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Influence of Sintering Temperature on Density, Hardness, Shrinkage and Microstructure of Alumina-Zirconia Cutting Tool

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ABSTRACT

The confederation of Alumina (Al_2O_3) and Zirconia (ZrO_2) has emerged as a promising ceramic framework for advanced machine tool applications. The mechanical properties of the Al_2O_3-ZrO_2 cutting tool are critically dependent on its density, hardness, and shrinkage, which are influenced by the powder preparation and sintering processes. The effect of sintering temperature on density, hardness, shrinkage, and microstructure of Al_2O_3-ZrO_2 is investigated in this study. Al_2O_3 powders (80% wt) and ZrO_2 powders (20% wt) were combined in a ball mill machine with polyethylene glycol (PEG) added as a binder agent. The samples were compacted using the cold isostatic pressing method at 300 MPa pressure after being pressed using a hydraulic hand press machine at 10 tons pressure. The green compacts were sintered at temperatures ranging from 1500°C to 1700°C, with soaking times ranging from 4 to 6 hours. The results show that as the sintering temperature increased, the density and hardness decreased. When the sintering temperature was set to 1500°C and the soaking time was 6 hours, the maximum hardness and density were recorded at 1321.23HV. Whereas, lowest hardness was recorded at 688.4HV when the sintering temperature was set to 1700°C and the soaking time was 6 hours. The shrinkage increased significantly as the sintering temperature increased, reaching a maximum of 9.95 percent at 1700°C after 4 hours of soaking. Microstructure analysis reveals that grain size increased as sintering temperature increased, with the smallest grain size presented 2 μm at 1500°C and 6 hours of soaking time.

Keywords: alumina, zirconia, density, hardness, shrinkage, microstructure

1. INTRODUCTION

Machining is the process of removing layers of material by shearing the workpiece material with a cutting tool. Machining is commonly used as a secondary finishing process prior to primary manufacturing processes such as casting, shaping, or forging. In machining, selecting the appropriate cutting tool is critical for producing products with high dimensional precision and fine surface finishing while minimizing operational costs. There are many different types of cutting tool materials on the market, including high-speed steels, cubic boron nitride (CBN), polycrystalline diamond (PCD), cemented carbide, ceramics and natural diamonds. Each of these cutting tools have advantages and disadvantages depended on the grades and application [1-2].

Ceramic-based cutting tools are one of the most commonly used in machining operations. Ceramic-based cutting tools are not only chemically stable, abrasive, and have high strength and hardness, but they can also work in dry conditions. As a result, the use of ceramic cutting tools can reduce the negative impact of cutting fluid on the environment, machinists, and operational costs. The use of ceramic cutting tool in high-speed machining is always subjected to a sudden increase in temperature and workload. Cracking is one of the failure risks, resulting in inefficient ceramic use for machining operations [3-4].

Sintering is a process that solidifies the particle packing of ceramics by heating the materials for a set period of time. This means that the ceramic body is strengthened by firing at specific temperatures so that coherent bonds between particles can be formed [5]. During the sintering process, heat convects through the ceramic structure, causing changes in grain size. The ceramic particles began to expand until they reached the grain boundaries. Due to the prolonged sintering time, the particles diffused each other at the grain boundaries, resulting in porosity removal and structure shrinkage. At the same time, the particles that diffused each other created an interlocked reaction to provide a high deformation resistance structure that increased the hardness of the particles compact. As a result, the sintering process was highly dependent on the heating mechanisms associated with temperature and soaking time.

Nowadays, Alumina (Al_2O_3) and Zirconia (ZrO_2) are used in the majority of ceramic products. Because of the nature of the two materials, a compact and sturdy structure can be created. Al_2O_3 has greater hardness, heat capacity, thermal conductivity, and elastic modulus than ZrO_2. However, Al_2O_3 still lacks of brittle resistance, flexural strength, compressive strength, and fracture strength. The sintered body of Al_2O_3, is fragile and extremely sensitive to surprise power and thermal shocks. To improve the mechanical properties and brittleness of Al_2O_3, secondary element such as ZrO_2 can be added by improving particle packing. The addition of ZrO_2 is thought to have improved the fracture toughness and improve some of the weaknesses in the Al_2O_3 structure, which can further strengthen ceramic cutting tools. According to Fan et al. [6], having a high ZrO_2 content in an Al_2O_3 structure increases fracture resistance by up to 50%. The recommended ZrO_2 content should be between 20% and 25% in order to produce a strong ceramic cutting tool with higher abrasion resistance [7]. ZrO_2 also acts as a barrier to the increase in Al_2O_3 grain size during the sintering process. This is to ensure that the grain size of Al_2O_3 is not too large, which can lead to easy particle slip at the grain boundary.

In order to produce a high-density ceramic body with fine grain size, some aspects must be controlled, such as the nature of the raw material, particle size distribution and shape, particle density, sintering time, sintering temperature, and sintering conditions [8-9]. Cutting tools with a density of 99% can be produced at low sintering temperatures with the addition of ZrO_2 [10]. By using the fine grains of ZrO_2, the mechanical properties and refractory of the cutting tool structure can be improved [11]. However, there are some challenging issues when nano-size ZrO_2 powder is used in product combinations as it has a high tendency to agglomerate and results in a decrease in the mechanical performance of the final product [12]. When a minor amount of ZrO_2 agglomerates in a structure, it causes segregation as the particle's distribution does not act uniformly on the entire structure [13].

Several authors have previously investigated the development of Al_2O_3 cutting tools in terms of microstructure, density and wear performance [4, 15]. Many preliminary research studies have been conducted on the addition of other materials to the structure of Al_2O_3-ZrO_2 to resolve its limitations, such as low fracture toughness, which is required for each cutting tool. Nonetheless, there has been little discussion of the density, hardness that related with grain size of Al_2O_3-ZrO_2. There is also a lack of discussion regarding shrinkage of ceramic body before and after sintering to relate with the precision of ceramic dimension. As a result, the primary goal of this paper is to investigate the impact of sintering temperature and soaking time on the microstructure and mechanical properties of an Al_2O_3-ZrO_2 cutting tool. The mechanical properties that being studied further are density, hardness and shrinkage after the sintering process.

2. MATERIAL AND METHODS

The production of Alumina-Zirconia (Al_2O_3-ZrO_2) cutting inserts began with the combination of 80 wt% Al_2O_3 and 20 wt% ZrO_2 powders, as well as 0.6 wt% binder agent, polyethylene glycol (PEG). To blend the mixture uniformly, a ball mill method was used with alumina grinding balls. The mixture was then extracted from the ball mill machine and pressed into the circularly formed mould using a manual hydraulic hand press machine. The green compacts were then compacted using the cold isostatic pressing (CIP) process at 300 MPa. Finally, multiple sintering temperatures and furnace soaking times were prepared for a series of sintering processes of the green compacts.

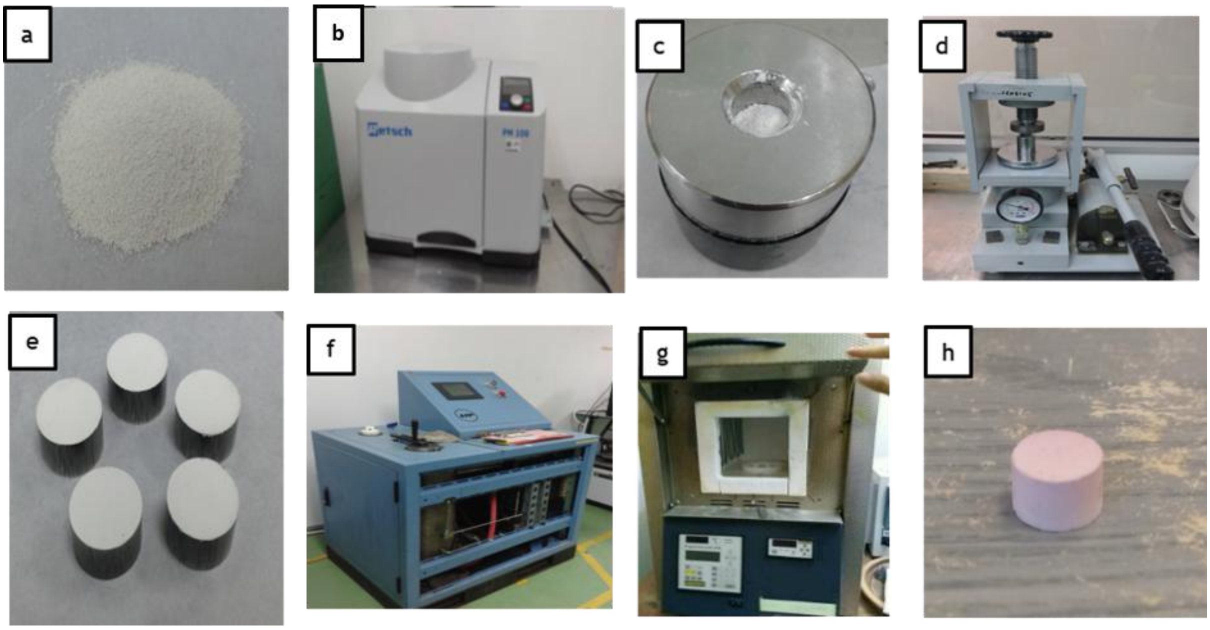


Figure 1. The procedure to fabricate Al_2O_3-ZrO_2 cutting tool (a) Preparing the powders (b) Blending in ball mill for 2 hours (c) Inserting powders into the mould (d) Pressing the mould by hydraulic hand press (e) Green body of ceramic compact (f) Pressing the powders by Cold Isostatic Press (g) Sintering the compacted powders (h) Finish product of ceramic cutting tool after sintering.

The sintering temperature and soaking time are two important sintering parameters that can impact the properties of sintered cutting tools. The sintering parameters and their intervals are described in Table 1. For density analysis, the samples were sintered at sintering temperatures ranging from 1500°C to 1700°C, with soaking times ranging from 4 to 6 hours. For hardness and microstructure observation, the comparison was made at the sintering temperatures ranging from 1500°C to 1700°C, with soaking time constant at 6 hours. Figure 2 depicts the furnace used for sintering process. Figure 3 depicts the sintering profile that represents the isothermal sintering process used in this analysis.

Table 1 Sintering parameters and their intervals

Parameters	1	2	3
Sintering temperature	1500℃	1600℃	1700℃
Soaking time	4 hours	5 hours	6 hours

Figure 2. Naberthem High Temperature Furnace.

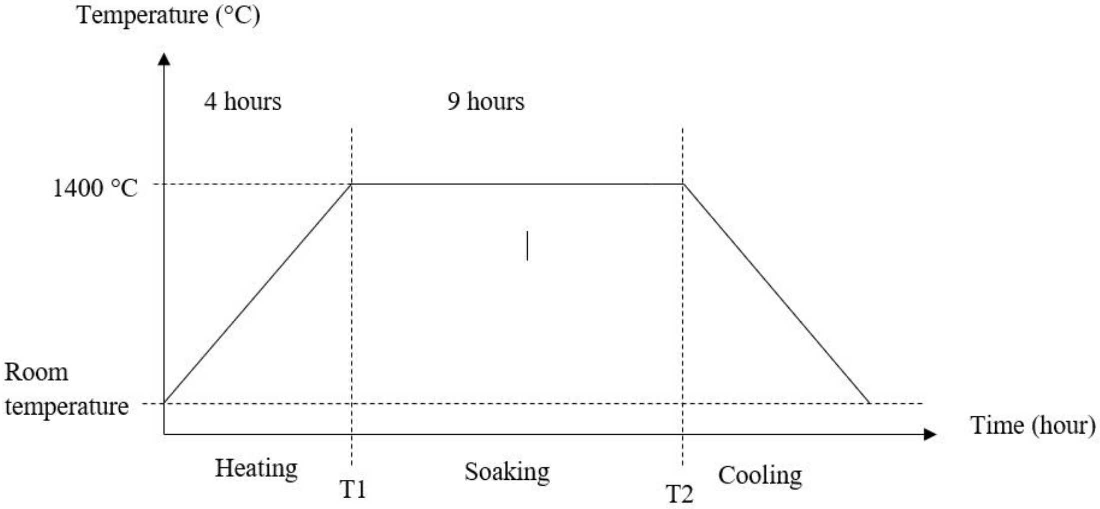


Figure 3. Example of sintering profile and soaking time when the temperature increases from room temperature to the sintering temperature.

3. RESULTS AND DISCUSSION

Table 1 shows the results of an experimental design with an average density outcome. There are seven runs of the sintering method that were completed. Five different sintered cutting tools were measured to determine the average density for each run.

Figure 4 shows the distribution of density when the sintering temperature changed, that plotted according to Table 2. It shows that as the sintering temperature increased, the density decreased. This is because as the temperature rises, the thermal expansion of the particles occurs, and the Al_2O_3 and ZrO_2 particles expand, resulting in larger particle sizes [14]. Although the particles grow larger at higher temperatures, the mismatch expansion between Al_2O_3 and ZrO_2 particles could leave tiny voids between the particles. This condition causes greater porosity and, as a result, lower density. In addition, when the sintering temperature exceeds 1600°C, there is possibility of ZrO_2 transformation from tetragonal to monoclinic, which could further contribute to lower density and gradually decreases its hardness. This finding, however, contradicts research study in which the higher the sintering temperature, the higher the density [6].

Table 2 Experimental design with average density result

Std Run	Sintering Temperature (℃)	Soaking Time (hr)	Average Density (g/cm^3)
1	1500	6	4.0916
2	1600	5	4.0808
3	1700	6	3.93
4	1600	5	4.05
5	1700	4	3.9384
6	1600	5	4.0376
7	1500	4	4.0856

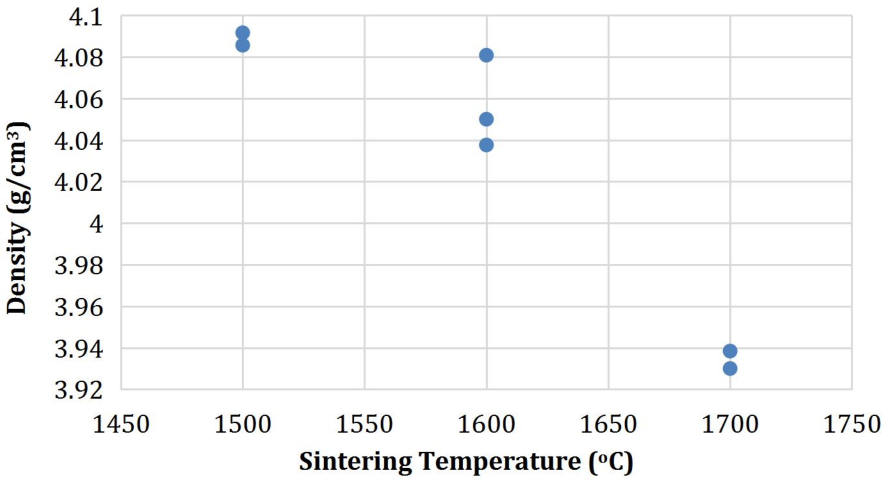


Figure 4. Effect of sintering temperature on density.

The ability to resist plastic deformation, which is normally accomplished by indentation, is referred to as hardness. In this experiment, the hardness was measured using the Vickers indentation process on the Mitutoyo Vickers Hardness Machine to obtain the average hardness value by taking three readings for each sample. Table 3 and Figure 5 show higher hardness of 1321.23 HV recorded after 6 hours of soaking time and sintering temperature of 1500°C. This phenomenon could be explained by the fact that the ceramic sintered at a lower temperature of 1500°C had a higher density of 4.0916 g/cm^3, which reflected better particles density that increased deformation resistance [15]. In addition, the grain sizes when sintering performed at 1500℃ for 6 hours soaking hours are measured to be as small as 2 μm as shown in Figure 6(a).

The decrease of hardness of ceramic compact when the sintering temperature is increased with the time remains constant could be explained by the grain growth that occurred at higher sintering temperatures, which resulting expansion of particle packing. This is shown in Figure

6(b) where the grain size of the sintered particles for cutting tools at 1700 °C and 6 hours is around 5 μm. This is evidence when sintering temperature that was held at 1700℃ and 6 hours soaking time resulting only 688.4HV hardness. Nonetheless, it contradicts the findings of Fan, who found that the Vickers hardness increased as the sintering temperature increased [6].

Table 3 Vickers Hardness results

Sintering Temperature, Soaking Time	**Hardness (Hv)**			
	1	2	3	average
1500℃, 6 hours	1336.4	1438.8	1188.5	1321.23
1700℃, 6 hours	688.4	685.8	700.5	691.57

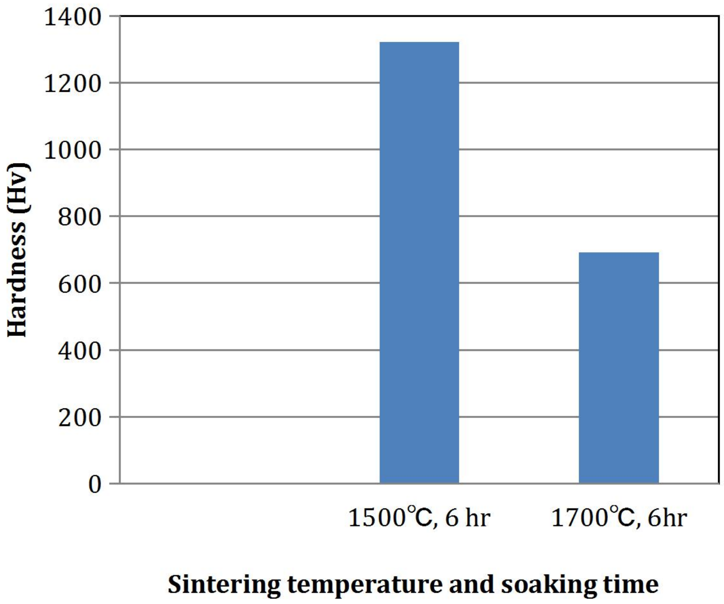
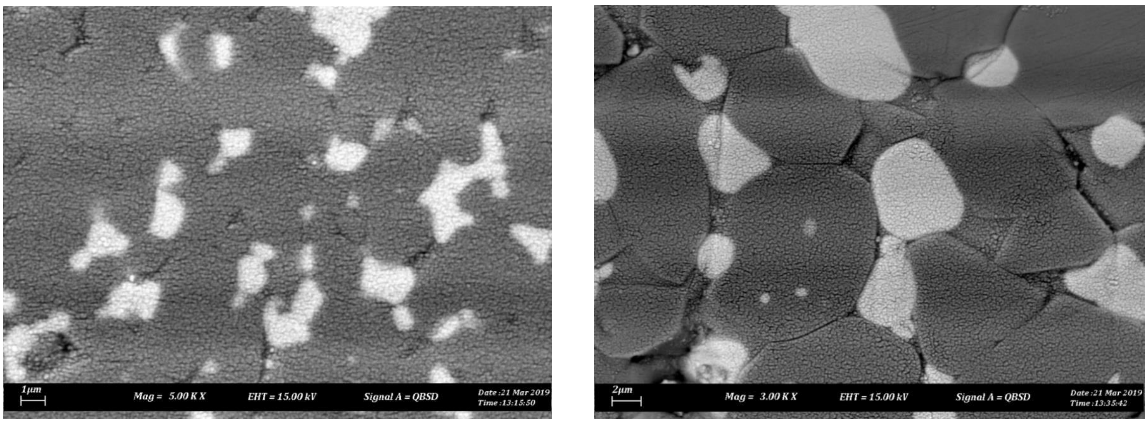


Figure 5. Graph of hardness versus sintering temperature and soaking time.



(a) (b)

Figure 6. (a) SEM image at temperature 1500°C and 6 hours soaking time using Backscattered Electrons Signal. Average grain size around 2 μm(b) SEM image at temperature 1700°C and 6 hours soaking time using Backscattered Electrons Signal. Average grain size around 5 μm.

Table 6 shows the shrinkage of cutting tools after being measured in terms of diameter and thickness before and after sintering. The shrinkage percentage was determined by multiplying the readings by five to get the average value of the shrinkage percentage from comparison measurement according to Table 4 and Table 5. Figure 7 shows Effect of sintering temperature and soaking time on shrinkage. Overall, the highest shrinkage percentage was found to be 9.95% and 4 hours of soaking time at 1700°C sintering temperature, while the lowest shrinkage percentage was found to be 8.36% and 6 hours of soaking time at 1500°C sintering temperature. Shrinkage is the reduction in the dimension of the cutting instrument. There are several factors that contribute to shrinkage or densification. The reduction of the liquid-vapour interfacial region is one of the catalysts for device shrinkage. During sintering process, the gas is removed from the body structure at the start of the sintering process. The empty spaces between the ceramic particles are caused by this condition. When the particles came into contact with each other, they began to restructure. Particles began to develop, resulting in the formation of grains and grain boundaries. Currently, the particle development orientation tends to fill the space along the grain boundaries. This causes the particles body to diffused at the grain boundaries. Once diffused, the separation distance of the mass centres between particles would also decrease, causing shrinkage along the sintered particles [16-18].

Table 4 Cutting tool dimension before the sintering process

Sintering Temperature (℃)	**Soaking Time (hr)**	**Average Diameter (mm)**	**Average Thickness(mm)**
1500	4	13.204	4.652
1500	6	12.99	4.59
1600	5	13.102	4.692
1600	5	13.068	4.694
1600	5	13.134	4.742
1700	4	13.204	4.652
1700	6	13.134	4.716

Table 5 Cutting tool dimension after the sintering process

Sintering Temperature (℃)	**Soaking Time (hr)**	**Average Diameter (mm)**	**Average Thickness(mm)**
1500	4	12.244	4.272
1500	6	11.904	4.24
1600	5	11.9	4.28
1600	5	11.922	4.316
1600	5	11.856	4.328
1700	4	11.89	4.29
1700	6	11.946	4.348

Table 6. Cutting tool shrinkage percentage

Sintering Temperature (℃)	**Soaking Time (hr)**	**Shrinkage Percentage (%)**
1500	4	7.27
1500	6	8.36
1600	5	9.17
1600	5	8.77
1600	5	9.73
1700	4	9.95
1700	6	9.05

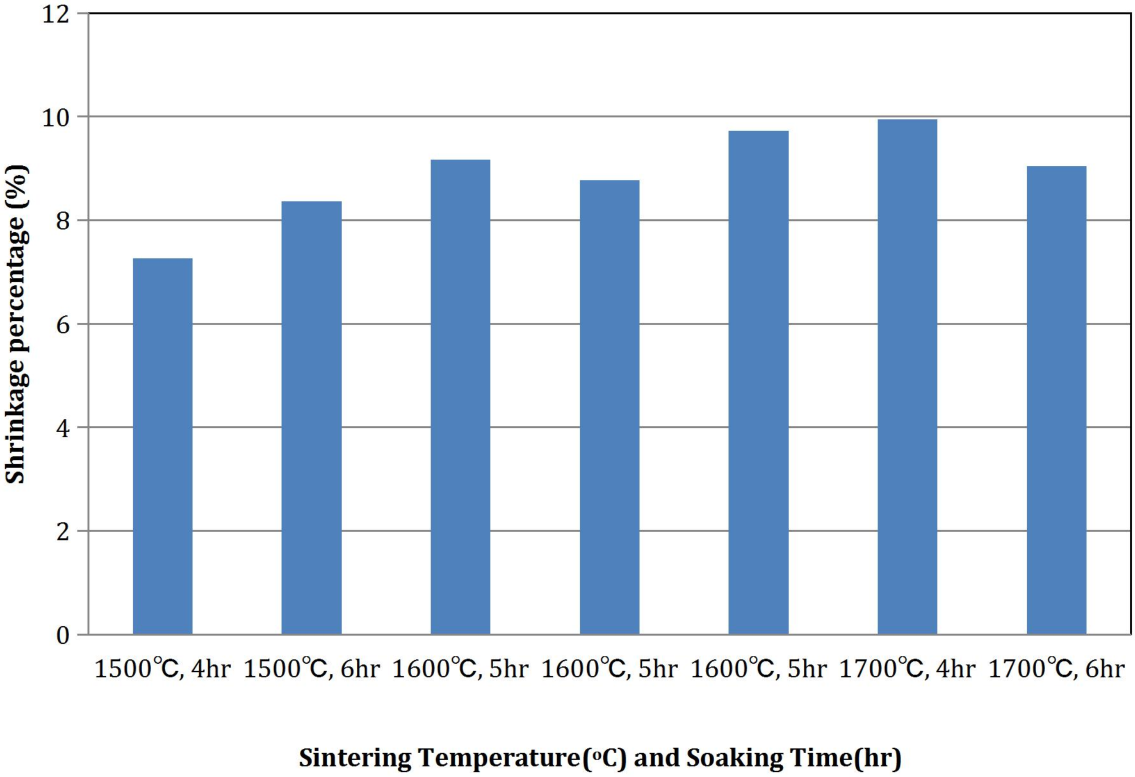


Figure 7. Effect of sintering temperature and soaking time on shrinkage.

4. CONCLUSIONS

This paper describes the fabrication of a ceramic cutting tool made of Alumina-Zirconia (Al_2O_3-ZrO_2) and sintered at temperatures ranging from 1500°C to 1700°C and soaking time ranging from 4 to 6 hours. Density, hardness, shrinkage, and microstructure were used to analyse the cutting tool's properties. AISI 1045 was used to machine a few cutting tools. The following conclusions can be drawn based on the experimental findings:

i. Al_2O_3-ZrO_2 had a maximum density of 4.0916 g/cm^3 after a sintering temperature of 1500°C and a soaking time of 6 hours. As the sintering temperature rose, the density fell.
ii. Maximum density and hardness are related to the diffusion and necking asperity of the grains between Al_2O_3 and ZrO_2. This phenomenon results in decreased porosity and grain interlocking at the grain boundary.
iii. The maximum shrinkage percentage is 9.95% at 1700℃ sintering temperature and 4 hours soaking time while the minimum shrinkage percentage is 8.36% at 1500℃ sintering temperature and 6 hours soaking time.
iv. The higher the sintering temperature, the larger the grain size.

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