

Effect of Temperature and Heating Resistant Time on Wear Properties of Molybdenum/Alumina Composites

Rizal Indrawan¹

¹Shipbuilding Institute of Polytechnic Surabaya, Teknik Kimia Street, Kampus ITS, Sukolilo, Surabaya, East Java, Indonesia

[†]rizal11307@ppns.ac.id

Submitted: January 2022; Revised: June 2022; Approved: June 2022; Available Online: October 2022

Abstract. The wear properties of metals are very important in manufacturing. This is evidenced in the many studies that analyze the wear properties of a metal. This study uses Metal Matrix Composites, where the manufacturing process uses metal as the matrix and ceramics as reinforcement with powder metallurgy manufacturing methods. Where the powder of the two composite materials is compacted and heated at a certain temperature to form a solid composite material. The purpose of this study is to ensure that the mechanical properties of the Mo/Al2O3 composite are greater than that of the constituent material. In this study, molybdenum powder was used as a matrix material and alumina as a reinforcing material. Mo has excellent mechanical properties, namely hardness and excellent wear resistance. Al2O3 has excellent wear resistance properties. Research variables include heating temperature of 900, 1000, 1100 °C and holding time of heating process for 0.5, 1, 1.5 hours with compaction pressure of 125 MPa and weight fraction of Mo by 80% and Al2O3 by 10%. Based on the test results, the highest sinter density was found at a sintering temperature of 11000C and a sintering holding time of 1.5 hours at 6.831g/cm3. The value of porosity and wear rate is getting lower, namely 28.8% and the wear rate is 0.0012 mm/minute.

Keywords: wear properties, Mo/Al₂O₃ composite, temperature, heating holding time

DOI : 10.15408/fiziya.v5i1.24455

INTRODUCTION

 Mo/Al_2O_3 composites were investigated with the hope that these materials have better mechanical properties than the natural mechanical properties of Mo and Al_2O_3 materials. This parameter depends on various factors. Including the type of powder material, particle size and shape, and the process of making the composite. In this study, Molybdenum (Mo) powder was used as a matrix material and Aluminum Oxide (Al_2O_3) as a reinforcing material. Mo has excellent mechanical properties, namely hardness, high modulus of elasticity, and excellent wear resistance. Meanwhile, Al_2O_3 material has high wear resistance.

Starting with the homogenization of the two powder materials using a planetarium ball mill, then the wet mixing process was carried out using a magnetic

stirrer. After that the cold compaction process with a pressure of 125 MPa. Then the sintering process is carried out at temperatures of 900, 1000, 1100 °C and the sintering process holding time is 0.5, 1, 1.5 hours in an argon atmosphere. The writing of this paper discusses the results of the wear-resistant properties of Mo/Al₂O₃ composite materials and the characterization of these materials.

METHODS

Molybdenum Powder Characterization

In the manufacture of Mo/Al₂O₃ composites, the Mo material is used as a matrix. Mo has a very high hardness and high elasticity value as well. Mo material is very resistant to high temperatures so it is very suitable to be used as a matrix in the manufacture of Mo/Al₂O₃ composites. The initial test on this Mo powder used SEM/EDX Testing. To analyze the Mo powder material using SEM. The following test results using SEM are shown in figure 1.

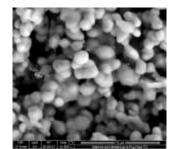


Figure 1. Mo Powder SEM Test Results.

In figure 1, it can be seen that the size of Mo powder is 0.9-3 μ m at 15000× magnification, so there is no need for sieving because the grain size is considered homogeneous. And it can be seen that Mo powder has a dendritic shape and is macroscopically gray in color. To identify any elements contained in Mo powder, the EDX test was carried out. The results of the EDX test are shown in figure 2.

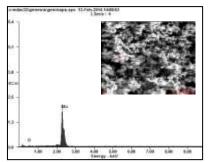


Figure 2. Test Results SEM-EDX Mo Powder.

Based on the results of the SEM - EDX test, it is known that the majority element content contained in the powder is Mo, which is 91%wt with 8.11%wt of O in it.

Element	Wt%	At%		
OK	08,11	34,6		
MoL	91,89	65,4		
Matrix	Correction	ZAF		

Table 1. Elemental Composition of Molybdenum.
--

Alumina Powder Characterization

In this research, alumina powder is used as a reinforcing material. Alumina has high wear resistance. Alumina is also resistant to high temperatures so it is very suitable as a reinforcement in the manufacture of Al_2O_3 composites. Therefore, Alumina material is used as a reinforcement in Al_2O_3 composites. To analyze the Al_2O_3 powder material using SEM. The following test results using SEM are shown in figure 3.

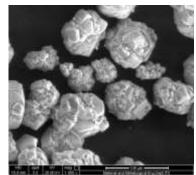


Figure 3. Alumina Powder SEM Test Results.

In figure 3 it can be seen that the size of the alumina powder is 40 - 90 m at 100 0× magnification, so it is necessary to enrich it so that the grain size can be homogeneous. And it can be seen that the alumina powder has a polygonal shape and is macroscopically white in color. To identify what elements are contained in alumina powder, the EDX test is carried out. The results of the EDX test are shown in figure 4.

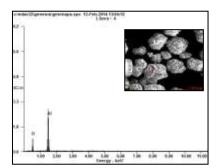


Figure 4. Test Results SEM-EDX Alumina Powder.

Based on the results of the SEM - EDX test, it is known that the majority element content contained in the powder is AI which is 56.67%wt with 43.33%wt of O in it.

Table	2. Element	al Compositio	on of Alı	umina
	Element	Wt%	At%	
	OK	43,33	56,32	
	AIK	56,67	43,68	
	Matrix	Correction	ZAF	

Mo/Al₂O₃ Composite Manufacturing Process with Powder Metallurgy

In the process of making the Mo/Al₂O₃ composite, the first step is to characterize each powder, namely molybdenum powder and alumina powder. The size of the molybdenum powder is smaller, namely 0.9-3 μ m compared to the size of the alumina

powder which has a size of 40-90 μ m. Therefore, a vibrator sieving process was carried out on alumina powder in order to obtain a smaller powder size. After that, the two powders were mixed using the wet mixing method and then a cold compaction process was carried out with a pressure of 125 MPa. After that, the heating process is carried out with variations in heating temperatures of 900, 1000, 1100°C and variations in the holding time of the heating process for 0.5, 1, 1.5 hours. Then the testing phase was carried out on the Mo/Al₂O₃ composite

RESULTS AND DISCUSSIONS

Mo/Al₂O₃ Composite Characterization

At the beginning of the heating process there is a rearrangement of the particles that are in contact so that the contact area between the particles will be better. Neck growth begins to occur in the contact area between particles. Furthermore, at the grain growth stage the porosity structure becomes finer, but remains interconnected until the end of heating. The growing grains will press against each other, causing the empty space for porosity to shrink. And at this final stage, the closed porosity will shrink as a result of the diffusion process. Parameters that affect the heating process include temperature and heating holding time. After the heating process is carried out, the composite is weighed and its mass calculated, then using the Archimedes principle, the density of sinter is obtained.

Table 3. Sintered Density Mo/Al ₂ O _{3.}				
Tomporatur (°C)	Hold	olding Time (h)		
Temperatur (°C)	0.5	1	1.5	
900	6.529	6.633	6.714	
1000	6.725	6.738	6.759	
1100	6.799	6.819	6.831	
ter, p (g/cm ³)		-		

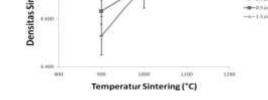


Figure 5. Graph of Relationship between Sintering temperature and Sintered Density of Mo/Al₂O₃ Composite.

Figure 5 shows the effect of the sintering temperature on the sintered density value. The highest density was shown by the Mo/Al_2O_3 composite which was heated at a temperature of 1100 °C for 1.5 hours, which was 6.831 g/cm3. As the sintering temperature increases, the sinter density will increase the heating holding time also affects the density of the sintered composite, where the highest density is found in the

heating holding time for 1.5 hours with a temperature of 1100 °C. The relationship between heating resistance time and sinter density can be illustrated in figure 6.

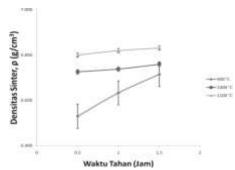


Figure 6. Graph of Relationship between Sintering Resistant Time to Sintered Density of Mo/Al₂O₃ Composite.

From figure 6, it can be seen that there is a relationship between the heating resistance time and the sinter density where the longer the heating holding time, the relative sinter density shows an increase.

In general, the effect of temperature and heating holding time on the sinter density can be seen in Figures 5 and 6 where the higher the heating temperature and time, the higher the sinter density. The results obtained are in line with the theory. The higher the heating temperature, the higher the activation energy, so that the driving force for grain growth is greater. Then as the holding time increases in the heating process, the greater the density of the sinter. This is due to the higher grain growth and coarsening. A long heating time will increase the heating rate. Shrinkage is based on the change in sample dimensions after heating. During heating, the bulk transport process will cause changes in the distance between particles due to neck growth. This causes shrinkage in the compacted powder

Table 4. Mo/Al ₂ O ₃ Composite Shrinkage.				
Temperature (°C) Holding Time (Hour)				
Temperature (C)	0.5		0.5	
900	3.85	900	3.85	
1000	4.85	1000	4.85	
1100	5.71	1100	5.71	
Land 100 100 100 100 100 100 100 100 100 10	eator (°C)	88 130	-se-0,5 µn -se-1 µn -se-1 3 µn	

Figure 7. Graph of Relationship between Sintering Temperature and Shrinkage of Mo/Al₂O₃ Composites.

Figure 7 shows the effect of heating temperature on the percentage of sample shrinkage after undergoing the heating process. The highest percentage of shrinkage was shown by the Mo/Al_2O_3 composite which underwent a heating process at a temperature of 1100 °C for 1.5 hours, which was 7.26%. As the heating temperature increases, the shrinkage percentage will increase. This increase can be seen in samples that have a heating holding time of 1.5 hours. At a temperature of 900 °C the percentage of shrinkage is only 3.85%, but at a temperature of 1100 °C it reaches 7.26%.

The heating resistance time also affects the shrinkage of the composite dimensions, where the highest percentage of shrinkage is found in the heating holding time for 1.5 hours with a temperature of 1100 °C, which is 7.26%. The relationship between heating resistance time and shrinkage percentage can be illustrated in figure 8.



Figure 8. Graph of Relationship between Sintering Resistant Time and Shrinkage of Mo/Al₂O₃ Composites.

From figure 8, it can be seen that there is a relationship between the heating resistance time and the shrinkage percentage where the longer the heating holding time, the relative shrinkage percentage shows an increase. This increase can be seen in the samples that have undergone a heating process at a temperature of 900 °C. However, the increase due to the effect of heating holding time is not so significant when compared to the effect of temperature. At a temperature of 900 °C with a heating holding time of 0.5 hours the percentage of shrinkage is only 3.85%, while with a heating holding time of 1.5 hours it only reaches 4.81%. It can be seen in Figure 7 and Figure 8 that the effect of temperature plays a greater role in shrinkage. The higher the heating temperature, the faster the heating rate.

Porosity is an incoherent part after the composite has undergone a heating process, in the form of a void filled with gas or lubricant. The manufacture of composites by powder metallurgy method can allow the occurrence of porosity. Porosity is also related to the value of the sintered density. The higher the porosity contained in the composite, the lower the sinter density.

So, then the relationship between temperature and heating resistance time on the level of porosity in the Mo/Al_2O_3 composite can be described in Figure 9 below:

Table 5. 100/Al2O3 Composite Porosity value.			
Temperature (°C) Holding Time (Hou			(Hour)
Temperature (C)	0.5		0.5
900	0.320	900	0.320
1000	0.299	1000	0.299
1100	0.292	1100	0.292

Table 5. Mo/Al₂O₃ Composite Porosity Value.

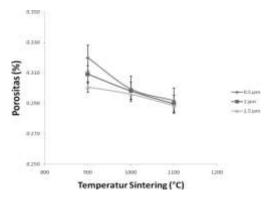


Figure 9. Graph of Relationship between Sintering Temperature and Porosity of Mo/Al₂O₃ Composite.

From figure 9 it can be seen that the lowest porosity is indicated by the Mo/Al_2O_3 composite which is heated at a temperature of 1100 °C for 1.5 hours, which is 28.8%. Then the porosity decreases with increasing heating temperature. Heating resistance time also affects the porosity level of the Mo/Al_2O_3 composite. The effect of heating resistance time on the porosity of the Mo/Al_2O_3 composite is shown in figure 10.

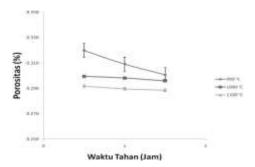


Figure 10. Graph of Relationship between Sintering Resistant Time and Porosity of Mo/Al₂O₃ Composites.

Based on figure 10, it can be seen that the highest porosity is found in the heating holding time for 0.5 hours with a heating temperature of 900 °C, which is 32%. From Figure 10 it can also be seen that the longer the heating holding time, the lower the level of porosity contained in the Mo/Al₂O₃ composite.

Mechanical Properties of Mo/Al₂O₃ Composites

The mechanical properties of a composite can be determined by providing a mechanical test. In this study, the mechanical test used is the Wear Test. Wear can be defined by how quickly a material is damaged during use. By using this wear test tool can provide information on the age of a material how long it can last and be used.

After the wear resistance test was carried out, the wear value was obtained from the influence of temperature and heating resistance time on the wear value of the Mo/ Al_2O_3 composite. This relationship can be seen in figure 11.

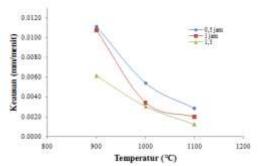


Figure. 11 Graph of Relationship between Sintering Temperature and Wear Value of Composite Mo/Al₂O₃.

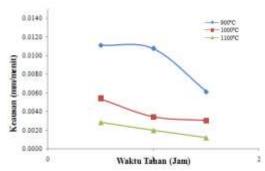


Figure 12. Graph of Relationship between Sintering Resistance Time and Hardness of Mo/Al₂O₃ Composites.

Figure 11 and figure 12 show the wear value of the Mo/Al₂O₃ composite, where the highest wear value is found at a heating temperature of 900 °C with a heating holding time of 0.5 hours, which is 0.0111 mm/minute. While the lowest wear value is found in the Mo/ Al₂O₃ composite which is heated at a temperature of 1100 °C and a heating holding time of 1.5 hours is 0.0012 mm/minute. From Figure 12 it can also be seen that the greater the heating temperature, the lower the wear resistance of the Mo/ Al₂O₃ composite.

Interface Analysis on Mo/Al₂O₃ Composites

Composites are closely related to the quality of the bond between the matrix and the reinforcement. The interface area is an area that can be identified by the presence of bonds between the matrix and reinforcement after the heating process, so that it can show differences in the interface area in the composite. Figure 13 shows the interfacial bonding of the composite at a sintering temperature of 900 °C with a sintering time of 0.5 hours.

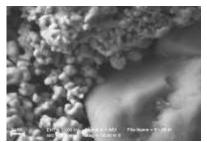


Figure 13. Results of SEM Observations in the Mo/Al₂O₃ Composite Interface with a Sintering Temperature of 900 °C and a Hold Time of 0.5 Hours.

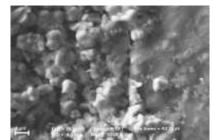


Figure 14. SEM Observation Results in the Mo/Al₂O₃ Composite Interface with Sintering Temperature 1000 °C and Holding Time for 1 Hour.

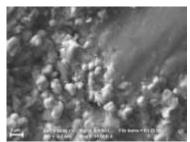


Figure 15. Results of SEM Observations in the Mo/Al₂O₃ Composite Interface with a Sintering Temperature of 1100 °C and a Hold Time of 1.5 Hours.

On SEM observations as shown in figure 13, it can be seen that the interphase surface has not yet formed tightly. The grains do not seem to blend with one another. When the heating temperature is increased as shown in figure 14 by 1000 °C and the holding time is added for 1 hour, it starts to appear that there are parts that are fused but still not tight. And when the heating temperature is 1100 °C and the holding time is 1.5 hours, it can be seen that the interphase surfaces have begun to form tightly and begin to coalesce as shown in figure 15. This is because the increase in heating temperature and the holding time of the heating process greatly affects the bond between powders. Mo and Al_2O_3 powder.

CONCLUSIONS

From the test results in this study, it can be concluded that:

- 1. The lowest wear value is at a heating temperature of 1100 °C with a heating holding time of 1.5 hours, which is 0.0012 mm/minute.
- 2. The highest wear value is found in the Mo/Al_2O_3 composite which is sintered at a temperature of 900 °C and a heating holding time of 0.5 hours, which is 0.011 mm/minute.
- 3. The density of the sintered composite Mo/Al₂O₃ shows that the higher the temperature and heating resistance time, the density of the sinter will increase. This is because the higher the heating temperature, the higher the activation energy, so that the driving force for grain growth is greater.
- 4. In SEM observation, it is known that the most perfect interfacial bond is in the sintering process of 1100 °C and the sintering holding time is 1.5 hours.

REFERENCES

- [1] U. A. Siagian, PENGARUH PENAMBAHAN MOLIBDENUM TERHADAP STRUKTUR DAN KETAHANAN OKSIDASI PADUAN FeCrAlsi. 2018.
- [2] S. E. E. Profile, "Multilayer Al2O3 / Mo composites," no. February, 2020.
- [3] K. Suarsana, I. M. Astika, and L. Suprapto, "Pengaruh Penguat Hibrid SiCw / Al 2 o 3 Pada Komposit Aluminium Matrik Terhadap Sifat Konduktivitas Termal," *J. Ilm. Tek. DESAIN Mek.*, vol. 6, no. 1, pp. 147–151, 2017.
- [4] A. Alhamidi, S. Suryana, and S. Husein, "Sintesa dan Karakterisasi Material Ceramic Metal Composite (Ti1-xMox)C-NiCr melalui Proses Pemaduan Mekanik dengan Variasi Komposisi Molibdenum," *Tek. J. Sains dan Teknol.*, vol. 11, no. 1, p. 11, 2015, doi: 10.36055/tjst.v11i1.6968.
- [5] M. Terhadap, S. Mekanik, D. A. N. Struktur, M. Pada, B. Tahan, and K. Tipe, "Pengaruh penambahan unsur MOLIBDENUM (Mo) TERHADAP SIFAT MEKANIK DAN STRUKTUR MIKRO PADA BAJA TAHAN KARAT TIPE AISI 410," vol. 2, no. 1, pp. 43–50, 2019.
- [6] J. Zygmuntowicz, A. Łukasiak, P. Piotrkiewicz, and W. Kaszuwara, "Al2o3-cu-mo hybrid composites: Fabrication, microstructure, properties," *Compos. Theory Pract.*, vol. 19, no. 2, pp. 43–49, 2019.
- [7] A. I. Nafili, S. M. B. Respati, and B. Santoso, "Pengaruh kandungan alumina terhadap kekasaran permukaan dan foto mikro permesinan pada komposit Al-Al2O3," *J. Tek. Mesin Indones.*, vol. 12, no. 2, p. 94, 2018, doi: 10.36289/jtmi.v12i2.79.
- [8] I. W. L. Suprapto, K. Suarsana, and N. Santhiarsa, "EFEK KOMPOSISI DAN PERLAKUAN SINTERING PADA KOMPOSIT AI/(SiCw+Al2O3) TERHADAP SIFAT FISIK DAN KEAUSAN," J. Muara Sains, Teknol. Kedokt. dan Ilmu Kesehat., vol. 1, no. 1, pp. 124–131, 2017.