



Preparation and characterization of bioinspired three-dimensional architecture of zirconia on ceramic surface

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ABSTRACT

Inspired by bioinspired coating with nanostructures, a heterogeneous zirconia (ZrO₂) ceramic membrane has been extensively aspired for changing its intrinsic surface properties. Hence, synthesizing specific nano-micro-scale architectures of ZrO₂ has achieved great interest due to its novel surface properties and application. Here we reported a sol-gel strategy that involved the organic of P123, Brij@C10 and inorganic of Zr-precursor. Impressively, the heterogeneously growth demonstrated that an ultrathin buffer layer could be utilized as the rational mediation of thermal expansion mismatches between their heterogeneously interfaces. This work will raise the potential of bioinspired membrane for emerging applications in industrial processes, such as surface adhesion.

1. Introduction

Many natural structures have much novel properties owing to their unique multiscale structures, such as low adhesion of lotus leaves [1,2]. Some researchers had found that materials that were less than 100 nm in size have properties that were either new or different from the corresponding bulk forms [3]. These nanoscale structures have inspired researchers to construct bioinspired nanostructures at pre-designed substrates [4–7]. Impressively, Owing to the dramatic increase of the surface area comparing with flat surface, these bioinspired surfaces changed their properties in response to environmental stimuli, such as temperature and certain chemicals [8–12]. However, owing to the high reactivity of ion precursors towards hydrolysis and condensation, there still has a prominent challenge to synthesize 3D bioinspired nanostructure on solid surface, especially for ceramic materials.

Above challenge has driven a continuing effort into the preparation of bioinspired 3D structures and exploring their novel properties. Some reported work had utilized the solution-based synthetic approach to constructed hierarchical structures, and they used self-assembled organic surfactants as stabilizing agent, such as diblock copolymers [13–16]. The self-assembly of chemically different moieties had been distinctly illustrated in coordination complexes, supramolecular structures, and colloidal lattices among other systems [17–20]. The mechanisms of mixing primarily depended on specific interfacing of chemically, physically or geometrically complementary objects [21].

Impressively, some researchers had found that adding a second small-sized copolymer could improve structural stability, such as Brij56. The intercalation of the second immiscible macromolecule increased the space between the chains [22,23]. Moreover, the well-defined diblock copolymer building blocks formed abundantly micellar skeletons in nanoscale, which was surrounded by macro-molecular precursors and stretched into the solvent phase [24]. Hence, we obtained perfect micelles of various morphologies by increasing the length ratio and the degrees of polymerization of the two insoluble-to-soluble blocks.

In this communication, we extended a parallel self-assembly strategy to rationally produce branched ZrO₂ nanowires with size from 30 to 60 nm and mesopores of 6–30 nm in diameter on ZrB₂ ceramic matrix composite surface. The non-ionic amphiphilic triblock copolymer P123 (EO₂₀PO₇₀EO₂₀) and Brij@C10 (C₁₆EO₁₀) were the template agents, and zirconium propoxide (Zr(OC₃H₇)₄) was the inorganic precursor. We reported the characterization of aligned and branched nanowire arrays with a controlled growth that would reinforce wettability, and a tentative mechanism of present system was proposed.

2. Experimental

2.1. Preparation of the reactant solution

A micellar solution (15–25 wt%) of P123 (EO₂₀PO₇₀EO₂₀, average molar weight of 5800, Aldrich) and Brij C10 (C₁₆(EO)₁₀, average molar

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weight of 683, Aldrich) were prepared by dissolving P123 and Brij® C10 with a weight ratio of 1:1 in completely deionized water with stirring for about 4 h under ambient conditions. A proper amount of zirconium propoxide ($\text{Zr}(\text{OCH}_2\text{CH}_2\text{CH}_3)_4$, molar weight of 327.57, Aldrich) was dropwise added into the above solution to offer a surfactant-to-Zr molar ratio of 0.35 with stirring for 1 h.

2.2. Preparation of samples

Acid treatment was performed by immersing the ceramic materials in a hot aqueous solution of concentrated sulfuric acid (H_2SO_4) and hydrogen peroxide (H_2O_2) with a weight ratio 7: 3 for 1 h at 90 °C. The pretreated samples were subsequently washed with deionized water. In order to manipulate the thickness of rough and heterogeneous coatings, micelles were transferred onto the medium by spin-coating at 3000 r/min rotating speed. These obtained samples were transferred into a Teflon-lined autoclave and then were heated at 130 °C for 20 h to produce a self-assembled nanostructure film. Thermal annealing was implemented at 300 °C for 1 h to remove the surfactant species, and then it was increased to 700 °C for 5 h to aggrandize crosslinking of the inorganic framework.

2.3. Characterization of ZrO_2 films

The morphology and structure of the ZrO_2 nanostructure film was characterized by a focusing ion/electronic double beam micro electron microscopy (FIB/SEM, HELIOS NanoLab 600i, FEI, USA) and transmission electron microscope (TEM, JEM-2100UHR, JEOL, Japan). Prior to increase surface conductivity, the samples were coated with thin gold films under vacuum conditions using a precision etching coating sputter (PECS™ 682, Gatan, USA). The crystallinity of the samples after annealing treatment was measured using an X-ray diffractometer with Cu target (XRD, Empyrean, Panalytical, Netherlands). Thermal behavior of the as-prepared ZrO_2 powder was analyzed with thermogravimetric and differential scanning calorimetry (TGA-DSC, SDTA600, TA, USA).

2.4. Surface wettability

Contact angle measurements were performed under room temperature (295 K) using a commercial hydrophobicity instrument (JC2000D5, China), with an experimental error of $\pm 1^\circ$. Five measurements were taken to accurately calculate the mean apparent contact angle (CA) of the bioinspired ZrO_2 membrane. The volumes of the denoised droplets were 1.0 μl . The contact angles were precisely measured by recording images with a camera after the droplet had rested on the surface for 2s and 30s, respectively.

3. Result and discussion

Three dimensional (3D) bionic surfaces upon substrate can be fabricated by sol-gel approach and spin coating technology. In the rational design of hierarchical structure, the pretreatment of substrate with hydroxyl bonds and graphite sheets were two pivotal elements for enhancing interactions between colloidal sol and ZrB_2 ceramic matrix composite (substrates). Fig. 1 is schematic illustration of preparing ZrO_2 hierarchical structure on ceramic surface. The surface of substrate was predominantly covered with hydroxyl (R-OH) functional groups by acid-based treatment (Fig. 2a–b). This treatment increased the reactive R-OH functional groups, removed organic contaminant and produced graphite nanosheets. Graphite nanosheets had ability to enhance their interfacial adhesion with hierarchical ZrO_2 structure (Fig. 2c–d). This pretreated process was confirmed by evidently downgraded CA (from 78 to 23°). Hence, a combination of graphite nanosheets and R-OH functional groups increases the interfacial adhesion between substrates and prepared sol. The thickness of the solution membrane is stipulated by the capillary number and rotational velocity of spin coating.

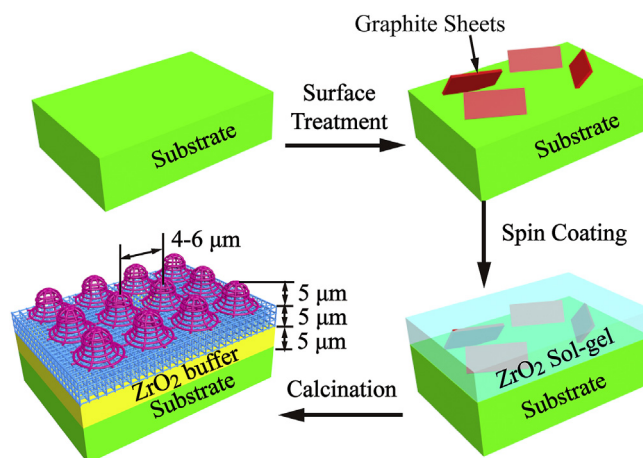


Fig. 1. Schematic illustration of preparation and surface treatment for constructing ZrO_2 hierarchical structure. The graphite sheets enhanced the assembly process of substrate and prepared gel.

The specific surface morphology of the 3D ZrO_2 hierarchical structure was characterized by SEM. The walls of the 3D hierarchical structure (Fig. 2e–f), viewing from the flank of the coating, were composed of successive protuberance. The height and bottom diameter of protuberance were 5 μm and 4–6 μm , respectively. The micrograph (Fig. 2d), observing from the top side of the membrane, depicted the multi-scale organization of the as-synthesized membrane. Fig. 2e–f exhibited high-resolution SEM images of hierarchical structures, which emerged the cross-linked bioinspired nanostructure. This hierarchical structure was composed of ZrO_2 nanowires (diameter 30–60 nm). The average interval of nanowires was 30 nm (half length). These characterizations illustrate that a combination of preparing reactant solution and high-temperature calcination constructs ZrO_2 hierarchical structures on substrates. Self-assembly sol-gel approach makes membrane have large surface area and stable morphology.

We further characterized tetragonal or monoclinic phase ZrO_2 crystalline with XRD and TEM analysis. The wide-angle XRD patterns of substrate and self-assembly ZrO_2 powder were exhibited in Fig. 3. Matrix material and the ceramic surfaces after acid treatment with concentrated H_2SO_4 and H_2O_2 were both composed of ZrB_2 , silicon carbide, and graphite (Fig. 3a and b). The percentage of synthesized tetragonal ZrO_2 powder was 39% (Fig. 3c), whereas it was 32% for oxidized tetragonal ZrO_2 on substrate (buffer layer) (Fig. 3d). Hierarchical ZrO_2 membrane were composed of monoclinic (65%) and small amounts of tetragonal ZrO_2 (35%) in Fig. 3e. A representative TEM image of synthesized ZrO_2 powder (Fig. 4a) revealed that hierarchical structure was apparently fluffy owing to their dark contrast. The corresponding selected area electron diffraction (SAED) analysis of the magnified image demonstrated that the pore walls, composing of nanocrystalline ZrO_2 , emerged diffuse electron diffraction rings. They were confirmed to be tetragonal ZrO_2 crystalline with high-percentage monoclinic. A HRTEM image (Fig. 4c) depicted a high crystallinity of the ZrO_2 with well-defined lattice planes. Combining with above XRD and TEM analysis, we conclude that prepared membrane and buffer layer are both composed of tetragonal and high-percentage monoclinic ZrO_2 crystalline (see Fig. 5).

The pore size distribution of ZrO_2 membrane was characterized by N_2 adsorption-desorption. Its curves depicted an IV isotherm with H3 hysteresis loop (Fig. 4). The pore volume and surface area were 0.063 $\text{cm}^3 \text{g}^{-1}$ and 31.62 $\text{m}^2 \text{g}^{-1}$, respectively. Quantitative data of hierarchical nanopores had been achieved using N_2 adsorption-desorption isotherms, and its radius were 6.8 nm, 14.5 nm and 23.8 nm, respectively. The pore size distribution of ZrO_2 powder in the range of 2.1–77.7 nm was performed using the density functional theory (DFT), as depicted in the inserted part of Fig. 4. These results illustrate that the

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