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Abstract

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Keywords

via, uniball, magneto-milling, uniaxial, hot, characterization, pressing, mechanical, properties, α -al₂o₃, particle, reinforced, aluminium, matrix, composites, synthesized

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Characterization and mechanical properties of α -Al₂O₃ particle reinforced aluminium matrix composites, synthesized via uniball magneto-milling and uniaxial hot pressing

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Abstract

Al-based composite powders containing 2, 4, 7 and 10 volume fraction of α -Al₂O₃ were prepared using the uniball controlled magneto-milling method and were then uniaxially hot pressed at (600±10)°C under 70MPa for 15 minutes. The resulting composites were greater than 99% theoretical density with enhanced mechanical properties. Detailed characterization was performed using: X-ray diffraction, scanning electron microscopy equipped with energy dispersive spectroscopy, electrical conductivity, compression, ultra-micro indentation testing and pin on drum wear testing at ambient temperature. Microstructure-mechanical property correlations were obtained as functions of α -Al₂O₃ volume fraction. It was found that controlled milling resulted in a uniform distribution of the hard α -Al₂O₃ particles within the Al, an acceleration of Al hardening and fracturing, and strain accumulation by the Al matrix. Hardness, strength, wear resistance and electrical resistivity of the monolithic products increased with increasing the volume fraction of α -Al₂O₃ up to 10vol.%. These were: HV=(1.84±0.26) GPa, maximum compressive strength=(845±33) MPa, compressive yield strength=(515±11) MPa. Outcomes were interpreted in light of the structural defects induced by milling, the presence of α -Al₂O₃, and dispersion of iron milling contaminants, with additional effects caused by oxygen introduced during the milling and/or the heat treatment.

Keywords: Al–Al₂O₃, Al-MMCs, uniball magneto-milling, metal matrix composites, mechanical alloying.

1. Introduction

Aluminium matrix composites (Al-MMCs or AMCs) have been developed and improved upon over the past century. They cover range of demands in aerospace, ground transportation, automotive, electronics and energy sectors [1–5]. Various reinforced phases in micro and nanoscale including; Al_2O_3 , AlN, SiC, TiC, B_4C , SiO_2 , BN, CuO, TiB_2 , graphene, graphite and intermetallics have been used to reinforce AMCs. These particulate phases create vital changes in the mechanical properties of the pure metals and their alloys [4]. Al_2O_3 is used as a reinforcement material in AMCs due to its low cost, stability at high temperatures, high oxidation resistance, and relatively high chemical stability and inertness [5]. Wear resistance and hardness of AMCs can be improved by Al_2O_3 [6–14]. It has been found that the breakup of Al_2O_3 into nanoparticles after longer milling times further increases the refinement process, resulting in more uniform dispersions. Al_2O_3 particles tend to enhance particle packing through the dissolution of the soft metal clusters and agglomerates during the milling process [15–23].

Several processing methods have been developed to synthesize and produce Al- Al_2O_3 micro and nano composites. These including solid state method or powder metallurgy and liquid base with stir casting methods. However, combinations of two or three methods were used for a high quality composite. In solid state milling processes, a process control agent (PCA), and cryo-milling Techniques were used to avoid problems associated with agglomeration and/or cold welding [22–47]. The homogeneous dispersion of hard particles within the Al matrix is considered one of the main problems encountered by researchers. AMCs still have some drawbacks including lower yield strength, lower strength and stiffness, poor wear and tear resistance [2,4]. Al possesses poor wear resistance which can be improved by addition of ceramic hard phase.

In the current investigation, we employed an advanced milling/mixing method, the uniball magneto-ball milling technique [48–50], in an attempt to prepare uniform precursor powders of Al+ Al_2O_3 , suitable for consolidation using uniaxial hot pressing. This technique involves relatively low energy milling with

control over ball-particle impact and particle shearing processes via positioning of external magnets. The method was selected because it can avoid the problem of cold welding of metal particles, common during high energy milling, and has also proved useful for synthesis of homogeneous dispersions of fine particles in a refined metal matrix with lower milling contaminations [48–50]. Our aims were (i) to produce Al+Al₂O₃ precursor powders with refined Al crystallite size and a uniform distribution of Al₂O₃ particles; (ii) to convert these composite powders to high density monolithic samples via uniaxial hot pressing; (iii) to determine optimum parameters for milling and densification, and (iv) to investigate the effects of the volume fraction of α -Al₂O₃ on the physical and the mechanical properties of the monolithic composites. With this in view, different volume fractions (2-10%) and particle size (200nm) were chosen. Fig.1 summarises the synthesis and characterisation approach used in purpose of this investigation.

2. Experimental

Aluminium fine powder of <180 μ m particle size and >91.5% purity was obtained from Sigma-Aldrich, and used as the base matrix. The α -Al₂O₃ starting powder (99.9% purity) of submicrometric particles size >200nm was obtained from Nanostructured and Amorphous Materials, Inc, Huston, USA. Stearic acid powder was used as process control agent and was obtained from Sigma-Aldrich Chemistry. The properties of the starting materials are shown in Table 1. The mechanical milling process was done using a magnetically controlled ball mill with a gentle low-energy shearing mode, which is induced by positioning magnet at the bottom of the rotating mill chamber to promote a relatively low-energy milling mode, where particle interactions are dominated by ball-particle shearing [48,49]. The milling parameters were listed in Table 2.

In an initial milling trail of Al+10 vol. % Al₂O₃ powder samples were taken after milling for 12, 24, 48, 72 and 120 hours. An optimum milling time of 55 hours was selected for 10g based on the results of X-ray diffraction and crystallite size calculation of composite powders. Different volume fractions 2, 4, 7 and 10 vol. % of α - Al₂O₃ were used for composites manufacturing for the purpose to study the effect of

reinforcement volume fraction on microstructure and mechanical properties. X-ray diffraction of milled powders was performed using a GBC diffractometer with Cu-K α radiation and graphite monochromator. The X-ray source operated 35kV and 26.8mA. The XRD outputs were analysed using TracesV6 software. Composite powders of Al-Al₂O₃ were consolidated under an argon atmosphere using uniaxial hot pressing (UHP) device with an induction heating. Fig. 2 shows the sample setup in the graphite die and hot pressing parameters. Compacts of (7 $\pm$ 0.05) mm diameter and more than (9 $\pm$ 0.05) mm length were produced.

The Archimedes density and relative density of the composite samples were measured using ASTM B962-15 [51]. The theoretical density was calculated using the rule of mixtures [52]. Metallographic preparation of compacts (i.e cutting, grinding and polishing) was performed using an automated Struers Accutom-50 sectioning device and Struers Tegramin-20 grinding and polishing system. Following this, Ion beam cleaning for 30 min was performed on mounted samples using a Leica EM RES101 ion milling system. Vickers hardness measurements were obtained using a Struers DuraScan-70 hardness testing machine, using 1kg load and diamond indenter. Hardness estimates were obtained by averaging results of 9 individual measurements and results plotted with standard deviation indicated. Field emission scanning electron microscopy (FSEM) was performed using a JEOL-JSM-7001F instrument equipped with 80 mm² XMax energy dispersive x-ray spectrometer with silicon drift detector. Electrical conductivity was measured using a TH2817B LCR device on samples with (7 $\pm$ 0.05) mm diameter and (3 $\pm$ 0.05) mm thickness. Prior to measurement, the surfaces of the samples were ground with 800 silicon carbide abrasive paper and cleaned with ethanol. Then, both surfaces are painted with high purity conductive silver paint and allowed to dry for 24 hours. An Instron testing device (Instron 5566) was used to perform compression tests on the cylindrical compacts of (3.4 $\pm$ 0.05) mm diameter and (8.5 $\pm$ 0.05) mm length (L/D=2.5) in accordance with standards [53,54]. The maximum displacement was kept close to 20% of the total specimen height and the load rate was 0.004mm/sec. The compressive yield strength was calculated using the 0.2% offset principle [55]. Ultra-micro indentation investigations were conducted

using UMIS-2000 device with a Berkovich diamond tip of 200nm radius manufactured by IBIS Fischer-Cripps laboratories, Pty Limited, Australia. The experiments were carried out using 25 indents of loading and unloading mode with a dwell time of 2 second and maximum load of 200mN [56]. Pin-on-drum abrasion wear testing was performed with the selected parameters shown in Table 3 and according to ASTM G 132-96 [57]. The wear rates were calculated in terms of volume loss. The worn surfaces of the composite samples were analyzed by low vacuum SEM using JEOL-6490 instrument.

3. Results and discussion

3.1 Starting materials

Figure 3 shows indexed XRD and corresponding field emission secondary electron micrographs (FSEM) obtained from the starting aluminium and α -Al₂O₃ powders. The fine Al particles shown in Fig.3b have irregular shape with sharp edges and the submicrometric α -Al₂O₃ particles shown in Fig.3d have irregular shape with rounded edges. Estimates of lattice strain and crystallite size after milling were based on the widths of the Al starting powder peaks.

3.2 X-ray diffraction analysis

Figure 4 shows XRD patterns obtained from Al+10%Al₂O₃ composite powders as a function of milling time. The main mechanisms operative during ball milling of the Al-Al₂O₃ system are believed to be a combination of agglomeration via cold welding and particle fracture. These mechanisms are believed operative during the early stages of the milling process when the alumina volume fraction was low [58]. XRD revealed only the Al and alumina peaks, with no evidence of additional peaks which might be associated with intermetallic phases arising from reaction during milling. With increasing milling times up to 120 hours the Al peaks decreased in intensity, increased in breadth, and shifted to lower diffraction angles.

The crystallite sizes and lattice strains were estimated using Williamson-Hall approximation based on the main 4 peaks of Al. The effect of milling time and alumina volume fraction on crystallite size and lattice strain were estimated. Measurements of alumina crystallite size could not be obtained because these peaks disappeared into the background as they broadened. Figure 5 a & b shows estimated crystallite size and lattice strain as functions of milling time. It was found that crystallite size decreases and lattice strain increases as the milling time increases up to 120 hours, consistent with increased energy absorption via defect and strain accumulation during collision with the balls [56].

Figure 6a shows the XRD patterns of Al-Al₂O₃ composites powders as a function of alumina volume fraction milled for 55 hours. Figure 6b shows the trends for the first Al (111) peak shifting and broadening affected by alumina volume fraction. The peak broadening is consistent with formation of a deformed nanostructure while peak shifting to lower angles is consistent with accumulation of strain within the Al-FCC lattice. The crystallite sizes and lattice strains calculations based on figure 6 are shown in figure 7. It was found that slight increases in crystallite sizes and slight decreases in lattice strain as the alumina volume fraction increases from 2 to 10 % at the same milling time 55 hours.

The results of figures 6 and 7 suggest that the presence of alumina enhances grain refinement during milling. This is likely due to increases in rate of defect accumulation due to interaction of the soft matrix with increasing the volume fraction of hard particles, resulting in increased strain and fracturing. In summary, the reduction of aluminium crystallite size with increasing alumina may attributed to; (i) higher dislocation densities and lattice defects formed by milling in the presence of increased amounts of hard phase, (ii) higher accumulated strain in the Al due to associated increases in FCC lattice imperfection.

3.3 Density, Vickers hardness and electrical conductivity measurements

The Archimedes density, theoretical density, Vickers hardness, resistivity and electrical conductivity of hot pressed Al-Al₂O₃ samples were measured. A summary of these results is presented in Fig.8. The theoretical density of the composites increases as the alumina volume fraction increases. This is because

the density of alumina is higher than that of the aluminium matrix. The measured Archimedes density is in the region of the theoretical density and gives an indication that a near full-density composite can be achieved using uniaxial hot pressing after the magneto ball milling process. This can be seen in Fig.8a where Al+4%vol. Al_2O_3 has no porosity and full density. Vickers hardness values (Fig.8b) of the UHP composites increased with alumina volume fraction. This might be attributed to; (i) a dispersion strengthening mechanism caused by the presence of submicrometric alumina particles, (ii) the high hardness of alumina compared to the Al matrix, and (iii) the commensurate increasing in lattice strain and dislocations densities with increases of milling time [59–62]. The increases in hardnesses values with particulate volume fraction is consistent with [62]. Figure 8c and d show the effect of alumina volume fraction on the electrical resistivity and conductivity respectively. The decrease in electrical conductivity as alumina volume fraction increases can be attributed to both the high dielectric properties of alumina particles and their high resistivity (alumina resistivity $<10^{14} \Omega\cdot\text{cm}$) [63]. The electrical conductivity decrease with alumina volume fraction can be attributed internal electron movement, where a higher volume fraction of particles results in increases in the number of scattering sites for conduction electrons.

3.4 Scanning electron microscopy

For particle reinforced composite materials it is vital to obtain a homogenous distribution with optimum dispersion of reinforcement in the matrix in order to obtain enhanced mechanical, electrical and thermal properties. Figures 9 and 10 show the FSEM backscattered and secondary electron images and associated EDS-mapping results obtained from of UHP samples of Al+2%vol. Al_2O_3 and Al+10%vol. Al_2O_3 respectively. The dark gray background is the aluminum matrix while the lighter areas represent the Al_2O_3 particles and agglomerates. The milling impurities from the balls and stainless steel vial comprise iron and chromium flakes and appear whitest in the backscattered images. The secondary and backscattered electron images show an objectively homogenous distribution of Al_2O_3 particles within the aluminium matrix. This is confirmed by the associated EDS mapping. EDS analysis of large areas indicated about 0.3wt. % Fe and Cr some of which is possibly dissolved within the Al grains. All the

samples have lower content of impurities compared to milling methods in literature [60] and as revealed by EDS mapping, this might be attributed to the milling with low energy shearing mode and using of stearic acid as process control agent during milling processes. The FSEM images show that no evidence of agglomeration of the hard phase and verification that milling reduces the reinforcement particle size which is believed to promote Al grain boundary pinning. The FSEM images shown that the aluminum matrix are smeared out with no clear porosity in the composites which may give an indication of the close full density composites measured previously.

The higher volume fraction of Al_2O_3 in aluminum matrix (10% Al_2O_3 as in Fig. 9) is associated with a finer grain microstructure in UHP samples. This is attributed to the fact that alumina hinders the grain growth by acting as barrier to grain boundaries movement [43,61]. It is reported that interface layers located between the reinforcement particles and the matrix have negative effect on the mechanical properties of the composites [61]. Figure 11 shows the high resolution backscattered images of both volume fraction of composites with no evidence of the reaction layer on the interfaces between the aluminium matrix and alumina particles. Image analysis using the ImageJ software of numbers of FSEM images was used for the purpose of estimating the reinforcement particle sizes of reinforcement after hot pressing. The calculations show that the average particle size of reinforcement particles is (160 ± 10) nm for Al+10%vol. Al_2O_3 . It is clear that alumina particle have been fractured slightly compared to the starting average particle size (200nm). This might be the fact that alumina is hard particles and the low energy processing method with low speed.

3.5 Uniaxial compression testing

Figure 12 shows compressive stress-strain curves result from compression tests for uniaxial hot pressed Al matrix and Al+ Al_2O_3 composites. The preliminary pre loading data (approximately 25-50 points) were eliminated due to offset errors associated with non-intimate contact during the initial stages of compression testing. This was done according to the procedure outlined in the literature [53,59]. It was

observed that the ultimate compressive stress increased as the alumina volume fraction increases and the compressive strain decreases. This may suggest that the dispersed alumina particles are useful in improving the ultimate compressive strength and modulus of elasticity of the composites compared to pure Al matrix.

The increasing in compressive strength of the Al+Al₂O₃ composites may be attributed to different phenomenon. These may include; particle grain refinements, dislocation density increments, the effect of Orowan strengthening mechanism, the load transfer from Al matrix to alumina, full density composites that may reduce the stress concentration regions [30,64,65]. The Young's moduli (E) of the composites and the matrix were calculated using the linear portion of the curves in Fig.12 and the results are summarized in Table 4. These results are compared with the elastic modulus of pure aluminium matrix manufactured by a similar processing method (UHP).

The observed ultimate compressive strength values of Al-10 % vol. Al₂O₃ composites in current work are higher than those reported in the literature. In previous publications, the maximum strength of the Al matrix reinforced with alumina nanoparticles composites produced via high energy milling and hot isostatic pressing was reported as 628MPa for the same volume fraction [6]. According to the results in Table 4, the ultimate compressive strength and yielding strength of the composites increased directly in proportional to the volume fraction of the reinforcement particles. The modulus values are lower than the theoretical values calculated by the rule of mixture. However, the trends are similar, showing an increase of moduli as the alumina volume fraction increases.

3.6 UltraMicro-Indentation hardness and modulus results (UMIS)

Table 5 shows the modulus of elasticity and microhardness results obtained from ultra-micro indentation testing using the UMIS device. These values are based on the statistical analysis of the average of 25 points. A sharp increase in the modulus of elasticity and hardness of the composites occurred as the alumina volume fraction increased. The microhardness test results are in agreement with the compression

test results and the Vickers hardness results (as reported in section 3.3). Figure 13 shows the load-displacement average curves for aluminium matrix and Al+Al₂O₃ composites. The elastic moduli of the aluminium matrix and the Al+Al₂O₃ composite samples were obtained from the load-displacement response using the indentation testing machine software.

3.7 Abrasion wear test (Pin on Drum)

The corrected abrasive wear value was calculated using ASTM standard [57] as weight loss and volume loss per unit wear path length versus load as shown in Fig.14 a & b respectively. Following the ASTM standard, each wear value is an average of three measurements taken from the wear tests. The wear resistance is seen as the inverse of the material loss due to wear; therefore, it is documented as the mass loss. There is a considerable decrease in mass loss and specific wear rate as a function of the volume fraction of the α -Al₂O₃ particles and applied load (Fig.14). The specific wear rate linearly increases with increasing load showing a direct connection between the wear rate and the load (Fig.14b). These results are consistent with abrasive wear phenomenon and the pin-on-drum wear testing standard. As the specific wear rate of composite samples declined as the volume fraction of α -Al₂O₃ increased suggesting that composites may be useful for applications where high-stress abrasion wear is problematic. It should also be noted that the wear resistance improves within the composite samples as the alumina volume fraction increases. This validates the theory that the addition of alumina hard particles has the potential to improve the wear properties of the aluminium matrix.

Figure 15 compares the worn surfaces of different composite samples and the aluminium matrix sample. The fracture surface of the pure aluminium is characterized by evidence of severe plastic deformation and smooth wear tracks while the composite samples have sharper wear tracks with less evidences of plastic deformation. This trend in behavior can also be attributed to the presence of hard particle and its volume fraction. The 10% volume fraction composite sample has less plastic deformation and sharper edges on wear tracks compared to the other composite samples. The greater surface damage detected in the Al

matrix, Al+ 2, 4, 7 and 10 vol. % Al_2O_3 , respectively, is consistent with the increase in specific wear rate as the Al_2O_3 content decreases. Long grooves along the sliding direction and dimple formation are observed in all samples. As the load increases and alumina fraction decreases, the width of the grooves and the size of the dimples increase. Irregular plastic flow lines can be seen, indicating the occurrence of extensive plastic deformation during wear. This indicates that the wear mechanism changes from mild to severe as the load increases. With low load, the small wear rate can be attributed to presence of a stable film of surface oxide that may gradually form due to frictional heat generated during sliding. At high loads, the test sample surface deforms plastically and fracture occurs.

Figure 16 shows the variation of the specific wear rate as a function of Vickers hardness of the Al+ Al_2O_3 composite and applied load with volume fraction of reinforcement. It can be seen that increases in hardness due to reinforcement volume fraction are associated with decreased in specific wear rate. In composite materials or multi-phase materials, there are various abrasive wear mechanisms. These include the equal pressure and phase wear mechanism or a combination of both of these or even an intermediate state between the two [66–68]. In the case of equal pressure, the softer component will be worn out quickly and cause the hard phase to protrude. During an equal phase wear mechanism, the pressure distribution on the harder phase will be several times higher than the softer one, depending on their hardness or wear resistance, and this leads to the same wear during all the phases.

4. Conclusions

1. Uniball magneto milling process was used successfully to produce composite precursor powders of Al- Al_2O_3 in the presence of stearic acid as process control agent.
2. Microstructural and XRD results revealed that Al particles fracturing and defect accumulation was improved during milling due to the presence of alumina.

3. Uniaxial hot pressing was performed to consolidate the composite powders of Al-Al₂O₃ into a dense monolithic cylindrical shape samples (<99% theoretical density) using 70MPa applied pressure and short processing time (15min).
4. Vickers hardness, ultimate compressive strength, maximum compressive strain, modulus of elasticity and ultra-micro indentation hardness increased with increasing the volume fraction of Al₂O₃ up to 10 vol.%. The high hardness values combined with good retention of compressive strength, HV=(1.84±0.26) GPa, compressive strength= (845±33) MPa for 10 vol.%. Al₂O₃ can be attributed to; (i) the homogenous and fine dispersion of α -Al₂O₃ in Al matrix, and (ii) the achievement of near full density hot pressed composites.
5. The composites of Al-Al₂O₃ samples show an improvement in the wear resistance with increasing the volume fraction of α -Al₂O₃ up to 10vol.%. The specific wear rate of Al-Al₂O₃ composites increases with the applied load showing a straight line relationship.

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Table 1:

Materials	Al	α -Al ₂ O ₃	Stearic acid
Density	2.7 g/cm ³	3.96 g/cm ³	0.847 g/cm ³
Purity	≥ 91.9 % (complexometric)	99.9%	99.99%
Particle size	Fine powder (150-180 μm)	200 nm	Powder
Melting point	660 °C	2045 °C	361 °C boiling point

Table 2:

Parameters	Value
Velocity	65 RPM
Atmosphere	Ar at 300 kPa
Ball to powder ratio	27:1
Milling media	Steel ball 25mm Ø
Magnet position	Base of cylindrical mill promotes shearing

Table 3:

Sample diameter	(6±0.1) mm
Applied load	20, 40, 60 N
Translation speed	0.04 m/s
Rotational speed	8.9 rpm
Sliding distance	6.02 meter
Abrasive material	150 grit garnet paper
Temperature and humidity	20 °C , 50%

Table 4:

Sample	Max. Compressive stress (MPa)	Compressive Yield Point (MPa)	Modulus of Elasticity (GPa)
Al-Matrix	484 $\pm$ 28	205 $\pm$ 20	12 $\pm$ 3
Al+2 vol.% Al ₂ O ₃	737 $\pm$ 33	385 $\pm$ 25	15 $\pm$ 2
Al+7 vol.% Al ₂ O ₃	815 $\pm$ 36	402 $\pm$ 35	16 $\pm$ 3
Al+10 vol.% Al ₂ O ₃	846 $\pm$ 33	515 $\pm$ 11	17 $\pm$ 2

Table 5:

Property / sample	Modulus of Elasticity (GPa)	Hardness GPa
Al	19 ± 2.58	0.23 ± 0.02
Al+2% vol. Al ₂ O ₃	31 ± 3.05	1.19 ± 0.07
Al+4% vol. Al ₂ O ₃	41 ± 3.65	1.27 ± 0.19
Al+7% vol. Al ₂ O ₃	87 ± 5.27	1.59 ± 0.10
Al+10% vol. Al ₂ O ₃	91 ± 9.03	1.84 ± 0.26

Figure 1

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