Preparation and Properties of Pre-Stressed Ceramic Plate

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Abstract. Ceramics are widely used because of their excellent mechanical properties, electrical insulation properties, high temperature resistance and corrosion resistance. Ceramic plate is one of the important branches of ceramic components. However, brittle fracture caused by low toughness and high sensitivity to surface defects limits the reliability of ceramics. Therefore, there are a lot of research on strengthening and reducing defects of ceramic plate. In this work, a pre-stressed ceramic plate was obtained by a simple method, which was impregnated with a layer of alumina slurry on the surface of zirconia substrate and sintered at 1500°C. Because the expansion coefficient of alumina is lower than that of zirconia, a compressive stress is formed on the surface of the plate, which can reduce the defect sensitivity and improve the bending strength of ceramic plate. Compared with the pure zirconia plate, the strength was improved with about 40%.

1. Introduction
Ceramic materials are widely used in the fields of aviation, aerospace, ship, weapon, electronics and nuclear industry because of their high insulation properties, excellent corrosion resistance and high mechanical strength. Ceramic plate, as an important branch of ceramic products, has important applications in armor protection, human body protection, mobile phone communication, integrated circuit and other fields [1-2]. However, brittle fracture caused by low toughness and high sensitivity to surface defects limits the reliability of ceramic plate. Therefore, there are a lot of research on strengthening and reducing defects of ceramic plate, such as Particle enhancement, fiber enhancement, grain refinement, surface enhancement, etc., have been proposed to enhance the mechanical properties of ceramics and obtained great achievements[3-6]. However, large-scale and economical fabrication technology has not been achieved.

In this paper, we apply a pre-stressed design model to realize the pre-stressed enhancement of ceramic plate by a simple process, which is expected to obtain industrial application.

2. Experimental procedure
ZrO₂ ceramic plate were made by the following steps: 1) gel injection molding to prepare green body; 2) binder removal at 800°C; 3) the substrate samples pre-sintered at 1100°C for 2 hours; 4) dip to the slurry to obtain Al₂O₃ coating; 5) drying; 6) both Al₂O₃ coated ZrO₂ and uncoated ZrO₂ samples were sintered at 1500°C for 2 hour.

The formulation gel injection molding and the alumina slurry are shown in table 1 and table 2 respectively. The preparation process of pre-stressedZrO₂ ceramic plate as shown in figure 1. The particle sizes of 3Y-ZrO₂ powders and Al₂O₃ powders were 0.4 μm and 0.5 μm respectively. In the
table 1 and table 2, dispersant, defoaming agent and curing agent are acrylic/methacrylic acid salts, organic silicon and ammonium persulfate respectively.

Table 1. The formulation of gel injection molding

| ZrO₂ (g) | Acrylamide premixed solution (g) | Dispersant (g) | Defoamer (ml) | Curing agent (ml) |
|----------|---------------------------------|----------------|--------------|-------------------|
| 500      | 120                             | 11             | 0.3          | 0.3               |

Table 2. The formulation of alumina slurry

| Al₂O₃ (g) | Water (g) | Dispersant (g) | Defoamer (ml) |
|-----------|-----------|----------------|---------------|
| 150       | 87        | 1.2            | 0.3           |

Figure 1. The preparation process of pre-stressed ceramic plate.

The coefficient of thermal expansion (CTE) of Al₂O₃ coating ($\alpha_c = 7.5 \times 10^{-6}$ K$^{-1}$) is smaller than that of ZrO₂ substrate ($\alpha_s = 10.5 \times 10^{-6}$ K$^{-1}$) and consequently a residual compressive stress was created in the coating after sintering.

Flexural strengths of the ZrO₂ substrate specimens were measured using a double ring testing method with a loading rate of 0.5 mm/min [7]. Pre-stressed and pristine specimens were tested and compared. The measured value was the average of 10 separate measurements. The cross section and fracture surface of the specimen were determined using scanning electron microscopy (SEM).

Figure 2. Schematics of the coated substrates with (a) square cross section and (b) circular cross section. s is the cross section and t is the thickness of coating.
3. Results and Discussion

3.1. Pre-stressed Design

To generate compressive stresses in the surface layer for ceramic plates, it is required that the coating material have a lower coefficient of thermal expansion (CTE) and similar sintering temperature relative to the base material. Since the CTE of the coating is lower than that of the base, the surface compression stress will be generated during the cooling process after high temperature sintering. Cross sections of square and circular substrates are shown in figure 2.

Due to the expansion coefficient of alumina is lower than that of zirconia, a compressive stress is formed on the surface of plate. The preliminary study shows that the pre-stressed can be pre-designed by adjusting the ratio of the coefficient of thermal expansion (CTE) and the ratio of the cross-sectional area of the base material and the coating material, so as to meet the specific engineering requirements. The pre-compressive stress can be calculated by equation (1)[8].

\[
\sigma_{pre} = \left( \frac{A_s}{A_c} \right) \cdot \left\{ 1 - \left[ \frac{E_s A_s}{E_c A_c} + \frac{\alpha_s}{\alpha_c} \right] \left[ 1 + \frac{E_s A_s}{E_c A_c} \right] \right\} E_s \cdot \alpha_s \cdot \Delta T_c
\]

Where, \( \sigma_{pre} \) is the pre-stressed; \( A_s/A_c \) is the ratio of the cross-sectional area of substrate to coating; \( E_s/E_c \) is the ratio of the elastic modulus of substrate to coating; \( \alpha_s/\alpha_c \) is the ratio of the CTE of substrate to coating; \( E_s \) is the elastic modulus of substrate; \( \alpha_s \) is the CTE of substrate; \( \Delta T_c \) is the difference between sintering temperature and service temperature.

3.2. Effect of pre-stress on flexural strength

We have designed and fabricated pre-stressed ZrO_2 ceramic plates as shown in Figure 3. The measured flexural strength of ten pre-stressed ZrO_2 plate specimens is 1288 MPa (Figure 4(a)), which is 40% higher than its uncoated counterpart (920MPa). Tight bonding between the coating and the substrate indicates a good sintering compatibility as shown in Figure 4(b).

![Figure 3. Pre-stressed ZrO_2 ceramic plates.](image)

3.3. Effect of cross-sectional area ratio of substrate to coating on flexural strength of the pre-stressed ceramic plate

We designed and manufactured pre-stressed ZrO_2 ceramic plates with different cross-sectional area ratio of the base material and the coating material, as shown in figure 5. The flexural strength of ceramic plates with different cross-sectional area of the base material and the coating material was measured. The influence of cross sectional area on the flexural strength of ceramic plates was consistent with the predicted value of equation (1), as shown in Figure 6.
Figure 4. The flexural strength of pre-stressed ZrO2 ceramic plates compared to their untreated counterparts as well as the morphology of their cross sections. (a) Measured in flexural strength by double ring testing method for ten ZrO2 substrate specimens, ten ZrO2 plate specimens coated with alumina, the flexural strength is enhanced by 40% due to the expansion coefficient of the coating is lower than the substrate. (b) SEM photo showing the morphology of the interface between the ZrO2 and the alumina coating, the grain size of the substrate ZrO2 is nanometer scale and the grain size of coating is about 5um.

Figure 5. Morphology of cross-section of pre-stressed ZrO2 ceramic plates with different cross-sectional area ratio. (a) The cross-sectional area ratio is As/Ac=30; (b) As/Ac=19; (c) As/Ac=15.

Figure 6. Effect of cross-sectional area ratio of substrate to coating on flexural strength of the pre-stressed ceramic plate.
According to equation (1), by changing the ratio of cross-sectional area of substrate and coating, the variation law of surface pre-stressed obtained is shown in Figure 6. It can be seen from figure 6 that the surface pre-stressed increased with the increase of the cross-sectional area ratio of the substrate and the coating. During the research, by controlling the cross-sectional area ratio, the change rule of flexural strength obtained by measurement is consistent with the calculation rule, as shown in figure 6. In figure 6, point a represents the flexural strength of pre-stressed ZrO₂ ceramic plate with a cross-sectional area ratio of As/Ac:30 (1288MPa); Point b represents the flexural strength of pre-stressed ZrO₂ ceramic plate with a cross-sectional area ratio of As/Ac:19 (1160MPa); Point c represents the flexural strength of pre-stressed ZrO₂ ceramic plate with a cross-sectional area ratio of As/Ac:15 (1050MPa).

4. Summary
Pre-stressed ZrO₂ ceramic plates were obtained by a simple dipping method. Compared with the pure zirconia plate, the flexural strength of pre-stressed ZrO₂ ceramic plate was improved with about 40%. The flexural strength of pre-stressed ZrO₂ ceramic plate is closely related to the As/Ac. Within a certain range, the larger the As/Ac, the more significant the enhancement effect.

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