Effect of Sintered Alumina Blended Polyethylene Glycol Binder on the Pin on Disc Test Parameter

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Abstract: This study presents the effects of sintering temperature and binder content of polyethylene glycol (PEG) on wear properties of compacted alumina using pin on disc wear test under dry sliding condition. Wear rate was measured at 20N load and sliding speed of 200rpm at 0.28 m/s sliding velocity. The wear test sample was tested at 3 different sintering temperatures (1000°C, 1100°C, and 1200°C) to produce different microstructures. The results show that a dry pressed alumina ceramic with 5 wt. % of PEG content and sintering temperature at 1200°C recorded the lowest wear rate and volume lost compared to other samples. SEM was carried out to investigate the microstructure how it affects the wear property. It was found that at high sintering temperature of 1200°C the particle formation was strengthened to each other hence promote highest wear resistance.

1. Introduction
These days because of a unique properties of ceramic phases, for example, high wear resistance of the ceramic has developed the enthusiasm among researchers about. Regardless of high hardness and chemical inertness, ceramic get high compressive quality and temperature opposition which permit great execution under outrageous conditions. This especially under sliding circumstance with present of high speeds and temperatures, accordingly unlubricated contact and forceful condition [1-3]. Ceramic material such as Al₂O₃(alumina), ZrO₂, Si₃N₄ and SiC have been utilized broadly in metal-use segments like metal roller, mechanical water seals, cutting devices, aides and dies in manufacturing industries have been traded for materials formed by ceramic phases.

Alumina is the most commonly used material in applications requiring wear resistance because of novel properties in a wide scope of uses and great cost-viability [3]. The wide range of Alumina is also in the ceramic composites and nanocomposites as Al₂O₃-TiC, Al₂O₃-ZrO₂ and Al₂O₃-SiC. Subsequently, numerous researchers about have been directed so as to upgrade the preparing of those ceramic production and especially on microstructure for development of ceramic wear properties. Wear is expulsion of material from a strong surface by the sliding activity of another strong and is brought about by friction, fatigue or vibration [3]. Subsequently, it causes dynamic harm including material loss and happens on the surface of the segment because of sliding.
In addition, the tribological properties of alumina and ceramic composites have present huge contrast in spite of the fact as the wide researches develop on friction and wear. This might be influenced by their properties, for example, hardness, fragility, thermal conductivity and chemical inertness. Many publications presented results wear behavior of alumina, and much of the focus has been on the role of wear parameter for example, contact geometry, sliding speed and distance, surface conditions, connected load, relative humidity or kind of condition, surface temperature, substance similarity, test device and specimen technique preparation. [4]. Other than that, there are some issue while analyse the alumina wear information is that habitually over the referenced test conditions factors are not depicted or even monitored. This paper will be discussed the particular effect of alumina pin-on-disc test to the effect of the wear rate test parameter including characteristics of the samples worn surface subsequently.

In addition, the effect test condition of alumina wear behaviour and wear properties of alumina has been studied comprehensively. Alumina or also known as Al$_2$O$_3$ is an engineering ceramic have relatively high density and purity, exhibit very high hardness about in the range of (Hv $>$ 16 GPa) and the value of toughness is prompt considerable for tribological applications in the range of (KIC $\geq$ 4 MPa.m$^{1/2}$). Furthermore, alumina properties such as its brittleness results low friction coefficient ($\mu$) of the un lubricated sliding contact pair of similar alumina. Bajwa [5] reported that the friction coefficient values in the range of 0.2 to 0.8 [1, 2, 4] considering the characteristics of alumina itself, environment conditions as well as the surface roughness and geometry.

For tribological applications, wear of alumina may be categorized into mild’ and ‘severe’ [1, 4-8]. There are several research associated from testing parameter where the wear mode result from normal load, temperature sliding velocity and distance and etc. Ravikiran [7] reported on the wear coefficient is reduced as the apparent area of contact increases. In addition, for such ordinary load and sliding rate, a mild wear mode which is lower than $10^{-6}$ mm$^3$/N.m produces a generally smooth worn surface thus the severe wear mode demonstrating a separate higher value of roughness which creates a harsh worn surface. Furthermore, since the particular wear rate is $<10^{-6}$ mm$^3$/N.m it is demonstrated that a ceramic will be helpful as a business tribological part. Therefore, surface examination is required to determine the wear mechanism. For pin on disc wear test the fundamental assessment is the friction procedure that repeated section of the pin on a similar wear track. Concerning the fragility of ceramic, the presence of the wear debris on the worn surface and powder formation is vital factor to get wear mechanism. [1, 4].

The contact stresses and the contact pressure involve in the formation of mechanical wear debris. The stress became related by normal load and surface roughness and frictional heating from the contact thermal stress. For the most part, the smooth wear surface related to a mild wear mode coming about because of flattening or polishing of surface grains and delicate crack. Thus, decreasing the wear rate and sustain in mild wear mode creates a preventive tribofilm with the repetition cycles of the pin subsequently.

2. Experimental procedure
The alumina powder (NM9620) with sub micrometer average particles size of 0.6µm and high purity (99,999% purity) used in this study was supplied by Vistec Technology. The surface area were 3m$^2$/g and density 3.98 g/cm$^3$ respectively. The pin and the disc sample was produce using mixing process of alumina powder blended with polyethylene glycol (PEG) and the formulation were varies in the percentage of PEG binder apply with 3 different sintering temperatures respectively. The cylindrical pin produced with a semispherical tip (radius of 6.5 mm) and was compacted by uniaxial followed by isostatic pressing at room temperature under the pressure of 24MPa according to ASTM G99-05. The sintering was performed for 20 h at 3 different temperature of 1000°C, 1100°C and 1200°C with a lower heating rate of 1°C/min. The disc was produced using the same method using compaction with PEG binder using isostatic pressed and form a disc with 40 in diameter and 8 mm of thickness and subsequently it was sintered at 1000°C,1100°C and 1200°C for 20 h process as in Table 1.
Table 1. Properties of alumina powder and percentage used.

| Formulation | % of PEG | Sintering Temperature (°C) |
|-------------|----------|---------------------------|
| F1          | 1        | 1000                      |
| F2          | 5        |                           |
| F3          | 1        | 1100                      |
| F4          | 5        |                           |
| F5          | 1        | 1200                      |
| F6          | 1        |                           |

The wear test was carried out using a pin-on-disc test machine (Model: Wear & Friction Monitor TR-20; Brand: MAGNUM). As in Figure 1, the wear test for all specimens was carried out under the normal loads of 20N and a test rpm of 200 for about 10 minutes. In this experiment all specimens were prepared for the wear tests for an absolute sliding distance of around 251 m and about 0.28 m/s sliding speed. The resulting wear rate was determined from the weight loss by deciding its volume loss per unit sliding distance. The wear volume loss was estimated by optical microscopy where it was resolved from the pin worn scar distance diameter. Sample tests with worn surface delivered in the mild wear mode and in the area of progress to severe wear, recognized by the wear rate in the scope of $10^{-6}$ to $10^{-5}$ mm$^3$/N.m were chosen for evaluation and microstructure characterization were examined by scanning electron microscopy (SEM).

Figure 1. Alumina pin and disc specimen after sintering process and schematic diagram of pin on disc test

3. Result and discussion

3.1. Wear Properties of Dry Pressed Ceramic

Figure 2 demonstrate the wear rate of progress transition from gentle to extreme or severe wear mode as capacity of consistent sliding speed and steady ordinary load as a primer endeavor to distinguish the change conditions. The test was implemented at constant load of 20N showed wear rate values for different formulation of alumina blended PEG. The wear rate at sintering temperature of 1000 °C for F1 about $2.89 \times 10^{-6}$ mm$^3$/N.m and decrease trend for highest sintering temperature at 1200 °C about $0.084 \times 10^{-6}$ mm$^3$/N.m. This result is supported by Todesco et al. [6], where the pin-on-disc test of alumina was carried out at 3 different load with 20N, 30N and 40N showed wear rate values between in the range of 2 to 25.10$^{-6}$ mm$^3$/N.m. The increasing wear at sliding speed of about 0.4 m/s for load of 30N result an ordinary transition from mild to severe wear was appeared.

Therefore, as can be observed for load of 20N in Figure 2, the typical transition was shifted from mild to severe wear where the wear rate showed an abrupt increase at a sliding speed of about 0.28 m/s. The chose zone for microstructure characterization in the middle of the mellow wear mode and
in the area of progress to severe wear, found by the wear rate between in the range of $10^{-6}$ to $10^{-5}$ mm$^3$/N.m. However, higher the wear rates will cause error increases too. This phenomena results an uncertain behaviour of the pin-on-disc apparatus that effect the wear volume measurement and also variation caused by load slip and controlled conditions [6]. On the other hand, the wear rates below $10^{-6}$ mm$^3$/N.m will exhibit smooth surfaces are corresponded with mild wear mode, while if there are grooves appear, micro-fracture and rate above $10^{-6}$ associated to the severe wear mode [2].

As illustrate in Figure 3 show the volume loses of alumina samples loaded with 1 wt. % PEG binder (F1, F3, and F5) & 5wt. % PEG (F2, F4, F5) show an decreasing trend associated with higher sintering temperature. The sample weighed before and after the test. The sample unable to withstand the 20 N load for 10 minutes, thus permit little evidence possessed a lot of weight lost with sliding speed of 0.28 m/s. This phenomena have a contradiction as reported by Ahmad et al. [8] in their finding where the weight loss of AMC (Alumina matrix composite) is increased when the load applied 100 N to 150 N accordingly. Hence, higher sintering temperature results the lowest volume loss of samples. The decrease in weight losses was due to highly formation of alpha alumina via transition alumina (gamma alumina) which occurs by nucleation and growth process during heating.

**Figure 2.** Wear rate as a function of constant sliding speed for different formulation of alumina blended PEG (F1,F2,F3,F4,F5,F6) of alumina/alumina in a pin-on-disc configuration.

**Figure 3.** Volume loss as function of constant load of 20N for different formulation of alumina blended PEG (F1,F2,F3,F4,F5,F6).
From the graph in Figure 4 shows bar chart of different wear resistance calculated from wear rate measured by pin-on-disc testing. Based on these values, the profound wear resistance was come from F6 where 5 wt. % of binder was used and sintered at 1200°C. The wear resistance can be defined as it is inversely proportional to the wear rate results. At F6 formulation increase wear resistance trend can be observed, this is due to the uniformity of alumina microstructure and results high mechanical properties. The use of fine alumina powder with bi-modal particle size distribution and higher specific surface, promotes microstructure uniformity with lower closed porosity. This is supported by Milak, [10] where the wear resistance is consequently of the fracture mode and mechanical properties. The greater wear resistance of the alumina occurs because the fraction of material removed during severe wear through the alteration of the intergranular fracture mode in Al₂O₃ to the transgranular mode undergoes less wear where it results a smoother surface. Therefore, the F6 formulation results in higher mechanical properties and wear resistance of the range studied ceramics.

| Formulation | wear resistance (m/mm²) |
|-------------|------------------------|
| F1          | 334.7                  |
| F2          | 467.4                  |
| F3          | 863.53                 |
| F4          | 2238.4                 |
| F5          | 2925.3                 |
| F6          | 11819                  |

Figure 5 shows the worn surfaces analyses under SEM characterization of the samples was carried out in order to observe and interpreted the transition phenomena. For F1 formulation at 1% PEG binder at 1000°C sintering temperature where the wear rates of about 2.9x10⁻⁶ shown a rough worn surface and increase the wear rate. In analysis at Figure 5 (F2) at 5% PEG binder, the grooves started to be found and produce wear scars. Both specimens show existence of grooves at disk and pin, and the peak and valley roughness merge well with the matting surfaces since the sliding pair is nearly the same hardness.

It can be seen that the scar produced at Figure 5 (F3) at 1% PEG binder at sintering temperature of 1100°C. However, the explanation should take into account that the scars depths have been found to be much greater. Figure 5 (F4) SEM micrograph show a lump defect was observed due to a not well recognize behaviour with the increasing of sintering temperatures, the worn surface. Increasing the sintering temperature Figure 5 (F5), at 1% PEG binder at 1200°C, it is clearly seen that the surface material is pushed to the sides of the scratch, as well as the formation of wear debris expelled to the edge of the scar. While Figure 5 (F6) show the amount of wear particles is much smaller, and the particles remaining adherent in scratching scars are small.
4. Conclusion

Based on the wear rate and volume loss of alumina tested at 20N load and sliding speed of 0.28 m/s, it was concluded that the increment of sintering temperature of alumina blended PEG results in lower wear rate between 2.9 to 0.08 x10^-6 mm^3/N.m in terms of the specific wear rate and worn surface analysed. However, the ratio of PEG binder give less significant effect to the pin on disc wear test due to the less amount of binder have been introduced in the test. It was observed the lower sintering temperature permit higher wear rate and the presence the grooves at the surrounding area. This phenomena probably happen that due to the surface roughening and existent of the wear debris. The microstructural studies show at high sintering temperate of 1200°C, the smooth contact surface was noted and contain strengthening of alumina particle that resist the wear. Applied sintering temperature
may affect the wear mechanism and need further improvement in projection with high content of PEG binder for comparable studies onwards.

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