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# Effect Of Pressure On Density, Porosity And Flexural Strength During Cold Isostatic Press Of Alumina-Ysz-Chromia Cutting Tool

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**ABSTRACT:** This study presented the effect of pressure on the density, porosity and flexural strength when cold isostatic press (CIP) was applied to compact the ceramic powders in the form of insert cutting tools. Specific composition of alumina ( $\text{Al}_2\text{O}_3$ ) wt.90%, yttria stabilized zirconia (YSZ) wt.10%, chromium oxide ( $\text{Cr}_2\text{O}_3$ ) wt.0.6% and polyethylene glycol (PEG) wt.0.6% were ball milled and hand pressed to form green body of ceramic inserts. These green body were undergone further compaction inside CIP with pressures variation of 200 MPa, 300 MPa, 400 MPa with 30 seconds and 60 seconds pressuring time. The ceramic compacts were then sintered at  $1440^\circ\text{C}$  for 9 hours before being assessed with density, porosity, Rockwell hardness (HRC) and bending test. The results show that CIP use with 300 MPa parameters with 60 seconds shows the best mechanical properties with relative density 95.5%, porosity 4.5% and HRC 65.5 hardness. Further assessment of microstructure revealed that the particles size distributed evenly along fracture surface with coarse grain and porosity dominant in the certain area.

**KEYWORDS:** Recycled-Unused Disposable Diapers; Recycled-Polymer Blend; Mechanical Properties; Thermal Properties; Morphological Properties

## 1. INTRODUCTION

To promote sustainable machining process, the use of ceramic cutting tools being an interested subject as their usage significantly related with dry cutting environment that support green technology in the manufacturing industry. Ceramic cutting tools reportedly not only has high stiffness, good machining ability and high wear resistance, but also high hot hardness which makes this cutting tools can be applied in machining process that require resistance at high temperature.

Among many ceramic based cutting tools that available in industry, alumina dominated the low cost category. When sintered, particles of alumina expanded and interlocked between grain



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boundaries to form solid and dense structure [1-3]. Densification of powders compact may benefit the improvement of hardness and flexural strength of powders compact. Both characteristics are important in machining as harder cutting tools may improve the wear resistance which makes the cutting tool able to be used at higher cutting speed. On the same time, better flexural strength may improve the resistance to breakage which make the cutting tool able to be used at higher feed rate and depth of cut.

Even though some researchers stated that alumina based cutting tools suffered with low fracture toughness [4-6] secondary material addition such as zirconia or yttria stabilized zirconia may enhance the mechanical properties up to the level that provided competency in wear performance. In recent study, tertiary addition such as chromia being added in alumina-zirconia cutting tools to improve their abrasive capability. The addition of chromia on alumina structure may increase fracture toughness of ceramic compacts [7-9].

In development of three phase ceramic cutting tools, specific powders should be carefully processed with adequate pressure to obtain high density other than the ceramic powder mixture itself. The usage of cold isostatic press (CIP) may assist the densification of ceramic cutting insert with adequate pressures. CIP works with pressurized air that blasted from all-round direction to compact the ceramic powders [5].

In this study, the properties of alumina-YSZ-chromia cutting tools have been assessed when compacted inside CIP under different pressures. In particular, the density, hardness and flexural strength have been presented together with the details of microstructure. The study provides initial findings to developed alumina-zirconia-chromia cutting tools for dry machining application.

## 2. Materials and Methods

### 2.1 Sample Preparation

This ceramic powder were weighed according to Table 1 by using the electronic weighing scale before mixing together. After that, the mixed powder were poured into a Teflon (PTFE) bottle. The ratio between the ceramic powder and the bottle space is 1: 3 and so alumina or stainless steel balls insert into the ceramic bottle sealed. The space in the bottle should be left 1: 3 for the grinding and milling process.

**Table 1:** Percentage of ceramic powder to be weight

Powder	Percentage (%)
$\text{Al}_2\text{O}_3$	90
YSZ	10
$\text{Cr}_2\text{O}_3$	0.6
PEG	0.6

### 2.2 Grinding and Seiving

The mixed powder were milled in a planetary ball mill machine. The purpose of the ball mill is to mix the powder until the powder becomes homogeneous. The ball mill machine also acts like a grinder that is used to combine and grind the mixture powder. The ball mill is partly filled with grinding media, which is alumina ball or stainless steel ball. The alumina ball works with the principle of shifting and impact, which fall off near the top of the skin when the machine rotates

at 40 RPM. Therefore, the size of the ceramic powder can be reduced after the effect of the ball mill process. This process was took about 9 hours to complete and the powder was dried below room temperature after the milling process.



**Figure 1:** Ball mill machine for grinding the mixed ceramic powder

After mixing and drying process, mixed powder were poured and the alumina grinding ball was cleared by a small brush. Then mixed powder followed by sieving with mesh. Although the grinding of the powder with the ball mill can provide a good particle, but refining is still needed to isolate the unwanted components. Samples will be placed on a sieve powder containing openings with a  $75\mu\text{m}$  (200 mesh) and the sieve help to separate the materials to be agglomerate in addition to isolating impurity fine particles can pass through the opening. Separation process was filtered out coarse particles and large chunks of materials; while removing fine particles.

### 2.3 Pressing and Cold Isolatic Press (CIP) Process

Before pressing process, each sample required 3.5 grams of mixed powder was weighted by using electronic weighing machine. After that, weighted powder was poured into the mold and pressed by mechanical press. The pressure was applied slowly until 5 tones. The pressing was hold for 1 minute for every 3 tones of the applied pressure. The purpose is to ensure there is enough time for particles to compact together and eliminate the pores within the green sample. The sample was taken out carefully from the mould to avoid the cracking of green sample. A pre-shaped of green sample was produced by this method. After the pre-shaped of green samples has been obtained, the sample was pressed by CIP machine as be seen Figure 2. The applied parameter was shown in Table 2.

**Table 2:** Parameter setup for CIP machine

CIP pressure (MPa)	Dwell time (s)
200,300,400	30,60



**Figure 2:** CIP Machine

#### 2.4 Sintering

The last process of sample preparation is a sintering process, sintering perform by using high temperature furnace. Sintering is a mechanical process of material densification through mass transfer likes atom diffusion and grain growth under a high temperature. In this experiment, temperature is rammed up at 3°C/min until reach 1400°C and followed by the soaking time as long as 9 hours. Once the sintering process is done, the samples are gradually cooled down at 3°C/min until the temperature cooling as surrounding before the furnace is opened.

#### 2.5 Material testing

After the specimens were fabricated, the effects of different CIP pressure and dwell pressure time on the material properties of samples was investigated through a few tests. Study on relative density, Rockwell hardness (HRC), bending test and microstructure of materials were carried out to determine the effect of CIP parameters on the alumina-based cutting tool.

### 3. Results and Discussion

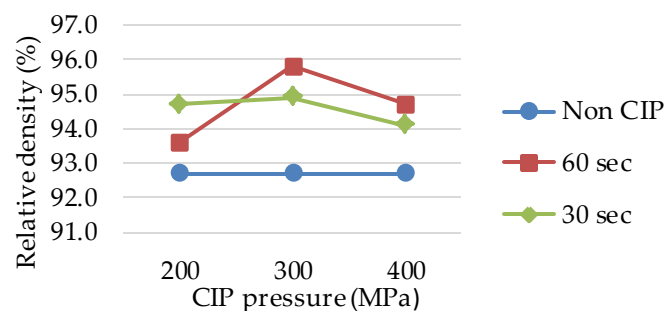
Thus, the experiment responses such as density, hardness, microstructure, flexural strength and machinability were evaluated and effects of the samples were analyzed.

#### 3.1 Effect of CIP on relative Density and Porosity

The graph shows that the relative density of samples with 30 sec and 60 sec dwell pressure times gradually increase with the increasing of pressure from 200 to 300 MPa. According to Amat et al. [10], stated CIP pressure will affect the density of alumina specimens. In the beginning stage of CIP, those huge pores will be eliminated and the orientation of alumina particles will be rearranged. The green body gradually increases the density and the reach highest densification rate. As the CIP pressure further increased, the alumina particles were crushed, compressed and filling into the smaller pores in the green specimen. The contacted area within particles also will increase as the isostatic pressed applied to the particles. This stage was occurring when the pressure applied from 200 to 300 MPa. The relative density of both samples (30 and 60 sec dwell pressure time) had decreased with the increasing of the CIP pressure from 300 to 400 MPa. This is because as the CIP pressure continued to increase, the phenomenon of strain hardening within the particles occurred. The increasing of surface contact area within particles had led to the growth of deformation resistance. Therefore, the densification rate had slowed down. The relative density should be increase as the increasing of CIP pressure [10]. However, cracks might be occurred in the sample as the CIP pressure is too high. Clearly, this study revealed that 300 MPa is the optimum dwell pressure time for CIP instead of 400 MPa and the

Figure 3 shows the effect of CIP pressure with relative density of ceramics mixing.

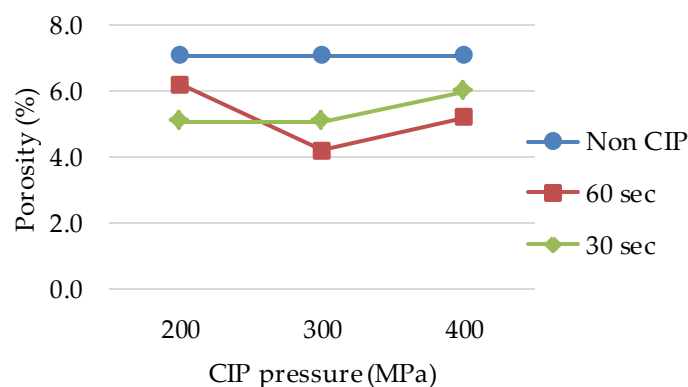
As overall, sample that is cold isostatic pressed with 60sec dwell pressure time had achieved the higher relative density, whereas above than the samples with 30sec dwell pressure time. The reason is because longer dwell pressure time had provided more sufficient time for small particles to fill in pores. This result has attempts to correlate that CIP specimens with 60sec pressing dwell pressure time is the optimum parameter to produce a high density sample. Yet, the relative density of the sample was higher than the 60sec dwell pressure time at 200 MPa.



**Figure 3:** The effect of CIP pressure with relative density of ceramics mixing

This result had opposed the general theory which could cause by experiment and equipment error. As a conclusion, green specimen that cold isostatic pressed at 300 MPa with 60 sec dwell pressure time is the best parameter among to the other parameter, whereas the sample had achieved 95% of the theoretical density when sintered at 1400°C temperature.

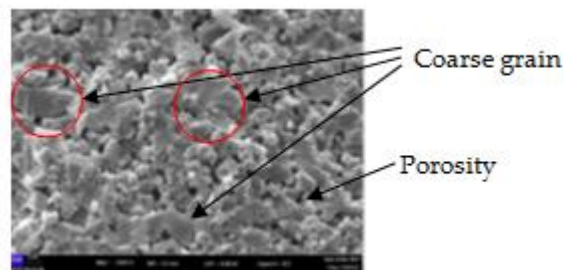
Figure 4 shows that the percentage of apparent porosity of samples. Non-CIP samples had shown the highest value percentage of porosity which is 7.2%. As the percentage of porosity is contrasted with the relative density; hence, the CIP samples showed much lower porosity value compares to non-CIP samples. The reason is same as the evaluation, which the CIP samples have better densification as compared to non-CIP samples. The pores within the particles are eliminated through the cold isostatic pressing process when the high pressure was compacted the particles from all the directions. Again, this result proved that the CIP samples had decreased the one percentage of porosity.



**Figure 4:** Porosity comparison percentage

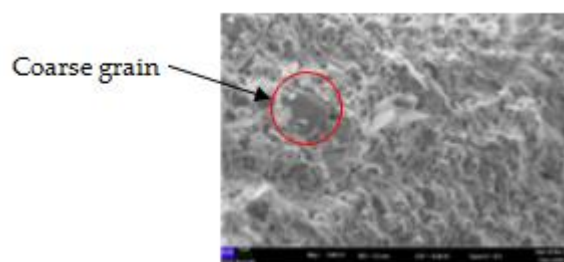
### 3.2 Microstructure

Microstructural studies between non-CIP samples and CIP samples were conducted and analyzed. Figure 5 shows the SEM cross section sintered at 1400 °C using focusing 2 $\mu$ m and 2000 mag. From observation, there are many grain microstructure that emerges and substantial porosity among ceramic grains and this gives the impression that cutting tools using mechanical pressure cannot contend that the binding between the grain perfectly. Coarse grain and uneven distribution are one of the reasons for weak bending strength in non-CIP samples [11,12]. The porosity in the sample can weaken the bending strength through the broken mode and make the sample more fragile and may interrupt on machining process.



**Figure 5:** SEM top surface by using uniaxial press samples

In the other hand, Figure 6 shows the SEM microstructure on the cross section of the ceramic cutting tool with a 30 sec dwell time at pressure 300 MPa. This is the sample with best density among other CIP samples, although there has some coarse grain, but the grain closes each other and eliminates porosity. From the observation, the microstructure is more homogenous than the non-CIP sample. The formed grains are fine & homogenous and seem bonded tighter than the non-CIP sample. The intragranular fracture and some of the grains are pulled out can be observed on the fractured surface. This is because the particles tend to be strengthened by binding together. This could be explained the reason that the flexural strength of this CIP-sample is better than the non-CIP [6]. In general, as the samples were cold isostatic pressed, pores will be eliminated as the high pressure compact the particles to bond together. However, some huge pores and intragranular pores still appeared within the particles or the grains. This was caused by the nonuniform agglomeration of initial powder during the uniaxial pressing [13].



**Figure 6:** SEM cross-section on 300 MPa at 30 sec dwell time

This present microstructure study also correlated to the densification result of samples. It could find that relative density of CIP samples higher than non-CIP samples. One of the reasons was that only few pore and intragranular pores could be found in the CIP sample as compared to non-CIP sample. The study had indicated that pores had such a huge influence on the density of the alumina material base.



### 3.2 Hardness

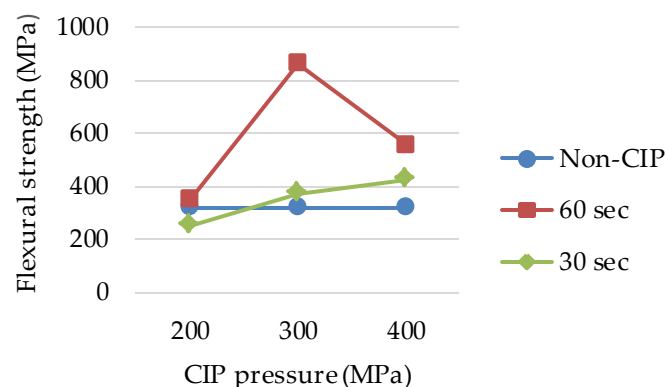
The effect of method of pressing on the Rockwell hardness of alumina samples is shown in Table 3. Based on the theory, the relationship between relative density and Rockwell Hardness (HRC) is directly proportional because the densification is the crucial that had influenced the mechanical properties of specimens. The CIP samples in 300 MPa and 400 MPa obtained higher values as compared non-CIP sample. The hardness for non-CIP sintered sample had shown the lowest HRC, which is 58 and this causes led low density behavior on ceramic cutting tool. This was because the non-CIP sample with low relative density contained of high quantity of pore that had weaken the strength in microstructure to withstand high load at 150 Kg/force. Moreover, the effect of relative density to HRC is more obvious for CIP sample with 30 sec dwell pressure time as compared to the CIP sample with 60 sec dwell pressure time at every particular CIP pressure.

**Table 3:** Rockwell hardness of ceramic cutting tool

MPa	Rockwell Hardness (HRC)	
	30 sec	60 sec
200	54.5	59.2
300	60.2	65.5
400	61.3	63.3
Non-CIP	58	58

### 3.4 Effect of CIP on Flexural Strength

Flexural strength are measured and analyzed to determine the effect of CIP parameters on the mechanical properties of the samples. From Figure 8, linear trend increases from both sets of CIP samples (30 seconds and 60 seconds of dwell pressure time) from 200 MPa to 300 MPa, however, for 400 MPa and 60 seconds of dwell pressure time it decreases significantly. This is because it depends on the relative density of the sample. The flexural strength relationship is directly proportional to the relative density of the sample [14].



**Figure 7:** The Effect of CIP Pressure on Flexural Strength of Alumina Sample

According to Ćurković et al. [3], stated the flexural strength of the non-CIP sample will affect by coarse grain. The formation of intragranular pores and holes will also reduce the grain boundary strength and cause fractures when the load is applied. Although the microstructure of the non-CIP sample is clear in the phenomenon, the flexural strength of the non-CIP sample with manual pressure of 9 tonnes shows a bending strength value of 320 MPa higher than the CIP 200 MPa pressure. This data shows that CIP pressure has a high impact on the green body with appropriate pressure conditions, even though manual pressure is charged with a high tone, so



300 MPa clearly shows the pressure strength along with the appropriate pressure time to form the green body strength and so it will suitable for developing cutting tool for machining apply.

#### 4. Conclusion

This paper presents the fabrication of the alumina-YSZ-chromia insert ceramic cutting tool that compacted with pressures variation of 200 MPa, 300 MPa, 400 MPa with 30 seconds and 60 seconds pressuring time. These green bodies were sintered at 1400°C in 9 hours soaking time. Based on the experimental finding the following conclusions can be drawn:-

1. Ball mill process help refining the particle size of alumina-YSZ-chromia powder and made it easier to be compacted in the mold as the shape of the powder is the same which help to fill any porosity while compaction process is done.
2. There are various trend line of relative density for each applicable parameter, however the highest relative density has been achieved is 95.5% and 4.5% of porosity where the CIP pressure was at 300 MPa with 60 sec dwell time.
3. It was found that the particle distributed evenly along the fracture surface with coarse grain and porosity dominant in certain area. The highest hardness value achievable by this process is 65.5 HRC hardness with CIP pressure 300 MPa at 60 sec dwell time.
4. Noted that the flexural strength trend are various upon the parameter usage and the highest strength was 865.2 MPa when the CIP pressure at 300 Mpa with 60 sec dwell time.

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