第 48 卷第 3 期
2020 年 3 月

硅 酸 盐 学 报
JOURNAL OF THE CHINESE CERAMIC SOCIETY

Vol. 48，No. 3
March，2020

http://www.gxyb.cbpt.cnki.net

DOI：10.14062/j.issn.0454-5648.20190285

基于振荡压力烧结的氧化锆陶瓷水热老化行为

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摘 要：采用振荡压力烧结法制备了致密度高、晶粒细化、力学性能优异的氧化锆陶瓷，研究了水热老化时间对两种氧化锆陶瓷(振荡压力烧结 OPS，热压烧结 HP)的显微结构、相变及力学性能的作用规律及机制。Raman 分析表明：随着老化时间的延长，2 种材料中单斜相氧化锆的含量缓慢增加，但 OPS 氧化锆在 178、189 cm^{-1} 处的峰值强度显著低于 HP 氧化锆。水热老化导致氧化锆表面微裂纹、颗粒粗化等现象，当老化时间为 18 h 时 OPS 氧化锆和 HP 氧化锆的抗弯强度分别为 1 216 和 878 MPa。纳米压痕测试表明：OPS 氧化锆的力学参数显著优于 HP 氧化锆。因此，OPS 烧结技术有望极大拓展氧化锆陶瓷在生物医学领域的应用。

关键词：振荡压力烧结；相变；抗弯强度；晶粒尺寸
中图分类号：TB332　**文献标志码**：A　**文章编号**：0454–5648(2020)03–0391–08
网络出版时间：2019–12–13



Hydrothermal Aging of Zirconia Ceramics Consolidated by Oscillatory Pressure Sintering

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Abstract: The use of zirconia ceramics in humid environments is limited by the phase transformation from the tetragonal phase to the monoclinic phase. In this study, an oscillatory pressure sintering (OPS) technique was developed for the preparation of zirconia ceramics that were characterized by high density, fine grain size and excellent mechanical properties. The hydrothermal aging behaviors of the oscillatory pressure sintered and hot-pressed zirconia were investigated. The Raman analysis showed strong peaks at 178 and 189 cm^{-1} in both the oscillatory pressure sintered and the hot-pressed specimens, but the peak intensities of the oscillatory pressure sintered specimen were much weaker than those of the hot-pressed specimen. When the specimens were aged for 18 h, the flexural strength of the oscillatory pressure sintered zirconia reached approximately 1 216 MPa, which is much higher than that of the hot-pressed zirconia. Therefore, the OPS technique is promising for the preparation of zirconia ceramics that are used in the biomedical field.

Keywords: oscillatory pressure sintering; phase transformation; flexural strength; grain size

1 Introduction

Yttria-stabilized tetragonal zirconia polycrystalline (Y-TZP) ceramics exhibit exceptional physical and mechanical properties due to the stress-induced transformation from the tetragonal phase to the monoclinic phase at room temperature[1–3]. Thus, they can be used for various applications that range from engine components, sensors and cutting tools to thermal barrier coatings. However, when zirconia ceramics are exposed in a humid environment at low temperatures from 100 to 300 ℃, this phase transformation also occurs due to the presence of water and/or water vapor. This phenomenon is known as hydrothermal aging or low-temperature degradation[4–7].

收稿日期：2019–04–28。　修订日期：2019–05–08。
基金项目：辽宁科技大学开放课题(USTLGCZX201804)资助。
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It is well known that hydrothermal aging results in microcracks on the surface of the materials and strength loss of the modules. Thus, a comprehensive understanding and characterization of the hydrothermal aging behaviors of zirconia has become a critical issue for the applications that are in contact with water and/or water vapor.

Hydrothermal aging of zirconia was first proposed by Kobayashi thirty years ago[8]; since then, many attempts have been made to obtain a reasonable explanation for the degradation mechanisms, thereby enabling the prevention of hydrothermal aging of zirconia[9–12]. Generally, hydrothermal aging of zirconia initiates at the surface that is in contact with water and/or water vapor and then progresses gradually towards the bulk of the material. The phase transformation of grains from $t \rightarrow m$ is accompanied by a volume expansion, so the volume expansion of transformation zone gives rise to severe defects, such as microcracks, grain pullout and surface roughening, which finally leads to a loss in the fracture strength of the ceramic. To date, although the hydrothermal aging mechanisms are still under debate, researchers believe that many factors, such as different grain sizes, residual or applied stresses, density, content of Y_2O_3 in the tetragonal phase, proportion of cubic phase, and doping with more than one oxide can influence the aging process[13–16]. Thus, the usual approaches to alleviate aging in zirconia ceramics are as follows: (i) reduce the grain size, (ii) increase the amount of the phase stabilizer, (iii) formulate the composites appropriately, (iv) modify the surface and (v) enhance the density[17–19]. For a given zirconia ceramic, the relative density, the average grain size and the homogeneity of the phase distribution are of primary importance[20–21].

To prepare Y-TZP ceramics with high relative density as well as uniform and fine grains, pressure-assisted sintering techniques are considered a practical and efficient approach, during which the external applied load can provide an additional force to accelerate densification while inhibiting grain growth. Currently, pressure-assisted sintering techniques generally consist of hot pressing (HP), spark plasma sintering (SPS) and hot isostatic pressing (HIP)[22–25]. However, only a constant pressure is applied for these techniques, and that cannot accomplish what the pressure is capable of during the sintering stage. It is believed that ceramic powders under an oscillatory pressure can be different from those under a constant pressure in terms of the densification route and grain growth since oscillatory pressure can enhance particle rearrangement at low temperatures and plastic deformation of grains at high temperatures.

In previous studies[26–29], we proposed an oscillatory pressure sintering (OPS) technique for the preparation of zirconia ceramics with high performance. When applying an oscillatory pressure during the sintering stage, the zirconia ceramics exhibited a high density and uniform and fine grains compared with those prepared by hot pressing (HP) under the same temperature and constant pressure conditions. Due to these microstructure evolutions, the flexural strength of the oscillatory pressure sintered specimen reached approximately 1 556 MPa, whereas that for the specimen prepared by hot pressing (HP) was 1 224 MPa. When applying an oscillatory pressure at high temperatures, several new densification mechanisms, such as particle rearrangement, grain plastic deformation and grain boundary sliding occurred, which provided a high driving force for the densification of the particles. Based on these results, the OPS technique is considered as an attracting technique for preparing zirconia ceramics.

In this study, the hydrothermal aging behaviors of zirconia ceramics using the OPS technique were investigated. The as-received ceramics were hydrothermally treated in autoclaves at 134 ℃ and 0.2 MPa for periods of 6 h, 12 and 18 h. It has been shown that 1 h of autoclave treatment of zirconia at 134 ℃ has the same effect as 3–4 a in vivo at 37 ℃[30–31]. Thus, this is an adequate accelerated program for life cycle studies of zirconia biomedical implants. As a comparison, zirconia ceramics were sintered by the hot-pressing method with the same starting powders and then aged in the same hydrothermal condition. The variation of the microstructure, phase composition, and mechanical properties of the oscillatory pressure sintered and hot-pressed specimens as a function of the the aging temperatures were investigated in detail.

2 Experimental procedures

2.1 Raw materials and sample preparation

Commercially available zirconia powder (Y-TZP with 3% (mole fraction) Y_2O_3 and an average size of 80 nm) prepared by the coprecipitation method was chosen as the starting powder. A brief description of the processing and hydrothermal aging of the Y-TZP ceramics is presented in Fig. 1. From the TEM image in Fig. 1, the zirconia particles exhibited a uniformly distributed size and regular morphology. Before sintering, the as-received powders were first granulated using the spray drying method.

The preparation of the oscillatory pressure sintered specimens was carried out using a self-developed oscillatory pressure sintering apparatus, as shown in Fig. 1. This apparatus could apply an oscillatory pressure with controllable amplitude and frequency to the powders in the graphite die. For the preparation of the oscillatory pressure sintered specimens, the raw particles were first placed in a graphite die and then heated gradually to 1 450 ℃ at a heating rate of 200 ℃/h. When the temperature reached 1 450 ℃, an

oscillatory pressure from 27.5–32.5 MPa was applied for 90 min. The shape of the oscillatory pressure is indicated by the brown curve in Fig. 1. After soaking at the peak temperature for 90 min, the sample was cooled to 900 ℃ with a cooling rate of 200 ℃/h; at the same time, the oscillatory pressure was gradually unloaded.

To prepare zirconia ceramics by hot pressing, the granulated particles were first placed in a cylindrical graphite die with an inner diameter of 50 mm and then sintered in a hot-pressing furnace (MULTI-5000, Fujidempa, Japan). The sintering schedule was similar to that of the oscillatory pressure sintered specimens, except that a constant pressure of 30 MPa was applied at 1 450 ℃ without any oscillatory pressure.

Both the hot-pressed and the oscillatory pressure sintered ceramics were post-processed by incising, grinding and polishing, and then, they were hydrothermally aged at 134 ℃ and 0.2 MPa in an autoclave. The hydrothermal aging periods in this study were limited to 6 h, 12 h and 18 h. According to the 6 h, 12 h and 18 h aging periods, the HP specimens were labeled as HP-6, HP-12 and HP-18, respectively; similarly, the oscillatory pressure sintered specimens were labeled as OPS-6, OPS-12 and OPS-18, respectively.

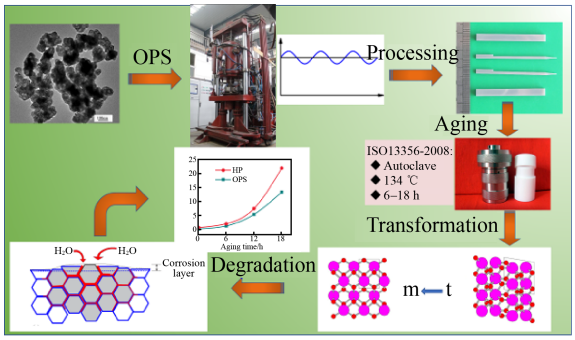


Fig. 1　A brief description of the preparation and hydrothermal aging of the zirconia ceramics

2.2　Characterization methods

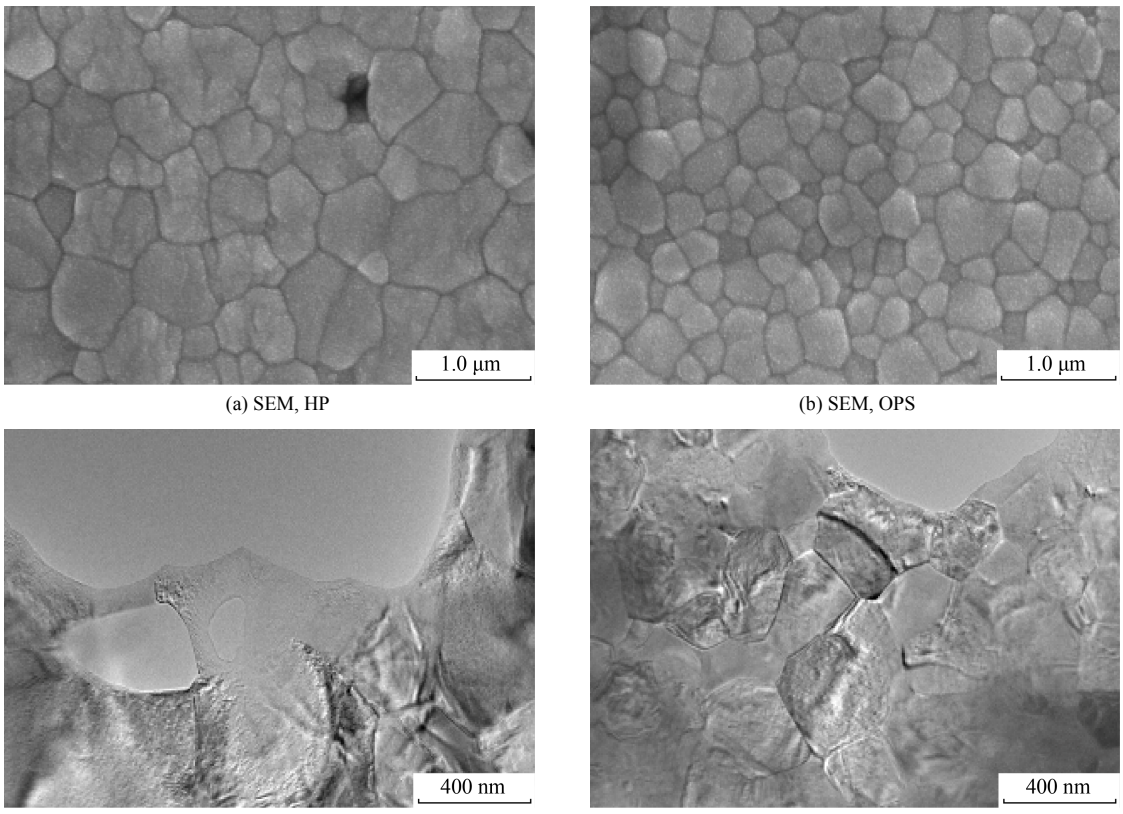
The morphology and size of the raw powders were characterized by field-emission transmission election microscopy (TEM; JEOL-2 011, Japan). Microstructure changes of the specimens before and after aging were characterized using field-emission scanning election microscopy (SEM; LEO-1 503, Oberkochen, Germany). Before observation, the polished surface of the specimens was thermally etched at 1 400 ℃ for 30 min. Composition changes of the specimen before and after aging were characterized by XRD and Raman spectroscopy. Flexural strength of the specimens was measured using a three-point bending method using testing bars with dimensions of 3 mm×4 mm×36 mm, which were polished with diamond slurry to a 1.0 μm finish. The hardness (H) and Young's modulus (E) of the specimens were analyzed by a continuous stiffness measurement using a nanoindenter (XP, MTS Systems Corp., United States) before and after hydrothermal aging. Indentation was performed on the polished surface of the specimens.

3　Results and discussion

To investigate the grain morphology and size of the two specimens, the as-sintered zirconia ceramics were first thermally etched at 1 250 ℃ for 30 min in a muffle furnace. The microstructures of the hot-pressed and oscillatory pressure sintered zirconia ceramics before aging are presented in Fig. 2. From the polished surfaces in Figs. 2(a)–2(b), it can be seen that the grains of the hot-pressed specimen were distributed over the size range from 200 to 400 nm, while those of the oscillatory pressure sintered specimen were distributed over the narrow range from 230 to 320 nm. In addition, pores and particle shedding occurred on the polished surface of the hot-pressed specimen, which is harmful to the mechanical properties of zirconia ceramics. The statistical analysis indicated that the average grain sizes of the hot-pressed and oscillatory pressure sintered specimens corresponded to 307 and 283 nm, respectively. In addition, the grain boundaries of the oscillatory pressure sintered specimen were cleaner and straighter than those of the hot-pressed specimen. The TEM images in Figs. 2(c)–2(d) further confirmed the grain refinement in the oscillatory pressure sintered specimen, which improved the resistance of the zirconia to hydrothermal aging. As a result of the microstructure evolution, the mechanical properties of the oscillatory pressure sintered specimen were significantly improved, which has been reported in the previous literature reports[26,29]. In particular, the flexural strength of zirconia increased from 1 224 to 1 556 MPa when the oscillatory pressure was applied at high temperatures.

Fig. 3 shows the Raman spectra and XRD patterns of the hot-pressed and oscillatory pressure sintered specimens before and after hydrothermal aging at 134 ℃ and 0.2 MPa. The Raman spectra were collected from a very thin layer on the surface, so even a very small amount of monoclinic phase can be detected. Moreover, X-rays could penetrate further into the bulk of ceramics to analyze the propagation of the phase transformation. A combination of XRD and Raman analyses was performed to investigate the initiation and propagation of the phase transformation.

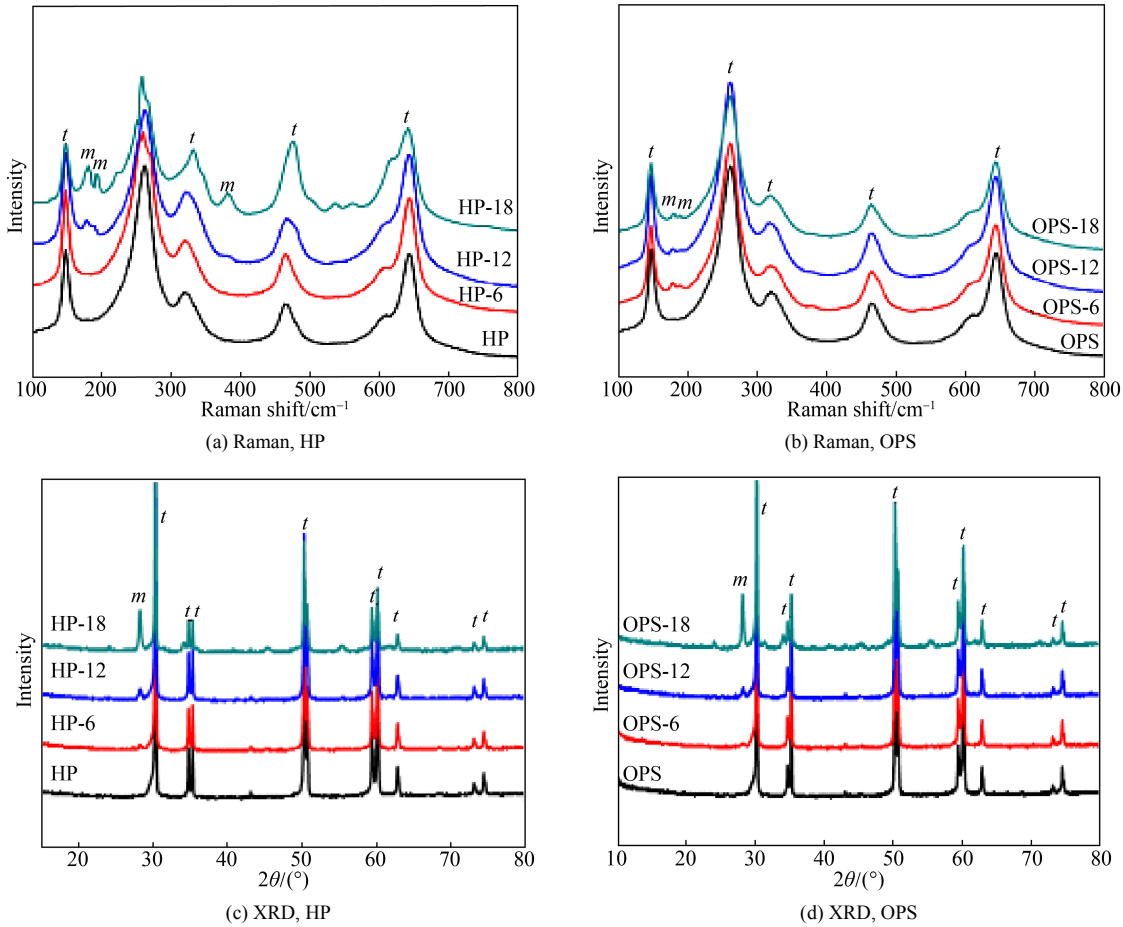
The Raman spectra in Figs. 3(a) and 3(b) present the initiation and propagation of the $t \rightarrow m$ transformation on the ceramic surface with increasing aging time. With the extension of the aging time, strong peaks at 178, 189 and 373 cm^{-1} were observed that correspond to monoclinic zirconia in both the HP-18 and OPS-18 specimens. However, compared



(a) SEM, HP (b) SEM, OPS

(c) TEM, HP (d) TEM, OPS

Fig. 2 Microstructures of the as-sintered zirconia ceramics



(a) Raman, HP (b) Raman, OPS

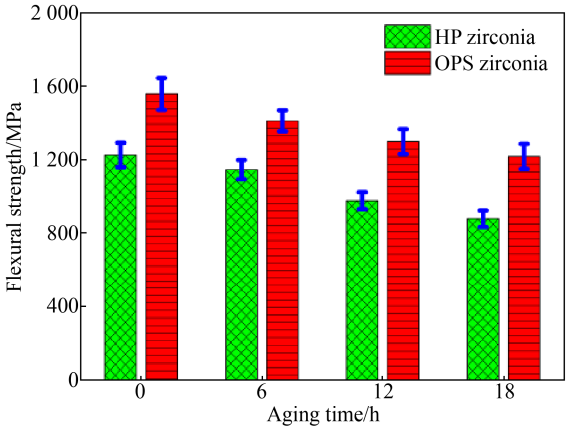
(c) XRD, HP (d) XRD, OPS

Fig. 3 Phase compositions of the zirconia ceramics before and after aging

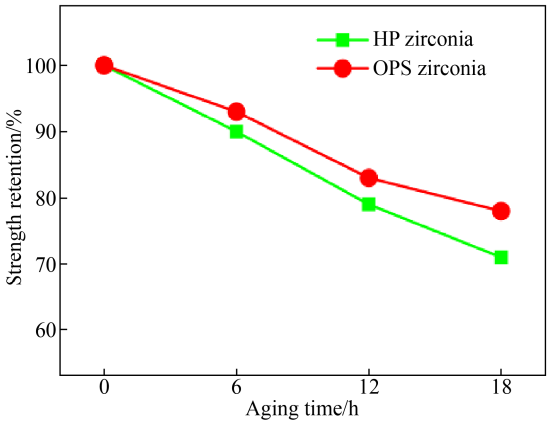
with that of the hot-pressed specimen in Fig. 3(a), the oscillatory pressure sintered specimen in Fig. 3(b) went through a lower rate of phase transformation, indicating a high resistance to hydrothermal aging. As shown from the XRD patterns in Figs. 3(c)–3(d), an obvious peak appeared at 2θ=27.8°, which confirmed the transformation from the tetragonal phase to the monoclinic phase. Because the fractions of monoclinic zirconia were low in both specimens, only one peak belonging to the monoclinic phase was detected in the XRD patterns.

As described above, hydrothermal aging leads to phase transformation and causes microcracks and high roughness on the material surface. From the failure theory by Griffith[32], the mechanical performances of ceramics are sensitive to surface defects. The flexural strength of the hot-pressed and oscillatory pressure sintered ceramics as a function of aging time is illustrated in Fig. 4. With increasing aging time, the flexural strength of the hot-pressed specimen declined from 1 224 to 878 MPa. Although the oscillatory pressure sintered specimen was nearly fully dense and had uniform and fine grains, it underwent an obvious degradation in flexural strength from 1 556 MPa for the unaged specimen to 1 216 MPa for the OPS-18 specimen, as described in Fig. 4(a). In spite of this decrease, the decrease in the strength of the oscillatory pressure sintered specimen was slower than that of the hot-pressed specimen at extended aging times, as described in Fig. 4(b). Therefore, the flexural strength of the oscillatory pressure sintered specimen was always higher than that of the hot-pressed specimen, indicating that the OPS technique is applicable for the preparation of high-performance zirconia ceramics.

The tetragonal grain size, sintering temperature, and yttria stabilizer are considered to be the pertinent factors for the aging behavior of zirconia ceramics. The decline in the flexural strength in both the hot-pressed and oscillatory pressure sintered specimens was ascribed to the degree of the phase transformation on the material surface that induced microcracks and grain coarsening as the hydrothermal aging occurred. Fig. 5 presents the microstructure variation of the oscillatory pressure sintered specimen with increasing aging time. The phase transformation from $t \rightarrow m$ in the zirconia was accompanied by a volume expansion of approximately 7% (volume fraction); thus, the oscillatory pressure sintered zirconia experienced grain coarsening in average grain size with increasing aging time, as shown in Fig. 5. From Hall-Petch relationship[33], the fracture strength of ceramics is inversely proportional to the average grain size. Specifically, as indicated by the red circles in Fig. 5, an increasing amount of coarse grains formed with increasing aging time, which is harmful to the mechanical properties of zirconia ceramics.



(a) Flexural strength

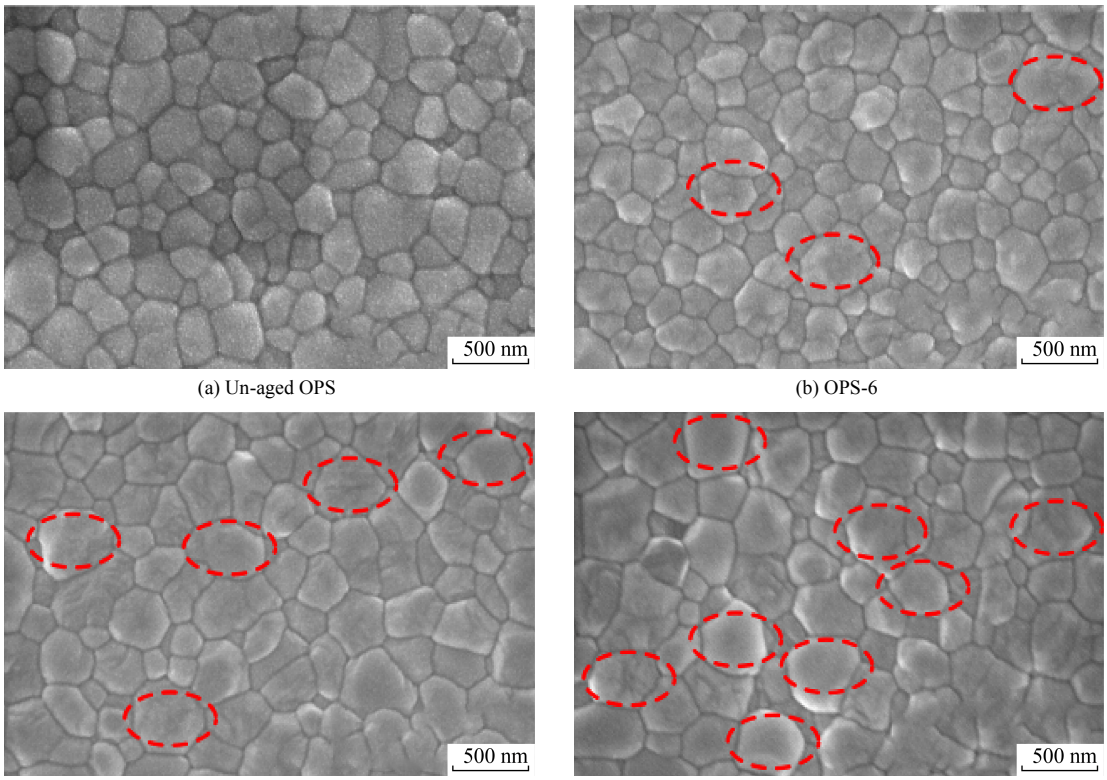


(b) Flexural strength retention

Fig. 4　Variation of mechanical properties of the zirconia ceramics with the extension of hydrothermal aging time

As discussed in Fig. 2, the average size of the hot-pressed specimen was higher than that of the oscillatory pressure sintered specimen; thus, a higher amount of phase transformation occurred in the hot-pressed specimen and led to a rapid decrease in the flexural strength of the hot-pressed specimen. Despite the decline in flexural strength for the oscillatory pressure sintered specimen, it was always higher than that of the hot-pressed specimen. Therefore, the oscillatory pressure sintered zirconia specimen can be used in extreme conditions.

Typical load-displacement curves of the hot-pressed and oscillatory pressure sintered ceramics by nanoindentation are shown in Fig. 6. The changes in the load-displacement curves is an indication of the specific surface condition of each specimen, so the load-displacement curve is considered as a practical method to analyze the influence of hydrothermal aging on zirconia ceramics. For the same penetration depth, the indentation load needed for the oscillatory pressure sintered zirconia is much higher than that of the hot-pressed zirconia, which is ascribed to the high relative density and fine grain size of the oscillatory pressure sintered zirconia. With extended aging times,



(a) Un-aged OPS (b) OPS-6

(c) OPS-12 (d) OPS-18

Fig. 5 Surface microstructures of oscillatory pressure sintered zirconia before and after hydrothermal aging

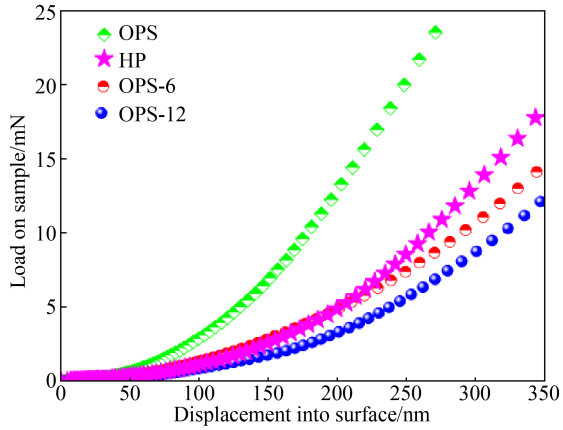
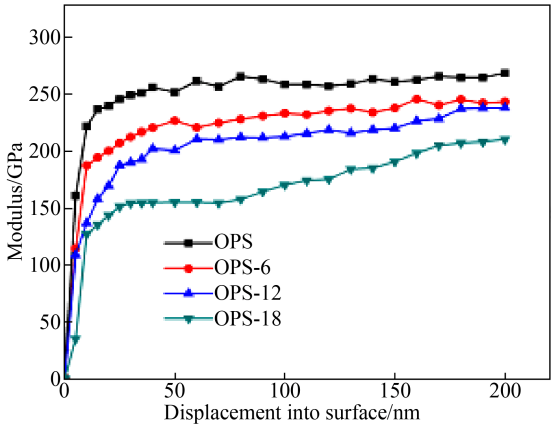
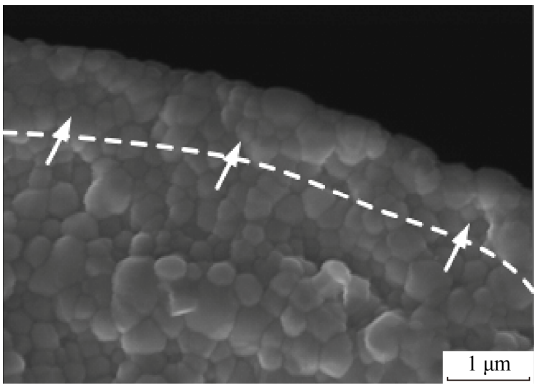


Fig. 6 Typical load-displacement curves of the zirconia ceramics by nanoindentation



(a) Young's modulus



(b) SEM of the surface

Fig. 7 Young's modulus as a function of indentation depth for OPS zirconia ceramics

the phase transformation led to a decrease in the indentation load for a given penetration depth. It is speculated that hydrothermal aging decreased the bonding strength between zirconia grains and even created microcracks in the grain boundaries. Therefore, the variation of the indentation load at the same surface penetration depth clearly indicated the corrosion degree of zirconia induced by hydrothermal aging.

Hydrothermal aging is an initiation-propagation process from the ceramic surface to the ceramic bulk and from the grain boundary to the grain center. To investigate the variation of corrosion depths with extended aging times, the modulus variation of the oscillatory pressure sintered specimens from the surface to the penetration depth of approximately 200 nm was investigated. In Fig. 7(a), the moduli increase rapidly

and then maintain a constant value, indicating that only a thin layer was corroded during hydrothermal aging. From the SEM image in Fig. 7(b), a coarsening layer can be observed on the material surface, which corresponds to the monoclinic phase induced by the phase transformation. Moreover, from Fig. 7(a), the modulus at the same displacement declined with increasing aging time, which can be explained by the variation in the phase composition, grain size and micro-crack-induced hydrothermal aging. With the extension of aging time, the formation of the monoclinic phase, the volume expansion of zirconia grains and the formation of microcracks were responsible for the decrease in the modulus.

4 Conclusions

A novel OPS technique was developed for the preparation of zirconia ceramics with a high density and uniform and fine grains. To expand the application of zirconia ceramics in the biomedical field, the aging behaviors of oscillatory pressure sintered ceramics were studied in an autoclave at 134 ℃ and 0.2 MPa.

With the extension of the aging time, strong peaks at 178, 189 and 373 cm^{-1} occurred in the Raman spectra, which corresponded to the monoclinic zirconia derived from the phase transformation. However, the peak intensities for the oscillatory pressure sintered specimen were lower than those for the hot-pressed specimen. Due to the microcracks and high roughness induced by the phase transformation on the material surface, the flexural strength of the oscillatory pressure sintered specimen declined from 1 556 MPa for the unaged specimen to 1 216 MPa for the specimen aged for 18 h. Even so, the flexural strength of oscillatory pressure sintered zirconia was always greater than that of the hot-pressed specimen. From the load-displacement curves, the indentation load needed for the oscillatory pressure sintered specimen was much higher than that of the hot-pressed specimen for a given penetration depth, which was ascribed to the high density and fine grains in the oscillatory pressure sintered zirconia. As a result, the zirconia ceramics consolidated by the OPS technique can operate in more extreme conditions than those consolidated by the HP technique.

Acknowledgements

This work was financially supported by the National Natural Science Foundation of China (Grant No. 51502120, 51802176 and 51872174), the Project of Shandong Province Higher Educational Science and Technology Program (Grant No. J17KA020) and SDUT & Zibo City Integration Development Project (2018ZBXC439).

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