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Effect of Yttria on the Synthesis, Microstructure and Mechanical Properties of Partially Stabilized Zirconia in α - Al_2O_3 Matrix

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ABSTRACT

The effect of Y_2O_3 addition on the phase transition, microstructure and mechanical properties of ultra-fine partially stabilized zirconia (Y-PSZ) in α - Al_2O_3 matrix prepared by co precipitation process has been studied. In the present work, the degree of phase stabilization of different mol% Y_2O_3 doped zirconia (Y-PSZ) was studied. 10 vol % Y-PSZ (2-3 mol% Y_2O_3) in α - Al_2O_3 matrix was prepared with the simultaneous co-precipitation process. The stabilization of crystallographic and thermal phases was characterized by XRD, TG-DTA. The samples were calcined in the range of 700-800°C. The fabricated samples were sintered at a temperature of 1600°C for 2-4 hrs. The sintered samples were characterized for their physical (such as density) and mechanical properties (flexural strength, hardness and fracture toughness). The morphology and microstructure have been observed by FESEM. It has been observed that the significant improvement in fracture toughness with retention of high hardness in the order of ~1700 HV. The fracture toughness was in the order of 12.0 $\text{MPa}\cdot\text{m}^{1/2}$ with the 10 vol% of Y-PSZ (2.5 mol% Y_2O_3) in α - Al_2O_3 .

Introduction

Zirconia based ceramics is one of the most promising structural material because of its high hardness, high corrosion and erosion resistance and high temperature strength. The toughness of this ceramics can be increased using the stress induced phase transformation [1-4]. Zirconia based ceramics are also well known for their excellent electronic, thermal, optical & mechanical properties [1-3]. Pure ZrO_2 exists in three crystalline forms namely monoclinic (m), tetragonal (t) and cubic (c). It also exhibit phase transformation: Monoclinic (m) $\xrightarrow{1170^\circ\text{C}}$ Tetragonal (t) $\xrightarrow{2270^\circ\text{C}}$ Cubic (c) [4] where (m) and (c) are stable phases and (t) is a metastable phase. The transformation of pure zirconia from the 'm' phase to 't' phase is accompanied by a volume increase of about 4-5% causing cracking and structural failure [4,5]. Due to this volume increase of pure Zirconia makes it unsuitable for any application [6]. For stabilizing the high temperature t-phase at room temperature, different additives/ dopant can be mixed like Y_2O_3 , CeO_2 , MgO , CaO [7-10]. Yttria was found to reduce t \rightarrow m transformation from 1100°C to approximately 550°C [11, 12]. In

yttria stabilized zirconia toughened alumina, the ideal microstructure is the single tetragonal phase as it increases the strength, hardness and fracture toughness of the sintered zirconia materials [12, 13]. The high toughness is due to a phase transformation induced by a stress relief due to crack propagating in the metastable t phase. The increase in volume accompanying transformation causes compressive stresses. These stresses effectively close the crack and block further crack growth, which can improve the strength, fracture toughness, and hardness of the ZTA [5]. In particular zirconia stabilized with 2-4 mol% Y_2O_3 has been used as a structural ceramics, with high hardness, strength [14] and high fracture toughness derived from stress induced phase transformation while 10-15 mol% of CeO_2 (Ce-PSZ) plays a vital role on hydrothermal stability and mechanical properties [15]. It has been reported that ceria doped zirconia polycrystals (Ce-PSZ) more than 10 mol% have high fracture toughness and high thermal stability compared to yttria doped zirconia polycrystals (Y-PSZ) ceramics [16]. Bravo et al reported that the trend towards increasing phase stability with decreasing particle-size of t- ZrO_2 could overcome by adjusting the yttria dopant concentration [17] whereas 2-3 mol% Y_2O_3 was found to optimize fracture toughness in submicron t- ZrO_2 dopant

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concentrations. To meet all these requirements, several powder processing, forming and sintering processes were investigated. For powder synthesis, methods including attrition milling, CVD synthesis, spray ICP, colloidal processing, hydrothermal reaction, evaporative decomposition, hydrolysis coating, plasma synthesis, sol gel processing and co predicated process were used [18-21] However, the phase transition and mechanical property evaluation 10 vol% Y-PSZ with different mol % Y_2O_3 in $\alpha-Al_2O_3$ in has not been explored significantly. The main purpose of the present investigation is to examine the effect of different mole % of Y_2O_3 addition in ZrO_2 on the phase transition and growth of Y-PSZ crystal and to find out the effect of different mole % of Y_2O_3 addition in 10 vol.% of Y-PSZ in $\alpha-Al_2O_3$ matrix for better physical and mechanical properties (flexural strength, hardness and fracture toughness) with respect to wear resistant characteristics as cutting tool materials.

Experimental

Synthesis of Y-PSZ (0-3 mol% Y_2O_3 doped) and 10 vol% Y-PSZ (2-3 mol % Y_2O_3 doped) in $\alpha-Al_2O_3$ Powder

Partially / fully stabilized zirconia by Y_2O_3 powders varying from 1.0 to 3.0 mol % was synthesized using wet chemical co-precipitation process of requisite amount of $ZrOCl_2 \cdot 8H_2O$ and $Y(NO_3)_3 \cdot 5H_2O$ solution for study of different degree of stabilization. The particular concentration of Y-PSZ with 2-3 mol% Y_2O_3 doped having more pseudo-tetragonal phase was chosen for 10 vol % reinforcement with $\alpha-Al_2O_3$ matrix for making advanced composites by co-precipitation method. These composites were made to correlate the phases with mechanical strength of the material i.e. to study the flexural strength, hardness and fracture toughness. The simultaneous co-precipitation process was done using requisite amount of zirconyloxochloride $ZrOCl_2 \cdot 8H_2O$ (>99%, BDH, India), $Y(NO_3)_3 \cdot 5H_2O$ (Aldrich, USA) and $Al(NO_3)_3 \cdot 9H_2O$ (LobaChemie, India). The homogenized mixed solution were prepared in a 5 lit glass beaker with a magnetic stirrer system, considering the desired stoichiometry of the metal oxides in the final composition of yttria doped zirconia toughened alumina (Y-ZTA) ceramics. The simultaneous co-precipitation was done with dilute NH_4OH at pH ≈ 9 in all the cases of 10 vol % yttria doped zirconia in α -alumina matrix. After complete precipitation, the stirring was continued for few hours to maintain the homogeneity of co-precipitated gel. The hydrated gelatinous precipitate was thoroughly washed with hot distilled water until complete elimination of anions in the solution phase. Then the precipitate was dried in air oven at $80^\circ C$ for several hours. The samples of 10 vol% Y-PSZ (2, 2.5 and 3 mol% Y_2O_3) in $\alpha-Al_2O_3$ were prepared. The dried mass was calcined at $700-800^\circ C$. The calcined mass of Y-ZTA was ball milled in alcohol media for 24 hrs using high alumina balls in a 500 ml high alumina jar contained planetary mill (Fritsch, Germany). The dried milled powders were uniaxially and hydraulically compacted at the pressure of 200-300 MPa in a suitable cylindrical (with 10 mm in diameter) die and the compacts were sintered at $1600^\circ C$ for 2-4 hours. The flow chart for the preparation of Y-ZTA is shown in Fig.1.

Sample Characterization Study

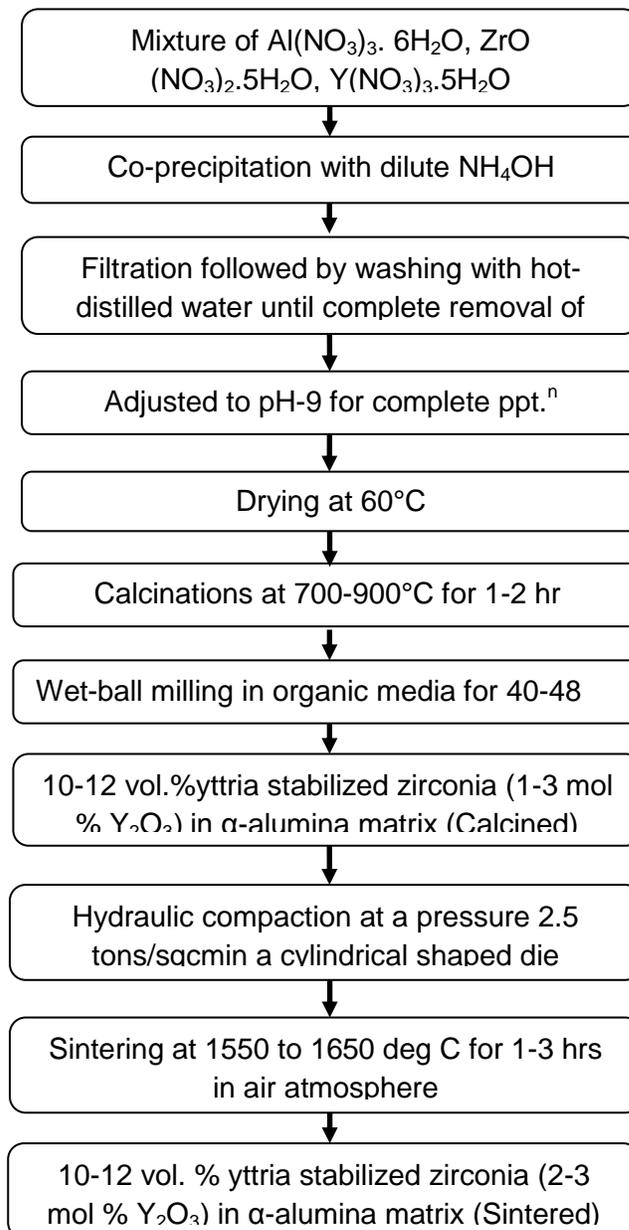


Fig (1). Flow Chart

XRD for different mol% of Y_2O_3 in PSZ was carried out over very short range from $27-33^\circ$ with a scan rate of $3^\circ/min$ to study the degree of phase stabilization. After visualizing more stabilized tetragonal phase within 2-3 mol% of Y_2O_3 in Y-PSZ system, the 10 vol% Y-PSZ (2.5 mol% Y_2O_3) in $\alpha-Al_2O_3$ matrix was studied by XRD ($CuK\alpha$, Ni filter) using a powder diffractometer (SHIMADZU Model. XRD 6000, Japan) to see whether the t- ZrO_2 phase exists or not in presence of $\alpha-Al_2O_3$ matrix. The XRD patterns were recorded over the angular range of $20-75^\circ (2\theta)$ with a scan rate of $2.8^\circ/min$. Thermal analysis of all the samples was performed in a simultaneous thermal analyzer (Netzsch, STA-449, Jupiter, Germany). Sample of about 30-40 mg were heated from 30 to $1550^\circ C$ at a heating rate of $10^\circ C/min$ and

a flow rate of 20 ml/min in a nitrogen atmosphere. Field Emission Scanning Electron Microscope (FESEM) (CARL-ZEISS-SMT LTD, Germany, Model: SUPRA 40) were used to examine the morphology of yttria based zirconia toughened alumina powders.

Mechanical Characterization Study

Weighing the samples and determining their volumes by Archimedes method bulk densities and porosities of sintered specimen were evaluated. The hardness and fracture toughness of the sintered polished specimens were measured by Vickers hardness testing machine (Matsuzwa, MXT-70). Fracture toughness was determined by the Vickers indentation technique. The fracture toughness K_{IC} was estimated using the equation

$$K_{IC} = 0.055Ha^{0.5} (l/a)^{-0.5}$$

Where H is the Vickers hardness calculated from $H = 0.464 P/a^2$, a is the half diagonal length of the indentation and l is radial crack length from the corner of the indentation. To find out the flexure strength of the specimen, the samples were prepared in the dimension of 2.5 X 3.5 X 30 mm². Then 3 pt bend test with a span of 20 mm at a cross head speed of 0.5 mm/min was conducted in Universal Testing Machine (Tinius Olsen, H50 KS).

Result & Discussion

Phase Characterization Study

Phase Characterization Study of Y-PSZ (0-3 mol% Y₂O₃ doped) powder

The XRD pattern of sintered samples at 1600°C of Y-PSZ (0-3 mol% Y₂O₃ doped zirconia) was shown in Fig. 2(a). The stabilization of the phases was studied at low angle (2θ between 27° and 35°) especially to distinguish between monoclinic and cubic/tetragonal phases and to determine the crystallite size of the samples. The phase transition, density, crystallite size and grain size is listed in Table 1. It has revealed that in case of 3 mol% Y₂O₃ additions in zirconia, the formation of stabilized zirconia specially tetragonal zirconia (t-ZrO₂) is predominant whereas in 2.0 mol% Y₂O₃ addition, the retention of t-ZrO₂ is moderate and metastable. It can be concluded from this study that Y₂O₃ had dissolved in ZrO₂ with the formation of stabilized ZrO₂ (t-ZrO₂) in case of 3 mol% Y-TZP and relatively small amount of monoclinic ZrO₂ (m-ZrO₂) presents in the same sample. The t-ZrO₂ retention also increases with Y₂O₃ addition with the trend similar to that observed for density variation. The maximum t-ZrO₂ retention is 92% at 3 mol% of Y₂O₃ addition. The t-ZrO₂ retention is very low in the composites prepared with 1.0 mol% Y₂O₃. The density also increases with increase in mol% of Y₂O₃ addition. To The ratio of the monoclinic to tetragonal phase is calculated with the integral intensities of the monoclinic peaks (111) and (111) versus the tetragonal peak (101) using the following relationship [22–24]

$$M (\%) = \frac{I_m (111) + I_m (111)}{I_m (111) + I_m (111) + I_t (101)}$$

Where M (%) the ratio of is monoclinic I_m is the intensity of

monoclinic and I_t is the intensity of tetragonal. The particular concentration of Y-PSZ with 2-3 mol% Y₂O₃ doped having more pseudo-tetragonal phase was chosen for 10 vol % reinforcement with α-Al₂O₃ matrix for making advanced composites by co-precipitation method. These composites were made to correlate the phases with mechanical strength of the material i.e. to study the flexural strength, hardness and fracture toughness of the composite.

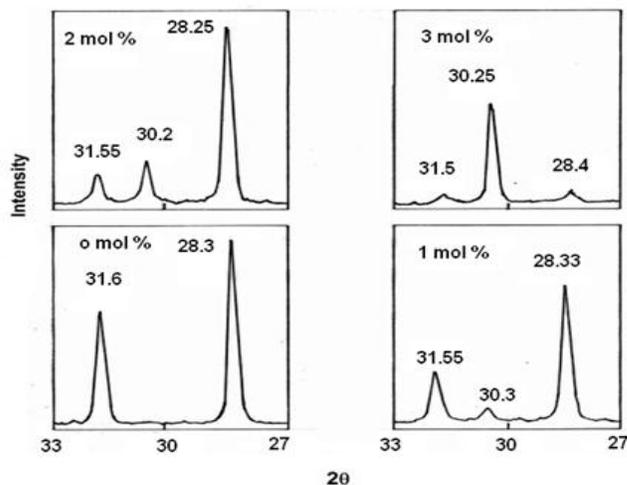


Fig 2(a). XRD patterns of Y-PSZ (0-3 mol% Y₂O₃ doped zirconia)

Phase Characterization Study of 10 vol% Y-PSZ (2.5 mol% Y₂O₃) in α-Al₂O₃ powder

The X-ray diffraction (XRD) of 10 vol. % Y-PSZ in α-Al₂O₃ matrix sintered at 1600°C was studied for determining the degree of stabilization of phases as shown in Fig. 2b. The stabilization of phases specially to distinguish between monoclinic and cubic/ tetragonal phases were identified at low angle (2θ between 27 and 35°) and also at higher angle (48- 52°). However, in case of 2 and 3 mol % Y₂O₃ (Y-PSZ) with same volume % in α-Al₂O₃ matrix were also carried out but not shown in the figure. The ZrO₂ particles are mainly of tetragonal phase embedded in α-Al₂O₃ matrix, which shows the presence of t-ZrO₂ and α-Al₂O₃ as major phases along with small amount of monoclinic zirconia in case of Y-PSZ system. The main peaks of t-ZrO₂ are sharp and high. The tetragonal/ phase in α-Al₂O₃ matrices play a vital role for enhancing the fracture toughness and hardness of the material.

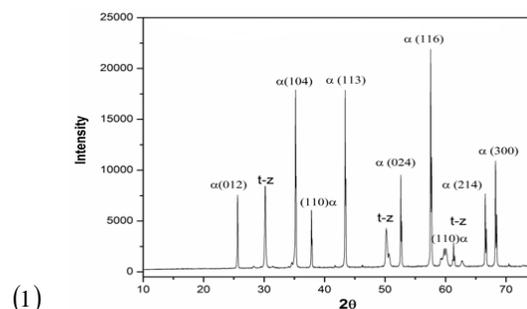


Fig 2(b). XRD patterns of 10 vol % Y-PSZ (2.5 mol% Y₂O₃) in α-Al₂O₃ matrix

Thermal Behavior of 10 vol % Y-PSZ (2, 2.5 and 3 mol% Y₂O₃) in α -Al₂O₃ matrix

The resulting dried mass (heated at 70°C) of co-precipitated powders of 10 vol.% Y-PSZ (with 2.0, 2.5, 3.0 mol% Y₂O₃) in alumina precursors were studied through thermal analysis (TA) as shown in Fig.3. In all the cases the endothermic peak range at 180-240°C corresponds to the decomposition and transformation of γ -AlOOH to γ -alumina in presence of 10 vol % Y-PSZ in alumina precursor. The sharp exothermic peak centered at around 460-472°C. The exothermic peak shifts towards high temperature with increase in Y₂O₃ content. These exothermic peaks are attributed to the transformation of pseudo tetragonal phase of zirconia. The broad endothermic peak ranging 600-800°C indicates the formation of various transition phases of alumina before conversion to α -alumina at ~1150°C. The phase transition of α -alumina starts at 900°C, fully transform to α -alumina at ~1200°C. No exothermic peak was observed in the higher temperature range around 1200 °C, which clearly indicates the absence of monoclinic to tetragonal phase transition.

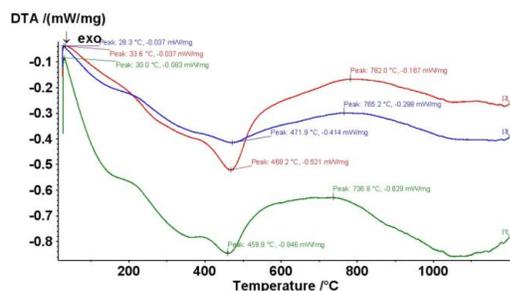


Fig 3 DTA curve of 10 vol % Y-PSZ (2-3 mol% Y₂O₃) in α -Al₂O₃ matrix

Morphology of Y-ZTA Powder

Fig. 4(a) and Fig. 4(b) showed the FESEM image of sintered surface and sintered fracture surface of 10 vol % Y-PSZ (2.5 mol% Y₂O₃) in α -Al₂O₃ matrix respectively. Homogeneous distribution of finer particles in the range of 20-40 nm was observed in the sintered powder. This sintered compact at 1600°C shows significant grain growth. A close-up view of the sintered Y-ZTA ceramics showed a very uniform dispersion of the zirconia grains within the alumina grain matrix. The average grain size of α -Al₂O₃ is ~ 1 μ m and a very fine grain of Y-TZP in the range of 0.2-0.3 μ m.

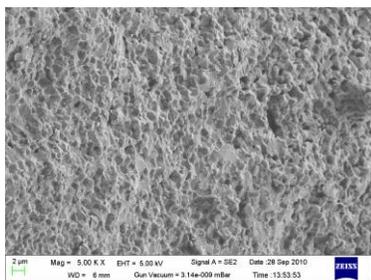


Fig 4 a. FESEM of 10 vol % Y-PSZ (2.5 mol% Y₂O₃) in α -Al₂O₃ matrix (sintered)

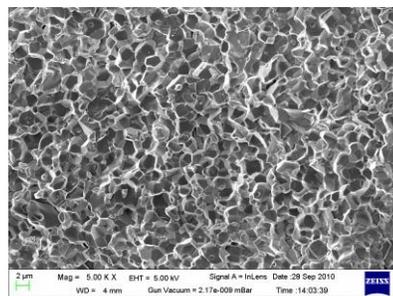


Fig 4 b. FESEM of 10 vol % Y-PSZ (2.5 mol% Y₂O₃) in α -Al₂O₃ matrix (Fracture Sintered)

Mechanical Characterization of Y-ZTA sintered samples

Study of different mechanical properties like flexural strength, hardness and fracture toughness for different mole % of Y₂O₃ addition in 10 vol% of Y-PSZ with α -Al₂O₃ matrix has been carried out and reported in Table 2. The fracture toughness of alumina-zirconia composites depends on the morphology and distribution of zirconia particles, their size, shape, location in alumina matrix and size distribution, which are critically influenced by the processing methodology and Y₂O₃ content. At 2.5 mol% Y₂O₃, not only the retention of t-ZrO₂ was higher but also sintered density also high as well as the grain sizes also finer. When the Y₂O₃ content increases from 2.5 to 3.0 mol %, then most of the Y₂O₃ doped ZrO₂ grains transformed to m-ZrO₂ during cooling and retention of t-ZrO₂ is less and reduces the hardness as well as fracture toughness of the sample. The flexural strength is also increased from 2 mol% to 2.5 mol% of Y₂O₃ and after that it also reduces. The sintered density and t-ZrO₂ retention also affects the flexure strength of the composite which is highest at 2.5 mol% Y₂O₃ added 10 vol% Y-PSZ in α -Al₂O₃.

Conclusions

The effect of different mole % of Y₂O₃ addition in ZrO₂ on the phase transition and growth of Y-PSZ crystal has been investigated by XRD using a powder diffractometer. After visualizing the tetragonality factor of ZrO₂ crystal, 2-3 mol% Y₂O₃ doped ZrO₂ has been selected for doping in 90 vol% α -Al₂O₃ to correlate the phases with flexural strength, hardness and fracture toughness of the developed composites. The following conclusions can be drawn from this study

- The retention of metastable tetragonal is predominant in case of 2-3 mol% Y₂O₃ doped ZrO₂.
- The developed sintered Y-ZTA ceramics showed a very uniform dispersion and microstructure of the zirconia grains within the alumina grain matrix.
- As the Y₂O₃ content increases, the exothermic peak in the DTA curves of Y-ZTA shifts to high temperature.

Significant improvement in fracture toughness, hardness & flexural strength has been observed in the order of ~ 12.20 MPa m^{1/2}, ~1700 HV and 510 MPa respectively for 10 vol% of Y-PSZ (2.5 mol% Y₂O₃) in α -Al₂O₃ which can be suitable with respect to wear resistant characteristics as cutting tool materials.

Table 1. Phase Composition, Density, Crystallite Size and Grain Size of different mol% of Y₂O₃ in Y-PSZ

Composition	Density (%) theoretical)	Crystallite Size (nm)	Phase Composition		Average Grain Size (µm)
			m-ZrO ₂	t-ZrO ₂	
Y-PSZ (0 mol% Y ₂ O ₃)	90.05	32.5	100	0	1.65
Y-PSZ (1 mol% Y ₂ O ₃)	91.35	30.0	96	4	1.50
Y-PSZ (2 mol% Y ₂ O ₃)	93.26	27.1	56	44	1.25
Y-PSZ (3 mol% Y ₂ O ₃)	98.89	24.0	08	92	1.20

Table 2. Density, Hardness, fracture Toughness and Flexural Strength of different mol% of Y₂O₃ in 10 vol% Y-PSZ in α-Al₂O₃

Different mol% of Y ₂ O ₃ in 10 vol% Y-PSZ in α-Al ₂ O ₃	Sintering Temperature (°C)	Bulk Density (g/cm ³)	% Theoretical Density	Hardness HV	Fracture ToughnessK _{IC} (MPa m ^{1/2})	Flexural Strength (MPa)
2.00	1600	4.30	98.4	1600±45	11.10±0.32	473±21.2
2.50	1600	4.40	98.5	1690±24	12.20±0.15	510±16.3
3.00	1600	4.49	98.5	980±35	7.90±0.26	498±18.2

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