

MECHANICAL PROPERTIES OF ZrO₂-TOUGHENED Al₂O₃ CERAMICS: EFFECT OF ZrO₂ CONTENT

MEHANSKE LASTNOSTI KERAMIKE NA OSNOVI Al₂O₃ OJAČANE S ZrO₂: VPLIV VSEBNOSTI ZrO₂

Zhihua Hu¹, Tingting Liao^{1,2*}, Shunying Chang¹, Yongfeng Chen¹, Siqi Song¹, Chenyang Zhu¹, Haoxiang Xu¹, Zhenchuan Wei¹, Peng Su¹, Jiehui Liu¹, Xi Zhang²

¹School of Materials and Environmental Engineering, Chengdu Technological University, Chengdu, Sichuan 611730, China

²School of Materials Science and Engineering, Southwest Jiaotong University, Chengdu, Sichuan 610031, China

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Alumina ceramics are important high-temperature materials with exceptional properties; however, their applications are limited by their low flexural strength and brittle nature. Adding a secondary phase effectively improves the toughness of alumina without compromising its hardness. In this study, as a cost-effective solution for the production of high-performance alumina composites, zirconia-toughened alumina (ZTA) composites with different ZrO₂ contents (3–20 %) were prepared using the vacuum hot-pressing sintering of micron-scale ZrO₂ and Al₂O₃ powders. Furthermore, the influence of the ZrO₂ content on the mechanical properties and fracture morphology of the composites was investigated. The density, hardness, fracture toughness and flexural strength of the zirconia/alumina composite ceramic increased with an increase in the ZrO₂ content from 3 % to 15 %. The ceramic exhibited the best comprehensive mechanical properties – a density of 98.15 %, a hardness of 887.3 HV and a bending strength of 326.28 MPa – at a ZrO₂ content of 15 %. This was attributed to the uniform distribution of the secondary phase at the fracture surface and toughening mechanisms of phase transformation and crack deflection. At ZrO₂ contents of ≥ 18 %, the agglomeration of the ZrO₂ particles caused stress concentration and other phenomena, which weakened the toughening mechanism. This study provided a feasible approach for the industrial-scale production and application of high-performance ZTA multiphase ceramics.

Keywords: vacuum hot-press sintering, zirconia-toughened alumina multiphase ceramics, toughening mechanism, mechanical properties

Keramika na osnovi aluminijevega oksida (Al₂O₃) je pomemben visokotemperaturni material z izjemnimi lastnostmi; vendar je njegova uporaba omejena zaradi nizke upogibne trdnosti in krhkosti. Dodajanje sekundarne faze lahko učinkovito izboljša žilavost aluminijevega oksida, ne da bi se pri tem ogrozilo njegovo trdoto. V tem članku avtorji opisujejo študijo stroškovno učinkovite rešitve za proizvodnjo visokozmogljivih kompozitov iz aluminijevega oksida z dodatkom cirkonijevega oksida (ZTA; angl.: zirconia-toughened alumina). Avtorji so Al₂O₃ dodajali različno vsebnost ZrO₂ (od 3 % do 20 %) in kompozite zgoščevali s postopkom vakuumskega vročega stiskanja in istočasnega sintranja mikronskih prahov ZrO₂ in Al₂O₃. Po zgoščevanju so avtorji ugotavljali vpliv vsebnosti ZrO₂ na mehanske lastnosti in morfolologijo loma kompozitov. Gostota, trdota, lomna žilavost in upogibna trdnost kompozitne keramike iz Al₂O₃ in ZrO₂ so se povečevale s povečevanjem vsebnosti ZrO₂ s 3 % na 15 %. Kompozitna keramika je pokazala najboljše fizikalne in mehanske lastnosti (gostoto: 98,15 %, trdoto: 887,3 HV in upogibno trdnost: 326,28 MPa) pri 15 %-ni vsebnosti ZrO₂. To so avtorji pripisali enakomerni porazdelitvi sekundarne faze na površini loma ter mehanizmom utrjevanja faze transformacije in nastalin oviram za napredovanje rapok. Pri vsebnosti ZrO₂ nad 18 % je aglomeracija delcev ZrO₂ že povzročila koncentracije napetosti in druge pojave, ki so oslabil mehanizme utrjevanja. S to študijo so avtorji omogočili razvoj novega materiala za industrijsko proizvodnjo in uporabo visokozmogljive večfazne ZTA keramike.

Ključne besede: sintranje z vakuumskim vročim stiskanjem, večfazna keramika na osnovi aluminijevega oksida, utrjevanje s cirkonijem oksidom, mehanizem utrjevanja, mehanske lastnosti

1 INTRODUCTION

Alumina (Al₂O₃) ceramics are important in the field of materials science because of their high hardness, high-temperature resistance, corrosion resistance, wear resistance and low cost.^{1–6} However, their low flexural strength and fracture toughness are the factors limiting their broad applications.^{7–11} Currently, research on toughening alumina has primarily focused on three approaches: introducing high-strength ceramic whiskers or

fibres to enhance toughness;^{12–14} incorporating nano- or micro-scale particles to improve toughness via pinning effects and crack deflection^{15,16} and achieving synergistic toughening by combining multiple mechanisms, such as phase transformation, whisker reinforcement and particle toughening.¹⁷ In this study, zirconia (ZrO₂) particles are incorporated into alumina.^{18,19} The transformation of the tetragonal phase (t-ZrO₂) to the monoclinic phase (m-ZrO₂) induces volume expansion and shear strain, which effectively absorbs the energy released during crack propagation, thereby enhancing the material's toughness. Zirconia (ZrO₂) is an important ceramic with exceptional properties, including high toughness, high wear resistance and good thermal stability.^{20–22} To over-

*Corresponding author's e-mail:
ltting1@cdu.edu.cn (Tingting Liao)



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come the limitations of alumina and expand its application scope, toughened alumina composite ceramics have been developed by adding some amounts of zirconia, SiC, Si₃N₄ and TiC powders/fibres. Through the synergistic effect of different toughening mechanisms, including stress-induced phase transformation, microcracks and surface strengthening, the fracture toughness of the composites is improved while retaining the desirable mechanical properties of alumina. This improves the service life and reliability of alumina ceramic materials.^{23–26} Cui et al.²⁷ examined the effect of the nano-ZrO₂ content on the microstructure and mechanical properties of nano-ZrO₂-toughened Al₂O₃ composite ceramics. However, the high cost of nano-scale raw materials, combined with the lack of standardisation in process parameters such as the sintering temperature and second-phase ZrO₂ content, limits the cost-effectiveness of equipment and renders large-scale batch production impractical. In this context, this study explored the use of micron-scale ZrO₂ particles, simple equipment (vacuum hot-press sintering furnace) and optimised processes (ball milling duration and sintering pressure) to reduce production costs while maintaining performance, thereby offering a practical solution for the industrial production and application of high-performance zirconia-toughened alumina (ZTA) multiphase ceramics.

To this end, ZTA composite ceramics with different mass fractions of ZrO₂ (3–20 %) were prepared to investigate the effect of varying mass fractions on the mechanical properties, such as the density, hardness, and flexural strength, of the ceramics. Additionally, the toughening mechanism of their fracture morphology was analysed.

2 EXPERIMENTAL DETAILS

2.1 Materials and methods

Zirconia and alumina powders with a particle size of 75 µm and purity of 99.99 % were used as the raw materials. The mass fractions of different samples are listed in **Table 1**. The powders (according to the proportions in **Table 1**) were mixed in a planetary ball mill (XQM-4-KL, Dandong Tongda) for 5 h and then placed in a dryer (101-0A, Beijing Kewei Yongxing Instrument) at 80 °C for 3 h. The mixed powders were sintered via vacuum hot-press sintering (ZR50B-8T, China) at 1300 °C and 20 MPa to obtain ZTA composite ceramic materials.

Table 1: Mass fractions of the zirconia-toughened alumina composite samples

ZrO ₂		Al ₂ O ₃		Total mass, g
Mass fraction, %	Mass, g	Mass fraction, %	Mass, g	
3	2.4	97	77.6	80.0
9	7.2	93	72.8	80.0
15	12.0	85	68.0	80.0
20	16.0	80	64.0	80.0

2.2 Performance characterizations and testings

The structure of the ZTA composite ceramic material was characterised by X-ray diffraction (XRD; TD-3500, Dandong Tongda). The density of the composite was measured based on Archimedes' principle using a direct-reading electronic densitometer (DH-300, Shanghai Taiming), and relative density was calculated as the ratio of the measured bulk density to the theoretical density predicted by the rule of mixtures. The Vickers hardness was evaluated using a digital microhardness tester (HXD-1000, LaiZhou Huayin), and the flexural strength and fracture toughness were measured using a universal testing machine (WDW-2.0, Shen Zhen Wance). A JSM-6510LV scanning electron microscope (JEOL) was used to observe the micromorphology of the fracture surface. In this study, at least five replicate specimens were fabricated for each composition, and mechanical and physical properties were reported as mean ± standard deviation.

3 RESULTS AND DISCUSSION

3.1 Structure

Figure 1 shows the XRD patterns of the ZTA composites with different ZrO₂ contents. The peaks corresponding to α-Al₂O₃ (2θ = 57°) and t-ZrO₂ (2θ = 35° and 50°) are observed in the XRD patterns of the composites; however, no peak attributed to the monoclinic phase of ZrO₂ is detected. The intensity of the ZrO₂ peak gradually increases with an increasing ZrO₂ content. Furthermore, all composites exhibit sharp and well-defined diffraction peaks with narrow half-widths and clear profiles, indicating high crystallinity. The test results confirm that the prepared samples exhibit typical crystal structure characteristics, with well-developed alumina grains.

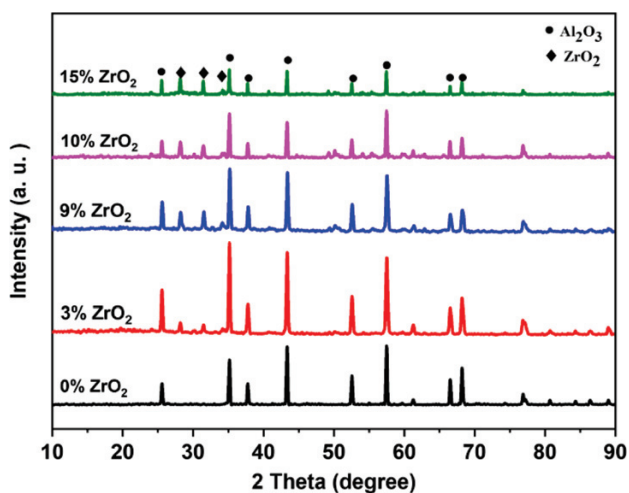


Figure 1: X-ray diffraction patterns of the zirconia-toughened alumina composites with different ZrO₂ contents

3.2 Density

Table 2 lists the theoretical, actual, and relative densities of the ZrO₂-toughened Al₂O₃ multiphase ceramics with different ZrO₂ contents, and **Figure 2** shows a density histogram. The density of the multiphase ceramics increases with an increasing ZrO₂ content, and the ratio of the actual to the theoretical density first increases and then decreases, reaching a maximum value of 98.15 % at 15 % ZrO₂. At low ZrO₂ contents, the density continues to increase because of an increase in the sintering driving force of the material caused by the ZrO₂ particles. With an increase in the ZrO₂ content, the contact area between the alumina matrix and secondary phase increases. Owing to the different valence electrons in zirconium and aluminium ions, vacancies are easily formed in the matrix and the lattice constant changes, which is favourable for increasing the density of the ZTA multiphase ceramics. However, at high ZrO₂ contents, the excess ZrO₂ agglomerates, weakening the pinning effect. The phase transformation of the ZrO₂ agglomerates causes the accumulation of microcracks, resulting in the formation of crystal defects and cracking of the crystal interface, thereby reducing the density of the multiphase ceramic composites.

Table 2: Density analysis of the zirconia-toughened alumina composites

Sample	0 % ZrO ₂	3 % ZrO ₂	9 % ZrO ₂	15 % ZrO ₂	20 % ZrO ₂
Actual density (g/cm ³)	3.31	3.44	3.61	3.76	3.78
Theoretical density (g/cm ³)	3.60	3.64	3.73	3.83	3.92
Relative density (%)	91.89	94.45	96.86	98.15	96.30

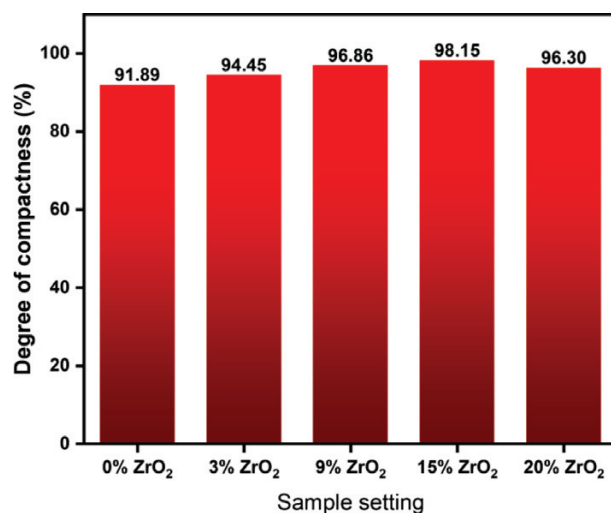


Figure 2: Bar graph of the densities of the zirconia-toughened alumina composites

3.3 Microhardness

Figure 3 shows the hardness values of the ZTA multiphase ceramics with different ZrO₂ contents. The hardness of pure alumina is the lowest (5.72 GPa), and that of the ZTA composite ceramics first increases, reaching a maximum value of 8.75 GPa at 15 % ZrO₂, and then decreases with an increasing ZrO₂ content. The dispersion of the ZrO₂ particles in the alumina matrix hinders the movement of dislocations and strengthens the second phase, thereby increasing the composite hardness. With a further increase in the ZrO₂ content (20 %), the excess ZrO₂ particles cause lattice defects in the matrix, which act as stress concentrators, thereby reducing the hardness of the multiphase ceramics.

3.4 Fracture toughness

Figure 4 shows the fracture toughness of the composite ceramics with different ZrO₂ contents. The fracture toughness of the single-phase Al₂O₃ ceramics is 4.3 MPa·m^{1/2}. With the ZrO₂ addition, the fracture toughness of the Al₂O₃/ZrO₂ composite first increases and then decreases; however, it is always higher than that of pure Al₂O₃ ceramics. This strengthening is due to the synergistic effects of the stress-induced and microcrack toughening mechanisms. When the ZrO₂ content is 9 %, the fracture toughness of the composite ceramics is the highest (4.91 MPa·m^{1/2}). This is because the tetragonal zirconia particles undergo martensitic transformation under the action of the stress field at the crack tip.²⁸ During phase transformation, volume expansion offsets a part of the stress, thereby reducing the fracture energy.²⁹ On increasing the ZrO₂ content, the lattice defects increase significantly and the toughening effect gradually weakens.

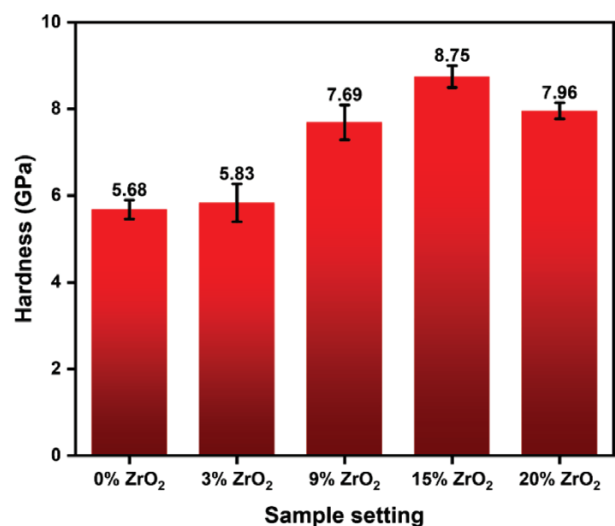


Figure 3: Hardness of the zirconia-toughened alumina composite ceramics with different ZrO₂ contents

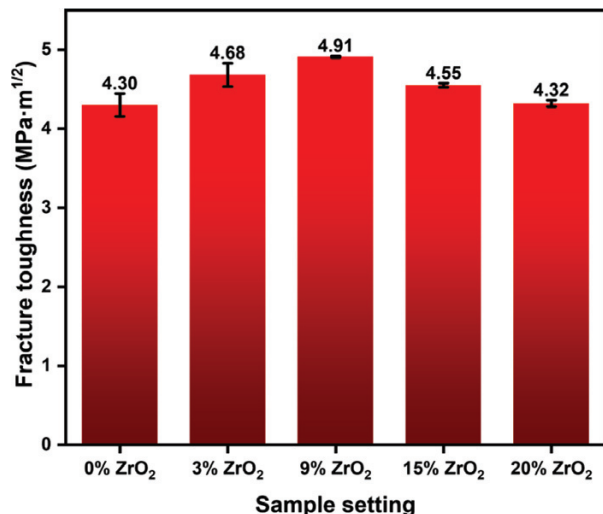


Figure 4: Fracture toughness of the zirconia-toughened alumina composite ceramics with different zirconia contents

3.5 Bending strength

Figure 5 shows the bending strengths of the ZTA multiphase ceramics with different ZrO₂ contents. The bending strength of the ZTA composite ceramics first increases and then decreases with an increasing ZrO₂ content. The maximum bending strength of 326.28 MPa is achieved when the ZrO₂ content is 15%. At ZrO₂ contents of < 15%, alumina has a stabilising effect on the metastable tetragonal zirconia crystals. The application of an external stress promotes the transformation of the tetragonal zirconia crystals into monoclinic zirconia. This phase transformation process is accompanied by a volume change of 5–8%. This volume change produced by the phase transformation inevitably causes microcracks inside the ceramic sample, thereby dispersing and reducing the energy of the main crack. This reduces the

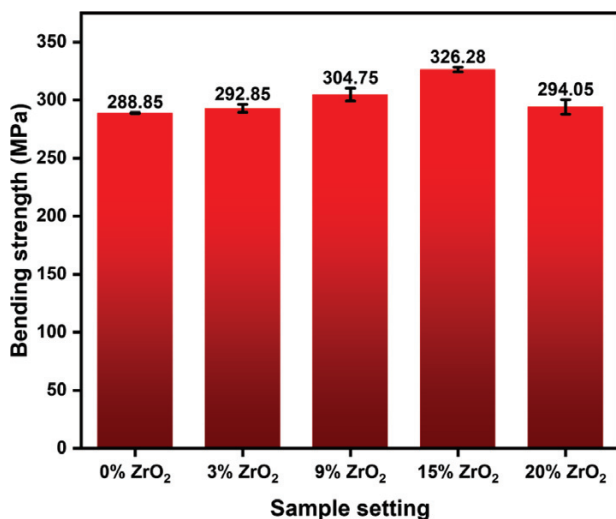


Figure 5: Bending strength of the zirconia-toughened alumina composite ceramics with different zirconia contents

stress concentration and improves the bending strength of the ceramic material via microcrack toughening. When the ZrO₂ content is 20%, the bending strength decreases because of the increase in the secondary phase content, attributed to agglomeration at high ZrO₂ contents. In the presence of an external stress, more microcracks are formed around the agglomerates; the accumulation of these microcracks causes matrix cracking and decreases the bending strength.

3.6 Fracture morphology

Figure 6 shows the fracture morphologies of the ZTA multiphase ceramics with different zirconia contents. The fracture observed in the ZTA multiphase ceramic with 15% ZrO₂ content is relatively flat and shows typical transgranular fracture characteristics. Several fine dimples are observed on the fracture surface, indicating that the material undergoes significant plastic deformation during the fracture process. For pure alumina, the fracture surface exhibits predominantly intergranular fracture characteristics and demonstrates significantly higher brittleness. At ZrO₂ contents of 3% and 9%, the zirconia particles are relatively sparse, and the cracks propagate mainly along the intergranular or transgranular alumina grains. In this case, the dimples are evenly distributed and plastic deformation is more significant. As the ZrO₂ content is increased to 20%, particle agglomeration becomes prominent, and the crack propagation path becomes more tortuous. This indicates that ZrO₂ particle agglomeration significantly hinders the crack propagation, causing stress concentration and other phenomena, thereby weakening the toughening mechanism. When the ZrO₂ content is 15%, the microcracks in the material are evenly distributed, effectively hindering crack propagation and toughening the material. Thus, 15% ZrO₂ content is the optimum amount for enhancing the comprehensive mechanical properties of a ZTA composite material.

In this study, the resulting balance of high hardness (8.75 GPa) and markedly improved fracture toughness ($K_{IC} \approx 5 \text{ MPa}\cdot\text{m}^{1/2}$) was achieved without compromising process simplicity or cost-effectiveness – underscoring the material's strong potential for real-world engineering applications. This synergistic property profile makes it especially promising for cost-sensitive, wear- and corrosion-prone structural components – including valve seats, slurry pump liners, and cutting tool substrates – where reliability, manufacturability, and lifecycle economics are paramount. Looking ahead, targeted enhancements – such as a judicious addition of trace sintering aids or controlled nanostructuring of the ZrO₂ phase – could further elevate mechanical performance while preserving the core advantages of scalability and affordability, thereby bridging the gap toward more demanding service environments.

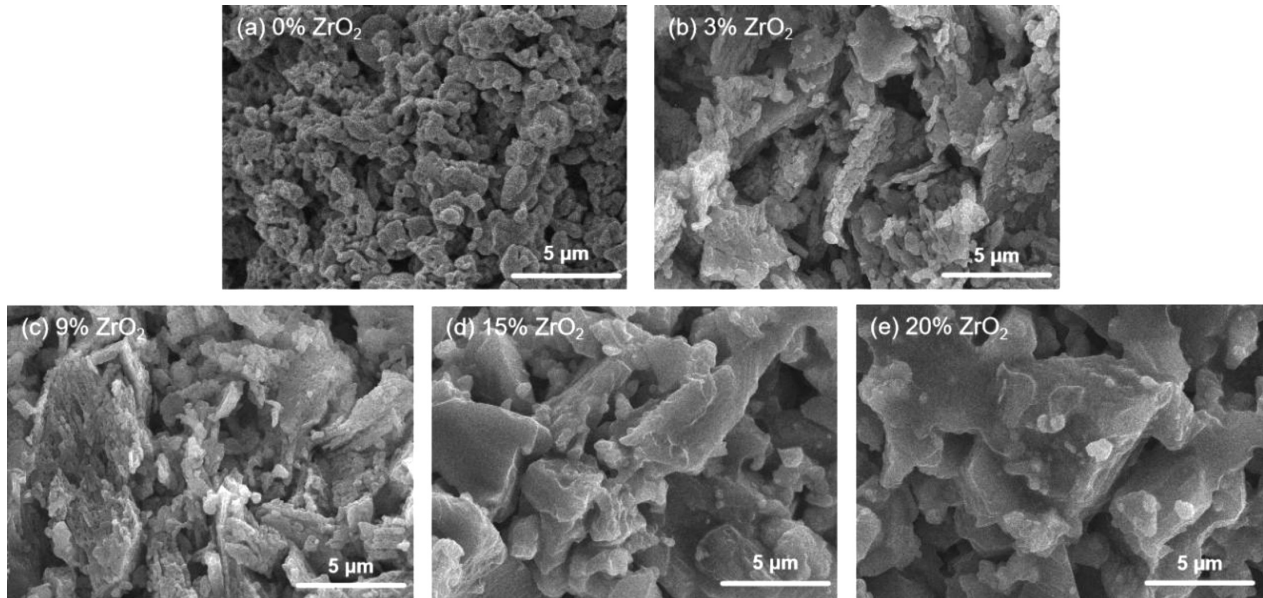


Figure 6: Scanning electron microscopy images of the fracture morphologies of the zirconia-toughened alumina composite ceramics with different ZrO₂ contents

4 CONCLUSIONS

The effects of different mass fractions of micron-scale ZrO₂ on the mechanical properties of the ZTA multiphase ceramics were investigated. The following conclusions are drawn from this study.

The zirconia/alumina multiphase ceramics with good crystallinity were successfully prepared using the vacuum hot-press sintering method.

The densities of the ZTA multiphase ceramics first increased and then decreased with an increase in the ZrO₂ content. The highest ceramic density of 98.15 % was observed at a ZrO₂ content of 15 %.

The Vickers hardness and flexural strength of the ZTA composites were significantly higher than those of pure Al₂O₃ ceramics, reaching maximum values of 8.75 GPa and 326.28 MPa, respectively.

The fracture toughness of the composite ceramics reached a maximum of 4.91 MPa·m^{1/2} when the mass fraction of ZrO₂ was 9 %. This is mainly attributed to the combined influence of the t→m phase transformation and microcrack toughening mechanisms. However, the toughening effect gradually weakened at higher ZrO₂ contents owing to the significant increase in the lattice defects.

The main toughening mechanisms of the ZTA composite ceramics are the phase-transformation-induced volume change and microcrack toughening.

When the mass fraction of the ZrO₂ second-phase added was 15 %, the comprehensive mechanical properties of the ZTA multiphase materials prepared from micron-scale raw materials reached their optimal values, and the phase transformation toughening and microcrack toughening effects were also maximised. This research result provides a solid theoretical foundation and valu-

able technical reserve for the industrial application of ZTA multiphase ceramic materials.

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