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ORIGINAL ARTICLE

Effects of di-ammonium citrate: polyvinylpyrrolidone as a complexion dispersants on 3Y-TZP slurries and sintered body properties

Pestaria Sinaga[1] · Sung-Hwan Bae[2]

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Abstract

A wide application of 3 mol% Yttria-stabilized Tetragonal Zirconia Poly-crystalline (3Y-TZP) makes the concern of ceramic processing required to produce strong and reliable engineering ceramic products. In this study, we successfully improved the 3Y-TZP mechanical properties by paying great concern to the early processing of 3Y-TZP powder. Here, we used the concept of colloidal processing by manipulating the 3Y-TZP inter-particle force as a combination of steric and depletion stabilization. Di-ammonium citrate (DAC) was used to induce steric stabilization utilizing the DAC's carboxyl group to strongly bond with the OH group of the 3Y-TZP. While Polyvinylpyrrolidone (PVP) which could not bond with 3Y-TZP, leaves it freely as an un-attached polymer in slurries and induces a depletion stabilization mechanism. Using the combination of steric and depletion stabilization, the YSZ slurries stability is improved, and a uniform and smaller particle size can be obtained. As the result, better microstructure and high mechanical properties of the sintered body, such as higher density and Vickers hardness, can be achieved. By comparing to the sintered body of 3Y-TZP containing just DAC and 3Y-TZP containing a combination of DAC and PVP, better-sintered body properties can be found for the 3Y-TZP containing the combination of DAC and PVP, and the results can be listed as follows: grain size reduced from 0.420 to 0.281 um, density is increased from 94.5 to 99.3%, and Vickers hardness increases from 1190 to 1305 HV.

Keywords 3Y-TZP · Steric stabilization · Depletion stabilization · Di-ammonium Citrate (DAC) · Polyvinylpyrrolidone (PVP)

1 Introduction

3 mol% Yttria-stabilized Tetragonal Zirconia Polycrystalline (3Y-TZP), which is zirconia partially stabilized with the addition of 3 mol % of yttria, is one of the most candidate ceramic materials in engineering ceramic application. The excellent mechanical properties of 3Y-TZP enabled their wide applications, such as body implants, dental crowns, fixed dental prostheses, engineering ceramics, ceramic crown restorations, and ceramic knives as well as ceramic balls, ceramics screws, etc. [1–5]

For the above-mentioned wide applications of 3Y-TZP, fine-grained and uniform microstructures of the products are required to produce strong and reliable structural parts [6]. However, to obtain a fine-grained and uniform microstructure of ceramics, careful powder processing to break up the agglomeration is highly necessary. A colloidal method is one of the promising powder processing which can be used to break the agglomeration and obtain a small and narrow size distribution of the dispersed phase in the dispersion medium [7–10]. A small and uniform particles size without agglomeration forming can be obtained by controlling the stability of the slurry in colloidal methods as called colloidal stabilization.

There are several types of colloidal stabilization, such as steric, electrostatic, electro-steric and depletion stabilization. In steric stabilization, some functional groups of the polymer can interact with the surface of the particles and act as a good anchor, especially carboxyl group turns out to be the most effective one because they are supposed to interact strongly with the OH sides that are often present on the surface of the particles. [11]. The electrostatic stabilization can be obtained by the addition of ion charge to the oxide

✉ Sung-Hwan Bae
shbae@kyungnam.ac.kr

1 Department of Mechatronics Engineering, Kyungnam University, Changwon, Korea

2 Materials Science and Engineering, Kyungnam University, Changwon, Korea

particles surfaces, so the particles will repel each other, particularly under the pH control. Further, another stabilization as a combination of steric and electrostatic stabilization is called electrosteric stabilization. The electrosteric stabilization can be obtained by the addition of polymer such as polyelectrolytes. The depletion stabilization occurs due to a high concentration of unabsorbed polymers which are placed freely in the suspension. When the polymer concentration increases beyond the concentration required for depletion flocculation, the oxides slurries become stable again, which is called as depletion stabilization [12–18].

The interaction between Polyvinylpyrrolidone (PVP) and oxide particles in aqueous suspension is highly affected by the Point Zero Charge (PZC). The higher the pH of PZC means the less the hydroxyl content in the particles' surfaces and the less the PVP adsorption into the oxide surfaces [19]. The high PZC of 3Y-TZP at pH 9 enables depletion stabilization and improves the stability of the 3Y-TZP slurry [11, 20].

The concept of this study is organized as follows by Fig. 1. First, an appropriate Di-Ammonium Citrate (DAC) is used to break up the agglomeration of 3Y-TZP by a steric stabilization mechanism. DAC is commonly used as a steric stabilization due to the carboxyl group contained [11]. Once the slurries are stabilized by DAC adsorption, a sufficient amount of PVP is added to improve the slurries stability by depletion stabilization mechanism. Using this concept, a complex stabilization as a combination of steric and depletion stabilization can be obtained. As a result, better slurries stability and better-sintered properties of 3Y-TZP can be achieved.

2 Materials and methods

3Y-TZP (3 mol% Yttria) (Cenotec Co.LTD) slurries were prepared by adding 3.5 wt% of DAC (Samchun Chemicals) through the attrition milled process with 1300 RPM for 5 h. Further, PVP (Sigma Aldrich) was added in two concentrations, 0.8 and 2.5wt%, and then stirred for another 15 h and dried in an oven. Further, the obtained powder from attrition

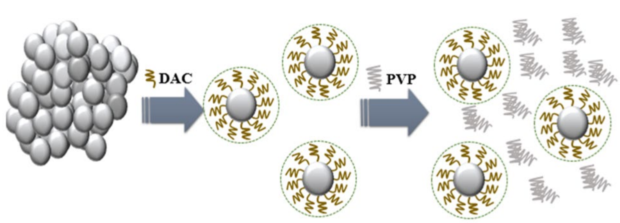

Fig. 1 Schematic of high slurries stability as a combination of steric and depletion stabilization mechanism achieved by addition of PVP into YSZ slurries containing DAC

milled was re-dispersed in the same solvent and re-milled through a ball-milled process at 200 RPM for 2 h. Further, both the powder obtained from the attrition milled process and the re-dispersion process were pressed into pellets. The pellets were sintered in a furnace at 1400 °C for 5 h.

Further, both the properties of slurries obtained by attrition milled and re-dispersed were investigated by viscosity (Brookfield, DV-II + Pro), particle size analyzer (Malvern Panalytical, Mastersizer 3000), and sedimentation rate test. Sedimentation tests were done by mixing 0.1 ml of each slurries into 32 ml of the solvent in a vial. Then the vials were put in a free vibration place and the sedimentation rate was investigated for 30 days tested. Further, FT-IR spectroscopy (JASCO, FT-IR-4200), was used to investigate the interaction between 3Y-TZP with DAC and PVP.

The effects of slurries stability on the sintered body properties were investigated through the Vickers hardness (Mitutoyo, HM-100), density, and microstructural analyses by Scanning Electron Microscopy (SEM) (ZEISS, Merlyn Compact).

3 Results and discussion

3.1 FTIR spectroscopy

The prediction of DAC adsorption and the inability of PVP to adsorb onto the YSZ surface was analyzed by the adsorption peak of FTIR. The FTIR analyses were performed on both YSZ samples before and after the PVP addition, also, the FTIR analysis of PVP and YSZ itself was conducted, as seen in Fig. 2a, b, respectively. The assignment of the various peaks as reported in this study has a reasonable agreement with the reported literature for the similar functional group. The FTIR spectrum of PVP shows a strong absorption peak at 1666 cm^{-1} indicates the C=O group. It also shows C–N stretching vibration at 1290 cm^{-1}, CH2 adsorption at 1436 cm^{-1}, and 1461 cm^{-1}. Further, a peak at 1378 cm^{-1} indicates C–H band is also observed [22–26]. Meanwhile, the FTIR spectrum of YSZ powders shows intense broad bands in the vicinity of 500 cm^{-1} and 1637 $^{cm-1}$, which are attributed to the stretching vibration of m-ZrO2 and t-ZrO2, as expected for ZrO2 samples. The adsorption of DAC into YSZ surfaces was observed by the peak shifted from 1637 $^{cm-1}$ to 1616 cm^{-1} and generation of a new peak at 1398 cm^{-1} respectively, indicating C = O and C–O stretching vibrations. The generation of C=O and C–O peaks indicates that DAC was adsorbed into YSZ surfaces by the carboxyl functional group. On the other hand, YSZ samples prepared with the combination of DAC and PVP as a complex dispersant agent were shown a similar FTIR spectrum with YSZ samples prepared with DAC only, which shows peaks at 1616 cm^{-1} and 1398 cm^{-1}. The absence of

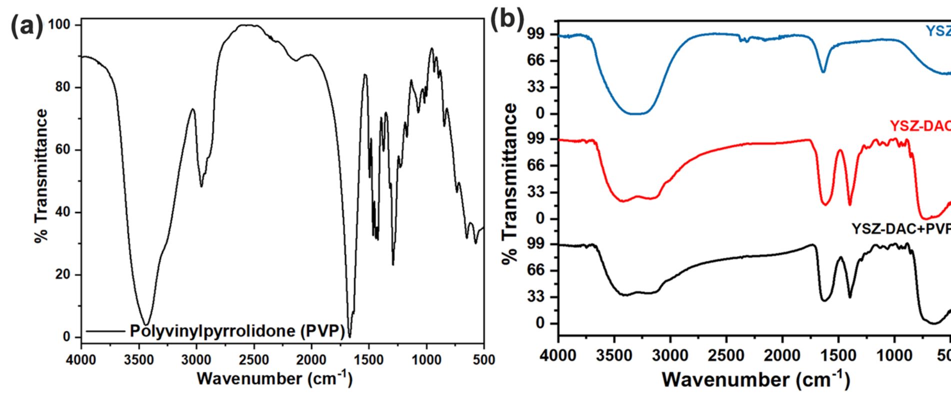

Fig. 2 FTIR spectra of **a** as purchased PVP. **b** Pure YSZ and YSZ containing 3.5wt% of DAC as dispersants with and without PVP addition

peak shifting or a significant change in the peak's intensity assumes that there is no bonding that has been built between PVP with DAC and YSZ surfaces. This assumption was supported by the previous study which confirmed that adsorption of PVP into the silica surfaces can be observed by the disappearance of C=O peak at 1661 cm^{-1} due to the hydrogen bonding between partial negative oxygen of C=O and hydrogen of Silanol [27]. Further, another study by Dieudonne R. Baganizi also reported the difference between unreacted PVP and carboxylate PVP by FTIR as shown by the change in the frequency of the characteristic band at 1650 cm-1 indicating the Pyrrolidone ring is opening due to the interaction with carboxyl [22]. According to the literature, and the results of this study which shown that there is no significant change in the FTIR peak of YSZ contained DAC before and after PVP addition indicates that PVP does not build any bonding either with DAC or YSZ surfaces and remained as a free polymer in suspension. This free PVP in the suspension can be used to improve the suspension stability by a depletion stabilization mechanism. Besides that, PVP has a longer molecular weight compared to DAC, thus making the repulsion between them to be more intense and generating better suspension stability.

The absence of adsorption of PVP onto YSZ containing DAC can explain due to the pH condition. According to the literature, which mentioned that suspension of YSZ containing DAC has a pH of 6.4 and the point zero charges occur at a pH of around 7 [11]. The suspension pH at 6.4 is higher compared to the pKa value of the carboxyl group, which is known at pH 4. The higher the pH of YSZ slurries compared to the pKa value of the carboxyl group, makes the carboxyl group highly ionized and leaves no interaction between PVP and DAC or 3Y-TZP [28].

3.2 YSZ slurries rheology

Figure 3a shows the rheological behavior of YSZ suspension with and without PVP addition and (b) YSZ contained DAC with and without the addition of PVP, respectively. As shown by Fig. 3a, the shear rate vs. viscosity of pure

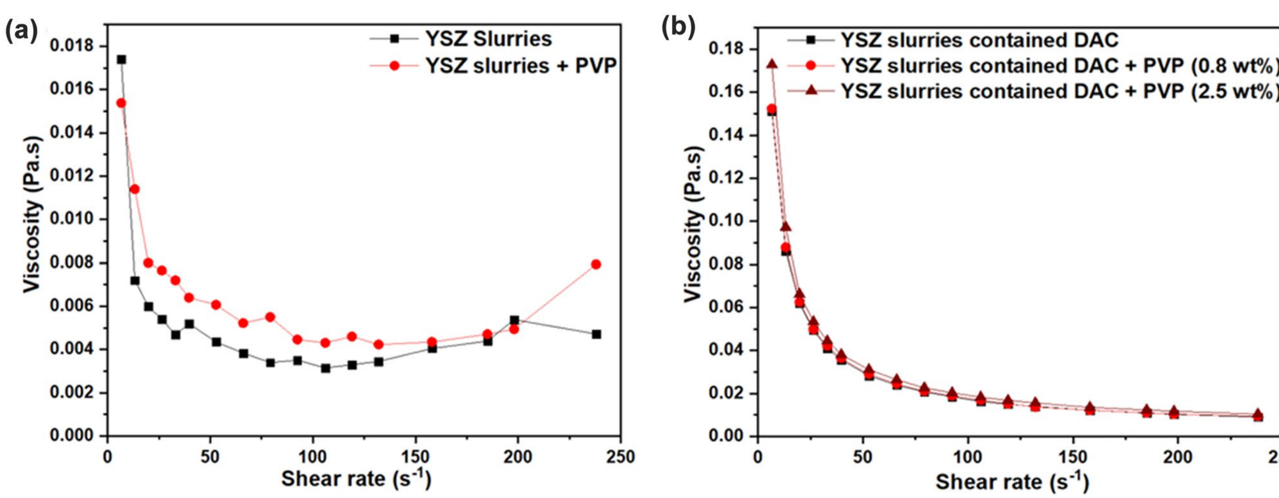

Fig. 3 Viscosities of **a** YSZ slurries with and without PVP addition. **b** YSZ slurries containing 3.5wt% of DAC with and without PVP addition

YSZ slurries before and after PVP addition shows a significant change. It shows that the addition of PVP into the YSZ slurries is generating higher viscosity values and present more viscosity peaks. The generation of more viscosity peaks after PVP addition indicates more agglomeration in the suspension. Meanwhile, Fig. 3b, shows the plot of the shear rate vs. viscosity of YSZ containing DAC with and without PVP addition. As noted in the experimental methods, that DAC concentration is decided to be 3.5wt% as it is known as the optimum concentration as previously reported [11]. By increasing the DAC concentration over 3.5wt% is known to increase the viscosity value significantly and leads to re-agglomeration as effects of the micelle's formation [11]. However, as shown by Fig. 3b, the addition of PVP into 3Y-TZP contained 3.5wt% of DAC does not result in any significant changes of viscosity value and there is no emergence of viscosity peak. The constant of the viscosity value and the vacancy of the viscosity peak indicates that the presence of PVP does not interfere with either DAC or YSZ. As a result, stability can keep maintained.

The slight increase in the viscosity value affirms the presence of free PVP in the YSZ solution [29]. With vacant of the viscosity peak as the shear rate is raised, this indicates that the stability of YSZ slurries has been not disturbed, even when the concentration of PVP is raised to 2.5wt%, as confirmed by the triangle-wine line in Fig. 3b. Since 0.8wt% of PVP concentration was found to be the optimum condition, thus it will be considered as the PVP concentration in the further results.

3.3 Particle size analysis

Figure 4, shows the particles size of YSZ which is confirmed by the particle size analyzer. It is shown that the addition of PVP into YSZ slurries containing DAC has reduced the agglomeration volume, as shown by the reduction of volume % at the second peak. By adding PVP into YSZ contained DAC has reduced the average particles size from 0.177 um for YSZ containing just DAC to 0.125 um for YSZ containing the combination dispersant of DAC and PVP as shown by the Table 1. These results indicate that PVP addition has successfully improved the YSZ slurries' stability by decreasing the particle size and agglomeration number. However, the re-dispersion state of YSZ containing just DAC shows a significant change in the particles size from 0.177 um to 0.349 um. The significant change of the particle size indicates that agglomeration has been reformed during the drying process. DAC stabilizes the YSZ by a short-length steric stabilization. However, during the drying process,

Table1 Average particle size of 3Y-TZP from PSA

Samples name	Average of particles size (um)	
	D(10)	D (50)
YSZ containing DAC	0.0412	0.177
YSZ containing DAC + PVP	0.0305	0.125
Re-dispersion state of YSZ containing DAC	0.168	0.349
Re-dispersion state of YSZ containing DAC + PVP	0.0275	0.114

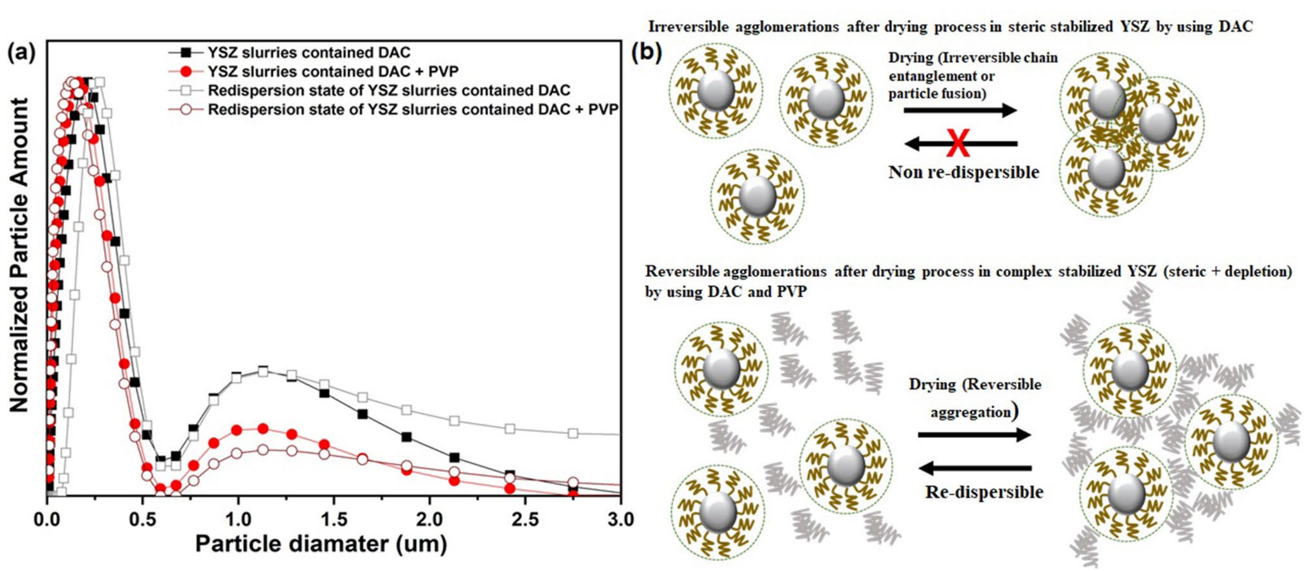

Fig. 4 **a** Particles size distribution of YSZ slurries containing 3.5wt% of DAC before and after PVP addition and **b** Schematic diagram of irreversible and reversible agglomeration formation of YSZ powder for YSZ containing DAC, and YSZ containing complex dispersants of DAC and PVP due to the solvent removal after the drying process

as the solvent is removed, the short-length chain of DAC becomes easier to entangle and makes the inter-particle bonding irreversible [30]. As the result of irreversible agglomeration, the particle size of YSZ in the re-dispersion state is increasing although the ball-milled has been applied. The schematic of irreversible agglomeration of DAC is shown in Fig. 4b. Meanwhile, the re-dispersion state of YSZ containing a complex dispersant of DAC and PVP shows the reduction of the particles size from 0.125 um to 0.114 um. The presence of unattached (free) PVP in the YSZ slurry containing DAC can prevent the entanglement of the DAC chain during the dried process. So, that is why when the solvent is removed, the unattached PVP will be placed between the particles and prevent the particles' inter-bonding formation as shown in the schematic of the Fig. 4b [30]. Also, the additional ball-milled during the re-dispersion process can break up the initial agglomeration of the particles, thus optimizing the dispersion and resulting in smaller particle size as shown in Table 1.

3.4 Sedimentation analysis

Sedimentation tests which are known as important physical testing for determining the stabilization properties [31] were conducted to compare the stabilization properties of attrition milled slurries and re-dispersed slurries. The sedimentation test was conducted in 1-month testing and the results are shown in Table 2. In the sedimentation test, slurries with better stability will show a slower sedimentation rate, as shown by the highest of supernatant height.

According to Table 2, the YSZ slurries that contain a combination of dispersant DAC and PVP show a higher supernatant height at 2.9 cm compared to YSZ slurries that contained DAC only at 2.68 cm. It is also shown that the re-dispersed YSZ slurries which contained a combination of dispersants of DAC and PVP can maintain their stability. However, the re-dispersed YSZ slurries containing just DAC were found to be settled down completely after one month of testing. The faster the sedimentation rate of YSZ slurries containing just DAC in the re-dispersion state must be occurred because of the lesser the stability with the largest particles size due to the presence of agglomeration, which is why the particles are easier to settle down.

3.5 Microstructure of sintered body

The sintered body properties can be determined by microstructure, and the microstructure can be determined by powder properties [31]. The presence of agglomerates will induce the heterogeneous microstructure with non-uniform pore size distribution and coarse pores of the sintered body [32]. In this study, to confirm the correlations between the YSZ slurry's stability and the sintered body properties, the microstructure was investigated by SEM as shown by Fig. 5 and Table 3. The grain size of 3Y-TZP was calculated using the Nano-measure 1.2 program. First, we set the scale using the scale bar of the SEM image, and then we calculated the size of every single grain (in this study, we measured for at least 150 grains, with every single grain size calculated crossly) and further the average of grain size was calculated. The grain size and grain size distribution of the sintered body of 3Y-TZP containing just DAC show a larger grain of 0.313 um (Fig. 5a), compared to 0.294 um for 3Y-TZP containing a complex combination of DAC and PVP dispersants (Fig. 5c). Further, in the re-dispersed case, 3Y-TZP containing just DAC shows the largest grain size at 0.423 um with the presence of a large volume of pores as shown in Fig. 5b. The increase of the grain size and the presence of pores in the re-dispersion case of 3Y-TZP containing just DAC can be induced by the presence of agglomeration. Although the dry-pressing was applied during the green body formation, the hard agglomeration will tend to remain and provide a powder compact with non-uniform particle packing, which results in deficient ceramics with excessive grain growth, presence of porous structure, or even the presence of cracks as shown in Fig. 5b [33, 34]. However, the re-dispersion state of 3Y-TZP contained a complex combination dispersant of DAC and PVP shows the smallest grain size of 0.281 um. The reduction of grain size in re-dispersed 3Y-TZP contained a combination of DAC and PVP can be obtained due to the optimization of 3Y-TZP stability during the re-dispersion process and less agglomeration. As the result, the particle packing is more uniform and resulted in a better microstructure at the sintered body, as shown by Fig. 5d.

This result becomes great and proves, that the sintered body properties of 3Y-TZP are highly affected by YSZ slurries' stability [35]. The presence of depletion stabilization of unattached PVP in steric-stabilized YSZ-contained DAC slurries is worked to prevent the agglomeration

Table 2 Sedimentation height of 3Y-TZP slurries and re-dispersed 3Y-TZP for one-month testing

Samples Name	Supernatant height (cm)
YSZ containing DAC	2.68
YSZ containing DAC + PVP	2.9
Re-dispersion state of YSZ containing DAC	Completely settled down
Re-dispersion state of YSZ containing DAC + PVP	3.1

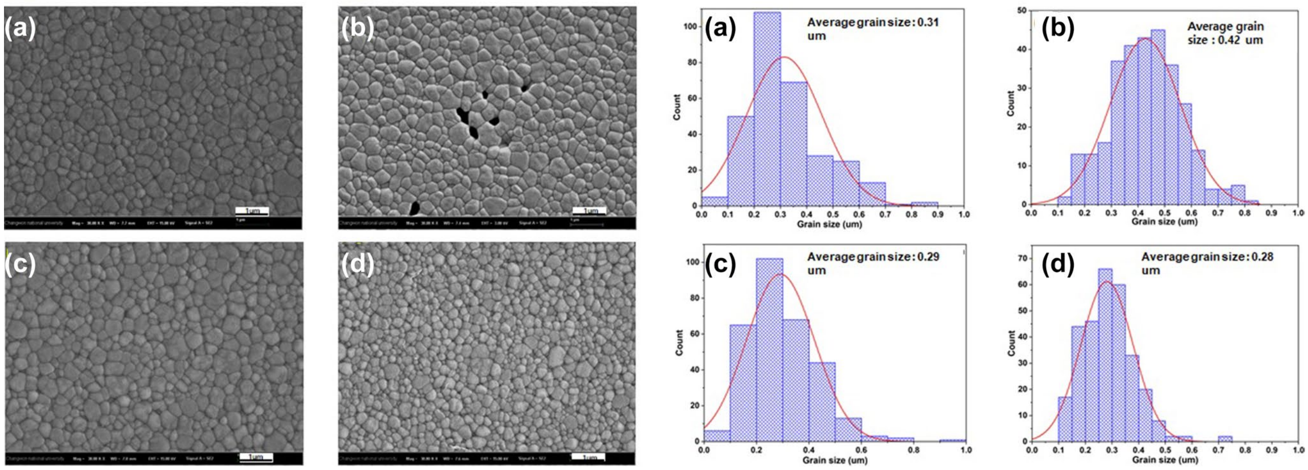

Fig. 5 Surface morphology of 3Y-TZP sintered body by SEM **a** 3Y-TZP containing DAC, **b** re-dispersed state of 3Y-TZP containing DAC, **c** 3Y-TZP containing DAC + PVP, **d** re-dispersed state of 3Y-TZP containing DAC + PVP

Table 3 Average of grain size of 3Y-TZP sintered body calculate from SEM image using Nano-measure 1.2 Program

Samples Name	Average grain size (um)
3Y-TZP containing DAC	0.313
Re-dispersion state of 3Y-TZP containing DAC	0.42
3Y-TZP containing DAC + PVP	0.294
Re-dispersion state of 3Y-TZP containing DAC + PVP	0.281

formation of particles during the drying process; as a result, powders compaction increases, and better sintering properties such as smaller grain size can be obtained.

3.6 Sintered body properties

Through, Fig. 6a, b, the mechanical properties of 3Y-TZP were investigated by density and Vickers hardness. It is shown that 3Y-TZP prepared with the complex dispersants of the combination of DAC and PVP has a higher density at 99.08% and Vickers hardness at 1275 HV compared to the samples prepared using just DAC which has a density at 98.66% with Vickers hardness value at 1268 HV. The mechanical properties of the re-dispersion state of 3Y-TZP prepared using just DAC also show the degradation of density at 94.5% with Vickers hardness at 1191 HV which is found as the lowest value as well. This degradation of mechanical properties of the re-dispersion state of YSZ containing just DAC is interpreted due to the presence of

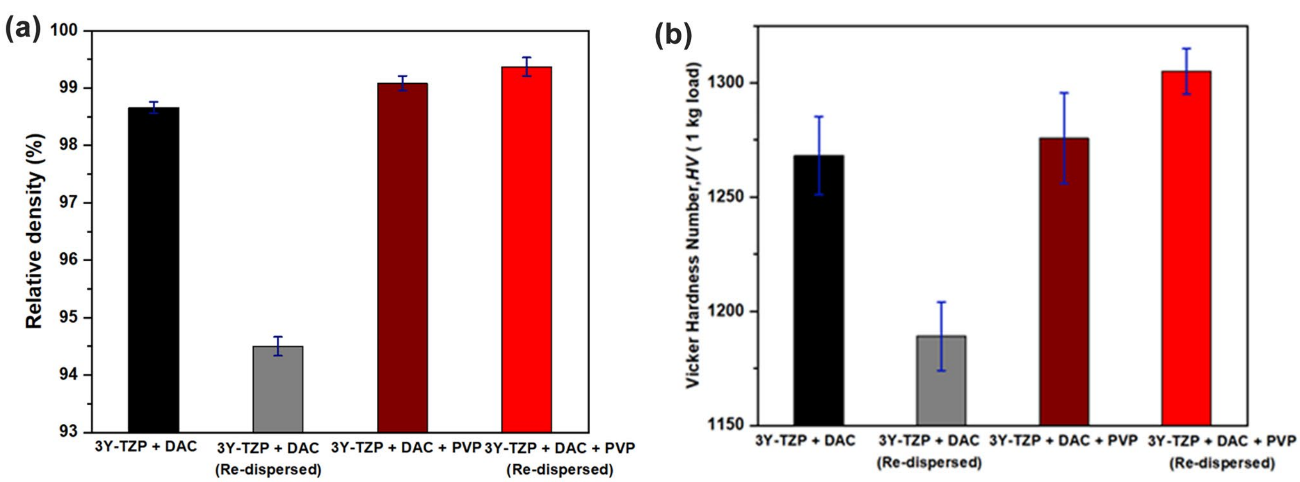

Fig. 6 **a** Average relative density of 3Y-TZP measured by Archimedes methods. **b** Average Vickers hardness of 3Y-TZP sintered at 1400 °C for 5 h

agglomeration in the sample, thus altering the density and Vickers hardness as well. The presence of agglomerates of powders leads to the formation of heterogeneous packing in the green body, thus leading to differential sintering during the sintering stage. This differential sintering occurs when different regions of the body shrink at different rates and lead to serious problems, such as the development of large pores and even crack in the sintered body; thus, it will alter the density and Vickers hardness as well [36]. Meanwhile, the re-dispersion state 3Y-TZP prepared with the complex dispersants of the combination of DAC and PVP has a superior value compared to the others samples as mentioned above. The best mechanical properties were found with density at 99.37% and Vickers hardness at 1305 HV. These results can be obtained due to the less agglomeration in YSZ powder, thus affecting the packing of the particles of the green body and altering the sintered properties, such as microstructure, higher density, and Vickers hardness number, as shown in Fig. 6. This result has a good agreement with the previous study, which emphasizes that the grain size shows a reverse correlation with mechanical properties of 3Y-TZP, the higher the grain size, the lower the hardness value [37].

4 Conclusion

YSZ slurries with high stability prepared with the combination of steric and depletion stabilization by utilization DAC and PVP as dispersant agents have been studied. DAC has enhanced the YSZ slurries stabilities by steric stabilization due to the adsorption of the carboxyl group of DAC's onto YSZ surfaces; further, PVP which cannot bond either with DAC or YSZ surface as the effects of pH thus placed it as a free dispersant in suspension. Further, due to the longer-length chain of PVP compared to DAC makes the repulsive force between the two become stronger and enhanced depletion repulsion to be more intense and induces the depletion stabilization. As the presence of the combination of DAC and PVP in YSZ slurries, a complex stabilization as a combination of steric and depletion stabilization can be obtained and resulting in better slurries stability. The presence of PVP as a depletion stabilization not only increases the YSZ slurries stability as shown by a uniform and smaller particle size distribution and lower the sedimentation rate, but also altered the sintered body properties as well. A later, we also re-dispersed the dry powder with solvent to form the original YSZ slurries and found that powder that stabilized with the combination of both DAC and PVP has a good stability properties and excellent sintered properties compared to the powder stabilized with DAC only. These results indicate that, even after dry process, the YSZ powder maintains its stability and agglomeration is not formed. Therefore, from these results, we expected that this study could help the previous technology of the ceramic powder and slurries market by realizing a high dispersion performance with less agglomeration of ceramic powders either in slurries or dried state.

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