Wear Resistance of Agglomerated and Dispersed Solid Lubricants in Aluminium

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Aluminium matrix composites containing up to 5% of solid lubricants, namely carbon black and molybdenum disulfide, were fabricated. The composites were either mixed of milled followed by cold compaction of the powders. Some of the billets were degassed in vacuum. Cold extrusion was achieved with an extrusion ratio and velocity of 25:1 and 0.5 mm/s, respectively. Micro-hardness of the loose *granulates* and Vicker's hardness and tensile tests were made for the *extrudate*. Wear test under the dry condition was carried out at constant sliding velocity and contact pressure of 1.54 m/s and 1 N/mm².

Degassing the billets, that have been dispersed with solid lubricant, is necessary to give products having constant hardness when annealed at 673 and 813 K for different times. A superior wear resistance of composites manufactured by agglomerating the solid lubricant and in the as-extruded condition is noticed. The wear resistance of Al-3 mass% C composites is higher than the other while the Al-5 mass% MoS₂ have the lowest wear rate for the agglomerated solid lubricant and in the as-extruded condition.

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I. Introduction

It was suggested and attempted some time ago⁽¹⁾⁽²⁾ to strengthen the relatively soft and rather weak aluminium metal by dispersion hardening. This development lead to SAP⁽³⁾⁻⁽⁵⁾, a material, in which very fine Al₂O₃ particles are embedded in an Al-matrix as a dispersoid. But SAP could not penetrate the commercial market due to relatively complicated production technology requiring controlled oxidation of the Al powder, hot working and milling of the powder, followed by extrusion.

A good bearing material should have good resistance to seizure and wear resistance as well as good compressive strength, fatigue strength and thermal conductivity⁽⁶⁾. Aluminium-base bearing alloys generally have good fatigue strength, yield strength, corrosion resistance and thermal conductivity. However, most of these alloys tend to seize when run together or against another alloy in the absence of fluid lubrication. Seizure or galling can be produced by suitable alloy addition. Seizure can effective-

ly be eliminated by a dispersion of a sufficient volume of graphite or molybdenum disulfide particles in the matrix of an aluminium alloy.

A number of materials mostly inorganic, are used as lubricants in particulate form. They are dusted dry on the surface to be lubricated or are dispersed in easily evaporated liquids or gases, in lubricating liquids, in greases, or in composite mixtures. These materials include graphite, molybdenum disulfide, talc, mica, vermiculite, teflon (PTFE) and other inorganic salts⁽⁷⁾. In general, they have lubricating abilities because of their layered lattice structure and they are usually anisotropic. Their structure allows them to be easily sheared, and therefore they lower the friction when placed between sliding metal surfaces and provide a shear plane of lower strength than those of the two sliding surfaces.

The object of this work is to determine the wear resistance of mixed (embedded) and milled (dispersed) graphite and molybdenum disulfide and mixture of them in aluminium for the densified billets by extrusion. These materials have shown recently considerable interest for aerospace applications. This is because of high specific strength, modulus and the potential low cost of the composites.

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II. Experimental

The composites constituents are listed in Table 1. They were either mixed for 0.5 h (1.8 ks) or milled for different times according to the composition, in a Planetary Ball Milling Mixer to have good dispersion. The powders were cold compacted in a floating die-set to 70% of the theoretical density. Some of the compacted billets were degassed in vacuum for 3 h (10.8 ks) at 723 K and the others were kept without degassing. Cold extrusion of billets was achieved using 12.5 MN vertical press. The extrusion velocity was 0.5 mm/s. The billets length and diameter were 100 and 60 mm, respectively. The extrusion ratio was 25:1 and the extrusion die has an angle of 180°.

Micro-hardness test was done for the loose powder after milling. Vicker's hardness test was performed on the extrudate after annealing at 673 and 813 K for 1, 3, 6, 24 and 72 h holding time at temperature. Tensile test was also carried out to some specimens for the extrudate and after annealing 6 h at 813 K.

Wear tests under dry condition were carried out on pin-on-disc type testing machine as de-

scribed by Hohmann⁽⁸⁾. The wear pins (composite) are 6 mm in diameter and 15 mm in length. The contact pressure and the sliding velocity were kept constant at 1 N/mm² and 1.54 m/s, respectively. The wear discs were made of cold worked tool steel (105 MnCr 4) and have hardness of 60–62 H_RC. The weight-loss due to the wear of the pins were measured on an electric balance with an accuracy of ±0.1 mg.

Scanning electron microscope (SEM) and X-ray diffraction were used to analyze the wear surfaces.

III. Results and Discussion

1. Blending the powders and the hardness of the granulates

It was possible to admix carbon black powder or molybdenum disulfide powder or mixture of them without any problem with aluminium powder. The uniform distribution of the dispersoid was accomplished during milling in a planetary ball milling mixer because the charge is constantly comminuted and rewelded. The initial Al-powder of a particle

Table 1 Composition, manufacturing conditions and extrusion pressure for the composites.

Material No.	Composition (mass%)	Mixing conditions	Milling time (h)	Degassing conditions T(K)	t(h)	Atm.*	Extrusion pressure (N/mm ²)
0	Al			823	3	V	820
1	Al+1C	Milled	8	823	3	V	1185
2	Al+2C	Milled	12–16	823	3	V	1414
3	Al+2C	Milled	12–16				1945
4	Al+2C	Mixed	0.5	823	3	V	1298
5	Al+3C	Milled	24	823	3	V	1768
6	Al+2MoS ₂	Milled	8	823	3	V	1150
7	Al+2MoS ₂	Milled	8				1768
8	Al+2MoS ₂	Mixed	0.5	823	3	V	1050
9	Al+5MoS ₂	Milled	18–24	823	3	V	1602
10	Al+3C+2MoS ₂	Milled	36	823	3	V	very high
11	Al+3C+2MoS ₂	Mixed	0.5	823	3	V	1121
12	Al+10Al ₂ O ₃	Milled	8	823	3	V	1390
13	Al+10Al ₂ O ₃	Milled	8				1945

* V=Vacuum of 10⁻⁴ Pa (10⁻⁶ Torr)

size 35 μm was first comminuted, but becomes coarser again with increasing milling periods. As soon as the solid lubricant particles has been completely milled into the charge, the fine particles disappear completely (i.e. full dispersion). With continued milling the particle size of the charge increases further. Increasing the dispersoid fraction leads to increase the milling time to obtain full dispersion strengthening, Table 1. Milling was stopped as soon as the granulates have reached 2–3 mm in size. Micro-hardness of the granulates after milling and agglomeration revealed that increasing the carbon fraction tends to increase the hardness, Table 2.

2. Effect of the composite material on the extrusion pressure

It can be seen from Table 1 for the blended and degassed powders that increasing the second-phase fraction (carbon or molybdenum disulfide) leads to an increase in the extrusion pressure. The extrusion pressure for the degassed powders is much lower than that for the powders extruded without degassing as seen for the specimens No. 2, 3 and 6, 7. This can

be attributed to the thermal softening of the powders during degassing at 723 K for 3 h in vacuum. The condition of blending the powder mixtures has a great effect on the extrusion pressure. When the powders mixture mixed for 0.5 h, the mixture required lower extrusion pressure than that required for the milled powders (granulates). This is because the second-phase (C or MoS_2) agglomerated on the aluminium particles surface. These agglomerates facilitate the extrusion process by reducing the friction between aluminium particles (for the mixed powders). While in the case of dispersing solid lubricant, the granulates were very hard and therefore have low formability and required high pressure for deformation.

3. Evaluation of the composite mechanical properties

(1) Hardness measurements

The micro-hardness of the dispersed granulates after milling the powders mixtures increased with increasing the second-phase fraction. Granulates of Al-3C have the highest hardness value. After extrusion, pure

Table 2 Vicker's hardness measurements for the composites after annealing for different times.

Material No.	$H_V0.025$ for loose granulates	H_V10 for rods	H_V10 after annealing at 673 K for $t(\text{h})$					H_V10 after annealing at 813 K for $t(\text{h})$				
			1	3	6	24	72	1	3	6	24	72
0		53	47	44.7	44.3	42.1	40.7	32.2	30.1	30.1	28.7	29
1	60	63.3	61	61.9	61.9	62.1	63.6	61.9	60	60.3	58.9	59.2
2	80	74.1	75	74.2	73.9	74	74.2	73.6	72.8	74.2	70	71.4
3	80	87.4	81.5	81	80.6	82	83.6	70.7	71.4	69.4	68.3	63.7
4		52.4	44.3	44.8	44.8	44.2	43.9	43.6	44.3	44.6	43.3	44.7
5	109	96.8	99.3	98.7	99.5	99.7	100.3	102.9	100.4	102.7	99.3	100.3
6	81	60	58.7	58.7	58.4	58.5	58.7	56.6	56.1	55.7	53.8	54.3
7	81	89.3	67	64.3	61.3	61.3	61.3	56.1	55.2	55.2	53.8	53.3
8		54.3	49.1	49.5	49.8	49.5	49.4	45.3	44.6	43.1	42.6	43
9	88.7	69.6	68	67.3	67.2	67.2	67.3	67.4	68.8	66.1	64.3	66.1
10	90.1	109.8	110.5	110.4	109.1	110.4	111.7	113.1	113.1	114.5	113.1	115.3
11		53.3	50.3	50	50.3	50.2	50.3	50.7	53.3	54.3	54.7	57.4
12	82.9	69.8	67.7	67.3	66.4	67.2	68.6	65.4	65.1	64.8	63	63.1
13	82.9	84	65.5	60.8	64.2	63	64.6	63.1	62.2	61.9	63.7	60.8

aluminium (material No. 1) has the lowest hardness value in comparison with any composites. The composite hardness increased with increasing second-phase fraction as seen in Table 2. The composites that have not been degassed before extrusion showed higher hardness than those degassed. The hardness of the dispersed composites are higher than that for the composites manufactured by mixing the powders. Annealing at 673 or 813 K for different times has no effect on the hardness of the composites that have been milled and degassed before extrusion. This can be attributed to the fact that during degassing the dispersed phase absorbed the rest of oxygen in the specimen and oxidized to either MoO_3 or CO and CO_2 . During annealing, the surface of the specimens can be oxidized but the whole specimen has no more oxygen for oxidation. Moreover, it can be seen that carbon is more stable than MoS_2 when dispersed in aluminium and subjected to annealing up to 813 K for a long time. The hardness of the composites, which have been dispersed by carbon, remains constant after several annealing operations for different times whereas dispersing by MoS_2 led to great changes in the composite hardness when subjected to high temperature annealing.

(2) Tensile properties

The mechanical properties of aluminium hardened by dispersing carbon black or molybdenum disulfide are noteworthy. The results were compared with aluminium dispersed with 10 mass% Al_2O_3 . The results given in Table 3 show that dispersion of 3% C in

aluminium gives higher tensile strength than the dispersion of 5% MoS_2 and 10% Al_2O_3 .

Tests by X-ray diffraction proved that the carbon black transformed to aluminium carbide Al_4C_3 during milling and subsequent heat treatment. The superior tensile strength of Al+3C is attributed to the volume per cent of the dispersoid. The volume per cent of the dispersoid in aluminium are: 12–15 vol.% Al_4C_3 , 10 vol.% MoS_2 , 8 vol.% Al_2O_3 for Al-3C, Al-5 MoS_2 and Al-10 Al_2O_3 , respectively⁽¹¹⁾. Milling 3% C as a second-phase produces higher amount of dispersion (Al_4C_3) than that produced by any other composites under test. Therefore, the tensile strength and yield stress of Al-3C composites have the highest value. The ductility, as measured by the elongation and reduction of area per cent, of the composites have reasonable values. On the other hand, the mechanical properties of aluminium mixed for only 0.5 h with carbon black, molybdenum disulfide and aluminium oxide gave lower strength and higher ductility than aluminium hardened by dispersion. These materials represent the aggregate type of two-phase structure in which the size of the second-phase particles is of the order of the aluminium particle size⁽⁹⁾. In this case, each phase contributes a certain role to the overall properties of the aggregate. Many factors must be considered for a complete understanding of strengthening from second-phase particles. These factors include the size, shape, number, and distribution of the second-phase particles, the strength, ductility, and strain hardening behavior of the matrix and second-phase, the crystallographic fit between the phases, and the interfacial energy and interfacial bonding between the phases.

Tensile tests for the composites after annealing at 813 K for 6 h showed the characteristic properties for the dispersion hardened alloys, i.e. the second-phase particles inhibit softening processes such as recovery, recrystallization and grain growth. Aluminium hardened by carbon-black did not suffer any changes in the tensile properties after annealing⁽¹⁰⁾, Table 3. While dispersion by MoS_2 and Al_2O_3 lead to changes in the tensile properties after annealing but these decrease are not too high in com-

Table 3 Tensile test results for Composites with milled powders.

Material No.	Conditions	$\sigma_{\max}$ N/mm ²	$\sigma_{0.2}$ N/mm ²	Elong. %	R.A. %
5	as-extruded	357.5	317.0	6.78	11.42
	annealed*	359.5	297	6.40	11.90
9	as-extruded	266	236	7.25	9.86
	annealed*	208	167	7.38	15.20
12	as-extruded	250	231	6.64	23.20
	annealed*	211	170.5	12.13	26.64

* Annealing is performed at 813 K for 6 h.

Table 4 Tensile test results for composites with mixed powders.

Material No.	Conditions	$\sigma_{\max}$ N/mm ²	$\sigma_{0.2}$ N/mm ²	Elong. %	R.A. %
4	as-extruded	230	215	2.0	7.0
	annealed*	140	110	9.0	30.0
8	as-extruded	245	225	3.5	13.0
	annealed*	142	120	10.0	35.0
11	as-extruded	240	220	2.5	7.5
	annealed*	138	125	12.0	38.0

* Annealing is performed at 813 K for 6 h.

parison with the mixed second-phase with aluminium, Table 4. The later materials suffered a high changes in the tensile properties after annealing which represents that these materials are two-phase aggregate.

4. Wear resistance of the composites

The wear rate for Al-3C, Al-5MoS₂ and Al-3C2MoS₂ and manufactured by either mixing or milling the powders mixture and tested in the as-extruded condition and after annealing is shown in Figs. 1-3. Generally for all the composites, the specimens manufactured by mixing powders show better wear resistance than those manufactured by milling powders. This is because the function of milling powders is the dispersion hardening of the composite, i.e. making the solid lubricant particles very fine and distributing them inside the aluminium particles. The hardness and strength of the dispersed composites in the as-extruded and annealed conditions show higher values than

those for the mixed powders composites, Tables 2, 3 and 4. These finely distributed particles have no effect on the wear resistance of the composites either in the as-extruded or annealed states. On the other hand, mixing of powder gave the opportunity to the second-phase to agglomerate over the surface of the aluminium particles which hindered the complete welding of the particles. This agglomeration has small effect on the composite hardness and strength but has a great effect on the wear resistance as shown in Figs. 1-3.

Moreover, the specimens tested in the as-extruded condition have better wear resistance than those tested after annealing. This can be attributed to the following:

- (1) Oxidation of MoS₂ begins at 573 K and produces MoO₃ which is not a lubricant⁽¹³⁾.
- (2) Carbon suffers from oxidation at elevated temperature; the rate of oxidation is greater for finer powder. Very finely ground (600 m²/g surface area) graphite powder

Fig. 2 Wear resistance of Al-5MoS₂ composites.

Fig. 1 Wear resistance of Al-3C composites.

Fig. 3 Wear resistance of Al-3C2MoS₂ composites.

Fig. 4 The oxide layer over the composite pins.

begins to oxidize above 723 K with the formation of CO and CO₂, whereas coarse powder (3 m²/g) survives to 873 K⁽¹³⁾.

These can explain the high wear resistance of the as-extruded specimens over the annealed specimens (6 h at 813 K). Moreover, during the wear test, the temperature rise at the contact surface may lead to oxidation of carbon and molybdenum disulfide. This temperature rise led also to oxidize the abraded iron particles from the steel counterface disks. This oxide layer is shown in Fig. 4 and has a black color and can take either the form Fe₃O₄ or τ -Fe₂O₃. Micro-hardness measurement of the black oxide layer on the longitudinal section of the pins gave value of $492 \pm 8\%$ Hv0.015. Galan and Opravil⁽¹⁵⁾ found that the micro-hardness value of Fe₃O₄ = 500 and τ -Fe₂O₃ = 1000. Comparing these results with the present results proved that the black oxide layer covers the pins was Fe₃O₄. This is in a good agreement with the results of Zamzam⁽¹⁴⁾ who found that the temperature at the contact surface between the pin (specimen) and the steel counterface did not exceed 843 K.

In steady state wear, Al-5MoS₂ specimens have good wear resistance while Al-3C specimens have the highest wear rate for the composites produced by mixing powders and for only the as-extruded condition. Severe wear of Al-5MoS₂ and Al-3C2MoS₂ specimens occurred at 8 km and 38 km, respectively, while Al-3C specimens suffered steady state wear up to 60 km without severe wear

(failure). This can be attributed to the increase in temperature at the contact surface during the test which led to oxidize both C and MoS₂. The oxidation of MoS₂ started at 573 K while those for carbon started above 723 K. When oxidation took place, a severe wear starts and a failure of the specimens is noticed, i.e. abrupt change in the wear rate of the specimens.

IV. Conclusions

From the previous results and discussions, the following conclusions can be drawn:

- (1) Increasing solid lubricant fraction tends to increase the extrusion pressure.
- (2) Agglomerating of solid lubricants in aluminium require lower extrusion pressure than dispersion.
- (3) Dispersion of solid lubricants tends to increase the hardness of the loose granulates.
- (4) Degassing the billets, that have been dispersed with solid lubricants, is necessary to give products having constant hardness when annealed at 673 and 813 K for different times.
- (5) The tensile properties suffers small changes for the dispersoid solid lubricants when annealed while these changes increase for the agglomerated (mixed) one. The tensile strength are superior for the dispersed solid lubricants.
- (6) A superior wear resistance of composites manufactured by agglomerating solid lubricants and in the as-extruded condition is found.

(7) The wear resistance of Al-3 mass% C composites is higher than the other while the Al-5 mass% MoS₂ specimens have the lowest wear rate for the agglomerated solid lubricant and in the as-extruded conditions.

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