MECHANICAL PROPERTIES AND DRY SLIDING WEAR BEHAVIOUR OF EXTRUDED Al-Al_2O_3 PM COMPOSITES

K. Milos, H. Danninger, G. Jangg, H.-P. Degischer

Abstract
Particle reinforced Al base MMCs offer improved mechanical properties and wear resistance compared to monolithic Al alloys. In the present work, Al-Al_2O_3 composites are produced by hot extruding green powder compacts of plain Al with 20...50 wt.% (14.5 ... 40.3 vol.%) fused alumina. The extruded bars are free of any macrodefects and are characterized mechanically and microstructurally. The dry sliding wear is studied on a pin-on-disc tester in dry run against ball bearing steel. With increasing content of hard phase the tensile and yield strength as well as Young’s modulus first increase but tend to drop >30 vol.% particles, as a consequence of increasing probability of direct oxide-oxide contacts to be regarded as defects. The wear behaviour, in contrast, exhibits a consistently decreasing wear rate with higher hard phase content. Generally, the wear rate is affected markedly more by the test parameters, i.e. normal load and sliding speed, than by the microstructure. A pronounced transition from mild to severe wear is encountered when exceeding a critical combination of parameters, as a consequence of thermal overloading of the matrix.

Keywords: Al matrix composites, alumina particle reinforcement, powder metallurgy, extrusion, dry sliding wear

INTRODUCTION
Al alloys have found widespread applications because of their low specific weight and high corrosion resistance, in particular in the aerospace industry, but also increasingly in automotive applications [1, 2]. Precipitation strengthened Al alloys offer strength levels similar to those of structural steels and much better strength-to-weight ratio. There are however some disadvantages that cannot be corrected by alloying or heat treatment, such as the relatively low Young’s modulus and high coefficient of thermal expansion (CTE) as well as the moderate wear resistance. Here, reinforcing by ceramic phases offering high E and low CTE as well as increased wear resistance is beneficial, and Al based composites have therefore found considerable interest very early in the 1970s [3]. Unless also high strength is a definite requirement, particle reinforcement is well suited, such materials being easier to manufacture than fiber reinforced grades, for which even distribution and retaining of undamaged reinforcing phases are difficult to attain [4, 5, 6].

Powder metallurgy is inherently well suited for manufacturing of discontinuously reinforced metals [7, 8] since the tendency to segregation and demixing, resulting from density differences and poor wetting, that causes severe problems in ingot metallurgy routes does not emerge here. Among the different PM production routes feasible, extrusion of...
compacts of mixed powder is particularly well suited, since the oxide layers covering the Al particles are destroyed due to the high shear forces, resulting in excellent interface bonding [9], and the intense deformation also results in virtually complete embedding of the reinforcing particles in the matrix, at least if the hard phase content is not higher than 25 vol.% [10, 11].

In the present work, Al-Al$_2$O$_3$ composites produced from powder mixes by extrusion have been studied. Al$_2$O$_3$ is less attractive than SiC if low CTE is aimed at, but since fused alumina powder is a mass product available in different grades, cheap starting materials can be used, and the high hardness of the alumina powder can be expected to result in significantly increased wear resistance of the composites.

**EXPERIMENTAL PROCEDURES**

As starting materials, plain Al powder Ecka AS-011S (< 45 µm, < 0.4 wt.% oxygen), supplied by Ecka Granulate GmbH, Velden, Germany, and a fused alumina grade ZESK (containing 3% Ti) from Treibacher Schleifmittel GmbH, Villach, Austria, were used, the latter being available in a fine and a coarse fraction with $d_{50}$ = 22.5 µm and 50 µm, respectively. As visible in Fig.1, the finer fraction exhibited a slightly more rounded shape than the coarser one, which is a consequence of the crushing and milling treatment the powders were subjected to. The content of the hard phase in the powder mixes was varied between 20 and 50 wt.%, i.e. 14.5 to 40.3 vol.%. 

![a) fine ($d_{50} = 22.5$ µm) b) coarse ($d_{50} = 50$ µm)](image)

Fig.1. Fused alumina powder (Treibacher grade ZESK).

The powders were dry mixed for 25 min in a tumbling mixer and cold compacted at 600 MPa in a pressing tool to form cylindrical billets with 40 mm diameter and about 60 mm length. The billets were wrapped in Al foil, preheated for 60 min at 500°C in air and extruded to cylindrical rods with 10 mm diameter, i.e. at an extrusion ratio of 16, using a custom-built 2000 kN (200 ton) horizontal extrusion press and a 180° die, the ram speed being 10 mm/s. Lubrication was afforded by a graphite-oil suspension. The forces required for extrusion were in the range 800 to 1200 kN, i.e. well below the capacity of the extrusion press.

The extruded bars had a smooth surface, those with alumina contents >30 wt% exhibited slightly higher surface roughness. All bars were free from externally visible defects such as cracks etc., with the exception of the end sections which were cut off. Metallographic longitudinal and cross sections were prepared from all materials, the embedded specimens being wet ground with SiC paper and then diamond polished at 7, 3 and 1 µm grit. Round tensile test bars 6 x 30 after DIN 50125 with a gage length of 30 mm were machined from the as-extruded specimens, machining of these particle reinforced
materials being surprisingly easier than of plain Al reference bars. Tensile testing was done on a Zwick 1474 universal testing machine with a clip-on extensometer, the Young’s modulus being measured in a loading-unloading cycle during the tensile test.

Compared to standard mechanical testing, wear testing is a procedure that suffers from the fact that there is no “wear resistance” as a material property as there is a “tensile strength”. Wear is a system property, depending on the wear partners as well as on the environment, and therefore, principal tests such as the pin-on-disc test done in the present work are suitable to generate a ranking of different materials, but the results are significant only for wear loading cases that are similar to those in the test. Nevertheless, pin-on-disc testing is a well established procedure in wear testing, and hardened ball bearing steels are very common as counter material.

![Fig.2. Pin-on-disc tester (schematic).](image)

The wear behaviour was studied using a pin-on-disc tester (see Fig.2) in dry run against discs made of ball bearing steel 100Cr6 (AISI 52100) heat treated to 63 ± 1 HRC. Cylindrical specimens of 40 mm length were cut from the extruded bars, carefully turned plane at one end and inserted into the specimen holder of the tester so that the turned end face of the specimens was as parallel to the disc as possible. However, absolutely parallel fit is not possible, and therefore some run-in period was to be expected during which the faces were re-shaped to contact each other at the complete cross section of the pin. To ensure that the run-in period was safely exceeded and stable wear conditions had been reached, the tests were carried out up to a sliding distance of typically > 20 km. The mass loss was determined by weighing the specimens in regular intervals, and the cumulative mass loss was plotted as a function of the sliding distance. In all cases, after some run-in period well defined linear wear graphs were obtained. Therefore, from the slope of the graph and the density the wear coefficient k was calculated for each test according to

\[ k = \frac{\Delta V}{F \cdot s} = \frac{\Delta m}{\rho \cdot F \cdot s} \text{ [mm}^3\text{N}^{-1}\text{m}^{-1}] \]  

\[ \Delta m \] being the mass loss, \( \rho \) the density, \( F \) the normal force and \( s \) the sliding distance. In parallel, the friction force \( F_f \) was continuously recorded using a sheet of spring steel equipped with strain gages at both faces, and the average friction coefficient \( \mu \) was calculated from the friction force and the normal load \( F_N \) according to

\[ \mu = \frac{F_f}{F_N} \]
MICROSTRUCTURES

Typical cross sections of the extruded specimens are shown in Fig.3. The specimens appear fully dense, and the distribution of the oxide particles in the Al matrix is very uniform, hardly any pronounced clustering of alumina except some statistical variation of the oxide density, being observed. This holds also for the specimens with higher oxide content. Furthermore, there is virtually no texture of the microstructure in the longitudinal direction, as indicated by Fig.4a. This confirms that the very simple and straightforward method of dry mixing, compacting and extrusion is well suited to produce uniform Al-Al$_2$O$_3$ composites.

![Metallographic cross sections of extruded Al-Al$_2$O$_3$ specimens.](image)
30 wt.% (22.8 vol.%) $\text{Al}_2\text{O}_3$ 22.5 µm

30 wt.% (22.8 vol.%) $\text{Al}_2\text{O}_3$ 50 µm

40 wt.% (31.5 vol.%) $\text{Al}_2\text{O}_3$ 50 µm

Fig. 4. Metallographic longitudinal sections of extruded Al-$\text{Al}_2\text{O}_3$ composites (extrusion direction parallel to longer axis of image).

MECHANICAL PROPERTIES

The mechanical properties are summarized in Table 1 and graphically shown in Fig. 5 as a function of the alumina content. As can be seen, the tensile strength is slightly increased by addition of the hard phase up to about 30 wt.% / 22.8 vol.% and then drops, slightly in the case of the fine alumina grade but very pronouncedly between 40 and 50 wt.% for the coarser one. A similar trend is observed with the 0.2% yield strength, though here the differences between the alumina fractions are still more pronounced: for the fine fraction, the values increase consistently right up to the maximum alumina content while...
for the coarser fraction a pronounced drop is observed between 40 and 50 wt.%. Apparently the larger oxide-oxide contacts, which, as mentioned above, act as defects, have a decidedly more adverse effect than the smaller ones in the case of the finer alumina, which is also indicated by the markedly larger scatter of the properties. Furthermore, it should not be ignored that there is no chemical bonding between the metal matrix and the reinforcing phase, which enhances separation already at low deformation rates. This can also clearly be seen from fracture surfaces (Fig.6): here the very large oxide surfaces in the case of the coarser alumina stand out quite clearly while the finer dimple structure in the case of the fine alumina fraction indicates significantly more microductile fracture.

Fig.5. Mechanical properties of extruded Al-Al₂O₃ composites.
Tab.1. Mechanical properties of extruded Al-Al$_2$O$_3$ composites; properties measured parallel to the extrusion direction.

<table>
<thead>
<tr>
<th>Alumina fraction [µm]</th>
<th>Alumina content [wt.%]</th>
<th>Alumina content [vol.%]</th>
<th>Tensile strength [MPa]</th>
<th>Tensile yield strength $R_{0.2}$ [MPa]</th>
<th>Elongation A5 [%]</th>
<th>Young’s modulus [GPa]</th>
</tr>
</thead>
<tbody>
<tr>
<td>22.5</td>
<td>20</td>
<td>14.5</td>
<td>145 ± 0</td>
<td>114 ± 1</td>
<td>10.0 ± 1.1</td>
<td>83 ± 6</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>18.4</td>
<td>144 ± 1</td>
<td>98 ± 0</td>
<td>13.2 ± 0.5</td>
<td>92 ± 1</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>22.5</td>
<td>153 ± 1</td>
<td>115 ± 2</td>
<td>7.9 ± 1.1</td>
<td>105 ± 4</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>31.1</td>
<td>157 ± 4</td>
<td>131 ± 0</td>
<td>2.5 ± 0.4</td>
<td>110 ± 3</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>40.3</td>
<td>150 ± 3</td>
<td>147 ± 1</td>
<td>0.8 ± 0.5</td>
<td>110 ± 3</td>
</tr>
<tr>
<td>50</td>
<td>20</td>
<td>14.5</td>
<td>139 ± 6</td>
<td>110 ± 2</td>
<td>7.9 ± 0.1</td>
<td>82 ± 5</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>18.4</td>
<td>138 ± 3</td>
<td>103 ± 5</td>
<td>10.4 ± 2.5</td>
<td>93 ± 1</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>22.5</td>
<td>147 ± 1</td>
<td>113 ± 1</td>
<td>7.7 ± 1.2</td>
<td>105 ± 2</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>31.1</td>
<td>142 ± 2</td>
<td>125 ± 6</td>
<td>5.1 ± 2.5</td>
<td>103 ± 7</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>40.3</td>
<td>50 ± 15</td>
<td>50 ± 15</td>
<td>0.2 ± 0.1</td>
<td>96 ± 4</td>
</tr>
</tbody>
</table>

However, it must be remembered that microductility is not necessarily macroductility, and this stands out quite clearly if the elongation to fracture is regarded: as indicated in Fig.5c, the elongation drops with increasing alumina content, as might be expected, but there is no significant difference between materials containing coarse and fine alumina particles, respectively. This can be explained by the fact that the fine dimples generated around the finer alumina particles produce little total deformation. The coarse particles of the larger fraction cause earlier presence of defects of critical size that cause unstable crack propagation.

The Young’s modulus, for which plastic deformation does not play a role, enables differentiating between the metal-oxide interfaces and the oxide-oxide contacts. The E-moduli are plotted against the oxide content in Fig 5d; the broken line indicates the theoretical graph calculated after the inverse rule of mixture [2]. Here it stands out clearly that E increases significantly with higher oxide content up to 30 wt.% alumina; from this level the increase is diminished for the finer alumina or even is reversed to a decrease for the coarser hard phase. This indicates that the oxide-oxide contacts tend to become significant above 30 wt.%, but their adverse effect is more pronounced in the case of coarser reinforcing phase, which can be attributed to the higher probability of oxide-oxide contacts without bonding with Al, which of course are the larger the coarser the alumina is.

The hardness, finally, shows a different trend: here the values consistently increase with higher oxide content, which is quite understandable since Vickers hardness testing means applying compressive loads, and in this case oxide-oxide contacts are no more defects, the hard alumina particles supporting one another. Also here, however, the finer oxide particles tend to result in higher hardness levels above 30 wt.% alumina content, which indicates that the formation of metal-free oxide-oxide contacts is not without effect on the hardness either.
Fig. 6. Tensile fracture surfaces of extruded Al-Al$_2$O$_3$ composites.
DRY SLIDING WEAR BEHAVIOUR

Effect of the test parameters

A critical feature of wear testing is the reproducibility. Therefore, as a first approach, parallel tests on the same material were carried out. As indicated in Fig.7a, the wear graphs of the parallel runs are in excellent agreement within ± 0.5 mg mass loss, which confirms that the reproducibility of the test can be regarded as satisfactory. Typically, there is a run-in period with significantly higher wear rate, and only after about 6 km stable wear regime is attained that enables plotting a linear relationship between mass loss and sliding distance. This underlines that in pin-on-disc testing involving mating flat faces, sufficiently long sliding distances are essential to obtain reliable data for the wear coefficient; otherwise, too high values for the wear coefficient are obtained and, since the run-in period is prone to be statistically determined, also a large scatter of the k values is encountered. The drop of the k value with extended testing is clearly visible from Fig.7b; it is evident that too short test runs will result in grossly overestimated wear coefficients (by a factor of 7±1). The friction coefficient, in contrast, does not vary too much; there is only a slight decrease both of µ\textsubscript{max} and µ\textsubscript{average} with progressive testing, indicating that the run-in period affects the material loss but not so much the transfer of load.

![Fig.7a. Wear graph (mass loss vs. wear track length) of Al-20 wt.% Al\textsubscript{2}O\textsubscript{3} (22.5 µm); load: 13 N; sliding velocity: 0.52 m·s\textsuperscript{-1}, parallel runs.](image1)

![Fig.7b. Friction coefficient µ and wear coefficient k as a function of the sliding distance. Al-20 wt.% Al\textsubscript{2}O\textsubscript{3} (22.5 µm); load: 13 N; sliding velocity: 0.52 m·s\textsuperscript{-1}.](image2)

Aluminium is a relatively soft and low-melting matrix. Therefore it could be assumed that thermal effects might play a major role, resulting in softening of the matrix and loss of stability, the hard phases simply flowing off with the matrix. Thermal effects are strongly linked to the test parameters, i.e. the normal force and the sliding speed. Therefore, test runs were carried out at a constant sliding speed of 0.52 m·s\textsuperscript{-1} but with widely varying normal force of 10 - 50 N, which corresponds to a pressure of 0.17 to 0.76 MPa. The wear graphs obtained are given in Fig.8: here it is clearly visible that at low to moderate normal loads, the wear coefficient in the stable regime does not vary significantly, only the run-in period is more pronounced. Only at a load of 60 N the wear rate dramatically increases, as is also visible from Fig.8b, in which the wear coefficient is plotted as a function of the normal load. Here the “jump” between 50 and 60 N, from k = about 3 to 36·10\textsuperscript{-6} mm\textsuperscript{3}/N·m is clearly evident for the composites with the smaller particles. This indicates that there is a transition from mild to severe wear as a function of thermal overloading of the matrix. Once more, the effect on the friction coefficient was much less pronounced; it slightly
increases at higher load, but the scatter of the values, as a consequence of stick-slip effects, is so large that it would be unjustified to define more than a trend (Fig.9).

Fig.8. Wear graphs and wear coefficient of Al-20 wt.% Al₂O₃ (50 µm) as a function of the normal load. Sliding velocity 0.52 m·s⁻¹.

Fig.9. Friction coefficient of Al-20 wt.% Al₂O₃ (50 µm); sliding velocity: 0.52 m·s⁻¹.

Similar experiments were carried out at constant load but with varying sliding speed: here, however, it was found that at 40 N normal load, there was not really a significant difference between 0.52 and 1.55 m·s⁻¹. Apparently for the transition to severe wear, higher speeds would be necessary – speeds that were not feasible with the tester, due to excessive vibrations - , which indicates that the dry sliding wear behaviour of the materials tested here is more sensitive to the normal load than to the sliding speed. It was however observed that the normal load threshold from mild to severe wear was lower at higher sliding speed: in the case of 1.55 m/s, already at 50 N normal load quite severe wear occurred.

Effect of the hard phase content

In order to study the effect of the hard phase content on the stable wear behaviour, medium wear loading conditions were selected, i.e. a normal load of 30 N and a sliding speed of 1.55 m/s. Selection of this speed enabled attaining sliding distances of up to 48 km within acceptable periods while avoiding the vibration problems encountered at still higher speeds.

In Fig.10, wear graphs for Al-Al₂O₃ with varying hard phase content are given for two alumina fractions. It is clearly evident that in both cases there is a consistent drop of the wear rate with higher alumina content. (Measurements with plain Al reference materials were not possible since in that case, severe adhesive wear resulted in seizure and
mechanical locking of the tester). This indicates that, similarly to the hardness measurement, also in sliding wear loading the oxide-oxide contacts, which in the case of tensile loading act as defects, do not play a detrimental role here, and also the comparatively weak metal-oxide interfaces are not detrimental.

Mass loss graphs Al-x% Al₂O₃ 22.5 µm. Mass loss graphs Al-x% Al₂O₃ 50 µm

Fig.10. Wear graphs of Al-Al₂O₃ with varying alumina contents. Load: 30 N (= pressure: 0.38 MPa), sliding velocity: 1.55 m·s⁻¹.

If the wear coefficients of both material types are compared (Table 2), it is even visible that here the composites that contain the coarser alumina particles exhibits slightly better wear resistance. The reason might be that the larger, very angular alumina particles are better bonded geometrically than the more rounded finer particles. This is in agreement with studies on particle reinforced sintered aluminium [12], which had shown that coarser hard particles result in better wear resistance since they are less easily extracted from the matrix during wear loading.

Tab.2. Wear and friction coefficients (stable wear regime) of extruded Al-Al₂O₃ in dry run against bearing steel 100Cr6 (63 HRC). Load: 30 N, sliding velocity: 1.55 m·s⁻¹.

<table>
<thead>
<tr>
<th>Alumina fraction [µm]</th>
<th>Alumina content [wt.%]</th>
<th>Alumina content [vol.%]</th>
<th>Wear coefficient k [10⁻⁶ mm³/N·m]</th>
<th>Max. friction coefficient µmax</th>
<th>Average friction coeff. µaverage</th>
</tr>
</thead>
<tbody>
<tr>
<td>22.5</td>
<td>20</td>
<td>14.5</td>
<td>4.6</td>
<td>1.12</td>
<td>0.94</td>
</tr>
<tr>
<td></td>
<td>25</td>
<td>18.4</td>
<td>3.9</td>
<td>1.39</td>
<td>1.18</td>
</tr>
<tr>
<td></td>
<td>30</td>
<td>22.5</td>
<td>3.1</td>
<td>1.58</td>
<td>1.31</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>31.1</td>
<td>2.2</td>
<td>0.93</td>
<td>0.78</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>40.3</td>
<td>1.6</td>
<td>0.94</td>
<td>0.81</td>
</tr>
</tbody>
</table>

|                       | 50                     | 20                      | 14.5                           | 4.0                         | 1.45                          | 1.10                          |
|                       |                        | 25                      | 18.4                           | 3.5                         | 1.31                          | 1.09                          |
|                       |                        | 30                      | 22.5                           | 2.9                         | 1.43                          | 1.18                          |
|                       |                        | 40                      | 31.1                           | 1.6                         | 1.40                          | 1.16                          |
|                       |                        | 50                      | 40.3                           | 1.0                         | 1.00                          | 0.85                          |
This hypothesis is supported by the appearance of the worn surfaces (Fig.11). Here it can be seen that the material containing coarse alumina exhibits flat surfaces of alumina particles that slightly protrude from the matrix. On the worn surface of the other material, in contrast, hollow tracks are found that seem to have been generated by reinforcement particles that have been torn out of the matrix and have been pulled across the surface, resulting in three-body abrasion. Also the alumina particles visible in the SEM image do not seem to be strongly embedded in the matrix. On the other hand, this observation should not be overestimated considering the relatively small differences in mass loss between the materials reinforced with different alumina fractions.

![Al2O3 22.5 μm](image1)
![Al2O3 50 μm](image2)

Fig.11. Worn surfaces of Al-25 wt.% Al2O3. Load: 30 N, sliding velocity: 1.55 m·s⁻¹.

The friction coefficients exhibited a somewhat irregular behaviour (Table 2), in particular for the finer alumina fraction with a maximum at 30 vol.%; however a trend to lower $\mu$ values with increasing hard phase content was discernible for both alumina fractions. If the friction coefficient is plotted as a function of the wear coefficient, it is evident that with decreasing wear (i.e. with increasing alumina content) the friction coefficient first slightly increases, but between 30 and 40 wt.% alumina there is more or less a break in the graph (Fig.12), higher alumina contents resulting in lower $\mu$ which indicates that the relatively hard and inert alumina inhibits adhesive effects if contained above a given volume content level.
Fig. 12. Relationship between wear coefficient and friction coefficient (average and maximum) in dry sliding of extruded Al-Al$_2$O$_3$ with varying oxide content (mass %) and particle size.

CONCLUSIONS

It has been shown that Al-base metal matrix composites reinforced with fused alumina can be easily and economically manufactured from commercial starting powders by dry blending, cold compacting and then hot extrusion with an extrusion ratio of 16. Fully dense, defect free bars with homogeneous, virtually texture-free microstructure could be obtained with both the coarse (50 µm) and the fine (22.5 µm) alumina grade.

The mechanical properties tensile and yield strength as well as Young’s modulus E increased within higher alumina content up to a maximum at 30 wt.% / 22.8 vol.% and then slightly increase further or dropped, the drop of E being markedly pronounced in the case of the coarser alumina. The elongation consistently dropped with higher hard phase content while the hardness consistently increased; in both cases the alumina particle size did not play a major role.

Dry sliding wear tests performed on a pin-on-disc tester against ball bearing steel showed that there is a critical combination of normal force and sliding speed at which there is an abrupt transition from mild to severe wear, which however does not affect the friction coefficient. The normal force threshold is lower at higher sliding speed, but in general the effect of the normal force is markedly more pronounced than that of the sliding speed. Increasing content of alumina results in consistently lower wear rates; here, coarse alumina is slightly more effective than finer one, apparently due to better geometrical fixture within the metal matrix. In any case, wear testing has to be carried out for sufficiently long sliding distance to attain stable wear conditions; otherwise, incorrect wear data will be obtained.
Acknowledgement

The authors wish to thank Ecka Granulate GmbH, Velden, Germany, and Treibacher Schleifmittel GmbH, Villach, Austria, for supplying the starting powders used as well as AMAG, Ranshofen, Austria, for funding this research work.

REFERENCES

[12] www.amc-mmc.co.uk