

Comparative study among Al₂O₃, ZTA, MgO/ZTA & CuO/ZTA composites in terms of Mechanical Properties

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Alumina (Al₂O₃) ceramics are one of the demanded materials in the current scenario due to its cheaper cost, abundance of availability and combination of high hardness and fracture toughness by reinforcement of yttria stabilized zirconia (YSZ), Magnesium oxide (MgO), and Copper oxide (CuO). The effect of such additives inside alumina ceramics are carried out from last several decades but still there are many more phenomenons that are still inconclusive. So, a comparative study among the said additive in terms of mechanical properties is carried out to reveal the effect of YSZ, MgO and CuO additives inside alumina ceramics. At first, 10 wt. % of YSZ is doped inside the alumina matrix followed by 0.6 wt. % of MgO and 1.5 wt. % of CuO. The developed composites are thoroughly examined in terms of microstructure, bulk density, hardness, and fracture toughness. The results of the microstructure clearly reveal the homogeneous distribution of all additives inside the alumina matrix. A positive effect with YSZ and MgO is noticed on the bulk density and hardness whereas an adverse effect of CuO is observed. The values of fracture toughness are increases with all the additives due to the synergistic effect of transformation toughening and crack bridging phenomenon.

Keywords: Alumina, ZTA, MgO, CuO, Mechanical Properties.

Introduction

In alumina refineries, pure alumina is extracted from bauxite ore using the Bayer process. In this process, crushed bauxite is placed in an autoclave, maintained at high temperature and high pressure followed by clarification, precipitation, washing and finally calcination to produce pure anhydrous alumina. After preparation, pure alumina has a high melting point, more than 2050 °C, and chemically very stable compound. The density of alumina which is in the range of 3.80 to 3.97 g/cm³ depends on the grade of purity. It has high hardness, high strength, high hot hardness, wear resistance, resistance to corrosion, as well as good dielectric properties. These inherent properties of alumina are still attractive to a large number of researchers [1-3]. The major drawback associated with alumina is its high brittleness or low toughness. Hence, the efforts of researchers are directed towards the improvement in ductility along with fracture toughness. In this context, making ceramic composites is one of the best ways to improve the properties of alumina. Therefore, the developed ceramic composites led to a great interest in the research community due to their outstanding

contribution in the improvement of mechanical, thermal and electrical properties. The potential of alumina in various fields have been increased with the advent of new advanced ceramic composites. These advanced ceramic composites consist of two or more compounds that not only improve the intrinsic properties but also make these composites suitable for highly specific applications. For example, silicon nitrides and tungsten carbides were mixed to obtain exceptionally hard material suitable for high-performance cutting tools [4]. Other material developed by mixing of aluminium oxide (alumina) and silicon dioxide to apply in fabrication of integrated circuits ("microchips"). Canikoğlu et al. [5] used alumina balls to synthesize the nano-sized AlN powder that showed superior mechanical properties. However, earlier researchers [6-11] have also found that when yttria-stabilized zirconia added inside alumina ceramics, the toughness of developed composite improves drastically. This improvement is attributed to transformation toughening mechanism. In the year 1975, an article was published by Garvie [12] with heading as "ceramic steel?". This article provided new insight into the field of ceramic research. The research showed the relationship between tetragonal and monoclinic phases of zirconia. The author proved that these phases were responsible for the transformation toughening mechanism which in turn improves the

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mechanical properties of ceramics. In general, the failure of ceramic materials occurs due to the propagation of cracks [13]. The propagation of crack inside the material can be restricted by increasing the energy or stress required for the same. Therefore, the basic mechanisms which enhance the energy or stress required for propagation of cracks are transformation toughening. In the transformation toughening mechanism, the stress-induced effect of metastable phase posed by zirconia particle is most prominent in the vicinity of a propagating crack [14, 15]. Pazarlioglu et al. [16] investigated the effect of yttria on hydroxyapatite/alumina composite in terms of phase stability, mechanical and *in vitro* bioactivity properties. Researchers showed impactful contribution of yttria inside the composites towards improvement of properties. Furthermore, earlier research also illustrated that the formation of larger grain size inside the composites deteriorates the mechanical properties. Hence, to obtain high mechanical properties and homogenize microstructure, it is very essential to control the grain growth of the specimen after sintering. According to Budworth [17], the control of grain boundary movement could be an approach to prevent the larger grain size at the time of sintering. Budworth also found that the mechanism associated to control the grain growth can be affected by second-phase particulate inclusions. These inclusions can mobilize the pores or developed grain-boundary film phase to control the grain growth. Hence, the most promising process to control the abnormal grain growth is the development of grain boundary films which moves along the boundary at a moderate speed. Kim et al. [18] developed 9 mol.% MgO reinforced zirconia further added with 10 mol.% alumina. The researchers demonstrated that the addition of alumina destabilizes the Mg-PSZ due to formation of bigger grains of forsterite (Mg_2SiO_4), and spinel ($MgAl_2O_4$) responsible for decrement in flexural strength and apparent density. The enlargement in grain size could be mitigated by second-phase particulate inclusions like MgO, ZrO_2 , CoO, ZnO and NiO known as grain growth inhibitor inside the matrix of ceramic materials. These additive forms spinel-type compounds having appropriate colours of spinels. The literature also suggested that these metal oxides segregate at the alumina grain boundaries during sintering and behave as grain growth inhibitor inside the matrix of alumina. The segregation of additives at the grain boundaries lowers the energy to form densified composites. Furthermore, the researches dedicated on the CuO added ZTA found an extensive interest for the researchers to use the same (self-lubricating property) in tribological application. It exhibits wide range of potentially useful properties like easily mixed with polarized liquids (i.e., water) and polymers, inert in terms of both chemical and physical properties. These useful properties make its application compatible with ceramics. It is found that CuO behaves

as a soft material when doped in ceramic and significantly enhanced the superplastic deformation of ceramic [19-21]. In this regard Kerkwijk [22] carried out a thorough investigation on different solid lubricants like CuO, ZnO, MgO, MnO_2 and B_2O_3 doped within alumina (Al_2O_3) and yttria-stabilized tetragonal zirconia (Y-TZP) matrix to study the influence of solid lubricant on the mechanical and tribological behaviour of the developed composites. The investigation clearly showed that CuO gives the best tribological properties among several metal oxides. The Sanusi et al. [23] successfully showed structural applications of alumina ceramic-steel composite. Later, Shahid et al. [24] showed the application of alumina composite in the armor industries.

Hence, the above studies revealed the effect of YSZ, MgO and CuO inside alumina matrix could solve the problem of high brittleness. So, an indeed comparison among the additive has been required to postulate the effect on alumina. To fulfill this, an attempt has been made that explore all possible phenomenons responsible for the improvement in mechanical properties by reinforcing additives inside the alumina matrix novel aspect of this study. So, at first ZTA composites is fabricated by adding alumina and YSZ in ratio of 90:10 respectively. Furthermore, 0.6 wt. % MgO was added inside ZTA followed by mixing of 1.5 wt. % CuO. The mechanical properties of developed composites are thoroughly investigated to explore a comparative study.

Methodology Opted to Prepare Composites and Experiments

In this investigation, precipitation and co-precipitation synthesis route were selected to prepare alumina and yttria stabilized zirconia (YSZ) respectively. The alumina ceramics were prepared by taking requisite amount of nitrite salts (Mark, India 99.9% purity) dissolve in distilled water. The dissolved salts were further treated with ammonia solution (0.1 Mol.%) to form the precipitates. In case of YSZ, yttria nitrate and zirconium oxychloride (Mark, India 99.9% purity) salts were dissolved in distilled water followed by mixing of ammonia solution (0.1 Mol.%). The formed precipitates were segregated on a filter unite using whatman filter paper. The segregated precipitated were thoroughly washed with warm water to remove chemical impurities. The washed precipitate was kept in an oven for next 24 h for drying. The dried lumped were crushed and calcined at temperature of 800 °C followed by ball milling process on a planetary ball mill for 8-10 h at 300 rpm. After preparation of alumina and YSZ ceramics, physical mixing process was opted to prepare the composites. In physical mixing process the requisite amount of powders were taken in a zirconia jar having 500 ml capacity. The powders were kept in alcoholic medium along with alumina balls having 8-12 mm diameter as a media. At first, 50 g Zirconia

toughened alumina (ZTA) composites were prepared by taking 45 g of alumina and 5 g of YSZ. The said amount of powders was kept in a zirconia jar mixed with methanol and 120 g of alumina balls. After placing the zirconia jar was closed and placed in a planetary ball mill for next 8-10 h. After proper mixing in planetary ball mill, automatic mechanical mixing systems were used for next 6 h to get properly blending of composites. The blended composites were kept in an oven followed by crushing process. After crushing ZTA powders were ready for use. Similarly, MgO and CuO reinforced composites were prepared through physical mixing process. The amount of MgO and CuO were selected on the basis of author earlier work [25, 26] that provide the optimized concentration of powders. In this case also the ball milling and automatic mechanical blender were used to fabricate the composites having 0.6 wt.% MgO/ZTA and 1.5 wt.% CuO/ZTA composites. The prepared powders of composites were calcined at a temperature of 800 °C with a soaking time of 2 h that remove all moisture and impurities. After blending, the requisite amount of the calcined powders was placed inside a die. The die and punch were cleaned thoroughly before pouring the powders. The die filled with powder was placed on hydraulic press. A pressure of 10 ton/cm² for 1 min as holding time was used to press the powders with help of punch. After compaction the samples were slowly ejected from the die with special care. After removal the samples were placed on a ceramic plate and kept inside the furnace for sintering. In case of CuO/ZTA a special arrangement was used i.e. instead of plate a ceramic crucible was used in which the samples were kept and completely filled with alumina powder. This arrangement was due to better sintering and restraining the flow-ability of CuO particles inside the matrix (liquid phase sintering). In this investigation, the sintering temperature was maintained as 1500 °C with holding time of 1 h. After sintering, the samples were cooled inside the furnace till the temperature of samples not reached room temperature. After cooling the samples were mirror polished using silicon carbide powder and Bain polisher. A semi-finishing polishing was carried out with different sizes of silicon carbide process obeying honing/lapping. After semi-finishing operation the fine polishing was carried out on a Bain polisher using diamond paste in kerosene medium. After polishing the samples were used for mechanical properties evaluation.

At first, sintered samples were used to investigate the microstructure on Field emission scanning electron microscopy (FESEM) instrument. The microstructure of all developed composites was used to analyze the uniform distribution of additive inside the alumina matrix. After microstructural investigation, bulk densities of all developed composites were carried out through Archimedes principal. The principle was exclusively described with all the empirical relation earlier by

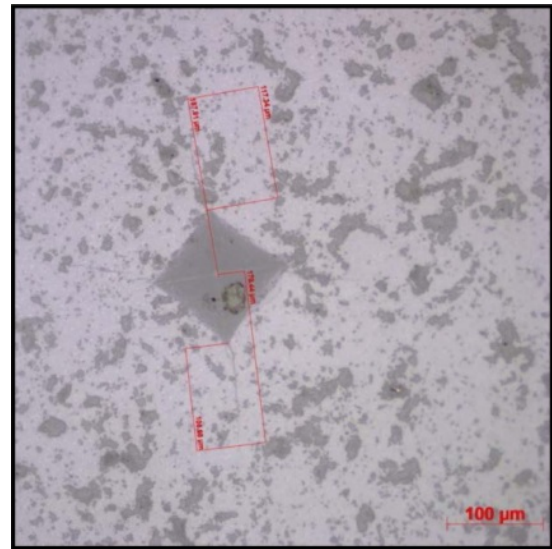


Fig. 1. Systematic representation of measuring parameter to evaluate Fracture Toughness.

Singh et al. [27]. After evaluation of bulk densities the hardness and fracture toughness of all developed samples were carried through Vickers hardness tester. A holding time of 10 sec at a load of 1 Kgf was used to investigate the hardness test. A pyramidal shaped indenter, in which apex edges are meeting at 136° was used to penetrate the sample for to investigate the hardness. After evaluation of hardness the fracture toughness was carried out at 2 Kgf load. The methodology and empirical relation opted to evaluate the fracture toughness (K_{IC}) (MPa·m^{1/2}) was shown through equation (1) [27].

$$K_{IC} = 0.16(c/a)^{-1.5} (Ha^{0.5}) \quad (1)$$

In the equation, c (microns) represents the average length of crack shown in Fig. 1 whereas a (microns) is the half average length of the diagonal. H (MPa) and P (Newton) are the hardness of the samples and measuring load respectively.

Experimental Results and Analysis

At first, morphological study based on FESEM images has been carried out for all the developed composites. The FESEM images are taken at same magnification for alumina, ZTA, MgO/ZTA and CuO/ZTA as shown in Fig. 2(a), (b), (c) and (d) respectively. The analysis suggested that the additives are uniformly distributed inside the cluster and have significant influence on the grain growth which in turn reflects on the mechanical properties.

Morphological Analysis

From Fig. 2(a), (b) and (c), it has been observed that the average grain sizes of alumina ceramics have larger than ZTA and MgO/ZTA composites. The earlier

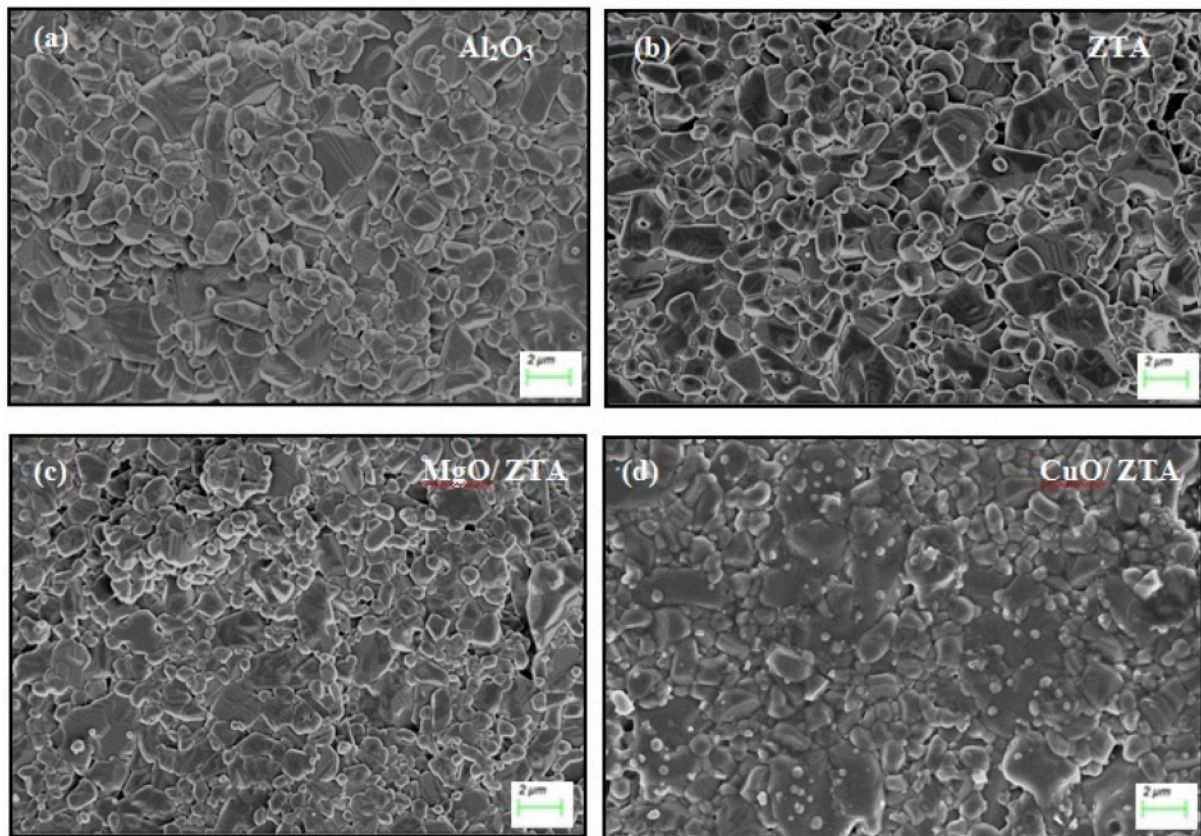


Fig. 2. (a) FESEM image of Alumina (b) FESEM image of ZTA (c) FESEM image of MgO/ZTA (d) FESEM image of CuO/ZTA.

studied dedicated on YSZ [28] and MgO [29] as additives inside the alumina matrix acts as a grain growth inhibitor that restrict the growth of alumina grain. Especially, the grain growth phenomenon inside alumina is highly influenced by MgO particles. The results are in agreement with Berry et al. [30] that described that the surface-diffusion-controlled pore drag during sintering was predominantly effective for grain growth. It was found that the presence of magnesia restricts the grain growth rate by a factor of 2.5 during the sintering process. Hence, the restriction in the grain growth rate mechanism was attributed to the surface-diffusion-controlled pore drag or due to rise in the pore mobility rate, enhanced by the presence of magnesia particle inside the composite. A similar kind of observation was also demonstrated earlier by Haroun [31]. The researchers explained that without any sintering aid, the pores exerted a decelerating effect on the grain boundary migration resulted in abnormal grain growth during the sintering process. But these pores were stable and uniformly moved on all grain boundaries during migration due to the presence of magnesia particles inside the matrix. The effects of pore mobility were easily recognized from the microstructure analysis of MgO/ZTA. Furthermore, the microstructure of CuO/ZTA clearly showed a reverse

morphology that the alumina grains are increased with reinforcement of CuO particles. The results are in agreement with the author earlier research Singh et al [32].

It was found that the CuO particles didn't show any effect on the pores mobility rate due to liquid phase sintering. The liquid-phase sintering exaggerated the formation of CuAlO_2 having larger grain and provide soft nature particles. Hence, the grain sizes of the CuO/ZTA has large size compare to other two composites. So, it can be concluded that the MgO particles have high influence on the grain growth compare to YSZ and CuO inside alumina matrix. Furthermore, the interpretation observed from the XRD plot shown in Fig. 3(a), (b), (c) and (d) justified the conclusion. From Fig. 3(b) and (c) the exaggeration in the retention of metastable phases of zirconia (111) at the cost of monoclinic phases (11-1) is clearly observed, in agreement with Raja et al. [33]. The light percentage (beyond the identification limit of XRD) and homogeneous mixing of MgO and CuO particles make difficult to identify through XRD spectra. However, the stable phases are observed for the MgO and CuO reinforced alumina matrix. For CuO doped ZTA no phase transformation is observed which is in accordance with Singh et al. [26].

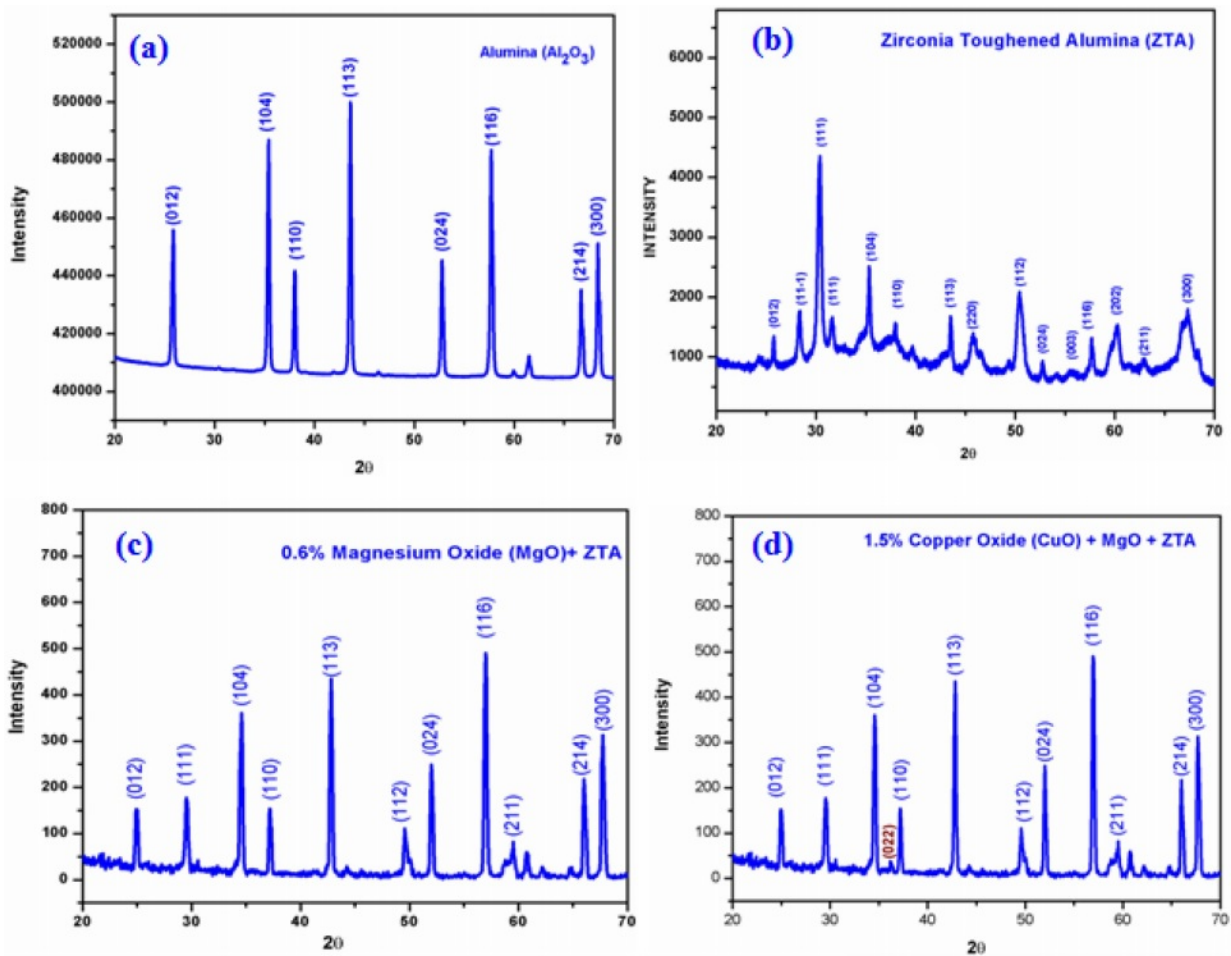


Fig. 3. (a) XRD plot of Alumina (b) XRD plot of ZTA (c) XRD plot of MgO/ZTA (d) XRD plot of CuO/ZTA.

Bulk Density Analysis

After micro-structural analysis the mechanical properties in terms of bulk density, hardness and fracture toughness of all developed composites are evaluated and summarized in Table 1.

From Table 1 an improvement in bulk density is observed with reinforcement of YSZ and MgO, whereas, decrement with CuO particles. A low hardness is observed in case of YSZ as the Ytria and zirconium particles are softer than alumina particles. But when the combination of Ytria and Zirconia i.e. yttria stabilized zirconia (YSZ) present inside the alumina matrix acts as a grain growth inhibitor. The restriction in grain

growth is also seen through micro-structural analyses that have influence on the development of porosity inside the cluster. The restriction in grain growth influenced the bulk density, consequences as maximum density for MgO reinforced alumina matrix. The above section showed dire effect of MgO on the grain growth influenced by pore mobility. The lower grain size creates low porosity inside the cluster results in high bulk density. In case of CuO/ZTA the formation of larger grain size clearly seen from micro-structural images develops high porosity inside the cluster shows a vital role in the decrement of bulk density. The result is in agreement with Singh et al. [32] that also

Table 1. Evaluated Results of Mechanical Properties.

Composites	Bulk Density ($g \cdot cm^{-3}$)	Hardness (GPa)	Fracture Toughness ($MPa \cdot m^{1/2}$)
Alumina (Al_2O_3)	4.18±0.16	17.68±0.58	3.68±0.21
Ytria Stabilized Zirconia (YSZ)	3.89±0.19	15.03±0.43	8.04±0.28
Zirconia Toughened Alumina (ZTA)	4.21±0.21	17.91±0.48	4.28±0.14
Zirconia Toughened Alumina (ZTA) + 0.6 wt. % Magnesium Oxide (MgO)	4.23±0.24	18.02 ±0.59	4.42±0.16
Zirconia Toughened Alumina (ZTA) + 1.5 wt. % Copper Oxide (CuO)	4.08±0.20	17.67±0.40	4.98±0.18

suggested that the incorporation of CuO particles inside ZTA matrix provide an exaggeration in the growth of grain due to inferior effect on pore mobility.

Hardness and Fracture Toughness Analysis

From Table 1 it can be observed that the YSZ have minimum hardness but maximum fracture toughness, but when YSZ incorporated inside the alumina matrix the hardness of alumina ceramics increases. The improvement in hardness of the alumina matrix is attributed to the transformation toughening phenomenon allied with YSZ matrix. The transformation toughening phenomenon was elaborately explained by Singh et al. [32] earlier work. Researchers disclosed that zirconia particles exist in monoclinic phase at the room temperature, but when heated to a temperature of 1200 °C it transforms into tetragonal phase and vice versa on the cooling. The research showed that the tetragonal particles of zirconia can be trapped at room temperature due to presence of Ytria and Magnesium particles present near to the tetragonal zirconia particles. The trapped zirconia particles are known as metastable tetragonal phase of zirconia inside the cluster. This metastable tetragonal phases of zirconia have unique characteristic of expansion around ~3-4% when it return back to monoclinic phase at a critical stress induced inside the cluster. It is found that when any crack initiated at the thresholds value inside the cluster and starts propagating results in failure of materials. The presence of metastabilized zirconia particles creates obstacles during propagation of crack by increasing its volume to some extent. The restriction in the propagation of crack result in the increment of hardness and fracture toughness as observed during examination of hardness and fracture toughness for ZTA as well as MgO doped ZTA. The said mechanism is known as transformation toughening mechanism. Hence, it can be illustrate that the presence of YSZ and MgO particles provide a beneficial effect on hardness and fracture toughness due to transformation toughening phenomenon accompanied with the small grain sizes of alumina [34, 35].

An interesting result is observed in case of CuO/ZTA i.e. hardness decreases and fracture toughness increases. The decrement in hardness is attributed to the impurity phases created by CuO particles at the grain boundary. The earlier research carried out by Ramesh et al. [36] and Ran et al. [37] showed that the presence of Cu at grain boundary developed Cu-rich phase inside the cluster. This Cu-rich zone acts as an impurity, provides no obstacle in crack propagation result in little inferior hardness. The earlier analysis also suggested that in case of CuO/ZTA the transformation toughening mechanism was completely obsolete. The microstructural investigation of CuO/ZTA composites shows bigger grain sizes, can also affect the hardness of the composite. So, due to impurity phase created at

the grain boundary alongside bigger size of grains results in low value of hardness for CuO/ZTA composites. In adverse, improvement in fracture toughness is observed in case of CuO/ZTA. This improvement is attributed to the crack bridging and bigger size of grains present inside the cluster. The bigger size crack forms larger intergranular cracks [38, 39] that further require high energy towards propagation of cracks consequences in high fracture toughness. It was also found that the larger porosity due to larger grain absorbed more local residual stress and emits the stress in form of acoustic wave results in the improvement of fracture toughness and the phenomenon is known as crack bridging phenomenon. So, it can be concluded that the effect of impurity phases decreases the hardness but crack bridging and bigger size grains improves the fracture toughness.

Conclusion

Homogeneous composites of YSZ, ZTA, MgO/ZTA and CuO/ZTA has been successfully developed to see the affect of YSZ, MgO and CuO inside alumina ceramic in terms of microstructure and mechanical properties. The micro-structural analysis reveals decrement in grain growth with incorporation of YSZ and MgO whereas, CuO shows adverse affect. Discussion reveals that the pore mobility rate inside the cluster plays major role towards development of grain growth. The bulk density shows improvement with incorporation of YSZ, MgO and decrement with CuO. This attainment of such result dedicated to the development of grain size. Furthermore, the contribution of transformation toughening phenomenon and crack bridging mechanism alongside grain size are predominant on the value of hardness and fracture toughness. The Cu-rich zone developed at the grain boundary with incorporation of CuO particles creates an impurity phases, consequences in unfavorable affect on the hardness of CuO/ZTA. The metastable tetragonal phases retain at room temperature due to presence of YSZ and MgO direly effect the transformation toughening phenomenon have direct affect on the value of toughness.

Acknowledgement

Authors heartily thanks to director and principal of Sir Eshwar college of Engineering for inconsistent support towards completion of this work.

Funding:

No fund received

Conflicts of interest/Competing interests:

No conflict between the authors.

Availability of data and material:

All data are presented in the manuscript

Authors' contributions:

G. Gokilakrishnan: Formal analysis, Methodology, Supervision Bipin Kumar Singh: Experimentation, Validation, Visualization, Vigneshkumar M: Write-up, Visualization

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