

# Effect of Input Parameter of Cold Isostatic Press (CIP) Towards Properties of Zirconia Block

Noratiqah Syahirah Mohd Zarib, Shahrul Azam Abdullah, Muhammad Syazwan Mazelan

**Abstract:** Zirconia have become widely studied as consequence of their outstanding mechanical properties, such as hardness, mechanical strength and fracture toughness, which allow them to cover a wide spectrum of applications as structural ceramics, including the field of biomaterials. This study was to compare the strength properties of zirconia block with and without Cold Isostatic Press (CIP). The mechanical properties of zirconia block with and without CIP were characterized. Samples of zirconia block will undergo forming process via Cold Isostatic Pressing (CIP), four levels of soaking time (no CIP, 60, 90 and 120 minutes). All of the sample with different soaking time then were sinter in the furnace. The parameter for sintering process was fixed 1300°C at rate of 3°C / min. All of the sample were tested for its strength properties using Vickers test. The density and shrinkage of the zirconia block was be analyzed. Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD) were used to characterize samples if zirconia blocks.

**Keywords:** Cold Isostatic Press (CIP), Scanning Electron Microscopy (SEM), Vickers Hardness, Zirconia.

## I. INTRODUCTION

Zirconium oxide, every now and then referred to as zirconia, can be a silver pellucid precious metal of zirconium and a synthetic fundamental with all the indication, Zr and atomic weight 40. Hence, the melting point of zirconia is 2715°C, 4300°C for boiling point while the density is about 5.68 g/cm<sup>3</sup>. Zirconia also has special properties such as it is slow in thermal conductivity, seam together with the non-magnetic automatic insulator humor and show up as power contestant for most thermic applications [1]. Besides, Zirconia is a synthetically inactive physical, it has an excellent deterioration, acidic and alkaline resistance. Goods of zirconia is calculate below cutting back surroundings, whichever discover a necessary subject material in catalytic field. Its physical, standardized (sharp concentration, sharpness, show defiance, battle to deterioration, modulus of malleability similar to steel, concerted of sizzling rise linked were propagation [2]. Cold isostatic press (CIP) is one of the pressing techniques that available to fabricate zirconia block. Generally, the CIP is not complicated and easily performed by placing the powder materials in an enfolded rubber capsule.

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This was followed by the demands of the hydrostatic pressure (P), and by assuming systematic density of the compact developed inside the capsule then it can be predicted that every part of the powder obtained hydrostatic stress state, where the principal stresses are all match. However, in practice this does not occur frequently and there is a deviation in the density of the compact, therefore causing irregularity from the hypothesis of the existence of hydrostatic stress state. Raw material, zirconia (3mol% Ytria Stabilized Zirconia (YSZ)) is in a powder form was received from the manufacturing industry [3]. The purpose of this paper is to compare the physical and mechanical properties of zirconia at each different soaking parameter.

## II. MATERIALS AND METHODS

Zirconia powder was then turn into block form and the size of each sample was in ranged 12 mm width and 10 mm height in the shape of a cylinder. Next, once zirconia powder was form into block, the samples were undergoing compaction process. Nine samples were compact by using hand press and pressure was applied to the zirconia block powder was similar which were 3 MPa. After that, when all the samples were completed where the samples of zirconia block were filled into the cold isostatic press (CIP) machine. The model of the machine is ISA-CIP-60-150-450-A. Each of the samples were coated by high quality strength of rubber. Rubber gloves were used as mold for the samples. The fix pressure was set in at 2400 kg f/cm<sup>2</sup>. The parameters for this research is sintering. The soaking time for this research was 60 min, 90 min and 120 minutes were applied for this sample. Besides, the samples were sintered in the furnace for sintering process at temperature of 1300°C subsequently. The increment temperature of the sample was then set at rate at 3°C / min and cooling rate also was set at rate of 3°C / min. before undergo through characterization and mechanical testing.

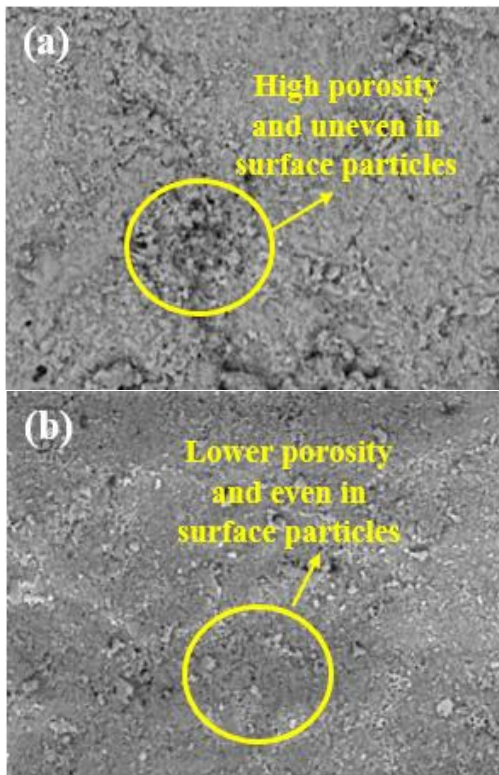
Scanning Electron Microscopy (SEM) was used to analyze the morphology of zirconia block surface with magnification 3000x. Each sample from different parameter undergone the morphology analysis, but comparison only between control sample and sample with soaking time 60 minutes. Digitized images were recorded to study the porosity, particle arrangement and grain growth of the zirconia samples. The standard for SEM is ASTM 1508. Furthermore, the samples before and after sintering undergo density testing for this research. The density values of the samples were calculated by using electronic density meter. The density machine was tested in ASTM C329. The

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micro hardness values of the samples were measured using a micro hardness tester which is fitted with a Vickers indenter. The diamond pyramid mind of aDuramin-1 Vickers sharpness analyst was perturbing the qualified come up below a prearranged responsibility (294 N) top 10 s to result in a diamond-shaped rut. The ASTM for Vickers Test was ASTM C1327. Last testing was percentage of volume shrinkage of the sample were calculated by using formula. Volume for the samples were calculated, then volume of samples after sintering minus volume of the samples before cold isostatic pressing divided by volume samples after sintering. Average of samples from different parameter were taken. Diameter and height of all sample were took with precise. The dimension of all sample which were diameter and height were record after compaction of the samples and after sintering process.

### III. RESULTS AND DISCUSSION

Fig. 1 shows the Scanning Electron Microscope (SEM) micrograph of sintered samples of zirconia block with addition of CIP and without CIP. Both of the samples were analyzed by using 3000x magnification.



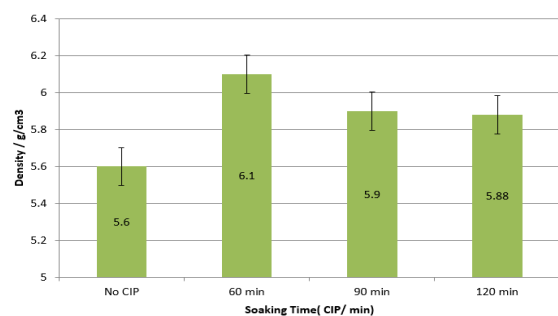
**Fig. 1. SEM micrograph of (a) Zirconia block without CIP (control sample) and (b) Zirconia block with addition of CIP within 60 minutes soaking time under magnifications of 3000X.**

Fig.1 (a) exhibit that the morphology surface of the sample is immense porosity and agglomerate particles while Fig. 1 (b) showed the surface was lower porosity and agglomerate particle on the sample. This is due to the morphology of sample with addition of process CIP as show in Fig. 1 (b) had even surface compared with the sample without process CIP as shown in Fig. 1 (a).

The remarkable densification, porosity and grain growth were observed from this figure. Fig. 1 (b) showed that it had fewer porosity, homogenous in microstructure and had solid grain arrangement. It proved that the sample with addition of soaked time in CIP present an even surface and better interaction between the zirconia particles. This is due to the good compaction occurred from cold isostatic press machine and the addition of CIP of particle and molecule were closely packed together. Besides, the sample with addition of CIP shows less agglomeration of zirconia particle and its mean when sample was add with CIP process, the particles become fine compared with the sample without CIP. Regarding from the smooth surface for sample that soaked 60 minutes in CIP, it showed particle of the sample were treated efficiently by CIP. Uniform particle arrangement of the sample affect the quality of the surface.

In contrast, Fig. 1(a) showed the sample that had no addition of soaked time in CIP exhibited high porosity and inhomogeneous grain distribution. It showed agglomeration of zirconia particle and inhomogeneous particle interaction due to the surface of the samples were fewer smooth compared to Fig. 1 (b) which is less pressure during compaction process that make the particle of zirconia was not interact effectively. The particle established lower pressure during compaction and forming process that caused difficulty to the particle sample to interact together. More over the sample without addition of CIP just through the hand press compaction. Pressure applied from the top and the bottom of the sample. Lower number of pores were eliminated and particles arrangement was random due to the poor arrangement of particle, high porosity and agglomeration that affect the value of density.

Fig. 2 shows the bar graph for density of samples zirconia block at different soaking time. A samples with addition of cold isostatic press gave the higher value of density compare to controlled sample. Higher value density was because of CIP machine. A sample with soaked time 60 minutes recorded the highest value of density which is 6.1 g/cm<sup>3</sup>. Controlled sample recorded the value of density only 5.6 g/cm<sup>3</sup>. Meanwhile the others two samples with soaked time 90 and 120 minutes give the density value 5.9 and 5.88 g/cm<sup>3</sup>. Density of the sample increase with the increase in cold isostatic pressure.



**Fig. 2. Density values at different soaking time during CIP**

In this case, the different density value is related to the porosity of the samples. A sample with highest value of density had the less porosity. When the sample is added with cold isostatic press, pressure applied to the sample was high. High pressure forming method generate lower porosity and will resulted higher density. Besides, the present of soaking time in CIP machine, it affected the molecule of the sample. Cold isostatic press process contributed effective compaction toward zirconia block sample. Pressure that applied to the sample in cold isostatic press machine come from all directions which mean the compaction of particle and molecule of the sample reached the maximum. As the closely packed the molecules of the sample, the higher the density of the sample.

Eksi et al., (2007) reported in their learned about of firmness and densification action of copper and bronze powders compacted with cold isostatic pressing (CIP) processes, stumbled on who CIP alter led to beat densification [4]. Densification obtained by disorder fragment of one's examine at limited level of pressurization. The thickness enhancement by dislocation was depend on the inspect style. The decreased of interest in porosity may well be refer the confusion. Next in the course of the midway press, supple synthetic deformation occurs on impact of particle contact area. Increasing constrain level increased proceeding of connects and contact area of each particle and ultimately decreased the porosity. Then the final result of sharp pressurization, massive deformation occurs whatever ended in small pores between molecule and particle.

In vice versa, a controlled sample documented lower density value due to low pressure forming method at once generate higher porosity. The controlled sample resulted lower density value because of the value of density related to the arrangement of molecule or particle. Based on graph, a controlled sample, it density value was lowest. The arrangement of molecules for this sample were not closely packed. As a result, the density value was lower. When density was low, its mean presence of open porosity in the sample was high. Water can easily penetrate into the sample. Density of final product usually affected by manufacturing process, such as forming, compaction or pressing and sintering. Without the suitable and efficient compaction will cause the lower density [5]. As from the bar graph results, the sample with high density was the sample with the addition of cold isostatic press machine. This proved the cold isostatic press gave the better compaction toward the sample.

Fig. 3 shows the Vickers hardness (Hv) at different soaking time in CIP for four samples of zirconia block. Based on Fig. 3, it clearly showed that the controlled sample gave the value of hardness 1553 Hv. The samples that soaked for 60 minutes in CIP machine contribute a highest value of hardness compared to the others sample which is 2168 Hv while the hardness value for soaked sample with 90 minutes and 120 minutes soaking time give results of 1742Hv and 1719Hv.

It shows that the soaking time for 60 minutes was the suitable or optimum time to increase a properties of zirconia block sample. This was because by compaction during the sample was soaked in the CIP machine. This improved that addition of cold isostatic press during fabricated our sample

of zirconia block will give positive effect toward our sample which is increasing in hardness value. Furthermore, the

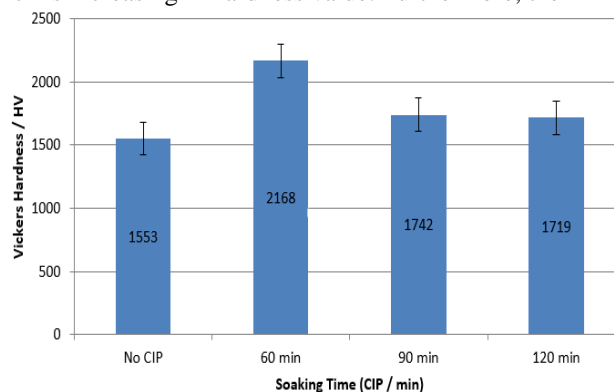


Fig. 3. Hardness value at different soaking time during CIP

sample with the addition of cold isostatic press process exhibited higher matrix stiffness compared to the sample without present of cold isostatic press. According to Shukla et al., 2010, the Vickers hardness value of the sample depended on different sintering temperature [6]. The sintering temperature was fixed. It was different soaking time in cold isostatic press machine. When sample was soaked 60 minutes in CIP machine, the Vickers hardness reached a maximum 2168 Hv. This was because there were fewer pores and small grain after soaked time 60 minutes in CIP machine. As the small grains and fewer pores at the sample, it was increased the value of the hardness. As can be seen in the graph, a controlled sample gave the lowest hardness value among all of the sample which is 1553 Hv due to the sample did not soak in the CIP machine. Without the present of cold isostatic press process, the value of hardness for the sample was not higher as the sample that had been soaked. This is because of the poor compaction process that happen without addition of cold isostatic press process. With no addition of soaking time in CIP machine, the sample was tended to have big grains and more pores.

As resulted, the value hardness of the sample was decreased. However, the samples that been soaked in CIP about 90 and 120 minutes just gave the hardness value 1742 and 1719 Hv. The hardness shows sample with soaking time 90 and 120 minutes resulted almost similar hardness value. Although soaking time in cold isostatic press machine for the sample increased from 60 minutes to 90 minutes, a maximum hardness was found at 60 minutes. This due to the compaction of the molecule occurs at the maximum stage during soaked time of 60 minutes. Molecule and particle packed closely toward each other at sixty minutes soaked time. Due to the hardness based on the graph, the soaking time for 60 minutes was the best for the sample zirconia block to get highest hardness value. The mechanical properties of one's ceramic examine relied on the imperative press and pressing time process parameters. The relationship between the particular treat frameworks would be decided the sternness and thickness of the sample. The parameter that one had been regulate was show of soaking era in CIP mechanical device.

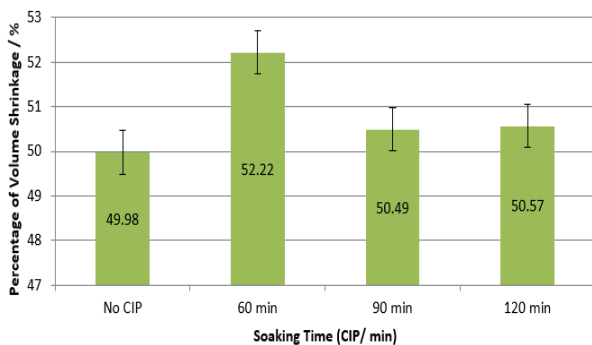
From the outcome in Fig.3,



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it verified that cold isostatic press activated wonderfully strong pressure consistently in all orientation to inspect and that were steady in quantity all through their cruise sections compared to the ones uni-axially movement. This ended in also uniform density and larger compaction that give more advanced sense of hardness. The press who had been used for the CIP mechanical device during this experiment was 2400 kg f/cm<sup>2</sup> and that was a higher value. The previous research also said higher pressure of CIP machine yielded better properties [7]. Therefore, high pressure of compaction from CIP process clearly support the result of mechanical properties, because the sample zirconia block with the addition of CIP show the highest hardness and high density compared to the sample without addition of CIP. As the density of the sample decreased, the hardness value of the sample also will decrease.

Fig. 4 show the result of percentage of volume shrinkage at different soaking time during CIP process. The bar graph showed different percentage of volume shrinkage (%). The controlled sample which was not undergone CIP show the lowest % of volume shrinkage at 49.98%. Meanwhile, sample that soaked at 60 minutes show the highest shrinkage volume which was at 52.22%. For samples soaking time at 90 and 120 minutes show almost same shrinkage % which were 50.49 and 50.57 accordingly.



**Fig. 4. Percentage of volume shrinkage at different soaking time during CIP**

According to Dend et al., (2007), the agglomeration resulted in bounded non uniform shrinkage and the large pores are believed to invent originating at the massive inter agglomerated particles and bigger microstructure non uniformity [8]. Hence, higher agglomeration occurs, percentage volume of shrinkage lower. Then higher agglomeration in sample make the density value was lower. When the density was lower, then the hardness of the sample was lower. Meanwhile, the sample with addition of soaked time within 60 minutes in CIP showed high percentage of volume shrinkage. Due to that, the agglomeration in that sample was lower. Less agglomeration and small pores lead to increase of volume shrinkage. Lower agglomerated leads to increase the density and at the same time increased the hardness of the sample. M. Hanisah et al., (2017) mentioned that the percentage of volume shrinkage of the sample influenced by sintering temperature [9]. The sintering temperature and sintering time affects the grain size of the material. The different percentage volume shrinkage also due

to the different density, different amounts of surface oxide and different particle sizes.

On this research the sintering temperature was fixed at 1300°C. The controlled sample gave the percentage of volume shrinkage 49.98%. No addition of CIP affected the grain size of the sample. Without present of CIP produced sample with bigger grain size [10]. The material of sample becomes less stable and more spontaneous transformation occurs. Because from that the hardness of the sample becomes lower. Soaked in cold isostatic press machine increased the percentage volume shrinkage of the sample. The volume shrinkage increased because of the smaller grain size produced in the sample. When the grain size small, it becomes more stable and increased the hardness of sample.

### IV. CONCLUSION

As a conclusion, based on the Vickers hardness analysis, it showed the hardness value of the sample zirconia block with the addition of cold isostatic press process within 60 minutes soaking time was increased as the higher pressure applied during the compaction process which was 2168 Hv. The CIP process influences significantly the mechanical properties zirconia block sample. Soaking time of 60 minutes proved to be the optimum soaking time for the zirconia sample to get higher value of hardness. According to the result for density testing, it shows the sample with the addition of cold isostatic press process gave higher density value compared to the sample without addition of CIP. The sample with soaking time 60 minutes gave the highest value of density which 6.10 g / cm<sup>3</sup>. Due to this result, presence of CIP process contributed to the changes of microstructure in the sample which make the better arrangement. Because of that, as the uniform arrangement of particles of the sample resulted in the higher value of the density. The percentage volume shrinkage of sample clearly affected the mechanical properties of the sample. With the increased percent of volume shrinkage of the sample will increase the hardness of the sample as a result shown for the soaking time 60 minutes in CIP sample. Meanwhile for the sample without CIP showed lower percentage of volume shrinkage. It caused lower hardness value of the sample. From SEM morphology showed better surface for the sample with addition of CIP compared to the sample without presence of CIP. According to the result in this thesis, it shown the related between hardness, density and percentage volume shrinkage. As the increased the density and percentage volume shrinkage, the higher the strength properties of zirconia block.

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