第 37 卷 第 4 期
2022 年 4 月

无 机 材 料 学 报
Journal of Inorganic Materials

Vol. 37 No. 4
Apr., 2022

Article ID: 1000-324X(2022)04-0467-06 **DOI:** 10.15541/jim20210412

Effects of Residual Stresses on Strength and Crack Resistance in ZrO_2 Ceramics with Alumina Coating

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Abstract: By covering a thin Al_2O_3 coating on ZrO_2 substrate, compressive stress caused by the mismatch of thermal expansion coefficients between the coating and substrate was introduced in the surface layer of Al_2O_3-ZrO_2 pre-stressed ceramics (marked as A_CZ_S pre-stressed ceramics). Vickers indentation test was carried out to check the crack resistance in the surface layer and substrate influenced by the residual stresses. The enhancement of the crack resistance in surface layer results in a high flexural strength and excellent damage tolerance. Both theoretical analysis and experimental results show that the compressive stress and crack resistance in the surface layer increase with the increasing ratio of the cross-sectional area of ZrO_2 substrate to Al_2O_3 coating. Due to the residual compressive stress existing in Al_2O_3 coating, a high flexural strength of (1207±20) MPa was measured for ZrO_2 specimens coated with 40 μm Al_2O_3. The flexural strength is 32% higher than that of monolithic ZrO_2, and about triple of the value of Al_2O_3. Meanwhile, compared to ZrO_2, the A_CZ_S pre-stressed ceramics exhibit superior thermal shock resistance.

Key words: Al_2O_3-ZrO_2 pre-stressed ceramics; compressive stress; flexural strength; damage tolerance

Zirconia (ZrO_2) materials have been widely applied in fuel cell, refractory materials, and construction materials due to the prominent mechanical and chemical properties such as high hardness, strength, good wear-resistant, fine oxygen ionic conductivity, excellent heat-insulating and corrosion protective[1-3]. With the rapid advances of engineering applications, ceramic materials with higher strength and reliability are greatly needed. Therefore, significant improvements are essential to fabricate zirconia components with higher strength and excellent damage tolerance to improve the lifetime and safety of ceramic components. Many efforts including reinforcement strengthening (such as the incorporation of fibers, particles, and whiskers) and transformation toughening were applied to improve the strength and toughness of ZrO_2[4-6]. Among them, the way to enhance the strength and damage tolerance of brittle ceramics by introducing residual compressive stress in the surface layer was more attractive because of its remarkable strengthening effect and convenience[7-8]. It is well-known that the fracture of ceramics is mainly caused by crack propagation in the surface layer. Thus, the key to improve the strength and damage tolerance of ceramics is to enhance the resistance to crack propagation in the surface[9]. By covering a coating with lower coefficient of thermal expansion (CTE) than the substrate, compressive stress generated in the coating layer after sintering. Then the compressive stress hinders the crack extension, thereby improve the strength and damage tolerance of ceramics.

In order to illuminate the strengthening mechanism, it is important to understand the effect of residual stress on crack propagation. As it is well known that indentation deformation strongly links to the crack initiation and crack propagation. For the crack propagation behavior investigation, Vickers indentation proposed by Palmquvist in the late 1950s has been widely used because of its simplicity and efficiency[10]. By comparing the growth of crack from the indentations made in unstressed and stressed materials, the effect of residual stress on crack propagation could be studied.

In this work, hot-pressed sintering (HP) was used to fabricate ZrO_2 ceramics covered by Al_2O_3 coating with

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Received date: 2021-07-01; **Revised date:** 2021-07-30; **Published online:** 2021-07-31

Foundation item: National Natural Science Foundation of China (52032011); State Key Laboratory of New Ceramic and Fine Processing Tsinghua University (KF 202007) ; Youth Fund of China Building Materials Academy (ZD-16)

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strong interface bonding and high density. For comparison, monolithic ZrO_2 ceramics were also prepared by the HP process using the same technical parameters. The effects of different ratios of cross-sectional area of substrate to coating (marked as S_s/S_c) on residual stresses and the crack resistance were further investigated.

1 Experimental

1.1 Material synthesis

Commercial zirconia with an average particle size of D_{50}=220 nm (3% Y_2O_3 (molar percent), G3Y-020OO, Shangdong Sinocera Functional Materials Co. Ltd., China) and alumina with an average particle size of D_{50}=220 nm (SAO-020A-M, Shangdong Sinocera Functional Materials Co. Ltd., China) were used as the raw materials in this research. Alumina powders, deionized water, and dispersant (Isobam104 with a molecular weight of 55000–60000, provided by Kuraray Co., Ltd., Osaka, Japan) were added to produce alumina slurry, and their mass contents were 30%, 69.2%, and 0.8%, respectively. The mixture was obtained using a planetary mill (YXQM-1L, MITR, China) for 24 h in zirconia containers with the ball-to-powder weight ratio of 10:1 and rotational speed of 150 r/min.

To fabricate the A_CZ_S pre-stressed ceramics by hot-pressed sintering, graphite die with a diameter of 50 mm was uniformly sprayed with the above alumina slurry. And the thickness of the Al_2O_3 coating was about 40 μm. Then zirconia powder was put into the Al_2O_3-coated graphite die followed by applying a constant pressure of 10 MPa. The specimens were heated up to 1450 ℃ at a heating rate of 15 ℃/min, and then a constant pressure of 30 MPa was applied for 60 min. For comparison, a monolithic ZrO_2 specimen was prepared by a hot-pressing sintering at 1450 ℃ with the pressure of 30 MPa for 60 min. Both the ZrO_2 specimens with a dimension of 3 mm×4 mm×36 mm and the A_CZ_S pre-stressed ceramics with the size of 3.6 mm×4 mm×36 mm were machined (the original thickness of A_CZ_S pre-stressed ceramics prepared by hot-pressed sintering was 3.6 mm).

1.2 Characterization

Morphology of the specimens was observed by using a scanning electron microscope (SEM) (Merlin, Zeiss, Germany). The flexural strength was measured by the three-point bending tests with a span length of 30 mm and a crosshead speed of 0.5 mm/min (according to ISO 14704:2000). The residual strengths were investigated to evaluate the thermal shock resistance after quenching the specimens from different temperatures (200, 250, 300, 350, 400 ℃) to 20 ℃ water (according to ASTM C1525-18). The measured value of strength is the average of 5 separate measurements. In order to study the effect of residual stress on crack propagation behavior, the specimens with Al_2O_3 coatings of different thicknesses (40, 30, 20 μm) were used, because the residual stresses change with the coating thickness. The Vickers indenter (Tukon2500B, Wilson, America) was used to generate the indentation and indentation cracks under different loadings.

2 Results and discussion

The A_CZ_S pre-stressed ceramics were designed as the schematic diagram for plates in Fig. 1(a). To generate compressive stresses in the surface layer, Al_2O_3 was applied as the coating material, because it has a lower coefficient of thermal expansion (CTE) than ZrO_2. Due to CTE difference between Al_2O_3 coating and ZrO_2 substrate, residual compressive stress was generated in the Al_2O_3 coating during the cooling process of the A_CZ_S pre-stressed ceramics. The way to fabricate pre-stressed ceramics is also available for any ceramic components of various shapes, including cylinders, tubes and spheres, as illustrated in Fig. 1(a). As for structural ceramics, the interface bonding generates significant impacts on mechanical properties[11]. Fine interface bonding manner was investigated from the continuous and tight interface illustrated in Fig. 1(b). The cross-sectional SEM image of the A_CZ_S pre-stressed ceramics indicates that the thickness of Al_2O_3 coating is near 40 μm (while the thickness for ZrO_2 substrate is 3.6 mm). Therefore, by employing HP sintering, A_CZ_S pre-stressed ceramics with a good interface bond between Al_2O_3 coating and ZrO_2 substrate can be successfully fabricated.

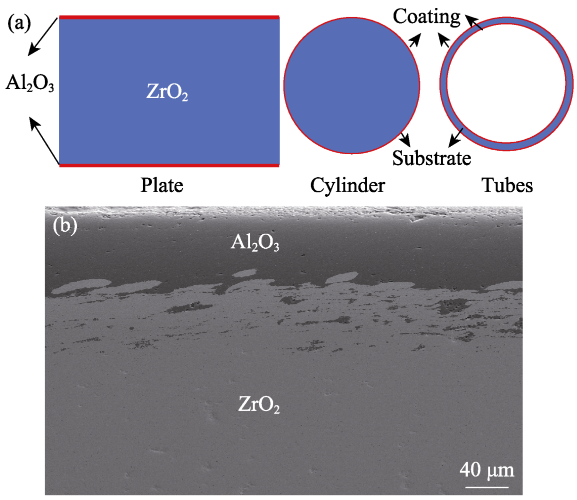

Fig. 1 (a) Schematic diagram of the A_CZ_S pre-stressed ceramics; (b) SEM image of interface between ZrO_2 substrate and Al_2O_3 coating

To investigate the effect of residual stress on crack propagation behavior, Vickers indentation tests were carried out on the ZrO_2 ceramics and the A_CZ_S pre-stressed ceramics with different ratios of sectional area (S_s/S_c is the ratio of substrate to coating in the schematic diagram of plates shown in Fig.1(a)). By comparing the length and expanded direction of crack in Fig. 2(b) and Fig. 3, the indentation diagonal half-length a and the crack length c (illustrated in Fig. 2(a)) can be easily obtained through the observation of indentation morphology. On this basis, the indentation fracture resistance ($K_{\mathrm{I,\ IFR}}$) used to describe the resistance to indentation cracking[12] can be calculated by Eq. (1)[13]. Since the residual stresses are distributed anisotropically, the test conditions for determine the crack resistance does not satisfy all the application requirements of Eq. (1) mentioned in ISO 14627. It is effective to evaluate the crack resistance at different directions in the surface layer and substrate by comparing the measured values of $K_{\mathrm{I,\ IFR}}$.

$$K_{\mathrm{I,IFR}} = 0.000978\left(\frac{E}{H_{\mathrm{V}}}\right)^{0.4}\left(\frac{P}{c^{1.5}}\right) \qquad (1)$$

Where, $K_{\mathrm{I,\ IFR}}$: the indentation fracture resistance value ($\mathrm{MPa\cdot m^{1/2}}$); E: modulus of elasticity (GPa); H_{V}: Vickers hardness (GPa);

$$H_{\mathrm{V}} = 0.001854\frac{P}{(2a)^2} \qquad (2)$$

P: pressing in load (N); c: half of average of crack length (mm); a: half of average of diagonal line length of indentation (mm).

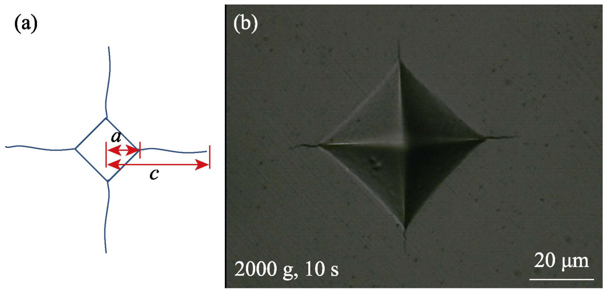

Fig. 2 (a) Typical Vickers indentation of ZrO_2 ceramics; (b) Optical photograph of the indentation morphology for ZrO_2 ceramics without residual stress

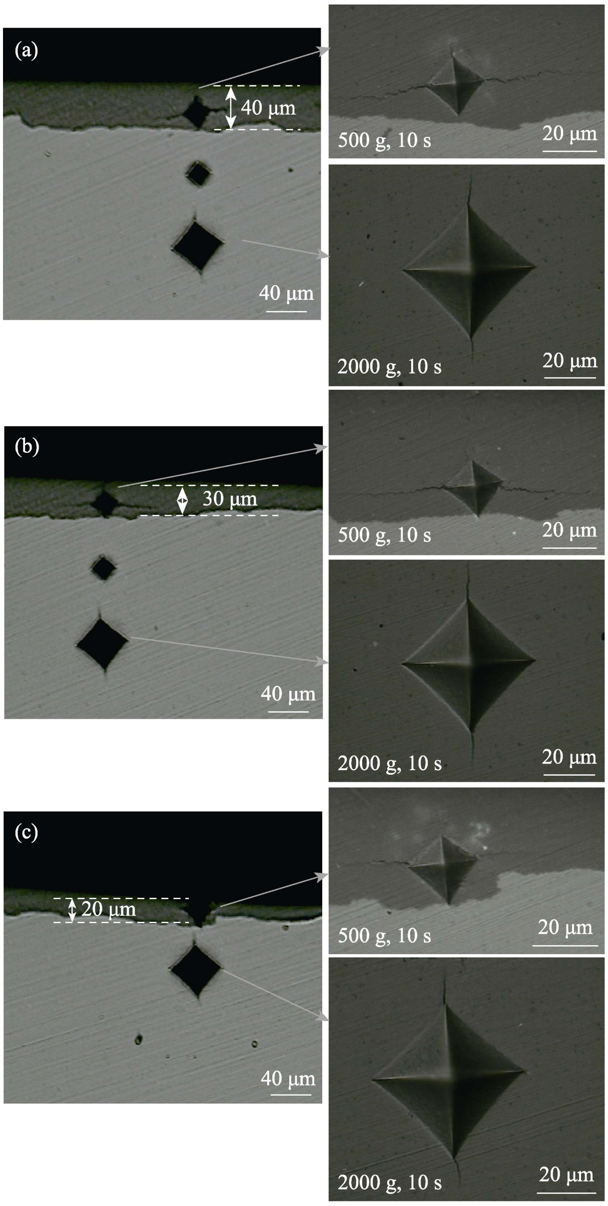

Fig. 3 Optical photographs of the indentation morphologies for A_CZ_S pre-stressed ceramics with different ratios of the cross-sectional areas, showing the effects of the compressive stresses in the coating and tensile stresses in the substrate

As for the indentation on the polished surface of ZrO_2 shown in Fig. 2(b), the cracks propagate outward along the diagonal direction. According to Eq. (1), the calculated $K_{\mathrm{I,\ IFR}}$ of ZrO_2 ceramics was 9.10 $\mathrm{MPa\cdot m^{1/2}}$ (as shown in Table 1). However, different indentation morphology was presented at the A_CZ_S pre-stressed ceramics with different thicknesses of Al_2O_3 coating in Fig. 3. As shown in Fig. 3(a-c), the crack in Al_2O_3 coating layer propagates along the direction parallel to the interface, while the crack in ZrO_2 substrate propagates along the direction perpendicular to the interface. The above phenomenon might be attributed to the compressive stress existed in the Al_2O_3 coating, while the tensile stress existed in the ZrO_2 substrate, as shown in Fig. 4. And it is well known that the compressive stresses could restrain crack extension and the tensile stress could promote crack initiation and growth[7-8]. Therefore, the cracks perpendicular to the interface were restrained in Al_2O_3 coating layer, while those were promoted in ZrO_2 substrates. Moreover, it was found that the length of crack in Al_2O_3 surface layer decreases with the increased value of S_s/S_c. The measured $K_{\mathrm{I,\ IFR}}$ of Al_2O_3 coatings and ZrO_2 substrates vary with the coating thickness, as shown in Table 1. The result suggests that the variations in the

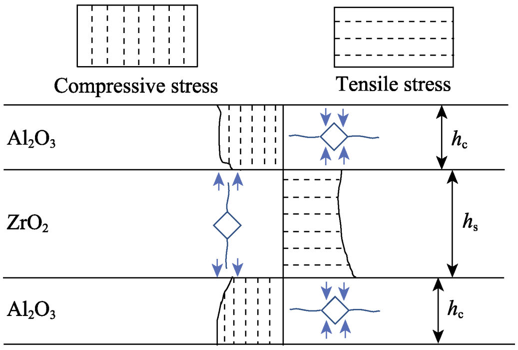

Fig. 4 Stress state and Vickers indentation of A_CZ_S pre-stressed ceramics

h_s and h_c: the thicknesses of substrate and coating

length of crack is the consequences of varied residual stress in the A_CZ_S pre-stressed ceramics with different values of S_s/S_c. Combined Eqs. (3) and (4), the residual stress in the Al_2O_3 coatings and ZrO_2 substrates could be calculated.

$$\sigma_c = \left(\frac{S_s}{S_c}\right)\cdot\left\{1-\frac{\left[\dfrac{E_sS_s}{E_cS_c}+\dfrac{\alpha_c}{\alpha_s}\right]}{\left[1+\dfrac{E_sS_s}{E_cS_c}\right]}\right\}\cdot E_s\cdot\alpha_s\cdot\Delta T_c \qquad (3)$$

In any cross-section of the sample, the tensile stresses in the substrate and the compressive stresses in the coating should keep balance,

$$\sigma_c S_c = \sigma_s S_s \qquad (4)$$

where $S_{s,}$ $E_{s,}$ α_s are the values of the cross-section areas, elastic modulus and CTE of the substrate, S_c, E_c and α_c are those of the coating, respectively. ΔT, the temperature difference, in this test: $\Delta T=T_{\text{sintering}}-T_{\text{room}}=1425$ ℃.

Depending on the calculations, the values of residual stress in the coatings (σ_c) and substrates (σ_s) were plotted in Fig. 5. As is shown, σ_c increased while σ_s decreased with the value of S_s/S_c increasing. It can be deduced that, with the value of S_s/S_c increasing, the compressive stress in Al_2O_3 coatings increased, while the tensile stress in ZrO_2 substrates presented opposite tendency. As compressive stresses could restrain crack extension and the tensile stress could promote crack initiation and growth, the crack resistance of A_CZ_S pre-stressed ceramics was enhanced as the value of S_s/S_c increasing. This is consistent with the crack propagation behavior shown in Fig. 2(b) and Fig. 3.

Fractographic features of ZrO_2 ceramics with and without Al_2O_3 coating (40 μm thick) were observed through SEM after bending tests. As shown in Fig. 6(a, c), the fracture morphology is smooth without ductile ridge marks. This means that the fracture mode of both ZrO_2 ceramics and A_CZ_S pre-stressed ceramics is brittle fracture. Grain sizes of uncoated ZrO_2 ceramic and ZrO_2 substrate of A_CZ_S pre-stressed ceramics displayed in Fig. 6(b, d) are in the range of 400 to 600 nm. Moreover, few pores can be found from the microstructure, indicating that the specimens prepared by the hot-pressing sintering method have high density.

The flexure strength of A_CZ_S pre-stressed ceramics with 40 μm thickness of Al_2O_3 coating is (1207±20) MPa, which is 32% higher than that of ZrO_2 ceramics ((821±15) MPa) fabricated by the same process and about 3-fold of the flexural strength of Al_2O_3[14]. According to the previous reports[15-17], the Al_2O_3/ZrO_2 composites fabricated by the conventional methods, such as ZTA or ATZ, or laminated Al_2O_3/ZrO_2, mostly possess a strength with the value between those of Al_2O_3 and ZrO_2. Generally, the strength of ceramic composites may be between the reinforcement material and matrix. However, the pre-stressing design endows the A_CZ_S ceramics with a higher strength than Al_2O_3 and ZrO_2. The results of the mechanical properties clearly show that the increase in strength is mainly attributed to the residual compressive stress in Al_2O_3 coating, thereby more energy was required for crack extension[18]. In addition, the robust interface between substrate and coating material also makes restrictions

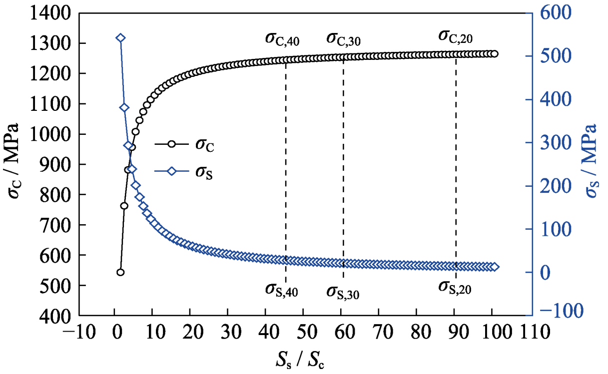

Fig. 5 Calculated residual stress in the Al_2O_3 coating and ZrO_2 substrate of A_CZ_S pre-stressed ceramics, as the function of the ratio of cross-sectional area of ZrO_2 substrate to Al_2O_3 coating

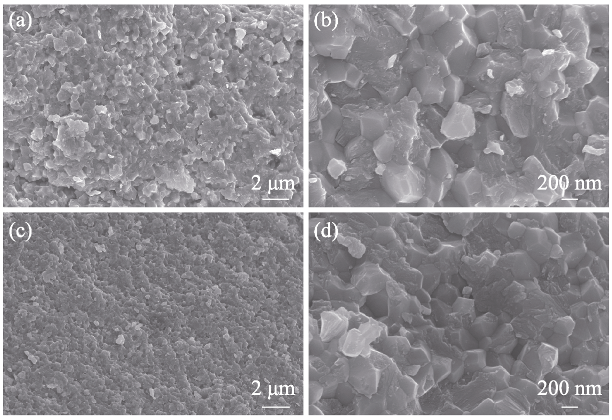

Fig. 6 SEM images of the fracture surfaces for monolithic ZrO_2 ceramics (a, b) and ZrO_2 substrate of A_CZ_S pre-stressed ceramics (c, d) fabricated by the hot-pressing sintering method

Table 1 Parameters used in formulas and the results of the experiment and simulation

		Elasticity modulus, E/GPa	p/N	a/μm	c/μm	H_V/GPa	$K_{I,\ IFR}$/(MPa·$m^{1/2}$)
ZrO_2	ZrO_2	220	19.62	26.80	35.20	12.66	9.10
A_CZ_S with 40 μm coating	Al_2O_3-40	350	4.91	11.25	47.50	17.98	1.52
	ZrO_2-40	220	19.62	26.16	36.88	13.28	8.33
A_CZ_S with 30 μm coating	Al_2O_3-30	350	4.91	11.09	44.38	18.49	1.67
	ZrO_2-30	220	19.62	26.08	36.05	13.37	8.59
A_CZ_S with 20 μm coating	Al_2O_3-20	350	4.91	10.94	25.00	19.02	3.89
	ZrO_2-20	220	19.62	26.01	35.87	13.44	8.64

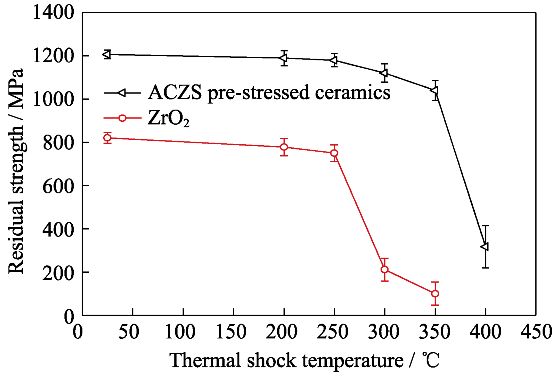

Fig. 7 Residual strength of A_CZ_S pre-stressed ceramics and ZrO_2 ceramics, after quenching at different thermal shock temperatures

against crack growth, which has a significant effect on the improvement of the strength and toughness[19].

Another important mechanical property observed for ceramics is the damage tolerance, which represents the ability to inhibit crack extension. The residual strength of two materials was tested after thermal shock with various thermal shock temperatures, as illustrated in Fig. 7. The residual strength of both ZrO_2 and A_CZ_S pre-stressed ceramics with 40 μm thickness Al_2O_3 coating were reduced with the quenching temperature increasing. Especially, the residual strength of A_CZ_S pre-stressed ceramics rapidly dropped from 1040 MPa to 317 MPa when the thermal shock temperature raised from 350 ℃ to 400 ℃, while the residual strength of ZrO_2 quenched at 350 ℃ was only 100 MPa. The result could be ascribed to that the compressive stresses existing in the coating layer can hinder the crack extension. Consequently, it is significant to introduce residual compressive stress in the surface layer of ceramic components to prevent crack propagation.

Owing to the high density and residual compressive stresses in the surface layer, A_CZ_S pre-stressed ceramics were endowed with high flexural strength and excellent thermal shock resistance. Moreover, considering the feasibility in economy and simplification in the fabrication, the pre-stressing design to improve mechanical properties has great practical value and prospect in the fields of structural ceramics and domestic ceramics. Besides, unlike the pre-stressed concrete and tempered glass, the pre-stressed ceramics can be machined to specific dimensions, which is more suitable for industrialized production.

3 Conclusions

Pre-stressing design was used to enhance the strength and damage tolerance of ZrO_2 ceramics by coating a thin Al_2O_3 layer. The magnitude and direction of the residual stress were examined by Vickers indentation tests, and the crack resistance varied with the residual stress was further verified. Experiments indicated that the crack resistance of A_CZ_S pre-stressed ceramics was enhanced with increasing ratio of the cross-sectional area of ZrO_2 substrate to Al_2O_3 coating.

On account of the residual compressive stress in the coating, the bending strength of ZrO_2 specimens coated with 40 μm thickness of Al_2O_3 was (1207±20) MPa which is much higher than those of ZrO_2 (850 MPa) and Al_2O_3 (400 MPa). After quenching from 350 ℃ to water, the residual strength of the A_CZ_S pre-stressed ceramics was measured as 1040 MPa, while the measured residual strength of the ZrO_2 was only 100 MPa.

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残余应力对涂覆 Al_2O_3 涂层的 ZrO_2 陶瓷的强度和裂纹扩展阻力的影响

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摘 要: 本研究在 ZrO_2 基体表面涂覆一薄层 Al_2O_3 涂层, 利用基体与涂层之间热膨胀系数不匹配, 在 Al_2O_3-ZrO_2 预应力陶瓷(简称 A_CZ_S 预应力陶瓷)表层引入压应力。采用维氏压痕法评价残余应力对 A_CZ_S 预应力陶瓷的表层和基体中裂纹扩展阻力的影响。理论分析结合实验结果表明: 表层的压应力使得 A_CZ_S 预应力陶瓷的裂纹扩展阻力增大, 最终导致强度和损伤容限提高; 且 A_CZ_S 预应力陶瓷表层的压应力和裂纹扩展阻力随着基体截面积与涂层截面积比值的增加而增大。当 ZrO_2 基体表层的 Al_2O_3 涂层厚度为 40 μm 时, 表层压应力使 A_CZ_S 预应力陶瓷的弯曲强度达到 (1207±20) MPa, 相比于同种工艺下制备的 ZrO_2 陶瓷强度提高了 32%, 同时也是 Al_2O_3 强度的 3 倍。此外, A_CZ_S 预应力陶瓷也表现出很好的抗热震性能。

关 键 词: Al_2O_3-ZrO_2 预应力陶瓷; 压应力; 弯曲强度; 损伤容限

中图分类号: TQ174 **文献标志码**: A