Synthesis and Characterization of Sintered Magnetic Abrasive Particles having Alumina and Carbonyl Iron Powder

Anupam Alok*¹, M.S. Niranjan², Abhinav Kumar¹, Manjesh Kumar¹, Manas Das¹
¹Department of Mechanical Engineering, Indian Institute of Technology Guwahati, India
²Department of Mechanical Engineering, Delhi Technological University, Delhi, India

E-mail: anupamalok59@gmail.com

Abstract. The present work is focused on compacting, sintering, and characterization of sintered magnetic abrasive particles, which is composed of equal volume fraction of alumina and carbonyl iron powder. Powder metallurgy method is a well-developed technique for manufacturing of ferrous and nonferrous parts. Al₂O₃–CIP composites are prepared through powder metallurgy method. Ball milling is used for mixing powders, and hydraulic jack with die is used for compacting purpose. Solid and liquid phase sintering is performed at a high temperature tubular furnace under an inert gas atmosphere of argon. Solid and liquid phase sintering is done at 1000°C and 1545°C, respectively in proper consecutive sintering cycle. After sintering, the sintered pallets are crushed using ball miller to obtain the required size of the sintered powder. Energy Dispersive X-ray spectroscopy is used for elemental composition of all sintered powders. Vibrating sample magnetometer is used to see the magnetization of the particles. The saturation magnetization of the sintered abrasive obtained at 9 ton compaction pressure is found to be highest. Different phases of all prepared samples are studied using the X-ray diffraction technique. The morphology, as well as particle size, are studied using a scanning electron microscope. Also, the microstructure of sintered powders is studied using an optical microscope. Compression strength test of all sintered pallets is carried out using Universal Testing Machine. Bulk density of the pallets is measured using standard Archimedean principle. It is observed that the bulk density value increases with the compaction load. Micro hardness of the sintered pallets is measured using a Vickers micro hardness measuring instrument. The sintered pallet, fabricated at a compaction pressure of 9 ton shows the highest hardness.

1. Introduction
Ceramics have been known to mankind since early civilization and have been played an important role in the evolution and development of human civilization. Generally, ceramics are defined as solid crystalline materials composed of oxides, carbides, nitrides, borides having important structural, mechanical, thermal, and electronic properties [1]. Metal Matrix composites have acquired substantial importance, as it is being used in many industries [2]. Matrix composites of iron metal with ceramic particles are of interest in the present study because of its various advantages in mechanical properties with easy fabrication. These ceramics are used in aircraft, aerospace, automotive, and other manufacturing fields [3]. Powder metallurgy is a low-cost technique to produce rich properties in materials. Also, it has the capability to fabricate complex alumina, which is technologically an important material for electronic and structural applications. Alumina is an important structural ceramic material having many desirable properties like high melting point (2046 ±5°C), high hardness (18 GPa), high elastic modulus (380 GPa) having excellent resistance to acids and alkalis. It has widely used as grinding media, textile thread guides, in paper manufacturing and cutting tool industries, etc.

1.1. Powder metallurgy
Powder metallurgy (PM) is a process of forming precision components from powders [4]. Powder metallurgy processes have technical as well as economic benefits as compared to conventional processes [5]. In the PM process, the following three steps are followed in sequence.
• Mixing (or blending): A homogeneous mixture of elemental metal powders or alloy powders is prepared.
• Compacting: A controlled amount of the mixed powder is introduced into a precision die, and then it is pressed or compacted at a pressure in the range of 100 – 1000 MPa.
• Sintering: During this step, the green compact is heated in a protective atmosphere furnace to a suitable temperature, which is below the melting point of the metal.

2. Material and method
The magnetic- abrasive powder machining is prepared using alumina abrasives and carbonyl iron powders (CIP).

2.1. Mixing
Powder mixing is carried out by ball mill with a steel ball with equal proportion, i.e. 20 vol. % of alumina and CIP each, as shown in figure 1.

Figure 1. Ball milling machine

2.2. Compacting
For compacting purpose, a 12–ton capacity hydraulic jack having 13 mm diameter die is used. For compaction, 5 gm powder is considered. Further, a load of 5, 7, and 9 ton are applied. In the process of making pallet, the holding time is 15 min. Figure 2 shows the powder compaction set up.

Figure 2. Hydraulic Jack with die for powder compaction

2.3. Sintering
Solid and liquid phase sintering is conducted in high-temperature tube furnace having capacity up to 1600°C. The inert environment of argon is considered to avoid any kind of atmospheric contamination. The most important factors involved during the sintering process are temperature, time, and furnace atmosphere. Increasing sintering temperature greatly increases the rate and magnitude of any change in the properties occurring during sintering. Two types of sintering cycles are used for sintering; one for solid phase and another one for liquid phase sintering. Maximum temperature of 1000°C is considered for solid phase and for liquid phase sintering.
1545°. In the present study, a heating rate of 3°C/min and cooling rate of 6°C/min are considered. Figure 3 shows the image of a programmable high-temperature tube furnace. Figure 4 shows that the sintering cycles which are used for (a) solid and (b) liquid phase sintering.

![High-temperature tube furnace](image)

**Figure 3.** High-temperature tube furnace

![Sintering cycles](image)

**Figure 4.** (a) solid and (b) liquid phase sintering cycle

### 3. Results and discussion

#### 3.1. Density

Figure 5 shows the plot of density at different compaction load for green and sintered pallets. Bulk density increases with increased compaction load of sintered pallets because when compaction load increases, the volume of the pallets decrease. Also, the density of sintered pallets is higher than the green pallets due to the shrinkage of pallets.

![Density plot](image)

**Figure 5.** Comparison of density for green pallet and sintered pallet
3.2. Micro-hardness

The micro-hardness values are measured at various points of pallets for different compaction load. Figure 6 shows the micro-hardness plot of solid and liquid phase sintered pallets. From figure 6, it is observed that 9-ton pallet has a maximum hardness value. The micro-hardness values increase with the compaction load. Hardness is the resistance offered by the crystal for the movement of dislocations. Practically, it is the resistance offered by the crystal for localized plastic deformation. Hence, from the experimental analysis, it is clear that resistance offered by the crystal increases with higher compaction load.

![Figure 6. Comparison of micro-hardness between the solid phase and liquid phase sintered pallets at different compaction loads](image)

3.3. Compressive stress

Figure 7 shows the plot of compressive stress at different compaction loads for sintered pallets. Compressive stress increases with compaction loads of pallets. Higher compressive stress is observed in the case of liquid phase sintering. Therefore, compressive stress depends on both the compaction load and temperature.

![Figure 7. Comparison of compressive stress of solid phase and liquid phase sintered pallet at different compaction load](image)

Average particle size is measured from SEM morphology of the sintered powders at different compaction loads; (a) 5-ton and (b) 7-ton solid phases as shown in figure 8. For the calculation of average particle size in the sintered powder, particle sizes are measured diagonally in the SEM image. Further, an average of all diagonally measured values is used to calculate the average particle size. Figure 9 shows the trend of average particle size at different compaction loads. Figure 10 shows the optical morphology of solid as well as liquid sintered powders.
Figure 8. Average particle size measured from SEM images at different compaction load; (a) 5-ton and (b) 7-ton solid phases.

Figure 9. Particle sizes of solid and liquid phase sintering of pallets at different compaction load (a) solid and (b) liquid phase sintering cycle.

From figure 10, it can be seen that the two phases are present in the sintered powders. One phase is due to alumina, and another phase is due to Fe powder. The white one is due to alumina, and the round shape one is due to iron powder.
3.4. XRD analysis

The XRD pattern of 5-ton solid sintered Al$_2$O$_3$–CIP is shown in figure 11 (a). From figure 11 (a), it can be seen that the first peak comes at 33.180°. This peak belongs to Fe$_2$O having (1, 0, 4) h, k, l values, and the plane is simple cubic. The second phase is at 33.280° of Al$_2$O$_3$ having (1, 0, 4) h, k, l values, and the plane is simple cubic. The third peak is at 35.452° of Fe$_3$O$_4$ having (3, 1, 1) h, k, l values, and the plane is FCC. The fourth pick is at 44.142° of Fe having (1, 1, 0) h, k, l values, and the plane is BCC. The fifth peak is at 54.23° of Fe$_2$O$_3$ having (1, 1, 6) h, k, l values, and the plane is simple cubic. For this sample, the FWHM value of the highest peak is 0.16.

The next XRD pattern of 7-ton solid sintered Al$_2$O$_3$–CIP is shown in figure 11 (b) and the first peak comes at 33.180°. This peak belongs to Fe$_2$O and has (1, 0, 4) h, k, l values, and the plane is simple cubic. The second phase is observed at 33.28° of Al$_2$O$_3$ having (1, 0, 4) h, k, l values, and the plane is simple cubic. The third peak is observed at 35.452° of Fe$_3$O$_4$ having (3, 1, 1) h, k, l values, and the plane is FCC. The fourth pick is observed at 44.142° of Fe having (1, 1, 0) h, k, l values, and the plane is simple cubic. The fifth peak is at 54.23° of Fe$_2$O$_3$ having (1, 1, 6) h, k, l values, and the plane is BCC. The sixth peak is at 54.23° of Fe$_2$O$_3$ having (1, 1, 6) h, k, l values, and the plane is BCC. The
fifth peak is observed at 54.233° of Fe$_2$O$_3$ having (1, 1, 6) h, k, l values with the simple cubic plane. The sixth peak is observed at 65.686° of Fe having (2, 0, 0) h, k, l values with BCC plane. The FWHM value for the highest peak is 0.127. Another XRD pattern of 9-ton solid sintered Al$_2$O$_3$–CIP is shown in figure 11 (c) where the first peak comes at 33.180°. This peak belongs to Fe$_2$O$_3$ having (1, 0, 4) h, k, l values with the simple cubic plane. The second phase is observed at 33.280° of Al2O3 having (1, 0, 4) h, k, l values with the simple cubic plane. The third peak is observed at 35.452 of Fe$_3$O$_4$ having (3, 1, 1) h, k, l values with FCC plane. The fourth pick is observed at 44.142° of Fe having (1, 1, 0) h, k, l values with BCC plane. The fifth peak is observed at 83.219° of Fe having (2, 1, 1) h, k, l values with BCC plane. The FWHM value for a highest peak is 0.188.

The XRD pattern of 7-ton liquid phase sintered Al$_2$O$_3$–CIP is shown in figure 11(d). From this figure it can be seen that first peak comes at 33.180°. This peaks belongs to Fe$_2$O$_3$ having (1, 0, 4) h, k, l values with simple cubic plane. Second phase is observed at 33.280° of Al$_2$O$_3$ having (1, 0, 4) h, k, l values with simple cubic plane. Third peak is observed at 35.452 of Fe$_3$O$_4$ having (3, 1, 1) h, k, l values with FCC plane. Fourth pick is observed at 44.142° of Fe having (1, 1, 0) h, k, l values with BCC plane. Fifth peak is observed at 83.219° of Fe has (2, 1, 1) h, k, l values with BCC plane. The FWHM value for highest peak is 0.174.

3.5. **EDX analysis**

Figure 12 shows that the important elements which are present in the sintered pallet measured from EDX. The most important elements are Fe and Al.

![Figure 12. Elemental compositions of sintered powder for 5-ton compaction load](image)

3.6. **VSM analysis**

Vibrating sample magnetometer (VSM) results, as shown in figure 13, shows magnetic properties of a sample. When a ferromagnetic material is magnetized in one direction, it will not relax back to zero magnetization when the imposed magnetizing field is removed. It must be driven back to zero by a field in the opposite direction. From the values of magnetic moment as shown in figure 13, it is clear that the value of magnetic moment increases with compaction load and sintering temperature. From figure 13, the highest magnetic moment is observed for 9-ton compaction load with liquid phase sintering power. In liquid phase sintering, the iron particles diffuse in a proper way into the alumina particle. Hence, the magnetic properties increases for liquid phase sintered powder as compared to the solid phase sintered powder.
4. Conclusions

In the present study, alumina and CIP based sintered magnetic abrasive powder is prepared by powder metallurgy method. The following conclusions are drawn based on above study on Al₂O₃–CIP composite.

- The micro-hardness result shows that its value increases with load at higher sintering temperature.
- The compressive stress increases with compaction load and sintering temperature. The maximum value of compressive stress is 67.95 N/mm² at 9-ton liquid phase sintered sample.
- Optical microscopy reveals that denser particles are obtained at higher compaction load.
- XRD results show crystalline structure and different phases present in the sample with lattice parameter. XRD study shows simple cubic, face cubic centered and body cubic centered planes present in the sample. FWHM value of sample is 0.16, 0.188, 0.127 and 0.174, respectively.
- From SEM analysis, size of particles has been seen diagonally on the micrograph and it is used to calculate average particle size. Maximum size of particle in diagonal is 53.2 µm and average particle size is 29 µm, 18.9 µm, 24 µm, 13 µm, respectively.
- From magnetic properties study of sintered abrasives using VSM, it has been found that saturation magnetization increases with compaction load of pallets and sintering temperature.

References

[1] Richerson DW, 2012, “The magic of ceramics,” American Ceramic Society, Wiley/American Ceramic Society.

[2] Saxena A, Singh N, Kumar D, Gupta P, 2017 “Effect of Ceramic Reinforcement on the
Properties of Metal Matrix Nanocomposites,” Mater Today Proc, 4:55, 61–70.

[3] E. Dörre, 1984, “Alumina - Processing, Properties and Applications”, Springer, XIV, 330.

[4] Champion Y, 2008, “Powder metallurgy of nanometric aluminium powders” Powder Metall, 51, 125–32.

[5] Abdel-Rahman M, El-Sheikh MN, 1995,”Workability in forging of powder metallurgy compacts,” J Mater Process Technol, 54, 97–102.