

# Reducing The Risk of Agglomeration and Shrinkage Ceramic Body from $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ Composition

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## ABSTRACT

*This study introduces the effect of ceramic composition that consists of Alumina ( $\text{Al}_2\text{O}_3$ )-Zirconia ( $\text{ZrO}_2$ )-Chromia ( $\text{Cr}_2\text{O}_3$ ) compositions on agglomerate and shrinkage for cutting tool development. Shrinkage is a problem in the development of ceramic cutting tools other than the occurrence of particle agglomerate on the body structure. Finer grain size significantly contributes to the ceramic body's shrinkage and agglomeration. This study analyzed grain size and its relationship with shrinkage and agglomerates. The powders were ball-milled with 80 wt%  $\text{Al}_2\text{O}_3$  -20 wt%  $\text{ZrO}_2$  -0.6 wt%  $\text{Cr}_2\text{O}_3$  and then compacted and sintered at 1400 °C to examine their shrinkage and investigate microstructure by scanning electron microscopy (SEM) machine. The results show that  $\text{ZrO}_2$  has a larger particle size of 6.10  $\mu\text{m}$  and  $\text{Cr}_2\text{O}_3$  has a finer measure of 1.24  $\mu\text{m}$ . When blended with the ball mill, the mix of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  was obtained is 7.30  $\mu\text{m}$ , showing that the ball mill can uniformly mix all the particles and reduce the risk of agglomeration. The microstructural analysis found that  $\text{Cr}_2\text{O}_3$  covers and fills up the space between  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  compared to without  $\text{Cr}_2\text{O}_3$ . The combination of agglomerate and shrinkage of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  was responsible for the performance of the ceramic cutting tool fabricated.*

**Keywords:** Agglomeration; Shrinkage; Ceramic; Cutting Tool

## Introduction

Engineering or advanced ceramics are inorganic non-metallic materials widely used in high-performance engineering applications. The production of advanced ceramic gives the ideal arrangement and a practical, superior other option to conventional materials, for example, metals and plastics. Ceramics can be called oxide-based, carbides, nitrides, and borides. According to Salamon [1] and Bala et al. [2], the desired properties of advanced ceramics are wear resistance, stability against thermal resistance, thermal insulation, electrical insulation, and non-magnetic. Through these properties, ceramic cutting tools are capable of machining AISI 1045 [3]. Besides that, industrial ceramics applications include automotive components, medical, insulation materials, shielding materials army, coating materials, nuclear reactors, and artificial bone.

Nowadays, most ceramic products use  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  materials because the nature of the two materials enables the production of a compact and sturdy structure. The use of  $\text{Al}_2\text{O}_3$  powder in the fabrication of ceramic cutting tools is sufficient to produce a robust structure to carry out lathe machining work [4]-[5]. According to Tong et al. [6], Shafeiey et al. [7], and Tuan et al. [8], the refined grains and mechanical properties of the  $\text{ZrO}_2$  structure can improve the strength of  $\text{Al}_2\text{O}_3$  ceramic cutting tools. However, there is a challenging issue when using nano-sized  $\text{ZrO}_2$  powder. A high tendency to agglomerate between grains can cause a decrease in the mechanical performance of ceramic cutting tools. As a result of the agglomeration,  $\text{ZrO}_2$  is not dispersed uniformly in the composition of the green body [9]. Agglomerate is inevitable due to the fineness of the ceramic powder used in the composition and the need to analyze the condition and its effects on the ceramic powder mixture. The additive material should be considered and studied to strengthen the composition of the  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  ceramic  $\text{Cr}_2\text{O}_3$  body, such as using a powder. One of the powerful mixtures and additives used for ceramic cutting tools is  $\text{Cr}_2\text{O}_3$  [3]. According to Manshor et al. [10] and Kunkun et al. [11],  $\text{Cr}_2\text{O}_3$  is added to  $\text{Al}_2\text{O}_3$  to provide fracture toughness because it can form a solid isovalent solution.

Green body shrinkage occurs when the particle size changes due to the effect of the sintering. Shrinkage refers to a reduction in the size of the compacted green body after sintering. It is caused by the closure of porosity inside the ceramic body during the sintering process [12]. The microstructure of the ceramic cutting tool is initially in the small grains form, then continues to expand during the sintering process. The sintering process affects the shrinkage rate due to the molecular and grain movement of the solid ceramic body during the sintering process [13]-[14]. There are three stages of transformation during the sintering of ceramic cutting tools. In the initial stage, there are some degrees of atomic mobility among grain particles, and sharply concave necks are formed between individual particles. The process occurs

when the grain size changes at the initial stage of sintering, where the microstructure of the ceramic cutting tool consists of porosity between the grains. As the sintering process continues, the pores between the grains continue to close as the grains expand to cover the spaces and voids. This condition causes shrinkage because the pores have been decomposed, and the grains grow to their maximum level (depending on the sintering temperature) [15].

As a result of this phenomenon, the structure of the ceramic cutting tool becomes denser, and it has better physical and mechanical properties. The shrinkage is calculated based on the diameter and thickness of the cutting tool, as the changes in diameter and thickness are more significant for the cutting tool to fit into the holder.

## Experimental Procedure

The mixing process of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Cr<sub>2</sub>O<sub>3</sub> compositions is done using the dry method. Each ceramic powder is weighed evenly with a 4 gram, then placed in a bottle/jar and mixed evenly using a ball mill machine with a 40 rpm rotation speed at 9 hours of rotation time. This ball mill machine produces a finer and uniformly powder mixture. The powder is then poured into a mould and compacted using a mechanical press and press up to 5 tons to get the determined shape of the green body. A Cold Isostatic Press (CIP) machine is used with 300 MPa for 30 seconds to further compact the green body. Then, the green body of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Cr<sub>2</sub>O<sub>3</sub> is sintered up to 1400 °C for 9 hours to obtain a solid and compact ceramic body. Figure 1 shows the process of fabricating ceramic cutting tools.



Figure 1: Process cutting tool development

X-ray diffraction (XRD) is an analytical method used to identify the phase of the crystalline material. In this study, XRD was used to detect the presence of  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ , and  $\text{Cr}_2\text{O}_3$  elements inside a single ceramic body. Figure 2 shows the XRD machine used in this study.

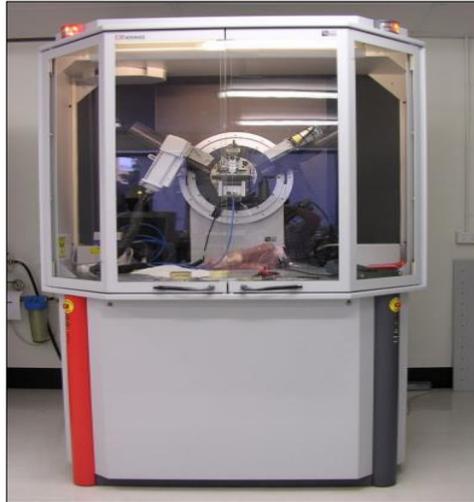


Figure 2: X-ray diffraction (XRD) brand D8 Advance model MSE 4003

### Measurement

The grain size and surface contact were measured on each ceramic powder, and then the ceramic mixture was analysed using Malvern master-sizer equipment, as shown in Figure 3. Combination focused 80 wt%  $\text{Al}_2\text{O}_3$ -20 wt%  $\text{ZrO}_2$ -0.6 wt%  $\text{Cr}_2\text{O}_3$ .



Figure 3: Malvern master-sizer

Specimens were measured at three places using a digital calliper for diameter and thickness before the sintering process was carried out and then averaged. The average diameter and thickness before the sintering process is

carried out are 12 mm and 6 mm, respectively. After the sintering process, the same measurement method is performed to obtain an average value to be compared before and after sintering and converted to a percentage. Figure 4 shows the specimen before and after the sintering process is carried out. While for analysis, microstructure and agglomerate identification were carried out using SEM machines, as seen in Figure 5.

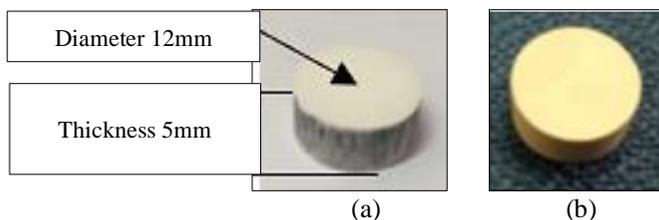


Figure 4: Ceramic cutting tool; (a) before sintering, and (b) after sintering



Figure 5: Scanning electron microscopy (SEM)

## Results and Discussion

The study was conducted by identifying the grain size, shrinkage rate, agglomeration, grain contact surface, and microstructure analysis of fabricated ceramic cutting tool by comparing two compositions of  $Al_2O_3$ - $ZrO_2$  and  $Al_2O_3$ - $ZrO_2$ - $Cr_2O_3$ . Shrinkage identification was performed to identify the effect on the composition of each powder. At the same time, microstructure analysis determines the causes and consequences of the composition used against powder agglomeration. Powder identification is the first step and the subsequent study about the structure's agglomeration and the effect that causes shrinkage.

### **Powder characteristics**

Figure 6 compares the average grain size after mixing  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  and  $\text{Al}_2\text{O}_3\text{-ZrO}_2\text{-Cr}_2\text{O}_3$ . Since the size of  $\text{ZrO}_2$  used in this study only presented around 20% compared to  $\text{Al}_2\text{O}_3$  (80%), it is expected that the fine  $\text{ZrO}_2$  covers the space between the necks of  $\text{Al}_2\text{O}_3$  grains.  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  materials can react chemically with each other. However, they have different grain sizes and shapes to produce a compact structure when compaction and sintering. While the properties of the ceramic body from the mixture of  $\text{Al}_2\text{O}_3\text{-ZrO}_2\text{-Cr}_2\text{O}_3$  powders depended on the grain distribution, which is critically influenced by the processing method. Adding  $\text{Cr}_2\text{O}_3$ , which has a finer grain size and can react chemically to the composition, can close the space between the  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  neck during the sintering process.

Agglomeration strongly depended on the surface contact area between particles. A bigger surface contact area increased the attraction between the particles due to stronger Van Der Waals forces [16]. As shown in Figure 7,  $\text{Cr}_2\text{O}_3$  has the largest surface contact area of  $4.84 \text{ m}^2/\text{g}$ , which means this  $\text{Cr}_2\text{O}_3$  has the strongest tendency to form an agglomerate. On the other hand, the particle size of  $\text{Al}_2\text{O}_3$  which is dominant in the mixture, has  $0.0574 \text{ m}^2/\text{g}$ , the lowest tendency to form an agglomerate. When  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  and  $\text{Al}_2\text{O}_3\text{-ZrO}_2\text{-Cr}_2\text{O}_3$  powders ball-milled together, the average surface contact area obtained is  $0.61 \text{ m}^2/\text{g}$  and  $0.82 \text{ m}^2/\text{g}$ , respectively, and slightly lower than  $\text{ZrO}_2$  ( $0.983 \text{ m}^2/\text{g}$ ). This indicates a tendency for  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  to agglomerate almost equivalent to  $\text{ZrO}_2$ .

### **Agglomeration**

Agglomerate can be prevented using the grinding/ milling method [17]-[18]. Using a ball mill machine is one of the grinding methods indirectly capable of reducing agglomerated powder with a rough surface to a finer material and can mix uniformly with other ceramic powders. During the ball milling process, the ball mill machine grinds and crushes the ceramic powder and mixes it evenly [19]-[20]. The rotational impact from the  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  mixture makes the particles blend homogeneously in the powder bed (bottle/jar) to produce uniform grain distribution for the whole cutting tool structure [21]. The reduction of the  $\text{Cr}_2\text{O}_3$  surface contact area from  $4.84 \text{ m}^2/\text{g}$  to  $0.82 \text{ m}^2/\text{g}$  after  $\text{Al}_2\text{O}_3\text{-ZrO}_2\text{-Cr}_2\text{O}_3$  ball milled shows that using this technique capable of removing soft agglomerate that appears in the powder mixture and Figure 8 shows an example of agglomeration of small powder.

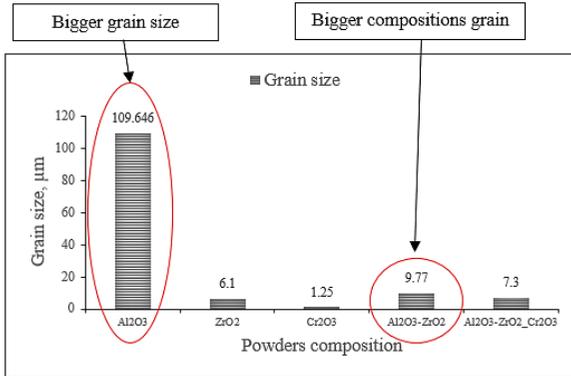


Figure 6: Comparison of average grain size

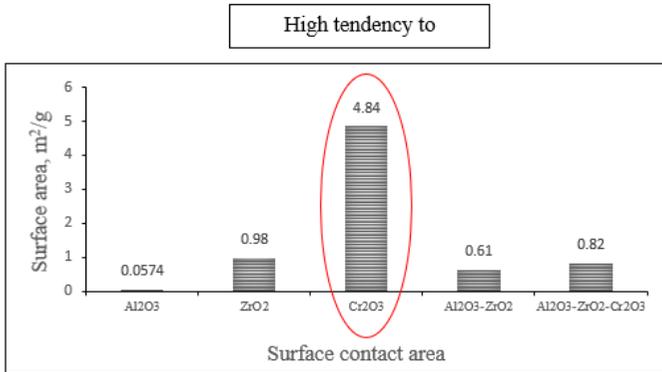


Figure 7: Comparison of surface contact area

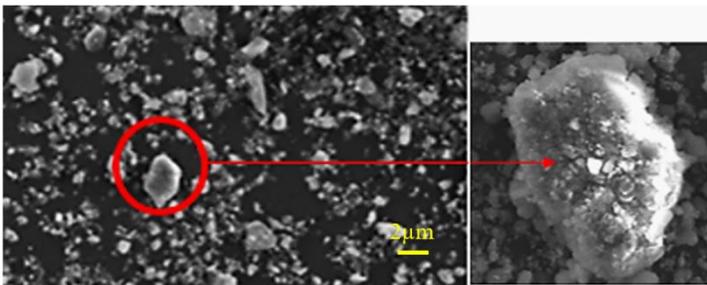


Figure 8: Agglomeration of small powder

Figure 9(a) shows an X-ray diffraction (XRD) pattern of the percentage difference between  $ZrO_2$  against  $Al_2O_3$  content. Analysis shows that  $Al_2O_3$  and  $ZrO_2$  can merge or consolidate because of chemical reactions during when sintering process. While Figure 9(b) indicates that  $Cr_2O_3$  can react with  $Al_2O_3$  when sintered. It's in line with the study conducted by Manshor et al. [10];  $Al_2O_3$  reacts with  $Cr_2O_3$  because it can form a solid solution isovalent. However, Figure 7 generally does not show  $ZrO_2$  and  $Cr_2O_3$  consolidating or responding. Since Cr is hardly soluble in Zr, the ion of Cr should be dissolved and diffuse through the surface of  $Al_2O_3$ .  $Cr_2O_3$  is added to the  $Al_2O_3$ - $ZrO_2$  composition that has been dissolved when the sintering process is carried out [22]. This vaporised  $Cr_2O_3$  could be heterogeneously distributed to the upper surface area of  $Al_2O_3$ - $ZrO_2$ , resulting in partial grain growth to the surface, and some of the  $Al_2O_3$  grain can consolidate and merge with  $Cr_2O_3$ .

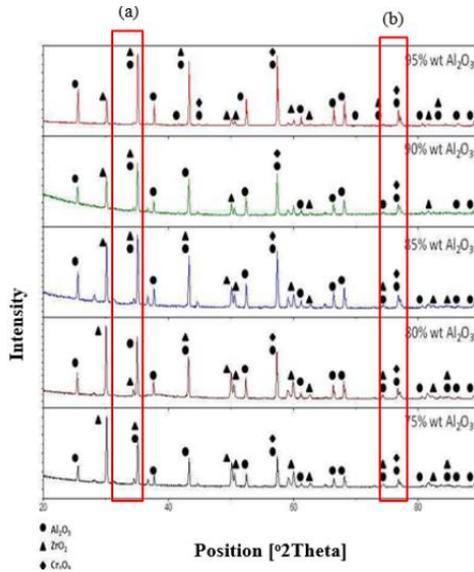


Figure 9: XRD pattern wt.%  $ZrO_2$  on  $Al_2O_3$

The microstructure composition in Figure 10(b) shows more compactness with a significant reduction in porosity on the surface of the ceramic cutting tool.  $Cr_2O_3$  can help cover the spaces between  $ZrO_2$  and  $Al_2O_3$  grains on the necks. The diagram can be interpreted clearly that 0.6 wt%  $Cr_2O_3$  is enough to accelerate grain growth and can help strengthen the composition of  $Al_2O_3$  80 wt% and  $ZrO_2$  20 wt%. Compared with Figure 10(a), there is much porosity on the surface, and proved by the cross-section in Figure 11(a), the cross-section has much porosity and is seen like patches inside the cutting tool.

However, each grain is seen to be firmly bonded to each other and is better than Figure 11(b). This phenomenon is due to the evaporation that occurs on  $\text{Cr}_2\text{O}_3$  in the ceramic body. The sintering process causes  $\text{Cr}_2\text{O}_3$ , in the composition of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  in the solid body, to evaporate and redeposit over the ceramic surface, which in turn reacts to the  $\text{Al}_2\text{O}_3$  on the ceramic body surface [23]. The addition of  $\text{Cr}_2\text{O}_3$  to  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  significantly affects the quality of ceramic cutting tools; the  $\text{ZrO}_2$  proportion is ideal for reinforcing the grain position, and the addition of  $\text{Cr}_2\text{O}_3$  helps to strengthen the surface of the cutting tool by covering the porosity between  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$ .

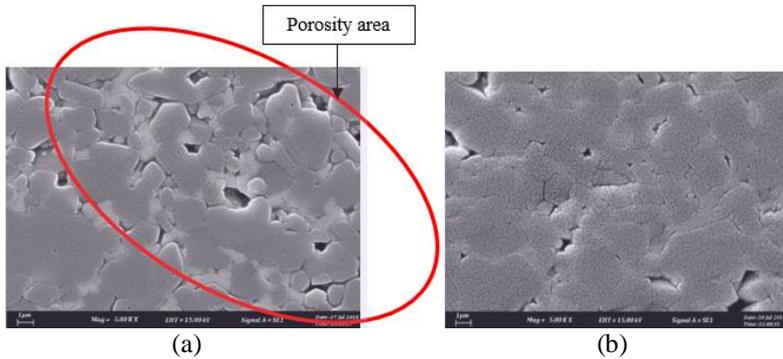


Figure 10: Surface comparison between (a)  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ , and (b)  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$

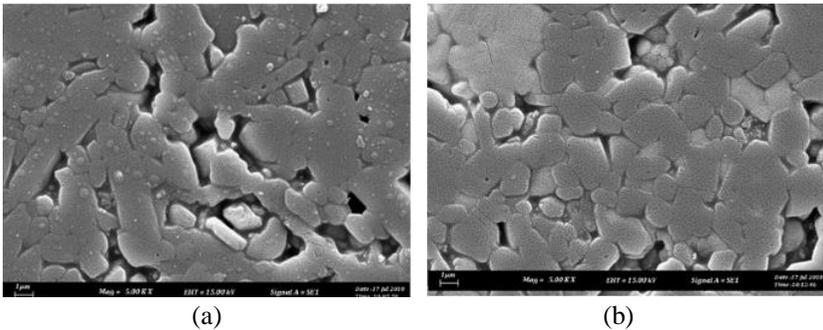


Figure 11: Cross-section comparison between (a)  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ , and (b)  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$

### Shrinkage

Table 1 shows a composition between  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$ , while the significant difference in diameter shrinkage between  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  can be seen in Figure 12. The shrinkage of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  composition is higher

than  $\text{Al}_2\text{O}_3\text{-ZrO}_2\text{-Cr}_2\text{O}_3$ . The apparent difference is due to the presence of  $\text{Cr}_2\text{O}_3$  on  $\text{Al}_2\text{O}_3\text{-ZrO}_2$ , which reinforces the surface of the ceramic mixture. The  $\text{Cr}_2\text{O}_3$  added to the  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  materials evaporates during the firing process. Then the  $\text{Cr}_2\text{O}_3$  covers the surface of the ceramic mixer, which becomes more robust and has the advantage of wear resistance and heat resistance [12]. According to Renold and Ramesh [24], the shrinking will change during the sintering process by adding additives to the ceramic mixture, and the size of the additive particles affects the order of ceramic shrinkage. Using  $\text{Cr}_2\text{O}_3$  as an additive to  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  is very helpful in toughening the synthesised ceramic body through the ceramic body observation, as seen in Figures 8 and 9.

Table 1: Powder composition between  $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ , and  $\text{Cr}_2\text{O}_3$

Composition	$\text{Al}_2\text{O}_3$ (%)	$\text{ZrO}_2$ (%)	$\text{Cr}_2\text{O}_3$ (%)
A	95	5	
B	90	10	
C	85	15	0.6
D	80	20	
E	75	25	

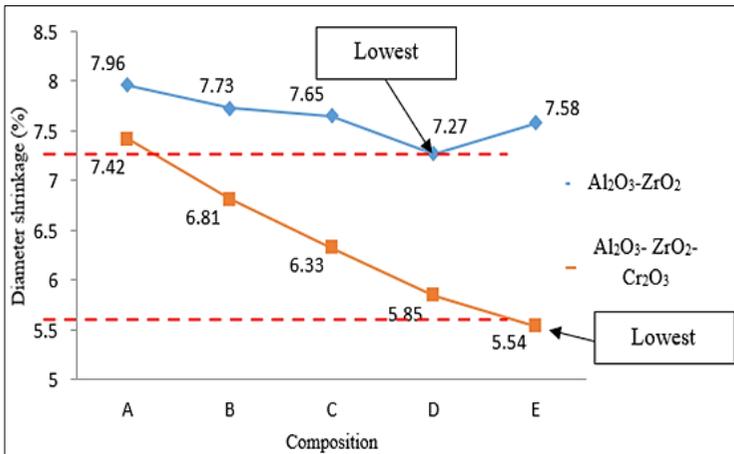


Figure 12: Comparison of diameter shrinkage

The addition of  $\text{Cr}_2\text{O}_3$  against  $\text{Al}_2\text{O}_3\text{-ZrO}_2$  is seen to have much impact on the thickness shrinkage, as seen in Figure 13. The addition of the  $\text{Cr}_2\text{O}_3$  powder increases the strength of the surface of the ceramic cutting tools. However, the thickness difference in the cutting tool diameter does not matter very much because it depends on the tool holder used. In many instances, the

thickness of the cutting tool can be changed on the tool holder, which is adjustable in the range of 5 to 7 mm [3]. Although the percentage of thickness shrinkage increases, it does not exceed 10% and is still in the range of shrinkage percentage for  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ . This shrinkage occurs due to adding  $\text{Cr}_2\text{O}_3$  to  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  powder. The addition of  $\text{Cr}_2\text{O}_3$  is seen to help overcome excessive shrinkage; however, the use of  $\text{ZrO}_2$  based on percentage impacts the shrinkage. Shrinkage cannot be avoided because  $\text{ZrO}_2$  blocks vary when sintered; the higher the  $\text{ZrO}_2$  percentage, the higher the percentage of shrinkage that will occur [25].

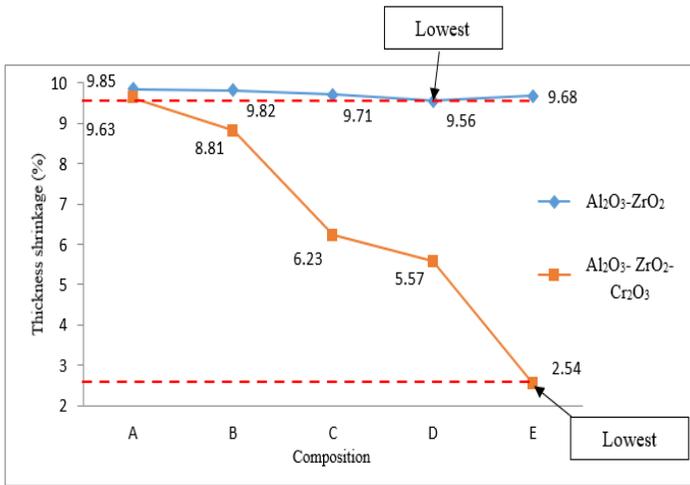


Figure 13: Comparison of thickness shrinkage in millimeters (mm)

## Conclusion

The shrinkage rate comparison between two ratio compositions of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  was studied to identify the effects of the mixture. Agglomeration occurred due to the grain size and the impact of the contact area. It was analysed in detail to determine the cause of agglomerate and shrinkage in the composition used. Further studies need to be carried out with a mixture of other materials that can reduce the shrinkage and porosity resulting from agglomerate by researching a mixture of compositions other than  $\text{Cr}_2\text{O}_3$ , for example, a mixture of Mg, B4N against  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ . Based on this study, it can be concluded that:

- i.  $\text{Cr}_2\text{O}_3$  grains are the smallest at 1.25  $\mu\text{m}$ , and the surface contact is the largest at 4.84  $\text{m}^2/\text{g}$  compared to the grain size of  $\text{Al}_2\text{O}_3$  and  $\text{ZrO}_2$  grains.

- ii. The tendency of agglomeration in single  $\text{Cr}_2\text{O}_3$  grain is very high because of the large surface area due to the small grain size.
- iii. The  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  mixture increased grain size to  $9.77 \mu\text{m}$  and recorded a surface area of  $0.61 \text{ m}^2/\text{g}$  compared to a single  $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$  that only recorded  $6.10 \mu\text{m}$  and  $0.98 \text{ m}^2/\text{g}$ , respectively.
- iv. The  $\text{Cr}_2\text{O}_3$  addition of as much as  $0.6 \text{ wt}\%$  against  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  can help reduce agglomerate, which is the grain size increases to  $7.30 \mu\text{m}$  and surface area of  $0.82 \text{ m}^2/\text{g}$ .
- v.  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  and  $\text{Al}_2\text{O}_3$ - $\text{Cr}_2\text{O}_3$  can chemically react with each other through consolidation. While  $\text{ZrO}_2$  and  $\text{Cr}_2\text{O}_3$  cannot react chemically because  $\text{Cr}_2\text{O}_3$  can't dissolve with  $\text{ZrO}_2$ , there will be evaporation.
- vi. Observation on the microstructure of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  is much denser than  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ , which features high porosity. The vaporation of  $\text{Cr}_2\text{O}_3$ , when sintered, shows that it redeposits to the surface and chemically reacts to the  $\text{Al}_2\text{O}_3$  on the surface of the ceramic body. While the microstructure on the cross-section or the inside of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$  shows porosity, it is still better than the  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  mixture.
- vii. The shrinkage rate of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  is higher than  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ - $\text{Cr}_2\text{O}_3$ , where the shrinkage is very significant with each addition of  $\text{wt}\%$   $\text{ZrO}_2$ . It's caused by the result of the evaporation processes of  $\text{Cr}_2\text{O}_3$  that occurs and leaves an empty space in the composition of the mixture.

## Contributions of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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## Conflict of Interests

All authors declare that they have no conflicts of interest

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