Sintered Properties of Stainless Steel-doped Y-TZP Ceramics

Kelvin Chew Wai Jin¹,*, Mervyn Marlon Anak Mathew Minggat¹, and Ramesh Singh²

¹Department of Mechanical Engineering, School of Engineering, Faculty of Built Environment, Engineering, Technology and Design, Taylor’s University, 47500 Subang Jaya, Selangor, Malaysia.
²Centre of Advanced Manufacturing & Material Processing (AMMP), Department of Mechanical Engineering, Faculty of Engineering, University of Malaya, 50603 Kuala Lumpur, Malaysia.

Abstract. Yttria stabilised Tetragonal Zirconia Polycrystalline (Y-TZP) has been extensively renowned for its vast outstanding contribution in medical field such as primary function for implant biomaterial which acts as restoration of damaged body parts. Nonetheless in the past decades, there have been various reported cases that point the failure of hip transplants involving material of zirconia ceramic. This eventually leads to the birth of this research project where it studied the properties effects of Y-TZP as it was added or doped with stainless steel 316 (SS316). In this research project, the parameter of weight percentage for stainless steel 316 (SS316) to be doped with Y-TZP were as follows; 0 wt%, 0.1 wt%, 0.5 wt% and 1 wt%. The outcome of this experiment then were measured and analysed as according to properties of relative density, Vickers hardness and fracture toughness. The experiment was conducted by firstly mixed the Y-TZP and SS316 powders together as according to the weight percentage of SS316 as well as addition of ethanol solution that act for medium of enhancement. The mixture then underwent compacting pressure bench press as well as Cold Isostatic Pressure (CIP) in order to produce the green sample. After that, the green sample underwent sintering process via pressureless sintering method at mentioned ranges temperature of 1250°C, 1300°C, 1400°C and up to 1500°C. Subsequently, the sample then underwent grinding and polishing process before proceeding to the measurement and analysis process. The outcome of the measurement and analysis process revealed that as the content of SS316 doped with Y-TZP increases as well as the increased in sintering temperature, the relative density and fracture toughness profound to be increased while Vickers hardness indicated as decreased.

1 Introduction

In this robust technological era, experimentation involving combination of two distinct materials which consist of different properties respectively often been performed via doping method in order to attained successful outcome of material with superior properties. In the past for medical field, there were numerous of different materials have been studied for the

* Corresponding author: KelvinWaiJin.Chew@taylors.edu.my
objective to produce a durable implant biomaterial which function as alternative substitution to repair damaged body parts. Zirconia ceramic in particular has gain enormous popularity and attention from surgeon and scientist across the world due to its advantage as the closest restoration for bone tissue [1]. Nevertheless, zirconia ceramic alone has been identified in which it does not exhibit a high toughness as comparison made with metals. Therefore, metals do gain consideration in which it can be added to zirconia ceramic through doping and creating metal doped ceramic or ceramic doped metal. This method and process aim to enhance the overall replacement implants toughness. Moreover, this doped material progressively replaced the prostheses that comprised of purely ceramic as taking concerned to improve the prostheses major aspects of strength and durability [2, 3]. Hence, the studies regarding doped material to produce advance biomaterial has developed rapidly.

In addition, zirconia ceramic has been described to exhibit different properties when taking into account that it exists at distinct phases. In this case, zirconia ceramic often added with dopant or sintering additive in order for the characteristics and phases do not alter or transformed at different temperatures. Nonetheless, the advantages of zirconia ceramic were such that include high density and strength, high tolerance towards corrosion as well as high resistance towards impact. A specific zirconia ceramic type known as Yttria-stabilised Tetragonal Zirconia Polycrystals (Y-TZP) often been used as implant material in human body due to its high compatibility. Therefore, it has been applied as prosthetics for numerous years and yet displayed negative feedback. Moreover, stainless steel usually comes with different types and graded as according to its production specifications, grade composition as well as mechanical properties. Customarily when comparing zirconia ceramic with stainless steel, stainless steel in this case has displayed to have high strength, ductility, toughness as well as high corrosion resistance. Thus, the doped material can be utilised as a substitution for pure ceramic in order to create a more durable implant material.

A study was conducted to examine the doped material relative density such that considering the factors of varied zirconia and stainless steel compositions as well as heating rates [4]. In this case, the samples relative density was determined based on different sintering heating rates. The results then revealed that the samples relative density decreased when the stainless steel weight percentage (wt%) falls below 50% as well as during the increased in sintering heating rates. Vice versa, samples relative density increased when the stainless steel weight percentage (wt%) exceeding 50% as well as during the increased in sintering heating rates. A study was performed to find out the fracture toughness and Vickers hardness of zirconia stainless steel composite [5]. In this investigation, the samples were made as according to stainless steel concentration with ranges from 15% to 30%.

The results displayed to obey the mixture law such that when stainless steel content inside the zirconia/stainless steel composite increased, the fracture toughness indicated as increased while Vickers hardness described as decreased [6, 7]. In addition when subjected to fractures, there was no observation of plastic deformation. As comparison to zirconia ceramic, zirconia/stainless steel composite do not exhibit brittle condition [8]. In terms of biomedical implants, these types of characteristics often denoted as vital for material enhancement. In the previous years, there were various cases had been reported that address the serious issue of zirconia ceramic hip transplant failure. In order to provide solution to this dilemma, various thorough studies have been conducted that primarily emphasize on sintering of zirconia doped material at low temperature as well as mechanical properties [9]. In this research project, it fundamental objective was to study the effects of properties when adding dopant stainless steel 316 to Y-TZP. This report presented the experimental flow process as well as results for the tested physical and mechanical properties.
2 Methodology

2.1 Powder preparation

The starting materials for this research project methodology begins with acquired and gathered the two essential material substances that may include Y-TZP and stainless steel 316 powders. Table 1 shows the Y-TZP and stainless steel 316 summaries of typical characteristics accordingly. The powder preparation process was initiated with powder synthesis process in which both Y-TZP and stainless steel 316 powders were been mixed together as well as measured based on the stainless steel 316 content. Before both of the powders to be mixed together, each of the samples weight percentage were fixed as according to the stainless steel 316 content which ranges from 0 wt%, 0.1 wt%, 0.5 wt% and 1 wt%. Ethanol with measurement of 100 ml was firstly poured into the beaker and only then followed by poured both of the stainless steel 316 and Y-TZP together into the beaker in order to undergo the mixing process.

Table 1. Characteristics of Zirconia and Stainless Steel powders.

<table>
<thead>
<tr>
<th>Material</th>
<th>Grade /Type</th>
<th>Composition (wt%)</th>
<th>Powder Particle Size (µm)</th>
<th>Particle Shape</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconia</td>
<td>Stabilized tetragonal zirconia, Y-TZP</td>
<td>5.2 Y2O3, HfO2 &lt;5, Al2O3 0.1-0.4, ZrO2 (remaining)</td>
<td>0.04</td>
<td>Spherical</td>
</tr>
<tr>
<td>Stainless Steel</td>
<td>316</td>
<td>Cr 16-18, Ni 10-14, Mo 2-3, C 0.08, Fe (remaining)</td>
<td>5-20</td>
<td>Spherical</td>
</tr>
</tbody>
</table>

The equipment of Digital Ultrasonic Bath (Powersonic UB-410, Thermoline Scientific, Australia) was utilised by filled up the equipment with distilled water. Next, the beaker then placed inside the Digital Ultrasonic Bath equipment for 15 minutes of thorough mixing process with continuous stirring and beaker later was removed from equipment itself once achieved completion of mixing. After that, the machine of Laboratory Attritor Mill (01-HD, Union Press, United States) was utilised by pouring the mixture inside it with the purpose for the mixture to undergo the attrition milling process where further break down of powder mixture for duration of 45 minutes with running at 500 rpm. Furthermore, 5 mm size of zirconia milling ball which account quarter of the mixture amount were added to the Laboratory Attritor Mill machine also with aim to enhance the entire milling process completion. A sieve utility of measurement mesh size 212 µm was used for the first time to sieve the mixture where zirconia ball were trapped and minimised the mixture to slurry conditions. The remaining mixture then created into a cake form and undergoes baking process where the mixture was stored inside the Universal Oven (UN 55, Memmert, Germany) which heated for 60°C for overnight duration and subsequently left dry. The cake mixture after the heating process was crushed into smaller form using hand tools. After that, a sieve utility of measurement mesh size 212 µm was utilised for second time with objective to produce the final powder composition of definite fine. Table 2 shows the summaries of typical properties of Y-TZP and stainless steel 316, respectively, at room temperature.
Table 2. Typical properties of Y-TZP and Stainless Steel 316 at room temperature.

<table>
<thead>
<tr>
<th>Materials</th>
<th>Density (g/cm³)</th>
<th>Young's Modulus (GPa)</th>
<th>Vickers Hardness (GPa)</th>
<th>Fracture Toughness (MPa.m⁰⁵)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconia (Y-TZP)</td>
<td>6.02</td>
<td>200</td>
<td>12.5</td>
<td>13</td>
</tr>
<tr>
<td>Stainless Steel 316</td>
<td>8.00</td>
<td>193</td>
<td>1.47</td>
<td>112–278</td>
</tr>
</tbody>
</table>

2.2 Green sample preparation

The machine of Hydraulic Bench Press (HP10-150, RMS Hydro, Malaysia) was utilised for the compacting process of the powder mixture in which the powder mixture was poured into a round shaped die with diameter measurement of 20 mm. The subjected high pressure bench press was 1000 bar for 10 seconds. After that, the Cold Isostatic Press (KJYU150, Shanxi Golden Kaiyuan Co., Ltd, China) machine was used for the CIP process where the green body round tablets were been placed inside a glove as well as placed it again inside desired square shaped container. On the other hand, the container in this case then to be stored under room temperature which in this case aim for neither heat nor pressure to penetrate and ultimately affect the green body round tablets during the pressing process. In overall for this current process stage, the green body round tablets indeed can likewise be referred as green samples.

2.3 Sintering

The type of applied sintering process method was indeed pressureless sintering method and to be performed after the formation of green samples. In this process, it begins by placing the green samples inside the crucible which to be stored inside the Electric Box Furnace (LT10, LT, Malaysia). The applied heating for sintering temperatures were distinct such that ranging from 1250°C, 1300°C, 1400°C and up to 1500°C where only one sintering temperature was performed in one day. The process heating rate was also conserved at 10°C per minute. In addition, the furnace holding time was kept for 2 hours and after the process, the heating was stopped. The samples then let to be cooled down at rate of at 10°C per minute where it explained that the samples cooled through natural cooling method until approaching ambient.

2.4 Grinding and Polishing

2.4.1 Grinding

After the completion of sintering process, the samples were vitally required to be grinded and followed by polished. The equipment of Polishing & Grinding Machine (GP2V, LT, Malaysia) was utilised for both of grinding and polishing processes. For grinding process, the Polishing & Grinding Machine was installed with grinding papers and operates at 50 rpm in which two minutes for each sample. The applied grinding papers range with five grit sizes in total such that namely 120, 240, 600, 800 and 1200 that consists with surface material of silicon carbide (SiC). The samples were grinded only on one side and the other side used as samples labelling for clear identification purposes. Furthermore, the grinding
results for each grade were displayed as visible one direction line and vitally covering the entire grinded side of samples surface. Each visible direction lines also represent each grade of grinding papers respectively.

2.4.2 Polishing

Polishing process was performed subsequently after the grinding process in which polishing clothes were utilised and injected with diamond paste as well as lubricant via syringe device. The polishing clothes were installed to the equipment of Polishing & Grinding Machine (GP2V, LT, Malaysia) and operated at 20 rpm. In addition, the diamond paste in this case consists of diamond bit with grades measurement of 1 µm and 6 µm. Moreover, the lubricant likewise been injected to the polishing clothes as well as to function by ease the spreading of diamond paste evenly across the entire surface of the samples. The polishing process was performed for duration of two minute only for each sample.

2.5 Measurement and Analysis

2.5.1 Relative density

The density of samples was determined in which the testing was conducted by adhering strictly according to Archimedes principle and water immersion technique. ASTM C-29 was used as reference of standard to relate with the determined relative density. In this process, an analytical balance with density kit (AY220, Shimadzu, Japan) was employed by filled up the equipment with distilled water that function as immersion medium. The value for density of water was declared as 0.99654 kg/m³. The stated Y-TZP theoretical density was 6.10 g/cm³ and the following formula (eq 1) was used to calculate the relative density of samples.

$$\rho = \frac{w_a}{w_a - w_w} \rho_w$$  

(1)

where

- $\rho$: Relative density of sample (g/cm³)
- $w_a$: Weight of sample in air (g)
- $w_w$: Weight of sample in water (g)
- $\rho_w$: Density of distilled water (g/cm³)

2.5.2 Vickers hardness

The samples Vickers hardness were experimented by utilising the equipment of Vickers hardness tester (MV-01, Matsuzawa, Japan) that consist of pyramidal diamond indenter as well as subjected load application of 10 kg. Only minimum duration of 10 seconds for held down implementation. ASTM E284-99 and ISO 14705 can likewise be used as a standard reference for all experiment parameter. In broad, the impression was indicated as square such that presence with two diagonals that has identical length. The indentation in this process was measured by using the Vickers hardness tester machine microscope that has been installed together with filar micrometer which consist advantage accuracy of 0.1 µm. The equation to calculate the samples Vickers hardness was presented below as (eq 2). Figure 1 displays the schematic diagram of a Vickers hardness indenter profile which
described as angle between the opposite faces at the pyramidal indenter vertex and consequent resulting indentation on the sample.

\[ H_v = \frac{1.854P}{(D)^2} \]  

(2)

where

- \( P = \) Applied load (kg)
- \( D = \) Average of two diagonals \( D_1 \) and \( D_2 \) (m)

**Fig. 1.** (a) Schematic diagram of Vickers hardness indenter with profile angles \( \alpha = 136^\circ \pm 0.5^\circ \) (angle between the opposite faces at the vertex of the pyramidal indenter) and (b) an indentation that is formed on the sample.

### 2.5.3 Fracture toughness

The samples fracture toughness were determined with an identical method of testing as the one of Vickers hardness due to it used similar indentation. The microscope filar micrometer was used to determine the crack length (\( L_1, L_2, L_3, \) and \( L_4 \)) and indentation length (\( D \)) after the formation of indentation. The equation for fracture toughness was indicated below as (eq 3). Figure 2 displays the schematic diagram regarding typical Palmqvist crack system that emerged after Vickers indentation. In this case also, “\( L \)” was described as the crack measured length which used to calculate the samples fracture toughness. The Vickers indent diagonals (\( D_1 \) and \( D_2 \)) were suggested to be identical with the length of 2a by referring to Fig. 1.

\[
\left( \frac{K_{IC}}{H_v a^2} \right) \left( \frac{H_v}{E \phi} \right)^{\frac{1}{2}} = 0.035 \left( \frac{L}{a} \right)^{\frac{3}{2}}
\]

(3)

where

- \( K_{IC} = \) Fracture toughness (MPa. m\(^{1/2}\))
- \( H_v = \) Vickers hardness (GPa)
- \( E = \) Young’s Modulus (GPa), taken as 200 GPa for Y-TZP ceramic
- \( a = \) Half-length of the average diagonal (m)
- \( L = \) Average crack length (m)
- \( \phi = \) Constraint factor taken as 3
described as angle between the opposite faces at the pyramidal indenter vertex and consequent resulting indentation on the sample.

\[ P = \text{Applied load (kg)} \]
\[ D = \text{Average of two diagonals } D_1 \text{ and } D_2 \text{ (m)} \]

Fig. 1. (a) Schematic diagram of Vickers hardness indenter with profile angles \( \alpha = 136^\circ \pm 0.5^\circ \) (angle between the opposite faces at the vertex of the pyramidal indenter) and (b) an indentation that is formed on the sample.

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\[ K_{IC} = 0.035 \]  
\[ K_{IC} = \text{Fracture toughness (MPa.m}^{1/2}\text{)} \]
\[ HV = \text{Vickers hardness (GPa)} \]
\[ E = \text{Young's Modulus (GPa), taken as 200 GPa for Y-TZP ceramic} \]
\[ a = \text{Half-length of the average diagonal (m)} \]
\[ L = \text{Average crack length (m)} \]
\[ \Phi = \text{Constraint factor taken as 3} \]

Fig. 2. Schematic diagram regarding the typical Palmqvist crack system that emerged after Vickers indentation. Parameter “L” refers to the measured crack length used for the computation of both hardness and fracture toughness. The Vickers indenter diagonals \( D_1 \text{ and } D_2 \) are equivalent to the length of "2a" as indicated in Fig. 1.

2.5.4 Accelerated Ageing Test

The first step for ageing test was obligatory required the samples to be stored inside the acid digestion vessel whilst being subjected to hydrothermal conditions for increasing time intervals. Consecutively, the samples were then be immersed inside distilled water with 20 ml in measurement. After the immersion process, it later required the samples to be transferred to an oven for heating and baking process as well as provided with temperature below 180°C for duration of five hours. Segregations of samples were later to be done as according to the corresponding ageing time intervals and subsequently examined using X-ray diffraction (XRD) to monitor the rate of zirconia phase transformation.
3 Results and Discussion

3.1 Relative Density

By referring to the plotted graph in Figure 3, the lowest identified relative density was 55% which consist of 1 wt% of SS316 as well as sintered at 1250°C. The relative density for 1 wt% of SS316 then continue to increased when sintering temperature increased from 1250°C to 1400°C and remained static for sintering temperature between 1400°C to 1500°C. For 0 wt% SS316 and 0.5 wt% SS316, the results showed that the maximum sintering temperature was 1400°C due to obtained the highest relative densities which were 93% and 92% respectively. In overall, considering sintering temperature between 1250°C and 1300°C, the relative densities increased when weight percentage of stainless steel increased. It can be observed that adding dopant SS316 by 0.5 wt% to Y-TZP was beneficial to enhance the densification but provided that it must be sintered at low temperature of 1300°C.

Nonetheless taking into account of sintering temperature between 1400°C and 1500°C, all of the samples exhibit no improvement or decreased in relative densities. This can be supported that grain coarsening could possible cause the decreased in densities as higher sintering temperature was applied. In addition, when a higher sintering temperature was applied such that exceeding 1500°C, zirconia underwent partial transformation such that transform into monoclinic or cubic phases [28]. The samples doped with 0.5 wt% SS316 indicated to exhibit the optimum benefit in terms of densification where it attained the highest relative density of 90% and 89% when sintered at temperature 1300°C and 1500°C. For the case 0 wt% SS316 which denoted as undoped or pure Y-TZP, the achieved density was 72% when sintered at 1300°C. Considering that sintering temperature was identical such that at 1300°C but different weight percentage of SS316 such that 0 wt% SS316 and 0.5 wt% SS316, the samples of doped 0.5 wt% SS316 displayed a higher relative density as compared to 0 wt% SS316. This in turn proved that adding SS316 into Y-TZP indeed contributed an enhancement for consolidation process but must in line with condition of lower sintering temperature such that below 1500°C.

Taking into account of sintering temperature between 1250°C and 1300°C which indicated nearly all samples showed an increased, as weight percentage of the dopant increased, the samples relative density indicated as increased. This was considered as truthful as compared to theory such that it mentioned stainless steel density increased when heating rate increased and therefore, promote the increased in samples densities [10]. This also means that samples with more weight percentage of stainless steel exhibits a higher density due to the total accumulated of an increased in densities based on the amount. However, considering the opposite case such that for 0.5 wt% SS316 which showed a decreased in relative density when sintered from 1250°C to 1300°C, the trend line was the opposite way. The reason for this may be due to method of processing for the samples such as shorter sintering holding time. A shorter holding time may cause the reverse of the material density value such that it became decreased [11]. Another possible reason for the decreased in relative density was due to uneven powder composition. It considered as extremely difficult to obtain the most ideal powder composition.
3 Results and Discussion

3.1 Relative Density

For the case 0 wt% SS316 which denoted as undoped or pure Y-TZP, the achieved density was 72% when sintered at 1300°C. Considering that sintering temperature was identical to 0 wt% SS316. This in turn proved that adding SS316 into Y-TZP indeed contributed an enhancement for consolidation process but must in line with condition of sintering temperature between 1250°C to 1400°C and remained static for sintering temperature between 1400°C to 1500°C. For 0 wt% SS316 and 0.5 wt% SS316, the results showed that the maximum highest relative density of 90% and 89% when sintered at temperature 1300°C and 1500°C. Considering that sintering temperature was identical to 1300°C, sample of 0.5 wt% SS316 revealed to achieved the highest optimum sintering temperature to obtain the highest sintering temperature was 1300°C. As sintering temperature continued to increase from 1300°C to 1400°C, all doped samples showed a rapid declined in Vickers hardness but for undoped sample on the other hand indicated as increased. When sintering temperature increased higher from 1400°C to 1500°C, all samples for both doped and undoped presented as decreased. Considering sintering temperature at 1300°C, sample of 0.5 wt% SS316 revealed to achieved the highest among all samples such that 13.3 GPa. In comparison with 0 wt% SS316 which denoted as pure Y-TZP or undoped sample, 0.5 wt% SS316 showed a higher Vickers hardness than 0 wt% SS316 with values 13.3 GPa and 12 GPa respectively. For all doped samples, the optimum sintering temperature to obtain the highest sintering temperature was 1300°C.

At sintering temperature of 1300°C, the Vickers hardness values for samples of 0.1 wt% SS316, 0.5 wt% SS316 and 1 wt% SS316 were 13 GPa, 13.3 GPa and 11.7 GPa. For undoped sample of 0 wt% SS316, the optimum sintering temperature was 1400°C in which it attained it highest Vickers hardness value of 13.5 GPa. Considering an increased sintering temperature between 1250°C to 1300°C, all samples exhibit improvement of Vickers hardness due to sintered body strengthens densification. Nonetheless, for the case of decreased in Vickers hardness as sintering temperature continued to increase from 1300°C to 1500°C, this issue occurred due to grain coarsening that related to cubic structure [12].

Fig. 3. Relative density of sintered Y-TZP ceramics doped with 0.1 to 1 wt% stainless steel SS316.

3.2 Vickers Hardness

As according to the plotted graph in Figure 4, all samples exposed to have an increased in Vickers hardness when considering sintering temperature was increased from 1250°C to 1300°C. As sintering temperature continued to increase from 1300°C to 1400°C, all doped samples showed a rapid declined in Vickers hardness but for undoped sample on the other hand indicated as increased. When sintering temperature increased higher from 1400°C to 1500°C, this issue occurred due to grain coarsening that related to cubic structure [12]. By referring at sintering temperature of 1500°C, Vickers hardness decreased as weight percentage of SS316 increased. This likewise follows the trend line as well as obeys the mixture law such that explained Y-TZP was harder than SS316.
3.3 Fracture Toughness

Based on the plotted graph in Figure 5, samples of 0 wt% SS316, 0.1 wt% SS316 and 1 wt% SS316 profound to exhibit an increased in fracture toughness when sintering temperature increased from 1250°C to 1300°C. On the contrast side, sample of 0.5 wt% SS316 displayed a decreased in fracture toughness when sintering temperature increased from 1250°C to 1400°C. At sintering temperature of 1300°C, it indicated as the optimum sintering temperature for samples of 0 wt% SS316, 0.1 wt% SS316 and 1 wt% SS316 as all samples showed their highest fracture toughness with values 5.5 MPa.m$^{1/2}$, 5.5 MPa.m$^{1/2}$ and 6 MPa.m$^{1/2}$ respectively. As sintering temperature increased from 1400°C to 1500°C, samples of 0 wt% SS316, 0.1 wt% SS316 and 0.5 wt% SS316 identified as increased in fracture toughness whereas sample of 1 wt% SS316 showed a decreased in fracture toughness. At sintering temperature of 1500°C, sample of 0.5 wt% SS316 has the highest fracture toughness with value of 5.7 MPa.m$^{1/2}$ and 1500°C was declared as its optimum sintering temperature.

Mismatched of thermal expansions occurred at the section between stainless steel and zirconia particles provided when there was an increased in stainless steel particles [13]. Hence, radial tensile stress was developed internally during cooling process and consequently contributed towards particles with inadequate interfacial bonds which cause debonding. In addition, crack also then formed in a method that it smoothly inseminates between the unsteady interface of zirconia and stainless steel particles [33]. This in turn leads to the enhancement of fracture toughness via the process of crack deflection. Sintering temperature on the other hand suggested that it marginally affected the fracture toughness and this also considering that the all wt% SS316 exhibit fracture toughness with values varied from 5.2 MPa.m$^{1/2}$ to 5.7 MPa.m$^{1/2}$ when sintered at sintering temperature of 1500°C. In this case, homogenization developed over stabilizer which also existed within the samples ZrO$_2$ grains and thus, produce an improved stability of tetragonal grains [14].
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3.4 Ageing Resistance

The accelerated ageing test was subjected to selected samples with aim to determine the samples ageing resistance due to low temperature degradation (LTD) when exposed towards humid environments. Generally, samples were subjected to superheated steam at temperature of 180°C as well as 10 bar of pressure with increasing duration time. In this case, XRD analysis was utilised with the objective to monitor the samples phase content as well as identified the monoclinic zirconia (m-ZrO₂) phase content. Figure 6 shows the monoclinic phase content for samples of 0 wt% SS316 and 0.1 wt% SS316 accordingly with increasing ageing duration up to 24 hours. The monoclinic phase content was monitored for ageing duration of 0, 1, 6, 12 and 24 hours respectively. Sample of 0.1 wt% SS316 with sintering temperature of 1300°C was chosen as reference due to optimum composition and consist of the highest mechanical properties. By observing the trend line on the plotted graph of monoclinic phase content against ageing duration in figure 6 above, it has been identified that pure Y-TZP or known as 0 wt% SS316 exhibit a rapidly continuous increasing trend line. The sample for 0 wt% SS316 then breaks during the ageing treatment for the next 12 hours which tells that data was absence for analysis.

After an hour of exposure, 0 wt% SS316 displayed 11 vol% of monoclinic phase content which was higher and drastic in comparison to 0.1 wt% SS316 that presented as 6.5 vol%. As approaching 24 hours of exposure, 0 wt% SS316 closed the monoclinic phase content at 91 vol%. This essentially means that the longer the ageing duration, the higher the development of monoclinic phase content [15]. In addition, this was considered as not ideal due to monoclinic promote failure of material. The more the monoclinic phase, the worst the material properties become. Moreover, development of crack and surface spalling were observed at the sample via visual inspection as results of progressive ageing [15]. On the other hand, sample of 0.1 wt% SS316 had presented a better ageing resistance but sample then disintegrated at 12 hours which closed the monoclinic phase content with 20 vol%. This can be proved based on the trend line for 0.1 wt% SS316 stopped at 12 hours.

The disintegrated of samples will be consideration for further study. By comparing both samples of 0 wt% SS316 and 0.1 wt% SS316 at similar 12 hours of exposure, 0.1 wt%
SS316 was considered ideal as compared to 0 wt% SS316 due to it showed significant low of monoclinic phase content. This proved that 0.1 wt% SS316 exhibit better ageing resistance and phenomena possibly due to finer grain size obtained through sintering process. Hence, sample of 0 wt% SS316 can be further upgrade to prevent the disintegration in the future works. Pure Y-TZP (0 wt% SS316) was considered as instable which leads to lower ageing resistance. Hence, it changes phases easily from tetragonal to monoclinic supposed that polar water molecules penetrate the sample particles. Phase transformation occurs in such a way that if one of the grain experienced stress, this will interfere the neighbouring grain and hence, phase changes take placed and promote low temperature degradation. This project also aims to achieve an ideal stable matrix in order to prevent phase transformation. By doing so, ageing rate likewise can be minimised via the reduction of deterioration rate, microcracking and surface roughening.

![Graph showing monoclinic zirconia phase content](image)

**Fig. 6.** Monoclinic zirconia phase content of undoped and 0.1 wt% SS316-doped Y-TZP samples at various accelerated ageing time intervals of exposure at hydrothermal conditions of 180°C and 10 bar.

**4 Conclusion**

The experiment has successfully been conducted to investigate the advantageous and effects of adding sintering additive stainless steel 316 to be doped with Y-TZP ceramics. It has been identified that the highest density would be 93% for the case of 0 wt% SS316 with sintered at sintering temperature of 1400°C. For Vickers hardness, the lowest value profound to be 9.8 GPa when taking into account of 0 wt% SS316. In addition, the optimum value for fracture toughness would be 6 MPa.m\(^{1/2}\) which considering for the 1 wt% SS316. By referencing to the all the plotted graph for all wt% of SS316 for sintering
temperature between 1400°C to 1500°C, the effects of adding stainless steel 316 to be doped with Y-TZP were such that the relative density nearly static, Vickers hardness decrease and fracture toughness increase. A low monoclinic phase content for samples was also the targeted outcome.

References