The effect of transition metal oxide doping on the sintering behaviour of yttria-stabilised tetragonal zirconia

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The effect of small amounts (0.2 wt% and 0.5 wt%) of transition metal oxide (CuO) doping on the sintering behavior of 3 mol% yttria-stabilized zirconia (Y-TZP) was investigated over the temperature range of 1250 °C to 1500 °C. Sintered samples were characterized to determine the phase present, relative density, microstructural evolution and Vickers hardness. The studies revealed that the addition of 0.2 wt% CuO dopant was most effective in enhancing the densification of the Y-TZP in particularly at low temperatures below 1350 °C. The phase analysis revealed that the tetragonal phase was disrupted as evident from the high monoclinic phase formation in the 0.5 wt% CuO-doped zirconia. This phenomenon was associated with mechanism involving transient liquid phase which believed to have destabilized the tetragonal structure. Nevertheless, the study revealed that compared to the undoped Y-TZP, the sample with addition of 0.2 wt% CuO resulted in enhanced hardness and finer grain sizes when sintered at relatively low temperatures.

Key words: Y-TZP, Copper oxide, Doping, Sintering additives, Vickers hardness.

Introduction

Yttria-stabilized tetragonal zirconia polycrystals or Y-TZP is known to exhibit attractive properties such as high fracture toughness, strength and good wear properties suitable for many industrial and biomedical applications. This has been attributed to its unique 'self-healing' mechanism known as transformation toughening. In this mechanism, the energy of an advancing crack through the matrix is absorbed by the tetragonal grains in the vicinity of the crack front and transform to a monoclinic symmetry. This phase transformation is accompanied by 3-5% volume expansion which induced a local compression surrounding the crack tip; as a result more energy would be required for crack propagation and hence enhancing the strength [1-3].

The widespread use of Y-TZP, however is hampered by the undesirable tetragonal to monoclinic phase transformation when exposed to steam environment at temperatures 60 °C to 500 °C, a phenomenon known as hydrothermal ageing or low temperature degradation (LTD) [4-8]. This devastating effect of zirconia was initially observed by Kobayashi et al. [4], and the ageing process was documented to induce formation of networks of micro- and macro-cracking, thus leading to

deterioration of mechanical properties. According to this study, when the Y-TZP was exposed to humid environment at temperatures between 150 °C and 400 °C, isolated tetragonal grains or nuclei at the surface begin to transform to the monoclinic symmetry by incorporating the hydroxyls in the oxygen vacancies of the zirconia lattice. This induces localized residual stresses which subsequently destabilized the tetragonal structure [7-10].

A nucleation and growth mechanisms have been proposed to explain the ageing kinetics although a consensus on the nature of the transformation in the interior of the sample i.e. whether the transformation proceed linearly or exponentially with time and/or reached a saturation limit after a certain depth have not been unequivocally resolved [11-15]. Nevertheless, factors such as grain sizes and the chemistry of the grain boundaries have been associated with the LTD phenomenon [16-19]. For instance, Hallmann et al. [17] reported that Y-TZPs with average grain size less than 0.3 µm was resistant against the ageing-induced phase transformation. The authors also found that grain boundary modification using dopants such as alumina, ceria and iron oxide were beneficial in suppressing the LTD in Y-TZP. Zhang et al. [19] investigated the influence of 0.25 wt% alumina addition and incorporating yttria coating of zirconia starting powder on the LTD behavior of the ceramics when exposed in an autoclave containing steam at 134 °C and 0.2 MPa up to 40 h. Their TEM analysis revealed that the Y/Zr ratio of the

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yttria coated zirconia was higher at grain boundary regions when compared to the grain core. Zhang et al. suggested that the improved ageing resistant observed for their Y-TZP was attributed to both, the segregation of Al^{3+} and the heterogeneously distributed of Y^{3+} at the grain boundaries.

Improvement in the sintering conditions has been demonstrated in the literatures to be viable in order to improve the densification and properties of Y-TZP as well as to control the LTD in zirconia. This includes using non-conventional sintering processes and the manipulation of the sintering conditions such as field-assisted sintering [20], hot-isostatic pressing [21], spark-plasma sintering [22], cold sintering [23], two-step fast firing [24], microwave sintering [25] and laser sintering [26]. All these methods have shown to be effective in enhancing the properties of zirconia, however this improvement is at the expense of high cost associated with the equipment used for powder consolidation. A more economical approach would be to aid the conventional sintering process using low melting sintering additives or dopants, added to the zirconia during powder processing and subjecting the doped powders to conventional sintering at low temperatures. This strategy has been demonstrated to yield highly dense bodies in several ceramic systems [27-33].

The present work aims to examine the effect of small additions of transition metal copper oxide (0.2 wt% and 0.5 wt%) on the mechanical properties and microstructural development of Y-TZP ceramics prepared by the pressureless sintering method.

Experimental

In the present work, 3 mol% yttria stabilized zirconia powder from Kyoritsu Japan was used and the different amounts of high purity CuO (0.2 wt% and 0.5 wt%) were mixed in an attritor-mill contained zirconia beads (1 mm in diameter) as the milling media and ethanol as the mixing medium. The speed of the rotation was kept constant at 600 rpm and each milling was carried out for 30 mins. After the mixing process, the slurries were separated from the milling media by filtration and then dried overnight in an oven at 60 °C. Finally, soft, ready to press powders, were obtained by sieving through 212 µm mesh sieve. For the study, green disc samples (20 mm diameter, 5 mm thick) were uniaxially compacted using a metal mold and subsequently subjected to cold isostatic pressing at 200 MPa.

Powder consolidation was accomplished by pressureless sintering under ambient conditions in a box furnace. The sintering was carried out at different temperatures ranging from 1250 °C to 1500 °C at a ramp rate 10 °C/min and holding time 2 h. The sintered samples were ground successively on one face by using silicon carbide paper of 120 (rough), 240, 600, 800 and 1200 (fine) grades, followed by polishing using 6 µm and 1 µm

diamond paste to obtain an optical reflective surface.

The bulk densities of sintered samples were determined by the water immersion method based on the Archimedes principle, and the relative densities were calculated by taking the theoretical density of tetragonal zirconia as 6.07 g/cm^3. Vickers Hardness (H_v) was measured on polished samples using Vickers indention method. The indention load was kept constant at 10 kgF with a loading time of 10 s. Phase analysis was conducted by X-ray diffraction (XRD) (Geiger-Flex, Rigaku Japan) under room temperature conditions using Cu-Kα as the radiation source. The fraction of monoclinic phase was determined using the method of Toraya et al. [34]. The effect of temperature on the microstructure evolution of the ceramic was examined by using scanning electron microscope (SEM). The average grain size of the polished samples was determined by using the line intercept method [35].

Result and Discussion

A typical XRD trace of the undoped and CuO-doped Y-TZP sintered at 1400 °C is shown in Fig. 1. A similar trend was observed at all temperatures. The undoped and 0.2 wt% CuO doped zirconia exhibited a fully tetragonal structure, however, the XRD analysis indicated that the addition of 0.5 wt% CuO has disrupted the tetragonal phase stability of the Y-TZP throughout the sintering regime employed. A high percentage of monoclinic phase was measured for the 0.5 wt% CuO addition. It is believed that the addition of high amounts of dopant could have developed excessive transient liquid phase during sintering which caused the depletion of yttria stabilizer from the zirconia lattice at around
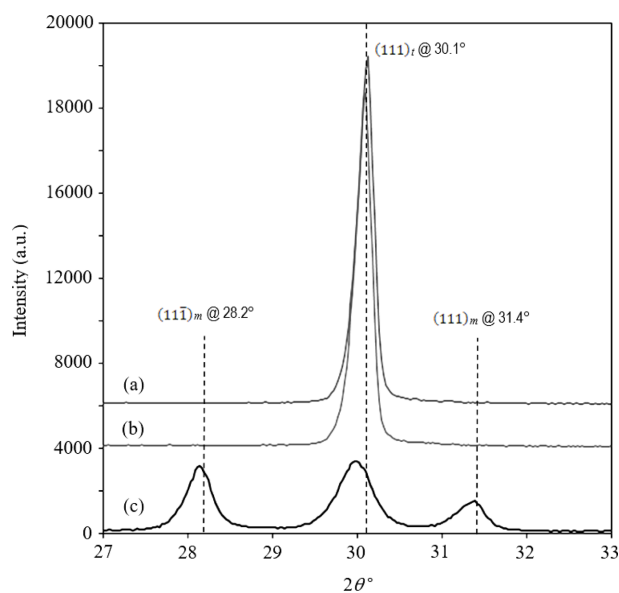

Fig. 1. XRD trace of Y-TZPs sintered at 1400 °C: (a) undoped, (b) 0.2 wt% CuO and (c) 0.5 wt% CuO. Note the shifting of the peaks resulting from copper doping.
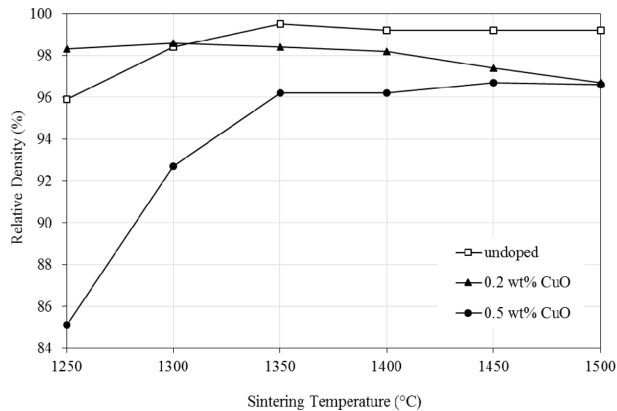
Fig. 2. The effect of sintering temperature on the relative density of Y-TZP.

1180 °C and on cooling destabilised the tetragonal structure [36, 37]. It was determined that the monoclinic phase content for the 0.5 wt% CuO-doped zirconia samples varied between 50 to 55% as the sintering temperature increased from 1250 to 1500 °C. In addition, the tetragonal peaks at $2\theta = 30.1°$ for this sample shifted slightly to the left by about 0.10-0.13° when compared to the undoped zirconia as shown by the inset picture in Fig. 1. This is an indication that the high amount of CuO had affected the stability of the tetragonal zirconia lattice.

The effect of sintering temperature on the relative density of undoped and CuO-doped zirconia is shown in Fig. 2. The result shows that the addition of 0.2 wt% CuO was beneficial in enhancing the density of Y-TZP sintered below 1350 °C. This sample exhibited > 98% relative density when sintered at 1250 °C when compared to 96% for the undoped zirconia. However, the relative density of the undoped Y-TZP started to increase and maintained above 98% as the temperature raised to 1350 °C and above. In contrast, the 0.5 wt% CuO doped Y-TZP exhibited the lowest density regardless of sintering temperature. It started low at 85% dense @ 1250 °C and increased steadily to 96% at 1350 °C and then remained almost constant with further increased in temperatures. This observation is in good agreement with the high monoclinic content observed for the sintered body.

The beneficial effects of transition metal oxides such as copper oxide and manganese oxide (MnO_2) in promoting densification and improvement in other properties were also observed in other ceramic systems such as alumina [38], cadmium [39] as well as SnO_2 [40] and ternary based ferroelectrics [41]. It is known that ceria and the tantalates are not easy to densify at moderate temperatures without sintering at elevated temperature above 1500 °C. Shimada et al. [42] demonstrated that 3 wt% MnO-doped $LiTaO_3$ promoted densification at low temperatures of 1125-1190 °C. They obtained a relative density of 85-90% when compared to 60-62% for the undoped ceramic but at

the expense of grain coarsening. The authors believed that the enhancement in densification in these ceramics could be linked to the reactive Mn-based liquid formation at low temperatures. In another research, Corker et al. [43] studied the effect of CuO doping on the sintering of PZT and they found the addition of 3 wt% of Cu_2O/PbO with the eutectic ratio of Cu_2O : PbO (1 : 4) was effective in reducing the densification temperature considerably by about 67%, from 1260 °C down to 850 °C. The authors attributed this remarkable improvement to the presences of a Cu_2O-PbO eutectic liquid at about 680 °C. This finding was supported by TEM studies [44] which showed the formation of a CuO-PbO rich grain boundary phase at 900 °C and the crystallization of nanosized CuO precipitates near grain boundary regions after sintering.

Zhang et al. [45] investigated the effect of transition metal oxide (MnO_2) doping in ceria and found that the addition of up to 1.5 wt% MnO_2 accelerated the densification rate of ceria when sintered at low temperatures. They went on to suggest the formation of a very thin Mn-amorphous layer during heating could have facilitated particle rearrangement thus leading to rapid consolidation during the early-stage of sintering. Similar observation on the effect of manganese oxide in aiding the sintering of zirconia was also reported in the literatures [46, 47]. These studies concur that the presence of a reactive liquid phase involving the transition metals during sintering was responsible for the improvement in sintered density. More recently, Watson et al. [41] concluded that CuO actively reacted with PbO to form a reactive eutectic liquid phase during sintering of PIN-PMN-PT ceramics, which reduces the activation energy for sintering to proceed at temperatures as low as 790 °C and hence retaining a fine grain microstructure on cooling. There have been different views on the sintering mechanism involving CuO as additive. For instance, Nie et al. [48] who studied the effect of CuO on the sintering of TiO_2 ceramics inferred that the enhancement in densification of CuO-doped TiO_2 at low temperatures was due to sub-eutectic activated sintering, rather than liquid-phase sintering.

The fact that the melting point of copper oxide (1150 °C) is lower than the sintering temperature employed in the present work and based on the literature findings as well as the results obtained, it is envisaged that a transient liquid phase mechanism was in operative. The results indicated that the addition of 0.2 wt% CuO was the optimum amount required to form sufficient reactive Y_2O_3/CuO-rich transient liquid phase which facilitated particle rearrangement and coalescence at low temperatures, below 1350 °C. As the doping level increased to 0.5 wt%, the excess amount of Cu-rich liquid proved to be detrimental as this has an effect to draw out too much of yttria from the zirconia matrix, resulting in phase transformation upon cooling from sintering as evident from the XRD analysis. This

tetragonal to monoclinic phase transformation was accompanied by micro-and macro-cracking observed on the free surface of the samples as typically shown in Fig. 3.

The effect of sintering and dopant addition on the average grain size of Y-TZP is shown in Fig. 4. The general trend is that the grain size increases with increasing sintering temperature for all samples. The
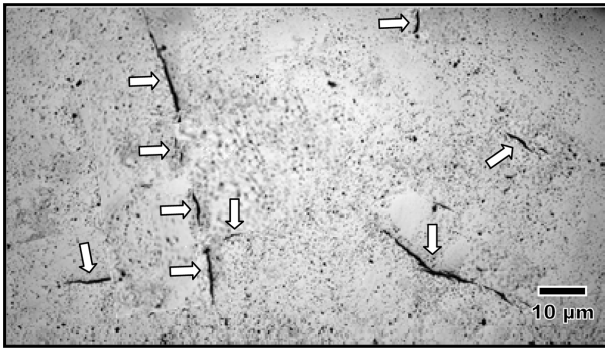

Fig. 3. Typical optical view of the free surface of the as-sintered 0.5 wt% CuO-doped Y-TZP revealing the presences of micro- and macro-cracks (as indicated by the arrows) associated with the tetragonal to monoclinic phase transformation upon cooling from sintering.
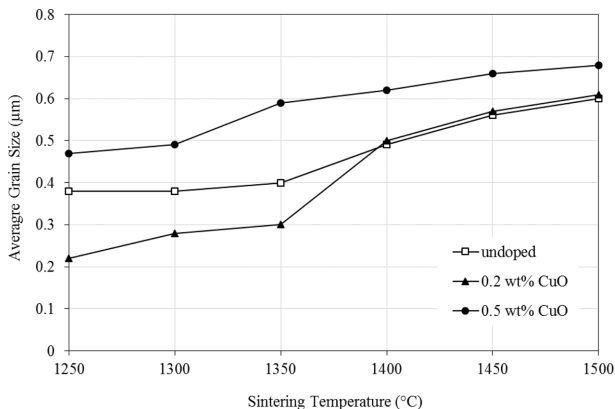

Fig. 4. Average grain size variation as a function of sintering temperature and CuO doping.

results showed that the 0.5 wt% CuO exhibited larger grain size when compared to the 0.2 wt% CuO and undoped samples for all temperatures investigated. The grain size of the 0.2 wt% CuO-doped zirconia was smaller (below 0.3 μm) than the undoped when sintered at 1350 °C and below.

A typical SEM microstructure of the samples sintered at 1450 °C is shown in Fig. 5. An equiaxed grain morphology was observed for all samples regardless of dopant additions. The general observation made was that the grain size increased with increasing CuO content. This phenomenon of grain coarsening has also been noted by other researchers. For instance, the effect of copper oxide doping on the electrical and micro-structural properties of SnO_2-based varistors (SCNCr) was studied by Mahmoudi et al. [40]. The authors found that the grain growth kinetics of the ceramic increased when the doping level exceeded 0.25 mol%. This grain growth was accompanied by a reduction in the activation energy from 594 kJ/mol (undoped) to 364 kJ/mol (0.25 mol% CuO-doped). They suggested that a solute drag force was the controlling mechanism of grain growth for samples with a low CuO content (< 0.25 mol%) and this growth mechanism changed to the Sn^{4+} solution-precipitation in CuO-rich liquid phase for higher CuO doping. On the contrary, Kim et al. [49] who studied the effect of CuO on the properties of $(K,Na,Li)(Nb,Ta)O_3$ ceramics showed that the exaggerated grain growth observed for the CuO-doped ceramics could be avoided by manipulating the sintering para-meters. The authors initially performed normal sintering at temperatures above 900 °C and obtained an in inhomogeneous microstructures, consisting of a bimodal distribution of small (several microns) and exaggerated grains (20-40 μm). However, when a two-step sintering (920-930 °C for 0 h in the first step and 880 °C for 1-12 h in the second step) was used, this resulted in a homogeneous fine-grained microstructure without com-promising on the piezoelectric properties of the ceramics.

The variation in Vickers hardness as a function of sintering temperature and dopant addition is shown in Fig. 6. The results show that both the 0.2 wt% CuO-
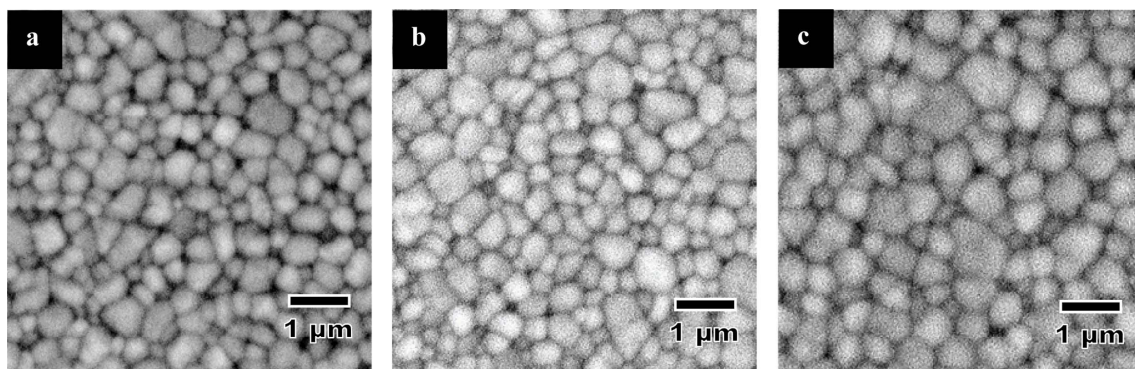

Fig. 5. Typical SEM microstructure of Y-TZPs sintered at 1400 °C: (a) undoped, (b) 0.2 wt% CuO and (c) 0.5 wt% CuO doped samples, respectively.
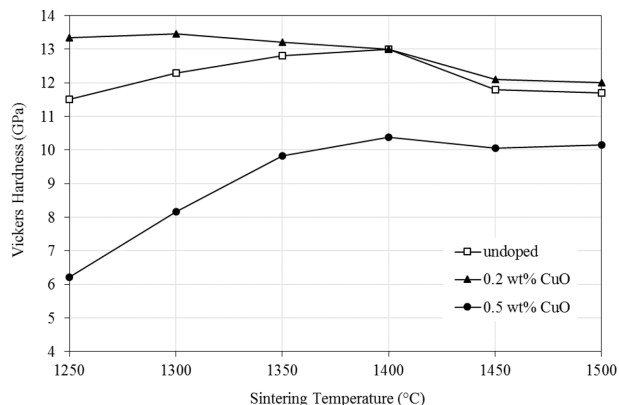
Fig. 6. The effect of sintering temperature on the Vickers hardness of Y-TZPs.

doped and undoped zirconia exhibited a similar trend with increasing temperature. However, in all cases, the hardness of the 0.2 wt% CuO-doped was higher than the undoped ceramic and this agrees with the density trend. The hardness of the 0.2 wt% CuO-doped zirconia was > 13 GPa when sintered at 1350 °C and below. Similar observation on the effect of CuO in enhancing the mechanical properties of Sm-doped CeO_2 ceramic was reported by Lu et al. [50]. The researchers found that the addition of CuO lowers the densification sintering temperature and enhances mechanical strength. They also noted that the hardness dramatically increases from 4.9 ± 0.5 to 8.8 ± 0.4 GPa after doping with only 0.5 mol% CuO which was attributed to a fracture transformation from intergranular to transgranular, as well as enhanced density. In the present work, however, the hardness of the 0.5 wt% CuO was low regardless of sintering temperature. The sintered body attained a maximum value of about 10 GPa when sintered at 1500 °C. This was expected due to the lower bulk density as well as the high monoclinic phase content measured in the sintered bodies as discussed earlier.

Conclusions

In this present work, the effect of small amounts of transition metal oxide i.e. CuO addition as a sintering aid on the densification and properties of Y-TZP ceramics were investigated. The results revealed that the addition 0.2 wt% CuO was beneficial in aiding densification with improved hardness when sintered at low temperatures, below 1350 °C when compared to the undoped ceramics. The tetragonal phase stability was also not affected by the 0.2 wt% doping and the sintered grain sizes were below 0.3 μm when sintered at lower temperature regime. In contrast, the addition of 0.5 wt% CuO was found to be detrimental as this excess liquid formation during sintering has disrupted the tetragonal phase stability and resulted in lower hardness and low relative density. This phase transformation on cooling to room temperature was also

accompanied by micro- and macro-cracking thus rendering the material unfit for application. Nevertheless, this study has shown the viability of aiding low temperature sintering of Y-TZP through doping with low amounts of copper oxide.

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