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Toughening Mechanism and Application Analysis of ZrO₂ Ceramics under High Speed Strain Condition

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Abstract. In order to study the impact properties of ZrO₂ ceramics and analyze the failure characteristics to improve the brittleness of ZrO₂ ceramics, we used a high-power glass pulsed laser to test the impact of ZrO₂ ceramics. Observing the fracture morphology by scanning electron microscopy, we found that the failure of ceramics is mainly caused by spallation. The cracked surface is dominated by typical brittle fractures, and there are more crystals appearing and cracking. The surface does not expand along a single plane, but forms a large number of steps. The direction of the crack and the density distribution indicate that the failure is caused by the reflected tensile wave. At the same time, the surface layer of the material generates a dense compression layer of about several tens of micrometers due to compressive stress, and no cracking occurs in the compression layer. This mechanism indicates that ZrO₂ ceramic materials with surface strengthening and matrix toughening can be formed under suitable laser energy.

1. Introduction

The inherent intrinsic properties of ceramics are brittle. Although various methods have been used to improve the toughness of ceramics, most of them are based on the study of composite materials [1-5]. Especially with the change of the loading mode, what kind of response and what kind of fracture mechanism is exhibited for pure ceramic materials under high-speed impact loading has not been well studied. There are no existing theories in this field at home and abroad.

Among the ceramic products, Al₂O₃ ceramics and ZrO₂ ceramics are the most common and the most widely used. The former has the best cost performance, while the latter has the best performance in single-phase oxide ceramics [6].

Laser shock loading of ZrO₂ ceramics, in addition to the fracture characteristics, can also analyze the phase transformation mechanism of ZrO₂ under tensile stress, the contribution of phase transformation to ZrO₂ toughening mechanism and the problem of phase change driving force.

2. Test materials and Test Methods

The test material is a single-phase tetragonal polycrystalline ZrO₂ ceramic customized in Changshu Huaxing Modern Ceramics Co., Ltd. After the finished product it is processed into a 45mm×45mm and 4mm thick sample using a grinding machine. The sample has a particle size of 0.5~1μm and the compressive strength is more than 1200MPa. Other major performance indexes are provided by the manufacturer as follows: density=6.02g/cm³, elastic modulus E = 365.2 GPa, fracture toughness 5.85 MPa m^{1/2}, Poisson's ratio 0.183.

The laser parameters used in the test are as follows: laser power adjustable range 10~49J; laser



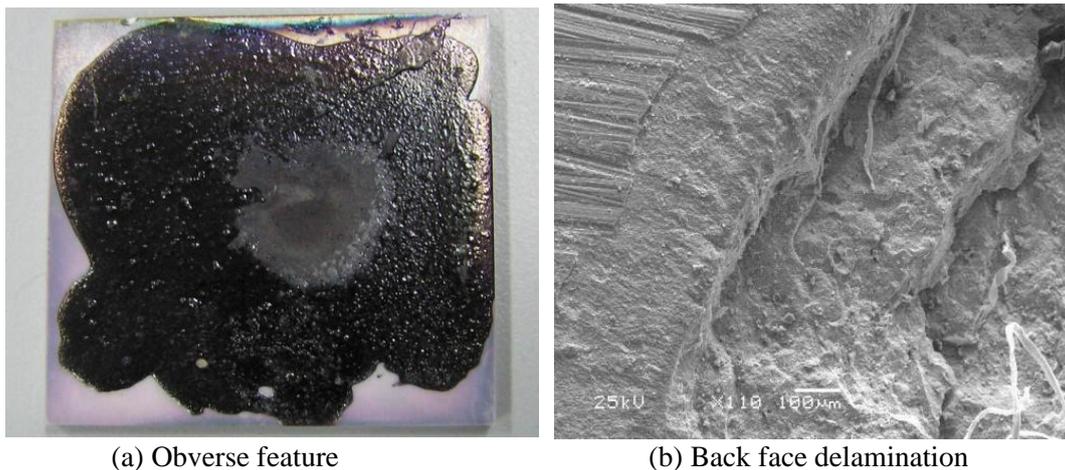
wavelength $1.054\mu\text{m}$; spot diameter 8mm, laser pulse width 23ns. Before the laser impact, we applied a black lacquer energy absorbing layer with a thickness of about 0.1 mm on the surface of the ceramic material. After the black paint is fully cured, the sample is placed on a five-axis console, and a spout is placed above the sample to make the water flow evenly through the area to be impacted. The thickness of the water layer is about 1 mm. The phase analysis was carried out on a D/MAX-rA type X-ray diffractometer. The test parameters were: tube flow 150 mA, tube pressure 50 kV, scanning speed $10^\circ/\text{min}$, $\text{CuK}\alpha$.

The JXA-840A scanning electron microscope (SEM) of Japan Electronics Co., Ltd. was used to observe the microscopic fracture morphology. In order to prevent the charging effect, the sample was sprayed with gold before observation and fixed on the sample holder with conductive resin.

3. Experimental Results and Analysis

3.1. Macroscopic Appearance

Figure 1 shows the macroscopic morphology of the ceramic material after the laser energy is 42J. It can be seen from the macroscopic characteristics that the material does not have large fragmentation and the front surface is intact. As shown in Fig. 1(a), no macro cracks are found after cleaning. However, the back side of the sheet is peeled off, and the thickness of the layer is about 0.1 mm, as shown in Fig. 1(b).



(a) Obverse feature

(b) Back face delamination

Figure 1. Delamination feature of ceramic by 42J laser shock processing

3.2. Hardness Analysis

This experiment tests the microhardness of ceramics, which reflects the ability of ceramic materials to resist damage. Table 1 shows the results of microhardness test of ZrO_2 ceramic samples:

Table 1. Hardness of ZrO_2 ceramic sample

Material status	Microhardness/HV				Average hardness
Original sample	1437	1400	1370	1380	1396.75
Laser shock	1570	1632	1700	1643	1636.25

The hardness of the sample after laser shock in the table is the hardness of the back surface after impact. It can be seen from Table 3-1 that the average hardness of the original sample without laser shock is 1396.75 HV. The average hardness of the back surface after laser shock is 1636.25 HV, which indicates that the hardness of ZrO_2 ceramics has been improved to some extent after laser shock. This is consistent with the results of XRD and SEM analysis. The increase in hardness is mainly due to the densification of the front surface of the ceramic and the phase transformation and toughening of

the back surface.

3.3. Toughness Analysis

In this experiment, the fracture toughness of ZrO₂ ceramics was measured by Vickers indentation method [7] (IM). According to the study by J. Lankford et al., a square indentation can be pressed on the ceramic surface by a microhardness tester, and cracks are radiated along the diagonal of the square indentation (Fig. 2). He proposed a formula for calculating fracture toughness:

$$\left(\frac{H}{E\Phi}\right)^{0.4} \left(\frac{K_{IC}\Phi}{H\sqrt{\alpha}}\right) = 0.129 \left(\frac{c}{\alpha}\right)^{3/2} \quad (1)$$

In the middle: K_{IC} —fracture toughness; H —Microhardness; Φ —Constraint factor (≈ 3); E —the modulus of elasticity of the material; α —Square indentation diagonal half length; C —half length of the crack radiated along the diagonal of the indentation.

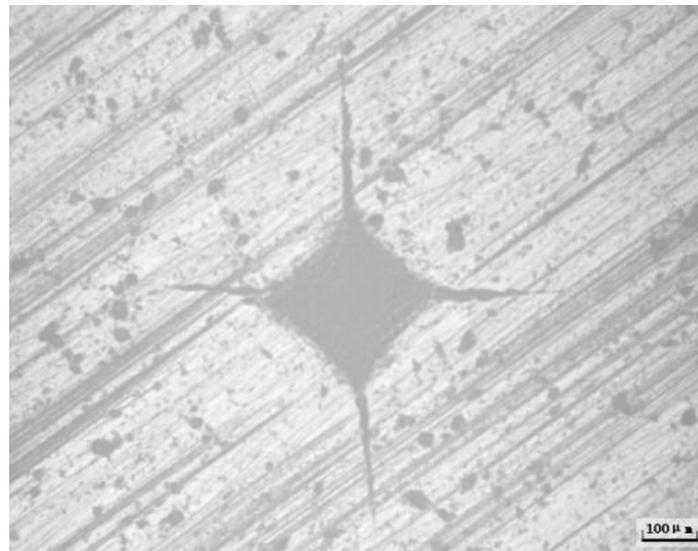


Figure 2. Photograph of the back surface indentation of the sample after laser treatment

These data are substituted into the above formula (1): $H=1636.25\text{kg/mm}^2 \approx 16\text{GPa}$, $E=230\text{GPa}$, $c=380\mu\text{m}=3.8 \times 10^{-4}\text{m}$, $\alpha=150\mu\text{m}=1.5 \times 10^{-4}\text{m}$,

$$\left(\frac{16}{230 \times 3}\right)^{0.4} \left(\frac{K_{IC} \times 3}{16 \times 10^9 \times \sqrt{1.5 \times 10^{-4}}}\right) = 0.129 \left(\frac{3.8 \times 10^{-4}}{1.5 \times 10^{-4}}\right)^{3/2} \quad (2)$$

solution $K_{IC}=9.5\text{MPa m}^{1/2}$.

It can be seen from Fig. 2 that the sample indentation after laser impact is square. Its edges are neat and linear, and cracks are radiated outward along the square diagonal.

Therefore, it can be seen that after impacting with a laser of 25J, the fracture toughness of the ZrO₂ ceramic material is changed from 5.85 MPa m^{1/2} to 9.5 MPa m^{1/2}, the fracture toughness is improved by more than 60%, and the toughening effect is ideal.

It is well known that ZrO₂ ceramics undergo such a phase change called martensitic transformation, and some places are called bainite transformation. The reason for obtaining the tetragonal phase at room temperature is mainly the binding effect of the surrounding materials on the zirconia particles, as shown in Figure 2 [8, 9].

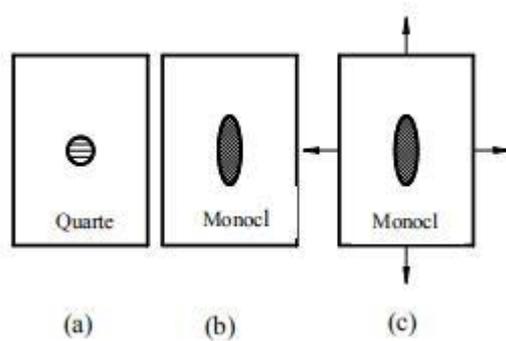


Figure 3. Sketch of transformation from cubic to diagonal

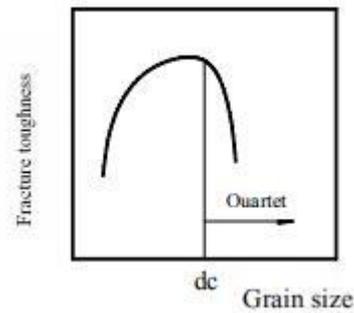


Figure 4. Effect on the fracture toughness of grain dimension

A large strain occurs from (a) to (b) during the phase transition, resulting in a change in the elastic energy that inhibits the phase change. This makes it possible to obtain a metastable tetragonal phase at temperatures below the "unconstrained" phase transition temperature. The phase change can be induced by applying stress (from (a) to (c)). It has been confirmed that in order to maintain the presence of the tetragonal phase, the size of the zirconia particles must be less than a certain critical value. It is generally believed that this value is about $1\mu\text{m}$. The size of the zirconia particles is very important for controlling the degree of stress-induced phase transition, as shown in Figure 3. In the case where the particle size is much smaller than the critical dimension d_c , the critical stress required to induce the phase transition is very high. Therefore, h is small and the toughening effect is poor. As the particle size increases, the stress-induced phase change becomes easier and the toughness increases accordingly. Maximum toughness is obtained when all particles have a size slightly smaller than d_c . It is obvious that the size distribution of zirconia particles has a certain effect in determining the volume fraction of phase transformation, and a narrower size distribution will cause more particles to undergo phase transformation. Particles larger than d_c have undergone a phase change before the force is applied. Correspondingly, the amount of $t\text{-ZrO}_2$ involved in the stress-induced phase transition process will decrease [10].

This theory can explain very well why the ZrO_2 fine powder particles can be processed to $0.05\mu\text{m}$. The ZrO_2 grain size used for phase transformation toughening is still maintained at $0.5\sim 1\mu\text{m}$. For ZrO_2 ceramic materials containing 2 mol% Y_2O_3 , the grain size larger than $1\mu\text{m}$ is difficult to ensure the formation of $t\text{-ZrO}_2$ at room temperature, while the grain size below $0.5\mu\text{m}$ makes phase transformation difficult, and it also increases the cost. In the experiment, the sample was destroyed by a laser with a higher power to study the toughening mechanism inside the laser-impacted ZrO_2 ceramic. However, if you want to use the toughening effect of laser impact ceramics in production applications, ZrO_2 ceramics can be impact processed with a laser of appropriate power. The energy of the shock wave is not enough to destroy the ceramic product, but it can make various toughening mechanisms work, so that the product can obtain excellent performance. Based on the above analysis, it can be found that the main damage of the material is caused by the tensile stress of the reflection. The compressive stress densifies the front surface of the material, which in turn strengthens the material. So we can improve the performance of ZrO_2 ceramics by controlling the magnitude of the reflected wave stress. According to the principle of wave propagation and reflection, if a substance that absorbs laser energy is attached to the back of the material, the energy of the reflected wave is inevitably reduced, and the magnitude of the tensile stress is correspondingly reduced. When the tensile stress is reduced below the tensile strength of the material, macroscopic cracks will not be caused inside the material, but the phase change caused by the tensile stress produces a micro-crack caused by the volume effect. These micro-cracks are non-expandable under the action of the load. Therefore, it does not significantly reduce the material strength. When the main cracks expand under the action of a large load and encounter these cracks, the extended crack will be diverted to absorb energy, which will increase the K_{IC} value, that is, microcrack toughening. At the same time, due to the small grain size of

ZrO₂ ceramics, the fracture toughness of the material is also increased. The dimple structure needs to absorb additional strain energy when it is pulled out to prevent the crack from continuing to expand, and also increase the fracture toughness of the material [11].

On the other hand, the reduction of the reflection tensile stress does not affect the compressive stress of the front surface, so the front surface of the material still forms a dense compression layer of a certain thickness. In this way, ZrO₂ ceramic materials with surface strengthening and matrix toughening can be formed, thereby expanding the application range and performance of ZrO₂ ceramics.



Figure 5. Annular fracture formed by weakened reflected wave (42J)

According to the above theory, the ZrO₂ ceramic was pre-modified. First, a uniform multi-point impact was applied to the ceramic surface using 10 J of laser energy, and then a single impact was performed with 42 J of energy.

It can be seen from the figure that the material has a macroscopic fracture at 42 J energy (Fig. 1b). After pretreatment, the material only has a tendency to crack, and a crack is formed on the back surface, and the degree of damage is not as large as that under 25 J energy. This process shows that pretreatment greatly increases the fracture toughness of the material and improves the catastrophic fracture resistance of the material. Compared with the previous toughening analysis, it can be found that in the overall toughening effect, in addition to the factors of phase transformation toughening, the microcrack toughening mechanism and the dense compression layer caused by the front side of the shock wave contribute to the overall toughening of the material.

4. Conclusion

- 1). The damage of the laser shock wave to the ZrO₂ ceramic material is mainly caused by the reflected tensile wave formed by the back surface, and its failure form is characterized by spallation.
- 2). The rear surface of ZrO₂ ceramic material undergoes stress phase transformation under the action of reflection tensile stress, which is transformed from t-ZrO₂ to m-ZrO₂.
- 3). Reducing the back reflection energy, thereby reducing the tensile stress level, can form a ZrO₂ ceramic material with surface strengthening and matrix toughening.

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