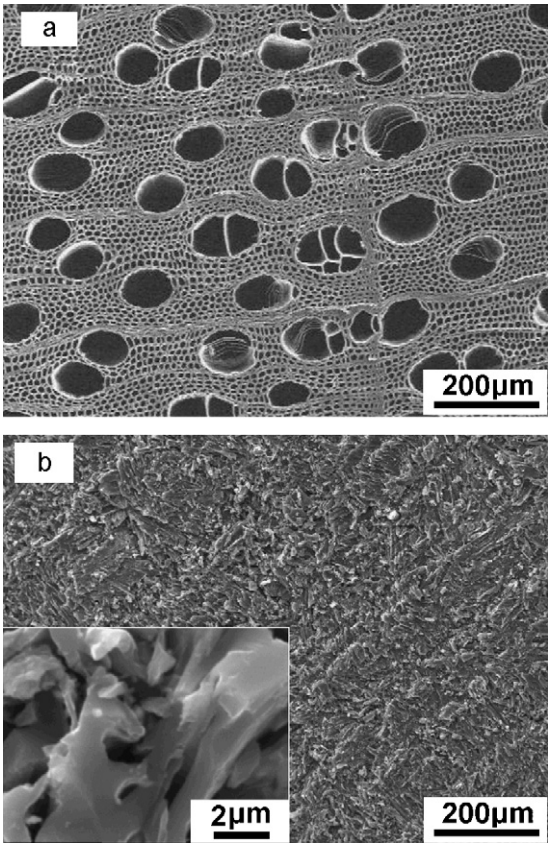

Fig. 5. SEM photographs of carbon templates derived from (a) birch block and (b) pre-template made from birch powder.

control the density of the carbon template by adjusting the density of the pre-template made from birch powder.

Figs. 5 and 6 show SEM photographs and mercury intrusion measurement results of carbon templates obtained from a pre-template made from birch powder and from a birch block. They show the different pore structures of the respective carbon templates.

For the carbon template obtained from a birch block, distinctly different pore diameters can be seen in the SEM photograph (Fig. 5(a)). Pores with diameters of 40–120 μm and 5–15 μm are inherited from vascular vessels and tracheidal channels, respectively. The mercury intrusion measurements were in good agreement with the SEM observations. Fig. 6(a) shows the heterogeneous pore size distribution of the carbon template obtained from birch. The cumulative pore volume percentages of pores with diameter larger than 4.5 μm and with diameter smaller than 0.5 μm are about 30.7% and 1.0%, respectively.

For the carbon template derived from the pre-template made from birch powder, no visible difference in pore diameter is evident from the SEM image (Fig. 5(b)). The mercury intrusion measurement shows a very narrow pore diameter distribution with an average value of about 3.0 μm (Fig. 6(b)), very similar to that of pure carbon template prepared by Hucke's method [1,44]. The cumulative pore volume percentages of pores with diameter larger than 4.5 μm and with diameter smaller than 0.5 μm are about 2.4% and 5.0%, respectively. Thus, about 92.6% of the pore volumes are attributed to pores with diameters between 0.5 μm and 4.5 μm.

Carbon templates of various densities were infiltrated with liquid silicon in a graphite furnace. In theory, for a pure carbon template, the ideal density of a Si/SiC ceramic can be calculated

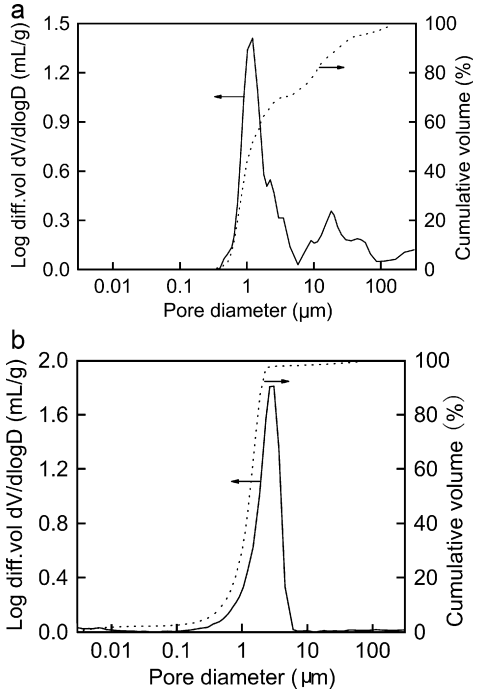

Fig. 6. Pore size distributions of carbon templates (a) from birch of density 0.58 g/cm^3 and (b) from a pre-template made from birch powder of density 0.70 g/cm^3.

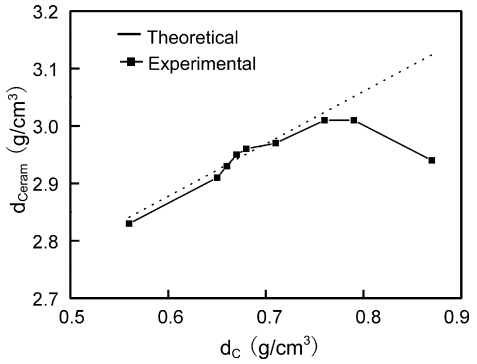

Fig. 7. Density correlation between the carbon template and the corresponding biomorphic Si/SiC ceramic.

by the following equation [13]:

$$d_{\text{ceram}} = d_{\text{Si}} + 1.038 \cdot d_{\text{C}}(d_{\text{SiC}} - d_{\text{Si}}) \quad (1)$$

where d_{ceram}, d_{C}, d_{Si}, and d_{SiC} are the densities of the Si/SiC ceramic, the carbon template, silicon, and SiC, respectively. From Eq. (1), it is apparent that when d_{C} is smaller than 0.963 g/cm^3, the higher the value of d_{C}, the higher the value of d_{ceram}. This implies that the density of the ceramic can be adjusted by controlling the density of the carbon template. In reality, using a carbon template with an ideal microstructure is unfeasible for dense SiC fabrication, since this leads to the presence of free silicon in RF-SiC and RB-SiC. A homogeneous pore diameter distribution and optimal density of the carbon template are

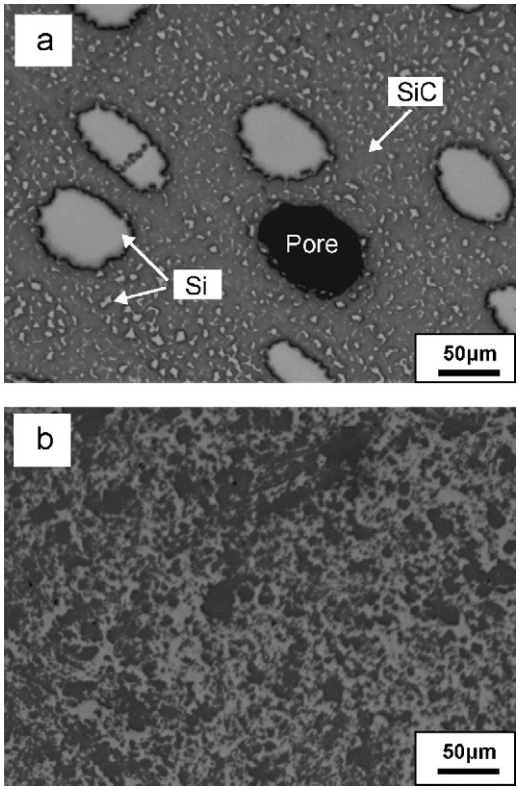

Fig. 8. Optical micrographs of the biomorphic Si/SiC ceramics from (a) birch block and (b) birch powder pre-template.

Table 1
Comparison of mechanical properties of biomorphic Si/SiC ceramics and RB-SiC.

Sample	Si/SiC ceramic from birch powder[a]			Si/SiC ceramic from birch[b]		RB-SiC
Density, g/cm^3	3.01	2.9	2.82	2.82	2.87	3.04
Vicker's hardness, GPa	19.6 ± 2.2	15.9 ± 1.7	15 ± 2.6	16.1 ± 3.4	18.5 ± 3.1	18.8 ± 1.9
Flexural strength, MPa	388 ± 36	272 ± 31	257 ± 25	243 ± 43	277 ± 39	330 ± 45
Elastic modulus, GPa	364 ± 22	330 ± 31	-	265 ± 17	314 ± 20	342 ± 14
Fracture toughness, MPa m$^{1/2}$	3.5 ± 0.3	3.1 ± 0.15	2.6 ± 0.2	2.8 ± 0.1	2.8 ± 0.2	3.3 ± 0.1

[a] The length of the specimen corresponds to the length direction or width direction of the pre-templates made from birch powder.
[b] The length of the specimen is parallel to the axial direction of the birch trunk.

beneficial for decreasing the amount of free silicon and improving the mechanical properties of the ceramic.

The correlation between the density of the carbon template (d_C) and that of the corresponding biomorphic Si/SiC ceramic (d_{ceram}) is shown in Fig. 7. The experimental d_{ceram} is consistent with the theoretical value obtained from Eq. (1) when d_C is lower than 0.76 g/cm^3, but a difference between them is evident when d_C is higher than 0.76 g/cm^3. This reveals that a carbon template with a density lower than 0.76 g/cm^3 can be completely infiltrated with silicon. For the carbon template with a density of 0.76 g/cm^3, the ceramic attained a density of 3.01 g/cm^3, which is very close to that of commercial RB-SiC [45].

When birch blocks were used to fabricate Si/SiC ceramics, the density of the carbon templates mainly depended on the density of the birch and the carbonization conditions. Under the carbonization conditions of this research, the density of the resulting carbon template was in the range 0.55–0.62 g/cm^3 when the density of the birch was 0.68–0.74 g/cm^3. Usually, the carbon template could be completely infiltrated with liquid silicon by virtue of the all-open pore structure of birch and our careful selection of birch blocks. The corresponding ceramic density was about 2.82–2.89 g/cm^3.

Fig. 8 shows optical micrographs of biomorphic Si/SiC ceramics from birch block and birch powder at 200× magnification. The ceramic obtained from birch retains the microstructure of birch. Silicon spots with obviously different diameters are distributed within the ceramic. There are also some pores from tracheas of the birch that are not filled with free silicon, although these are not observed in every micrograph at 200× magnification. For the ceramic from birch powder, the distribution of the two phases (silicon and silicon carbide) differs greatly from that of the ceramic derived from birch, but this sample has the characteristics of RB-SiC and RF-SiC reported in the literature [44,46]. Silicon spots are uniformly distributed in the ceramic and the only pores present are of diameter smaller than 5 μm.

The mechanical properties of the two as-fabricated biomorphic Si/SiC ceramics and a commercial RB-SiC sample were preliminarily investigated, and the results are shown in Table 1. For the ceramic from birch powder, the higher the density, the better the mechanical properties, which is consistent with the results of previous research by Martínez Fernández [16]. For biomorphic Si/SiC ceramic from birch powder with a density of 3.01 g/cm^3, the Vicker's hardness, flexural strength, elastic modulus, and fracture toughness were 19.6 ± 2.2 GPa, 388 ± 36 MPa, 364 ± 22 GPa, and 3.5 ± 0.3 MPa m$^{1/2}$, respectively, which are higher than those of the biomorphic Si/SiC ceramic from birch and the commercial RB-SiC. These superior mechanical properties may derive from the improved microstructure of the pre-templates made from birch powder. These results suggest that the mold-pressing process is effective for enhancing the mechanical properties of biomorphic Si/SiC ceramics.

4. Conclusion

The newly developed process can enhance the density, microstructure homogeneity, and mechanical properties of biomorphic Si/SiC ceramic when compared to biomorphic Si/SiC ceramic from birch. The fabricated biomorphic Si/SiC ceramic has a maximum density close to that of commercial RB-SiC, a homogeneous microstructure similar to those of RB-SiC and RF-SiC, and superior mechanical properties to those of biomorphic Si/SiC ceramic from birch and RB-SiC. The maximum density of biomorphic Si/SiC ceramic from birch powder is 3.01 g/cm^3. The corresponding Vicker's hardness, flexural strength, elastic modulus, and fracture toughness are 19.6 ± 2.2 GPa, 388 ± 36 MPa, 364 ± 22 GPa, and 3.5 ± 0.3 MPa m$^{1/2}$, respectively.

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