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Mechanical Properties and Constitutive Model of Toughening Ceramics

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Key words: Zirconia toughened alumina(ZTA), mechanical property, dynamic behaviors.

Summary: Mechanical properties of toughening ceramics were investigated under quasi-static loadings and shock loadings. The stress-strain curves of ceramics under quasi-static compressions showed an approximately linear relationship and an obvious brittle failure appeared in specimens. Furthermore, fracture toughness and flexural strength of zirconia toughened alumina were improved significantly because of the addition of ZrO2. Dynamic failure characteristics were studied using split Hopkinson pressure bar (SHPB) with strain rate ranging from $780s^{-1}$ to $1190s^{-1}$, suggesting that dynamic behaviors of ZTA ceramics are sensitive to the strain rate and dynamic compressive strength increases with rising strain rate. Meanwhile, due to the nucleation and growth of damage, the obvious nonlinear feature could be found from the stress-strain curves of ceramics. Hence, on basis of composite mechanics, a constitutive model was developed and its predicted results about elastic modulus and fracture toughness were in a good agreement with that of the experiments.

Introduction

Ceramic material has good mechanical and physical properties, such as high strength temperature resistance and corrosion resistance. Zirconia toughened alumina (ZTA) ceramic composite is the first developed Al_2O_3 ceramic matrix composites [1]. ZrO_2 has characteristic of phase transformation, while the toughening of cracks and residual stresses caused by ZrO_2 itself phase transformation can significantly improve the toughness of ZTA, and the improved ZTA is widely used to be the key parts of aircrafts, vehicles and ships.

When cooled from a high temperature to room temperature, ZrO_2 undergoes a c-t-m isomerization homogeneous transition in which t-m phase transformation tends to produce 3% to 5 % expansion in the volume. In 1976, Claussen [2] firstly applied the phase transformation toughening of ZrO_2 to obtain ZTA through purifying ZrO_2 particles and adding suitable amount of Y_2O_3 into Al_2O_3. The composite material exhibited high fracture toughness at room temperature ($11MPa.m^{1/2}$) when cooled from 1275℃. Wang and Stevens [3] performed experiments of ZTA ceramics with different content (0 ~ 20vol %) of unstable ZrO_2, from which they concluded that the size of micro-cracks generated by ZrO_2 phase

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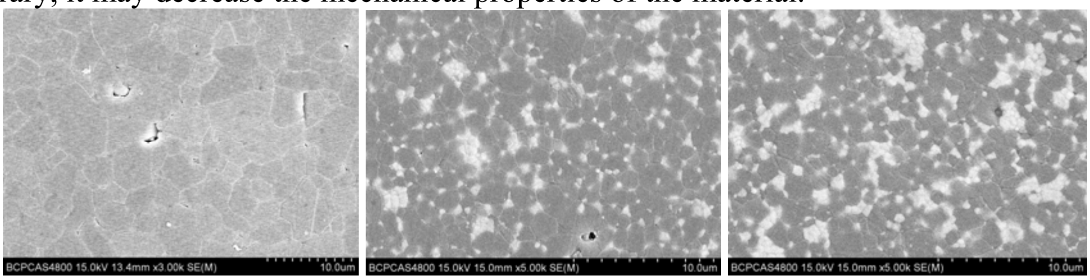
transformation is related to the content of stabilizer. Additionally, when the volume fraction of ZrO_2 is smaller than a critical value, the micro-cracks will be stable and will not be coalesced. Feng Wei [4] prepared 15% ZTA ceramic by using pressureless sintering method and studied its mechanical properties at low temperatures, obtaining that the flexural strength, fracture toughness and Vickers hardness at 77K are higher than those at 293K. Li Tingkai [5] investigated ZrO_2/Al_2O_3 ceramic composite materials with different contents of stabilizer Y_2O_3. Based on DOP, Zhang Xianfeng [6] conducted a series of experiments of the anti-shaped jet penetration performance of ZTA and AD95, and the results indicate that anti-jet penetration capability of ZTA is better than that of AD95.

In this paper, AD995 alumina, 15% ZTA and 25% ZTA were prepared by hot pressing sintering process, and a systematic study was made on their microstructure, mechanical properties and dynamic behaviors under impact loadings. According to the theory of composite material, a constitutive model for toughened ceramic was developed and the predicted results of the model about elastic modulus and fracture toughness were consistent with experimental results.

1 Micro-structure of ZTA

Two kinds of toughened ceramic samples were sintered at high temperature and consisted of Al_2O_3, a small amount of stabilizer Y_2O_3 and 15% and 25% ZrO_2 respectively. After sintered, the density of 15% ZrO_2-Al_2O_3 was 4.3g/cm^3 and 25 % ZrO_2-Al_2O_3 was 4.5g/cm^3. The toughening phase was not added to AD995 and the density of AD995 was 4 g/cm^3.

Figure 1 (a) -1 (c) are the microscopic scanning electron micrographs of three ceramic materials. As seen in the figures, the size of Al_2O_3 matrix grains in ZTA ceramic is smaller than that of AD995. On the one hand, ZrO_2 grains are evenly pinned between the Al_2O_3 matrix grain boundaries which restrict the growing of Al_2O_3 matrix grain and refine the grain. On the other hand, because volumetric expansion and shearing deformation of the ZrO_2 inhibit the original micro-cracks from growing, the average size of micro-cracks and micro-holes is effectively reduced in the sintering process. As a result, the densification of the toughened ceramics is further improved. Figure 1 (c) displays that some agglomerates are formed by white ZrO_2 grains and agglomeration is intensified with the increasing of ZrO_2 content. Related studies showed that when the volume of agglomerate exceeds a certain critical size, it will cause the micro-defects in ZTA ceramic, which decreases fracture toughness and strength considerably [6]. Therefore, considering the influence of processing technology, higher content of ZrO_2 may not exert the toughening influence over ZTA; on the contrary, it may decrease the mechanical properties of the material.



a) AD995 b)15%ZrO_2/Al_2O_3 c) 25%ZrO_2/Al_2O_3

Figure 1: Typical structure of SEM for three ceramics.

2 Mechanical Properties for Quasi-static loading

2.1 Compression strength

The schematic diagram of the experimental apparatus is shown in Figure 2. The equipment used in our experiment is WDW-300 electronic universal testing machine whose maximum loading force and loading speed are 300kN and 0.2mm/min.

Ceramic material at room temperature is a typical brittle material with characteristics such as high strength, high hardness, high sensitivity to deformation and small deformation to brittle fracture, which makes it difficult to obtain effective results through traditional compressive method. Consequently, two improvements were performed during the experimental measurement. Firstly, the axial deformation of the specimen was directly measured by the attaching strain gauges on the specimen, rather than calculated through the crosshead displacement of the testing machine. Secondly, considering that the hardness of ceramic is much higher than that of testing machine plate, two tungsten carbide pads were placed between the plate and specimen in order to protect the plate and prevent it from being embedded in by the specimen.

According to the stress-strain curves of ZTA shown in Figure 3, compressive properties of ceramic materials can be summarized as follows, which are that: (1) ceramic material are brittle material with small deformation and its failure strain is only a few thousandths reflecting the obvious feather of the brittle failure; (2) the stress-strain curves reveal the approximately linear relationship without significant plastic deformation during the loading process; (3) the addition of ZrO_2 particles significantly increases the compressive strength of the ceramic material, leading the fracture strength of AD995, 15% ZTA and 25% ZTA ceramic to be about 1.96GPa, 2.25GPa and 2.11GPa, respectively; (4) the elastic modulus of ZTA ceramic decreases with the increasing content of ZrO_2.

Figure 2: Compression test.

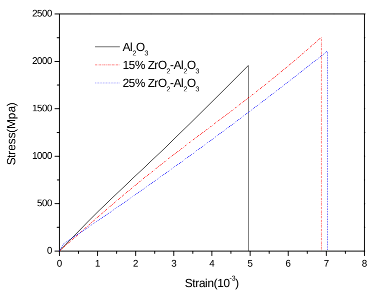


Figure 3: Stress-strain curve of ceramics.

2.2 Flexural Strength and Fracture Toughness

Three-point bending experiment was conducted to measure the flexural strength and fracture toughness of ceramics with the help of RGM-3100 electronic universal testing machine, whose maximum load was 100kN and loading rate was 0.2mm/min. The size of ZTA specimen was 40mm × 5mm × 10mm and the specimen in fracture toughness test was processed with a 4mm-in-depth notch in the middle. The experimental results in Table 1 apparently indicate that the flexural strength and fracture toughness of ZTA ceramics are much higher than those of AD995 ceramic, which suggests that ZrO_2 plays an indispensable role in increasing the fracture toughness of toughened ceramic.

	Al_2O_3	$15\%ZrO_2\text{-}Al_2O_3$	$25\%ZrO_2\text{-}Al_2O_3$
Flexural strength/MPa	372.4	583.5	598.3
Fracture toughness/$Mpa{\cdot}m^{1/2}$	5.65	6.55	8.42

Table 1: Flexural strength and fracture toughness.

3 Dynamic behaviors of ZTA

The split Hopkinson pressure bar apparatus with the diameter 37mm was used to explore the dynamic behaviors of ZTA. The strain gage was located at the center of each bar to record stress waves propagating in the bar. Two one-centimeter-thick spacers with the same diameter and material composition with the bar, were added between the specimen and the incident and transmission bar, so as to prevent the end surfaces of the two bars from being damaged. In order to guarantee the complete contact between the specimen and spacer, some butter was smeared between them before the experiment. The size of ZTA ceramic specimen is $\phi 14 \times 6\text{mm}$ and more details of the equipment can be seen in the schematic of split Hopkinson bar apparatus (Figure 4).

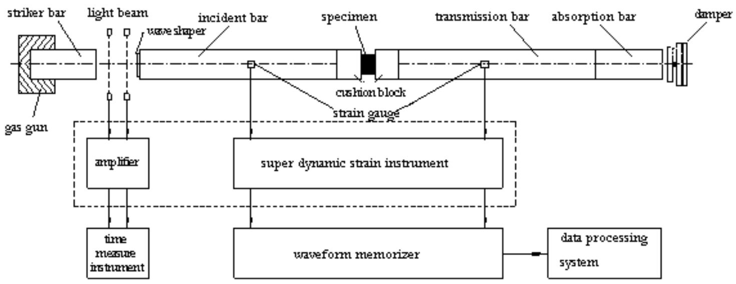
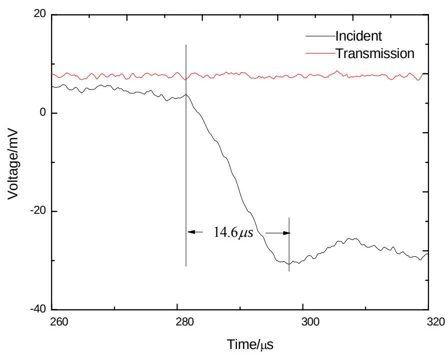
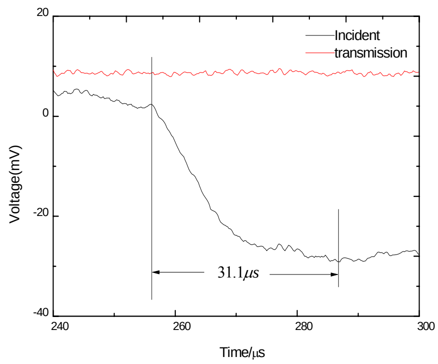


Figure 4: Schematic of modified SHPB.

The copper disc with a diameter of 14mm was used to increase the rising time of incident wave in order to get stress balance between the two sides of the specimen [7]. Figure 5 shows two sets of obtained waves, one with pulse shaper and the other without pulse shaper. We can find that the rise time of incident wave increases from 14.6μs to 31.1μs after adding the shaper, while the peak load on the specimen is not greatly affected.





(a) Without a pulse shaper (7.27m/s) (b) With a pulse shaper (7.69m/s)

Figure 5: The effect of pulse shaper on rising edge.

Base on the one dimensional stress wave theory for axial impacted bar, the stress-strain curves of the specimen can be calculated through the incident wave, reflected wave and transmitted wave of pressure bar [8]. The formula of strain rate, strain and stress can be written as

$$\begin{cases} \dot{\varepsilon}(t) = -\dfrac{2c}{L_0}\varepsilon_R(t) \\ \varepsilon(t) = -\dfrac{2c}{L_0}\displaystyle\int_0^t \varepsilon_R(\tau)d\tau \\ \sigma(t) = \dfrac{A}{A_0}E\varepsilon_T(t) \end{cases} \tag{1}$$

Where $\varepsilon_R(t), \varepsilon_T(t)$ respectively presents reflected wave and transmitted wave; c is the velocity of stress wave; L_0 is the thickness of the specimen; A and A_0 respectively present the cross-sectional area of the bar and specimen.

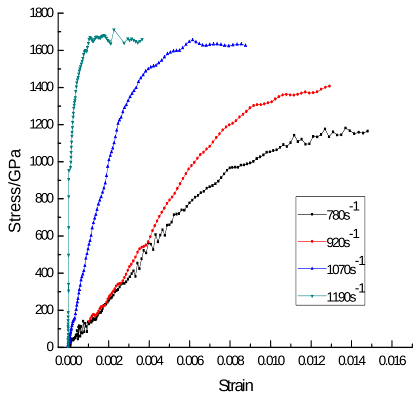


Figure 6: Dynamic stress-strain curves of 15% ZTA.

The stress-strain curves of 15% ZTA ceramic specimen under different impact velocities can be seen in Figure 6. The curves show that: 1) dynamic compressive strength increases with increasing strain rate, while failure strain decreases with increasing strain rate; 2) dynamic stress-strain curve presents obvious nonlinear characteristics, which is caused by the nucleation and growth of micro-cracks. The volume change and shear effect occurred because of the ZrO_2 particle transformation from tetragonal to monoclinic. A number of micro-cracks were formed around the phase-transform particles which played the role to disperse the tip energy of main cracks and improve the strength and toughness of ZTA ceramics. However, the nucleation and growth of large number of micro-cracks would result in the strength decrease of the material, and when damages were accumulated to a certain extent the material would fracture [9].

4 Constitutive Model Based on the Criteria of Phase Transformation

Two toughened ceramics were sintered at high temperature with matrix Al_2O_3 powder, ZrO_2 and a small amount of stabilizer. From the view point of the material microstructure, ZTA ceramic could be considered as a kind of composite material consisted of matrix phase, ZrO_2 mixed phase and a small amount of micro-cracks phase [10]. In the theoretical analysis, the following assumptions had been made: (1) Young's modulus of ZrO_2 was invariant before and after the phase transformation; (2) phase transformation of unit volume was constant; (3) ZrO_2 was spherical inclusion and micro-cracks were the type of coin. According to the Mori-Tanaka method, assuming an average distribution of micro-cracks in all directions, the relationship between macroscopic stress $\bar{\sigma}$ and macroscopic strain $\bar{\varepsilon}$ of the composite material is

$$
\begin{aligned}
&\bar{\sigma}=\{C_0L_0+C_2L_2[I+S_2L_0^{-1}(L_2-L_0)]^{-1}\}\\
&:\{C_0I+C_1[I-S_1]^{-1}+C_{2I}[I+S_2L_0^{-1}(L_2-L_0)]^{-1}\}^{-1}\\
&:(\bar{\varepsilon}-C_{2II}<\varepsilon>^T)=M^{-1}:(\bar{\varepsilon}-C_{2II}<\varepsilon>^T)
\end{aligned} \tag{2}
$$

where C_0, C_1, and C_2 respectively present the volume ratio of Al_2O_3 matrix phase, micro-cracks phase and ZrO_2 mixed phase; L_0, L_1, and L_2 respectively present the stiffness tensor corresponding to the above phases; $C_2 = C_{2I} + C_{2II} : C_{2I}$ present the volume fractions that do not occur in phase transformation, C_{2II} presents the volume fraction that occurs in phase transformation; $<\varepsilon>^T$ presents the abrupt strain tensor, which equals to the sum of ε^T caused by phase transformation and ε_r^* caused by volume expansion of new micro-cracks generated during the phase transformation, which should be noted that we only consider the volume expansion of phase transformation; S_1 and S_2 respectively present Eshelby tensors corresponding to micro-cracks and ZrO_2 inclusion.

The relationship between the mechanical parameters of ZTA and the content of ZrO_2 can be found in Figure 7. With the increase of ZrO_2 content, the fracture toughness of ZTA rises; however, elastic modulus of ZTA ceramic decreases which is in accord with the rule of composites. Three groups of the experimental and theoretical results are in good agreement with the each other.

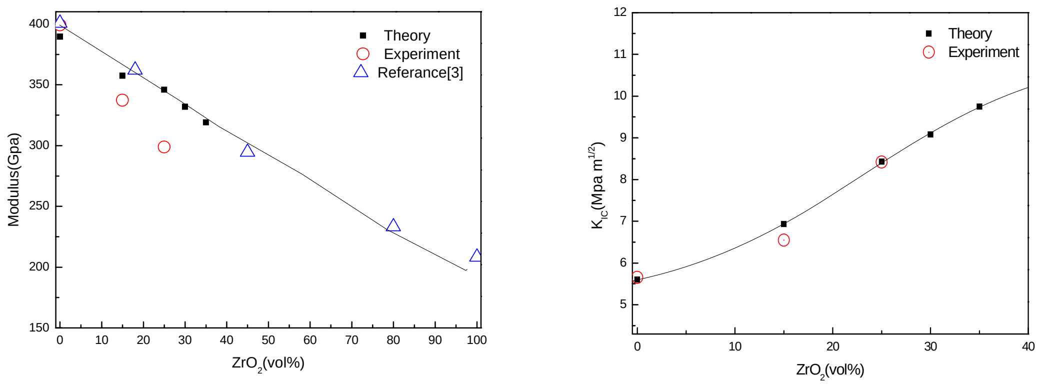


Figure 7: Mechanical behaviors with a function of ZrO_2 content.

5 Conclusions

The mechanical property of ZTA ceramic processed through hot pressing sintering was studied by using testing machine. Experimental results indicated that ZTA ceramic had higher flexural strength and fracture toughness than pure alumina ceramic and the approximately linear stress-strain curves of ceramic under quasi-static compression were obtained. From the micro-structural view point of toughened ceramic, ZrO_2 particles pinned between the grain boundaries induced the growth of matrix particles and played an important role in improving the compressive strength and fracture toughness. Therefore, the phase transformation of ZrO_2 is the main physical mechanism for improving the mechanical properties of ZTA ceramic.

Dynamic behaviors of ZTA were investigated using modified split Hopkinson pressure bar, whose pulse shaper was used to increase rising time of incident wave in order to obtain the stress equilibrium of specimen. We came to conclude that the fracture strength of ZTA increases with increasing strain rate and dynamic stress-strain curve reveals obvious nonlinear characteristics after elastic stage, which means that there are lots of nucleation and growth of the damage in the specimen before fracture.

Based on the theory of composite mechanics, ZTA ceramic was viewed as composite material consisted of matrix Al_2O_3 , ZrO_2 mixed phase and micro-cracks phase, and the constitutive model of ZTA ceramics was established considering the phase transformation strain of the inclusions and the growth of micro-cracks. According to the experimental results, the fracture toughness, Young's modulus and the strength of ZTA ceramic material increase with the increase of ZrO_2 content. The results of theoretical analysis are in good agreement with the experiments.

Acknowledgments

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References

[1] Ge Qilu, Lei Tingquan. Progress of Al_2O_3-ZrO_2 ceramic composites. Metal Science and Technology,1992,01:85-100.

[2] Claussen N. Fracture toughness of $Al_2O_3$with an unstabilized ZrO_2dispersed phase,Journal of the American Society,1976,59(1-2):49-51

[3] Wang, J. and R. Stevens. Zirconia-toughened alumina (ZTA) ceramics. Journal of Materi als Science, 1989,24(10): 3421-3440

[4] Feng Wei, Xie Zhipeng, Xue Weijiang, Xue Wendong. Mechanical and thermal properti es of Al_2O_3-ZrO_2 composite ceramics at cryogenic temperatures. Bulletin of the Chinese ceramic society,2012,03:379-384

[5] Li Tingkai, Shen Zhijian, Ding Zishang. Mechanical properties of Al_2O_3-ZrO_2composite ceramics. Bulletin of the Chinese ceramic society,1990,01:39-46

[6] Zhang Xianfeng, Li Yongchi,Yu Shaojuan. An Experimental study on anti-penetration pr ocessof zirconia toughened alumina ceramic target to shaped charge jet. Journal of Exper iment-al Mechanics,2007,06:631-636

[7] Chen W, Song B, Frew D J, et al. Dynamic small strain measurements of a metal specimen with a split Hopkinson pressure bar. Experimental Mechanics,2003,43(1):20-23

[8] Wang Li li. Foundation of Stress Wave. Beijing:National Defense Industry Press, 2010

[9] Ning J G, Ren H L, Fang M J. A constitutive model based on the evolution and coalescence of elliptical micro-cracks for quasi-brittle materials.Chinese Science Bulletin,2012,57(28-29):3773-3781

[10] Long B. Mechanics and toughening mechanism of zirconia toughened alumina ceramics[D]. Beijing:Beijing Institute of Technology, 2013(in Chinese)