PHYSICAL PROPERTIES OF AL/CALCINED DOLOMITE METAL MATRIX COMPOSITE BY POWDER METALLURGY ROUTE

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Abstract
Calcined dolomite is dolomite powder that had been calcined at high temperature. This paper describes the fabrication of aluminum metal matrix composite reinforced with calcined dolomite by powder metallurgy route. The effect of weight percent of reinforcement (10 wt%, 20 wt%, 30 wt%) on physical properties of Aluminum composites are studied and presented in this paper. Calcined dolomite was synthesis from dolomite powder at 1100°C for 4 hours. After weighed, mixed and blended in planetary ball mill, the powders were compacted at 300 MPa and heated in tube furnace under nitrogen environment at sintering temperature 610°C. After 7 hours heated in furnace, it was cooled in furnace until room temperature. The results of the study showed that, the hardness increased with the increment of dolomo content. Study also showed that the density decreased upon increasing the calcined dolomite content.

Keywords: Powder metallurgy, Aluminum/calcined dolomite, macrohardness, density

Introduction
Particulate reinforced metal matrix composites (PRMMC) comprise a new class of materials whose properties can be adjusted to suit a particular application. The main purpose of producing PRMMC is to achieve lightweight, low costs materials with added properties like hardness, high thermal stability, high stiffness and high ultimate strength [1]. The superior properties of these MMCs make them ideal futuristic materials for a variety of applications in automobile, structural, aerospace industries and critical defence-oriented fields [2]. Composites that use particles or whiskers as reinforcement can be obtained easily by P/M than by other alternative routes; moreover, particles are cheaper than continuous fibers of the same composition. Another advantage of P/M is the uniformity in the reinforcement distribution. This uniformity not only improves the structural properties but also the reproducibility level in the properties [3]. Interest in aluminium matrix composites (MMCs) continues to grow, especially from the transport industries, where component weight reduction is a key objective [4]. Al matrix composites reinforced by ceramic particles such as SiC, Al2O3, CuO, Si3N4, AlN, ZrB2 have widely been study however no research have been carried out for Al reinforced calcined dolomite. This paper will present the effect of weight fraction of calcined dolomite particulate to physical properties of pure Al metal.

Calcined dolomite is a heated dolomite where above 1000°C the structure and phase of dolomite changed and consist of a phase mixture of lime (CaO) and periclase (MgO). They have extremely high melting points, as the eutectic for the CaO -MgO binary system occurs at 2370°C [5]. Dolomite is attractive because of its potential cost effectiveness and worldwide abundance and has high thermal stability when calcined above 950°C.

Experimental
Materials
Calcined dolomite powders was produced by heating the dolomite powders (particle size < 65µm) at 1100°C for 4 hours at atmosphere environment to decompose the carbonate. Figure 1 shows XRD spectrum of calcined dolomite where above 1000°C dolomite structure has changed to lime (CaO) and periclase (MgO) phase follow below equation:

\[ \text{CaMg(CO}_3\text{)}_2 \rightarrow \text{CaO} + \text{MgO} + 2\text{CO}_2 \]

Pure Al (99.0%) manufactured by MERCK with average particle size 26.18µm was used as a matrix.

Composites processing
Calcined dolomite and aluminum powder were weighed and mixed in planetary ball. The powder mixture was ball milled for 2 hours rotating at 300 rpm speed using zirconia balls of 20 mm and 10 mm diameter. The composite powder then were compacted into cylindrical pellets having a diameter of 12 mm using a stress of 300 MPa by cold pressing using manual hydraulic compaction with the upper punch moving freely along the axial direction. The composition of the compacts is shown in Table 1. The composite pellets were then sintered in argon at 610°C for 7 hours to initiate the reactions and was cooled in furnace.
Table 1: Composition of Al/calcined dolomite composite

<table>
<thead>
<tr>
<th>Samples name</th>
<th>Al (%weight)</th>
<th>Calcined dolomite (%weight)</th>
</tr>
</thead>
<tbody>
<tr>
<td>AlCD</td>
<td>100</td>
<td>0</td>
</tr>
<tr>
<td>AlCD10</td>
<td>90</td>
<td>10</td>
</tr>
<tr>
<td>AlCD20</td>
<td>80</td>
<td>20</td>
</tr>
<tr>
<td>AlCD30</td>
<td>70</td>
<td>30</td>
</tr>
</tbody>
</table>

**Dimensional and weight change**
Change in diameter, height and weight was measured to determine percent shrinkage or expand of sample after sintering process. It was measure by dividing the average changes of 6 samples by average of initial sample before sintered.

**Density measurement and porosity**
The Shimadzu BX4200H electronic balance was used to determine the bulk density and apparent porosity. It was measured according to Archimedes principle as describe in ASTM standard C20-97. The theoretical density of calcined dolomite was calculated based on weight percent of lime and priclase present in the phase taken from XRF analysis. Table 2 shows wt% of lime and priclase.

Table 2: Weight percentage of priclase and lime phase in calcined dolomite obtained by XRF

<table>
<thead>
<tr>
<th>Materials</th>
<th>Bulk density (g/cm³)</th>
<th>Wt% in calcined dolomite</th>
</tr>
</thead>
<tbody>
<tr>
<td>mgO</td>
<td>3.65</td>
<td>59%</td>
</tr>
<tr>
<td>CaO</td>
<td>3.32</td>
<td>41%</td>
</tr>
</tbody>
</table>

The theoretical density has been calculated using the rule of mixture according to the weight fraction of calcine dolomite particles. Calculated porosities of the produced composites were also measured to compare with the apparent porosity composites. It was evaluated from the difference between the theoretical and the observed bulk density of each sample.

\[ P = (1 - (\rho_A / \rho_T)) \times 100 \]  
where \( P \) is the porosity, \( \rho_A \) the bulk density and \( \rho_T \) is the theoretical density of the sample.

**Hardness and compression test**
The hardness of the composite has been measured using a Vickers's microhardness tester at an applied load of 10N for an indentation period of 10 to 15 seconds. At least nine hardness values from different locations well distributed on the sample surface were measured for each sample in order to eliminate possible segregation efforts and the average of the measurement was considered to be microhardness of the sample.

**Results and Discussion**

**Dimensional and weight changes after sintering**
Figure 1 shows average changes in weight and diameter measured in percent after sintering process. Overall sample shows an obvious expansion in both diameter and length after sintered. Weight of sample also slightly increased compared to green compact. The addition of reinforcement particulate results in the increment of percent changes especially in dimension. Therefore slightly change in weight and dramatically change in volume can cause the density of the composites drop with higher weight % of reinforcement. Monitoring the sintering process with the linear shrinkage data is important not only for the manufacturing of precision components but also for determining the sintering mechanism.

Ordinarily, sintered metal undergo shrinkage cause by amalgamation of powder metal particles and closing void while sintering process. Expansion occurs because pure Al properties will swallow while sintering process. This phenomenon depends on sintering atmosphere and the strength of green compact. An addition of reinforcement content will raise linear expansion percentage. The factor of this explanation is this composite has many air void and gases trapped on the sample before sintering process. After sintering, these gases will expand and affect the dimension of sample.

Figure 2 shows comparison between bulk density of sample after sintered and before sintered and theoretical density of composite Al reinforced with different weight percent of calcined dolomite. The results show that the average density of green compact sample and sintered sample drop with the increment of reinforcement while theoretical density increase with increasing weight percent of calcined dolomite. Partial decomposed of carbonate during calcination of raw dolomited will results the evolution of CO₂ gas during prolonged sentering. There is some appreciable volume changes observed during the sintering of the composites causing density to decrease. The incorporation of the calcined dolomite powder has reduced the bulk density of the matrix powder. This is because the reinforcement particles are
bigger than the voids between the aluminium particles where it will interfere with the powder packing, decreasing the apparent density. On the other hand, the higher theoretical density of the reinforced particles produces the opposite effect, and this may explain why a higher content of reinforcement produces a lower decrease of the bulk density [5]. Measurement the density of powder metallurgy products are essential since material properties such as elongation, yield strength and hardness are closely related to density. Full density materials possess significantly better properties.

![Figure 1: Effect of calcined dolomite reinforcement to linear expansion of AlCD composites in nitrogen atmosphere.](image1)

**Density and porosity**

By using XRF data, the theoretical density of calcined dolomite = 3.51(g/cm³)

<table>
<thead>
<tr>
<th>Materials</th>
<th>Average bulk density (g/cm³)</th>
<th>Theoretical density (g/cm³)</th>
<th>Measured porosity %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>2.45</td>
<td>2.700</td>
<td>6.25</td>
</tr>
<tr>
<td>AlCD10</td>
<td>2.29</td>
<td>2.781</td>
<td>2.11</td>
</tr>
<tr>
<td>AlCD20</td>
<td>2.31</td>
<td>2.862</td>
<td>1.44</td>
</tr>
<tr>
<td>AlCD30</td>
<td>2.23</td>
<td>2.943</td>
<td>3.16</td>
</tr>
</tbody>
</table>

![Figure 2: Theoretical and measured density of composite sample with calcined dolomite content](image2)

Figure 3 shows the results of calculated porosity, green compact and sintered sample. The porosity was measured in percent. Percent of porosity decrease with the increasing of reinforcement up to 20 wt% calcined dolomite. However percent of porosity slightly increase for 30 wt% of reinforcement particulate. The presence of the reinforcement reduces the porosity by preventing the grain growth thereby reducing pore break-away from grain boundary. In contrast calculated porosity increased with increment of filler. This is because the percent of apparent porosity for green and sintered sample was measured without considering the closed pore that was existed inside the sample.
Hardness

Figure 5 displays the relationship between the weight percent of calcined dolomite and the micro hardness of Al/calcined dolomite composite for both conditions of samples before and after polish. Both conditions show an upward pattern. However sample before polish shows average 13.7% higher hardness compared to sample after polish. The figure shows that the hardness increases with the increasing weight percent of calcine dolomite. The average of Vickers hardness of the aluminum is 35.1HV, whereas for 10wt%, 20wt% & 30wt% MMC materials are 40.4 HV, 417 HV and 51.3 HV respectively. The hardness increased near 50% for sample with 30 wt% calcined dolomite compared to pure Al. This can be attributed to increasing the presence of harder calcine dolomite particulate in the matrix causes higher constrains to localize matrix deformation during indentation. High dislocation density originating from a strong interface between the particulates and the matrix also contribute to the increase in hardness. If the porosity could be decrease it was expected that the hardness will be higher than the hardness obtain from this research.

Figure 3: shows calculated porosity and measured porosity for composite samples

![Figure 3](image3)

Figure 4: Relationship between weight percent of calcined dolomite and hardness

![Figure 4](image4)

Conclusion

Incorporation of calcined dolomite particles in the Al matrix improves the hardness properties and produced more light material. The hardness properties of the composite can be higher than the results obtain if the porosity can be reduced during the process. The dimensional changes occur about 4.5%. Calcined dolomite which can easily obtain in lower cost is a potential filler to replace the usage of alumina or other expensive ceramic fillers since it has been proved can increased the hardness of Al matrix and can contribute to light materials.
References